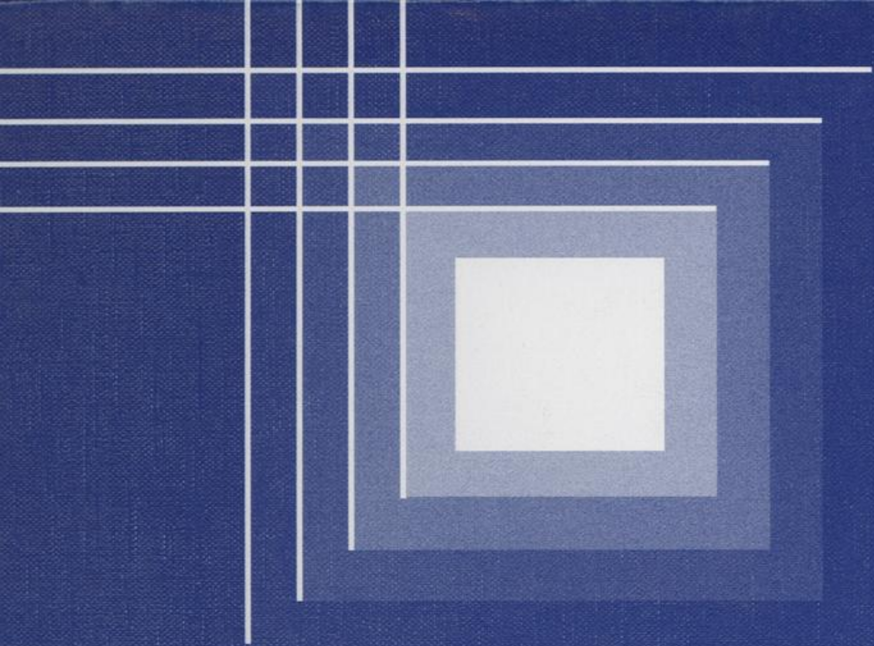

Electrical Insulating Materials

International Issues

Marcelo M. Hirschler, editor

STP 1376

STP 1376

Electrical Insulating Materials: International Issues

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Foreword

This publication, *Electrical Insulating Materials: International Issues,* contains papers presented at the symposium of the same name held in Seattle, Washington, on 15, March, 1999. The symposium was sponsored by ASTM committee D27 on Electrical Insulating Liquids and Gases and D09 on Electrical and Electronic Insulating Materials. The symposium Chairman was Marcelo M. Hirschler.

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Overview

The *ASTM Symposium on Electrical Insulating Materials: International Issues* (*1999*) took place in Seattle, WA, on March 15, 1999, and led to this STP. It was sponsored by Committee D09, on Electrical and Electronic Insulating Materials, in cooperation with Committee D27 on Electrical Insulating Liquids and Gases, to commemorate the 90th anniversary of the creation of Committee D09 (in 1909), which in turn was the source of Committee D27 fifty years later.

Electrotechnical products rely on electrical or electronic insulating materials to provide the protection required for the safety of people and the insulation of conductors from one another. Solid electrical insulations are often based on plastic materials, either thermoplastic, thermoset, or crosslinked, and they represent a major use of such materials. Electrical insulating liquids and gases are, however, also essential when used singularly or as combinations as electrical insulation or as an environment for electrical insulation. Standards have been issued, both in the United States and internationally, dealing with specifications for such materials, and with test methods for assessing a variety of properties for those materials. In particular, such standards may address a variety of safety issues which help to ensure the proper protection of the public. Moreover, international harmonization of electrical standards has profound effects on international trade and may present opportunities for export as well as removing barriers to trade. The sympsoium was intended to address all of these issues. In effect, it addressed five major types of issues: standards, properties of electrical insulating fluids, fire properties, and electrical issues.

Standards: The first section of this STP addresses writing standards and contains five papers. The first paper, by Kenneth Mathes, a winner of the prestigious Arnold Scott award and one of the most senior members of the committee, addresses the history of committee D09, and mentions the creation of committees D20, on plastics (in 1937), and of committee D27, on Electrical Insulating Liquids and Gases (in 1959). The history of ASTM D09 is associated with the rapid and increasing development, evolution, and complexity of the overall electrical industry, which depends on ASTM D09 for standard tests and specifications. This paper includes a list of those having received honors and awards from ASTM and from D09. David Baker discusses the present-day structure of committee D09, which now administers almost 150 standards, within 17 subcommittees. Interestingly, the 1999 membership of the committee barely exceeds the number of its standards. Joseph Kelly describes the activities of committee D27, which promotes knowledge pertaining to insulating liquids and gases, whether of synthetic or natural origin, and develops standards and specifications pertinent to these materials. The fluid materials covered include oils of petroleum origin, synthetic liquids, and halogenated and other gases used as electrical insulation in transformers and other electrical equipment. The paper gives examples of the work undertaken by the committee. Standards for electrical insulating materials are written by organizations outside of ASTM, both internationally and in the USA. International organizations include the International Electrotechnical Commission (IEC), while the National Electrical Manufacturers Association (NEMA) is a US trade association producing standards. Nicholas Maennling and Daniel Strachan, respectively, explain the process by which IEC TC89 (Technical Committee on Fire Hazard Testing) and NEMA develop standards and give an overview of the standards available (and under development) in 1999. IEC TC89 is a technical committee addressing the reduction of the risk of fire in electrotechnical products. It is a "horizontal" committee within the IEC, with a safety pilot function which helps other committees in search of fire safety answers through its nine working groups. IEC TC89 standards are of particular interest nowadays with the globalization of trade and the consequent need for harmonization of requirements outside of the North American borders. The importance of IEC standards is heightened by the fact that ASTM D09 bylaws now specify that every new standard should carry a statement addressing whether it is, or is not identical or even technically similar to some IEC standards. The paper explains that NEMA has a history of issues that are of particular interest to ASTM D09, including military specification conversions for electrical materials and the New York State smoke toxicity data base for electrical materials and products.

The remaining sections of this STP deal with specific individual issues, ranging from electrical insulating fluids, to fire issues and electrical issues. The papers cover a broad range of investigations, giving a thorough overview of active research on electrical materials. The issues include testing (for moisture content, fire properties, discharge properties, thermal endurance, physical stability), biodegradability, transformer maintenance, and new materials and products for the associated industries (including new electrical cables and new additives for cable materials).

Electrical Insulating Fluids: The second section of this STP addresses electrical insulating fluids and contains five papers addressing a variety of subjects. Clair Claiborne, T. V. Oommen, and E.J. Walsh explain the development of a fully biodegradable dielectric fluid based on a vegetable oil (with high oleic acid content) for use in electrical equipment, and its implications. A consequence of developing a novel material with different advantages is that specifications for the existing materials are often unsuitable for the new material which can hinder its commercialization until new standards are created. In this case, the existence of biodegradable insulating fluids is leading ASTM to work on new standards specific to such liquids to permit their field application to electrical transformers. Marius Grisaru gives six case histories in which transformers were withdrawn from service in Israel, by a power utility. The decisions were analyzed afterwards by the Central Chemical Laboratory who looked at the transformer's operational conditions based mainly on the concentration of gases and on the operation of Bucholtz relays. Based on this consideration it was found that four transformers had been properly removed from service and showed real faults while the other two had been removed prematurely. The two other transformers were then opened, tested, and returned to service. Samuel Margolis discusses an analytical technique to test for moisture using the Karl Fischer method in petroleum products. The paper explains that the results are affected by the method (volumetric or coulometric) used to make the measurement, the composition of the solution in the titration vessel, the degree of solubilization of the oil in the solvent, and the presence of trace amounts of materials in the solvent. The results are, however, independent of the ability of the instrument to titrate the water in a water standard. The investigation also suggests that the measurement of a falsely reduced moisture content is likely to be related to the ability of the undissolved oil in a heterogeneous system to sequester the water in a form that is not accessible to the Karl Fischer reagent. John Sabau and Rolf Stokhuyzen propose a new test to diagnose faults in high-voltage transformers. The test determines the physical stability of insulating oils by measuring changes in the absorption spectra and free radical concentration of transformer oils after they have been subjected to ionization in a discharge cell. Existing tests tend to be based on the analysis of gaseous degradation products which are thought to result from incipient electrical failures inside the windings. Paul Griffin, Joseph Kelly, and Thomas Rouse review the specifications for electrical insulating mineral oils, as well as the more common such products available. Emphasis is placed on comparisons between the ASTM and IEC standards (ASTM D 3487 and IEC 60296, which address physical, electrical, and chemical properties of the fluids.

Electrical Tests: The third section of this STP contains three papers dealing with test methods for properties of electrical interest. Hans-Jörg Mathis, Martin Baur, Rudolf Blank, Rudolf Woschitz, and Thomas Überfall introduce a test method for assessing disruptive discharge in lquid and gaseous insulation. The dielectric breakdown voltage is a variable with a statistical distribution of results so that any test procedure must be based on a set of single test sequences with minimal effect of one sequence on others to get reliable results. The article describes new ways to recognize dielectric breakdown voltages as well as new switching-off techniques. The article also discusses the advantages and disadvantages of various test methods. Ray Bartnikas addresses standardized testing procedures in partial discharge measurement. This comprehensive review has about 60 references and presents a comparison of the different standardized methods of discharge detection and measurement on electrical components, apparatus and cables. In particular, the standard methods from ASTM, IEC and IEEE (Institute of Electrical and Electronic Engineers) are compared. The ASTM approach is shown to constitute the basis for most standards, with the exception of those on rotating machines and compressed gas cables where very high frequency test methods are employed. Stephen Giannoni and Gian-Carlo Montanari present a study on accelerated aging of organic electrical insulating materials, particularly those used at elevated service temperatures. They determined long-term thermal endurance using traditional accelerated aging techniques alone and in combination with isothermal differential scanning calorimetry measurements of oxidation induction times. As there are practical limits on the maximum test temperatures for traditional aging, and as there is always an interest in decreasing test times while ensuring an accurate representation of the service degradation of the material, the combined DSC-aging technique results in a reduction in test times without compromising the accuracy of the determined service limit.

Fire issues: The final section of this STP has three papers addressing the response of electrical materials and products to fire. Ronald Markezich discusses the flame retardancy of various nylon (Nylon 6 and Nylon 66) and poly(butylene terephthalate) (PBT) insulation materials (with and without glass reinforcement), using mixtures of flame retardants which contain a chlorinated material. He addresses the various options available with special emphasis on the use of synergists. The paper also discusses effects of the additives on the electrical properties, especially the comparative tracking index which is improved by some of the systems used. Richard Whiteley and David Gardner present a study on ways of decreasing the mass and volume of electrical cabling systems, which is particularly important for transportation environments. The size and weight of wire and cable installations can be decreased by moving from thick and medium wall insulations to thin wall insulations. The paper compares the various fire properties of a number of wires based on ignition resistance, reaction to fire, and the production of smoke, acid/corrosive gases, and toxic fire products, together with the functional properties of the wires. Marcelo Hirschler presents a review of fire tests for electrical materials and products, both in the USA (ASTM, National Fire Protection Association, NFPA, and Underwriters Laboratories, UL) and internationally (International Organization for Standardization, ISO, and IEC). The properties discussed in this review (with almost 70 references and a large bibliography of fire standards) include ignitability, flame spread, heat release, (now considered to be the most critical fire property) and smoke release (smoke obscuration). Smoke toxicity and smoke corrosivity are also addressed to some extent, as is the need to conduct overall fire hazard assessments to get the best understanding of the potential problems associated with any electrical system.

Much goes into organizing a symposium and its documentation into an STP. The editor acknowledges and extends his thanks to Monica Siperko and Kathy Dernoga (of ASTM's publications group), Pat Picariello (staff manager to both committees ASTM D09 and ASTM D27), Dorothy Fitzpatrick (of the ASTM Symposia group), Joseph Kelly (chairman of committee ASTM D27), Loren Caudill (chairman of subcommittee ASTM D09.97, on planning), Thomas Robertson and David Baker (past and present chairman of committee ASTM D09, respectively) and Mark Marcus (from the Committee on Publications), as well as all of the authors.

Marcelo M. Hirschler
GBH International
Symposium Chairman and Editor

Standards

K. N. Mathes[1]

ASTM Committee D09—A History of Success

Reference: Mathes, K. N., "**ASTM Committee D09 - A History of Success,**" *Electrical Insulating Materials: International Issues, ASTM STP 1376,* M. M. Hirschler, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

Abstract: *Historical Aspects:* A long history of continued, active use is in itself a measure of success. Methods D 149 (1922), D 150 (1922), D 257 (1925) on electrical tests have been used extensively and even today are being periodically revised and updated. Much the same is true for D 115 (1941) varnish, D 202 (1924) paper and D 229 (1925) rigid sheet and plate plastics. D 495 (1938) dry tracking is generally now recognized as a technical failure, but a commercial success. This standard is still widely used and referenced! Standards D 69 (1920) friction tape, D 295 (1928) varnished cotton fabric and D 748 (1961) shellac, were once very important, are still needed, but today are seldom used.

Technical Concepts and Materials: In 1948 two technical papers, one by Ken Mathes on functional tests and another by Tom Dakin on thermal aging provided a basis for the adequate acceptance and use of the many new insulating materials that were becoming available. First, the Electrical Institute of Electrical Engineers, AIEE (now the Institute of Electrical and Electronic Engineers, IEEE), and then ASTM committee D09 followed with the much needed new approaches and standards—then and still outstanding successes. Tremendous growth in the use of plastics led to the formation of Technical Committee ASTM D20 from subcommittee status in D09. Committee D27 on Insulating Liquids and Gases also split away from D09. New elastomeric materials for cable led to their incorporation as Subcommittee 18 of D09 from their origins in ASTM D11 on rubber.

The subjects of treeing and surface failure (tracking) are extremely important, but today are still, to some extent, goals rather than completed accomplishments. *Instrumentation:* The Electronic revolution has been successfully incorporated by D09 in many electrical, mechanical, and physical tests for insulating materials. Methods for very high resistance are an example. *Organizational and Government Support:* Public Utilities, the Electrical Industry, NIST (originally NBS), the US Navy, NEMA, NASA, and UL have periodically been responsible for outstanding accomplishments in D09. UL continues in the forefront especially in thermal aging. D09 participation in the International Electrotechnical Commission (IEC) is of particular importance. *Publications:* Symposia, Publications like STPs and Research Reports have often highlighted D09 activity and success.

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[1] Consultant on Insulation and Plastics, Schenectady, NY 12309-4132.

Individuals: Success is possible only with the technical and organizational skills of dedicated individuals. Many ASTM awards have recognized such accomplishment.
Summary: This abstract can only touch on the foregoing aspects, which are described with more detail in the following paper.

Keywords: ASTM D09, ASTM D20, ASTM D27, ASTM D11, NIST, NBS, NASA, NEMA, US Navy, IEC, industry, public utilities, ASTM publications, STP, ASTM Report, ASTM symposia, ASTM award, insulating material, dielectric, electronics, plastic, history

Introduction

The author has been an active member of ASTM Committee D09 (the American Society for Testing and Materials on Electrical and Electronic Insulation) since 1942 (57 years!). The word "Electronic" was added to the title of D09 only in recent years. The wonderful personal association with many members of the committee over so many years and my involvement in many ways has provided the background and much of the information for this paper. Ed McGowan has been of special help.

Electrical insulation is vital to the operation of many types of electrical equipment from fingernail-size electronic components to huge turbine generators. A large variety of insulating materials with very different characteristics and often properties specific to the application is needed. The growth and increasing complexity of the electrical industry and the associated materials have led to a huge number of materials which need to be evaluated. In 1937 General Electric tried to standardize and limit its use to 8 kinds of insulating varnishes _ now there are thousands, for example, polyurethanes, polyesters, epoxies, silicone and so on! The development and required consensus for needed standards in such a complex arena is an ever-ongoing, technically challenging and time-consuming process _ not the "mature technology" often mistakenly envisioned by much industrial top management following the example of General Electric.

The two aspects of this paper _ "history" and "success" _ need definition. Here history is defined for methods by the date of "original issue," the dates on other documents and sometimes approximately by personal memories. Success is defined as technical excellence, especially when accompanied by commercial acceptance. (Occasionally, commercial interests may be involved without meritorious technical accomplishment.) Contributing factors to success include economic, scientific and engineering evolution and development.

Early Standards

Standards under the jurisdiction of ASTM D09 are found in the Annual Book of ASTM Standards Volumes 10.01 and 10.02. Volume 10.01 includes 79 standards issued from 1920 to 1965. All of these standards are reviewed, but in the absence of active technical interest some just may be edited and not revised. For example, friction tapes still have limited use without much technical interest. So D09-96 (1920)[2] on Test Methods for Friction Tapes is included in 10.01 with only editorial editing in 1996. Much the same is true for D295 (1928) for Varnished Cotton Fabrics Used for Electrical Insulation and D411-94 (1935) on Shellac Used for Electrical Insulation. A specification D784 (1961) for Orange Shellac and Other Indian Lacs for Electrical Insulation is also maintained. Just recognizing these seldom used methods and keeping them in the book for so many years, is in itself a measure of their *success.*

In marked contrast, D149-95a (1922), on AC Loss Characteristics and Permittivity, and D257-93 (1925) on DC Resistance or Conductance have been constantly technically revised and updated over many years of very active use. D150 and D257 have lists of references to technical papers written over many years by the most technically competent experts in prestigious organizations: among them H. L. Curtis (National Bureau of Standards, NBS now NIST in 1915 and 1939); R. F. Field (General Radio in 1944, 1945, 1946 and 1954); A. H. Scott (NBS in 1939. 1962 and 1965). Unreferenced work by world famous engineers, including Steinmetz of GE on voltage breakdown between 1910 and 1925, is reflected in D149. Each of these methods have important detailed appendices, which also have been continuously updated technically, as new concepts and measurement techniques developed. These three methods on electrical properties are referenced in many other standards in more than ten categories under the jurisdiction of D09 and separately by several other ASTM Technical Committees, as well. D149, D150 and D257 illustrate not only historical, but, also ongoing, outstanding *success.*

Other standards for which great *success is* measured by continuing technical update and very active use over many years include as examples: D115-96 (1941) for Solvent Containing Varnishes for Electrical Insulation; D202-98 (1924) for Untreated Paper Used for Electrical Insulation; D229-96 (1925) for Rigid Sheet and Plate Materials for Electrical Insulation; D348-95 (1932) for Rigid Tubes Used for Electrical Insulation; D374-94 (1933) for Thickness of Solid Insulation; D47093 (1937) for Crosslinked Insulations and Jackets for Wire and Cable; D876-95 (1946) for Non-Rigid Vinyl Chloride Polymer Tubing Used for Electrical Insulation and D902-95 (1946) for Flexible Resin Coated Glass Fabric and Tapes Used for Electrical Insulation.

Technical content of D495-94 (1938) for High-Voltage, Low Current, Dry-arc Resistance is now very questionable, but this method is often referenced. Thus D495 is a commercial success, that can not be removed from print.

New Materials and Technical Concepts

Originally, many insulating materials were based on natural products such as wax, mica, glass, ceramics, cements, cotton, silk, rubber, asphalt, shellac and oils like linseed. Early in the 1920's and 30's synthetic materials including first urea and then rayon, acetates, phenolics (Bakelite), saturated polyesters and poly(vinyl chlorides) began to appear Needed methods for these materials, as electrical insulation, were

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[2] The date in parenthesis indicates the year of original issue

developed in ASTM D09 on insulating materials and to some extent in D11 on rubber and D13 on textiles.

Starting with silicones in the late 1940's and continuing today new chemistry has led to an accelerated, tremendous, rapid growth in new materials. Many of these were developed first for application as electrical insulation. Examples include silicones, polyesters for wire enamel and films (polyethylene terephthlate and polycarbonates). As use of these materials expanded into broader applications, their costs fortunately decreased. Other materials initially were developed for other applications like polytetrafluoroethylene for gaskets and epoxies for adhesives. These materials were subsequently used for electrical insulation. Many methods were developed to meet new requirements for these materials _ a *success* in D09.

The use of polymeric materials has always extended beyond electrical applications. For example, urea based resins were first used for billiard balls. Phenolic resins were used for many molded products in the early 1920's. ASTM standardization of these products, however, was largely carried out in D09 emphasizing the electrical applications. With the very rapid growth of plastics in the 1940's, it became apparent that a new committee devoted to the non-electrical applications of plastics was needed. The formation of D20 on plastics from its roots in a subcommittee of D09 was overall a very great *success.* Somewhat later D27 on insulating fluids was another outgrowth from D09, organized primarily by Frank Clark of General Electric. Clark felt that the area of fluids, including dielectric gases, was not being adequately developed by D09.
An opposite trend occurred, when individuals in D11 on rubber felt, that their needs for electrical applications (especially in electric cables) were not being met adequately. In consequence, these aspects of D11 were moved to form D09 Subcommittee 18. This logical addition to D09 brought with it important new technology and technical expertise _another *success* for D09.

At the same time electrical technology was expanding rapidly in new areas like electronics and aircraft. New conditions such as high humidity in the tropics had to be met. Many examples can be listed. D1000-93 (1948) for Pressure Sensitive Adhesive coated Tapes for Electrical Applications is illustrative. B. H. Thompson at General Electric recognized the importance of electrolytic corrosion especially for adhesive tapes. He developed needed tests including improved means for controlling very high humidity with glycerine solutions. Many other tests to meet specific needs, like adhesion and blocking, were included in D1000 _ again illustrative of *success.*

Technical concepts had to change to meet the needs of new materials and their application. The concept of functional tests to meet application needs was introduced in a paper by Ken Mathes of General Electric in 1948. In the same year, Tom Dakin of Westinghouse introduced concepts of thermal aging based on reaction rate theory (the Arrhenious equation). A tremendous amount of activity followed in AIEE (now IEEE) and ASTM D09. It became apparent that temperature limits based on the broad description of insulating materials (first defined by Steinmetz of GE and Lamme of Westinghouse in 1913) were no longer technically adequate or appropriate.

First in IEEE generally and then in ASTM D09, heat aging tests based on reaction rate theory were developed. These tests provided thermal *indices* based on change of electrical properties, mechanical properties or weight loss for many insulating materials including magnet wire, varnishes, tubing and sheet. Higher thermal limits for electrical insulation based on these tests permitted the design of smaller and otherwise improved electrical equipment. The *success* achieved in this case is, perhaps, the most important of all achievements in D09 over the years.

The problem of surface failure on electrical insulation in electrical apparatus had been recognized. Surface resistivity (described in D257) and voltage breakdown over an insulation surface (flashover) in some test for solids evolved in D09 over the years. Detroit Edison developed a wet tracking (a kind of surface failure) test in about 1925. This test was difficult to control and failed to gain the acceptance of D09. However, a variation of the Detroit Edison wet tracking test was considered by the British Admiralty and then adopted by the British Standards Institute (BSI). Further variations of the test were developed in Germany and Switzerland. A much improved version was developed in Norway and finally adopted by the International Electrotechnical Commission (IEC) Committee TC15 as IEC 112. Underwriters Laboratory (UL) adopted IEC 112 and sponsored it's acceptance in D09 as D5288-97 (1992). Unlike D495, D5228 is both a technical and commercial *success.* However, it should be recognized that no test can meet all of the requirements for every application.

In illustration, an arc-tracking problem was encountered by the US Navy in aircraft hookup wire. Frank Campbell of the Naval Research Laboratory made an extensive investigation of this previously unrecognized problem. Fortunately, D09 Subcommittee 16 had been developing tests for many kinds of properties in D3032-97 (1972) Methods for Hookup Wire Insulation. After a number of years, including considerable controversy, methods for both wet and dry arc-tracking test were developed and included in D3032. Many interests were involved: including the US Navy, the US Air Force, British interests including the BSI, manufacturers of materials for hook-up wire, manufacturers of hookup wire and aircraft manufacturers. The US Navy refitted all of its aircraft at a cost of millions of dollars with hookup wire not subject to the arc-tracking problem. This wire was already in use for a number of applications. However, Boeing, McDonnell Douglas and the Air Force did not refit their planes. In this case, even though the technical success of the ASTM arc-tracking test was rather generally recognized, it received only limited commercial acceptance and use. D3032, with the inclusion of many test methods, was overall an outstanding *success.*

Instrumentation

In early years electrical, mechanical and physical tests were limited in sensitivity, accuracy and ease of use. Anyone who remembers the measurement of small currents using a string galvanometer with a mirror, which caused a light spot to move on a curved scale, will attest to these problems. D257 now lists electrometers, dc amplifiers and much improved galvanometers with at least one thousand times the sensitivity of the best string galvanometer. Greatly improved instrumentation for many other needs was developed also over the years by several equipment manufacturers and adopted by D09.

Most of the instruments needed for ac loss characteristics and permittivity (see D150), including especially the original Schering bridge for measurements at high voltage, at first lacked sensitivity, range and direct reading. Again several manufacturers progressively developed sensitive, direct reading instruments. Some of these could automatically balance, print results and interface with computers.

Much the same types of improvements were made in instruments for many mechanical and physical tests, which were standardized mostly in other ASTM committees, but used in many D09 methods.

Without question, development of improved instrumentation has led to more successful use of test methods under the jurisdiction of D09.

Organizational and Government Support

In the foregoing, reference has been made to organizations involved in specific standards for electrical insulation. Without the support of these organizations, the work of D09 would have been impossible. From the early days, public utilities (especially Consolidated Edison, Detroit Edison and Commonwealth Edison) had been deeply involved. The principal electrical manufacturers in the United States were also committed, carrying out round robin and other investigations in their engineering and research facilities. General Electric and Westinghouse often sent ten or more individuals to D09 meetings to represent their many types of interest. The same interest and activity was carried on in some commercial laboratories such as the Electrical Testing Laboratory and in government agencies like the US Navy and especially the National Bureau of Standards. The dedication of these organizations and their representatives (see below) was responsible for the outstanding success in the development of many standards in D09. Unfortunately, almost all of this representation is no longer active. Perhaps, a belief that electrical insulation is a mature technology may be involved. Moreover, much of the electrical industry has been subject to "downsizing, outsourcing, mergers and acquisitions" with emphasis largely on profits. This industry trend has often led to smaller laboratories and reduced technical personnel with little support for standardization.

Commercial competition and controversy in standardization always brings active participation, but may not be marked at present. However, insulation technology continues to advance and change. For example, new ceramic superconductors of interest in the power industry can operate in liquid nitrogen. Associated electrical insulation will need special tests to determine their suitability at low temperatures.

D09's important involvement with international standardization in IEC committee TC15 on solid electrical insulation and TC55 on winding (magnet) wire deserves recognition. Arnold Scott of NBS led the US delegation to the newly formed TC15 at Munich in 1956 and at New Delhi in 1960. For a number of years after that Ken Mathes acted as Technical Advisor for the US to IEC T15 and led the delegations with financial support from D09 and NEMA (Charlie Willmore). Later, UL became active and Howard Reymers replaced Ken Mathes as the Technical Advisor. Landis Feather and now Steve Giannoni followed Howard. Material suppliers like DuPont and Raychem have also supplied technical support and representation. IEC support from UL and manufacturers of electrical insulation continues.

In D09, also, many suppliers of electrical insulation and components, like magnet wire and cable continue active support, but it is often difficult to obtain the balanced support between suppliers and users required by ASTM. UL is still very active and of great help to the work in D09.

Individuals

The leadership, technical competence and dedication of many individuals are responsible for the success of D09 over the years. These individuals have been involved in many D09 sponsored activities including the technical papers at the symposia listed below in table 3. All of these individuals can not be recognized here. Special recognition was given to Arnold Scott of NBS for his tremendous contributions over many years with the establishment by D09 of the Arnold H. Scott award in 1965. This award recognizes outstanding achievement in the science of electrical insulation. Recipients are listed in Table 1.

Table 1 _ *ASTM Arnold H. Scott Award*

1968	Thomas Hazen	1982	Philip E. Alexander
1969	Harold S. Endicott	1983	Paul F. Ast
1970	Herbert G. Steffens	1984	Carl F. Ackerman
1971	Kenneth N. Mathes	1985	Ray Bartnikas
1972	Edward B. Curdts	1986	Edward J. McGowan
1973	Joseph R. Perkins	1987	Wendell T. Starr
1974	Lester J. Timm	1988	Charles J. Saile
1975	Howard A. Davis	1989	Robert E. Kraus
1976	Edmund H. Povey	1992	Thomas R. Washer
1977	Eugene J. McMahon	1993	Edmund J. Zalewski
1978	William P. Harris	1996	Mark Winkler
1979	Celine Paul	1997	Loren Caudill
1981	Landis Feather		

ASTM itself recognizes individuals for overall achievement with the ASTM Award of Merit. Individuals sponsored by D09 for this award are listed in Table 2 on the following page.

The ASTM Dudley Award honors an outstanding contribution that has a widely recognized impact on the particular field of ASTM interest and has been documented in ASTM literature. The Dudley Award in 1985 was given to Ray Bartnikas for his editing and contributions to the several ASTM Special Technical Publications (STP) "Engineering Dielectrics" (see table 2 below).

The contributions of individual members from D09 to other activities are also important. Over the years D09 members have served in IEEE, NEMA and other technical organizations. Organizations like the US Navy and NASA have often recognized and utilized the expertise of D09 members. For example, two D09 members served in the investigation of the tragic fire in the Apollo spacecraft at Cape Canaveral. One of these D09 members had a number of follow up consulting assignments on the fire and other problems at NASA facilities in Huntsville, Alabama and Houston, Texas.

Table 2 _ *ASTM Award of Merit*

1958	Arnold H. Scott	1984	William P Harris
1964	Kenneth G. Coutlee	1985	Ray Bartnikas
1966	Harold H. Graves	1987	Carl F. Ackerman
1968	Harold S. Endicott	1998	Herbert G. Steffens
1970	Edward B. Curdts	1992	Howard E. Reymers
1972	Ernest 0. Hausman	1994	Thomas Robertson
1976	Kenneth N. Mathes	1998	Edward J. McGowan
1976	Joseph R. Perkins	1999	Robert E. Kraus

Publications

Many D09 methods have supporting "Research Reports" include round-robin and other data which are kept on file by ASTM headquarters.

Over the years, D09 has sponsored many technical sessions with contributed technical papers on selected technical subjects. Many meetings have been documented with Special Technical Publications (STP) listed in Table 3.

Table 3 _ *ASTM Symposia and STPs Sponsored by D09*

STP 59 - 1944 Symposium on Plastics (before D20 was organized)
STP 95 - 1949 Symposium on Insulating Oils
STP 135- 1952 Symposium on Insulating Oils
STP 152- 1953 Symposium on Insulating Oils
STP 161- 1954 Symposium on the Temperature Stability of Electrical Insulating Materials
STP 172- 1952 Symposium on Evaluation of Insulating Oils
STP 188- 1956 Symposium on Minimum Property Values of Electrical Insulating Materials
STP 198- 1955 Symposium on Corona
STP 218- 1957 Symposium on Insulating Oils (The last on liquids before the transfer to D27)
STP 276 - 1959 A three part part symposia on Materials in Nuclear Applications - D09 and D20 jointly sponsored one part, "Postirradiation Effects on Polymers".
STP 420 - 1966 Symposium - Measurement of Dielectric Properties under Space ConditionsHard Cover Books Edited by Ray Bartnikas in a series"Engineering Dielectrics"
STP 669- 1979 Vol. I - Corona Measurements and Interpretation
STP 783 -1983 Vol. IIA - Electrical Properties of Solid Insulating Materials: Molecular Structure and Electrical Behavior
STP 926 -1987 Vol. IIB - Electrical Properties of Solid Insulating Materials: Measurement Techniques

A number of symposia have been held by D09 without publications of STP's including an important "Symposia on Electrode Systems for Dielectric Measurements". Ed McGowan has taken the lead to include the papers from that symposium, as a Research Report for D09 _ an illustration of how an individual's dedication can lead to another *success* for D09.

Most of the individuals listed in tables 1 and 2 have authored papers for the symposia and deserve special recognition. Among them are: Arnold H. Scott of NBS, Kenneth G. Coutlee of Bell Laboratories, Harold S. Endicott of General Electric, Joseph R. Perkins of DuPont, William P. Harris of NBS, Ray Bartnikas of the Centre for Research for Quebec Hydroelectric, Landis E. Feather of Westinghouse, Edward J. McGowan of Raychem, Thomas Hazen of Bakelite and W. T. Starr of Raychem. The names of the organizations supporting these individuals have been included to emphasize the importance of organizational support.

Many individuals have contributed to the support and success of D09 in other ways. Lester J. Timm of the Brooklyn Naval Shipyard and later in retirement served as the dedicated secretary for D09 over many years. Celine Paul of Western Electric is the only woman to have served as a subcommittee chairman in D09. (Fortunately, a few women are now members of D09.) A number of individuals have been so dedicated to D09, that they have continued to serve after retirement at their own expense including among them: Harold Endicott, Carl Ackerman, Landis Feather, Bob Kraus, Ed McGowan and Wendell Starr.

Summary and Conclusions

Electrical equipment can be described in simple fashion as a combination of conductor, structural components and *electrical insulat*ion. The design, manufacture and use of each of these components include the need for development, selection, test and control for the properties of the materials used. Standardization of test methods for properties and associated specifications of insulating materials has been and continues to be vitally needed. Historically ANSI (American National Standards Institute), ASTM, NEMA, UL and individual companies have written related standards. Organizations such as IEEE and government agencies (MIL specifications for example) have all been involved, also. Internationally IEC TC15, TC55 and to some extent other IEC committees have had similar activity. Obviously, such a complicated mix of responsibility may lead to overlap and duplication of effort. This paper describes some of the ways in which ASTM D09 has cooperated with many of these organizations. Not mentioned earlier is the overall understanding between ASTM and government agencies to use ASTM test methods and specifications where possible. D09 also often references and coordinates its standards with those of IEC TC15. These are areas of *success* for both organizations. The need for continuing such effort is obvious in an ever expanding and changing technology.

The success achieved in D09 over past years from the combined effort of dedicated individuals and associated organizations will be a "hard act" to follow in the future. Unfortunately, direct participation in D09 by large electrical manufacturers and government agencies, so important in previous years, is at low ebb. Hopefully, more effective effort can be directed to solve this problem and continue the success of previous years. Perhaps the direction of industry will change again. (It took twenty years from the passage of the Sherman antitrust act to 1910, when President Roosevelt dismantled the oil trust.) Evidence of disenchantment with profits, as almost a sole motive, is beginning to appear. Moreover, government agencies can be expected to continue their efforts to adopt standards, like those in D09, because such action is cost effective. More direct involvement of individuals in these agencies would be helpful and should be encouraged.

It remains for ASTM and D09 to convince industry and government that insulation is not a mature technology. The mere addition of the word "Electronics" to the D09 title is not sufficient. The present low level effort in the electronics area does need to be expanded. Here, the advance in technology and the need for standards is obvious. D09 must adapt to this need.

Even in more traditional electrical equipment such as generators, transformers and cable, change is coming. For example, superconductivity at liquid nitrogen temperatures with ceramic superconductors may now be feasible for such apparatus. New standards for materials and test procedures will be needed. A paper, "Problems with Dielectric Measurements at Cryogenic Temperatures" by K. N. Mathes and E. J. McGowan, was included in *STP 420*, published 33 years ago in 1966! This paper considered tests in liquid nitrogen and helium. Then superconductivity could be achieved only in expensive liquid helium or perhaps in dangerous liquid hydrogen. Recent articles in the literature show a resurgence of interest. It will be up to today's industrial management, government agencies and D09 to meet such new challenges in the future.

David K. Baker[1]

Current Structure and Activities of ASTM Committee D09

Reference: Baker, D. K., "**Current Structure and Activities of ASTM Committee D09,**" *Electrical Insulating Materials: International Issues, ASTM STP 1376,* M. M. Hirschler, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

Abstract: Over the last 14 years, the span of my direct involvement with ASTM, many changes have occurred not only in the membership and goals of Committee D09, but also in ASTM itself. This paper will highlight the various Subcommittees within Committee D09 and some of the activities that have been completed over the past several years. In addition, the current topics of discussion within these Subcommittees will be pointed out. Committee D09 has a working relationship with other ASTM committees and standards organizations and some of these activities will be covered as well.

D09 membership is made up of approximately 160 individuals representing users, manufacturers, and general interest groups. Members may participate in as many subcommittees as they desire. In D09 the 160 members are responsible for 194 standards.

The scope of D09 is to develop test methods, practices, terminology, specifications, and promote knowledge and research relating to solid and solidifying fluid electrical insulating materials.

The principal materials included are grades designed primarily for electrical insulating or dielectric purposes of the following:

- Varnishes and resins
- Reinforced, laminated, filled and unfilled themoplastic and thermosetting plastics
- Metal-clad dielectrics
- Glass and Mica
- Embedment resins, compounds, and waxes

Keywords: Cable, ceramic, dielectrics, fiber, glass, insulation, jacket, mica, paper, plastics, resin, rubber, semi-conducting, sheet, tape, tubing, varnishes, waxes and wire.

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- Coated, Extruded, Cast, and Calendered Sheet, Tape, and Tubing,
- Organic and Inorganic Papers, Coated and Uncoated,
- Plastic, Resin, Paper, Fiber, Thermoset, Rubber and Ceramic Insulation for Wire, and
- Thermoplastic and Thermosetting Insulations, Jacket, and Semi-Conducting Materials for Insulated Wire and Cable.

Within the scope are standards for the evaluation of all electrical, mechanical, chemical, and thermal properties needed to define these materials, either alone or in simple combinations representative of the way these materials are combined in use; and of the effects upon the properties of these materials, or combinations, of operating environments including the presence of heat, thermal shock, vibration, outer space conditions, and ionizing radiation.

Electrical test methods may include procedures for measurement at direct voltage and at all frequencies, including optical frequencies.

Standards for insulated conductors, including fiber optics, are within the scope insofar as these standards evaluate properties of the electrical insulation and jacket materials.

The scope of D 9 does not include materials that are fluid in normal use except for their use as controlled ambients or impregnants for testing purposes.

At the present time D 9 is divided into sixteen subcommittees as listed below. An example of the standards developed by each group is also listed. The particular standards shown are the oldest and most recent standards issued by the subcommittee. These standards give an indication of when the subcommittee was formed, and its most recent activity. Subcommittees are divided into Task Groups (TG) which are responsible for formulation of initial drafts of new-work documents and for keeping existing standards up-to-date.

D09.01, ELECTRICAL INSULATING VARNISHES, POWDERS, AND ENCAPSULATING COMPOUNDS: The formulation of specifications and preparation of test methods for varnishes, paints, and lacquers that are applied in the form of liquids or in suspension and then hardened by evaporation or chemical action to produce either insulating films or insulating binders. (24 Standards)

D 176, Test Methods for Solid filling and Treating Compounds Used for electrical Insulation (1923)

D 6053, Test Method for Determination of Volatile Organic Compound (VOC) Content of Electric Insulating Varnishes (1998)

D09.07, FLEXIBLE AND RIGID INSULATING MATERIALS: The development of methods of test and specifications for flexible electrical insulation comprising treated braided sleeving, extruded plastic tubing, fabrics, films, pressure sensitive and bondable adhesive tapes and rigid electrical insulation comprising plates, tubes, rods and molded materials. (50 Standards)

D 69, Test Methods for Friction Tapes (1920)

D 5213, Specification for Polyimide Resin film for Electrical Insulation and dielectric Applications (1991)

D09.10, MAGNET WIRE INSULATION: The preparation and development of methods of test for mechanical, electrical, chemical, and thermal properties of magnet wire insulations and the formulation of appropriate specifications. (4 Standards)

D 1676, Test Methods for Film-Insulated Magnet Wire (1959)

D 3353, Test Methods for Fibrous-Insulated Magnet Wire (1974)

D09.12, ELECTRICAL TESTS: The development and preparation of test methods to determine the electrical properties of materials used as electrical insulation. (32 Standards)

D 149, Test Method for Dielectric Breakdown Voltage and Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (1922)

D 6194, Test Method for Glow-Wire Ignition of Materials (1977)

D09.14, ELECTRIC HEATING UNIT INSULATION: The development of standard test procedures and specifications for granular insulating materials as used in sheathed electrical heating elements. (11 Standards)

D 2755, Test Method for Sampling and Reduction to Test Weight of Electrical Grade Magnesium Oxide (1968)

D 5569, Test Method for the Determination of Static Flow of Electrical Grade Magnesium Oxide for Use in Sheathed Heating Elements (1994)

D09.15, acts as the U. S. Technical Advisory Group (TAG) to the to the Technical Advisor to the U. S. National Committee of the International Electrotechnical Commission (USNC/IEC) on matters related to IEC Technical Committee 15 on electrical Insulating Materials, and its subcommittees.

D09.17, THERMAL CAPABILITIES: Develop and maintain test methods, practices, terminology, specification, and the promotion of knowledge and research relating to the thermal characteristics of electrical insulating materials. (4 Standards)

D 3850, Test Method for Rapid Thermal Degradation of Solid Electrical Insulating Materials by Thermogravimetric Method (1979)

D 5642, Test Method for Sealed Tube Chemical Compatibility Test (1997)

D09.18, SOLID INSULATIONS, NON-METALLIC SHIELDINGS, AND COVERINGS FOR ELECTRICAL AND TELECOMMUNICATION WIRES AND CABLES: The development of test methods, practices, definitions and specifications for materials used as insulations, non-metallic shieldings and covers for electrical and telecommunications wires and cables. (42 Standards)

D 470, Test Methods for Crosslinked Insulations and Jackets for Wire and Cable (1937)

D 6096, Specification for Poly(vinyl chloride) Insulation for Wire and Cable, 90°C Operation (1997)

D09.19, DIELECTRIC SHEET AND ROLL PRODUCTS: The formulation of standards for materials used for electrical insulation in the form of paper, nonwovens, boards, flexible laminate composites, or mica products; and the raw materials for these products. Not included are rigid laminates, vulcanized fiber or polymeric films alone. This scope includes materials in either unimpregnated state or impregnated with liquid or resin combined with materials which themselves are not part of this scope. (17 Standards)

D 202, Test Methods for Sampling and Testing Untreated Paper Used for Electrical Insulation (1924)

D 6343, Test Method for Thin Thermally Conductive Solid Materials for Electrical Insulation and dielectric Applications (1998)

D09.20, acts as the U. S. Technical Advisory Group (TAG) to the to the Technical Advisor to the U. S. National Committee of the International Electrotechnical Commission (USNC/IEC) on matters related to IEC Technical Committee 20C on Burning Characteristics of Electric Cables.

D09.21, FIRE PERFORMANCE STANDARDS: Develops fire performance standards that describe the performance of electrical and electronic insulating materials, components and constructions. This committee will provide the technical support to the liaison subcommittee to IEC, but will not write specifications.

This subcommittee will work closely with other ASTM committees such as E 5 to assure that the ASTM fire policy is followed. It is expected to work with other organizations outside of ASTM through joint task groups to develop the best possible standards through the consensus process. (5 Standards)

D 5424, Test Method for Smoke Obscuration of Insulating Materials Contained in Electrical or Optical Fiber Cables When Burning in a Vertical Cable Tray Configuration (1993)

D 6113, Test Method for Using a Cone Calorimeter to Determine Fire-Test-Response Characteristics of Insulating Materials Contained in Electrical or Optical Fiber Cables (1997)

D09.89, acts as the U. S. Technical Advisory Group (TAG) to the to the Technical Advisor to the U. S. National Committee of the International Electrotechnical Commission (USNC/IEC) on matters related to IEC Technical Committee 89on Fire Hazard Testing and its subcommittees.

D09.90, EXECUTIVE: Composed of subcommittee chairmen or their designated alternate.
D09.92, AWARDS: This subcommittee helps to recognize individuals who promote the ideals of ASTM.

D09.94, NOMENCLATURE, SIGNIFICANCE, AND STATISTICS: Responsible for editorial review of all existing or proposed standards and documents under the jurisdiction of ASTM Committee D 9. (3 Standards)

D 1711, Terminology Relating to Electrical Insulation (1960)

D 3636, Standard Practice for Sampling and Judging Quality of Solid Electrical Insulating Materials (1977)

D09.97, LONG RANGE PLANNING: Develops and maintains the long range plan of D 9, promotes acceptance of standards worldwide, develops and administers International Standards Activity and funding, promotes the growth of D 9 membership, coordinates activities with other standards writing organizations to obtain harmonization and prevent duplication of effort and coordinate and promote technical seminars.

Conclusion

Through the efforts of approximately 40 to 45 attendees at the two meetings of ASTM Committee D09 each year and the comments received as a result of letter balloting, the 194 existing standards are kept up-to-date and new standards and test methods for new materials are created.

J. J. Kelly[1] and Dennis M. Getz[2]

Structures and Activities of ASTM Committee D-27

Electrical Insulating Materials STP 1376

Reference: Kelly, J. J., and Getz, D. M., "**Structures and Activities of ASTM Committee D-27,**" *Electrical Insulating Materials: International Issues, ASTM STP 1376,* M. M. Hirschler, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

Abstract: ASTM Committee D-27 on Electrical Insulating Fluids and Gases was formed as Subcommittee IV under Committee D-9 in the year 1916 and was designated as a separate technical committee in 1959. The scope of D-27 is the promotion of knowledge pertaining to insulating liquids and gases, whether synthetic or natural in origin, and the recommendation of standards and specifications pertinent to these materials.

The principal materials included in this arena are oils of petroleum origin, synthetic liquids, and halogenated and other gases when used singularly or as combinations, as electrical insulation or as an environment for electrical insulation in transformers, capacitors, and other electrical equipment.

Standards peculiar to solid insulating materials and varnishes, and the development of standards pertaining to non-electrical uses of liquids and gaseous materials are excluded from our scope.

The D-27 membership is made up of technical people from the refining and chemical industries, electrical equipment manufacturers, electrical utilities, and laboratories that test and analzye both new and used fluids and gases.

The paper presents in some detail the responsibilities of the various subcommittees and the harmonization efforts to work with international standards organizations.

Some specific examples of our work are cited in terms of the events that preceded the need to standardize a procedure.

Keywords: Transformer oil, insulating oil, insulating liquid, cooling liquid, dielectric liquid, functional properties, synthetic fluid, insulating gas

What we know today as a transformer was modeled after a device patented in England by Gaulard and Gibbs in 1882. George Westinghouse brought this idea to the United States a few years later. These first transformers were made of oil, copper, cardboard and steel[1]. Over 100 years later, we are still using the same materials but utilizing the benefits that come from the improvements in technology and standardization activities.

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Some of the early ASTM committees were formed in order to deal with the different aspects of various materials. As many of the materials were put into greater use, the need for standardization was recognized. These activities were formalized to reflect the specialized areas of interest and expertise. During the first decade of ASTM, these committees were formed:

Committee A-1 on Steel and Related Alloys	1898
Committee B-2 on Non Ferrous Metals	1902
Committee A-6 on Magnetic Properties	1903
Committee D-2 on Petroleum and Lubricants	1904
Committee E-5 on Fire Standards	1904
Committee D-9 on Electrical Insulating Materials	1909
Committee B-1 on Electrical Conductors	1909[2]

Elihu Thomson, working for Westinghouse, patented the first mineral oil insulating fluid in 1887[3]. It was originally referred to as "transformer oil" but was eventually used in other electrical devices. The primary functions of this oil were to provide dielectric strength and to cool the transformer from heat generated while in operation. Today, we have established that the oil is also in use to protect the insulating paper and it can also be used as a diagnostic tool, used to evaluate the condition of the equipment that it is in.

The 1890s saw a need to identify the functional properties of oil and how these could be adjusted to fit in situations presented to transformer operators. These properties represented electrical, physical and chemical characteristics of the oils in question. In addition to oils of petroleum base, animal fat tallow rendered into liquids and vegetable based seed oils were tried in transformers. The qualities shown by the petroleum based mineral oils proved to be an advantage over the animal and vegetable oils.

In the 1900s, an attempt was made to find one oil that would work under a variety of conditions in electrical equipment rather than the dozen products that were in service at the time[4]. Committee D-9 was involved in these issues and saw that they needed special consideration. To properly address these issues, Subcommittee IV on Liquid Insulation was formed in 1916. In 1959, this Subcommittee was elevated to full Committee status and given the designation D-27 on Electrical Insulating Liquids and Gases

During the early history of insulating liquids, oils named Diamond Paraffine and 300 Degree Mineral Seal Oil were in service[5]. The names of these oils portend some of the issues that had to be resolved both then and today in regards to critical considerations in the application of various oils as their composition and functions undergo the changes brought on by industry changes. Wax formation, a problem brought on by paraffinic-based oils, can be a problem in its cooling function in the transformer. Dewaxing may solve this problem, as long as the integrity of the oil as a dielectric medium is not compromised in the process. Flammability ranges of some of the early oils also presented some risks.

An early standard published by the Liquid Insulation Subcommittee that was first issued in 1921 and is still in use today is D 117-96, Standard Guide for Sampling, Test Methods, Specifications, and Guide for Electrical Insulating Oils of Petroleum Origin[6]. This Standard Guide currently lists 52 tests and specifications and describes the scope and significance of the methods. Some of the tests listed fall under the jurisdiction of Committee D-2, not Committee D-27.

The 1930s brought new interest in vegetable oils for use as a dielectric medium. The thought at the time was that they might be acceptable fluids because their chemical composition could be easily identified, especially when compared to the chemical composition of mineral oil, which may contain thousands of hydrocarbons[7]. It was eventually determined that mineral oils were still the fluid of choice for transformers.

STPs (Special Technical Publications) were a method that Committee D-9 used to discuss issues in the 1940s and early 1950s. During this time, STPs 146, 149, 95, 135 and 152 were published. They were published in the order listed. The first four documents pertained to testing of in service insulating oils[8] with the fifth document; STP 152, as presented by F. C. Doble, promoting the reclamation of used oils[9].

As different crude stocks began to be used in the refining of transformer oil and refining methods began to change, Committee D-27 formed from its parent Subcommittee. Certainly, both main committees have a common beginning and common interests but both have now specialized in their unique fields of electrical insulating materials both in the United States and in the international arena.

The next focus for D-27 was to look beyond the issues of oils as a dielectric and coolant and to realize that a more important function that they play is in preserving the cellulose insulation in a transformer. Original oils were of paraffinic base. These needed to be dewaxed to ensure proper operation in cold climates. Over time, these oils would oxidize and form sludges, which were insoluble and abrasive to the paper insulation. To deal with the sludge problem, two solutions were proposed. One solution was the use of naphthenic crudes. These crudes did not have wax, solving that problem. The sludges formed in oils of this stock were softer than those in paraffinic stock were. They were also soluble. The other solution was the design of sealed transformers with a nitrogen gas cushion or an air filled bladder. In these transformers, contact of the transformer oil with outside air was controlled, causing oxidation to be minimized. The addition of oxidation inhibitors in the dielectric fluid further slowed the aging process. Oil specification D 1040 for uninhibited oil was published in 1949 followed by D 3146 for inhibited oil. The latest specification D 3487 deals with both types of oil and is currently being updated[10].

In 1987, D-27 held a symposium on Electrical Insulating Oils and produced STP 998 from the symposium. STP 998 is a collection of 10 papers that deal with oil manufacturing, PCBs (polychlorinated biphenyls) as an old synthetic with new problems, acceptance tests, dissolved gas in oil analysis, significance of oil properties, new problems and new analytical techniques. As good as this coverage was, much work still has to be done.

As voltages increase, so do the challenges for the transformer operator. Some of the latest tests look for the breakdown products of paper in oil. Electrical stressing can affect the gassing tendency of the oil and alter its dissipation factor. Certain gases are used as dielectric media in transformers and other electrical equipment but SF6 (sulfur hexaflouride) gas has been labeled as a greenhouse gas. Synthetic fluids are becoming bigger players in the transformer arena. Vegetable oils have once again entered the market place with emphasis on a quality other than that of a dielectric or cooling medium. That quality is biodegradability.

As the world has gotten smaller, D-27 has gone international. Institute of Electrical and Electronics Engineers (IEEE) guides and standards refer to ASTM for test procedures and specifications used in transformer maintenance. For instance, Standard Test Method D 3612 for Analysis of Gases Dissolved in Electrical Insulating Oil by Gas Chromatography is one of the most powerful tools used for the detection of fault conditions in a transformer. It is referenced in C57.104, the IEEE Guide for the Interpretation of Gases Generated in Oil-Immersed Transformers. D-27 also maintains its position as the official liaison with its international counterpart, Committee TC 10 of IEC, headquartered in Geneva, Switzerland.

Up to this point, the United States has relied on naphthenic crudes as its source for mineral oils but many countries use paraffinic crudes. The U. S. market is in a state of flux and we may see paraffinics again.

It appears that the work of D-27 is not complete. D-27 must remain dedicated to the progress of helping to provide reliable power through the consistent use of safe materials that protect human health and the environment.

The main committee D-27 is made up of nine technical subcommittees along with three administrative subcommittees. Most of the technical work is carried out at the subcommittee level. Their work is contained in volume 10.03 of the Annual Book of ASTM Standards. The technical subcommittees are as follows:

D-27.01 Mineral Oils

This subcommittee writes the specifications for electrical insulating oils of petroleum origin, studies the performance of these fluids and refers test procedure requirements to the appropriate test method subcommittee. An example of a specification that this subcommittee is responsible for is D 3487, Standard Specification for Mineral Insulating Oil Used in Electrical Apparatus.

D-27.02 Gases and Synthetic Liquids

This subcommittee carries out the same activities as subcommittee D-27.01 but does it for non-petroleum based insulating media such as askarels, silicones, polybutenes and sulfur hexaflouride. An example of a specification that this subcommittee is responsible for is D 4652, Standard Specification for Silicone Fluid Used for Electrical Insulation.

D-27.03 Analytical Tests

This subcommittee develops new methods and improves current methods and practices that are used to determine composition and properties of insulating liquids and gases. This includes all analytical test methods, which are not specifically physical, chemical or electrical tests. An example of a test method that this subcommittee is responsible for is D 5837, Standard Test Method for Furanic Compounds in Electrical Insulating Fluids by High-Performance Liquid Chromatography (HPLC).

D-27.04 Nomenclature and Definitions

This subcommittee writes definitions of terms used in the committee's methods and specifications. It works with ASTM's Committee on Terminology to standardize definitions between the technical committees of ASTM. An example of a standard that this subcommittee is responsible for is D 2864, Standard Terminology Relating to Electrical Insulating Liquids and Gases.

D-27.05 Electrical Tests

This subcommittee develops and updates test methods and practices that measure the electrical properties of insulating liquids and gases. An example of a test method that this subcommittee is responsible for is D 877, Standard Test Method for Dielectric Breakdown Voltage of Insulating Liquids Using Disk Electrodes.

D-27.06 Chemical Tests

This subcommittee develops new methods and improves current methods and practices used to determine the chemical nature and chemistry of insulating liquids and gases. An example of a test method that this subcommittee is responsible for is D 1533, Standard Test Methods for Water in Insulating Liquids (Karl Fischer Reaction Method).

D-27.07 Physical Tests

This subcommittee develops new methods and improves current methods and practices used to determine the physical properties of insulating liquids and gases and the methods used to sample these materials for testing and analysis. An example of a test method that this subcommittee is responsible for is D 971, Standard Test Method for Interfacial Tension of Oil Against Water by the Ring Method.

D-27.08 Symposia

This subcommittee exists to invite speakers to make formal presentations on new or improving techniques and to introduce candidate materials for acceptance as insulating liquids and gases. An example of the work that this subcommittee is responsible for is the symposium held at the October 1998 meeting on biodegradable and fire resistant dielectric fluids.

D-27.91 Advisory to IEC, TC 10

This subcommittee exists to make sure that the work done by Committee D-27 is recognized in the international arena and to have input in the development of international standards regarding insulating liquids and gases.

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Nicolas N. Maennling[1]

Fire Hazard Testing in the International Electrotechnical Commission Standards: An Overview of IEC TC 89 Activities

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Reference: Maennling, N. N., "**Fire Hazard Testing in the International Electrotechnical Commission Standards: An Overview of IEC TC 89 Activities,**" *Electrical Insulating Materials: International Issues, ASTM STP 1376,* M. M. Hirschler, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

Abstract: This paper presents an overview of the activities of IEC TC 89—Fire hazard testing. It covers the history of the committee, its status within the IEC and its philosophy of risk reduction. It describes the current publications and projects of IEC TC 89, plus new work as well as the description and activities of its eight working groups.

Keywords: flammability, IEC, safety

In the mid-1970s there was growing concern about the fire risks associated with the increasing use of plastic materials in the construction of electrotechnical equipment.

The IEC Central Office in Geneva reported the work on flammability tests by various IEC committees. The result of this report was a meeting in The Hague, The Netherlands that represented approximately 20 IEC committees. A working group was formed to consider how to proceed with this concern. The working group reported that "the important subjects for future work were definitions, guidance on aspects and measures related to fire protection, preparation of fire hazard assessment tests and guidance for their selection and application."

In 1977 new Subcommittee SC50D "Fire Hazard Testing" was formed and within two years the first publications of the IEC 695 series were published. This rapid progress was made possible by the transfer of experts from IEC and ISO to this new committee as well as previous work that had been done since the late 1950s by the CEE (International Commission on Rules for the Approval of Electrical Equipment).

In 1989, SC50D was elevated to full technical committee status as TC 89.

Status Within the IEC

TC 89 has a horizontal safety function and is given the task to prepare basic safety publications.

The work of a technical committee with a horizontal safety function directly affects the publications of many IEC product committees from the aspect of safety and serves to ensure consistency within the IEC.

Basic safety publications comprise fundamental concepts, principles and requirements with regard to general safety aspects and are applicable to a wide range of products, processes

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and services. It is the responsibility of a product committee to make use of basic safety publications.

It is intended that bi-directional liaisons are set up between committees with a horizontal safety function and product committees to keep each other informed about the progress of relevant work. This liaison is formed, preferably, at an early stage of the work.

Risk Reduction

The risk of fire needs to be considered in any electrical circuit. With regard to this risk, the objective of component, circuit and equipment design, and the choice of material is to reduce the likelihood of fire even in the event of foreseeable abnormal use, malfunction or failure. The primary aim is to reduce ignition due to the electrically energized part but, if ignition and fire do occur, to control the fire preferably within the bounds of the enclosure of the electrotechnical product. In cases where surfaces of the electrotechnical products are exposed to an external fire, care will be taken to ensure that they do not contribute to the fire growth to a greater extent than the building products or structures in the immediately surrounding areas.

It is important to understand and maintain the difference between "fire hazard" and "fire risk." The primary concern for fire assessment is to minimize the risk of fire within electrotechnical products and, should one start, to limit fire propagation.

Consideration shall be given to heat release and smoke opacity, toxicity and corrosivity of the smoke from a burning product, and any necessary ability to function under fire conditions. These hazards are all related to ignition and fire growth. The emission of gases may also lead to a risk of explosion under certain conditions.

Certain electrotechnical products, such as large enclosures, insulated cables and conduits, may, in fact, cover large portions of surfaces and finishing materials of building construction or may penetrate fire-resisting walls. In these circumstances, electrotechnical products, when exposed to an external fire, shall be evaluated from the standpoint of their contribution to the fire hazard in comparison to the building materials or structure lacking the installation of electrotechnical products.

The transmission, distribution, storage and utilization of energy of any type has the potential to contribute to fire in most buildings.

The most frequent causes of ignition are overheating and arcing. The frequency of ignition will depend on the types of materials used in the construction of the system.

Contrary to the commonly held belief that most electrical fires are caused by a "short circuit," electrical fires may be initiated from one or a combination of circumstances, including external nonelectrical sources. These circumstances can include improper installation, utilization or maintenance conditions (for example, operation under overload for extended periods, operation under conditions not provided for by the manufacturer or contractor, inadequate heat dissipation, clogged ventilation systems, etc.)

The fire hazard of electrotechnical products depends upon the selection and arrangement of materials, components, and subassemblies and upon the processing, assembly and relative position of these items.

The required properties can generally be achieved by using parts and/or circuit design and protection techniques that, in conditions of overload or failure, avoid ignition or restrict the propagation of fire and spread of flame should ignition occur.

The best method for testing electrotechnical products with regard to fire hazard is to duplicate exactly the conditions occurring in practice. In most instances this is not possible. Accordingly, for practical reasons, the testing of electrotechnical products with regard to fire hazard is best conducted by simulating as closely as possible the actual effects occurring in practice.

Parts of electrotechnical equipment which might be exposed to excessive thermal stress due to electric effects, and the deterioration of which might impair the safety of the equipment, shall not be unduly affected by heat and by fire generated within the equipment.

Parts of insulating material or other solid combustible material which are liable to propagate flames inside the equipment may be ignited by glowing wires or glowing elements. Under certain conditions (for example, a fault current flowing through a wire, overloading of components, or bad connections) certain elements may attain a temperature such that they will ignite parts in their vicinity.

Electrotechnical products frequently become involved in fires. However, except for certain specific cases (e.g., power generating stations, mass transit tunnels, computer rooms, etc.), electrotechnical products are not normally present in sufficient quantities to form the major source of fuel for a fire.

The tolerable risk is the result of a balance between the ideal of absolute safety and demands to be met by a product, and factors such as benefit to the user, suitability for purpose, cost and effectiveness, etc. Tolerable risk is achieved by the iterative process of risk assessment which combines risk analysis and risk evaluation.

When developing a standard, it is the task of a committee to consider both the intended use and reasonable foreseeable misuse of a product.

To prepare a standard the committee has to identify the hazards arising from all stages and conditions for the use of the product including installation, maintenance and disposal. They have to estimate and evaluate the risks arising from the hazards identified and judge if the risks are tolerable. If the risks are not tolerable they have to be reduced to a tolerable level.

Safety is dealt with in standards work in many different forms in all areas of technology and for most products. With the increasing complexity of products entering the market, it is obvious that safety has gained considerable importance in our contemporary world.

There can be no absolute safety—some risk will remain—and therefore a product can only be relatively safe.

The practical side to this risk philosophy has been published as a group of tests developed by IEC/TC 89 in the IEC 60695 series of publications. They cover the following:

Preselection tests—combustion characteristics tests made on materials, piece parts or assemblies;

Nonflame tests—a glowing-wire test to simulate the effects of localized thermal stresses;

Flame tests—Bunsen burner type flame tests (50 W to 1 kW) to simulate the effects of an open flame;

Heat release and surface spread of flame—guidance on methods to determine the effect of heat release and surface spread of flame;

Corrosivity tests—assessment of corrosion damage from fire effluent;

Smoke opacity tests—assessment of the obscuration hazard of vision caused by smoke opacity;

Toxicity tests—guidance on the applicability of the methodology for minimizing the toxic hazard from fire effluent;

Mechanical tests—resistance to heat of nonmetallic materials and parts, and the resistance to complete products to abnormally elevated ambient temperatures.

Working Groups

IEC/TC 89 currently has eight working groups represented by 68 experts from 14 countries. They are:

WG1—Terminology
To give guidance on the definition of new terms and approved definitions.
WG 2—General Guidance
To prepare general guidance including guidance on fire scenarios and total fire hazard analysis.
WG 3—Smoke Opacity and Corrosivity of Fire Effluent
To prepare guidance on the use of appropriate test methods for evaluation of the effects of fire effluents on electrotechnical products and the effects of fire effluents from electrotechnical products as a hazard to life and property.
WG5—Nonflame Sources
To prepare methods and guidance for fire hazard assessment tests using nonflame ignition sources. To study and compare tests for the resistance to abnormal heat of parts of electrotechnical equipment made from nonmetallic materials.
WG 6—Flame Sources
To prepare methods and guidance for fire hazard assessment tests using flame (ignition) sources.
WG 7—Toxic Hazard
To prepare guidance on the minimization of toxic hazards from electrotechnical products involved in fires, and in pyrolysis, including abnormal/fault operating conditions.
WG 8—Heat Release and Surface Spread of Flame
To prepare guidance on methods for the determination of heat release and surface spread of flame for electrotechnical products involved in fires.
WG 9—Burning Properties of Insulating Liquids
To prepare guidance on the minimization of fire hazard arising from the use of insulating liquids in electrotechnical products.

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IEC 60695-4:1993, Terminology concerning fire tests.

IEC 60695-7-1:1993, Guidance on the minimization of toxic hazards due to fires involving electrotechnical products—General guidance.

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APPENDIX

Overview of Current Publications

IEC Publication 60695-	Title	IEC Publication Date
1-1	Guidance for assessing fire hazard of electrotechnical products—General guidance	1995–03
1-2	Guidance for the preparation of requirements and test specifications for assessing fire hazard of electrotechnical products—Guidance for electronic components	1982–01
1-3	Guidance for the preparation of requirements and test specifications for assessing fire hazard of electrotechnical products—Guidance for use of preselection procedures	1986–09
2-1/0	Test methods—Glow-wire test methods—General	1994–03
2-1/1	Test methods—Glow-wire test methods—Glow-wire end-product test and guidance	1994–03
2-1/2	Test methods—Glow-wire test methods—Glow-wire flammability test on materials	1994–03
2-1/3	Test methods—Glow-wire test methods—Glow-wire ignitability test on materials	1994–03
2-2	Test methods—Needle-flame test	1991–05
2-4/0	Test methods—Diffusion type and premixed type flame test method	1991–08
2-4/1	Test methods—1 kW nominal premixed test flame and guidance	1991–04
2-4/2	Test methods—500 W nominal test flames and guidance	1994–03
2-20	Glowing/Hot wire based test methods—Hot-wire coil ignitability test on materials	1995–08
3-1	Examples of fire hazard assessment procedures and interpretation of results—Combustion characteristics and survey of test methods for their determination	1982–01
4	Terminology concerning fire tests	1993–10
5-1	Assessment of potential corrosion damage by fire effluent—General guidance	1993–06
5-2	Assessment of potential corrosion damage by fire effluent—Guidance on the selection and use of test methods	1994–12
6-30	Guidance and test methods on the assessment of obscuration hazard of vision caused by smoke opacity from electrotechnical products involved in fires—Small scale test method: Determination of smoke opacity—Description of the apparatus	1996–10
7-1	Guidance on the minimization of toxic hazards due to fires involving electrotechnical products—General	1993–11
7-3	Toxicity of fire effluent—Use and interpretation of test results	1998–12
7-4	Guidance on the minimization of toxic hazards due to fires involving electrotechnical products—Unusual toxic effects in fires	1995–05
9-1	Surface spread of flame—General guidance	1998–12
10-2	Guidance and test methods for the minimization of the effects of abnormal heat on electrotechnical products involved in fires—Method for testing products made from non-metallic materials for resistance to heat using the ball pressure test	1995–08
11-10	Test flames—50 W horizontal and vertical flame test methods	1999
11-20	Test flames—500 W flame test methods	1999
707	Methods of test for the determination of the flammability of solid electrical insulating materials when exposed to an igniting source	1998–12

Current Projects

IEC Publication 60695-	Title	Stage
1	General guidance	
1-1	General guidance—Second edition	under review
1-1	Amendment No. 1—Informative annex: Illustrative examples of hazard assessment	CDV*
1-30	Use of preselection procedures	CD†
1-40	Insulating liquids	preliminary
2	Glowing/hot-wire based tests	
2-10	Glow-wire apparatus and common test procedure	CDV
2-11	Glow-wire flammability test method for end products	CDV
2-12	Glow-wire flammability test method for materials	CDV
2-13	Glow-wire ignitability test method for materials	CDV
3	Assessment procedures	
3-1	Combustion characteristics test survey	under review
5	Corrosivity of fire effluent	
5-1	General guidance	under review
5-2	Summary and relevance of test methods	under review
6	Smoke obscuration	
6-1	General guidance	CDV
6-2	Summary and relevance of test methods	CDV
6-31	Small scale static test: Materials	final
7	Toxicity of fire effluent	
7-1	General guidance	under review
7-2	Summary and relevance of test methods	preliminary
7-50	Toxic potency: Apparatus and test method	preliminary
7-51	Toxic potency tests: Calculation and interpretation of test results	preliminary
8	Heat release	
8-1	General guidance	CDV
8-2	Summary and relevance of test methods	CDV
9	Surface spread of flame	
9-2	Summary and relevance of test methods	CD
10	Abnormal heat	
10-2	Ball pressure test—Amendment	CD
10-3	Mould stress relief distortion test	CD
11	Test flames	
11-3	500 W flames: Apparatus and confirmation test method—Amendment No. 1	CDV
11-4	50 W flames: Apparatus and confirmational test methods	CDV
11-30	History and development 1979–1995	CD

† CD—Committee Draft
* CDV—Committee Draft for Vote

Daniel J. Strachan[1]

NEMA Activities and Their Relation to ASTM Committee D09

Reference: Strachan, D. J., "**NEMA Activities and Their Relation to ASTM Committee D09,**" *Electrical Insulating Materials: International Issues, ASTM STP 1376,* M. M. Hirschler, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

Abstract: This paper details the history of the National Electrical Manufacturers Association. It discusses the three core competencies of NEMA. It also goes into detail of the NEMA Committee structure and the process of getting a document published as a NEMA standard. Also discussed is NEMA's ongoing relationship with the U.S. Department of Defense and the working relationship that various NEMA product sections have had with ASTM D09 over the years. The paper concludes with a presentation of current working projects in NEMA that might be of interest to ASTM D09 members.

Keywords: NEMA, electroindustry, core competencies, standard(s), section ballot, Codes and Standards Committee, military specifications, ASTM D09, forum

What Is NEMA?

In 1926, several members of the electrical industry in New York City founded NEMA, short for the National Electrical Manufacturers Association. These gentlemen thought that the electrical industry was getting more and more prominent in American society and there needed to be an organization to watch out for the best interests of the industry and to develop technical standards for the industry. For many years NEMA did not have a paid staff, but rather had volunteers from NEMA member companies. This practice was done away with many years ago as it was thought that paid staff would not have conflicts of interest.

NEMA was headquartered in mid-town Manhattan until 1976 when it was moved to Washington, DC. This move was due to the fact that lobbying was becoming more and more important to the electrical industry.

In 1995, NEMA once again moved its headquarters from Washington, DC to Rosslyn, VA. Rosslyn is the area of Arlington that is on the Potomac River. NEMA presently resides on two of the upper floors of a skyscraper and has one of the best views of Washington, DC in the area.

Today NEMA has grown from a small association to one of the most noted and influential trade associations in North America. There are presently 93 full-time staff members representing over 60 product areas from lighting to cable to MRIs. Over 600 companies claim membership in NEMA. There is a great diversification of companies in NEMA. Some are huge such as GE, Litton, Siemens, and Leviton, and there are small "mom and pop" companies with just 20 or fewer employees. NEMA gives all these companies an equal voice.

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NEMA's Core Activities

Under Malcolm O'Hagan, NEMA's president, the association was reorganized in 1993 to emphasize three core competencies: government affairs, economics, and standards development.

NEMA works under the auspices of its Board of Governors, which is composed of representatives from each of the nine product divisions of NEMA.

The Government Affairs Department of NEMA handles all lobbying activity for the association. Lobbying is not just confined to Capitol Hill. NEMA has lobbied for members' interest on state and local levels as well as federal. Trade and environmental issues fall under the Government Affairs Department. NEMA is well known to EPA and the Department of Commerce. NEMA was very instrumental in the NAFTA Agreement and also played a role in the GATT talks. NEMA's Vice-President of Government Affairs, Tim Feldman, is one of the most recognized people on the "hill."

The Economics Department, formerly known as the Statistics Department, is responsible for several different areas. Many product sections in NEMA have statistical market reports that reflect sales of certain products for a certain period of time. These reports are based upon sales and market data provided to NEMA by member companies. These reports are voluntary and are only provided to those companies that participate. The data that are reported to the NEMA Economics Department are kept confidential, even to the point that only employees of that department are allowed into that portion of the NEMA office.

The NEMA Economics Department also handles economic forecasting. Working with NEMA member companies, this department develops forecasting models for certain product groups. This provides data to NEMA member companies that cannot be obtained anywhere else.

NEMA Standards

The third core competency of NEMA is its standards development. Standards development is the primary responsibility of the NEMA Engineering Department. This is the largest department in the association, representing 6 of every 10 NEMA employees.

Frank Kitzantides, NEMA's Vice President of Engineering, heads the NEMA Engineering Department. He has several technical directors who report to him. Each one of these technical directors has several program managers who report to them. Most of those NEMA staff who one would find facilitating a NEMA technical committee are program managers.

NEMA has over 300 standards presently in publication. Although the majority of these standards are owned and copywritten by NEMA, a fair number of them are joint standards with other standards developing organizations. In the lighting division, the vast majority of standards are done in cooperation with ANSI. In the power and control cable section, joint standards between NEMA and the Insulated Cable Engineers Association (ICEA) are the norm. NEMA also has joint standards with IEEE in several product sections.

Each product area is known as a "Section" and has a committee comprised of one representative from each member company on that section. The section members have jurisdiction over what activities are taken upon by that particular section's technical committee, subcommittees, and task forces. The section also approves the yearly budget for operations of that section.

NEMA and ASTM standards are developed in much the same way. Most start out as an outline developed by a task force of some product section. This outline is researched and filled in until a working draft is done. At this point, a subcommittee or technical committee usually handles the working draft. This draft is reworked and reworked, sometimes over a period of months, sometimes over a period of years, until it is ready for letter ballot. Consensus of the committee is usually achieved before this draft standard is sent to letter ballot.

The draft standard is finally sent out to the product area's section for, what is known as, "section ballot." This is a 30-day ballot. After the ballot period, the responses are tallied, all comments are documented, and the ballot results are given back to the program manager.

NEMA requires that all comments received on a ballot be addressed. These include comments that are received on an affirmative ballot. All comments are to be reviewed and the disposition of each comment must be recorded in the minutes of the meeting of the committee that developed the letter ballot. It is the duty of the program manager to see that this process is strictly followed.

It is the goal of every technical committee in NEMA that there be no negative comments on a ballot. This is probably the goal of every committee in ASTM D09 also! Negative comments are received on many NEMA ballots. Unlike some other standards developing organizations, NEMA does not require that all negatives be resolved prior to final association approval, only that they be addressed. Most of the time, the committees do address the negative in such a way that the company submitting the negative vote changes their vote.

The final hurdle for a NEMA standard is approval by the NEMA Codes and Standards Committee. This committee is comprised of technical experts from each of NEMA's nine product divisions. Although the NEMA Codes and Standards Committee approves most standards, it is not unheard of for this committee to order a draft standard to be sent back out for approval or to have it changed. When this committee approves the draft of the standard, it is considered a de facto NEMA standard. It is a common misconception among people that a NEMA standard is official when it is published.

At this point the NEMA Communications Department formats the standard; staff and members review a "blue line" of the standard for editorial clarity, and the standard is published.

NEMA and the Department of Defense

In the early 1990s the "Perry Memorandum" was introduced to the DoD. This stated that the Department of Defense would use commercial specifications whenever possible in lieu of military specifications ("mil specs").

NEMA has been on the forefront of the conversion of mil specs to NEMA standards. This can happen either by taking an existing mil spec and rewriting it into a NEMA standard format or by revising an existing NEMA standard to meet the needs of the DoD. Further, new standards being developed in NEMA take into account the needs of the DoD. When these standards are published, the DoD adopts them.

This process can be cumbersome at times. NEMA will not just do a word-for-word conversion of a mil spec and republish that as a NEMA standard. Rather, a mil spec is put into a NEMA standard format and then a technical committee begins to make technical changes to this standard. This is done with the participation of DoD personnel. It is not uncommon for a 300-page mil spec to be converted to a 70-page NEMA standard.

The majority of this work has occurred in the High Performance Wire and Cable Section, but has also occurred in other NEMA sections such as Flexible Insulation and Mica.

NEMA and ASTM D09

NEMA has had a good working relationship with ASTM D09 for many years. Dan Strachan is the liaison between NEMA and D09.

The scope of ASTM D09 covers several product sections of NEMA, High Performance Wire and Cable, Tubing and Sleeving, and Flexible Insulation and Mica. The vast majority of active members of these NEMA product sections are active in D09. This is evident in the fact that the Flexible Insulation and Mica Technical Committee will only meet in conjunction with ASTM D09 meetings.

NEMA has participated in ASTM D09.20 and D09.18 meetings. The information that is gathered at these meetings is reported back to like NEMA technical committees. The work that goes on in ASTM D09 is of great interest to NEMA members. The reverse is also true.

Although the scope of D09 covers several NEMA product sections, the majority of NEMA's interest in the activities of D09 comes for the NEMA High Performance Wire and Cable Section. This section, known as 7-HW, is one of the largest product sections of NEMA, with over 34 member companies. There are four major product groups that fall under this section: premise wiring, airframe wiring, shipboard cable, and electronics cable. Unlike most other NEMA sections, letter ballots are only sent to section members with representation on the particular subcommittee that developed the standard. By having balloting done in this manner, companies are voting on letter ballots for products that they manufacture.

There are five technical subcommittees, with four of them having task forces under them. It is not uncommon for this section to have a 3-day battery of meetings where each task force and each subcommittee meets. Each meeting is done separately, with its own agenda and minutes.

The Test Procedures Subcommittee of the NEMA High Performance Wire and Cable Section is the one subcommittee who has the greatest interest in the activities of ASTM D09. This subcommittee handles all issues relating to flame testing, flame propagation, etc. It also provides comment on IEC and UL ballots dealing with cable testing and develops NEMA High Performance Wire and Cable Section standards dealing with test methods.

Presently, this subcommittee has just completed development of appendices to NEMA WC 61 and NEMA WC 62, the high frequency spark test and impulse spark test standards, respectively, that were published as similar standards. However, readers would be reading one standard and would not know that there were additional test methods that appeared in the other standard. These appendices will alert the reader to tests that appear in each standard and will harmonize the language and flow of each of the two standards. The revisions to these two NEMA standards were expected to be published in mid-1999.

NEMA Standards of Interest

NEMA WC 69-2000 is entitled "Standard for Cable Power, Electrical, and Cable Special Purpose, Electrical Shielded and Unshielded." This standard is being rewritten to supercede MIL-W-27500 of the same title. The Military Specification Task Force of the NEMA High Performance Wire and Cable Section's Aerospace Subcommittee is developing this standard. Work on this standard began in 1998, and it is expected to be published in late 2000.

NEMA WC 72-1999 is entitled "Continuity of Coating Testing for Electrical Conductors." The Polysulfide Task Force of the NEMA Test Procedures Subcommittee developed this standard which is a guide for testing the coating of electrical conductors. It is scheduled for letter ballot and publication this year.

NEMA WC 73-1999, "Wire Selection Guidelines for Wires Rated at 200–450°C," is a joint project of NEMA and SPI. The Electronics Subcommittee is in charge of this standard which is being developed to standardize wire selection for certain high temp applications (i.e., hot runner systems).

Presently, NEMA is getting ready to publish NEMA WC 66-1999 "Performance Standard for Category 6 and Category 7 100 Ohm Shielded and Unshielded Twisted Pair Cables."

This is the first North American standard to address "Category 6" cables. The Premise Wiring Subcommittee of the NEMA High Performance Wire and Cable Section developed WC 66. DoD approached NEMA several years ago with a request for such a standard due to the fact that Category 5 cable was not applicable to many of the DoD's top-secret applications. It is expected that this standard will be published in 2000. Presently, work is being done on Revision 1 to NEMA WC 66-1999.

Participation in NEMA

In the past, participation in NEMA technical committees and subcommittees was restricted to NEMA member companies. That is no longer true, especially in the wire and cable division of NEMA. Although membership in NEMA is only open to manufacturers, NEMA understands that in order to have a standard that can be valid, the input from suppliers and customers must be considered in the development of that standard.

It is not uncommon for more than 50% of the attendees at NEMA technical committees to be from companies that are not NEMA members, but are interested in the work of that committee. Non-NEMA member companies can become official members of NEMA task forces. It is by having the input from end-users, suppliers and others from the very beginning of the development of a standard that NEMA can assure that the standard meets the needs of all parties.

There is no registration fee to attend a NEMA technical committee meeting; the only requirement is that you contact NEMA before the meeting and let them know that you are interested in attending.

In 1998, NEMA launched its FTP site called "NEMA Forum." Drafts of standards are posted here for comment. This enables one to provide comment on a standard between meetings. It also enables one to provide comment and not have to attend a meeting in order to supply that comment. As with NEMA meetings, Forum is open to any interested party and is free of charge. It can be accessed through NEMA's web site: www.nema.org.

Conclusion

NEMA, like ASTM, is changing with the times. Some standards are in a constant revision process due to changes in technology, while others are quite dormant. More and more drafts are being reviewed and developed electronically. Letter ballots are being done electronically. NEMA knows that its members' time is valuable and is not to be wasted by doing busy work, but by providing results to actions taken by its members.

Although NEMA and ASTM differ in the composition of their committees and the responsibilities of their staffs, both NEMA and ASTM will always be member-oriented and member-driven. It is by working together that NEMA and ASTM have been able to develop world-class standards that are the cornerstone of today's electroindustry.

Electrical Insulating Fluids

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Specification Issues Associated with the Development of an Agriculturally Based Biodegradable Dielectric Fluid

Reference: Claiborne, C. C., Oommen, T. V., and Walsh, E. J., "**Specification Issues Associated with the Development of an Agriculturally Based Biodegradable Dielectric Fluid,**" *Electrical Insulating Materials: International Issues, ASTM STP 1376,* M. M. Hirschler, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

Abstract: A fully biodegradable dielectric fluid based on high oleic acid vegetable oil has been developed for use in electrical equipment. The development of such a fluid for use in equipment such as electrical transformers has naturally caused it to be compared to more conventional mineral oil based insulating fluids and to high-temperature hydrocarbons and silicones for which specifications already exist. The selection of the base oil and its modifications relied heavily on preexisting standards such as ASTM D 3487 and ASTM D 5222. Because such a vegetable based fluid in practical terms does not completely match any of these fluids exactly in its characteristics, commercial implementation may lead to a desire for a standard specification describing its unique set of properties.

In addition, the biodegradable nature of the fluid has required that standard tests not used in the electrical industry, such as CEC L-33-A-94, developed for two-stroke-cycle outboard engine oils, be applied to the material in question. ASTM has subsequently developed a standard guide, ASTM D 6006 for hydraulic fluids, but relatively little work was performed on dielectric fluids at the time of the development of the biodegradable fluid discussed in this paper. Comparisons with other potential tests will be discussed and a discussion of the relative merits of these tests for dielectric fluids will be presented.

Keywords: dielectric fluid, agricultural, vegetable, biodegradable, specifications, standards, transformers

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Background

In recent years there has been increased interest in the development of a dielectric fluid of a more environmentally benign nature than conventional mineral oil based fluids for use in electrical equipment such as transformers. This new fluid should have characteristics which would reduce the perceived and legal problems of release into the environment in the event of spills and leaks. From the outset, it was recognized that this fluid should possess a high degree of biodegradability, something for which conventional mineral oil was not known. At the same time, it must have the functional characteristics for long term performance in equipment, on the order of 20 years or more. For use in electrical equipment, it must possess both sufficient dielectric strength to be an effective insulating fluid and also have thermal properties enabling its function as an effective heat transfer agent and cool the electrical equipment during operation.

Significant steps have been made by several companies in developing dielectric fluids for electrical uses which have inherent properties leading them to be regarded as environmentally friendlier than conventional fluids. As these new fluids near commercialization, there will be a desire for specifications which will enable and direct the use of these fluids in electrical apparatus.

The development of these fluids relied heavily on the existence of standards for dielectric fluids such as "Standard Specification for Mineral Insulating Oil Used in Electrical Apparatus", ASTM D 3487-88 (Reapproved 1993), and its associated standards. This standard was published originally in 1976 and was an outgrowth of previous standards such as "Specification for Uninhibited Mineral Oil", ASTM D 1040, published originally in 1949. The traditions of the use of mineral oil in transformers date back to the late 1800's when electrical transformers were first invented and used. The existing standard ASTM D 3487 together with "Standard Guide for High Fire-Point Electrical Insulating Oils of Petroleum Origin", ASTM D 5222-92, provided very important guidelines in the development of a new dielectric fluid destined for electrical apparatus. In a similar manner, "Standard Specification for Silicone Fluid Used for Electrical Insulation", ASTM D 4652-92, describing a non-hydrocarbon based dielectric fluid is useful in considering new dielectric fluids. It is obvious that these existing standards were developed to fulfill the needs of the industry and provide benchmarks for choosing fluids which would function satisfactorily in electrical equipment. In many senses, ASTM D 3487 was relied upon in the development of high fire-point fluids because they served similar purposes in a functional sense with mineral oils. Thereafter, ASTM D 5222, the guide for high fire-point insulating oils (also known as high temperature hydrocarbons or HTH's), and D 4652, the specification for silicone fluids, were developed using ASTM D 3487 as a basis for many of the tests that were carried out on these dielectric fluids to determine their suitability in the electrical apparatus for which they were designed and developed.

Similarly, it was to be expected that any proposed specifications for new environmentally friendly fluids should heavily rely upon the existing specifications and guides in their development. With that in mind, the remaining discussion will describe some differences and similarities that have been discovered in the course of the development of a dielectric fluid based upon agricultural sources or in other words, a dielectric fluid refined and produced from vegetable based oils rather than from mineral based oils.

Standard Specifications for Dielectric Fluids

ASTM D 3487 specifies three types of properties that shall be considered when evaluating a new mineral insulating oil: physical, electrical and chemical. In developing a new dielectric fluid, one criteria is stated in the scope section of ASTM D 3487: "...satisfactorily maintain its functional characteristics in its application in electrical equipment." It other words, such a new fluid must not only possess desirable functional characteristics in the test tube, it must also retain them after filling the electrical equipment and finally after operating the equipment over an extended period of time.

Physical Properties

Considering the physical characteristics, D 3487 has one parameter that has no merit for a vegetable based oil. The aniline point, "Test Method for Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents", ASTM D 611 - 82 (1993), is most often used to estimate the aromatic hydrocarbon content of petroleum products. Because vegetable oils suited for dielectric uses are not generally expected to contain aromatic compounds, this test is not expected to be necessary for the evaluation of agriculturally based fluids.

Furthermore, as is the case for silicone fluids, interfacial tension, "Interfacial Tension of Oil Against Water by the Ring Method" ASTM D 971 - 91, is inappropriate for a vegetable oil based fluid. In both cases, while a measurable value of the interfacial tension can be determined, fundamental characteristics of these fluids cause the fluid to be inherently more miscible with water than petroleum hydrocarbons. In the case of vegetable oil based dielectric fluids this is due to the more hydrophilic nature of the triglycerides such as oleate esters and other such fatty acid esters comprising the oil.

The significance of this method is that it gives an indication of the presence of hydrophilic compounds which contaminate the fluid being studied. In the case of hydrocarbon fluids, the immiscibility of the fluid with water leads to a relatively high, greater than about 40 mN/m (dynes/cm), value for a new, clean dielectric fluid. When this fluid becomes contaminated with hydrophilic substances, this value can drop significantly as the contaminants encourage the oil to mix with water. For silicone fluid and vegetable oil based dielectric fluids, it has been determined that the starting value of interfacial tension is on the order of about 20 mN/m which reduces the margin of measurement by approximately half for contaminated material.

The remaining tests in the physical section, color "ASTM Color of Petroleum Products (ASTM Color Scale)", D 1500-96, flash point, "Flash and Fire Points by Cleveland Open Cup", D 92-97, pour point, "Pour Point of Petroleum Oils", D 97-96a, visual examination, "Visual Examination of Used Electrical Insulating Oils of Petroleum Origin in the Field", D 1524-94, and viscosity, "Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)", D 445-

97, all have functional validity and are generally considered in measurements on the vegetable oil based fluid. There may be notable differences in some of these values for the vegetable fluid as compared to D 3487 which is dedicated to petroleum based fluids. Fundamental physical properties of the vegetable oil such as coefficient of expansion, dielectric constant, specific heat and thermal conductivity may also be directly compared to the values given in the Appendix X1. of D 3487.

Electrical Properties

As the vegetable oil is destined for electrical apparatus, it is reasonable to assume that the tests specified in D 3487 in the electrical section will all have relevance to the new fluid. Dielectric breakdown voltage at 60 Hz, "Dielectric Breakdown Voltage of Insulating Liquids Using Disk Electrodes", D 877-87 (1995), and "Dielectric Breakdown Voltage of Insulating Oils of Petroleum Origin Using VDE Electrodes", D 1816-97, "Dielectric Breakdown Voltage of Insulating Oils of Petroleum Origin Under Impulse Conditions", D 3300-94, "Gassing of Insulating Oils Under Electrical Stress and Ionization (Modified Pirelli Method)", D 2300-85 (1997), and "Test Method for Dissipation Factor (or Power Factor) and Relative Permittivity (Dielectric Constant) of Electrical Insulating Liquids", D 924-92, can all be readily measured on an agriculturally based dielectric fluid. Indeed, some of these same tests were used to choose candidates for production of the vegetable oil based dielectric fluid. Although the fluid has not yet been tested according to "Stability of Insulating Oils of Petroleum Origin Under Electrical Discharge", D 6180-97, this test should also prove satisfactory as a measurement technique for this fluid.

Chemical Properties

For chemical tests, only one test is not strictly applicable to our formulation of the vegetable oil. The tests for oxidation inhibitor, "Test Method for 2,6-Ditertiary-Butyl Para-Cresol and 2,6-Ditertiary-Butyl Phenol in Electrical Insulating Oil by Infrared Absorption", D 2668-96, or "Analysis of 2,6-Ditertiary-Butyl Para-Cresol and 2,6-Ditertiary-Butyl Phenol in Insulating Fluids by Gas Chromatography", D 4768-96, are linked inextricably to the use of 2,6-ditertiary-butyl para cresol (DBPC) and 2,6-ditertiary-butyl phenol (DBP). Both of these inhibitors are not used in our formulation of the vegetable oil based dielectric fluid. Other food grade antioxidants, presently being treated as proprietary, are used to protect this fluid. Appropriate tests are being developed to monitor the levels of inhibitor present in the starting fluid and in the fluid as it ages in the electrical apparatus. As a consequence, both of these particular ASTM tests, appropriate for mineral oil based fluids, were not considered in the development of the new dielectric fluid nor are they expected to be of any significance in the characterization of the fluid.

Several of the remaining chemical tests, oxidation stability "Oxidation Stability of Mineral Insulating Oil", D 2440-97, and "Oxidation Stability of Mineral Insulating Oil", D 2112-95, water content, "Water in Insulating Liquids (Karl Fischer Reaction Method)", D 1533-96, and neutralization number, "Acid and Base Number by Color-

Indicator Titration"), D 974-97, certainly have significance in choosing a vegetable based dielectric fluid. Two other tests, corrosive sulfur, "Corrosive Sulfur in Electrical Insulating Oils", D 1275-96a and PCBs in oil, "Analysis of Polychlorinated Biphenyls in Mineral Insulating Oils by Gas Chromatography", D 4059-96, have validity from a functional standpoint, but not necessarily from their origin in the dielectric fluid. In the case of corrosive sulfur, there is a possibility that sulfur found in the crude oil from which the mineral oil is refined can be of a corrosive nature. For vegetable oils, it is possible that the plant from which the seed has been taken can take up sulfur from the soil in which it is grown. It is therefore not desirable to exclude such a measurement in the qualification of the vegetable oil. For PCB contamination, the source of vegetable oil contamination would again be uptake from a contaminated site in which the plants were grown. While not very likely, it is reasonable to assume that the PCB contamination level will continue to be a legal requirement by virtue of the vegetable based oil's function as a dielectric fluid. For mineral oil, PCB contamination is no longer as likely in production of the mineral oil based dielectric fluid as it once was when transfer vessels and reuse of the oils could lead to cross contamination. This once nearly ubiquitous nature of PCB contamination may also also still play a small role for vegetable fluids but it is less likely to be a problem with food grade production of the vegetable oil.

Biodegradability

When developing and choosing an agriculturally based dielectric fluid, one of the most important characteristics of these fluids is their biodegradability. No standard yet exists for dielectric fluids as regards this parameter. A few studies have been carried out which investigated the biodegradability of mineral oil based fluids and found them to have relatively low biodegradability. The test which has most commonly been used worldwide for such oils was developed originally for two stroke cycle outboard engines in water, CEC L-33-A-94, published by the Coordinating European Council (CEC) in Paris, France.

This test method uses test flasks which contain a mineral medium (aqueous solutions of various salts), the test oil and an inoculum (obtained from the effluent of a sewage treatment facility) which are incubated along with poisoned blanks for up to 21 days. Control blanks with reference materials (specific to the method) are also run in parallel. At the end of the incubation period, the flask contents are sonically vibrated, acidified and extracted with 1,1,2-trichlorotrifluoroethane. IR analysis is used to measure the C-H stretch maximum absorption of the CH_2-CH_3 band at a wavelength of 2930 cm^{-1}. These absorption values are then used to calculate the residual oil contents of the test and poisoned flasks. This residual oil value is converted to a biodegradability percentage by comparing the results from the poisoned and test flasks.

In addition, ASTM has approved a "Standard Guide for Assessing Biodegradability of Hydraulic Fluids", D 6006-97a, which describes two other European Standards, from "OECD Guidelines for the Testing of Chemicals", OECD 301B, (the Modified Sturm Test), and OECD 301F (the Manometric Respirometry Test), both promulgated by the Organization for Economic Cooperation and Development, also based in Paris, France, as well as several other tests. It also references some other biodegradability standards for lubricants, such as the "Test Method for Determining the Aerobic Aquatic Biodegradation of Lubricants and Their Components", ASTM D 5864-95.

If biodegradability is to become accepted as a specification for any new dielectric fluid based on vegetable oil, then the accepted method will probably be developed with these methods as background considerations. Such an acceptance of the type of biodegradability test will be an example of the consensus building process of the committee considering the specification, most likely ASTM D 27, Electrical Insulating Liquids and Gases.

Oxidative Stability

Oxidative stability is an important parameter that has a long tradition in the electrical industry. Not only have transformers been constructed which intake oxygen rather freely, so-called free-breathing transformers, but also oxidation degradation of transformer oils is a well known phenomenon because it is extremely difficult to banish oxygen entirely from an operating transformer.

It is generally accepted that oils stemming from agriculturally produced sources such as grains and seeds will have an oxidative stability inferior to oils produced from crude mineral oil. This lower oxidation stability stems primarily from the presence of double bonds (unsaturation) within the molecular chains. It depends in part on the degree of unsaturation and in part on the positional arrangement of the double bonds, e.g., conjugated double bonds. The oxidation proceeds primarily by a free radical mechanism reacting with oxygen to form lower molecular weight entities, some of which may be volatile, and cross-linked chains which may lead to polymerization of the material. There are two means of lessening the impact of this lower oxidative stability: (a.) formulation of the oil itself, such as choosing an oil with high monounsaturated content, thereby reducing the number of unsaturated sites which can react with oxygen and avoiding the conjugated double bonds and (b.) addition of oxidation inhibitors which sacrificially and preferentially react with oxygen present within the transformer.

Oxidation inhibitors have been used for many years in mineral oil filled transformers with a high tendency toward oxidative degradation. such as distribution transformers, which may take on oxygen when pressure buildup due to stressing the transformers causes pressure relief valves to open, briefly allowing air to flow into the transformers. In the previously mentioned free-breathing transformers where the admission of oxygen is only slightly limited by a desiccant breather, which actually does little to prevent oxygen admission but rather serves to limit moisture intake, oxidation inhibitors are also used in the mineral oil.

Measurement of the oxidative stability of transformer oil has traditionally been achieved in one of two ways, with D 2440 and by D 2112, the latter sometimes referred to as the RBOT test. In addition, we chose to measure the oxidation stability of our agriculturally based dielectric fluid employing two methods used in the vegetable oil and meat industries, the Oil Stability Index (OSI) test specified by the American Oil Chemists Society in AOCS Test Method Cd 12b-92 and to a lesser extent, the Active Oxygen Method (AOM) specified in AOCS Method Cd-57. The Active Oxygen Method measures the time required for a sample of fat or oil to attain a predetermined peroxide value which is in turn determined by titration. The AOM test has been relegated to a "surplus" status by the AOCS and consequently has less importance as a measure of the oxidation stability. The OSI test utilizes a method of collecting the effluent volatiles from a specified volume of oil bubbled with air and held at 110°C into a vessel containing deionized water. The conductivity of the water is measured and when the acidic products from the oxidized oil suddenly increase, the OSI time is noted. This method allowed for a more automated means of determining the oxidation stability of the initial trials of oil and various oxidation inhibitors.

Aside from the choice of the base vegetable oil, one has a variety of oxidation inhibitors to choose from. The conventional transformer oil inhibitor, 2,6-ditertiary-butyl para cresol (DBPC), was found to be rather wanting as a suitable inhibitor for any vegetable based oil that we studied. It must be recognized that different mechanisms of oxidation may require different inhibitors and numerous studies of various other inhibitors were necessary to arrive at a formulation which proved satisfactory. Eventually a suitable package of inhibitors was chosen as the ideal candidate for the oxidation inhibitor. This proprietary package of inhibitors was developed with several considerations in mind: The total content was deliberately held to a *de minimus* level in order to avoid affecting the biodegradability and at the same time limiting the inhibitor's influence on the electrical properties of the dielectric fluid.

Many of the trials of various inhibitor packages were evaluated with the vegetable oil industry methods mentioned above for the purpose of reducing the time involved in the testing. Reasoning that acceptance of the final formulation would be stronger if an industry accepted oxidation stability method were utilized, numerous tests were performed on the final choice of inhibitor package using ASTM D 2440 and ASTM D 2112, resulting in the parameters demonstrated in Table 1. Thus it can be recognized that agriculturally based oils can be formulated, which in the case of oxidation stability, meet the requirements of ASTM D 3487, Type I oils. Any specification developed for these oils may well include this as the oxidation stability limit to be met.

Suitability of Existing Specifications

Considering the values in Table 1, we can make certain generalizations as compared to mineral oils (D 3487), high temperature hydrocarbons (D 5222) and silicone fluids (D 4652). For agricultural fluids, the flash and fire points will far exceed the specification for mineral oils and will more nearly approach that of high temperature hydrocarbons (HTH's) or silicone fluids. Similarly, the higher viscosity of the vegetable fluid, differing from both HTH's and silicone, is significantly above that of mineral oil.

Both of these physical properties alone argue that a separate specification for vegetable based fluids should be developed. This specification will most likely incorporate select values that are present in the existing specifications with certain modifications where the fluid has properties that differ from those already specified in the existing specifications. The origin of the vegetable fluid argues most strongly that a separate specification is needed because many of the tests in the previous specifications are specific to the fluid of concern.

Table 1 - Typical Values of Agricultural Fluid vs. Specification/Guide Values for Mineral Oil, High Temperature Hydrocarbons and Silicone

Property: ASTM D 3487, D 4652 or D 5222 as appropriate[A]	Min. Oil	Agr. Oil	HTH	Silicone
Dielectric Strength, ASTM D 877, kV @ 25°C	30	42	42	35
ASTM D 1816, kV @ 25°C, 2 mm	56	74	61	
Dissipation Factor, ASTM D 924 @ 25°C	0.05	0.03	0.01	0.01
Dielectric breakdown, impulse, kV, ASTM D 3300	145	112		
Gassing tendency, D 2300, (µl/min), max	+30	+1		
Neutralization number (Acidity), (mg KOH/g), ASTM D 664	0.03	0.06	0.01	0.01
Color, ASTM D 1500	<0.5	<0.5	0.5-2	
Pour Point, ASTM D 971, (°C), max	-40	-15[B]	-24	-50
Flash Point, ASTM D 92, (°C), min	145	300		300
Fire Point, ASTM D 92, (°C), min	-	320	304-310	340
Specific gravity, ASTM D 1298, max	0.91	0.91	0.87	
Viscosity, kinematic, (cSt)				
@ 0°C	76	300	1800-2200	81-92
40°C	12	45	100-140	35-39
100°C	3	10	11.5-14.5	15-17
Biodegradability (CEC L-33-A-94, 21 days), %	-	97	-	-
Oxidative Stability				
72 hr acid num., ASTM D 2440, (mg KOH/gm)	0.15	0.3	0.2	
% sludge	0.5	none	0.01	
164 hr acid num., ASTM D 2440, (mg KOH/gm)	0.6	0.5	0.4	
% sludge	0.3	none	0.02	
Oxidative Stability, ASTM D 2112, (minutes)	195	200	800+	
Corrosive Sulfur, ASTM D 1275	none	none	none	
PCB's, ASTM D 4059, ppm	none	none	none	none
Nonmandatory Information, Appendix X1, D3487				
Coefficient of expansion, $°C^{-1}$, 25-100°C, $\times 10^{-4}$, ASTM D 1903	7-8	6.88	7.1	-
Dielectric constant, 25°C, ASTM D 924	2.2-2.3	3.2	2.2	-
Specific heat, cal/g, 20°C, ASTM D 2766	0.44	0.57	0.46	-
Thermal conductivity, cal/cm s °C, 20-100 °C $\times 10^{-3}$, ASTM D 2717	0.30	0.41	0.35	-

[A] Absence of a value indicates that it is not included in the specification or guide. Values for agricultural oil are typical values.

[B] The pour point specified here is without the use of pour point depressants. The use of such depressants or blends with other fluids may lower this value but care must be taken that other properties are not adversely affected.

Another large difference can be seen with the case of pour points. While vegetable fluids have a pour point even higher than that of high temperature hydrocarbons, we have studied the phenomenon carefully with distribution transformers. Distribution transformers have been chilled and held at temperatures as low as -50°C and energized without any apparent problems. Large power transformers which often have pumps to circulate the fluid will undoubtedly present special situations that must be addressed when the use of such a higher pour point dielectric fluid is contemplated.

Table 1 shows that the value obtained for the impulse breakdown strength, tested according to "ASTM Standard Test Method for Dielectric Breakdown Voltage of Insulating Oils of Petroleum Origin Under Impulse Conditions", D 3300-94, for the needle negative to sphere grounded configuration, is relatively lower than that for a mineral oil conforming to ASTM D 3487. This value is not included as part of the specification or guide for either silicone and high temperature hydrocarbons although it has been measured and found to be higher than that for mineral oil in both cases. For the vegetable oil, the opposite configuration (positive polarity) has been determined to be 94 kV/mm. This is attractive from a transformer design standpoint because even though the impulse strength is most commonly measured in the negative configuration because of consistency of measurement, the positive impulse strength is also important to the design. If the negative and positive impulse strength ratios are closer to unity one does not need to design from a worst case standpoint. This is not the case for other dielectric fluids where the needle positive values are much lower than the needle negative values.

We have found it useful to consider two other properties in the development of the vegetable oil dielectric fluid. Resistivity, "Test Method for Specific Resistance (Resistivity) of Electrical Insulating Liquids", ASTM D 1169-95, and conductivity "Standard Test Method for Electrical Conductivity of Liquid Hydrocarbons by Precision Meter", ASTM D 4308-95 can be quite useful in evaluating incoming raw materials and in the production of the final product. It is entirely possible that these may be desirable properties to consider in any specification of this fluid.

Vegetable oils, unlike mineral oils, have a much higher water solubility which is close to 1,200 ppm at room temperature. To set limits on dryness levels, we should use the concept of relative saturation. For example, if 10 ppm moisture is considered suitable for dry mineral oil (moisture saturation at 60 ppm at room temperature), the equivalent water content of vegetable oil would be approximately 200 ppm. Mineral oil as received may contain as much as 35 ppm water according to ASTM D 3487 which would be equivalent to about 700 ppm in vegetable oil. However, the acceptance level for vegetable oil should be much tighter and it is possible to have oil delivered at 150 ppm or below. We have strived to keep moisture levels below 100 ppm in electrical grade vegetable oil and something on the order of 100-150 ppm may be desirable for any specification of such a fluid.

Accelerated Aging Tests

In the course of developing and investigating this agriculturally based dielectric fluid, accelerated aging tests on actual transformers containing this fluid were conducted according to "Test Procedures for Thermal Evaluation of Oil-Immersed Distribution Transformers", ANSI/IEEE C57.100 (1986) [1992]. These so-called life tests were successfully completed. Over the course of these tests, samples of the oil were taken from the transformers and analyzed in an effort to more accurately characterize the life tests themselves. In addition to several of the tests mentioned previously and included in Table 1, gas-in-oil measurements were also conducted on the units at end-point intervals throughout the course of the test program. These measurements were conducted using "Standard Test Method for Analysis of Gases Dissolved in Electrical Insulating Oil by Gas Chromatography", ASTM D 3612-96, as the method of choice. No modifications of the procedure proved necessary for this fluid, other than the recognition of the higher viscosity of the fluid.

Conclusion

It has been seen that existing ASTM specifications for dielectric fluids can be used to guide the development of a new agriculturally based dielectric fluid as has been done previously with high temperature hydrocarbons and silicone fluids When there are large differences between such an insulating fluid and already existing specifications or guides, it may prove desirable to develop a new specification or guide for this fluid. This new specification may incorporate standards and values from already existing specifications as well as incorporate new standards and new values into the new specification. Many of the specifications that have been studied for use for this agricultural fluid are based on petroleum-based hydrocarbons but they are equally appropriate for a vegetable-based dielectric fluid. As acceptance of such a fluid grows, it may prove necessary to amend the titles of these specifications to reflect the broader scope of their application.

Marius Grisaru[1] *and Harry Friedman*[2]

The Big Question: When Should One Recommend Taking a Transformer Out of Service?

Reference: Grisaru, M. and Friedman, H., "**The Big Question: When Should One Recommend Taking a Transformer Out of Service?,**" *Electrical Insulating Materials: International Issues, ASTM STP 1376,* M. M. Hirschler, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

Abstract: This paper examines the goals and difficulties of dissolved gas analysis (DGA) implementation to evaluate the condition of transformers. Although the technique is quite old and well-known in many utilities throughout the world, diagnosis is still complicated and lies in the domain of the state of the art. The published guides indicating the limits for concentration values, or correlating gas ratio and failures, are too broad to be applicable in practice. Gas ratio guides refer to a single failure only, while in practice faults build up one on top of the other until the critical failure appears. This paper reviews some parameters liable to affect normal concentration values. The authors offer a different approach: to develop a database including all failures and unusual cases versus the gases developed at each stage. From the case histories, which took place at the Israel Electric Corporation, we have drawn some recommendations which help to find the right time for scheduled outage of the transformers. (*Note:* Israel Electrical Corporation acquires transformers on the free market. Therefore, the "gassing" transformers are different manufacturers.)

Keywords: dissolved gas analysis, diagnosis, transformer outage, oil, failures, faults

The Israel Electric system consists of an isolated ring, without interconnections to the outside. It includes all the components of a typical electric system, from generation and up to distribution to consumers. The system comprises mainly 400 kV, 161 kV and 22 kV. We still have a few old 110 kV and 33 kV lines, which will disappear in a few years. The whole electric system utilizes about 800 power transformers (above 5 MVA). Ninety percent of the transformers belong to the Electric Corporation, and the remaining ones to industrial consumers. In 1999 the electric system comprises approximately 60 one-phase, 400 kV transformers, 15 3-phase, 110 kV, and the remainder also 3-phase 161 kV units. In this paper we will refer mainly to the 400 kV and 161 kV units. All 400 kV transformers, and almost all 161 kV transformers are sealed by means of membranes or sacs. All generation transformers and step-up transformers are equipped with off-load tap changers, and transmission transformers have on-load tap changers (OLTC) from the high voltage side.

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Israel Electric Corporation DGA Practices

Eight years ago we decided to run periodical DGA tests on all power transformers. During this period we analyzed approximately 1500 dissolved gases in oil tests. In 90% of the transformers we found low combustible gas concentration; in 10%, the dissolved gas concentration was a cause for further concern. In 40 cases, DGA helped to decide upon further operations, as well as maintenance. In 10 cases, DGA helped to avoid unscheduled transformer outage, thus saving both costs and our good name. At present, we are making use of all those interesting cases, and working in close cooperation with our local manufacturer—ELCO—and with transformer users, to discover the actual correlation between oil-dissolved gases and possible transformer failures.

Israeli Conclusions

We conclude that the analysis of dissolved gas in transformer oil is the main, and in many cases, the only method with which it is possible to monitor, analyze and diagnose the condition of power transformers in our country. This method aims mainly to find the right time at which to take the transformer out of service, with minimum costs. Although these are well-known premises throughout the world, these tests are not simple to perform. Literature on the subject is vast and includes many basic theories and different tolerance limits for the amount of gas dissolved as gas-in-oil [*1*].

Further, to the basic principles stated in those papers, we gradually found that almost none of the data and recommendations can be literally applied in many suspicious cases. This situation probably applies to other utilities as well. Attempts to apply one of the many sets of cutoff values for gas concentration, or gas rate trends, can lead to erroneous conclusions. The unaware user of the DGA method looks for fast and easy solutions, but it is only a matter of time until the owner of the transformer is faced with the practical problems of adopting and implementing one of the types of diagnosis that appear in literature [2].

What Is the Best Method to Diagnose and Infer the Condition of the Transformer from DGA Values?

For this dilemma, two types of solutions can be found: the easy one, which consists of choosing the most adequate gas limits already established and recommended in the literature, while the hardest one, which is to develop your own diagnosis and gas concentration limits. The first one is suitable during the first period of application of the DGA method, and the latter one is to be applied in the long term. The disadvantage of the first solution could be likened to the difference between a ready-made and a tailored suit. The long-term solution requires performance of two tasks:

- To find the right method whereby to develop a suitable procedure for transformer diagnosis.
- To have a database large enough to draw significant conclusions and expertise.

During the past 8-year period, we switched over from the standard methods to in-house diagnosis. The main obstacles were to identify each of the transformers' particularities, and to establish the appropriate categories according to their properties.

The main goals of DGA-based diagnosis methods are:

1. To suggest the severity of the failures and their location.
2. To establish a prognosis, which consists of inferring the behavior of the apparatus and probable evaluation of the malfunctions in time. Since it is a forecast, prognosis will have, necessarily, subjective aspects. Today we still have no specific method, and prognosis remains a question for experts.

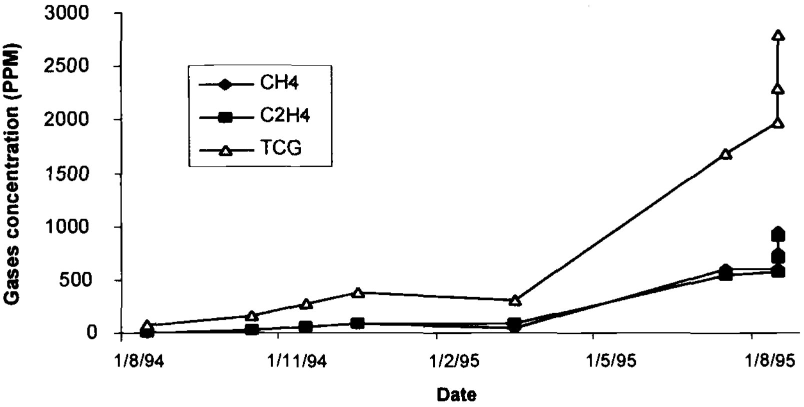
The major advantage of the DGA method is its capability to detect incipient transformer malfunctions using highly sensitive detectors; but it involves disadvantages as well. During the first stage the transformer can produce a high gas concentration without any visible internal marks. At this stage, inspection or repair may be both expensive and disappointing. This stage can span from a few hours to several years, and here comes to the fore the experience and intuition of DGA experts. Most guides and standards distinguish among three parameters: gas concentration level, trend gassing rate (according to ANSI IEEE C57-107 1991 "*IEEE Guide for Interpretation of the Gases Generated in Oil Immersing Transformers*": ppm/day or according to IEC 599 78 "*Interpretation of the Analysis of the Gases in Transformer and Other Oil-Filled Electrical Equipment in Service*": percentage per month) and different ratios between gases. The first two indicate the severity of the failure, and the latter one, the failure pattern. Some papers [*3*] correlate the stage of the transformer's life with its gas concentration level.

Transformer Parameters with Significant Impact on DGA Evaluation

Many transformer users are intuitively aware of many other important parameters that should be considered before evaluating the transformer's "health" [*4*]. Among them we can find:

Type of Tap Changer—The internal TC design, and its integration in the transformer operation, can dramatically affect the dissolved-gas concentration of the oil in the main tank. On-load tap changer oil is separated from the main tank oil. In some OLTCs oil leaks into the tank, while in others it does not. Also, off-load tap changers behave differently. Many thermal faults that produce large amounts of ethylene are caused by overheated contacts in off-load TCs (Fig. 2: Hedera 40, Table 1: Hev). As some of the leading utilities in your country do, we too are now starting to perform DGA in the OLTC compartment.

Manufacture and Construction Technology—Upon ordering a series of transformers, the owner may request all sorts of specifications, without knowing exactly the type of material he receives. Transformers comprise a long list of building materials that develop together with technology and in the wake of the manufacturers' search for new markets. One possible phenomenon caused by that new material can be a different gas rate during the operation. We have a series of mobile transformers that produce a particularly high CO concentration without any other malfunction indication. As a routine of the manufacturing procedure, each 161 kV and 400 kV transformer is tested for a partial discharge (P.D.), and found to be P.D. free. From the DGA point of view, the oil preservation system is important. As stated before, we use mainly sealed transformers. The main consideration in using this preservation system is to prolong the useful life of the transformer by preventing penetration of pollution and moisture into the transformer. Special care is to be given to the oil filling process at site prior to the operation. The ANSI/IEEE C57.106 91 "*Guide for Acceptance and Maintenance of Insulating Oil Equipment*," recommends 0.5% total gas for >345 kV transformers. Griffin [*5*] also recommends to keep the total gas content low. Together with our transformer manufacturer, ELCO, we decided to de-gas the oil to values below 2.0% for 161 kV, and below



Date	TG (%)	H_2	CH_4	C_2H_2	C_2H_4	C_2H_6	CO	CO_2
19/10/94	2.2	13	24	0	26	9	93	151
17/08/95	3.1	280	954	0	902	418	241	329

Transformer removed. Findings: transformer core touched cover.

FIG. 1—*History Case: Hedera SAT—92, 40 MVA, sealed, 14T oil, 161 kV, 1 year, OLTC.*

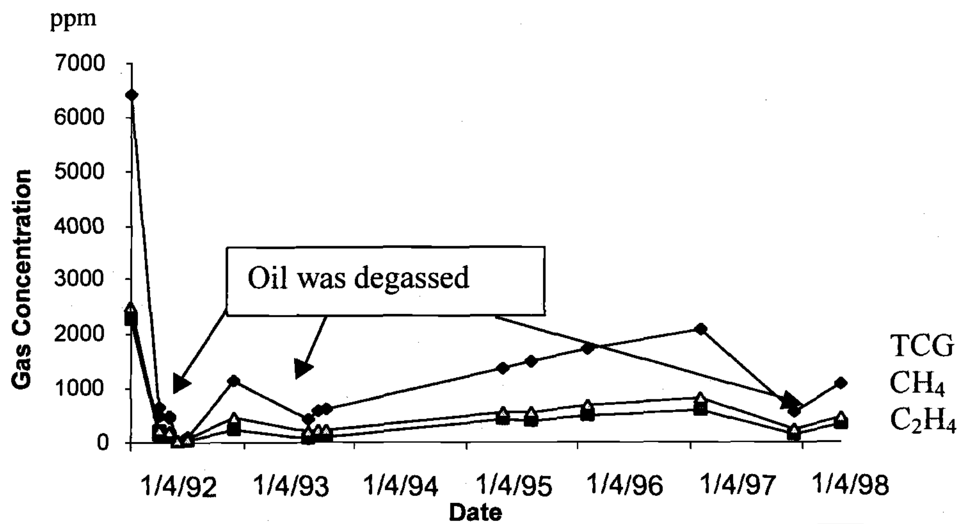
0.5% for 400 kV. We found that this accuracy provided important advantages in addition to those mentioned by Griffin.

Moisture penetrates into solid insulation either during transformer manufacture, or during its installation in the substation. The water content test is performed either immediately, or at a maximum of 6 h after oil filling (according to IEEE C57.106 91). From our experience, the time needed for achieving gas equilibrium inside a transformer is one month. Gas, like moisture, is absorbed deep into the solid insulation; thus, if the transformer is free of gases, it also remains dry.

The second point is described in history cases (Fig. 5; Ramat Gan, Table 1: Telo 1&3). During the overload period if two identical transformers have the same load, but one of them has a significantly higher gas concentration, then bubbles will be emitted first from the oil from the transformer containing a higher amount of total gas. Here we have two opposite and competing processes [*6*]: the speed with which gas develops in unusual situations, and the general trend of mineral oil to contain more hydrocarbon gas as the temperature rises. This characteristic is critical when the transformer is equipped with a Buchholz relay. In this case, gas bubbles can trigger operation of this relay, which in turn will trigger an alarm, or even stop the transformer. All Israel Electric Corporation's sealed transformers are equipped with Buchholz relays. As shown in these case histories (Table 1: Telo 1&3), if two identical transformers are subjected to overload, only the transformer with high nitrogen and oxygen content will cause triggering of the Buchholz relay. Keeping the value of gas low will reduce false Buchholz relay triggering. In our case Buchholz-relay triggering was erroneous.

In transformers with free breathing or blanketed nitrogen, it is more plausible that certain gases will not remain dissolved, as is the case in sealed transformers.

Oil Type—For gas production, the main material inside the transformer is the insulating liquid. At present there are different types of insulating liquids on the market. It is a well known fact that during regular or irregular transformer operation, different types of liquids



Transformer continues to operate. Off load TC was tilted.

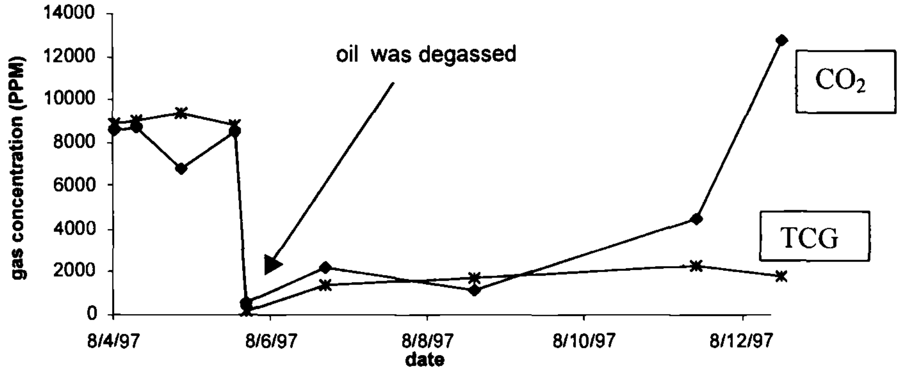
Date	TG (%)	H_2	CH_4	C_2H_2	C_2H_4	C_2H_6	CO	CO_2
30/04/92	4.3	135	2272	1	2494	744	769	5617
07/12/93	3.8	28	102	0	226	48	175	1014
04/03/98	1.9	51	135	0	211	40	122	1271

FIG. 2—*History Case: Hedera 40, 450 MVA, sealed, 44T oil, 161 kV, 17 years.*

produce different gases. One of the problems when introducing new liquids is to develop an appropriate standard equivalent to those developed for mineral oil. Our experience is limited to the most common liquid: mineral oil. Even in the mineral oil family, the market offers at least five types with different behaviors for different purposes. Every utility and/or transformer manufacturer has different transformer oil specifications. The amount of gas produced

TABLE 1—*Israel Electric Corporation partial database.*

	Hev.	Dim.	Pet-ti	Hef.	Atarot	TA. ce	Telo-1	Telo-3	Sprint
Total gas (%)	9.0	2.5	2.2	4.2	5.6	8.6	4.4	8.7	8.8
Hydrogen H_2 (ppm)	1412	637	815	134	71	458	44	61	102
Methane CH_4 (ppm)	5105	125	6	252	244	944	1495	2009	37
Acetylene C_2H_2 (ppm)	183	7	0	1	0	7	6	0	0
Ethylene C_2H_4 (ppm)	6333	10	1	26	19	1108	1241	1593	15
Ethane C_2H_6 (ppm)	1235	27	2	63	47	343	677	860	11
Carbon monoxide CO (ppm)	338	126	205	1223	859	474	400	990	583
Carbon dioxide CO_2 (ppm)	2388	5924	1929	3006	177	8925	1159	2940	10580
Oxygen O_2 (%)	1.7	0.2	0.1	0.1	0.1	0.1	0.1	0.1	0.2
Nitrogen N_2 (%)	5.0	2.0	1.8	3.5	4.6	7.0	3.4	7.3	6.9
TCG (ppm)	14606	932	1029	1700	1239	3335	3863	5513	748



Date	TG (%)	H_2	CH_4	C_2H_2	C_2H_4	C_2H_6	CO	CO_2
08/04/97	6.9	739	2615	205	3533	1272	331	8585
23/12/97	2.8	402	356	44	673	178	165	12733

Transformer removed. Finding: Burned OLTC contacts between the diverter switch compartment and main tank.

FIG. 3—*History Case: Cabri 524, 30 MVA, sealed, 16T oil, 161 kV, 22 years, OLTC.*

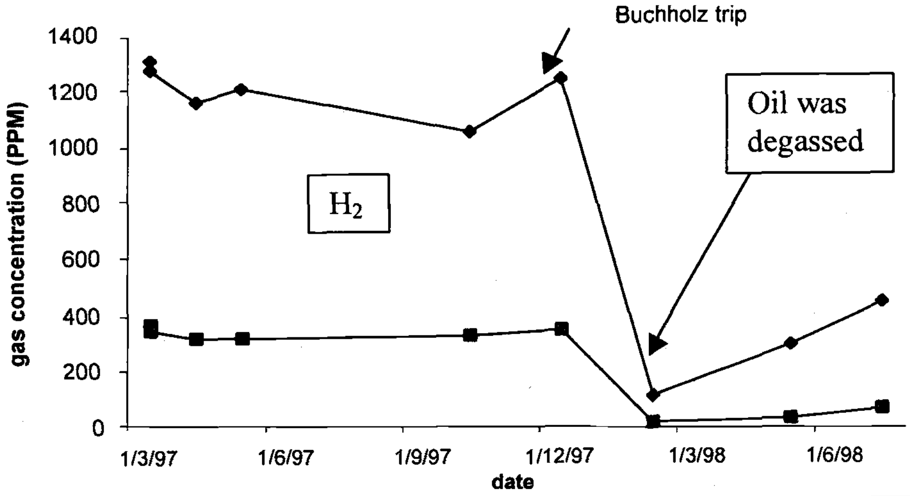
in oil depends on the type of oil (naphthenic or paraffinic), aromatic content, oxidation stage, water content, viscosity, and finally, gas emission tendency. Theoretically, two different types of mineral oils can produce, under similar conditions, two significantly different gas patterns.

Loading Condition

This is the main parameter affecting the gas evolving process during the transformer operation. When the demand for electrical power increases faster than expected—as happened in Israel during the past ten years—the economical way to supply it is to load the transformer almost to the limit. Overloading stresses all internal problems. The rationale behind large dissolved gas amounts in the oil of a well-designed and properly maintained transformer is also loading. One of the obstacles in DGA diagnosis after overload periods is to get from the transformer operator the exact overload time and rate. Sometimes, the urgent need for electricity supply contravene transformer specifications.

We can distinguish between three overload cases:

- The transformer is "healthy," and the large amounts of combustion gases detected in the oil are only due to its overload condition (Fig. 5: Ramat Gan, Table 1: Atarot). Once the gas is removed from the oil, the gas concentration should remain low.
- The transformer already has a minor internal failure, and the excessive load worsens the problem. In this case, the transformer produces a higher gas concentration as compared to another "healthy" unit (Fig. 3: Cabri 524). One of the signs indicative of this



Transformer removed. Finding: Burned OLTC contacts between the diverter switch compartment and main tank.

Date	TG (%)	H_2	CH_4	C_2H_2	C_2H_4	C_2H_6	CO	CO_2
05/03/97	8.9	1314	371	0	7	135	433	9123
19/07/98	4.1	448	70	0	16	19	94	2015

FIG. 4—*History Case: Bet Yosua 521, 30 MVA, sealed, 16T oil, 161 kV, 22 years, OLTC.*

situation is a strong correlation between load and gas evolution, even below 100% load values.

- The third case is even more complicated. Here the transformer was originally "healthy," but due to a long and heavy overload period, it sustained damages (Table 1: Hev). Once the gas was removed from the oil, gas concentrations continued to increase rapidly. In such cases, the transformer is faulty.

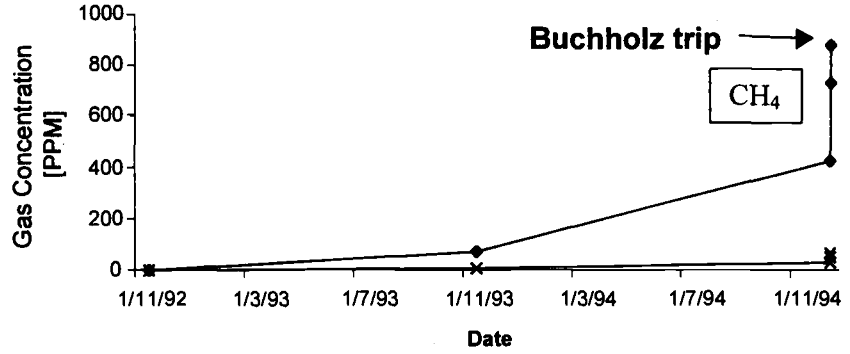
Decision Flow Chart Based on the Analysis of Dissolved Gas in Power Transformers

Figure 8 shows our general conception when using the DGA tool to monitor power transformers in Israel. The important differences as compared to the similar flow chart presented by Savio [*1*] are:

—Events lead to perform a dissolved gas analysis—DGA on transformer oil.

—Under severe conditions, the decision concerning the status and future operation is to be adopted jointly between three representatives:

1. The owner of the transformer, who is acquainted with the operating conditions;
2. The manufacturer of the transformer, who is acquainted with the design and limitations of the unit; and



Transformer removed. Intense inspection and test did not reveal any failure. After oil filling and degassing the transformer operated normally for 4 years.

Date	TG (%)	H_2	CH_4	C_2H_2	C_2H_4	C_2H_6	CO	CO_2
21/12/94	2.8	315	426	0	32	96	1170	1968
29/12/94	6.2	511	873	0	64	193	2732	3739

FIG. 5—*History Case: Ramat Gan 524, 45 MVA, sealed, 23T oil, 161 kV, 1.5 years, OLT.*

3. The DGA expert, who is acquainted with the chemistry and instruments required to get DGA results.

As a general rule, before adopting any critical decision it is imperative to consult with the manufacturer.

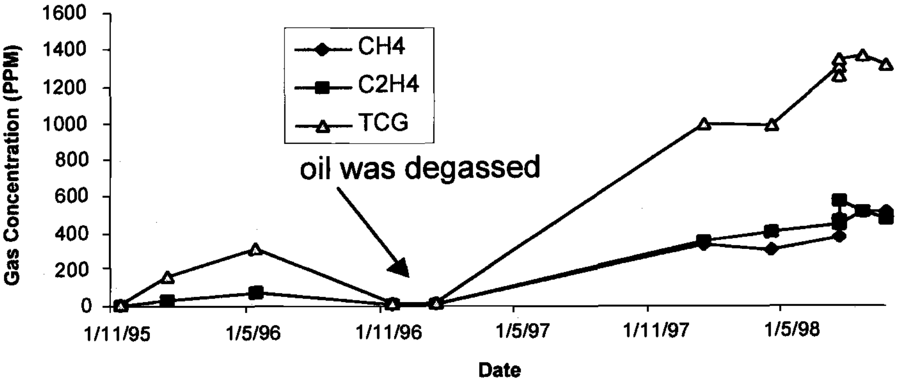
Remarks

Normal Gas Concentration

Instead of using overall cut-off values, we prefer to compare the transformers' concentration values with similar parameters (age, size, manufacture, design, load, etc.) Here a large database would be an advantage, as suggested by Savio [*1*], because the probability of finding another transformer having the same parameters is higher. As a general rule, in order to make a more accurate comparison, we need transformers that are similar, inasmuch as possible, to the suspect one. If the value changes by more than 10% (DGA accuracy), we consider that the gas concentration is increasing. This also facilitates the establishment of sampling intervals.

Distinction Between Severe and Deferrable Failure Conditions

Deferrable Failure Conditions—(a) Excessively high concentration either of hydrogen alone, or of high methane and/or ethylene and/or ethane without major amounts of hydrogen and acetylene. (Examples: H_2: Table 1 Dim., Pet-ti; CH_4+H_2: Table 1: Atar, Hef; CH_4+C_2H_4:



During planned outage the transformer was superficially inspected. Off load contacts suspected.

Date	TG (%)	H_2	CH_4	C_2H_2	C_2H_4	C_2H_6	CO	CO_2
29/01/96	1.8	10	27	0	27	11	82	212
26/09/98	1.7	30	489	1	456	145	169	1022

FIG. 6—*History Case: Hedera 50R, 217 MVA, sealed, 50T oil, 420 kV, 4 years.*

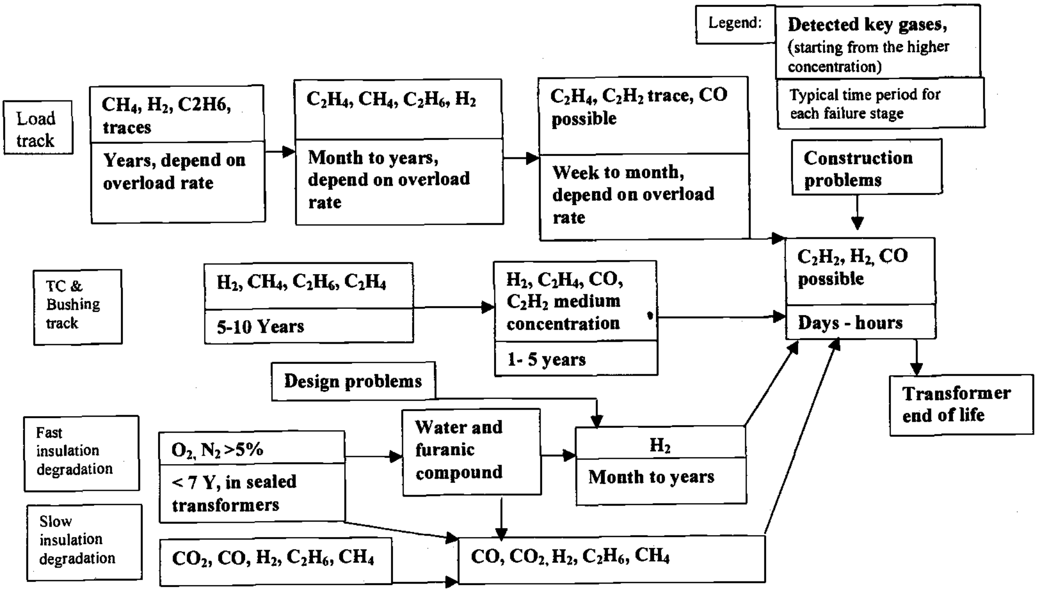


FIG. 7—*Fault propagation stages.*

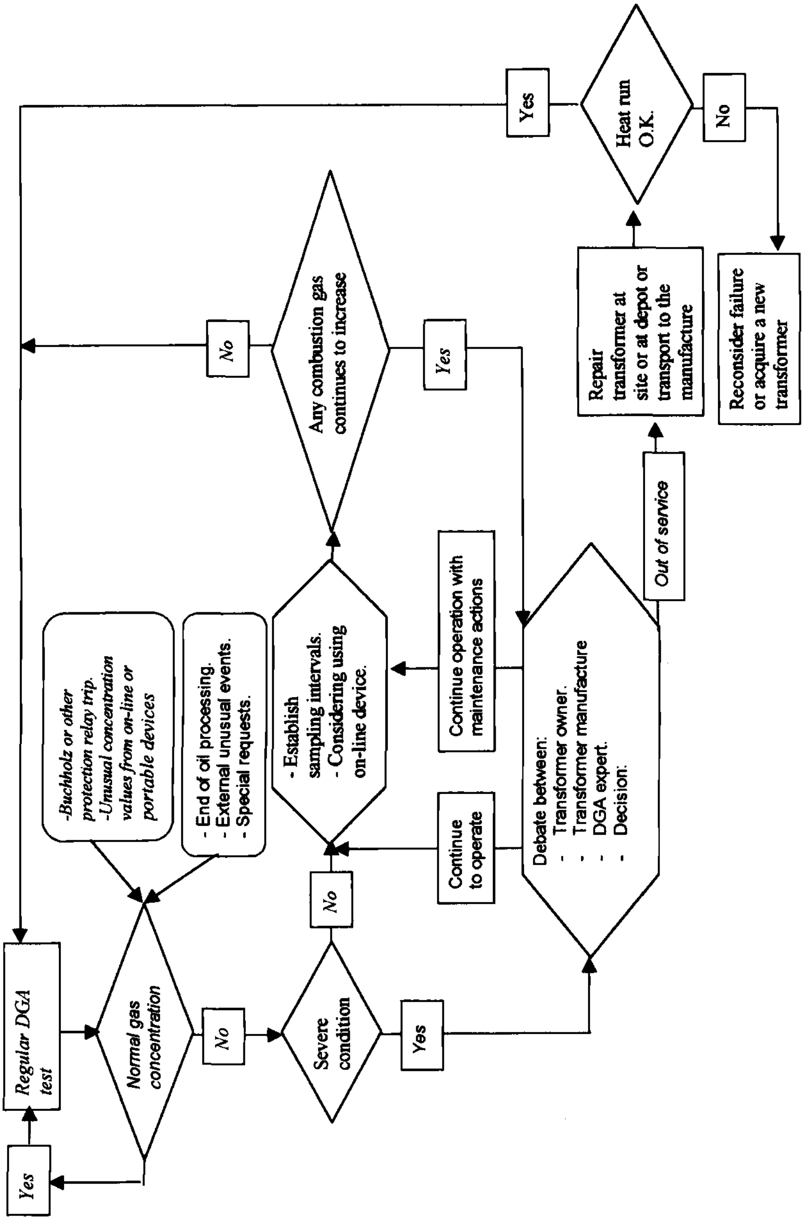


FIG. 8—*Decision flow chart based on dissolved gas analysis in power transformer.*

Fig. 5: Ramat Gan; $CH_4+C_2H_4+C_2H_6$: Table 1: Telo 1 & 3.) (b) First significant increase in the concentration of the gas mentioned in paragraph (a). (Examples: Fig. 5: Ramat Gan, Table 1: Telo 1 & 3.)

Severe Conditions—

- Excessively high increases (above 10% compared to "healthy" units) in the concentration of one of the following gas combinations: acetylene and hydrogen or ethylene and hydrogen or hydrogen and carbon monoxide. (Examples: Table 1: TA-Ce.: $H_2+C_2H_4+CH_4$.)
- Continuous significant increase (above 10%) of any of the dissolved gases (including nitrogen, oxygen and carbon dioxide). (Fig. 3 and 4: Cabri 524, Bet Yosua 521.)
- Abnormal increase in gas concentration: the relation between concentration value rate and failure severity is complicated. It depends on too many parameters: temperature, transformer size and design, oil preservation system, oil quantity, external conditions such as load short circuits, lightning and ambient temperature (Fig. 1 and 6: Hedera Sat-92 and Hedera 50 R).
- Gas concentration increase only if the values exceed the previous value by 10%.

Fault Propagation

Fault propagation is characterized by different failure stages. Faults are revealed in most cases by regular DGA regime. Usually small failures lead to severe faults. We could observe transition between those stages by close surveillance. Each stage has its unique gas profile (Figs. 1–6). Figure 7 provides rough time estimation for each failure stage. Naturally this time depends on transformer design and operation (load) conditions.

These parameters can assist the transformer owner to recognize the origin and severity of any unusual situation exposed by DGA and a convenient time schedule for the next step: resample or investigation.

Conclusions

Unlike blood tests performed on human beings, the design and operation of transformers can be highly varying. In such conditions, *general concentration limits are not applicable.* Our approach consists of developing a database that includes all failures and unusual cases versus the gases developed in each stage. Learning, and the conclusions derived from this database, pertains to state-of-the-art DGA diagnosis.

When Should One Recommend To Have a Transformer Taken Out of Service?

Even though the overall message of this paper speaks against prefabricated receipts, we have also prepared a concise guide based upon our present experience. It helped us to establish several cases in which the decision to take the transformer out of service was correct, and we believe it to be helpful for other utilities as well. If the transformer meets one of the seven following conditions, our recommendation would be to take the transformer out of service:

1. Total combustion gases (TCG) lie below 2000 ppm, and the curve of one gas concentration versus time changes abruptly—usual shift from linearity to exponential function, and

hydrogen concentration is higher than methane. If the first two conditions are true, and the third is false, then the failure is not a malfunction and the transformer should be resampled.

2. Sudden increase in acetylene and hydrogen or ethylene and hydrogen or hydrogen and carbon monoxide, and each of the pair of gas concentrations reach unusual values. Slow concentration growth or stable high values are deferrable conditions and only require resampling at short intervals.
3. Sudden increases in carbon dioxide or carbon monoxide and their concentrations are above 12 000 ppm and 1200 ppm, respectively (Table 1: Sprint).
4. TCG concentration > 2000 ppm and there is a significant increase in one of the combustion gases or carbon dioxide.
5. TCG concentration > 2000 ppm and the hydrogen concentration is higher than methane.
6. If the transformer has TCG > 2000 ppm, and its oil was degassed, it can still be judged according to paragraphs 4 and 5, even though the actual TCG concentration is smaller.
7. In sealed transformers, the total gas concentration increase exceeds 2% per year.

These are, of course, only a part of the risk situations which might appear, and each case should be discussed in depth by the three parties—the owner of the transformer, the manufacturer of the transformer, and the DGA expert.

Acknowledgments

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[2] Kelly, J. J., Myers, S. D., and Parish, R. H., *A Guide to Transformer Maintenance* Table 4.6. p. 347, published by Transformer Maintenance Institute, 1981.

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[*4*] Samat, J., "Interpretation of DGA and Its Use in the Detection of Malfunction," *IEE Colloquium on Assessments of Degradation Within Transformer Insulation System* (Digest 1991/184), London, UK, Date: 6 December 1991, pp. 3/1–3/4.

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[*6*] "Analysis of Dissolved Gas and Free Gases for Monitoring Performance of Oil-Filled Transformers," *The Brown Boveri Review*, Vol. 54, No. 2/3, February/March 1967.

Sam A. Margolis[1]

Sources of Systematic Bias Reflected in an ASTM Collaborative Study on Water in Oil Measured by the Karl Fischer Method

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Reference: Margolis, S. A., "**Sources of Systematic Bias Reflected in an ASTM Collaborative Study on Water in Oil Measured by the Karl Fischer Method,**" *Electrical Insulating Materials: International Issues, ASTM STP 1376,* M. M. Hirschler, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

Abstract: A collaborative study on the precision and bias of the measurement of water in transformer oils indicated that an acceptable level of precision was achieved using the revised ASTM Test Method for Water in Insulating Liquids by Coulometric Karl Fischer Titration (D 1533-96) and a sample size of 7 mL. Analysis of the distribution of the laboratory results indicates the existence of a systematic bias in the accuracy of the measurement which is related to the instrument design, solvent composition, and calibration of the instrument. Additionally, evidence is presented that indicates that a portion of the water in the oils is not titrated by the coulometric method.

Keywords: water, Karl Fischer method, systematic bias, transformer oil, coulometry, precision, D 1533-96

When an electrical transformer is put into service it is essential that fluids used for the insulation contain very little water (≤10 mg/kg) and that the water content of the insulating oil remains low, otherwise the transformer may malfunction. Several methods have been used or are now in use for making this measurement. These include: ASTM Test Method for Water in Insulating Liquids by Coulometric Karl Fischer Titration (D 1533-96) and ASTM Test Method for Water in Liquid Petroleum Products by Karl Fischer Reagent (D 1744-92), azeotropic distillation [1, 2], headspace/gas chromatography, [3], and oven evaporation [4].

About 10 years ago NIST was asked to prepare reference materials RM 8506 and RM 8507 to help resolve the basis for the large interlaboratory variation in the measurement of water in oil. A preliminary collaborative study carried out in 1996 indicated that the interlaboratory variation was still unacceptable. Because of this variability it was necessary to develop a stable standard to evaluate the accuracy of the instruments and the measurement methods. NIST has recently issued SRM 2890, Water Saturated 1-Octanol, which is a stable standard for the calibration of instruments that measure water. Using water saturated 1-octanol (WSO) we were able to demonstrate several types of systematic errors in the volumetric and coulometric Karl Fischer methods. The sources of bias for the volumetric method are the accurate estimation of

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the endpoint of the titration and the use of an accurate standard [5]. These biases could be surmounted by using the graphical method of endpoint determination [6] and by using WSO as a standard [5]. The sources of bias for the coulometric method include the accuracy of the water standard, the non-adjustable instrumental bias, the operator adjustable instrumental parameters, the solvent composition, the cell design, and the sample composition [5]. Procedures for overcoming some of these biases were defined.

After optimizing the instruments that are used in our laboratory and establishing their accuracy by using WSO, we demonstrated that sample composition has a significant effect on the amount of water measured in refined oils including transformer, mineral (paraffin), and hydraulic oils [7, 8]. The water titers were measured by the volumetric method using either a pyridine or imidazole based titrant and a vessel solution composed of chloroform:methanol 6:1 (v/v). The chloroform content of this vessel solution after being titrated to dryness with the Karl Fischer reagent was between 66% and 76% of the total volume depending on the reagent used [7]. The highest water titers for the oils were obtained by the volumetric method using either titrant. Hydrocarbons such as octanol and xylene gave the same water titers by both volumetric and coulometric methods. However, the water content of all of the other hydrocarbons and hydrocarbon mixtures measured by the coulometric method using a two compartment cell was less than that observed volumetrically. With the exception of one transformer oil the water content of all the oils tested coulometrically, using a one compartment cell, decreased relative to that obtained by the volumetric method. The water content of transformer oil, in which the coulometrically measured water did not decrease, was the same as that measured volumetrically. The objective of this study is to examine the sources of bias detected in an ASTM collaborative study on three samples of a new transformer oil containing different amounts of water and to interpret these results in terms of the results obtained at NIST using a variety of oils.

Experimental Method

Collaborative Study

Diala A oil was obtained from Shell Oil (Houston, TX) and prepared at CSC Scientific Co. Inc. (Fairfax, VA). It was dried by bubbling dry nitrogen through the oil until the water content measured by the coulometric Karl Fischer method was approximately 10 mg/kg (Diala A1). The resulting material was then divided into three equal lots, two of which were rehydrated by bubbling air through the oil until the water content of one lot (Diala A2) was approximately 25 mg/kg and the other lot (Diala A3) was 40 mg/kg. Each lot was then subdivided into two parts and each part was ampouled under argon as a separate sample in 20 mL ampoules and glass sealed. Fifty-five ampoules were prepared for each individual sample and numbered in order of filling. All ampoules used in this study were numbered according to the order of filling and distributed to the laboratories in random order.

Each laboratory received two ampoules of each sample of Diala A oil and two ampoules of SRM 2890, Water Saturated 1-Octanol. All test oil samples were numbered.

Samples were analyzed in pairs and the order of analysis of each pair was randomized. One WSO sample was analyzed before and the other after the oil samples were analyzed. Each laboratory used the coulometric method as described in the revised ASTM Test Method D 1533-96. Each laboratory used their in house coulometric instrument and solvent system. These included instruments manufactured by Mitsubishi (Cosa Instruments, Norwood, NJ), EM Science (Aquastar, Cherry Hill, NJ), UMM Electronics (Aquatest, Indianapolis, IN), Brinkmann Instruments (Westbury, NY), and Orion Research (Beverly, MA). The solvents were Watermark (GFS Chemicals, Powell, OH), Aquamicron (Cosa Instruments, Norwood, NJ), Coulomat A and Coulomat AG-H, (Brinkmann Instruments, Westbury, NY), Photovolt (UMM Electronics, Indianapolis, IN), and Aquastar (EM Science, Cherry Hill, NJ). The data were analyzed according to the ASTM Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method (E 691-92).

NIST Analyses[2]

NIST volumetric and coulometric analyses which were not included in the collaborative study were preformed as previously described [4].

Results and Discussion

The primary purpose of this ASTM sponsored collaborative study was to evaluate the precision and accuracy of ASTM Test Method for Water in Insulating Liquids by Coulometric Karl Fischer Titration (D 1533-96). Thirty four laboratories participated in this study and three laboratories submitted two sets of data for a total of 37 data sets. Analysis of the distribution of water content of each vial as a function of ampoule number indicated that within the accuracy of the measurement there was no bias due to filling sequence. The water content of the three lots of Diala A oil, each containing a different amount of water, is summarized in Table 1. The first column represents the mean and repeatability (SD of the measurements of a single laboratory using one method and one instrument as defined in E 691-92) of all of the data, the second column the mean and repeatability minus the outlier values, and the last column the reproducibility of the means in column 2. It is evident that the small number of outlier values (three complete sets of data and six isolated values) have little effect on the mean but a great effect on the repeatability which represents the overall within laboratory standard deviation. The values for the reproducibility (among laboratory SD) were calculated using the outlier adjusted data and represent the among laboratory standard deviation. The reproducibility is 1.9 to 2.1 times the repeatability and is similar to many such studies that under the

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[2] Certain commercial equipment, instruments and materials are identified in this paper to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the equipment or material is necessarily the best for the purposes.

Table 1 - *Summary of the Statistical Analysis of the Data of the Collaborative Study.*

Sample	Mean		Reproducibility
	All Data[a]	Outlier Adjusted[b]	
	Water (mg/kg)		
Diala A1	12.4 (7.9)[c]	11.1 (2.3)	4.3
Diala A2	28.8 (15.1)	24.0 (3.1)	6.3
Diala A3	42.2 (4.9)	40.7 (2.5)	5.1

[a] This includes thirty seven sets of data submitted by 34 laboratories
[b] Three sets of data were dropped because more than 30% of the values were outliers. In addition three Diala A2 samples from two additional laboratories were dropped as outliers.
[c] The numbers in parentheses represent the standard deviation or the repeatability.

conditions of measurement are relatively independent of materials and methods. This independence applies only to the limits of the sample size (7 mL) and restrictions of the method tested. The statistical analysis of these results indicates that 7 mL of transformer oil can be used to obtain measurements with acceptable repeatability and reproducibility with the D 1533-96 method in this type of oil sample with water concentrations of the order of 10 to 50 mg/kg of oil (10 to 50 ppm).

Although the precision of this method is acceptable according to E 691-92, the minimum amount of sample required for each measurement is large and it would be desirable to be able to use a smaller sample in order to reduce reagent consumption. The reduction of the sample size requires an understanding of the systematic biases inherent in the use of multiple instruments and reagents. This study was designed to assess some of the sources of bias arising from the use of a variety of instruments, solvents, and a stable standard.

Each laboratory that participated in this study evaluated their laboratory accuracy by measuring the water in 10 μL of NIST SRM 2890, WSO, which contains 39.24 ± 0.85 mg water/mL of solution, before and after the entire cohort of samples was analyzed. As illustrated in Figure 1, the measurement of water in the SRM by any given laboratory was consistent. Some laboratories were consistently low while others were consistently high indicating that either the instruments were improperly calibrated or that some laboratories were unable to measure 10 μL of WSO accurately. To resolve the source of this bias, we compared the WSO measurements made before any oil samples were assayed to the values obtained for the water in the oil samples. Figure 2 demonstrates that the values for the water in the oil exhibited a large degree of variation even for those laboratories that accurately measured the water in WSO. However, the variation in the oil assays was even greater for the other laboratories. Thus not only was there a bias due to the ability to

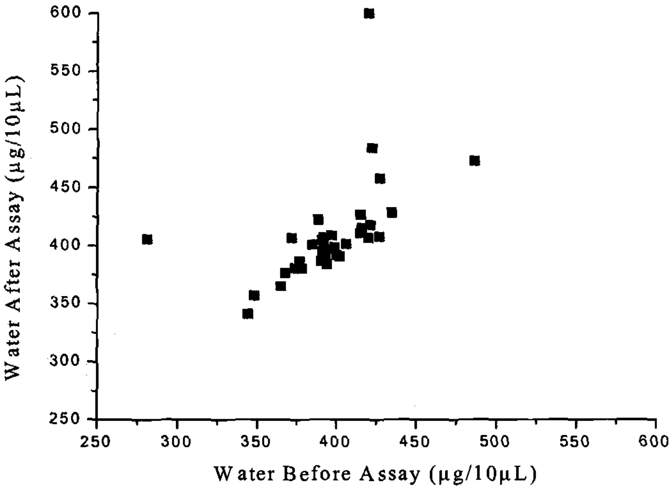

Figure 1. Comparison of the Measurement of Water in SRM 2890 Before and After the Assay of Water in Oil Samples. Each point represents the results of a single laboratory.

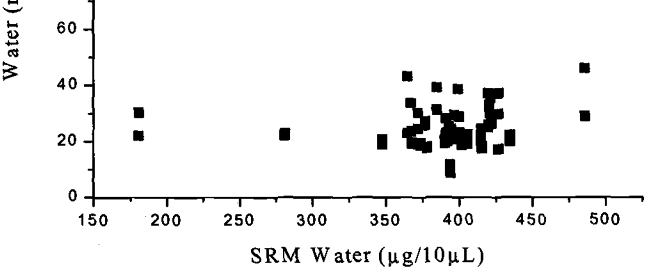

Figure 2. Distribution of Measurement of Water in Diala A2 Oil Sample as a Function of Water Measured in SRM 2890 (WSO). Each point represents the results of a single measurement by a single laboratory.

measure the water in a standard but also a predominantly unrelated large variation in the measurement of the water in the oil samples.

The design of the study and the large number of participants allow the evaluation of the systematic bias in the measurement of the water in the oil that was related to the instrument design and the coulometric anode reagents. All major instrument and solvent manufacturers are represented in this study. Figure 3 illustrates the distribution of the means of the reported water measurements for the Diala A2 oil sample as a function of the instruments and anode reagents. Each point represents the average results of a single laboratory. It is very clear that for reagent 1 there are two populations of results; one set (solid symbols) that is approximately 20% below the mean of 24.0 mg of water/kg of oil and another set (open symbols) that is approximately 30% above the mean. Furthermore except for one laboratory using an Aquatest instrument, each grouping of the measurement results using reagent 1 contains the results from a different group of instrument models. Similarly the results with the other reagents (particularly the Aquamicron and Coulomat AG-H reagents) appear to be grouped. A comparison of the values reported for the Aquamicron and Aquapal III instruments for the Watermark, Aquamicron, Coulomat A, and Coulomat AG-H reagents reveals that the reported values for each laboratory fall within a major grouping of data. Some of these groups are close to the overall mean and others are not. The wide distribution of values for the Aquatest group of instruments is probably the result of the variation in the calibration of the three different instrument models from the same manufacturer [5]. A similar distribution pattern of the water in the oil samples was observed for all three oil samples. Thus this bias is independent of water concentration, but is dependent on the model of the instrument and upon the reagent. This suggests that although the precision of these measurements can be assessed, their accuracy is questionable.

These observations concerning the accuracy of the Karl Fischer methods are consistent with our earlier observations: a) there is a bias attributed to the type of anode solution used in the titration vessel [5] and b) the amount of water measured in a given oil sample is dependent upon the Karl Fischer method that was used to make the measurement [7, 8]. The highest water content was obtained by the volumetric Karl Fischer method. When the coulometric Karl Fischer method was used, smaller amounts of water were measured that were of variable size for a given sample, depending on the type of method that was used. Table 2 illustrates this phenomenon with the Diala A oils. Three different concentrations of water were measured in the two ampoules of the Diala A1 sample by the three different methods. The difference between the results obtained by the two different coulometric instruments was observed only for the lowest concentration of water. For the other two samples with higher water concentrations the measurements were similar. A similar pattern is seen in Table 3 (laboratory 1 vs laboratory 2) except that lower water concentrations were observed for the samples with the two lower concentrations of water whereas at the highest water concentration the results were similar. Both of the laboratories in Table 3 used the same anode reagent but different instruments. Laboratory 1 used two different instruments from the same manufacturer, and laboratory 2 made two independent sets of measurements on the same instrument. These results indicate that the systematic bias of the measurement can be attributed to the instrument and not to the anode reagent. This supports the conclusion based on the distribution of the data in Figure 3 and upon our earlier observations [5].

Table 2 - *Summary of the NIST Analysis of the Collaborative Study Samples.*

Method	Volumetric	Coulometric	
Cell Type		Diaphragmless[a]	Diaphragm[b]
Reagent	Pyridine[c]	Coulomat AG-H	Coulomat A + CG
Oil	Water (mg/kg)[d]		
Diala A1	33, 28	19, 17	11, 13
Diala A2	46, 50	23, 29	30, 25
Diala A3	58, 55	41, 44	44, 46

[a] The instrument used is a Brinkmann Model 737 titrator.
[b] The instrument used is a Aquapal III titrator.
[c] The vessel solvent was chloroform:methanol 6:1 (v/v).
[d] Two vials of each oil were evaluated. A single sample from each vial was evaluated by each method.

Table 3 - *Summary of the Data from the Laboratories that Performed Duplicate Sets of Analyses.*

Laboratory	Oil Sample	Water (mg/kg)[a]	
		Data Set 1	Data Set 2
1[b]	Diala A1	15, 13	17, 15
2[c]	Diala A1	8, 8	8, 7
1	Diala A2	33, 31	37, 30
2	Diala A2	19, 20	23, 19
1	Diala A3	52, 43	43, 44
2	Diala A3	39, 43	42, 41

[a] Two vials of each oil were evaluated. A single sample from each vial was evaluated by each method.
[b] These data were acquired with a Coulomat solvent and an Orion AF7 instrument which is made by the same manufacturer as the Aquamicron instrument
[c] These data were acquired with a Brinkmann 652 instrument and Coulomat A reagent.

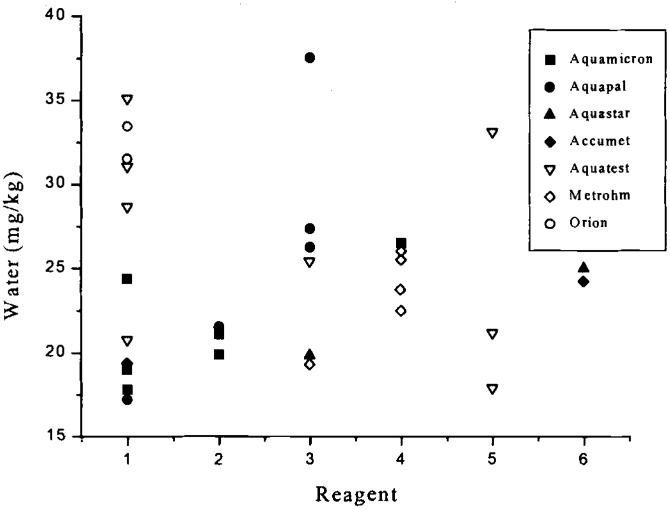

Figure 3. Comparison of Distribution of Water Content of Diala A2 Oil Samples as a Function of Instrument Type and Anode Reagent. Each point represents the average of the 2 values reported by an individual laboratory. The symbols represent the type of instrument; the anode reagents (x axis) are (1) Watermark, (2) Aquamicron, (3) Coulomat A, (4) Coulomat AG-H, (5) Photovolt, and (6) Aquastar.

These results indicate that the magnitude of the amount of water measured is dependent upon the type of instrument (Table 3) and may also be dependent on the composition of the anode reagent (Figure 3). The consistently higher water measurements obtained by the volumetric Karl Fischer method in earlier studies [7, 8] and in Table 2 also suggest that a systematic bias exists in the accuracy of the Karl Fischer methods. To resolve the nature of this systematic bias we measured the water coulometrically in WSO followed by six successive 5-mL samples of an oil sample and then another WSO sample (Table 4). We also measured the water volumetrically in three successive samples of the same oil samples using a vessel solution of chloroform: methanol 6:1 (v/v) (Table 4). It is evident that more water was measured by the volumetric method than by the coulometric method in all three oils that were evaluated. To determine the basis for the difference between the results of the two Karl Fischer methods, we measured the water in the two phases of the coulometric anode solution by the volumetric method. We inserted a syringe into the coulometric vessel and rinsed it with the solution to dry the syringe. After letting the coulometric instrument titrate the water added by rinsing the syringe, the stirrer was stopped and the oil phase was allowed to separate from the anode solution. Three 5-mL samples were taken from each layer and assayed by the volumetric method. The results of this experiment are summarized in Table 5. Columns A-C represent the total amount of water detected in 30 mL of oil by the volumetric method, the coulometric

Table 4 - *Summary of the Measurement by the Coulometric and Volumetric Karl Fischer Methods of the Water in Oils analyzed for the Distribution of Water in the Two Phases of the Coulometric Anode Solution.*

Oil	Water (mg/kg)	
	Volumetric[a]	Coulometric[b]
Univolt N61 RM 8506	40.6 (1.5)	20.6 (1.3)
Coray 22 RM 8507	87.1 (3.1)	35.2 (2.9)
Mineral Oil	73.0 (1.0)	42.9 (2.9)

[a] These measurements were made using chloroform:methanol 6:1 (v/v) as the titration vessel solvent. Each value represents the average of three measurements and the numbers in parentheses represent the standard deviation of a single measurement.

[b] Each value represents the average of six measurements and the numbers in parentheses represent the standard deviation of a single measurement.

method and in the 30 mL of the upper oil layer of the anode compartment. No water was detected in the lower solvent layer of the anode compartment. It is clear that the sum of the water measured in the oil in the initial coulometric measurement (column B) plus the water measured volumetrically in the upper oil layer of the coulometric cell (column C) equals the water measured volumetrically in the oils (compare columns A and D). The material that was measured in the oil layer of the coulometric anode solution appears to be water and not an artifact of the measurement process for the following reasons. In all the volumetric measurement sequences the water content of WSO was measured before and after the total water in the oil was measured, and the water in upper anode-compartment oil-layer samples was measured. No change was observed in the water titer of the WSO between the beginning and the end of the measurements. Furthermore, the amount of water detected volumetrically in the WSO was the same regardless of whether methanol or chloroform: methanol 6:1 (v/v) was used as the vessel solution. Thus, the amount of water measured does not appear to be an artifact of the solvent composition of the volumetric vessel solution, the oil matrix, or the ability of the Karl Fischer reagent to react with free water by either the coulometric or volumetric methods.

The three oils that were evaluated were selected to demonstrate that antioxidants or other oil additives were not responsible for the reduced amount of water detected by the coulometric methods. The Coray 22 oil contained no antioxidants, and the highly refined white mineral oil contained negligible amounts of the types of compounds that react with the Karl Fischer reagent to give spurious results. Furthermore, the titration curves, which were recorded during both the coulometric and volumetric assays, revealed no evidence of secondary reactions. None of the solvents used including solvent used for the volumetric assay, which contained a high concentration of chloroform, affected

Table 5 - *Distribution of Water in the Upper and Lower Phases of the Coulometric Anode Solution.*

Oil	Total Water (μg)			
	A	B	C	D
	Volumetric Method[a]	Coulometric Method[b]	Upper Phase Coulometric Vessel Solution	Column B + C
Univolt N61 RM 8506	1218 (45)[c]	552 (35)	672 (25)	1224
Coray 22 RM 8507	2613 (93)	1543 (87)	1032 (37)	2575
Mineral Oil	2280 (31)	1159 (65)	1190 (16)	2346

[a] The instrument used was a Brinkmann Model 633 titrator, the vessel solvent chloroform:methanol 6:1 (v/v).

[b] The instrument used was a Aquapal III titrator. The anode solution was Coulomat A the cathode solution was Coulomat CG.

[c] The number in parentheses represents the standard deviation of a single measurement. The values in column A represent the mean of three measurements and the values in column B represent the mean of six measurements.

the measurement of the water in WSO. Quantitative results for water in WSO were obtained in all cases. All of the evidence suggests that the increased water measured by the volumetric Karl Fischer method is not the result of the ability of the method to measure the free water introduced as WSO in the absence or presence of chloroform or oil. There is also no evidence that the lower values obtained coulometrically are the result of the inability of the instrument to detect all of the water introduced as WSO either in the presence or absence of the oil samples. Finally, none of the results indicates that antioxidants or other substances in the oils contributed to the difference between the coulometric and volumetric measurements.

The collaborative study indicates that the precision of the coulometric Karl Fischer method is acceptable within the limits of this study as defined in E 691-92. The data also indicate that a measurable amount of systematic bias exists. This bias is related to both the instrument design and the type or composition of the anode reagent. It is also evident that there is a large variation in the ability of some laboratories to measure accurately the water in NIST SRM 2890, WSO. This variation is consistent with the previously demonstrated sources of systematic bias [8]. The discrepancy between the results obtained by the coulometric and volumetric Karl Fischer methods appears to be related to the inability of the coulometric method to measure all of the water in the oil that is suspended in the anode solution. The physical basis for the unavailability of this water is not clear. Both methods are capable of measuring all of the water in WSO in both the presence and absence of oil in the titration vessel. Furthermore, the presence of chloroform (66% - 76% of the solvent) in the volumetric titration vessel does not modify the ability of the instrument to measure all of the water in WSO. All of the results indicate that the coulometric Karl Fischer method is not *accurate*. However, this method exhibits an acceptable level of *precision* and therefore can be used to assess routinely the relative water content of transformer oils for the purpose of evaluating the stability of the electrical insulating fluids and the transformers in which they are used.

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The Electrochemical Stability of Mineral Insulating Oils

Reference: Sabau, J., Stokhuyzen, R., **"The Electrochemical Stability of Mineral Insulating Oils,"** *Electrical Insulating Materials: International Issues, ASTM STP 1376,* M. M. Hirschler, Ed., American Society of Testing and Materials, West Conshohocken, PA, 2000.

Abstract: Successful detection of an incipient electrical failure within the windings of power transformers is based upon the possibility of determining the amount and nature of the gases evolved by the oil when subjected to the impact of a strong electric or thermal stress. The cause of the deficiency can be established by analyzing the gases dissolved in the oil. While under normal operating conditions the amount of gas evolved is minimal and therefore of little importance, a significant increase in the concentration of gaseous decomposition products gives cause for concern.

Since a large amount of vulnerable hydrocarbon molecules can also be the source of dissolved gases, the gassing tendency of oil should be taken into consideration. The lack of a reliable procedure capable of establishing a quantitive relationship between laboratory results and the performance of oil in service can influence the interpretation of dissolved gas analysis (DGA). Just as the chemical stability of oil is accepted as an indication of the ability of insulating oils to resist oxidation, the concept of electrochemical stability is introduced as a measure of the capacity of the oil to counteract gas evolution under the impact of electrical stress. Knowledge of the amount of gas generated by a sample of oil, when subjected to the influence of a standard high-voltage field, can improve the interpretation of DGA of that oil. This concept also provides a better understanding of the mechanism by which the high-voltage field negatively affects the chemistry of insulating fluids.

Keywords: transformer oil, gassing tendency, physical stability

Dissolved gaseous decay products are generated in electro-insulating oils in high-voltage transformers by incipient electrical failures of various types and intensities. Local overstressing of the oil-paper insulation inside the windings can supply sufficient energy to break certain carbon-carbon and carbon-hydrogen bonds of some components of the oil's complex blend of hydrocarbons to form gaseous compounds. Understanding and measuring the gassing tendency of insulating oils under thermal and electrical stress are therefore of major importance in predicting and preventing transformer failure.

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Over the past decade, the laboratory methods available for testing gas evolved from oils have been the Merrell Test (The American Society for Testing and Material - Designation: D 2298-81) which was discontinued in 1987, and replaced by the Modified Pirelli Method (ASTM Designation: D 2300-85). Analysis of dissolved gases in transformer oils is used to diagnose the type and degree of incipient failure [*1*]. However, the results of this analysis do not always correlate reliably with the actual failure rate under service conditions [*2*]. Possible oil breakdown mechanisms based on the principles of photochemistry are discussed in this paper and a method for pre-service analysis of insulating oils is presented. This analysis, which measures the production of gasses, charge carriers, free radicals and x-waxes formed during high-voltage impact, provides an alternative approach to the prediction of oil degradation. Its application has potential for significant savings to the electrical power industry by preventing transformer failures and extending their life expectancy.

2. THEORETICAL BACKGROUND

The build up of gaseous decay products in insulating oils during service results mainly from the breakdown of weak valence bonds. It is important to understand the process by which this breakdown occurs and to identify the energy source which initiates the decomposition of vulnerable hydrocarbon molecules.

The influence of transient voltage surges, trace impurities, hot spots and partial discharges make the decay process very complex. These factors all favour the degradation of both the insulating oil and the cellulose insulation. It has been observed that the degree of polymerization of the cellulose slowly decreases because of thermal stress. Formation of high molecular weight products (x-waxes) and their deposition in the paper insulation may reduce its permeability, resulting in impaired heat transfer and the creation of hot spots. While the breakdown of solid insulation requires further study and clarification, the focus of this research has been on the stability of insulating oils.

The hydrocarbon blend consists of more than 3000 components. Excited molecules produced by the primary process of energy absorption can undergo a variety of secondary processes, both physical and chemical. Typical processes which affect the electrochemical stability of hydrocarbons are the dissociation of excited molecules into smaller fragments, their rearrangement via bond-breaking, by transfer of energy to other molecules, or their return to the ground state by releasing absorbed energy.

The aliphatic carbon-carbon bond energy of hydrocarbon oils is about 90 kcal/mol (about 4 eV). Thermal excitation of molecules becomes sufficient to break these bonds at temperatures of 350 to 550 °C. Thermal cracking of petroleum products is carried out in this temperature range [*3*]. Below 100 °C, the normal maximum operating temperature of high-voltage transformers, there is insufficient thermal energy to excite valence electrons.

Our research has focused on the effects of free electrons, which provide a powerful source of energy. During transient (especially commutation) field enhancements [*4*], invisible asperities on the copper windings may eject free electrons. With potential gradients of 0.3 to 0.4 kV/mm, these free electrons possess sufficient energy to endanger the electronic structure of chemically inert hydrocarbons. Although the duration of these transient events is only a few microseconds, their cumulative effect cannot be neglected since commutation field enhancements occur during normal service conditions and because asperities in copper windings are unavoidable. In high-speed Schlieren studies of electrical breakdown in liquid hydrocarbons no streamer was observed originating from within the liquid itself. A rapid rise in light emission occurred just before breakdown [*5*] which suggests that this process begins in the immediate vicinity of metal surfaces.

Forster's work at high stresses on very pure and clean liquids [*6*] suggests the need to clarify the role played by vulnerable hydrocarbons in insulating oils. Even at normal operating temperatures, less stable molecules could accumulate sufficient energy for electronic transitions during the elastic collisions caused by thermal agitation. Such electronic transitions may result in the homolytical breakdown of weak valence bonds. Of the weakly-bonded molecules, peroxides are a frequent source of free radicals which can serve as initiators of auto-oxidation reactions.

$ROOH \xrightarrow{hv} RO\cdot + \cdot OH$

$RH + RO\cdot \rightarrow ROH + R\cdot$

Free radicals can be transformed through reduction-oxidation reactions into charge carriers, thus increasing the power factor of an insulating oil.

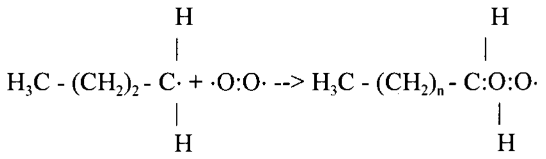
$R\cdot + [\text{cation}]^{+} \rightarrow R^{-} + [\text{cation}]^{++}$

Since free radicals are electrically neutral, they do not directly contribute to the value of the power factor or the interfacial tension. However, by capturing an electron a free radical can readily become a charge carrier.

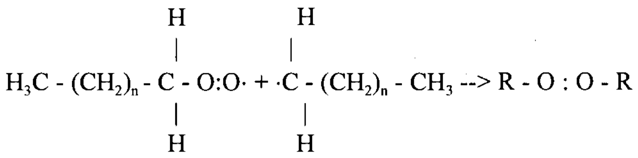
$R\cdot + e^{-} \rightarrow R^{-}$

It would therefore be desirable to determine the concentration of the free radical precursors. The alternative method presented in this paper can be used to determine the content of free radicals arising from these precursors and provide an indirect measure of the concentration of vulnerable molecules that negatively influence the oil stability. Having the potential to increase the power factor, the presence of free radicals accelerates the aging process of the oil because diradical oxygen molecules from the naturally-dissolved air content (about 10% by volume) in free breathing power transformers are able to initiate chain reactions.

Finally, the oxidation of free radicals remaining in the liquid phase after a small part of the broken hydrocarbon chain evolves as a gas, generates dissolved decay products and ultimately, sludge:

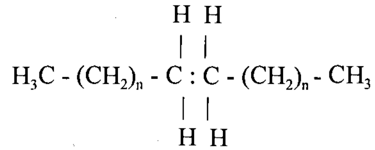


$$H_3C - (CH_2)_2 - \underset{H}{\overset{H}{C}}\cdot + \cdot O{:}O\cdot \dashrightarrow H_3C - (CH_2)_n - \underset{H}{\overset{H}{C}}{:}O{:}O\cdot$$



$$H_3C - (CH_2)_n - \underset{H}{\overset{H}{C}} - O{:}O\cdot + \cdot\underset{H}{\overset{H}{C}} - (CH_2)_n - CH_3 \dashrightarrow R - O : O - R$$

When a chemical reaction occurs between such liquid free radicals of large molecular weight without the involvement of oxygen, insoluble x-waxes are formed:



$$H_3C-(CH_2)_n-\underset{\underset{H}{|}}{\overset{\overset{H}{|}}{C}}:\underset{\underset{H}{|}}{\overset{\overset{H}{|}}{C}}-(CH_2)_n-CH_3$$

Both sludge and x-waxes favour the formation of hot spots by clogging the pores of paper insulation.

3. EXPERIMENTAL

The chemistry of mineral insulating oils uses the term "stability" to define two different properties of this blend of hydrocarbons, as follows:

- oxidation stability intended to portray the ability of oil to avoid chemical reactions with oxygen--the object of ASTM Designation D 2440-97, and
- stability under electrical discharge meant to characterize the behaviour of oil when submitted to the impact of a high voltage electrical field, as described by ASTM Designation D 2300-85 and D 6180-97.

During the oxidation stability test the properties of liquid insulation are deteriorated by a chemical cause, or more exactly, the aggressiveness of dissolved oxygen, that generates soluble decay products and sludge. In contrast, the stability of oil measured under conditions of electrical discharge assesses the damage inflicted by a physical cause, namely, the impact of a high voltage electrical field capable of breaking down a hydrocarbon chain. In the following chapters, the limitations of existing testing procedures of a physical nature are revealed and progress made towards enhancing the reliability of these laboratory techniques is presented.

3.1 ASTM Test Method D 2300-85

The unique goal of this testing procedure entitled, A Standard Test Method for Gassing of Insulating Oils Under Electrical Stress and Ionization (Modified Pirelli Method) is to determine the amount of gases generated by an oil sample under the effect of electrical stress. When the breakdown of a hydrocarbon molecule occurs, a small portion evolves as a gas, while the rest of the chain remains in the liquid phase as a free radical. However, the large content of these highly reactive species reduces the accuracy of quality determinations as mentioned in Section 4.2. According to this section, "In this test method, hydrogen (along with low molecular weight hydrocarbons) is generated by ion bombardment of some oil molecules and absorbed by chemical reaction with other molecules. The value reported *is the net effect of these competing reactions.*"

Moreover, in section 4.3 the fact is underscored, "There has been no quantitative relationship established between the gassing tendency as indicated by this test method and the operating performance." In order to overcome these shortcomings an alternative stability test method was developed over the past years and approved by Technical Committee D 27 two years ago as Designation D 6180-97.

3.2 ASTM Test Method D 6180-97

Unlike the gassing tendency test, this procedure, in addition to measuring the quantity of gases evolved, evaluates the changes that occur in the physical and chemical properties of the analyzed sample due to the decomposition of some hydrocarbon molecules. As a result, this test better simulates the process that takes place under real-life conditions and illustrates the manner in which high voltage fields affect the stability of mineral insulating oils.

3.2.1 The Gases Evolved Under Electrical Stress

One major advantage of this test method is that it measures the amount of gases evolved under electrical stress by monitoring the increase of pressure in the discharge cell. After five hours of electronic bombardment under vacuum conditions, the pressure developed indicates the intensity of the decomposition process. Although all hydrocarbon molecules are electronically excited, only a very small number decompose. The vast majority lose absorbed energy in the form of light, as shown by the visible violet glow on the surface of the oil. The unchanged value of the refraction index, measured before and after the stability test, confirms the validity of this conclusion. The qualitative relationship between the amount of gases evolved and the pressure inside the cell as it is illustrated in Fig. 1 provides a means by which the gassing tendency of insulating oils may be characterized. A small amount of evolved gases is normal even for an oil of high quality, but such a good gassing tendency should be consistent with other important physical and chemical properties that affect the insulating properties of tested oil samples.

3.2.2 The Dissipation Factor

As mentioned previously, the evolution of gasses generated by the decomposition of long hydrocarbon chains leaves molecules with a broken covalent bond in the liquid phase. When the split of the electronic doublet takes place homolitically both components are electrically neutral. When such a decay product catches a free electron it becomes a charge carrier that increases the dissipation factor of the oil. Therefore, besides a low gassing tendency, a high quality oil also requires a slight increase of dissipation factor, as measured after the stability test. According to the existing database, a low gassing tendency is not always consistent with a small value of dissipation factor. Table 1 indicates the dissipation factor of oil samples shown in Fig. 1.

Table 1 - *Dissipation Factor Increase of Oils Subjected to an Electrical Stress*

Oil Type	Dissipation Factor at 100°C, %	
	Before Test	After Test
Reclaimed Sample 1	0.38	0.72
New Sample 2	0.32	0.94
Aged Sample 3	0.83	3.36

3.2.3 Free Radicals

After the homolitical breakdown of a covalent bond, two molecules each having an unpaired electron arise. Usually, the small part is a gas that dissolves in the oil, but the large section of the chain remains as a component of the liquid phase. These chemically highly reactive species called free radicals [*7*] are the forerunners of soluble and insoluble decay products. The presence of free radicals in new or aged oils can be experimentally confirmed using the very sensitive reaction of the stable free radical 2,2-diphenylpicrylhydrazyl (DPPH). Solutions of DPPH, even at a concentration of 10^{-5} M, are intensely blue-violet coloured with maximum absorbance at 530 nm. When the DPPH free electron forms a doublet with the electron of another free radical, the solution changes colour from blue to yellow, with a corresponding decrease in absorbance. This change allows determination of the reaction kinetics [*8*]. Based upon this process a laboratory technique for the quantitative assessment of free radicals was developed and submitted to ASTM for consideration by Technical Committee D 27.

In addition to determining the gas evolved from an insulating oil, the standard method D 6180-97 can be used to determine the free radical concentration before and after the stability test. In order for free radicals to be formed the oil must contain their precursors, which may be either vulnerable hydrocarbons that were not eliminated during the refining process or contaminants accidentally introduced into the oil. The sensitivity of this precise analytical technique permits stability comparisons among different brands of insulating oils. A significant increase of DPPH consumption before and after exposure to electrical stress, as shown in Fig. 2, indicates a large concentration of vulnerable hydrocarbons.

3.2.4 The Buildup of X-Waxes

The formation of insoluble decay products is a major concern related to the purity of transformer oils in service. They reduce the breakdown voltage of oil and can clog the pores of paper insulation, hampering the dissipation of heat. For this reason the measurement of oil turbidity before and after the stability test is considered to be of paramount importance. This determination became possible by the development of a new ASTM procedure entitled, "Standard Test Method for Measurement of Turbidity in Mineral Insulating Oil of Petroleum Origin," approved two years ago as Designation D 6181-97. Thus, the gassing tendency test becomes more relevant, demonstrating that the breakdown of vulnerable hydrocarbons can have an additional negative effect on the insulating properties of oil. In real-life conditions hot spots and incipient electrical failures cause such deterioration, while their effect is detected by analyzing an average oil sample in which the decay products are highly diluted. For this reason, the tendency of oil to generate colloidal suspensions during the stability test should be as low as possible.

Table 2 contains the value of turbidity in several oil samples before and after the stability test.

Table 2 - *The Turbidity of Oil Samples in NTU**

Oil Type	Before Test	After Test
New Oil A	0.12	0.99

New Oil B	0.45	1.64
New Oil C	0.5	7.2
Highly Deteriorated Aged Oil	27.8	23.0

*Nephelometric Turbidity Units

3.2.5 Shift of Absorbance Curve in the Visible Spectrum

The recorded absorbance curve of a new oil sample, when scanned in the visible spectrum, lies in the region between 350-400 nm. When the oil is slightly oxidized, as is the case in free breathing power transformers, a shift of this curve takes place towards longer wavelengths. On the other hand, when an aged insulating oil is reclaimed, the selective removal of dissolved oxidation products shifts the absorbance curve towards shorter wavelengths, as shown in Fig. 3. Artificial oxidation as well as the reclamation of oil in laboratory conditions confirm this pattern. This methodology therefore offers a means of quantitatively determining the extent to which aged oils have suffered oxidation decay and the effectiveness of purification processes, such as the reclamation of aged oils. The same technique permits the assessment of changes that occur during the exposure of oil to electrical discharges. Due to the accuracy of this spectral analysis, the oxidation decay of oil can be monitored in its early stages. For instance, at the end of a stability test when air is introduced into the discharge cell, the absorbance curve of a new oil shows a slight shift towards longer wavelengths, as can be seen in Fig. 4.

4. ECONOMIC CONSIDERATIONS

By means of a laboratory simulation of the free electron injection process that provides the energy capable of breaking down hydrocarbon chains under real-life conditions, it is possible to establish a quantitative relationship between the results of the stability test and performance of the oil in service. Improved knowledge regarding the mechanism by which a high voltage field interferes with the chemistry of insulating oil provides a new means of maximizing the effectiveness of reliability-centered preventive maintenance procedures while minimizing their cost.

4.1 Cost Saving Predictive Maintenance

In order to avoid the expenses involved in aged oil purification procedures used *to prevent* an electrical failure, it is essential *to predict* the premature deterioration of fluid insulation while in service. It is important that this be accomplished to ensure the operational safety of power transformers. The newly developed stability test D 6180-97 now offers a means of making this prediction. Not only can the behaviour of a new oil under electrical stress be characterized by this laboratory technique; the effectiveness of aged oil reclamation procedures can also be determined. The results of the stability test also convincingly demonstrate the salutary effect of reclamation on the gassing of oil.

4.2 Improved Fault Gas Diagnosis

The dissolved gas analysis (DGA) method plays an important role world-wide in detecting a hot spot or an incipient electrical failure inside the windings. Although there are several methods for interpreting the results of DGA, none of them offers full satisfaction. Without knowing the initial gassing tendency of oil, it is difficult to establish whether the deterioration of purity has reduced its ability to resist decomposition under the same electrical stress, or whether an incipient failure is the cause of increased gas content. Since the reliability of transformers is a major concern for the electrical power industry, the determination of oil stability under electrical discharge should be the first priority if costly consequences of an erroneous fault, gas diagnosis are to be avoided.

4.3 Extension of Transformer Life Expectancy

It is an established fact that the auto-oxidation decay process of oil involves a species of intermediary reaction products called free radicals. As mentioned earlier, these intermediaries can be generated both by chemical means, such as the attack of oxygen, a diradical, and by the impact of electrical stress. Therefore, it is believed that a close relationship exists between the stability of oil under electrical stress and its oxidation stability.

When oil is less vulnerable under the effect of high voltage field, thus generating a small amount of gases and free radicals, it is also more resistant to oxidation. The stability test designated D 6180-97 was developed to provide information regarding the quality of insulating oils. Knowing that under in-service conditions an oil generates free radicals, in free breathing transformers, reaction with the dissolved oxygen is inevitable, even in the presence of oxidation inhibitors.

An additional reason for selecting the most stable oils, especially for very high voltage transformers, derives from the fact that free radicals are *paramagnetic*. Being attracted by the magnetic core of a transformer, the concentration of these free radicals may be lower in an average oil sample taken from the bottom of the tank than in samples taken from a region close to the windings. By preventing the deterioration of oil in service, the ageing of paper insulation is minimized and the life expectancy of power transformers significantly extended.

5. CONCLUSIONS

5.1 The gassing of oil under electrical stress is the aftermath of vulnerable hydrocarbon decomposition, caused by the free electron injection process into the liquid insulation. E. O. Forster extensively studied this phenomenon.

5.2 An important side effect of gassing is the formation of liquid-phase decay products such as free radicals, charge carriers and x-waxes. These contaminants contribute to the deterioration of insulating oils.

5.3 The ASTM Standard Test Method D 6180-97 was developed to simulate the impact of free electrons on the stability of mineral insulating oils, under laboratory conditions.

5.4 In addition to measuring the total gases evolved, this test method also allows for the determination of the concentration of free radicals, charge carriers and x-waxes (large molecular weight hydrocarbons).

5.5 Because this test is predictive in nature, the effectiveness of both different refining technologies of new oils and the reclamation procedure for aged oils can be reliably assessed.

5.6 Since the gassing process increases the free radical content in insulating oil, the oil should be kept free of dissolved oxygen, as in sealed or nitrogen blanketed power transformers.

5.7 The paramagnetic nature of free radicals, resulting from their unpaired electrons, as well as their chemical reactivity, suggests that any free radicals in the solid and liquid insulation of power transformers are considered to be potentially harmful.

5.8 Depletion of free radical forerunners results during their oxidation or ionization reactions. The selective removal of these decay products by reclamation improves the stability of oil under electrical discharges.

5.9 An improved understanding of the mechanism by which insulating oil deteriorates and a more advanced purification technology provide the means by which the reliability of power transformers may be enhanced, by maintaining the purity of the liquid insulation close to its initial value for the expected lifetime of these expensive electrical machines.

6. RECOMMENDATIONS

6.1 Finalization of the development process for the new ASTM "Standard Test Method for Measurement of the Relative Free Radical Content of Insulating Oils of Petroleum Origin," currently under consideration by Technical Committee D 27.

6.2 To initiate an inter-laboratory co-operative project for determining the reproducibility of the Relative Content of Free Radicals.

6.3 To develop a data base upon which to determine the stability and turbidity of different brands of insulating oils, according to the newly approved standard methods D 6180-97 and D 6181-97.

6.4 To study the formation of x-waxes (large molecular weight hydrocarbons) during the stability test under electrical discharge, by using more accurate analytical techniques such as particle counter and size characterization.

6.5 To develop an accurate spectrometric procedure for determination of the removal of non-acidic oxidation decay products during the reclamation of aged insulating oils, and the effect of oxidation reactions in the early stages of the decay process.

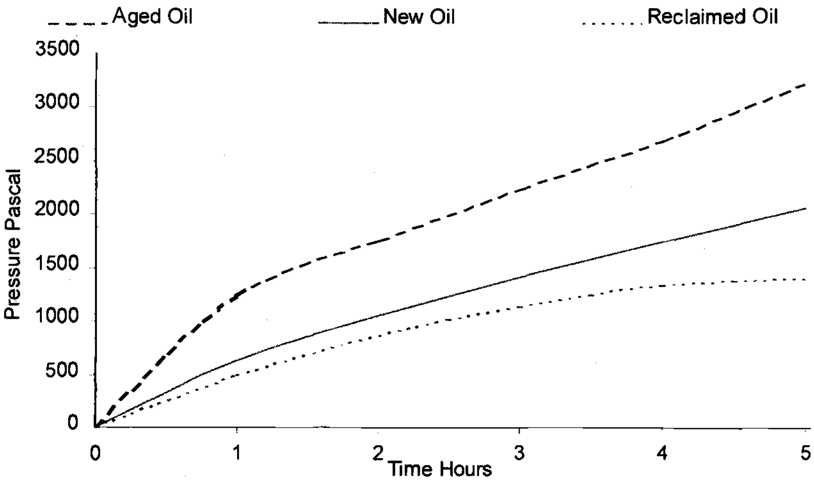


Fig. 1 Pressure Increase With Time for Insulating Oils Subjected to Electrical Discharge

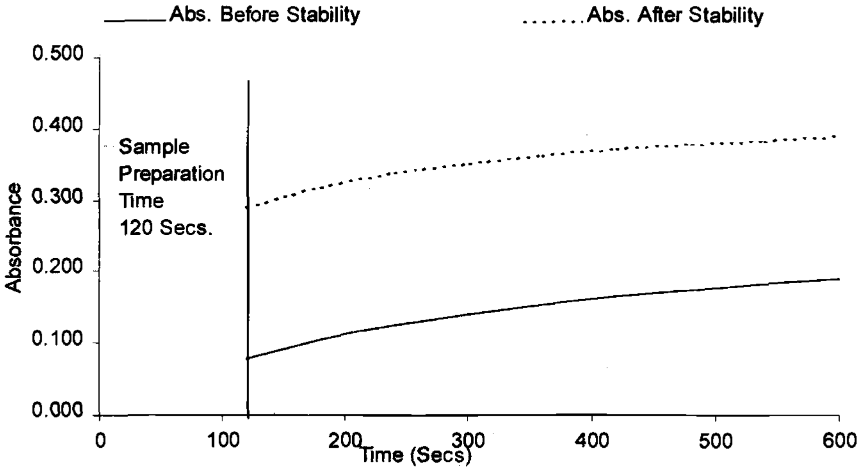


Fig. 2 Comparison of Insulating oil DPPH consumption before and after the stability test under electrical discharge

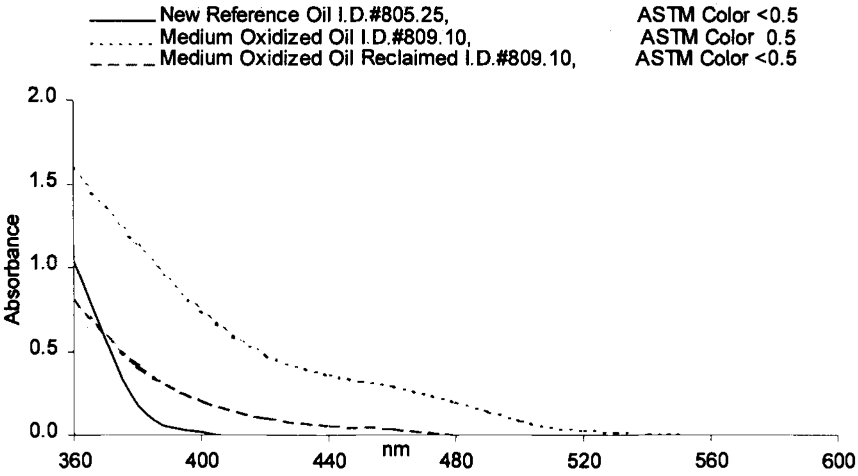


Fig. 3 Shift of Absorbance Curve in the Visible Spectrum

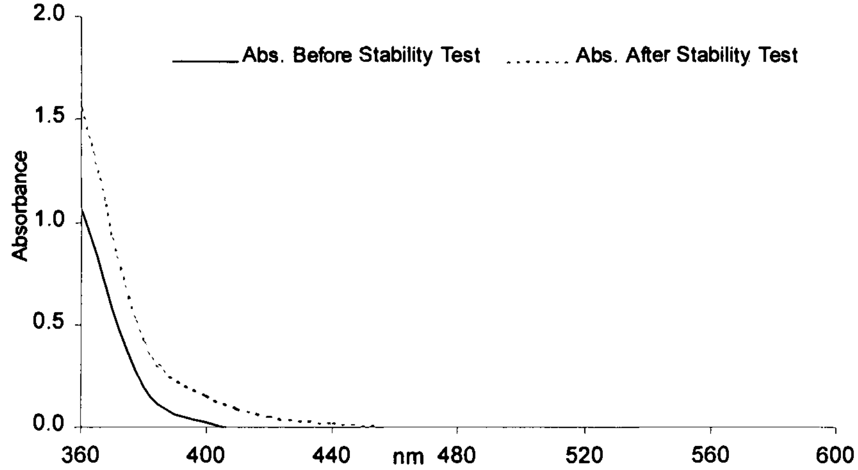


Fig. 4 Shift of absorbance for new oils towards longer wavelength after stability test

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This Applied Research project, intended to clarify the mechanism by which the high-voltage field deteriorates the dielectrical properties of mineral insulating oil, was initiated by INSOIL Canada Ltd. A decade ago and carried out with the solid support of the Southern Alberta Institute of Technology (SAIT). Special recognition must be given to Mr. Dave Walls, Dean of the Energy and Natural Resources Department, as well as Mr. Dan Violini, the coordinator of applied research activities, who played a decisive role in the successful conclusion of the project.

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Electrical Insulating Mineral Oils: Specifications and Products

Reference: Griffin, P. J., Rouse, T. O., and Kelly, J. J., "**Electrical Insulating Mineral Oils: Specifications and Products,**" *Electrical Insulating Materials, International Issues, ASTM STP 1376,* M. M. Hirschler, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

Abstract: This paper provides a review of two specifications for electrical insulating mineral oils by ASTM and the International Electrotechnical Commission (IEC). The physical, electrical, and chemical properties of these fluids are specified for use in electric apparatus such as transformers. The specified test methods are compared and their differences are presented. Some properties of commercial oils are shown.

Keywords: electrical oil, oil, insulating oil, specification, electrical properties, physical properties, chemical properties, oxidation stability

Electrical insulating mineral oils were developed and first used late in the 19th century. Nearly all load-bearing transformers in the electric power delivery systems around the world are filled with some type of this liquid. The liquid functions both as a dielectric insulator and as a heat transfer fluid. Today, there is on the order of a billion gallons of these petroleum-based insulating oils in use, in transformers, in the U.S. alone [*1*].

Transformers in electric power distribution and transmission systems are expected to function reliably and efficiently and to do this for many years. The quality of the oil in a transformer plays an important role in performing this function. Mineral oils used in electric apparatus are specified to have certain characteristics that permit reliable performance for many years. Oils to be used in transformers are made by many regional and international refining companies, each using a particular crude oil(s) and refining technology. The chemical, electrical, and physical characteristics of the transformer oil produced by each supplier then are, to some degree, unique to that oil. To assure that an oil is acceptable for use in specific apparatus, relevant characteristics are defined in specifications agreed to by manufacturers and users of electrical equipment and refiners of oils. These standard specifications or guides are developed by groups made up of experts in transformers and transformer materials. This paper compares and discusses two specifications used by many transformer owners worldwide for the procurement of electrical insulating mineral oils, ASTM D 3487 and IEC (International Electrotechnical Commission) 60296. Differences and similarities in the test methods specified are listed in order to properly compare limit values. Examples are given of commercial oils produced to meet one or both of these specifications but which have characteristics, such as very low pour points, to meet local requirements. Recent reviews

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provide more details on the manufacture, testing, and qualities of electrical insulating mineral oil [*1–3*].

ASTM Committee D-27 is responsible for the Specification for Mineral Insulating Oil Used in Electrical Apparatus (D 3487). IEC Technical Committee 10, made up of delegates from a number of national committees (including that for the U.S.) is responsible for the IEC 60296, Specification for Unused Mineral Insulating Oils for Transformers and Switchgear. ASTM D 3487 and IEC 60296 differ in a number of ways, as do the standard test methods developed by Committees D-27 and TC10.

It should be noted that limited quantities of mineral oils are produced for higher fire-point less flammable applications. A number of the characteristics of high fire-point oil (also referred to as less flammable hydrocarbon fluids) do not meet the limits set for conventional mineral transformer oil. For this reason there is a separate ASTM Specification for High Fire-Point Mineral Electric Insulating Oils, D 5222. For example, the viscosity of "high fire-point" oil is substantially higher than that specified in D 3487 or IEC 60296. As a result, the transfer of heat by this oil is less efficient than by conventional mineral transformer oil, and transformers must be specially designed for its use.

The ASTM and IEC specifications have tests for functional characteristics, quality control measures, and continuity or consistency of production. The relevant characteristic of the oil may be an intrinsic property used in the design of transformers or it may be the concentration of a specific component or impurity. In some cases, the characteristic is the response of an oil to a carefully controlled test procedure in a small laboratory test fixture intended to simulate actual behavior of an oil in an operating transformer. In still other cases, the protocol of a specified standard test does not simulate performance of an oil in a transformer; instead it provides a similar means for which a significant characteristic of liquids can be compared. In many cases ASTM and IEC test methods are so similar that values obtained should be within the stated precision of the test methods for the same property. However, for some characteristics the measurements are performed in a sufficiently different manner that direct comparison is not advisable. Electrical insulating oils produced to different countries' specifications may have characteristics which have been tailored to suit local operating conditions or performance criteria. It is of interest to see the interdependency of some characteristics, when modifications are made to insulating oils to adapt to local requirements.

Specifications and Tests

The ASTM D 3487 and IEC 60296 specification values and tests are compared in Tables 1 through 18. The test methods given in these specifications are classified (adjacent to the test method) as being:

- Similar (S) if they are essentially the same and are expected to yield equivalent results.
- Different (D) if the methods are so dissimilar that the results from the two tests are not expected to be consistently the same.
- Similar but different (SD) if the test method is somewhat different but, nonetheless, the data is expected to be similar.

Significant differences are noted when the test methods are different. General discussion of the properties described by these specifications is included. All of the specified tests in D 3487 and IEC 60296 are ASTM and IEC test methods respectively unless otherwise indicated.

Physical Characteristics

Aniline Point—The aniline point is the temperature at which a mixture of aniline and oil separates. It provides a rough indication of the total aromatic content, and relates to the solvency of the oil for materials in contact with it. The aniline point does not provide a measure of the type of aromatics present or their relative composition. The lower the aniline point, the greater the solvency effects. There is a requirement for aniline point in ASTM D 3487 listed in Table 1, but not in IEC 60296.

Although not specified by the IEC, many of those affected by European directives include a 3% by weight limit for polycylic aromatic compounds (PACs) as measured by IP 346 so that the oils purchased do not require special hazard labels. PACs are also called polycyclic aromatics (PCAs) or polynuclear aromatics (PNAs). In the IP 346 method, solvent extraction with dimethyl sulfoxide (DMSO) is used to isolate PACs from the portion of the oil in the boiling range of 300°C and higher. The amount of PACs is determined gravimetrically. The method is not suitable for used oils.

It is desirable to have a test that better characterizes the composition of aromatic compounds than aniline point. Changes in crudes and refining techniques can affect the aromatic content of a product, and therefore aniline point can be used to provide a measure of the consistency or continuity of product. One of the difficulties of specifying aromatic content is that generally similar, overall aromatic content in different crudes with dissimilar compositions of PACs, polar compounds, heterocyclic nitrogen, and sulfur compounds, etc., may yield oils with different performance characteristics. Gassing tendency under partial discharge conditions, impulse breakdown voltage, and oxidation stability are all affected by the amount and types of aromatics.

The rationale for the lower limit for aniline point is to provide an oil that is compatible with materials of construction such as gaskets. The upper limit is recommended, by some, to restrict over-refining. Further research is needed to select or develop a better method for determining aromatic content for which a consensus could be reached for creating a new specification.

Color—The color of a new oil is used as a general index of the degree of refinement. For oils in service, an increasing or high color number is an indication of contamination, oxidative or thermal deterioration, or both. Visual inspection of transformers, which are exhibiting evidence of an internal problem, can be performed more easily when the oil is not darkly colored. There is a requirement for color in ASTM D 3487 but not in IEC 60296 as given in Table 1.

Flash Point—The flash point is the minimum temperature at which heated oil gives off sufficient vapor to form a flammable mixture with air. It is also an indicator of the volatility of the oil. Minimum values need to be above the maximum anticipated operating temperatures of the bulk oil in the apparatus. This removes the risk of forming a flammable vapor should the oil be exposed to sufficient air while in service. Specified values are listed in Table 2. For the IEC specification there are three classes of oils with different physical

TABLE 1—*Specifications for aniline point and color.*

	ASTM D 3487	IEC 60296
Aniline Point, °C	63–84	None
Test Method	D 611	
Color, max.	0.5	None
Test Method	D 1500	

TABLE 2—*Specifications for flash point.*

	ASTM D 3487	IEC 60296
Flash Point, °C Min.	145	140 Class I 130 Class II 95 Class III
Test Method	D 92(D)	ISO 2719 (D)

TABLE 3—*Test methods for measurement of flash point.*

	ASTM D 3487	IEC 60296
Cup	Cleveland Open Cup	Pensky-Martins Closed Cup
Temperature rise	14–17°C/min up to 56°C below expected flash point, then 5–6°C/min	5–6°C/min
Test with flame	Every 2°C starting 28°C below expected flash point	Every 2°C starting at 17–28°C below expected flash point
Stirrer	No	Yes
Repeatability	8°C	6°C
Reproducibility	17°C	8.5°C

TABLE 4—*Specifications for interfacial tension.*

	ASTM D 3487	IEC 60296
Interfacial Tension Dynes/cm or Millinewtons/m, min	40	None If desired 40
Test Method	D 971(S)	ISO 6295(S)

TABLE 5—*Specifications for pour point.*

	ASTM D 3487	IEC 60296
Pour Point, °C Max.	−40	−30 Class I −45 Class II −60 Class III
Test Method	D 97 (S)	ISO 3016 (S)

TABLE 6—*Specifications for relative density and density.*

	ASTM D 3487	IEC 60296
Relative Density 60/60°F 15.15°C	0.91 max.	None
Density @ 20°C, kg/dm^3, Max.	None	0.895
Test Method	D 1298(S)	ISO 3675(S)

property requirements. Class III oils are intended for certain circuit breaker applications. The specified test methods for measurement of flash point are quite different as shown in Table 3. With the closed-cup test, values for a given oil will be lower than the results from the Cleveland open cup test. [There is an ASTM Pensky-Martins closed cup method (ASTM D 93) that is very similar to ISO 2719. There is also an ISO method 2592 that is quite similar to the ASTM Cleveland open cup method D 92.]

Interfacial Tension—The interfacial tension (IFT) of an oil is the force in millinewtons per meter required to rupture the oil surface existing at an oil-water interface. It is a measure of the degree of refining and contamination of new oils, and serves as a benchmark to evaluate in-service oils. For oils in service, a decreasing value indicates the accumulation of contaminants such as metal soaps, paints, varnishes, or oxidation byproducts. Also the IFT is reduced by precursors of oxidation products that may attack the insulation and interfere with the cooling of transformer windings. The ASTM and ISO methods for measurement of IFT are very similar. IEC 60296 does not have a mandatory requirement for IFT. Where it is used as an optional test the limit is the same as that for the ASTM specification (Table 4).

Pour Point—The pour point is the lowest temperature at which oil will just flow. The test is performed by determining the temperature below which a liquid will not move on tilting the container in which the oil has been cooled. It is a measure of the flow characteristics at low temperatures under variable and undefined shear rates and stresses. The geometry of the container and the cooling protocol are specified in the standard test method. Care should be used in extrapolating pour point temperatures to temperatures at which oil "solidifies" in actual equipment in service. Variations of up to 15°C have been found in the results of the test if the standard protocol is not followed exactly. A low pour point is important, particularly in cold climates, to ensure that the oil will circulate and serve its purpose as an insulating and cooling medium. In warmer climates, higher pour points may be permissible. The ASTM limit for pour point given in Table 5 is between the requirements for IEC Class I and Class II oils. The Class III oils are produced for minimum oil circuit breakers and not for use in transformers.

Relative Density (Specific Gravity) and Density—The relative density of an oil is the ratio of the weights of equal volumes of oil and water determined under specific conditions. The density is the mass of a material per unit volume.

In extremely cold climates the specific gravity or density has been used to determine whether ice, resulting from the freezing of water in oil filled apparatus, will float on the oil and possibly result in flashover of conductors. The purpose of the specification limits is to minimize this risk. ASTM and IEC limits shown in Table 6 are similar if corrected for the differences in the specified temperatures of the measurements and reported in equivalent units. The test methods both use hydrometers and are similar.

The coefficient of thermal expansion, i.e., the change in density with temperature, has a major role in heat transfer.

Viscosity—The viscosity of the insulating oil is a principal parameter in design calculations for heat transfer by either natural convection in smaller self-cooled transformers or forced convection in larger units with pumps. Dynamic viscosity is a measure of the resistance of a liquid to flow, the ratio of shear stress to shear rate. Kinematic viscosity is the ratio of the dynamic viscosity of a liquid to its density. Generally, the viscosity of a mixture increases as the molecular sizes and molecular weights of compounds in the ensemble increase. It has been pointed out that the electrical insulating liquid in a transformer must function both as a heat transfer fluid as well as a dielectric material. This is because oil is used alone as electrical insulation only in regions in a transformer where, by design, voltage stresses are relatively low.

A primary objective in the design of a reliable transformer is to limit internal temperatures. In an effective design, a balance is reached between the heat generated by electrical loading (and overloading) the core and coils in the transformer and the capacity of the internal oil circulation system to remove this heat to cooler external surfaces. The standardization of limits on kinematic viscosity at several temperatures allows transformer manufacturers to develop standard design practices for the sizing and placement of oil ducts, reservoirs, baffles, pumps, radiators, etc.

The ASTM and IEC test methods for viscosity are quite similar. Specified measurement temperatures listed in Table 7 are only the same at 40°C. Again the IEC has three classes of oils with different physical properties. The Class III oils are for use in certain types of circuit breakers.

Dielectric Characteristics

Dielectric Breakdown Voltage—The dielectric breakdown voltage is the minimum voltage at which electrical breakdown occurs in a specified oil gap. It is a measure of the ability of an oil to withstand electrical stress at power frequencies. There are two different ASTM tests and one IEC test. The test conditions of these methods have been chosen to emphasize the role of water and particles in the breakdown of the test specimen. Conducting particles, wetted or dry, can reduce the ac breakdown strength of an oil in a gap below the intrinsic strength of the hydrocarbon ensemble. Methods D 1816 and IEC 60156 are more sensitive to low concentrations of water and particles than is Method D 877. The voltage stress is highest in the liquid-filled gap between spherical caps at the center of the electrodes used in the D 1816 and IEC 60156 tests. The contouring allows the stress to fall off smoothly toward the electrode edges. The stress is relatively uniform in the gap between the caps where the breakdown occurs.

In Method D 877, the breakdown voltage of an oil sample is measured between parallel circular disks with flat faces. The face and the barrel of a disk are orthogonal and the edge at the rim of the face is sharp. There is a substantial voltage stress concentration at this edge and the electrical field in the gap between electrode faces is quasi-uniform. Breakdown is dominated by events occurring in the liquid in the regions near the edges of the disks where the electrical field is enhanced.

In some cases, tests are performed only on the oil as received, whereas in other cases, the requirement is for the testing after treatment (Table 8). Clearly, a requirement is needed for the oil as received so that purchasers and sellers have a means for completing a transaction.

TABLE 7—*Specifications for viscosity.*

	ASTM D 3487	IEC 60296
Viscosity, cSt (SUS)		16.5 Class I
40°C, max.	12.0	11.0 Class II
	(66)	3.5 Class III
100°C, max.	3.0	None
0°C, max.	76.0	None
	(350)	
−15°C, max	None	800 Class I
−30°C, max.	None	1800 Class II
−40°C, max	None	150 Class III
Test Method	D 445(S)	ISO 3104(S)
	(D 88)	

TABLE 8—*Specifications for dielectric breakdown voltage.*

	ASTM D 3487	ASTM D 3487	IEC 60296
Dielectric Breakdown Voltage, kV, min	30* (2.5 mm gap)	28* (1 mm gap) 56† (2 mm gap)	≥30* (2.5 mm gap) ≥50† (2.5 mm gap)
Test Method	D 877(D)	D 1816(D)	156(D)

* As delivered.
† After treatment.

However, it is worthwhile to have a limit for the treated oil as a measure of its inherent capability. It should not be a requirement to test the oil this way before shipment, so there is not an undue testing burden. There are numerous other differences between the test methods, some of which are outlined in Table 9.

Impulse Breakdown Voltage—The impulse breakdown voltage is the voltage at which electrical breakdown occurs in an oil under a specific shaped transient-voltage wave in a highly divergent field geometry. The results are dependent on electrode polarity. The ASTM limit for impulse breakdown voltage using method D 3300 is 145 kV with the needle negative and the sphere grounded, and a 1-in. (25.4-mm) gap. There is no corresponding IEC requirement for impulse breakdown strength. The impulse breakdown voltage of oils tends to decrease with increasing concentration of PACs, with the effect being more pronounced with increasing number of rings. With the pressure today for oils to have lower aromatic contents, and particularly lower PAC contents to alleviate environmental concerns, the impulse breakdown voltage of electrical insulating oils easily meets this requirement.

Gassing Under Electrical Stress—The gassing tendency can be defined as the rate of gas evolved or absorbed by an insulating oil when subjected to electrical stress of sufficient intensity to cause ionization. The oil sample in the test cell is saturated and blanketed with hydrogen gas (rarely, nitrogen). The characteristic is positive if gas is evolved and negative if gas is absorbed. The use of different saturating gases can affect the absolute value of the gassing tendency but typically does not affect the relative ranking of oils.

TABLE 9—*Test methods for measurement of dielectric breakdown voltage.*

	ASTM D 877	ASTM D 1816	IEC 60156
Oil	New or service aged	Processed or service aged	New or service aged
Cell volume	Not specified	400–1000 mL	300–500 mL transparent preferably closed
Electrode geometry	Disk	VDE*	VDE*
Electrode spacing	2.5 mm	1 or 2 mm	2.5 mm
Voltage rise	3 kV/s	0.5 kV/s	2 kV/s
Breakdowns	5 or 10	5 or 10	6
Time to begin test after filling cup	≥2 but ≤3 min	≥3 min	About 5 min, and no gas bubbles in gap
Stirring	No	Yes	Optional
Time between breakdowns	1 min	At least 1 min	At least 2 min, and no gas bubbles in gap

* Circular electrodes whose faces are contoured according to Verband Deutscher Electrotechniker Specification 0370 (VDE).

The overall rate of change of gas volume in an ASTM D 2300 test cell is the result of the rates of two processes in the oil/gas froth where the partial discharge occurs. Activated products, primarily hydrogen, are produced by the decomposition of the oil. Activated hydrogen species are formed at the same time in the hydrogen blanket. The active species formed in both processes hydrogenate aromatic hydrocarbons in the oil and are incorporated into the condensed phase at a rate limited by the concentration and type of aromatics present. Excess activated species revert to hydrogen gas in its ground state and add to the volume of gas in the cell. Oil in which the rate of uptake of active hydrogen exceeds the rate of its production from the decomposition of the oil in the discharge "absorbs" hydrogen from the gas blanket. Oil in which active hydrogen is generated by the decomposition of hydrocarbons in the discharge at a greater rate than the rate of uptake of hydrogen by aromatic hydrocarbons "evolves" gas to the blanket. Gas evolution in this test increases as the aromatic hydrocarbon contents of oils decrease.

The wide variety of conditions under which partial discharges occur in operating transformers cannot be reproduced in a standard test performed on oil in a laboratory. However, molecular hydrogen is the principal degradation product of low energy electrical discharges found in mineral oil-filled transformers. The solubility of hydrogen is low in transformer oils, and gas bubbles readily can form in the oil in the vicinity of a discharge. Gas bubbles are weak dielectrics and their presence can intensify the partial discharge or initiate further breakdown in highly stressed regions nearby. No specific correlation has been established between the rates of gas evolution of transformer oils measured by the D 2300 test and the performance of these oils in transformers. The likelihood of damage to the insulation system in a transformer by low energy discharges is clearly higher in oil in which a high gas evolution rate is measured in test method D 2300. For some equipment, such as cables, bushings and instrument transformers, certain capacitors, or for insulation which is only partially impregnated, low gassing tendency oils may mitigate the effects of partial discharges. The ASTM specification lists limits for two test methods, D 2300 procedure A (maximum of 15 μL/min) and procedure B (maximum of 30 μL/min). There is no requirement in the IEC specification.

Power Factor and Dissipation Factor—The power factor of an insulating oil is the cosine of the phase angle (sine of the loss angle) between a sinusoidal potential applied to the oil and the resulting current. Dissipation factor is the tangent of the loss angle and is often referred to as tan delta. The relationship between the two values is given by

$$PF = D/(1 + D^2)$$

where PF = power factor, and D = dissipation factor.

For practical purposes the power factor and dissipation factor are equal numeric values for new oils. Both the power factor and dissipation factor indicate the dielectric loss of an oil and, thus, the dielectric heating. Dielectric losses in the insulation system of transformers are very small and normally ignored as a source of heat, except in the case of some specialty transformers. A high dissipation factor is an indication of inadequate refining or contamination of new oils. For in-service oils, increases in power factor or dissipation factor may be caused by the presence of byproducts of oxidation, contamination or deterioration products such as moisture, carbon or other conducting matter, and metal soaps. The power factor and dissipation factor increase exponentially with temperature. ASTM and IEC requirements are at different temperatures and are different limit values with ASTM being stricter by its 100°C requirement (Table 10). ASTM and IEC tests may be routinely performed at different power frequencies. The test methods for these measurements are similar and have enough tolerance that it is possible to perform a test in such a way as to meet the conditions of both

TABLE 10—*Specifications for power factor.*

	ASTM D 3487	IEC 60296
Dissipation Factor, or Power Factor, %, max.		
100°C	0.30	None
90°C	None	≤0.5
25°C	0.05	None
Test Method	D 924(S)	60247(S)

ASTM D 924 and IEC 60287. Differences include the amount of description of the test cells, cell cleaning procedures, heating requirements, and voltage stress. An example of the similarity of the test methods can be seen by examining the range of voltage stresses permitted. The ASTM test allows for voltage stresses equal to or between 0.2 to 1.2 kV/mm while the IEC requirement is from 0.03 to 1 kV/mm.

Chemical Characteristics

Accelerated Oxidation Tests—There are several accelerated oxidation tests which, in common, use a copper catalyst, high temperatures (between 100 and 140°C), and a supply of oxygen (pure or from air). These tests provide a reasonable measure of the continuity of production, and a simple measure of acceptable oxidation stability. Most of these tests use some measure of acid or sludge formation, its incipience, or the amount of byproducts of aging formed over a standard time. It is, of course, desirable to minimize sludge and acid formation in transformers. The variation in test conditions and properties measured to assess performance are listed in Table 11.

It is important to know if an oil contains added oxidation inhibitor for selection of the proper test and for evaluation of the test results. In the IEC classification, oils are either uninhibited and contain no added antioxidant, or are inhibited. In contrast, D 3487 classifies oils as either Type I, which contains up to a maximum of 0.08% of 2,6-ditertiary butyl phenol (DBP) or 2,6-ditertiary butyl para-cresol (DBPC), or Type II, which can contain up to 0.3% antioxidant. For IEC 60296, if the oil is uninhibited, then the oxidation stability test would be 61125A (formerly IEC 74). IEC 61125C can also be used to test uninhibited oils but is not a specification test. Any of the IEC oxidation stability tests can be used for inhibited oils; however, only IEC 61125B (formerly IEC 474) is specified. ASTM D 2112 is a rapid test method strictly for inhibited oils, while the sludge and acidity test D 2440 can be used with either inhibited or uninhibited oils.

The primary means for detecting and quantifying the most commonly used antioxidants, DBP and DBPC, are ASTM D 2668 and IEC 60666. Infrared spectroscopy is the common instrumentation used in both methods, although the IEC method has alternative techniques using thin layer chromatography and spectrophotometry to detect DBPC and other antioxidants. The ASTM and IEC methods using infrared spectroscopy are similar, showing modest differences in choice of reference oils, calibration, and precision and bias statements. An alternative method of measuring oxidation inhibitor content is listed in the ASTM specification, D 1473, but uses antiquated techniques and is likely to be replaced by a more recently published method using gas chromatography, method D 4768.

The limit values for the various accelerated oxidation tests are difficult to compare, as the test conditions are not the same. For the sake of completeness, the limit values for oxidation stability for the ASTM and IEC specifications are listed in Tables 12–14.

TABLE 11—*Comparison of accelerated oxidation tests.*

Factor	IEC 61125A	IEC 61125B*	IEC 61125C	ASTM D 2440	ASTM D 2112*
Temp., °C	100	120	120	110	140
Oil, wt.	25 g	25 g	25 g	25 g	50 g
Oxygen	99.4 min. purity @ 1 L/h	99.4 min. purity @ 1 L/h	Air @ 0.15 L/h	99.4 min. purity @ 1 L/h	99.5 min. purity 90 psi
Copper/ oil†	45.9	114	114	38.1	307§
Catalyst	Abraided copper 1.0–1.02 × 365 mm	Abraided copper 1.0–1.02 × 900 mm	Surface area of 28.6 cm^2	Abraided copper 1.01 × 300 mm	Abraided or acid etched copper 1.628 × 3000 mm
Property Measured	Acidity & sludge @ 164 h	Time to dev. NV‡ of 0.28 mg KOH/g for volatile acidity. Sludge may also be determined.	Determine sludge, and acid. Can use time volatile acids reach 0.1 mg KOH/g. Test can be for 164 or 500 h.	Sludge and acidity 72 and 164 h.	O_2 consumption estimation of induction period.

* Tests are specifically for oils containing added oxidation inhibitors.

† Area of copper mm^2/g of oil.

‡ NV = Neutralization value.

§ All of the copper is not exposed to the oil simultaneously as the bomb, while rotating, is held at an angle and, therefore, the copper/oil ratio is exaggerated.

TABLE 12—*Specifications for oxidation stability for uninhibited oils.*

	ASTM D 3487	IEC 60296
Oxidation Inhibitor, wt. %	Type 1 0–0.08	0 max.
Test Method	D 2668 (DS) D 1473 D	60666 (DS)
Sludge and Acid Accumulation		
72 h		
Sludge, wt. %, max.	0.15	None
Acid, mg KOH/g max.	0.5	
164 h		
Sludge, wt. %, max.	0.3	0.10
Acid, mg KOH/g max.	0.6	0.40
Test Method	D 2440 (D)	74 (D) (61125A)

TABLE 13—*Specifications for oxidation stability for Type I oils, with inhibitor.*

	ASTM D 3487	IEC 60296
Oxidation Inhibitor, wt. % max.	0.08	None
Test Method	D 2668 (DS) D 1473 (D)	60666 (DS)
Sludge and Acid Accumulation		
72 h		
Sludge, wt. %, max.	0.15	None
Acid, mg KOH/g, max.	0.5	
164 h, Sludge, wt. %, max.	0.3	
Acid, mg KOH/g, max.	0.6	None
Test Method	D 2440	None
Induction Time	None	120 h Induction period (not required)
Test Method	None	474 (61125B)

TABLE 14—*Specifications for oxidation stability for Type II oils.*

	ASTM D 3487	IEC 60296
Oxidation Inhibitor, wt. %, max.	0.3	None
Test Method	D 2668 D 1473	60666
Slude and Acid Accumulation		
72 h		
Sludge, wt. %, max.	0.1	None
Acid, mg KOH/g, max.	0.3	
164 h		
Sludge, wt. %, max.	0.2	None
Acid, mg KOH/g, max.	0.4	
Test Method	D 2440	None
Rotating Bomb, Minutes, min.	195	None
Test Method	D 2112	None
Induction Time	None	120 h Induction period (not required)
Test Method	None	474 (61125B)

Corrosive Sulfur—This test detects the presence of objectionable quantities of elemental sulfur and thermally unstable sulfur-bearing compounds in an oil. When present, these compounds can cause corrosion of certain transformer metals. Corrosive sulfur can sometimes be detected in transformers as a black sulfide coating on copper and silver surfaces. Both specifications require a noncorrosive condition at the conclusion of the corrosive sulfur tests given in Table 15, which are very similar.

TABLE 15—*Specifications for corrosive sulfur.*

	ASTM D 3487	IEC 60296
Corrosive Sulfur	Noncorrosive	Noncorrosive
Test Method	D 1275(S)	ISO 5662(S)

TABLE 16—*Specifications for corrosive sulfur.*

	ASTM D 3487	IEC 60296
Water Content, ppm, mg/kg, max.	35	None If desired 30 Bulk, 40 Drums
Test Method	D 1533(S)	60814(S)

TABLE 17—*Specifications for neutralization number.*

	ASTM D 3487	IEC 60296
Neut. No. Mg KOH/g, max.	0.03	0.03
Test Method	D 974(SD)	IEC 60296(SD)

TABLE 18—*Test methods for the measurement of neutralization number.*

	ASTM D 974	IEC 60296
Indicator	p-Naphtholbenzein	Alkali Blue
Solvent	Tolune/Isopropanol-water	Toluene/Ethanol
Blank	Run solvent as blank	Neutralize solvent
Precision Statement	Yes	No

TABLE 19—*Physical properties of some naphthenic oil.*

Oil	Viscosity, cSt	Flash Pt., °C	Pour Pt., °C
A	7.80	158	−51
B	7.97	146	−57
C	8.94	150	−61
D	9.30	154	−43
E	9.36	162	−60
F	9.51	148	−57
G	9.91	146	−59
H	10.45	152	−54
I	10.85	152	−61
J	13.54	168	−40

Water Content—A low water content is specified in D 3487 as necessary to obtain and maintain acceptable dielectric breakdown strength. Newly delivered oils that exceed the limit require more extensive processing of the oil before use in electric apparatus. There is not a mandatory requirement for water content in IEC 60296. When given in IEC based specifications the requirement is similar to that in D 3487 (Table 16). ASTM method D 1533 B is similar to IEC 60814 although there are modest differences for sampling, sample storage, test specimen size. Perhaps the biggest difference is that, for method 1533 B one test is performed and the result reported, whereas with IEC 60814 the average of duplicate tests are reported.

Neutralization Number—The neutralization number of an oil is a measure of the amount of acidic or alkaline materials present. New oils should have a very low acidity when properly refined. As oils age in service the acidity, and therefore the neutralization number, generally increases. Used oil having a high neutralization number indicates that the oil is either oxidized or contaminated with materials such as varnish, paint, or other foreign matter. A basic neutralization number results from an alkaline contaminant in the oil. The requirement for maximum neutralization numbers is the same (Table 17). Both methods are an acid-base titration with a colored indicator to visually detect neutrality. However, there are differences in the specific solvents and indicators as listed in Table 18.

Polychlorinated Biphenyls (*PCBs*)—PCBs are regulated in most countries and should not be present in detectable quantities in new mineral oil products. The ASTM specification states that PCBs should not be detectable when tested in accordance with method D 4059, a gas chromatography technique using either a packed or megabore column. There is no requirement in the IEC specification.

Characteristics of Commercial Products

Transformer oils are produced by refining a fraction of the hydrocarbons collected during the distillation of a petroleum crude oil. Transformer oils are tailored to meet specifications, and the boiling temperature range over which this fraction is collected determines many of the properties of the finished oil. For example, viscosity, pour point, and flash point are physical characteristics that are primarily determined by the boiling range of the collected portion of crude oil. The physical properties of a distillate of a given median boiling point, and boiling range may vary with the crude. The requirements on viscosity largely determine the selection of the median boiling point of the portion of the distillate used in the manufacture of transformer oil. Flash point and low temperature properties are determined respectively by the initial and final boiling points of the fraction; the higher the initial boiling point, the higher the flash point; and (for a wax-free crude), the higher the final boiling point, and the higher the pour point. In a very cold climate, a lower pour point requirement may be stricter than for warm regions. To meet these requirements refiners may need to select a portion of the distillate that has a lower viscosity and flash point than for oils with less stringent requirements for cold temperature flow characteristics. Selected data from the Doble Engineering Company Survey (Spring, 1997) [*4*], of naphthenic-based electrical insulating mineral oils without pour point depressants was examined. The data is shown in Table 19 tabulated by increasing viscosity. There is no obvious correlation in the data. Instead what the data suggests is that the ability to produce a very low pour point oil depends upon the crude source, and the distillate range available to a refiner for production of transformer oil. These factors may influence how easy it is to manufacture very low pour point oils without reducing the flash point below minimum specification values.

When a similar tabulation of extreme values for gassing tendency is compared with impulse breakdown voltage and aromatic content values as shown in Table 20, the relationship

TABLE 20—*Comparison of electrical properties and aromatic content.*

Oil	Gassing Tendency µL/min D 2300 Proc. B	Impulse Breakdown Voltage, kV D 3300	Aromatic Content % D 2140
K	−12.4	156	12.2
L	−12.2	198	10.0
M	−12.0	184	10.2
C	+25.6	260	7.4
N	+20.9	210	5.8
O	+17.4	184	4.8

between these properties is revealed. Oils with lower gassing tendency also tend to have lower impulse breakdown voltages and higher total aromatic contents. There appear to be other factors which have an influence, as Oils M and O both have impulse breakdown voltages of 184 kV and yet have very different gassing tendencies and aromatic contents. This may be due to differences in the relative amounts of mono- and poly-aromatic species present in the oils. The method for determining aromatic content, ASTM D 2140, does not provide an indication of the relative portions of the different types of aromatic hydrocarbons.

Conclusions

In today's world of increasing international trade it is important for manufacturers of apparatus and electrical insulating oils to be aware of specifications and requirements used by different organizations. These differences include those in the specified test methods and the limit values. As members of standards organization it is important for us to harmonize international standards where possible and to help to make interested parties aware of the differences.

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Electrical Tests

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New Ways of Disruptive Discharge Recognition in Insulation Testing Devices

Reference: Mathis, H.-J., Baur, M., Blank, R., Woschitz, R., and Überfall, T., **"New Ways of Disruptive Discharge Recognition in Insulation Testing Devices,"** *Electrical Insulating Materials: International Issues, ASTM STP 1376,* M. M. Hirschler, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

Abstract: The dielectric testing of gaseous and liquid insulation has a long tradition and devices for breakdown testing have improved during the past 40 years. Special liquids like silicone oil, have made it necessary to develop special methods. As a result, methods to recognize the disruptive discharge have changed, as have methods to determine switchoff characteristics and to generate test voltages.

The dielectric breakdown voltage is a variable with a statistical distribution. The test procedure involves a number of single test sequences. To get reliable results, it is of paramount importance to have a minimum of influence from one sequence to the next. This depends on breakdown energy and therefore on breakdown recognition, maximum short-circuit current and also on the switch-off characteristic of the testing device. The development of new, sensitive testing devices enable the energy which is sent into a test sample to be kept at a very low level. This permits a clear differentiation between partial discharge and disruptive discharge.

Investigations in breakdown characteristics and switch-off methods of devices with different techniques across several gaseous and liquid samples with special consideration of the comparability of testing results have been made to improve testing.

This article describes new ways of breakdown recognition and switch-off techniques, discusses advantages and disadvantages of the new and better-known methods and discusses the problems of standardization.

Keywords: disruptive discharge, partial discharge, insulating liquids, insulating oil, testing devices, optical sensors

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Introduction

The optimization of the workload of electrical equipment in the high voltage range, which has been encouraged more and more during the past few years for reasons of profitability, requires a precise knowledge of permissible loadability, in order to guarantee the operational safety and to avoid excessive follow-up costs. Consequently, the testing of insulating materials shows a clear trend to on-site assessment.

Increasingly, complex monitoring systems are installed on high-voltage power transformers to measure the most important operating parameter on-line and to quickly provide a survey of the available load reserves and anomalies in the device.

In accordance with this development, dielectric breakdown testing devices have made their evolution away from network-operated laboratory equipment and towards compact portable and battery-powered instruments for on-site and in-situ testing. In these cases, not only the method of the voltage generation has changed, but also the turn-off characteristic following a breakdown has been improved.

A fast breakdown recognition method and switch-off after a breakdown play an important role in the determination procedure of the breakdown voltage mean value on the basis of several measuring sequences in which the result of following sequences depend on the breakdown energy of the previous sequences. It is also of special importance in the monitoring systems. Here the decomposition products of every breakdown test remain in the insulation liquid circuit. Dielectric test instruments that maintain the short-circuit current during a breakdown for a unnecessarily long time lead to an increase of hydrogen gas production as well as hydrocarbon gases. This hazard can be overcome using modern devices with fast, secure breakdown recognition and switching-off.

Although it is not possible in monitoring systems to observe each breakdown event the way an expert would in laboratory research, the same reliability of results is required. Therefore, new possibilities of redundant breakdown recognition principles are examined to compensate for this disadvantage.

Breakdown Recognition

The breakdown recognition can be based on different principles all of which must be capable of distinguishing real breakdowns. Therefore on the one hand these mechanisms must be immune against interference sources, such as partial discharges or electromagnetic interferences from the environment and on the other hand, they must guarantee fast and sensitive extinguishing of real breakdowns.

Fast recognition and switching-off of a disruptive discharge is fundamentally important but it is of special interest in the case of standardized tests that provide for no stirring. After an occurred breakdown, stirring serves for distribution of produced gases and other degradation byproducts within the insulating liquid. If these breakdown products remain between the electrodes, they have negative effect on following measuring sequences [3][7]. Therefore, at tests without stirring the influence of one breakdown on the results of the following breakdowns is stronger.

The following parameters can be used as indicators for breakdown recognition in insulating liquids:

- test voltage
- test current
- light emission of the arc
- breakdown sound

Voltage Collapse as Switch-off Criterion

The definition of a disruptive discharge via voltage collapse (according to IEEE 4-1995) leads to difficulties, when used as a switch off criterion.

Since the alternating test voltage is periodically zero at crossover points, a system that responds purely to falling below a voltage level is unsuitable. Consequently, at least the time which is required in order to distinguish a breakdown from a periodic zero crossover must be used as a second criterion. However, this means that switching off of the testing device can be executed earliest after a time in the range of a half period. Over this time, the arc and the short-circuit current of the testing transformer is maintained. This means, that considerable values of energy are converted in the insulating material specimen.

Alternatively, the test voltage can be measured continuously in its process and it can be compared with the reference value. However, such a detection system is sensitive to influences of partial discharges, harmonics and frequency variances, which would lead to deviations of the characteristic from the sine form. In this case, a reliable breakdown recognition is only practicable by application of complex, intelligent and therefore expensive evaluation systems.

Certainly a simpler possibility to recognize a disruptive discharge, is the assessment of the test voltage gradient dV/dt, which can be used as a switch off criterion. This is distinctive in the breakdown case and develops a maximum which can be registered easily.

However, the testing current correlates well with the test voltage gradient in the first moment of the breakdown, so the problem can be reduced to a current measurement.

Test Current Rise as Switch-off Criterion

The current rise in the test circuit as breakdown recognition criterion has proven itself over many years. The switch-off techniques have been refined. Compact, portable dielectric test instruments today are constructed on the basis of resonance transformers and the switch-off according to the breakdown recognition is executed so fast that the minimum energy determined physically is achieved. This means that only the capacitive energy, stored in the insulating material between the electrodes is converted in a disruptive discharge.

Real Breakdown Monitoring (RBM) - A clear distinction between partial discharge and disturbance on the one hand and disruptive discharge on the other hand must be guaranteed by choice of the trigger-level of the breakdown recognition and selective time criteria. An increased testing current caused by low direct current resistivity of the insulating material must not be recognized as a disruptive discharge. This must be indicated by a fault report.

This means that a recognition system may only respond to events that occur suddenly and not to continuous changes in the continuously increasing current with the test voltage. The capacitive test currents of an insulating material specimen (relative permittivity $\varepsilon_r < 3$) are in the range of < 500µA. Therefore, a sudden rise of the test current values of several milliamps is a clear indication of a disruptive discharge.

However, for the distinction between partial and disruptive discharges it is necessary to introduce a supplementary time criterion during which the current through the insulating material specimen must have exceeded the preselected limiting value in order to exclude a false release by the switch-off device.

By definition, a partial discharge is an event that does not result in a complete voltage collapse. Additionally partial discharges in liquid insulating materials are of very short duration (<1µs). Therefore, the two distinction criteria which are available are both necessary for a clear separation of partial and disruptive discharge.

Therefore, if one uses the simultaneous exceeding of a minimum time interval (e.g.>10µs) and a minimum current of (e.g.> 3mA) as a criterion for breakdown recognition, a good distinction between partial and disruptive discharge is possible since only a breakdown fulfills both criteria (i_{min}, t_{min}).

In practice, this method has proven itself over many years. If the limits (i_{min}, t_{min}) are well chosen, they deliver unambiguous results for the wide band of application cases of insulating material testing (Fig. 1).

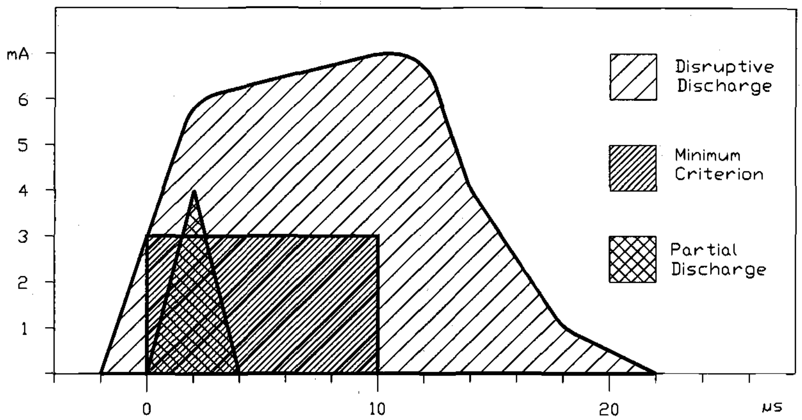


Figure 1.: Recognition criterion for current dependent switching off

Light Emission of the Arc as Switch-off Criterion (Optical Breakdown Recognition)

The emission of electro-magnetic radiation in the visible range originating in the arc at a breakdown represents a possible criterion for the breakdown recognition. In this case a great advantage is to be seen in the reaction rate (<250 nsec) and the galvanic separation of the measurement from the test circuit.

The light emission is an effect which occurs at every breakdown but is not unique to a disruptive discharge since partial discharges also have lighting appearances.

Investigations at the institute for High-Voltage Engineering of the Technical University of Graz (Austria) dealt with the problem to recognize disruptive discharges in insulating materials with the aid of an optical sensor and to distinguish them reliably from partial discharges and disturbances.

Parameters of the studies were different qualities of the insulating material (mineral oil) with different aging, turbidity and moisture and different electrode arrangements.

The chosen optical sensor consisted of a connected phototransistor (T2) with comparator unit, which generated a Transistor Transistor Logic-signal.

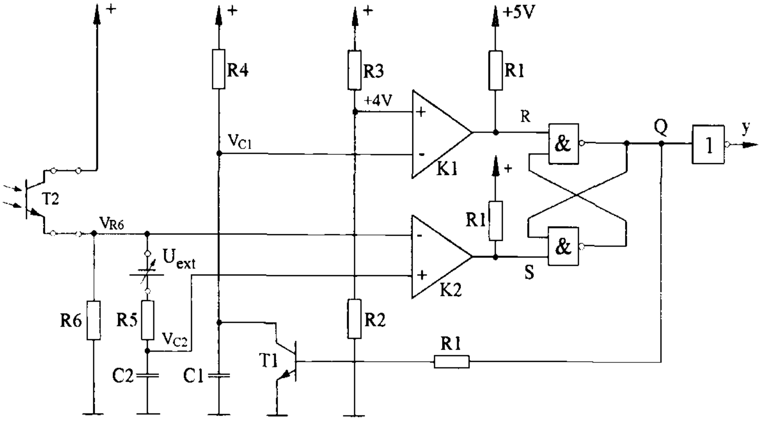


Figure 2.: Equivalent circuit diagram of the optical breakdown recognition [6].

First studies showed that the following factors had an influence on the measurement:

- **Electro-magnetic interference of the optical sensor**

  In the course of the studies it turned out that a complete shielding of the sensor circuit against electro-magnetic interferences was necessary.

- **External optical influences**

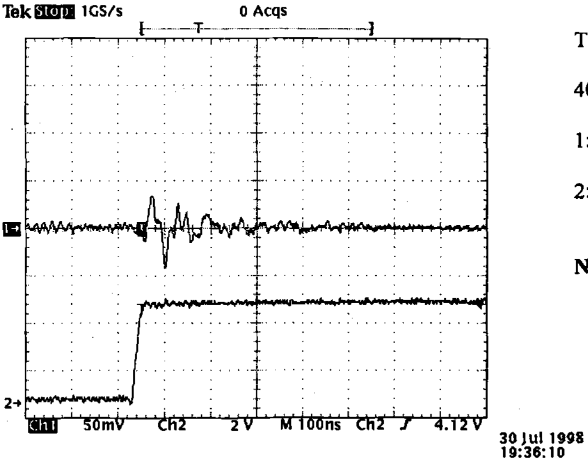
  The optical shielding proved equally to be necessary against outer light interference since strong light sources led to malfunctions of the sensor.

- **Constant light source**

  On the other hand, a moderate constant light showed advantages for a reliable response of the sensor to discharge processes, because it brought the sensor into a favorable operating range. This was a precondition for the recognition of low energy disruptive discharges in gas at low test voltages.

The sensitivity could be changed by selection of the emitter resistance of the phototransistor and of a supplementary external voltage source:

Influence of partial discharges -



Time range 100ns/DIV

40kV withstand voltage

1: current characteristic

2: signal output of the
optical breakdown recognition

No disruptive discharge!

Figure 3.: Response of the optical breakdown recognition at partial discharges and to high sensitivity [6].

Figure 3 shows the PD-influenced test current characteristic. These partial discharges triggered the optical breakdown recognition at to high sensitivity.
As expected, distinguishing the light emission of partial discharges from those from disruptive discharges represented the most difficult problem to solve.
Especially under consideration of the oil turbidity and the value of the breakdown voltage, extreme conditions could be created, under which distinction difficulties were to be expected:

A) Low energy disruptive discharge in insulation oil with high turbidity

B) High energy partial discharge in new clear insulating oil

During the studies it turned out, that by favorable selection of the emitter resistance (R6) and the external voltage V_{ext} (at the comparators reference input) a broad-scale insensitivity to high-energy partial discharges could be achieved. On the other hand with the exception of the electrode form according to ASTM D877, all disruptive discharges >10 kV could be recognized optically even for oil of high turbidity.

In tests with the disc-electrodes according to ASTM D877 the breakdown does not preferably occur in the center, that is in the electrodes axis, unlike all other electrode forms, but rather occurs in the area of the edge.

In the most unfavorable case, when the breakdown appeared on the turned away edges of the electrodes, there was too little light emission for recognition. This was due to the increased turbidity of the insulating oil. Therefore, during the tests, breakdowns up to a maximum of 15kV occurred that were not registered by the optical breakdown-recognition. The sensitivity for those events was too low.

Dynamic Sensitivity Adjustment as a Function of the Test Voltage Level- In order to achieve a clear separation between interference sources and disruptive discharges of low energy in the case of high turbidity one can make use of the voltage dependence for occurrence of different types of discharge activity:

Therefore, it is useful to change the sensitivity of the optical breakdown recognition dynamically with test voltage.

- **Low**-energy breakdow at **low** test **voltage** → **high** recognition sensitivity
- **High**-energy partial discharge at **high** test **voltage** → **low** recognition sensitivity

In this way, the interruption circuit can be desensitized to high-energy partial discharges, while the sensitivity for low-energy breakdowns can be increased.

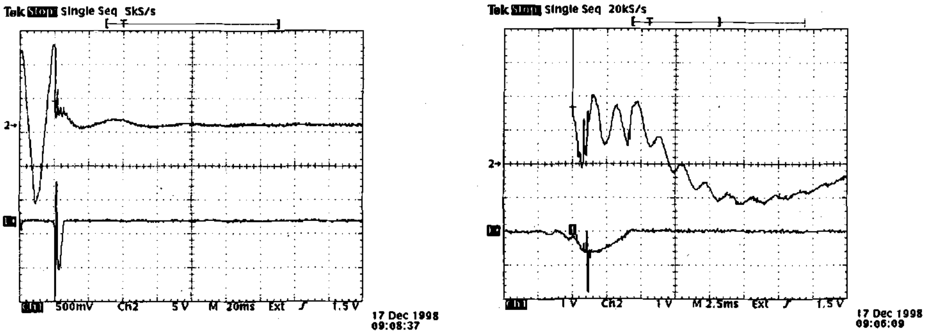
An optimized breakdown recognition system like this is quickly reacting and fulfills the requirement to exclusively react to disruptive discharges for all IEC and ASTM test standards (including ASTM D 877).

Switch-off Techniques

Dielectric Test Instrument with Network Powered HV-Transformer and Relay Off-Switching

The network powered dielectric test instruments with relay off-switching, which were manufactored in the past, have a switch-off characteristic, which maintains the short-circuit current over some milliseconds in the case of a breakdown.
The consequence of this switch off technique is a relatively high energy conversion within the specimen, which can be in the range of **several hundred mJ**. In this case, the short-circuit current is limited exclusively by the short-circuit impedance of the testing transformer. If the spark gap between the electrodes improves its dielectric strength, also multiple ignitions can occur.

Characteristic of Current and Voltage during the Disruptive Discharge -



Time range 20 ms/DIV

1: Current characteristic
500mV are 50mA

2: Voltage characteristic
5V are 50kV

RMS-value of the
breakdown voltage 79kV

Time range 2.5 ms/DIV

1: Current characteristic
1V is 100mA

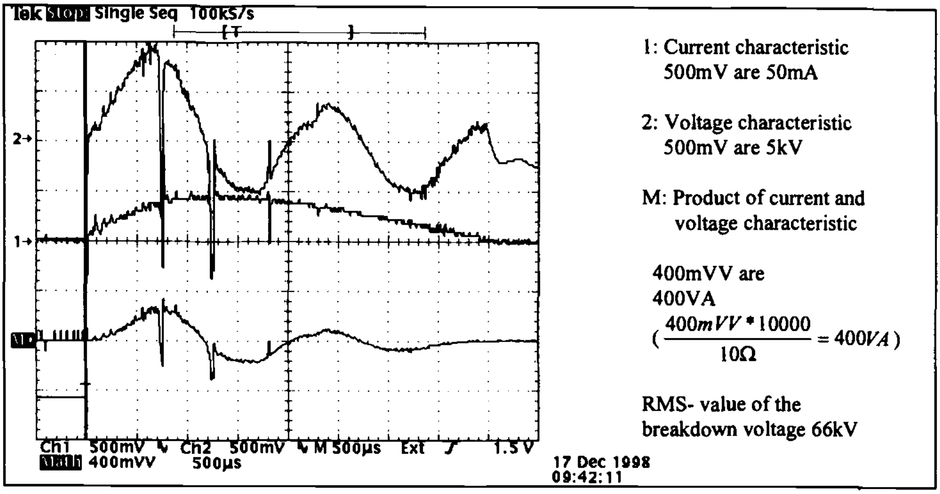
2: Voltage characteristic
1V is 10kV

RMS-value of the
breakdown voltage 78kV

Figure 4.: Characteristic of voltage (2) and current (1) during a disruptive discharge at a dielectric test instrument with network powered high-voltage transformer and switching off by relay (to the right a repeated record zoomed into) [6].

As one can see from Figure 4., the short circuit current remains for several msec, which results in remarkable energy conversion.

Energy Conversion during the Disruptive Discharge -



1: Current characteristic
500mV are 50mA

2: Voltage characteristic
500mV are 5kV

M: Product of current and
voltage characteristic

400mVV are
400VA

$$\left(\frac{400mVV*10000}{10\Omega}=400VA\right)$$

RMS- value of the
breakdown voltage 66kV

Figure 5.: Characteristic of current, voltage and power during a disruptive discharge at a dielectric test instrument with network powered high-voltage transformer and switching off by relay [6].

If one integrates the characteristic of power, one receives the energy converted in the specimen. The example above in Figure 5. results in a value in the range of 100 mJ. However, energies of 500 mJ and more can also be achieved. These energies stress an insulating material specimen. Extended hydrogen concentration and formation of simple molecular hydrocarbon connections is the result. This affects considerably subsequent breakdown tests particularly if it is not stirred during the sequences.

Dielectric Breakdown Test Instrument with Software Generated Test Voltage and Resonant Transformer

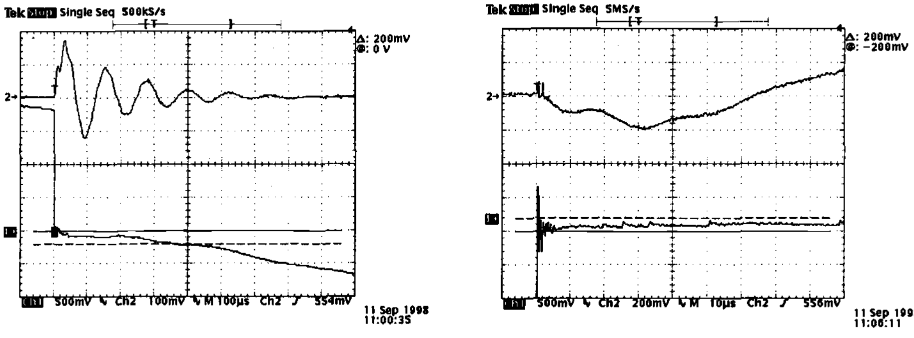
Unlike laboratory tests, on site testing needs network-independent, portable dielectric test instruments. For weight reasons, it is useful to implement the test voltage generation on the basis of resonance transformers with supply via amplifier and batteries.

In the normal testing operation the test circuit is compensated, that means the capacitive current via the natural capacitance of the high voltage winding and the capacity of the insulating specimen becomes compensated by an inductive current via the main inductance of the transformer. The main inductance is adapted by an air gap in the iron core of the transformer.

For the operation of such a resonance transformer it is sufficient to implement a relatively compact, lightweight power unit since only the losses of the test circuit must be supplied, but not the reactive power.

In the power unit, the sinusoidal primary voltage is generated and reinforced artificially from a d.c. voltage. If the breakdown recognition has responded, the electronics immediately stops the power supply to the testing transformer. As a result, switched-off times of clearly less than 100 μsec are achieved. Compared to relay disconnection (approx. 5 msec) this is an improvement of clearly more than factor 50.

Characteristic of Current and Voltage during the Disruptive Discharge -



Time range 100μs/DIV

1: Voltage characteristic u_{El}
200mV are 2.8kV

2: Current characteristic i_L

RMS-value of the
breakdown voltage 20.8kV

Time range 10μs/DIV

1: Voltage characteristic u_{El}
200mV are 2.8kV

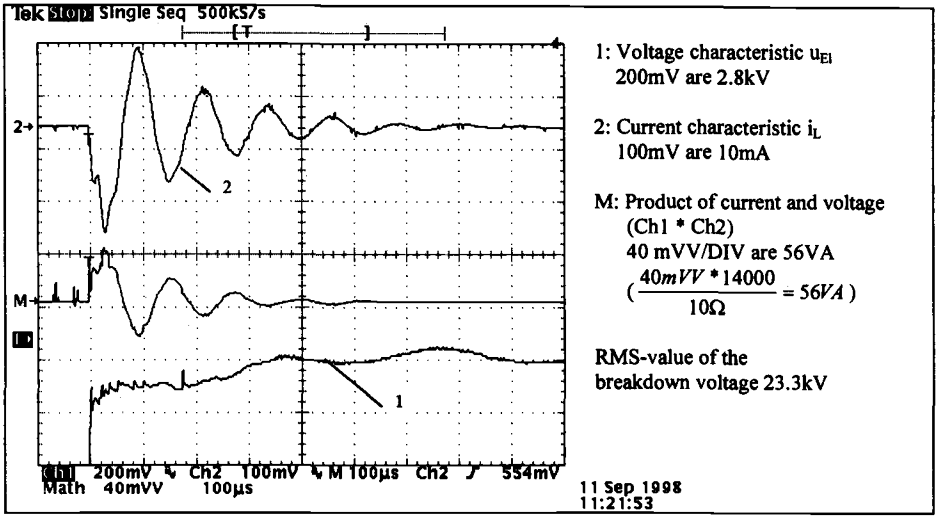
2: Current characteristic i_L

RMS-value of the
breakdown voltage 22.3kV

Figure 6.: Characteristic of current (2) and voltage (1) during a disruptive discharge at a dielectric test instrument with resonant high-voltage transformer and electronic switching off, electrodes according to ASTM D877 [6].
(to the right a repeated record, inverted and zoomed into)

As Figure 6. shows, the time to switch off is in the range of 20μsec, which results in very low breakdown energy conversion. In this case, the breakdown recognition was current-triggered, but optical triggering would have had the same result.

Energy Conversion during the Disruptive Discharge -



1: Voltage characteristic u_{El}
200mV are 2.8kV

2: Current characteristic i_L
100mV are 10mA

M: Product of current and voltage
(Ch1 * Ch2)
40 mVV/DIV are 56VA
$(\frac{40mVV * 14000}{10\Omega} = 56VA)$

RMS-value of the
breakdown voltage 23.3kV

Figure 7.: Characteristic of current, voltage and power during a disruptive discharge at a dielectric test instrument with resonant high-voltage transformer and electronic switching off (electrodes according to ASTM D877) [6].

The integration of the power characteristic only results in a breakdown energy value in the range of 2.5 mJ. If this is compared to the capacitive energy content of electrode arrangement with mineral insulating oil at the actual voltage value in the moment of the breakdown, an energy value in the same range is the result.

$$\mathbf{W_c = 0.5*C*V^2 \quad = \quad 0.5 * 10\ pF * (29\ kV)^2 = 4.2\ mJ}$$

W_c = converted breakdown energy
C = capacitance of the test arrangement
V = actual test voltage in the moment of the breakdown

This means that with modern, fast switch-off techniques the energy of an insulating material breakdown can be limited to the range of its capacitive intrinsic energy.
Therefore, the physically possible limit is achieved. As investigations at ETG Elin Transformers in Weiz, Austria proved that these energy levels have no essential influence on the hydrogen concentration in situ monitoring systems in high voltage power transformers. By conversion of the test results a hydrogen rise of less than 1ppm/year was calculated [5].

Conclusions

The design of the test circuit as a resonant circuit and the design of modern switching off techniques with dynamic optical or current-dependent releasing now makes it possible to reduce the converted energy during the breakdown test and therefore the decomposition effect in the insulating material specimen to a minimum. In this way, multiple breakdown tests within a testing procedure have a minimum influence on each other.
This is especially important during standardized tests that do not provide for stirring.

The optical breakdown-recognition method offers an alternative to the physical observation of the breakdown. A clear distinction between partial and disruptive discharges is achieved by dynamic adjustment of the sensitivity of optical sensor.
The optical breakdown-recognition method can be used with in-situ monitoring systems additionally to the well proven current criterion. Based on redundant remote sensing with different recognition principles, supplementary sureness of the assessment is created for the person responsible for operation and planning.

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Standardized Testing Procedures and Developments in Partial Discharge Measurement

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Abstract: A comparison of the various standardized methods of partial discharge detection and measurement on electrical components, apparatus and cables indicates that, for the most part, the methods are more concerned with the detection of the presence of partial discharges rather than the determination of arbitrary discharge energy thresholds that may induce degradation and eventual breakdown of electrical insulating systems. Particular attention is given to IEEE and IEC partial discharge measurement standards and their relation and similarity to the ASTM standard. The ASTM test method is shown to constitute the basis for most other standards and existing measurement techniques, with the exception of those on rotating machines and compressed gas cables where higher frequency methods are utilized. A discussion is presented on certain advanced topics, dealing with partial discharge pulse-height and discharge epoch distribution analysis and the associated digital measurement techniques, none of which have yet attained any standardization status. In addition, a number of partial discharge site location techniques, which may have some potential for standardization, are described.

Keywords: Partial discharge detection, measurement, apparent charge transfer, standard measurement procedures, partial discharge site location, pulse-height analysis, discharge pattern recognition

Introduction

The occurrence of partial discharges in cavities, occluded within the insulating systems of electrical apparatus and cables, was already well recognized in the early 1900's [*1*]. Although the initial observations of partial discharges were made either visually or aurally, their electrical detection was first effected in the 1920's by means of the Schering bridge in terms of the dissipation factor (tanδ) increment with applied voltage [*2, 3*]. Following shortly the invention of the cathode ray tube in the 1920's, it became possible using oscilloscopes in the 1930's to record the form of the detected partial discharge pulses [*4-6*]. The 1940's saw the extension of partial discharge measurements on actual electrical equipment, such as transformers [*7*] and cables [*8*]; in

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these applications the partial discharge detection circuits were of the RLC-type, with which the recorded discrete partial discharge pulses assumed the form of damped oscillating pulses. The stretched pulses, because of their relatively long decay time, were easy to discern on the relatively long 60 Hz time base normally preset on the oscilloscope screen; the use of an inductive detection impedance in the RLC circuits also provided higher sensitivity than that obtainable with RC-type partial detection circuits [*6*], which are more suitable for research orientated work where a more faithful reproduction of the actual form of the discharge pulses is required. Nevertheless, RC-type circuits were and are still commonly employed industrial practice for radio noise (radio influence voltage) measurements to determine the effect of discharges upon radio reception. During the early period of partial discharge investigation, data relating the occurrence of partial discharge to the physical and chemical degradation and the resulting reduction in the life of insulating systems continued to accumulate, thereby demonstrating its long term detrimental effects [*9, 10*]. It thus became abundantly clear that exposure of electrical insulation to partial discharges must be avoided and that partial discharge tests must be incorporated as an integral part of the electrical test protocol necessary to assess the quality of electrical insulating systems.

In the 1950's, cable and transformer manufacturers were already performing routine partial discharge tests on their products. Tests on transformers were carried out, using RC-type circuits to determine the radio influence voltage along the lines described in the early NEMA Standard (NEMA Publication 107, Methods of Measurement for Radio Influence Voltage of High Voltage Apparatus, 1964; also see IEEE C57.12.90-1987, IEEE Standard Test Code for Liquid Immersed Distribution, Power and Regulating Transformers and Guide for Short-Circuit Testing of Distribution and Power Transformers). However, the quality of cables was assessed in terms of the apparent charge transfer associated with the detected discharge pulses by means of RLC-type detection circuits. The immediate necessity of standardized partial discharge test procedures in conformance with a specified sensitivity led to considerable activity in ASTM Committee D.09 [*11*], which culminated in 1961 with the development of a partial discharge measurement standard (ASTM D1868 Standard Method for Detection and Measurement of Partial Discharge Pulses in Evaluation of Insulation Systems). Wide interest in partial discharge measurement standardization was subsequently reflected in the appearance of a similar and almost identical IEC Standard (IEC Publ. 270, Partial Discharge Measurement, First Edition) in 1968 [*12*]. In the present paper we shall examine the evolution of partial discharge (PD) measurement related standards and discuss the various reasons why certain PD quantities and measurement techniques can be standardized vis-à-vis others, that cannot, as concerns different types of specimens such as transformers, rotating machines, cables and capacitors.

Preliminary Considerations

Although partial discharge measurements on high-voltage electrical apparatus have now been performed over many decades, there has been little success achieved in attempts to relate the measured discharge pulse charge transfers, expressed normally in pC, to the deterioration rate of electrical insulating materials subjected to these discharge magnitudes. Much of this difficulty has been inextricably associated with the observed variations of the discharge pulse magnitudes themselves as a function of time. That is, as the recorded discharge pulse magnitudes may either increase or diminish with time in a quasi random manner, the discharge pulse magnitudes observed at the time of breakdown of the insulating material or system will in general bear little or no definitive relationship to the magnitude of the discharge pulses measured incipiently. Hence for similar reasons, as the insulating systems are not aged under a time invariant discharge pulse intensity (a constant pulse amplitude and discharge rate or energy loss), evanescent relationships between the initially recorded partial discharge energy loss and the time-to-failure are also found to be, in most cases, of little practical consequence. Accordingly, the common approach to practical discharge evaluation procedures on insulating materials having low resistance to the degradative effects of partial discharge (*e.g.* polymeric power transmission and distribution cables) is to insist on the complete absence of partial discharge in the insulating system at the operating voltage levels [*13*]; this point of view is borne out in the AEIC specifications on polymeric cables, namely: AEIC Specification CS5, Specifications for Thermoplastic and Cross-Linked Polyethylene Insulated Shielded Power Cables Rated 5 Through 46 kV, AEIC Specification CS6, Specifications for Ethylene Propylene Rubber Insulated Shielded Power Cables Rated 5 Through 69 kV and AEIC Specification CS7, Specifications for Cross-Linked Polyethylene Insulated Shielded Power Cables Rated 69 Through 138 kV. Evidently, in specifying complete absence of discharges at given applied or operating voltage levels, requires the establishment of the charge transfer levels associated with the minimum detectible discharge pulse (*e.g.* pulses having associated apparent charge transfers of ≤ 3 to 5 pC). With dielectric liquid filled insulating system structures, such as dielectric liquid filled transformers which tend to tolerate discharges better that solid plastic insulation, apparent charge threshold levels up to 500 pC may be deemed to be acceptable (c.f. Revised IEEE Standard 62, Guide for Diagnostic Field Testing of Power Transformers). Once such permissible *ad arbitrium* discharge levels are established, the partial discharge tests become simply go-no go type testing procedures whereby the specimens are tested to determine the inception and subsequent partial discharge extinction voltages – *i.e.* to obtain voltage levels at which the insulation is free of discharge pulses having charge transfers less than say 5 pC, depending upon the specimen under test. It is interesting to note that current IEEE guides (IEEE C57, 113-1991, IEEE Guide for Partial Discharge Measurement in Liquid-Filled Power Transformers and Reactors, and IEEE Standard C57.124-19991, IEEE Recommended Practice for the Detection of Partial Discharge and Measurement of Apparent Charge in Dry-Type Transformers), concerned with partial discharge tests for transformers, do not explicitly specify a maximum acceptable PD level, though an acceptable background noise level of less than 100 pC is specified, thereby inferring tacit acceptance of a discharge level above 100 pC.

As partial discharge acceptance tests on insulating components, electrical apparatus and cable specimens require a discharge-free condition at some arbitrarily specified sensitivity level in pC, the partial discharge procedure essentially consists of a measurement of the partial discharge inception and extinction voltages (PDIV and PDEV). Since the accuracy with which these values are determined is contingent upon the accuracy of the charge transfer determination in pC associated with the maximum detected discharge pulse amplitude, routine PD measurement necessitates calibration of the scale of the measuring instrument in pC. For this purpose either analog or digital circuitry may be employed. This is in contrast with other PD tests that may involve more analytical measurements such as discharge rate, discharge pulse-height, phase and time interval distributions, where utilization of digital circuits becomes essential. Common use of digital circuits is also made in partial discharge site location measurements. Computer software is frequently employed in conjunction with the foregoing tests, because it provides added latitude to the display format of the results and permits statistical treatment of the test data.

Standardized Routine Partial Discharge Test Procedures

ASTM Standard D1868 and its subsequent close relative, IEC Publ. 270, form the basis for all routine standard partial discharge tests. It is only for the tests on compressed SF_6 gas cables and rotating machines that the test protocol is different, since it involves the use of ultra wideband detectors for operation at ≥ 300 MHz. In contradistinction polymeric cables, transformers and capacitors are evaluated at much lower frequency bandwidths (≤ 250 kHz), using the well established apparent charge transfer and PDIV and PDEV measurement procedures.

Solid Dielectric Extruded Cables

Standard routine partial discharge tests on solid dielectric extruded power cables are normally carried out using RLC circuits of the type depicted in Figure 1. The cable specimen represented by a lumped capacitance, C_p, may be connected in series with an RLC detection impedance Z_d shunted by a discharge-free coupling capacitance, C_c, usually in series with another discharge-free capacitance C_M required to form a capacitive voltage divider for the voltammeter V as indicated in Figure 1(a); however, more commonly, the cable specimen is stressed directly between the high voltage terminal and ground, shunting the C_c and C_M combination placed in series with the detection impedance Z_d as shown in Figure 1(b). The latter is the more popular configuration, because it always results in a signal to noise ratio of unity and permits the specimen to be directly grounded. The *rf* choke is employed to prevent interference signals originating in the transformer windings from entering the detection circuit. It is well to emphasize here that the two circuits delineated in Fig. 1(a) and (b) are virtually identical to those described in ASTM D1868, except that in the latter the specimen is shown as a capacitive element and refers in general to any insulating system under test. In that sense ASTM D1868 is perfectly general in its application, since the insulation system may be that of a capacitor, cable, transformer, bushing or machine bar specimen for which the apparent charge, PDIV and PDEV must be determined.

The center of the frequency band of commercially available partial discharge detectors is made to coincide with the resonant frequency, f_o, of the RLC circuit, which is given by

$$f_o = \frac{1}{2\pi}\left[L\left\{\frac{C_c C_p}{C_c + C_p}\right\} + LC_d\right]^{1/2} \qquad (1)$$

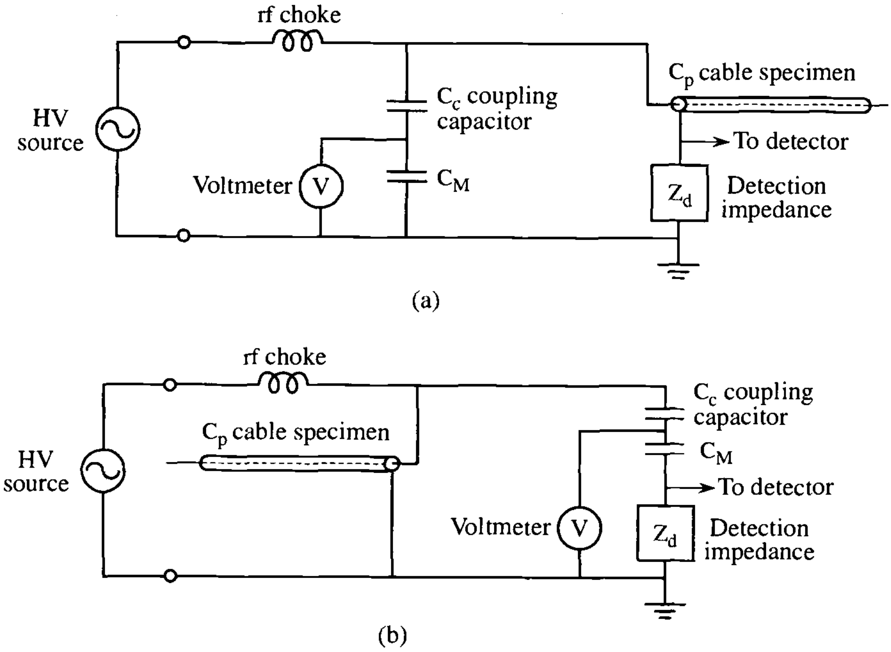


Figure 1 – *Two most common RLC-type partial discharge pulse detection circuits: (a) with specimen grounded via the detection impedance, Z_d and (b) with specimen directly grounded*

where L is the inductance and C_d the effective capacitance of the detection impedance Z_d. With the exception of very high capacitance test specimens, such as capacitors with high capacitance, the resonant frequency is determined by the values of L and C_c of the detection circuit itself, since ordinarily $C_c >> C_p >> C_d$. For most commercial PD detectors, the value of f_o is designed to fall normally within the range of 30 to 90 kHz. Since the frequency spectrum of partial discharge pulses may extend to approximately 1 GHz, partial discharges can be detected at any frequency within this range. However, partial discharge detection on polymeric cables, which are extremely susceptible to partial discharge induced degradation effects and in which the high frequency components in the PD pulses are rapidly attenuated by the lossy semiconducting conductor and insulation shields as they propagate along the cable, PD detection is carried out at low frequencies.

The principal advantage of low frequency PD detectors is that, as a consequence of their charge integrating nature, they respond to the apparent charge transfer, ΔQ, associated with the discrete discharge pulses. It has been demonstrated that with distributed parameter cable specimens, the signal response of low frequency RLC-type PD detectors consists of a low and high frequency component; the former, which contains explicitly the apparent charge transfer term, ΔQ, is given by *[14]*

$$\Delta V_d = 4\pi f_o \left\{ \frac{C_c}{C_c + C_d} \right\} \frac{Z_o \Delta Q}{2} \exp\left[-(R/2L)t\right] \sin(2\pi f_o t) \qquad (2)$$

where Z_o is the characteristic impedance of the cable specimen under test and R is the equivalent resistance of the detection impedance, Z_d. Cables in excess of 100 feet exhibit a definite transmission line behavior, causing signal reflections to occur from the far unterminated end of the cable. These can be eliminated by the use of termination at the far end, consisting of a purely resistive characteristic impedance element in series with a discharge-free capacitor equal approximately in value to C_c. However, in the routine testing of cable specimens, it is more expedient to leave the specimens unterminated. In order to minimize the effects of pulse superposition errors, arising either with reflected pulses from the far end of the cable specimen or between propinquitous pulse signals in dense discharge patterns, the exponentially decaying oscillatory discharge signals are damped to reduce their length and pulse shaping amplifiers of adequate bandwidth are employed to ensure that the first peak of the signal oscillation exceeds in amplitude the subsequent oscillatory peaks. This form of discharge pulse is commonly referred to as the α-response in contrast to the so-called β-response type pulse, whose first peak amplitude due to narrow bandwidth constraints is less than that of one or more of the successive oscillations [*15*]. Hence, superposition of α-response type pulses due to pulse integration or overlap results in added amplitude errors leading to a larger detected pulse amplitude than the true amplitude of the discharge pulse, whereas superposition of β-response pulses yields measured amplitudes that are smaller than the actual discharge pulse amplitudes. Since routine discharge tests on cables are go-no go type tests whose purpose is only to determine at what value of applied voltage certain level discharges ($\geq$ 5 pC) initially occur and at what voltage they become extinguished, an α-response inherently augments the sensitivity of the partial discharge detection circuit.

Calibration of RLC-type circuits utilized for partial discharge pulse detection in cables is usually carried out by injecting an excitation square pulse at the far end of the cable as delineated in Figure 2. The injected calibration charge, ΔQ, is given by

$$\Delta Q = \Delta V . C_s \qquad (3)$$

where ΔV represents the amplitude of the injected square pulse; C_s is a capacitor in series with the square pulse generator, whose value may extend from 50 to 150 pF. The width of the excitation pulse must be sufficiently wide to prevent overlap of the exponentially decaying pulses, simulated at the front and the trailing edge of the square pulse. The rise and decay time of the square excitation pulse is specified to be 100 ns as in the case of ASTM D1868. Some commercial PD detectors with internal calibration sources often employ an excitation pulse with a steep rise front, having a gradual exponential decay at the trailing edge of the pulse. In such circumstances a response pulse is only simulated at the abrupt step-like voltage change at the front of the pulse. A criterion frequently used to specify the rise time, t_r, of the excitation square pulse is [*16*]

$$t_r \leq 1/25\, f_o \qquad (4)$$

where f_0 is the resonant frequency of the RLC-type detection circuit. Equation (4) imposes an upper limit to the rise time of the excitation pulse, because the response of partial discharge detectors is a monotonically decreasing function of the measured pulse rise time. Thus discharge pulses with unduly long rise times (*e.g.* long rise time pseudo-glow discharges and pulseless glow discharges) cannot be detected by standard pulse discharge detectors. These type of discharges may occur in physically real cavity occlusions, depending upon the conditions existing within the cavity such as gas pressure, cavity diameter and wall conductivity.

Capacitor and Bushing Specimens

Partial discharge tests on capacitor specimens are in some respects the least complicated as they involve test objects that behave as simple lumped capacitance circuit elements. In this regard a very short length of a cable specimen and a bushing specimen may also be considered as lumped capacitance specimens. When a capacitor specimen is substituted for the cable specimen portrayed in Figure 2, the detected discharge voltage pulse amplitude due to a discharge in the capacitor specimen assumes the form

$$\Delta V_d = \frac{\Delta Q}{C} \exp\left[-(R/2L)t\right]\cos(2\pi\ f_o t) \tag{5}$$

where C denotes the total capacitance within the resonant RLC circuit and is equal to

$$C = C_p + \{C_c C_d / (C_c + C_d)\} \tag{6}$$

in which C_p represents the capacitance of the capacitor specimen undergoing test. Equation (5) indicates that the signal response of the RLC circuit with a lumped capacitance specimen assumes the form of an exponentially decaying cosine pulse as opposed to the exponential decaying sinusoidal pulse with a longer distributed parameter cable specimen as defined by Equation (2).

The major difficulties with tests on capacitor specimens arise when capacitor specimens having very large capacitance must be tested. In such circumstances partial discharge detection becomes very difficult, as is palpably apparent from the nature of Equation (7)

$$\Delta V_d = \Delta Q / C_p \tag{7}$$

which demonstrates that for a given charge transfer ΔQ, the magnitude of the detected peak pulse voltage, ΔV_d, across the detection impedance decreases with the capacitance C_p. In practice, the PD detection problems intrinsic to very large capacitance specimens may be circumvented by either testing the individual capacitor units that comprise the overall capacitor or by employing acoustical methods, using transducers placed at various positions on the surface casing of the capacitor specimen.

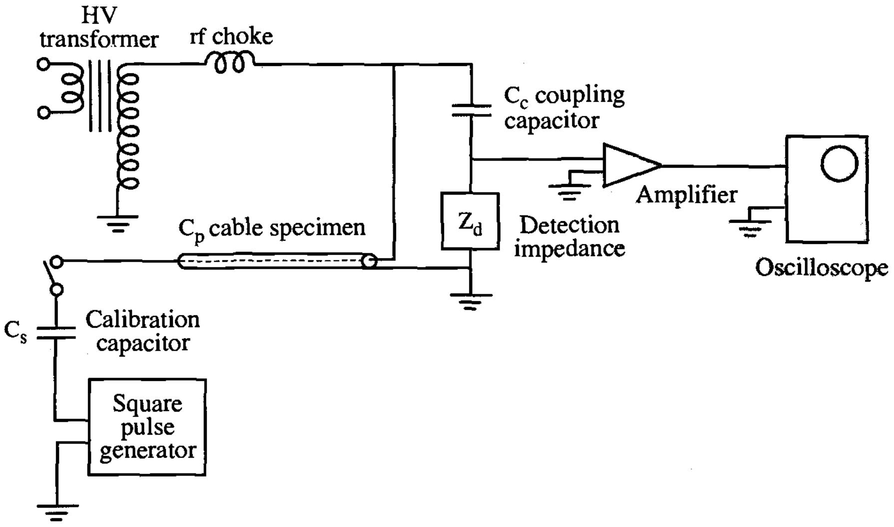


Figure 2 – *Calibration of RLC-type detection circuit with cable specimen under test*

Transformer Specimens

When testing transformer specimens for partial discharge, the voltage may be imposed either by performing what is generally referred to the induced voltage test or the externally applied voltage test. In the former instance, the voltage is simply applied across the low potential winding, whereby a voltage is developed between the individual turns and sections of the windings in the same manner as occurs under normal operation of the transformer [*17*]. Since in the induced voltage test, the transformer specimen may be subjected to voltages above the rated voltage level, the partial discharge tests are normally made with either a voltage generator source of 180 Hz (third harmonic) or 400 Hz in order to avert harmful saturation effects in the transformer's core. The induced voltage test dispenses with the necessity of the coupling or blocking capacitor, C_c, by making use of a high voltage capacitor tap located in the normally discharge-free H.V. bushing of the transformer specimen itself as portrayed in Figure 3.

In the applied voltage test, the voltage is imposed across the high voltage winding and the grounded low voltage winding and iron core of the transformer specimen as depicted in Figure 4. The externally applied high voltage test is generally used on smaller transformer specimens and is more popular on instrument transformers and dry-type transformers. A 60 Hz high voltage discharge-free testing transformer constitutes the standard voltage source in the applied voltage test; in this test a separate discharge-free coupling or blocking capacitor, C_c, is required.

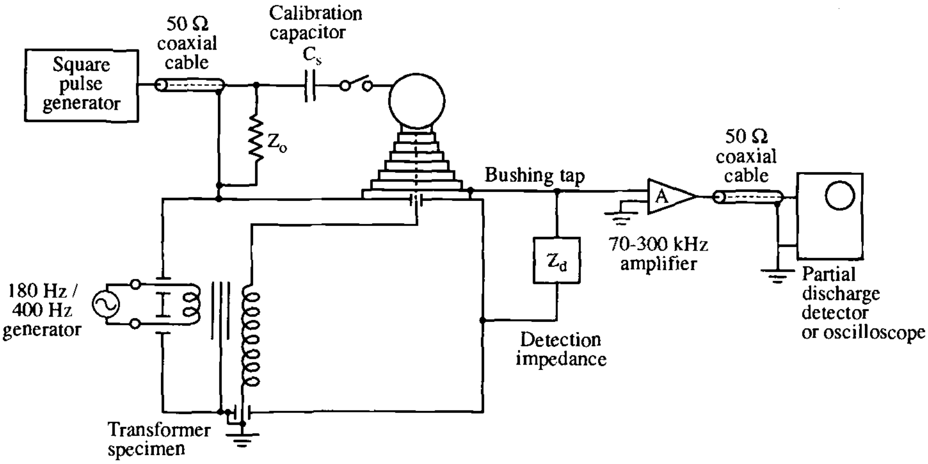


Figure 3 – *Schematic circuit arrangement for an induced voltage partial discharge test on a power transformer specimen after IEEE C57.113-1991 and [17]*

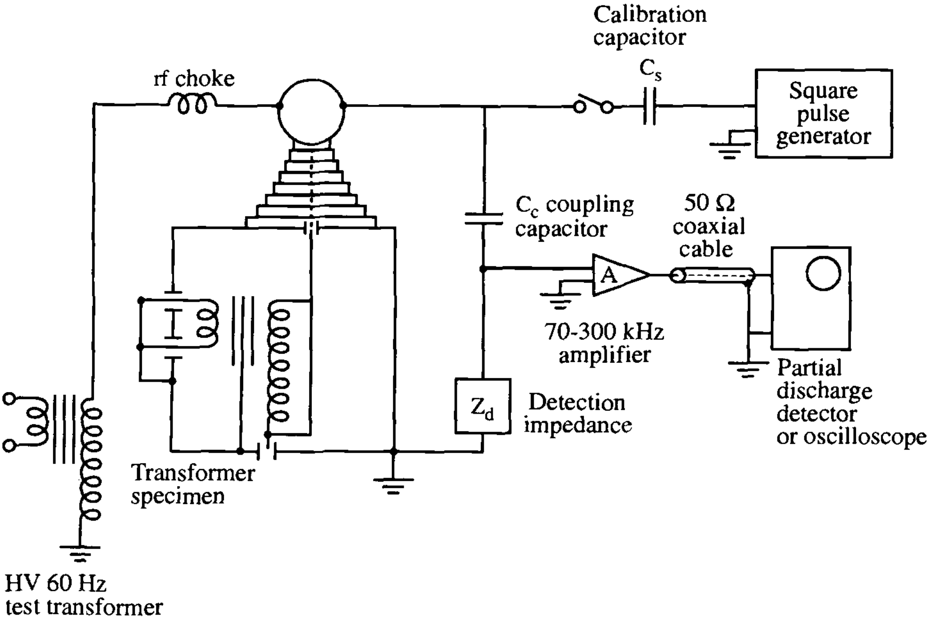


Figure 4 – *Schematic circuit arrangement for an externally applied voltage partial discharge test on a power transformer specimen after [17] and IEEE C57.113-1991 and IEEE Standard C57.124-1991.*

Rotating Machines

Although partial discharge measurements on rotating machines have now been performed for a number of decades, there are at the present no standardized methods available. However, an IEEE test guide (IEEE P1434 – Guide to Measurement of Partial Discharges in Rotating Machinery) is now under preparation, which lists a number PD test methods currently used on rotating machines. To describe each one of these methods would involve a great amount of detail, however, it is deemed appropriate to discuss here at least one of the more popular methods to illustrate some salient differences in the approach to PD measurements on rotating machines

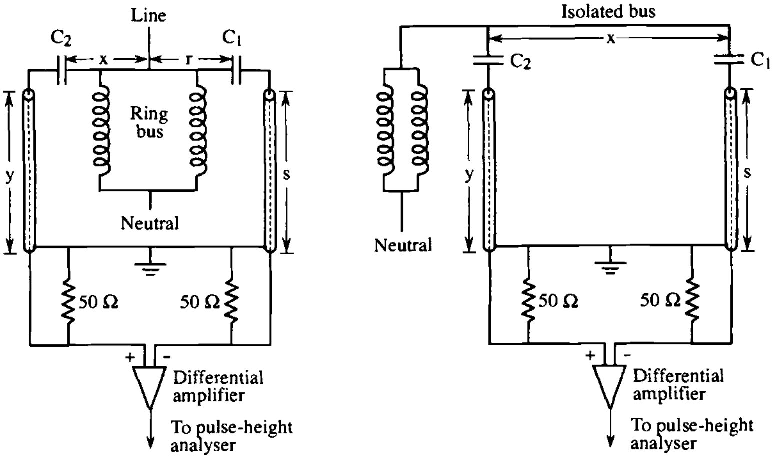
Since the complexity of the overall insulating systems of rotating machines has prevented thus far the development of a meaningful calibration procedure in terms of the apparent charge as achieved with other electrical apparatus, PD measurements on rotating machines are being carried out using the mV as the unit of PD pulse amplitude. Consequently, as the method, to be described, records the distributions of the discharge pulse heights expressed in mV, it cannot compare the discharges intensities of two different machines, though it may be effectively deployed to monitor the changes of discharge intensity with time and compare the discharge intensities of discharge patterns of machines having identical coil or winding designs.

The method utilizes coaxial signal delay lines in conjunction with a balanced capacitive coupler measurement scheme to reject common mode noise and static excitation interference [*18-20*]. Figure 5(a) shows that in the case of a water-wheel generator specimen, two identical permanent couplers (80 pF) are installed in each phase; whereas for a hydro-cooled steam generator, the two permanent couplers must be inserted on the isolated bus as depicted in Figure 5(b). In Figure 5(a), x and r denote the respective bus lengths with y and s being the corresponding coaxial cable lengths, leading to the equality

$$x+(y/0.65)=r+(s/0.65) \qquad (5)$$

where the factor 0.65 reflects the reduction in signal velocity of the solid polyethylene insulated coaxial cables -- *i.e.*, the propagation velocity in the 50 Ω coaxial cable medium is equal to 65% of the that *in vacuo*. The symmetrical permanent coupler pairs C_1 and C_2 together with the respective coaxial cable lengths present equivalent electrical lengths to the input of the differential amplifier where extraneous noise rejection is effected as the incident interference signals of opposite polarity are cancelled as a result of their equal travel times. Consequently only the actual partial discharge pulses are amplified by the differential amplifier. As these pulses emerge from the amplifier, they are sorted by means of a microprocessor into different window threshold levels of a single channel pulse-height analyzer, which yields a pulse-height distribution characteristic. The measurement technique employs a monostable multivibrator circuit similar to that described elsewhere [*21*]; its pulse resolution capability is of the order of 3 μs. The technique uses bandwidths up to 300 MHz, which decrease signal distortion, since the high frequency signals are transmitted by means of capacitive coupling along the windings. However, it is precisely the use of the very high frequency detection scheme that renders calibration in terms of the apparent charge transfer extremely difficult if not impossible.

Earliest application of digital techniques to partial discharge studies occurred with the use of analog to digital (A/D) converters in the work first reported on partial discharge pulse recurrence rate measurements and pulse-height analysis on simulated cavities and physical cavities occluded within the insulating systems of cables [*16, 22-24*]. Notwithstanding, these early encouraging results with digital circuitry, the introduction of digital techniques into standard routine measurements of partial discharges, as concerns routine go- no go type tests involving the determination of the maximum apparent charge transfer in pC and the associated partial discharge inception and extinction voltages, has been less marked and slower. For many years commercial partial discharge pulse detectors continued to employ entirely analog circuitry and it is only relatively recently that digital partial discharge pulse detectors have become commercially available.



a) b)

Figure 5 – *Balanced permanent coupler method for partial discharge measurement in waterwheel (a) and steam turbine (b) generators [20]*

Digital Techniques in PD Measurements

When digital PD detectors are employed to measure the apparent charge transfer associated with a given partial discharge pulse, it is important to be cognizant of certain features inherent with the digital PD detector that distinguish its response from that of an analog PD detector. The A/D converter, which forms an integral part of a digital PD detector, consists essentially of a sample and hold circuit followed by a quantizer and an encoder. This implies that a digital detector will display only the PD pulse amplitude variations within a given sampling step, which may be selected to extend, for example, over either one cycle (or period) or ten cycles of the applied voltage wave or 60 cycles, -- *i.e.*, over a one second time period. Consequently, the digital PD detector can only approximate the maximum charge transfer value; hence for this reason a statistically representative average of the maximum determined PD apparent charge transfer value should be employed so that it is in reasonable agreement with the corresponding value obtained with an analog PD detector. The calibration procedure is identical to that of an analog PD detector: a charge, ΔQ, is injected via a calibrating capacitor, C_s, by means of a generator, which provides a square pulse of voltage, ΔV, such that $\Delta Q = C_s \Delta V$. The pulse resolution characteristics are ascertained similarly by variation of the square excitation pulse width to determine the simulated pulse overlap limits between the simulated response pulse at the front and trailing edge of the excitation square pulse. With respect to pulse resolution requirements, it should be emphasized that poorly selected signal triggering levels in conjunction with inadequate data acquisition periods of the digital measuring system may lead to data artifacts when the PD pulse separations are less than the pulse resolution time of the relatively narrow bandwidth conventional detection system [*25*]. In addition, digital processing of under damped PD pulses having overshoots may result in pulse-height distributions with significant pulse polarity errors.

By means of digital filters, the signal-to-noise ratio characteristics of PD detection circuits may be appreciably ameliorated [*25, 26*]. However, a digital filter must be preceded by an A/D converter, while in tandem with the digital filter, an analog reconstruction filter (digital-to-analog converter) must be employed to provide an analog output signal. There are a number of different types of digital filters available; for example, a digital filter may transform the PD signal from the time domain into the frequency domain in which the frequency components of the noise spectrum are reduced to zero, so that the inverse Fourier transform of the noise-free frequency domain signal to the time domain yields a PD pulse form free of distortion due to extraneous interference.

Pulse-height analysis of the PD pulse patterns is frequently employed in research oriented studies to provide a distribution of the discharge pulse-heights, wherefrom, by means of a suitable calibration a distribution in the associated apparent charge transfers may readily be derived. The distribution consists of a plot of the discharge rate, n, in discharge pulses per second versus the apparent charge transfer, ΔQ, whose integral is numerically equal to the total partial discharge current. In an all hardware pulse-height analysis system, a multichannel analyzer is utilized whereby the incoming partial discharge pulses associated with a given discharge pattern having different pulse amplitudes are sorted by a A/D converter into one of a number of possible heights, with this number being determined by the channel capacity of the multichannel analyzer. Figure 6 delineates a hardware arrangement of this type with a high voltage characteristic impedance terminated cable specimen undergoing test; the termination is necessary to eliminate the reflected pulses from influencing the pulse distribution analysis of the actual pulse discharge pattern [*24*].

In addition to the partial discharge pulse-height distribution, other distributions, as for example, the discharge epoch or phase distribution with respect to the sinusoidal applied voltage wave as well as partial discharge pulse interval distribution may be determined [*27, 28*]. With the aid of computer software, the partial discharge pulse-height and discharge epoch distributions may be displayed simultaneously in a three dimensional format. Such an arrangement is portrayed schematically in Figure 7.

The circuit delineated in Figure 7 allows the simultaneous determination of the apparent charge transfer associated with a discrete discharge pulse and the instantaneous voltage value at which the particular pulse occurs, wherefrom it is possible to determine the power dissipated by all the partial discharge pulses taking place over a one second time interval. However, thus far it has not been possible to establish a direct relationship between partial discharge power loss and the ensuing time to failure of an insulating system. The reasons for the inability to arrive at such a relationship may be attributed in part to unpredictable sporadic changes in the measured discharge power loss as a function of time as well as the possibility that a certain amount of the discharge power loss may result from pulseless or pseudo glow type discharges, which a PD pulse detector is incapable of detecting [*29, 30*]. Hence, although the pulse-height analysis technique has been recognized as a powerful analytical research means for the study of partial discharge processes ever since its development [*22-24*], it is only in the area of tests on rotating machines that its practical usefulness as a possible standard has been recognized and implemented.

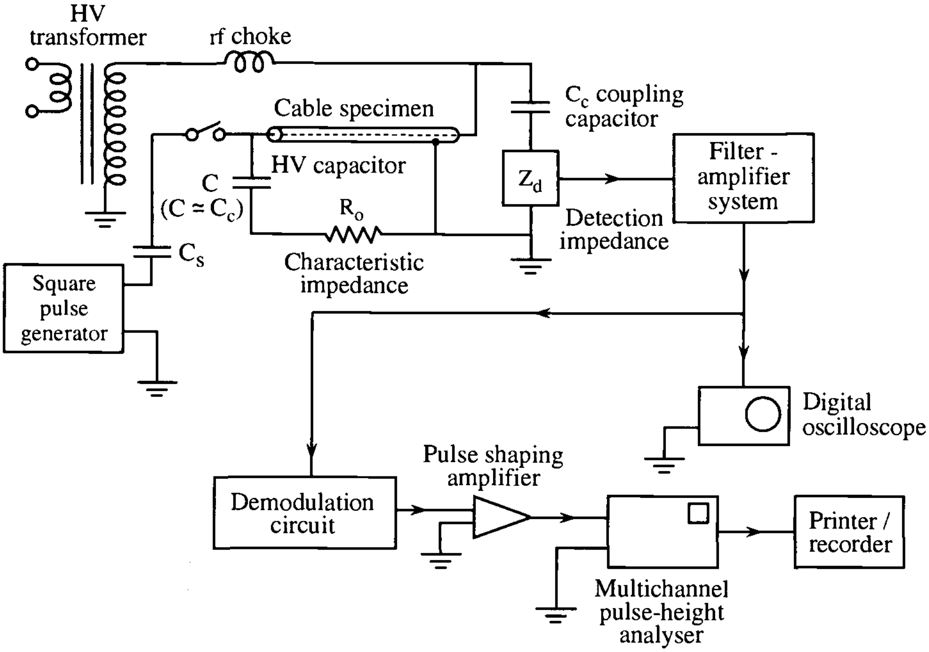


Figure 6 – *Multichannel pulse-height analyzer system for partial discharge analysis on a cable specimen with a HV impedance termination [22-24]*

Plots of partial discharge pulse and epoch distributions provide discharge records that may be regarded as fingerprints, which are characteristic of certain types of voids and their location within the insulating systems of electrical apparatus and cables [*31*]. These characteristics as well as other features, such as partial discharge pulse form (*e.g.*, rise time, peak value, area subtended by the pulse, etc.) may be used in conjunction with intelligent machines to identify the nature of the discharge sources and their location.

Artificial neural networks appear to hold considerable promise in this area [*32*]. A neural network will readily recognize a partial discharge pattern (*i.e.*, pulse-height distribution, discharge pulse shape, etc.) if it has been taught to recognize the same type of pattern that may characterize a known cavity size or distribution and cavity location within a particular insulating system such as that of a cable or a transformer. Evidently, its discharge pattern identification capability will be determined by the number of specific learning case scenarios to which it has been exposed. When presented with an unknown discharge pattern, which it had not been taught previously to recognize, the neural network will either simply not recognize it or, in the worst case, will confuse it with another pattern having some similar attributes. The cognitive efficacy of a neural network is also influenced adversely by the statistically controlled nature of the discharge processes. For instance, statistical variations in the breakdown voltages of the cavities lead to changes in the magnitude of the discharge pulses and result in a precession of discharge epochs [*33, 34*].

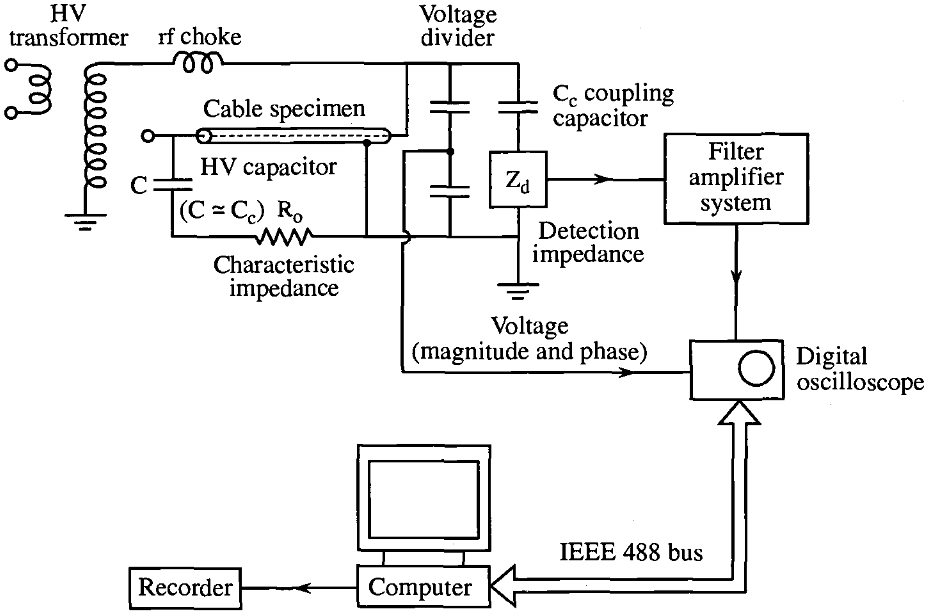


Figure 7– *Schematic circuit diagram for partial discharge pulse-height distribution analysis with the measurement of the discharge epoch and the corresponding instantaneous voltage of occurrence*

One of the most common types of neural networks utilized in cognitive partial discharge pattern studies is the multilayer perceptron (MLP) neural network, which consists of an organized topology of processing elements or neurons as depicted in Figure 8. The processing elements are represented in the form of circles and W_i, W_e and W_o denote the input, hidden and output layers respectively while $a_1, a_2, \dots a_n$ an are features or attributes of the partial discharge pattern. Thus a_1, a_2 and a_3 may be the rise time, amplitude and decay time of the PD pulse, etc., or, alternatively, we may select them to be the pulse amplitude, repetition rate and discharge epoch in a given PD pulse probability density function. The data describing the PD signal or pattern propagates through the processing elements in the forward direction. The neural network learns to identify a given PD test pattern by a changes in its weights, which gradually converge to given values so that each input vector produces the correct output -- *i.e.*, each processing element learns how to modify its weight in response to a particular stimulus. The overall learning procedure is accomplished by a back propagation step whereby the neural network is made to compare its own output with the desired response, making the necessary adjustments of the weights over a series of iterations, if required. The overall ability of a neural network to carry out the recognition task effectively depends also upon the design of the neural network itself, namely the number of processing elements and layers in its structure. Once the neural network has been taught to recognize PD pulse patterns exhibiting certain features, it is ready to be used on similar input patterns on which it should be capable of assigning correct classifications. Evidently, if it has been taught to recognize simple single cavity related PD patterns then, it should not be expected to be able to identify much more intricate discharge sources in complex structures, as for example, transformers. In the latter case, its learning process would have to include a multiplicity of different discharge sources, distributed over a variety of sites along the three phase windings of a power transformer specimen of a particular design. From the practical point of view, since such extensively detailed information is not available currently, any attempt to reject or accepted transformers on the basis of relatively simple PD discharge patterns that neural networks are taught to identify, would be, to say the least, highly premature. Nevertheless, work on PD pattern recognition with neural networks continues; recently some efforts have been also directed to the application of fuzzy logic systems to PD pattern recognition.

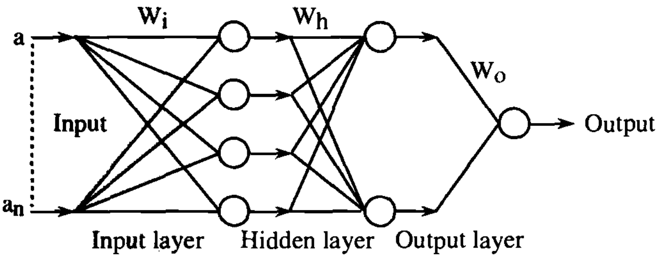


Figure 8– *A possible topology of a multi-layer perceptron neural network for partial discharge pattern recognition [32]*

Discharge Site Location

Although there are virtually no standards on partial discharge site location methods in electrical apparatus and cables, there exist a number of well established testing techniques; this represents an area of considerable activity and interest to electrical power utilities *[35]*. Most of the work is directed towards cables, transformers and rotating machines.

Compressed SF_6 gas insulated cables act essentially as lossless transmission lines and, consequently, high frequency time domain reflectometry methods, with frequencies between 300 MHz and 1 GHz, may be employed to both detect and locate partial discharge sites [*36*]. However, in actual compressed SF_6 gas cables or bus ducts, multiple reflections from the various joints render a travelling wave method less effective for discharge site location. Accordingly, acoustical transducer sensors have become the method of choice for locating discharge sites, particularly in view of the excellent ultrasonic transmission characteristics of SF_6 lines at elevated gas pressures of 5 atmospheres [*37*]. The acoustical sensor is simply moved along the SF_6 cable or duct and the discharge site is located by observing the positions of discharge intensity maxima along the line. Although signal attenuation is smaller at lower frequencies, it is common practice to employ detection frequencies above the audio frequency band in order to evade the high level of ambient noise at frequencies ≤ 20 kHz. Detection sensitivities generally range between 10 and 25 pC, but sensitivities as high as 2 pC at a detection frequency of 50 kHz have been reported [*38*]. There is a certain degree of uncertainty with partial discharge site location in SF_6 lines, since some discharge sites are mobile in the sense that the detected partial discharges may be associated with moving dust or metallic particles within the line.

Much of the partial discharge monitoring and site location on cables is carried out on solid dielectric extruded power distribution cables, which, together with their accessories, are appreciably more susceptible to partial discharge degradation than the equivalent oil-impregnated-paper insulated cables. Although polymeric cables are tested to be discharge-free to a sensitivity level of 5 pC according to standards, discharge sites may develop due to damage incurred during installation or load cycling whilst in service; the same situation applies to their accessories such as splices and terminations. Discharge site location procedures fall into two categories, namely those involving on-line and off-line tests. On-line tests are carried out on cables under normal operating conditions, whilst off-line tests require the cables to be disconnected from the remainder of the distribution system thereby requiring interruption of service to utility customers.

On-line discharge site location measurements are performed most effectively with probes that may be of the antenna, capacitive or inductive type. The antenna probe detects the rf radiation emitted by the discharge site and it functions well along the cable where there are gaps in the concentric neutral shielding such as at unshielded splices and terminations, locating the partial discharge source to approximately within a foot. A schematic circuit diagram of an antenna probe PD location system, operating at a frequency of 7 MHz with a basic sensitivity of 5 pC, is delineated in Figure 9 [*39*]. More precise location of the partial discharge sites necessitates the use of either capacitive or inductive type probes [*40*]. The limitations on the capacitive probes is that they must be placed in contact directly with the semiconductive layer, which is extruded over the polymeric cable insulation -- *i.e.*, they must be inserted underneath the metallic concentric neutrals, unless they are situated at a concentric neutral free surface – *e.g.*, a splice. In contrast, the inductive probes may be applied directly over the concentric neutral, thereby permitting scanning of the entire exposed cable surface. Discharge site location requires that both capacitive and inductive probes be used in pairs in conjunction with a digital oscilloscope to permit the comparison of the pulse arrival times from the discharge source [*loc. cit.*]. Manipulation of the position of the two probes and the use of the known speed of signal propagation along the cable allows the locations of the discharge site at signal sensitivity levels of 2-20 pC, depending upon the ambient noise level.

Present off-line discharge site location is principally carried out with time domain reflectometry (TDR) techniques. While these measurements must be made with the cables temporarily disconnected, necessitating the use of a separate power supply, they have the advantage of being able to view the entire cable length including often a major portion of the length that may generally not be accessible to probe scanning methods.

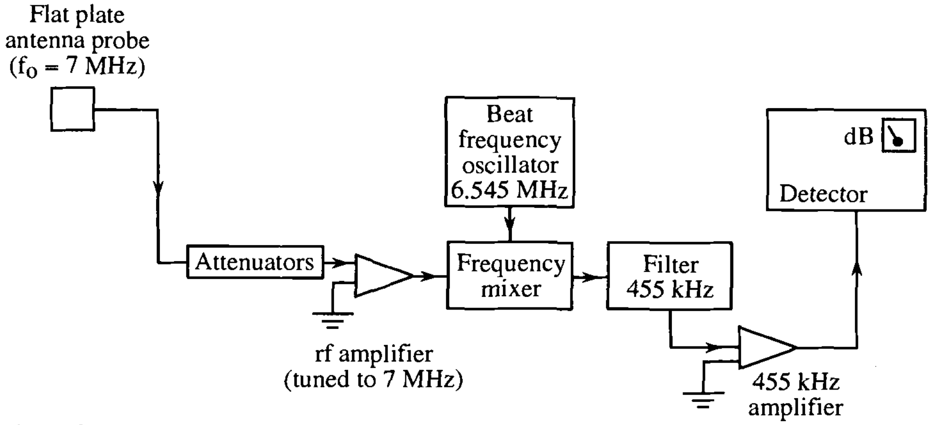


Figure 9 – *Schematic circuit diagram of an antenna probe type PD detector for discharge site location in polymeric cables [39]*

TDR measurement procedures have been recently greatly improved by digital signal processing and can locate discharge faults with good precision; the measure of time delay between the incident pulses and their multiple reflections forms the basis of the discharge site locating procedure [*41, 42*]. The rise time and pulse width of the PD pulses together with pulse broadening and attenuation as well as the signal to noise ratio exert the main influences on the accuracy of the site location. TDR systems suffer from the constraint in that they are generally uncalibrated and the PD pulse amplitudes are recorded in mV in lieu of pC. Figure 10 shows a schematic circuit diagram of a TDR type PD site locating system [*loc. cit*].

Partial discharge site location in transformers using electrical techniques are contingent to a large extent upon full knowledge of detailed pulse transmission characteristics in the windings of the transformer [*17*]. However, such information is generally not readily available. Furthermore, the high ambient noise level with transformer specimens often complicates the problem further, though if the discharge signals are sufficiently repetitive they may be retriewed from a dense but random noise pattern by means of digital signal processing techniques and signal averaging. Nevertheless, in practice discharge site location in transformers is usually carried out employing the simpler route of acoustical techniques, which tend to yield more tangible practical results.

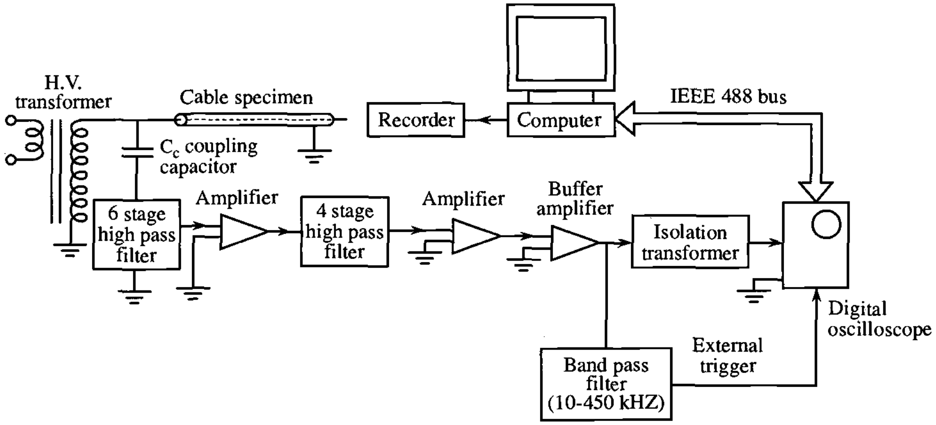


Figure 10 – *Schematic circuit diagram of a TDR system for PD site location in polymeric cables [41, 42]*

Partial discharge site location by acoustical techniques represents a relatively well developed area, that has been effectively utilized since the 1950's [*43*]. The developed triangulation method [*loc. cit.*] utilizes three acoustical transducers mounted upon the face of a transformer tank with their three positions selected by trial and error. The discharge pulse arrival times at the three transducers are recorded, with the first arriving pulse used to trigger the delayed pulses at the two remaining transducers, so that a comparison of the PD pulse arrival times at the three respective transducers provides information as to which transducer is the most proximate to the discharge fault. The method works adequately well for a single discharge site, but, in the presence of numerous discharge sites, difficulties are encountered. In order to circumvent ambient interfere centered at *ca.* 40 kHz as a result of Barkhausen (magnetostriction) noise in the vicinity of substations and aerial HV transmission lines, acoustical detection is carried out at substantially higher ultrasonic frequencies when in-service transformer specimens are involved. For these tests, ultrasonic transducers are generally employed within the frequency band of 100 to 150 kHz [44], or with suitable high pass filters to frequencies as high as 200 kHz [*45*]. The sensitivity of acoustical transducers is generally of the order of 70 pC/mV and is a function of the attenuation properties of the acoustical waves propagating along the transformer windings and the remaining structure of the transformer. Thus, discharge sites, which are situated deep in the inner portion of the windings, are difficult to locate with the acoustical probe method. The coupling efficacy of ultrasonic transducers to the tank or container vessel of the transformer is enhanced with the immersion of the transducer devices in a dielectric liquid medium, thereby simultaneously also reducing their susceptibility to extraneous acoustical noise.

Discharge site location in rotating machines requires the removal of the rotor to provide access for the scanning probes that may then be moved freely without any impediment along the bars or coils of the stator [*17*]. Either capacitive or inductive rf type probes may be deployed; in fact, both probes saw early application to PD site location on rotating machines [*17, 46, 47*]. For safety reasons, however, it was the inductive rf probe, which became the more popular of the two probes [*17, 47*]. The inductive probe is of the ferrite core design, whose semicircular or horse-shoe geometry is shaped to make contact with the two edges of the individual iron slots of the machine to permit completion of the magnetic flux line loop. The inductive of probe is usually designed for operation at 7 MHz, which is substantially above the standard AM radio broadcast band interference; it operates normally in conjunction with a quasi-peak voltmeter, providing an indication of the PD intensity in μV. Since the probe scans an energized stator with the rotor removed, some cyclic slot gap discharge sites, which appear only intermittently due to electromagnetically induced bar vibrations with the machine in operation, are not detected in this type of stationary test.

Concluding Remarks

Present standardization of partial discharge measurements on transformer, solid dielectric extruded cables and capacitors is centered on go no-go type tests and involves essentially the determination of the partial discharge inception and extinction voltages at specified sensity values of apparent charge transfer expressed in pC. The basis for these types of standardized tests rests firmly with the conviction that any presence of partial discharge under operating voltage conditions is detrimental to the life of the insulation and cannot, therefore, be tolerated. More sophisticated partial discharge measurement methods involving partial discharge pulse-height and discharge epoch distribution analysis have not fully attained the level of standardization, primarily because of their complexity and some unresolved interpretational considerations. One noted exception in this area is the quasi-empirical PD pulse-height analysis technique, which is widely accepted and extensively used on rotating machines where apparent charge calibration procedures have encountered great difficulty. Also, only very recently some effort has been expended to extend pulse-height analysis techniques to compressed SF_6 gas bus and cables [*48*].

Use of intelligent machines on PD pattern recognition is still limited to simple cases involving isolated and often artificially created cavities. Their application to more complex discharge sources in actual power apparatus and cables has been hindered by the inability of providing a range of well defined discharge sites and sources in such equipment, which could be employed for the purpose of teaching the intelligent machines to recognize and distinguish between these discharge sources and their location. Stated explicitly in simple terms, one ought not expect intelligent machines to classify correctly discharge patterns, if they are not taught first to recognize such patterns. On the other hand, there are now available a number of well proven and established techniques for partial discharge site location, which could conceivably meet standardization criteria. However, thus far, little effort has been made to proceed with such work.

Glossary of Partial Discharge Terms

Partial discharge is an electrical discharge that only partially bridges the insulation between conductors.

Partial discharge inception voltage (PDIV) represents the lowest voltage at which continuous partial discharges above a given magnitude may be sustained.

Partial discharge extinction voltage (PDEV) is the value of the highest voltage at which partial discharges above a given magnitude cease to recur as the applied voltage is gradually decreased from above the PDIV.

Partial discharge pulse voltage is the measured terminal pulse voltage across a specimen resulting from a partial discharge event within the insulating system of the specimen.

Apparent charge transfer represents the measured charge associated with a given detected partial discharge pulse resulting from a partial discharge event in the insulting system of a specimen.

Partial discharge pulse is a voltage or current pulse that results from a partial discharge event within the insulating system of a specimen and that is sensed externally by a partial discharge detection circuit.

Partial discharge pulse pattern is a display of partial discharge pulse amplitudes with respect to the applied sinusoidal waveform; it is normally displayed on a power frequency time base.

Partial discharge pulse epoch represents the phase of the discharge pulse occurrence with respect to the applied sinusoidal voltage wave.

Partial discharge rate is the average number of discharge pulses that occur per second or over some other specified time interval.

Partial discharge pulse height distribution represents a display of charge transfers associated with each discrete discharge pulse as a function of its recurrence rate.

Partial discharge pulse epoch distribution represents usually a three dimensional display of the discharge pulse amplitudes as a function of their respective recurrence rates and discharge epochs.

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Long-Term Endurance Determinations Using Traditional Accelerated Aging in Combination with Oxidative Stability Testing Resorting to Isothermal DSC Measurements of Oxidation Induction and Maximum Times

Reference: Giannoni, S. G., Montanari, G. C., Motori, A., and Hasan, O. A., "**Long-Term Endurance Determinations Using Traditional Accelerated Aging in Combination with Oxidative Stability Testing Resorting to Isothermal DSC Measurements of Oxidation Induction and Maximum Times,**" *Electrical Insulating Materials: International Issues, ASTM STP 1376,* M. M. Hirschler, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

Abstract: During the service life of electrical equipment, organic electrical insulating materials used at elevated service temperatures can degrade as a result of progressive chemical reactivity. It is therefore essential that normal operating temperature limits be established for such materials. The traditional approach is to accelerate the degradation process with simultaneous testing at moderately elevated temperatures above the intended service temperatures of both the candidate material and a control reference material. With proper application of a chemical kinetic model, a service limit is established for the candidate material based on the comparative test performance, with the control having known long-term thermal service capabilities. There are practical limits on the maximum test temperatures and corresponding minimum test times to ensure an accurate representation of the service degradation mechanism during the test program. This results in an onerous, unavoidably protracted test time for these thermal aging studies. A number of relatively rapid analytical techniques have been proposed to reduce the test times without compromising the accuracy of the determined service limit. The most successful of these often involve hybrid testing, as a combination of a rapid analytical technique and part of the traditional program at the higher test temperatures and shorter test times, to result in a desirable and substantial reduction in test program times. This paper will review the principles of the traditional program, and the theory and initial results of a proposed hybrid technique based on oxidative stability testing using oxidation induction.

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Keywords: thermal aging, thermal endurance, Arrhenius, oxidation induction time, oxidation maximum time, oxidative stability, asymptotic kinetic model, isothermal DSC

Introduction

The evaluation of electrotechnical products requires an assessment of long-term service performance as potentially affected by limiting stress factors. Since such products normally dissipate electrical energy as the production of heat, resulting in elevated normal operating temperatures for critical organic insulating materials, the predominant long-term stress factor is often thermally activated chemical degradation. An essential element of the assessment of material suitability is determining maximum normal operating temperature limits, at which long-term material performance will not be unacceptably compromised.

The assessment of material long-term performance capability usually involves the intensification of the predominant stress factor to accelerate the degradation process to produce manifestations of progressive material changes in a practical short test time. The study is completed by establishing a relationship between the rate of material change as a function of the level of the stress factor. The process is greatly enhanced if there is a reliable model as a basis for analysis of the degradation process. For thermally activated chemical degradation, the intensification of the predominant stress factor is achieved by exposure of material samples to moderately elevated temperatures above the normal service temperatures. Progressive material changes are measured by appropriate diagnostic property tests and the chemical kinetic model is applicable for the analysis, consisting of an extrapolation of performance from test temperatures down to service temperatures. A control reference material is simultaneously tested together with the candidate to eliminate systematic errors, and to establish a link between test performance and service performance via the known long-term thermal capability of the control.

In principle, ever higher test temperatures could result in desirably shorter test times. In practice, maximum test temperatures are severely limited, in that different degradation processes are likely to predominate at excessively higher test temperatures, than at service temperatures. Also, the greater extrapolation of performances from the range of higher test temperatures to service temperatures will tend to introduce unacceptably greater extrapolation errors. Consequently, typical program times range from nine months up to 1½ years or more. These onerous test times impose a severe hardship that is unacceptable for material producers and product designers alike, where product development cycles are much shorter, and require reliable material long term thermal capability assessments times, not exceeding three to four months.

A number and variety of test protocols have been proposed to shorten the material evaluation time. Many of these are analytical techniques such as thermogravimetry using very high test temperatures, yet relying on the same degradation process to be operating at these extreme temperatures as at the service temperatures. Some even resort to an oxygen free test environment to prevent material combustion; clearly absurd considering that oxidation is often the predominant service degradation mechanism. Yet the sophistication and sensitivities of the analytical hardware suggest the tantalizing possibility of developing some new reliable protocol for reducing the traditionally long test time.

One such new method uses the technique of isothermal differential scanning calorimetry (DSC), at much lower temperatures, that are only moderately above the test temperatures of the traditional isothermal method. It is observed that once thermal stability is established at the elevated test temperatures in an oxygen free environment, then introducing oxygen will initiate an oxidative degradation sequence, with heat flow measurements clearly indicating times of progressive degradation. These temperature time data are then used, as in the traditional conventional analysis to determine the activation energy parameter of the oxidation degradation processes, or the slope of the degradation curve. The position of the curve is then fixed by the results of at least one of the higher temperatures of conventional thermal aging, hence dramatically shortening the program test time.

Conventional Isothermal Protocol – UL 746B Relative Thermal Index (RTI)

An understanding of the theoretical basis for expecting the classical thermal endurance line, for log times to reach an end-point state as a linear function of reciprocal absolute temperatures, is important since this relationship is applicable for both the traditional and oxidative stability methods. These thermal endurance lines are fundamental to several preeminent protocols, including standards IEC 216 Guide for the determination of thermal endurance properties of electrical insulating materials, UL 1446 Systems of Insulating Materials and UL 746B Polymeric Materials – Long Term Property Evaluations. The UL 746B protocol has been selected for the following discussion since it illustrates some important characteristics of test developed thermal endurance lines, when used to determine the long term thermal capability of materials in electotechnical applications.

During UL 746B material evaluations, simultaneous destructive testing of a candidate material and a control reference material, is used to study the degradation of selected diagnostic properties of standard specimens during exposure to moderately elevated aging temperatures, above the maximum intended use temperature. For the property, an end-point at each elevated aging temperature is defined as that time required to produce a reduction in the property to some fraction (e.g. 50%) of the unaged value. These test properties serve as diagnostic indicators of the point where cumulative thermal degradation has progressed to the same (though arbitrary and in fact unknown) extent at each aging temperature. With proper application of chemical kinetic theory and subject to the limitations imposed by necessary underlying assumptions, then the log of these end-point times should be a linear function of the reciprocal of the absolute aging temperatures. This linear relationship is the thermal endurance line for the material, and is commonly and incorrectly known as the Arrhenius equation.

Following is a brief and simplified derivation of the linear thermal endurance equation. The true Arrhenius equation relates only specific reaction rate and absolute temperature :

$$K = Ae^{-\frac{E}{RT}} \tag{1}$$

where:
$K =$ Reaction Rate Constant
$E =$ Degradation Reaction Activation Energy
$R =$ Gas Constant
$A =$ Constant
$T =$ Absolute Temperature

Specific reaction rate is generally defined by the following differential equation:

$$dQ/dt = -Kg(Q) \tag{2}$$

where:
$Q =$ Quantity of unreacted good material
$t =$ Time
$g(Q) =$ Characteristic Function. For simple n(th) order reactions, $g(Q) = Q^n$. The form of $g(Q)$ has no effect on the linearity or otherwise of the thermal endurance equation.

Separating variables of (2) and integrating :

$$\int_{Q_0}^{Q_f} \frac{dQ}{g(Q)} = \int_0^{t_f} -Kdt \tag{3}$$

where:
$Q_0 =$ Initial Quantity of unreacted material
$Q_f =$ Quantity of unreacted material at the end-point
$t_f =$ Time to reach the end-point

The left side of (3) is only a function of Q_0 and Q_f, say $-C(Q_0, Q_f)$ and is constant since Q_0 and Q_f are constant (i.e. - the state of as received specimens is the same at each aging temperature, as is the state of end-point specimens). Computing (3) and substituting (1) yields:

$$t_f = \frac{C(Q_0, Q_f)}{A} e^{\frac{E}{RT}} \tag{4}$$

Taking common logs and changing variables yields the linear thermal endurance equation :

$$Y = mX + b \tag{5}$$

where :
$Y = log(t_f)$
$m = (E/R)log(e)$
$X = 1/T$

$$b = \quad log[C(Q_0, Q_f)/A]$$

The thermal endurance equation relating test times and temperatures, in either the exponential form (4) or the more common transformed linear form (5) has two parameters. The slope and intercept terms, m and b of (5), are commonly referenced as the activation energy and pre-exponential terms respectively. (m) is clearly directly proportional to the degradation reaction activation energy E through the Arrhenius equation. Most importantly, the defined relationship between test end-point times and exposure temperatures is not directly related to material service life and use temperature. Please recall in the analysis, that the definition of the test end-point performance level is arbitrary (as is to some extent the choice of the diagnostic property) and only for determining the exposure times at each temperature to produce an equal, though unknown, extent of cumulative thermal degradation.

A primary function of the control reference material is to establish a link between test performances and maximum service use temperatures. The control must have an accepted maximum service temperature limit in general electrotechnical products with respect to long term thermal chemical degradation of a class of critical property. Parallel testing of this material, with a candidate material, is conducted using a suitable diagnostic property having a similar character as the class of critical service property, to define thermal endurance lines. A test correlation time is defined as that time corresponding to the control maximum service temperature, and the analysis is completed by assigning a relative temperature index to the candidate at that temperature corresponding to the correlation time. Another important function of the control is to reduce systematic errors. Due to a variety of uncontrollable and often undetectable factors, the resulting thermal endurance lines can vary considerably between valid programs, in spite of every effort to maintain consistency. This translates into differing test program correlation times, but the effect on the determined candidate maximum service temperature is minimal if the control is tested under identical conditions.

Oxidation Stability Techniques

In the typical mode of operation, the Differential Scanning Calorimeter (DSC) subjects a small specimen of material to a programmed temperature increase and monitors the rate of heat exchanged to the specimen. The data is useful in determining a number of material characteristics, especially physical transition temperatures, such as the glass transition temperature (Tg). The program normally spans temperatures into very high ranges.

If instead, the DSC is set to maintain a constant temperature (isothermal mode) with a flow of inert gas (usually N_2) until thermal stability, and then O_2 is introduced at time zero, a characteristic sequence of heat exchange as a function of time can result as shown in Figure 1, indicating results on testing of cross linked polyethylene at two temperatures [1]. These characteristics reflect definite stages of the onset through stability of oxidation, as the oxidation induction time (OIT) and the oxidation maximum time

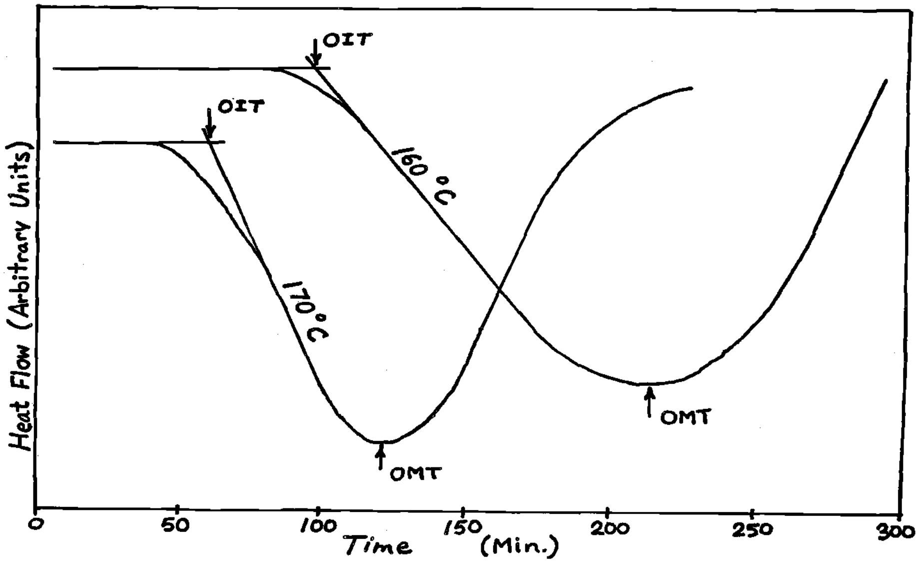

Figure 1 – *Typical Isothermal DSC Characteristics*

(OMT) as indicated. OMT is a useful characteristic time required for the material to progress to a defined state of oxidation at the test temperature.

If the test is conducted at several temperatures and oxidation is the predominant mechanism of material degradation at service temperatures, then this technique can serve to establish the activation energy of the material degradation reaction via the determined slope (m in Equation 5) of the resulting thermal endurance line [2]. It then remains only to fix the position of the line via a conventional aging test, at a single temperature chosen sufficiently high, to result in the desired significant decrease in total test program time. The practical isothermal DSC temperatures are fortunately sufficiently close above the corresponding temperatures of conventional aging, to insure that the oxidation mechanism is likely to be the similar to the service degradation mechanism.

Verification Study

Conventional Thermal Aging

A conventional isothermal study was conducted on a typical Polybutylene Terephthalate (PBT) engineering molding resin according to the standard UL 746B protocol, using a similar material for simultaneous testing as a control reference. The material was tested in several thicknesses and using several diagnostic property indicators, since both these factors are known to influence results in these long term thermal endurance studies. Experience has shown that non-impact mechanical properties

Table 1 – *Summarized Results of Conventional Thermal Endurance Program*

Temps.->	140 C		150 C		160 C		170 C	
	Hours	MPa.	Hours	MPa.	Hours	MPa.	Hours	MPa.
	0	3.515	0	3.515	0	3.515	0	3.515
	2160	3.555	1008	3.319	504	3.178	216	3.385
	4320	2.457	2016	2.354	1008	2.162	432	2.626
	5760	1.958	3024	1.957	1176	1.944	504	2.419
	6480	2.099	4032	1.71	1344	1.797	576	2.408
4^{th} Order Curve Fit Constant \\\|/	7200	1.722	4368	1.335	1512	1.386	672	2.198
	7920	1.212	4704	1.422	1680	1.1	720	1.739
			5040	1.325			792	1.241
A0 ->	3.51323		3.53082		3.51593		3.53082	
A1 ->	1.07989 E-03		2.80304 E-04		0.0017435		0.000280304	
A2 ->	-7.40555E-07		-7.60717E-07		-7.39168E-06		-7.60717E-07	
A3 ->	1.3363E-10		2.19178E-10		5.9035E-09		2.19178E-10	
A4 ->	-7.82393E-15		-1.92132E-14		-1.56876E-12		-1.92122E-14	
50% End Points ->	7178 hours		3337 hours		1317 hours		735 hours	

in larger thicknesses are more consistent diagnostic indicators of thermal degradation, hence for the purpose of this paper, only the tensile strength property in the 3.2 mm thickness will be considered. The summarized results of aging at four temperatures are included in Table 1.

The values listed in Table 1 are the mean of five destructive measurements at each indicated test aging time on samples tested at standard laboratory ambients, according to the ASTM Test Method for Tensile Properties of Plastics, D 638.

UL 746B suggests using ploynomial regression to develop a smooth curve through the degradation data points. In this case, fourth order polynomial coefficients A0 through A4 are shown in the table, together with the computed 50% times. Of course there is no theoretical basis for a polynomial representation of the degradation data, but this technique has been extensively used as an automated drawing tool in UL 746B thermal aging programs. The corresponding technique of IEC 216 is a linear regression of property as a function of log aging time for data points in the immediate interval around the end point. Likewise, there is no theoretical basis for this form of the curve fitting technique. In fact, under the ideal assumption, that property will be proportional to unreacted good material left in the sample, then a time exponential decay of property would result, and log property would be a linear function of time.

Figure 2 is a plot of the data points together with the resulting thermal endurance line, computed using the method of least squares linear regression. The line in the form of equation (5) is $Y = 6180X - 11.10$ with a correlation coefficient of 0.99. Normally in accordance with the standard UL 746B, the resulting Relative Temperature Index

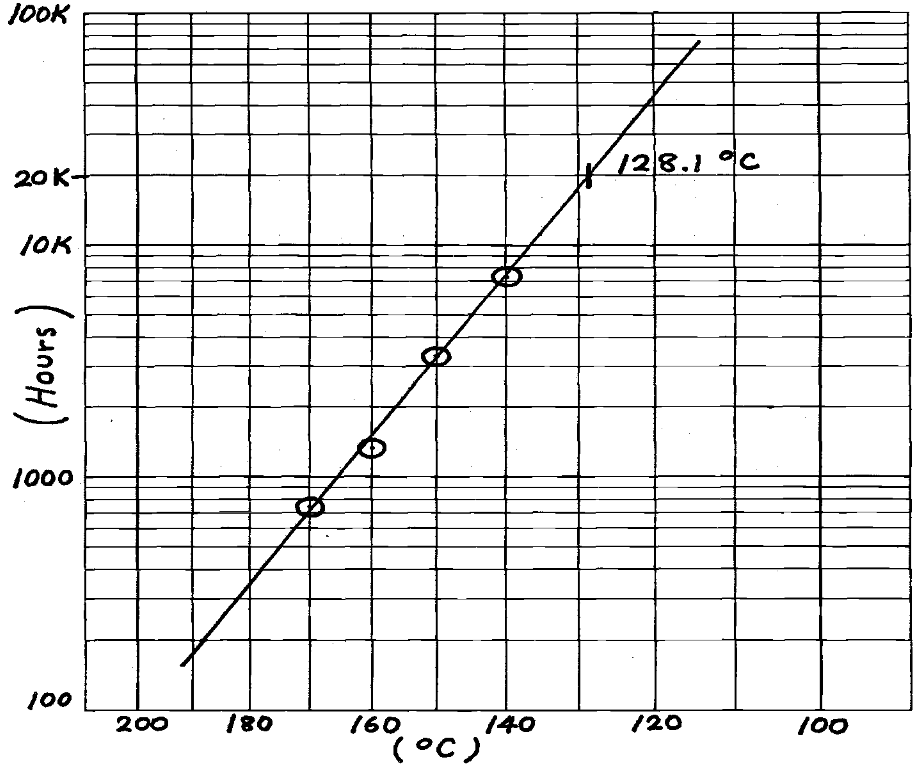

Figure 2 – *Thermal Endurance Line with 20,000 Hour TI*

(RTI) is computed as that temperature on the thermal endurance line at the determined correlation time taken from the thermal endurance line of the control material at the RTI of the control material. In this study, only the candidate material was tested according to the oxidation stability technique, and therefore an absolute correlation time of 20,000 hours will be applied as prescribed in the standard IEC 216, yielding a Thermal Index (TI) temperature of 128.1°C.

Oxidative Stability Aging

Measurements of oxidation maximum times were conducted on specimens from the same sample of PBT candidate material, using an isothermal technique with the ASTM Standard Test Method for Transition Temperatures of Polymers by Thermal Analysis, (D3418). The mean results of a minimum of three determinations at each temperature are

Table 2 – *Data of Oxidative Stability Study*

Temperature (°C)	Time (minutes)
175	3340
180	1881
185	1224
190	718.3
195	387.3
200	364.7
205	489.3
210	160
215	68.5
220	40
225	16.6
230	11.3

summarized in Table 2. Again, applying the method of least squares linear regression to all data points with times in hours, yields a line in the form of equation (5) of $Y = 9768\,X - 19.97$ with a correlation coefficient of 0.97. Passing a line with the same slope through the conventional aging point at 160°C, 1317 hours, results in a temperature at 20,000 hours of 138.4°C which deviates approximately 10°C above that determined from the conventional aging study.

The results of the oxidative stability are plotted in Figure 3 together with the regression line on all points. Significant nonlinearity is readily apparent on inspection, attributable to different degradation mechanisms predominating in higher temperature intervals. If data points above 195°C are rejected then a more linear expression in the form of equation (5) results as : $Y = 9600\,X - 19.67$ with a correlation coefficient of 0.99. Passing a line with this slope through the conventional aging point at 160°C and 1317 hours results in a slightly better temperature of 136.0°C at 20,000 hours.

Discussion of Results

Aging Temperatures and Extrapolations

Both the conventional aging and oxidative stability techniques are based on the same chemical kinetic model for the predominant degradation process, which in this case is assumed to be oxidation. Other analytical techniques are also based on this model but tests are conducted at much higher temperatures and shorter times. The problem with all techniques is that different degradation mechanisms are more likely, at ever greater temperatures above service temperatures and with greater extrapolation errors from the high test temperatures to the service temperatures.

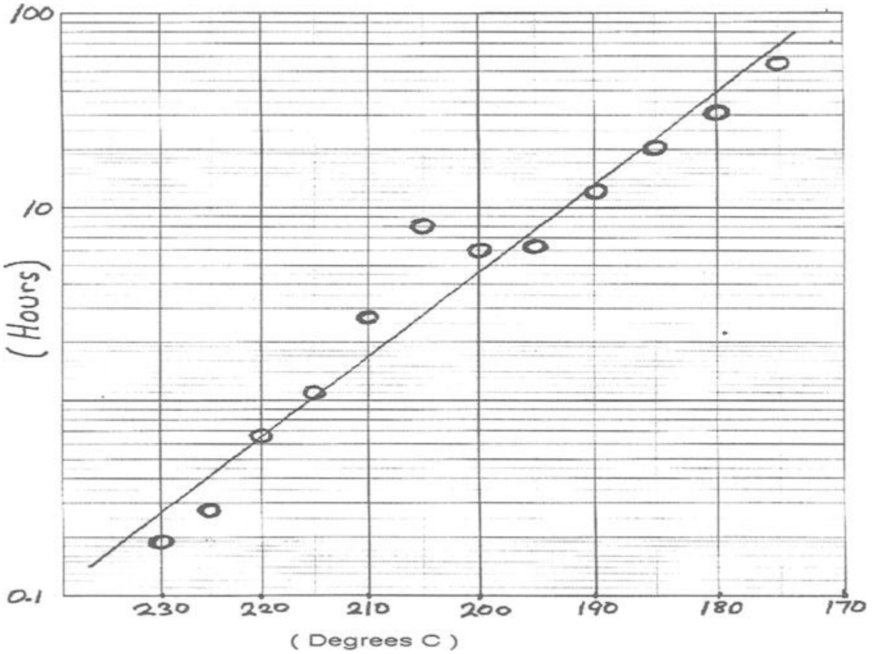

Figure 3 – *Plot of Oxidative Stability Data*

The oxidative stability technique has an important advantage over other analytical techniques in using test temperatures that are relatively low, and only moderately greater than those of conventional aging. The corresponding oxidation maximum times are in a range up to 55 hours, and are relatively long compared to other analytical techniques. Still, in this study, the highest temperatures have resulted in oxidation maximum times down to only fractions of an hour, and there was a clear departure from linearity in the data above 195°C. It would seem prudent to impose a minimum limit on useful oxidation maximum times by rejecting data in nonlinear regions.

Asymptotic Kinetic Model for Nonlinearity

The traditional degradation model is actually based on a homogeneous system of gaseous reactants. The success in applying this model to thermally degrading solid insulation is surprising. As a result of complex fixed intermolechular geometries, there is an effective distribution of degradation reaction activation energies rather than a single quantity assumed as in the case of the ideal system. This distribution of activation energies, will impart non-linearity to the thermal endurance line.

Also, as shown in the oxidative stability data, different degradation mechanisms resulting from physical and other changes will tend to predominate in the higher temperature ranges. Changes in crystallinity and a greater mobility of reactants above glass transition are examples of such changes.

These factors contributing to nonlinearity tend to result in uncharacteristically shorter end point times at ever higher aging temperatures, imparting a downward curvature to the thermal endurance line, increasingly at the highest temperatures.

It has been proposed to expand the Arrhenius equation in the form of equation (5) by the addition of an asymptote temperature term ***T**** and a kinetic exponent term ***n***, in an enhanced form of equation (5').

$$Y = m'\left(\frac{1}{T} - \frac{1}{T^*}\right)^n + b' \tag{5'}$$

If T* = ∞ and n = 1 then the traditional Arrhenius equation results. T* is a temperature where the reaction rate will tend toward infinity. In real terms it is a very high temperature where total degradation would tend to be instantaneous. The effect is of moving the infinite temperature point down to T* and in a general way accounts for the nonlinear increases in reaction rate at the highest temperatures. The term·n is intended to take account of the effective distribution of activation energies in the solid material. The terms m' and b' can be interpreted as the corresponding parameters in equation (5) but will naturally take on different values in (6).

Software has been developed to produce the now four parameter curve fit to aging data points using a hybrid, linear regression / 2-parameter grid search technique [3]. For each grid search selection of parameters *T** and *n*, a linear regression is performed using the converted variable data to optimize the parameters *m'* and *b'*, and the optimal combination of all four parameters yielding the best least squares fit is chosen. To eliminate an "outlier" point, the process is repeated with each of the data points sequentially ignored by the calculations, and noting which of the twelve sets yields the best least square fit. The result for the OMT data is shown in Figure 4. The 205°C point is clearly the outlier as determined by this technique, and is indicated by gray coloring. The observed deviations near this point have been attributed to polymer recrystallization. The computed 4 parameters are *m' = 401.6, T* =513.2 °K = 240.2* °C, *n = 0.577* and *b' = -1.924.*

Interestingly though, perhaps of limited significance, is that the computed slope of the curve at the conventional aging temperature of 150°C is 6643, and passing a line as before with this slope through the conventional aging point, yields a temperature of 130.0 °C at 20,000 hours. This is considerably closer to the conventional aging result than either of the previous oxidative stability linear estimates.

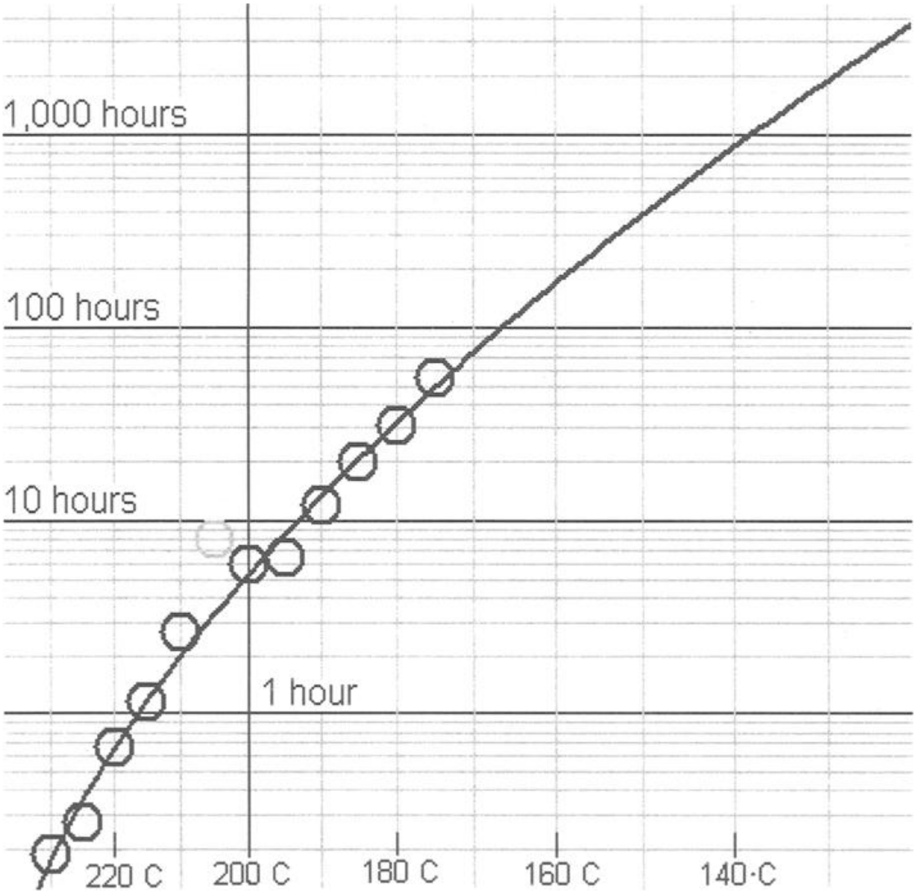

Figure 4 – *Asymptotic Kinetic Curve Fit to OMT Data*

Practical Methodology

The asymptotic kinetic analysis is included as a promising new method for dealing with nonlinearities in thermal endurance curves as can be observed especially in higher temperature tests like the OMT measurements. If tests are restricted to linear ranges, then the OMT analysis can be used in a straightforward approach with conventional aging to determine thermal endurance characteristics in significantly reduced test program times.

A number of additional studies will be needed to verify the oxidative stability technique, perhaps as relatively small additions to the many ongoing conventional aging programs.

Also, predominan degradation mechanisms other than oxidation may need to be addressed, e.g. – hydrolytic depolymerization on crosslinking. It is possible that the isothermal DSC technique could be applied to these other mechanisms. Where an environmental component is identified as an active element of the predominant degradation process, then this component could be introduced at time = 0, much the same as with O_2 in the oxidative stability technique. In cases where there is no identified environmental component of the predominant degradation reaction, it might still be possible to bring the specimen and system to elevated thermal equilibrium in a relatively short time, and note the longer term thermal characteristic to identify times at different temperatures of equal cumulative thermal degradation.

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Fire Issues

Ronald L. Markezich[1]

Comparative Tracking Index of Flame-Retardant Nylon and PBT

Reference: Markezich, R. L., "**Comparative Tracking Index of Flame-Retardant Nylon and PBT,**" *Electrical Insulating Materials: International Issues, ASTM STP 1376,* M. M. Hirschler, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

Abstract: Flame-retardant nylons and PBT have been used in the electrical and electronic industry for many years. To flame retard these materials, halogens can be used with different synergists. The choice of synergist and also the halogen flame retardant has a great effect on one of the electrical properties, the comparative tracking index (CTI). The choice of resin, PBT, nylon 6 or nylon 66 also has an effect on the CTI.

Keywords: CTI, FR-PBT, FR-Nylon, UL-94, synergists, tracking resistance, antimony oxide, zinc borate, iron oxide, teflon, flame retardants, dechlorane plus

Flame-retardant nylons and polybutylene terephthalate (PBT) have been used in the electrical and electronic industry for over 20 years. They are available as both glass-reinforced or non-reinforced compounds. The flammability requirement met is a V-0 as measured by the UL-94 test procedure.

The flammability requirements are met by the addition of additives. Materials of choice for nylons have been red phosphorus, brominated additives, and a cycloaliphatic chlorinated compound known as Dechlorane Plus® (CFR). This latter material may be used with various metal oxide synergists, which can have a major effect on the electrical and physical properties of the FR-nylon compounds. To flame retard PBT, various brominated additives and also the chlorinated flame retardant "CFR" have been used with the synergist antimony oxide.

The electrical test we use is one designed to measure sensitivity to surface tracking, called comparative tracking index (CTI). The CTI test measured is either Kc or Kb, depending on the electrolyte solutions used for the test. The Kc solution is 0.1 g NH_4Cl in 100 mL of distilled water, while the Kb solution is 0.1 g of NH_4Cl and 0.5 g cationic surfactant in 100 mL of distilled water.

Materials and Procedures

The additives used and their sources:

CFR	Dechlorane Plus®	Laurel Industries
Antimony Oxide	Thermoguard® S	Laurel Industries
Zinc Oxide	Kadox 15	New Jersey Zinc
Zinc Borate	Firebrake® ZB	U.S. Borax
Ferric Oxide (Fe_2O_3)		J. T. Baker
Iron Oxide-Black (Fe_3O_4)	Akrochem's E-8846	Akron Chemical

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[1] Laurel Industries, 30195 Chagrin Boulevard, Cleveland, OH 44124.

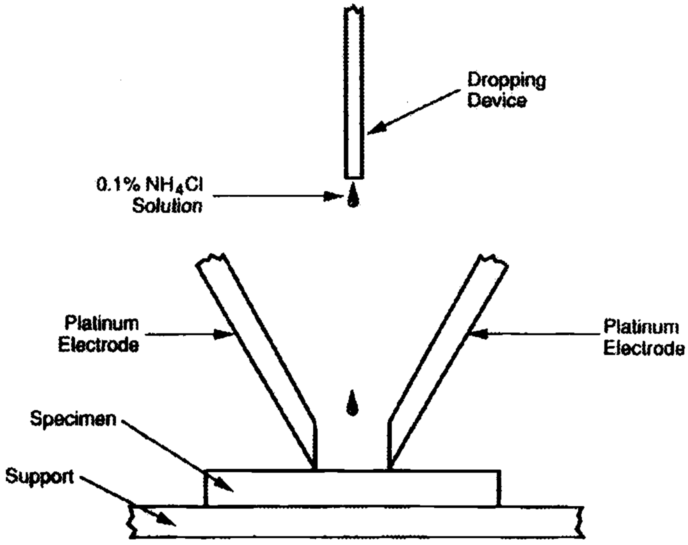

FIG. 1—*Tracking resistance.*

The resins were dry blended with the additives, extruded, pelletized, dried, and then molded into test bars. The conditioned bars, 1/8 in. (3.2 mm), 1/16 in. (1.6 mm), 1/32 in. (0.8 mm), and 1/64 in. (0.4 mm) were then tested for flammability using the UL-94 vertical burning test.

Several closely related methods are available for measuring the tracking index of materials [*1–3*]. All these procedures consist of dropping an electrolyte at defined time intervals to the

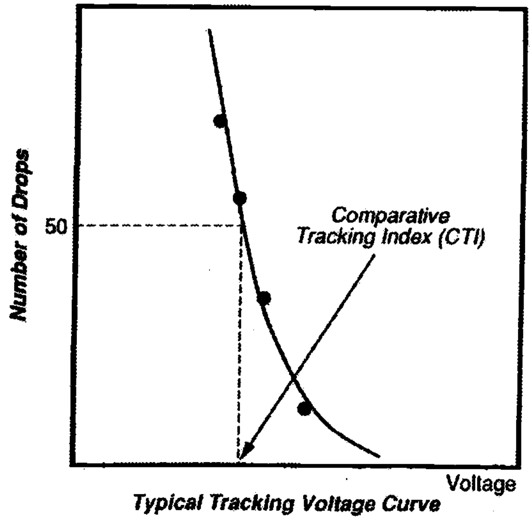

FIG. 2—*Tracking resistance.*

TABLE 1—*FR-Nylon 66 using different synergists.*

Formulation (Weight %)	1	2	3	4	5	6	7
Nylon 66	70	70	70	82	78	70	85
CFR	20	20	20	14	16	20	12
Sb_2O_3	...	10	...	...	...	...	...
Zinc Borate	...	10	...	...	4	5	1.5
Zinc Oxide	...	...	10	...	...	5	...
Ferric Oxide	...	...	...	4	...	...	1.5
UL-94 3.2 mm	V-0	V-0	V-0	V-0	V-0	V-0	V-0
1.6 mm	V-0	V-0	V-0	V-0	V-0	V-0	V-0
0.8 mm	V-0	V-0	NC	V-0	V-0	V-0	V-0
0.4 mm	V-0	V-0	NC	V-0	V-0	V-0	V-0
CTI Kc (Volts)	275	300	600	275	425	375	350
Kb (Volts)	...	...	600	...	...	...	...
Tensile Strength (MPa)	58.6	59.3	57.3	71.8	66.2	57.3	72.5

surface of the plastic molded part between two electrodes to which a voltage is applied. Figure 1 depicts this schematically.

The inclusion of the surfactant is to provide a harsher environment for the test. When a voltage is applied, it has been found that the number of drops which can be applied before a surface tracking that conducts substantial current is formed is a function of the voltage. The voltage which will cause failure at 50 drops of electrolyte has been selected as a measure of susceptibility of a material to tracking. It is defined as the Tracking Index or Critical Tracking Index (CTI). This phenomenon is shown in Fig. 2.

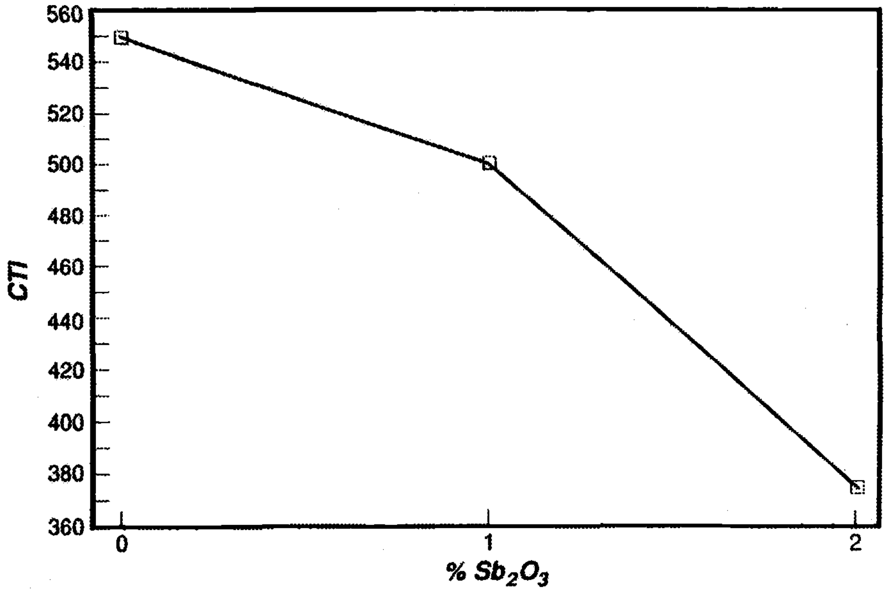

FIG. 3—*CTI of FR-Nylon 66 vs. Sb_2O_3 levels, 14% Dech Plus/Zinc Borate 4%–%Sb_2O_3.*

TABLE 2—*FR-Nylon 66 (25% glass-reinforced) using different synergists.*

Formulation (Weight %)	1	2	3	4
Nylon 66	49	48	48	60
Fiberglass	25	25	25	25
CFR	18	18	18	12
Sb_2O_3	8	...	...	...
Zinc Borate	...	9	...	...
Zinc Oxide	...	...	9	...
Ferric Oxide	...	...	...	3
UL-94 3.2 mm	V-0	V-0	V-0	V-0
1.6 mm	V-0	V-0	V-0	V-0
0.8 mm	V-0	V-1	V-1	V-0
0.4 mm	V-0	V-1	NC	V-0
CTI Kc (Volts)	225	375	325	225
Kb (Volts)	...	200	200	...
Tensile Strength (MPa)	122.8	121.4	97.2	98.3

In Nylon 66, ferric oxide is the most effective single synergist with CFR (Table 1). It gives a UL-94 V-0 material with only 14% of the flame retardant with 4% of the synergist, but the CTI (Kc) is only 275 volts. These are the same values that are obtained when antimony oxide is used as the synergist. The highest CTI obtained is when zinc oxide is used as the synergist, both Kc and Kb are 600 volts.

By using mixed synergist with zinc borate and CFR, the loading level needed for a UL-94 decreases and the CTI increases, Table 1, Formulations 5 and 7. Formulation 5 has a CTI (Kc) of 425 volts and only needs a 22% level of flame retardant package to achieve a V-0

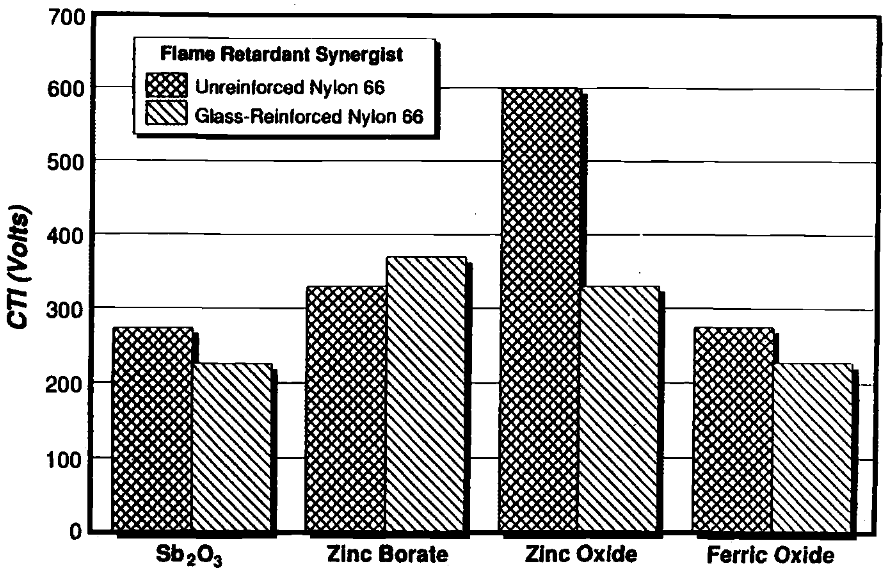

FIG. 4—*CTI for FR-Nylon 66, unreinforced vs. glass reinforced.*

TABLE 3—*FR-Nylon 66 (25% glass-reinforced) using different synergists.*

Formulation (Weight %)	1	2	3	4
Nylon 66	55	49	60	62
Fiberglass	25	25	25	25
CFR	16	18	12	10
Sb_2O_3	2	...	...	...
Zinc Borate	2	4	1.5	1.5
Zinc Oxide	...	4	...	...
Iron Oxide	...	...	1.5	1.5
UL-94 3.2 mm	V-0	V-0	V-0	V-0
0.6 mm	V-0	V-0	V-0	V-0
0.8 mm	V-0	NC	V-0	V-0
0.4 mm	V-0	NC	V-0	V-0
CTI Kc (Volts)	350	375	225	225
Kb (Volts)	200	200	...	...
Tensile Strength (MPa)	124.5	103.5	112.2	114.7

at 0.4 mm in a white material. Formulation 7 is a V-0 with only 15% total flame retardant package and has a CTI of 350 volts.

The replacement of zinc borate by antimony oxide has a negative effect on CTI (Fig. 3). Replacing zinc borate with antimony oxide drops the CTI from 550 volts with no antimony oxide to 510 volts with 1% antimony and 370 volts with 2% antimony oxide.

When a single synergist is used in glass-reinforced Nylon 66, the loading level of CFR and synergist needed to achieve a UL-94 V-0 is lowered relative to the nonreinforced systems (Table 2). Instead of using 20% flame retardant with 10% antimony oxide in the nonreinforced system to obtain a V-0 at 0.4 mm, one only needs 18% of the flame retardant with 8% of antimony oxide. Using ferric oxide as the synergist, an 11% less flame retardant package is needed to obtain the same flame retardancy. The CTIs of these two formulations are only 225 volts. The highest CTIs obtained in Table 2 are when using zinc borate or zinc oxide as the synergist, 375 volts and 325 volts, but these two formulations are only V-0 at 1.6 mm.

The addition of glass fibers to the flame retardant nylons in most cases brings about a deterioration of the tracking resistance (Fig. 4). With the ferric oxide and the antimony oxide, there is a small drop in CTI, 50 volts, while in the zinc oxide case, there is a 275-volt drop. In the case of the zinc borate synergist, there is an increase of 75 volts.

TABLE 4—*FR-Nylon 6 using different synergist.*

Formulation (Weight %)	1	2	3	4
Nylon 6	73	70	75	74
CFR	18	20	20	21
Sb_2O_3	9	...	...	2.5
Zinc Borate	...	10	...	2.5
Iron Oxide	...	...	5	...
UL-94 3.2 mm	V-0	NC	V-0	V-0
1.6 mm	V-0	NC	V-0	V-0
CTI Kc (Volts)	325	NA	~275	425
Tensile Strength (MPa)	47.6	52.4	48.5	50.7

TABLE 5—*FR-Nylon 6 (25% glass-reinforced)*

Formulation (Weight %)	1	2	3
Nylon 6	47	50	45
Fiberglass	25	25	25
CFR	22	20	20
Zinc Borate	...	...	7
Sb_2O_3	6	...	3
Ferric Oxide	...	5	...
UL-94 3.2 mm	V-0	V-0	V-0
1.6 mm	V-0	V-1	V-0
CTI Kc (Volts)	250	125	350
Tensile Strength (MPa)	100.1	84.9	113.2

Table 3 gives the results of glass-reinforced Nylon 66 using mixed synergists. The combination of antimony oxide with zinc borate and CFR gives a UL-94 V-0 material at 0.4 mm and a CTI of 350 volts. Using the mixed antimony oxide/iron oxide system, one obtains a V-0 at 0.4 mm with only 13% total flame retardant loading, flame retardant plus synergist, but the CTI is only 225 volts.

In Nylon 6, zinc borate is not an effective enough synergist with the flame retardant to achieve any level of flame retardance (Table 4). But the use of either antimony oxide or iron oxide gives a UL-94 V-0 material. In the case of antimony oxide, the CTI is 325 volts while with iron oxide, the CTI is 275 volts. The use of a mixed antimony oxide/zinc borate synergist gives a UL-94 V-0 material with a CTI of 425 volts.

Table 5 gives the results of 25% glass-reinforced Nylon 6 using CFR and various synergists. A UL-94 rating of V-0 at 1.6 mm and a CTI of 350 volts can be obtained depending on the formulation.

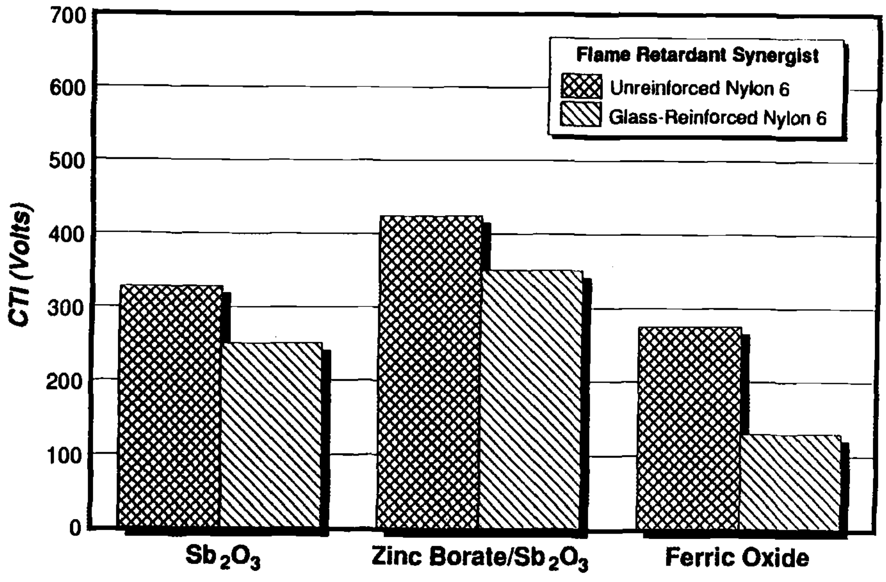

FIG. 5—*CTI for FR-Nylon 6, unreinforced vs. glass reinforced.*

TABLE 6—*FR-PBT.*

Formulation (Weight %)	1	2	3
PBT	78.9	54	55.9
Fiberglass	...	30	30
CFR	15.75	12	8
Sb_2O_3	5.25	4	6
Teflon 6C	0.1	...	0.1
UL-94 3.2 mm	V-0	V-0	V-0
1.6 mm	V-0	V-0	V-0
0.8 mm	V-2/V-0	V-1	V-0
CTI Kc (Volts)	325	225	250
Tensile Strength (MPa)	46.7	110.7	108.3

A graph of CTI value of FR-Nylon 6 is shown in Fig. 5. As in the case of FR-Nylon 66, glass reinforcement has a negative effect on CTI.

The use of CFR to flame retardant PBT is shown in Table 6. The only synergist that is effective with PBT is antimony oxide.

Figure 6 is a graph of CTIs of FR-PBT versus FR-Nylon 66. In all cases, the CTI values for FR-Nylon 66 are higher. Figure 7 is a graph of CTIs of FR-PBT versus FR-Nylon 6. In both the glass and the non-glass reinforced case, the CTI of the FR-Nylon 6 is 100 volts higher than the FR-PBT.

Table 7 gives a comparison of FR-PBT CTI values versus FR-Nylon 66.

Flame retardancy in nylons can be achieved with CFR using synergists other than antimony oxide. Nylon 66 can be flame retarded using the chlorinated flame retardant and the following synergists: antimony oxide, zinc oxide, zinc borate, or iron oxide. Mixed synergist

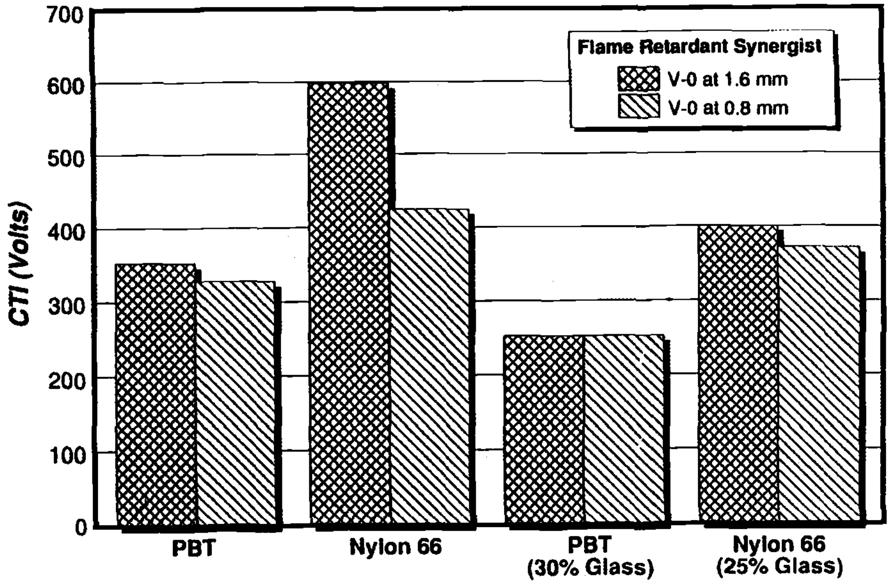

FIG. 6—*CTI of FR-PBT vs. FR-Nylon 66, unreinforced vs. glass reinforced.*

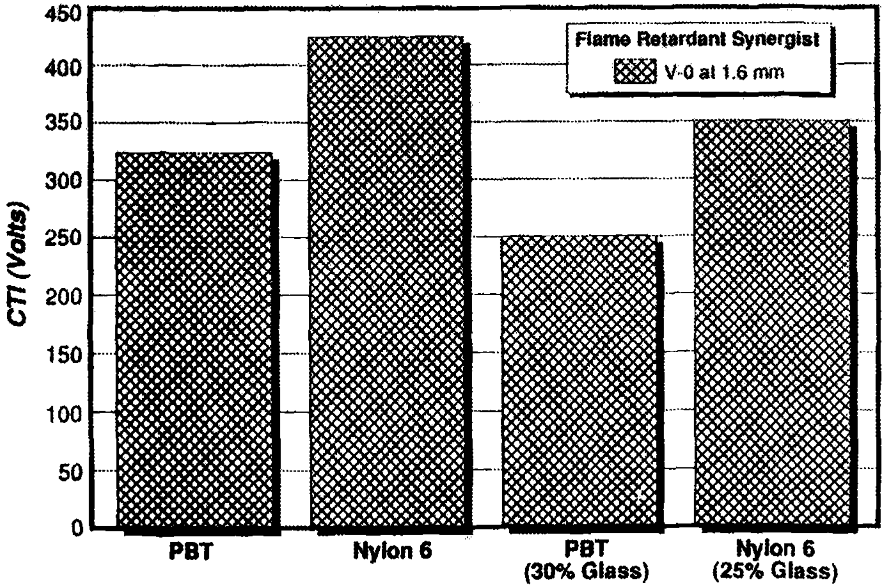

FIG. 7—*CTI of FR-PBT vs. FR-Nylon 6, unreinforced vs. glass reinforced.*

can be used that lowers the levels of flame retardants needed and also improves the electrical properties of the nylons. By the choice of the right synergists, CTI values of up to 600 volts can be obtained.

Nylon 6 can be flame retarded using the single synergist antimony oxide or ferric oxide, or using the mixed synergists:

1. Antimony oxide with zinc borate.
2. Antimony oxide with iron oxide.
3. Zinc borate with iron oxide.

The addition of glass fibers to the FR-nylons and FR-PBTs in most cases has a negative effect on the CTI.

TABLE 7—*Comparison of FR-PBT vs. FR-Nylon 66.*

	CTI of FR-PBT vs. FR-NYLON 66	
	V-0 (1.6 mm)	V-0 (0.8 mm)
FR-PBT	325 volts	325 volts
FR-PBT (30% Glass)	250	250
FR-Nylon 66	600	425
FR-Nylon 66 (25% Glass)	400	375

PBT can also be flame-retarded with CFR with the synergist antimony oxide; the other synergists are not effective. The CTI values for FR-PBTs are lower than both the FR-Nylon 66 and FR-Nylon 6.

References

[*1*] *International Electrochemical Commission Publication,* Vol. 112, 2nd edition, 1971.
[2] *DIN 53 480*
[*3*] *ASTM D-3638,* Specification for High Fire-Point Mineral Electrical Insulating Oils.

David Gardner[1] and Richard H. Whiteley[2]

Modern, Low Fire Hazard, Thin Wall Insulated Wire—A Complex Balance of Properties

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Reference: Gardner, D. and Whiteley, R. H., **"Modern, Low Fire Hazard, Thin Wall Insulated Wire—A Complex Balance of Properties,"** *Electrical Insulating Materials: International Issues, ASTM STP 1376,* M. M. Hirschler, Ed., American Society of Testing and Materials, West Conshohocken, PA, 2000.

Abstract: The complexity of electrical systems required for modern mass transport systems have been increasing over recent years. The size and weight of wire and cable installations are now important issues. The technical solution to these issues has been to move from thick and medium wall insulations to thin wall insulations. In addition, increased safety levels with respect to fire hazard properties are now demanded by the industry. Ignition resistance and reaction to fire are now considered to be of critical importance, especially for underground transit systems. The production of smoke, acid/corrosive gases and toxic fire products must all be minimized.

The functional properties of the wire, however, must not be compromised by these additional requirements of thin wall insulation and low fire hazard properties. A typical specification for such a wire therefore reflects a complex balance of properties.

This paper describes the elements of such specifications and reviews some of the test methods used to define the properties of the wire.

Keywords: acid gas, cable, cone calorimeter, corrosivity, fire, fire hazard, flame propagation, flammability, heat release, heat release rate, insulation, low fire hazard, rail, railway, reaction to fire, rolling stock, safety, smoke, thin wall, wire.

Introduction

Considerable developments have taken place during recent years in the type of wiring used in the rail transport industry. Modern railcars can contain between 20 and 50 km of wire. This represents a two- to threefold increase over the past 10 years. The reason is the growing complexity and sophistication of the electrical systems used in rolling stock.

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Three key issues arise from this growth in wire usage:

- Space for wire is at a premium
- Wire weight is negatively impacting performance and operating costs
- Combustible insulation material has increased dramatically, thus increasing the potential fire hazard

The challenge of minimizing the fire risk from the evolution of smoke and toxic or corrosive fumes is compounded by the need to also reduce heat release (which increases with more combustible material). The rail and mass transit market is therefore demanding smaller, lighter and safer wire to address these issues, while minimizing any compromise to in-service properties of the wire.

Existing systems such as air conditioning, toilet facilities, automatic doors and braking systems have become more sophisticated. New systems including computerized control and diagnostics, advanced public address, telephone, computer and even entertainment systems have increased wiring dramatically. Greater sensitivity to electromagnetic interference has also increased the use of bulkier screened cables.

External dimensions of rolling stock are fixed by track geometry and tunnel size while no extra space is available in the car for extra wiring. Conductor size is fixed by electrical requirements, so the only way to gain space is by a reduction in the wall thickness of the insulation.

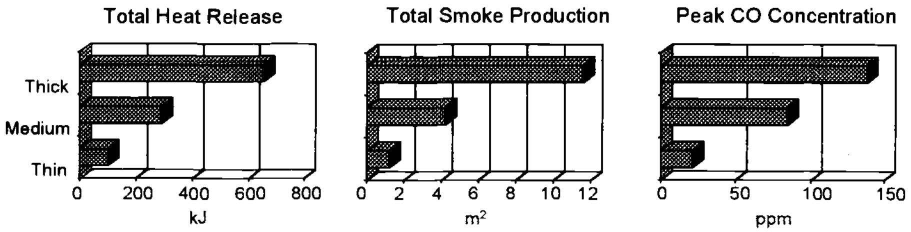
The Impact of a Thinner Wall Insulation

Combustible Material

Wall thickness can make a dramatic difference to the volume of insulation on a wire. For example, reducing wall thickness from a traditional value of 0.8 mm to 0.2 mm on a 2.2 mm diameter conductor results in an 80% decrease in insulation volume. This translates to a reduced fuel load which has the following obvious benefits during a fire: a reduction of heat released, and lower levels of smoke, toxic gases and corrosive species. Cone calorimeter measurements of the fire properties of wires having similar insulations but different wall thickness clearly illustrate this point (Figure 1) [*1*].

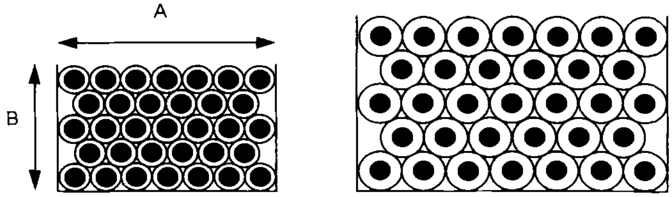
Size and Weight

Consider the case of two wiring channels each containing thirty-three wires of 1.5 mm^2 conductor and equivalent current carrying capacity (Figure 2). One channel contains thin wall wire and the other contains medium wall wire. The thin wall wire occupies an area of 136 mm^2 compared with 321 mm^2 for the medium wall wire. Alternatively, 67 thin wall wires would fit into the space occupied by the 33 medium wall wires - a 103% capacity increase that allows extra wiring to be installed. The weight reduction from 23.0 g/m to 15.2 g/m (-34%) is also very significant and can impact the total weight of wire on a rail car by several hundred kilograms. The benefit is maximized on smaller gauge wires where



Cone calorimeter data (Heat flux = 40 kW/m^2). Data are average values from three tests. All wires are halogen-free. Test specimen = 3.5 m of wire.

Figure 1 - *Cone Calorimeter Data*



	Thin wall	Medium wall
Wire Diam.	2.08 mm	3.20 mm
A	14.6 mm	22.4 mm
B	9.3 mm	14.3 mm
X-Section	136 mm^2	321 mm^2 (+ 136%)
Wire Wt.	15.2 g/m	23.0 g/m
Bundle Wt.	502 g/m	759 g/m (+ 51%)

Figure 2 - *The Effects of Wall Thickness on Size and Weight*

the insulation to conductor ratio is higher. Because of this the use of thin wall wiring in many countries has increased from a level of less than 10% of installations to more than 60%.

Who Cares About Size and Weight?

Both builders and operators are concerned about size and weight. The impact is felt on costs and performance. Total harness weight can be lowered by more than 40%, making installation easier. Conduit and cable trays can accommodate extra wire without costly size increases and higher density connectors can be used where space is limited.

Weight is a concern for both builders and operators. Builders have to meet imposed axle weight limits, and for operators weight is a cost-of-ownership issue. A 2 tonne decrease in insulation from a train significantly reduces energy consumption and also results in a lower level of infrastructure wear-and-tear. This can save millions of dollars over the life of a train fleet. This is especially true for metro systems having frequent stops and starts.

Additional Design Constraints

Given the obvious benefits associated with a reduced wall thickness it is reasonable to ask why all wires are not thin wall. This is because a number of additional factors have to be considered during wire design.

- In-service functionality of the wire
- Handling characteristics during installation
- Low fire hazard characteristics of the insulation

Wire used in rolling stock has to meet many and varied requirements. Its purpose is to carry electrical signals or power so the basic requirements demand that the dielectric properties of the insulation are good and must continue to be adequate after exposure to environmental influences such as heat, cold, moisture, fluids and mechanical handling. Furthermore the wire should be easy to handle and to strip so as to make installation as trouble-free as possible. Rail car manufacturing techniques can expose the wire to severe abuse during "pulling-in" or may result in pinching where overcrowding is evident. The issue of fire safety places additional constraints on the wire designer who must work with defined classes of materials that exclude elements generating excessive smoke or noxious gases during combustion. In many cases improvement of the insulation in one area often causes a deterioration of other properties, and therefore a complex balance of properties is required.

Test Categories

A typical wire specification will contain several different test categories. These can be divided into five main types:

- Electrical
- Physical
- Environmental
- Thermal
- Low Fire Hazard

Electrical Tests

The fundamental purpose of wiring is to carry either electrical signals or power and therefore electrical tests are essential. Conductor dimensions, weight per unit length, stranding and electrical resistance are all defined, as is insulation resistance - often at elevated temperature as well as at room temperature. Electrical withstand and/or electrical breakdown tests are also required. Some specifications require electrical tests to be carried out while the powered wire is immersed in hot salt water for several days, e.g. the CENELEC (Comitée Européen de Normalisation Electrotechnique) draft European Standard "Railway applications - Railway rolling stock cables having special fire performance: Test methods" (prEN50305:1998) has a test which runs at 300 V for 240 h at 85 °C in 3% salt solution.

Voltage withstand tests are also routinely used to check the electrical integrity of the wire after various other thermal and environmental exposures.

In the USA, the ASTM "Standard Test Methods for Hookup Wire Insulation" (D3032) is commonly referred to for many key electrical tests, whereas in Europe reference is often made to the International Electrotechnical Commission (IEC) standard, "Rubber insulated cables - Rated voltages up to and including 450/750 V - Part 2: Test methods" (IEC 60245-2).

Physical Tests

A major concern of potential users of thin wall insulated wire is that the resistance of the insulation to physical abuse may be unduly compromised and so tests such as scrape abrasion resistance, dynamic cut-through resistance and static cut-through penetration are important. The Association of American Railroads Wire and Cable Specification (AAR S 501) describes a scrape abrasion test in which a blade of defined geometry is scraped back and forth along the test specimen under a defined load and the number of cycles to cause wear through to the conductor is measured.

AAR S 501 also contains a test for static cut-through penetration in which the same blade rests on the specimen under a defined load at an elevated temperature for ten minutes and the degree of penetration through the insulation is measured.

Environmental Tests

These tests involve exposures of the wire to a variety of different liquids and also to ozone. The liquids may be oils, hydraulic fluid, brake fluid, cleaning solvents, de-icing fluid, and aqueous acid and alkali solutions. Various exposure times and temperatures are defined.

In ASTM D3032 test specimens are wrapped around a mandrel after exposure and examined both visually and electrically for failures in the insulation.

Thermal Tests

Resistance to heat and cold are both important. ASTM D3032 contains a simple cold bend test in which the wire is wound onto a mandrel at the test temperature and then is tested for electrical integrity.

ASTM D3032 also contains a more complex test which is used to determine how long the insulation will maintain its electrical integrity at elevated temperatures. Batches of wires are put in ovens at different temperatures and samples are repeatedly tested over time until failure occurs. A plot of the logarithm of the failure time versus the reciprocal of the absolute temperature is then drawn. This normally gives a straight line from which the lifetimes at particular temperatures can be obtained.

ASTM D3032 also contains a short term quality control test which is used to confirm that at a relatively high test temperature the wire is not inferior to that tested in the more complex and longer term test.

Low Fire Hazard Tests

These tests are generally of the "reaction to fire" type although there are other tests related to special circumstances associated with electrical wiring which can lead to smoke production and/or fire, e.g. tests under current overload conditions which cause abnormal heating, and tests for both wet and dry arc tracking.

Reaction to fire tests include heat release, flame propagation, smoke production, acid gas production and toxic gas production.

Heat release tests - Heat release is widely accepted by fire scientists as the single most important parameter which defines the fire hazard of a material, and the cone calorimeter has now become the instrument of choice to measure heat release on a laboratory scale. Some of the heat released during combustion is radiated back onto the fuel and thus directly affects fire growth. None of the other products of fire (smoke, toxic gases, acid gases) has this effect and this is why heat release data are so important. The ASTM "Test Method for Heat and Visible Smoke Release for Materials and Products Using an Oxygen Consumption Calorimeter" (E1354) and the International Standards Organisation (ISO) "Fire Tests - Reaction to Fire - Part 1: Rate of heat release from building products" (ISO 5660) describe the cone calorimeter test method. The ASTM "Test Method for Using a Cone Calorimeter to Determine Fire-Test-Response Characteristics of Insulating Materials Contained in Electrical or Optical Fiber Cables" (D6113) describes a test method for wire and cable products.

Flame propagation tests - The IEC "Tests on electric cables under fire conditions" (IEC 60332) tests flame propagation. "Part 3: Tests on bunched wires or cables" (IEC 60332-3) is a large scale test in which a number of cables or bundles are mounted vertically on a 3.5 m high ladder and a 20 kW propane flame is directed onto the specimen near the bottom of the ladder for twenty minutes. If cable damage extends more than 2.5 m the cable is deemed to have failed. "Part 1: Test on a single vertical insulated wire or cable" (IEC 60332-1) is a small scale test on a single vertical wire and is also a simple pass/fail test. No correlation has been found between small scale tests of this type and real fire behaviour so this test is best used for quality control purposes.

Smoke tests - The IEC "Measurement of smoke density of electric cables burning under defined conditions" (IEC 61034) tests smoke production. A one metre long horizontal specimen is exposed to the flames from one litre of burning alcohol in a 3 m x 3 m x 3 m enclosure and the attenuation of a horizontal light beam is measured.. Tests such as this in which the smoke accumulates in a closed chamber are known as static smoke tests. A smaller scale 0.51 m^3 chamber (sometimes referred to as the "NBS smoke chamber") has been used for many years and numerous test methods are based on the use of this chamber. The ASTM "Test Method for Specific Optical Density of Smoke Generated by Solid Materials" (E662) is typical of such tests in which a vertical sample is exposed to a radiant heat source of 25 $kW{\cdot}m^{-2}$ with or without flame impingement on the sample. The attenuation of a vertical light beam is measured. The ISO "Determination of optical density by a single chamber test" (ISO 5659) is a more recent development using this chamber in which a horizontal specimen is exposed to radiant heat from a truncated cone heater at 25 $kW{\cdot}m^{-2}$ or 50 $kW{\cdot}m^{-2}$, with or without a pilot ignition flame. This test has recently been adopted by the International Maritime Organisation (IMO) in its Fire Test Procedures.

The principle purpose of smoke tests such as those described above is to determine the potential obscuration of light and hence reduction in visibility that could be caused by the burning material. However, traditional ways of analysing and reporting smoke data have made it difficult to relate such measurements to the potential hazard. A recent British Standard, Guide to smoke measurement units - their basis and use in smoke opacity test methods (BS7904) gives some useful guidance.

Acid gas/corrosivity tests - Acid gas production is of concern because of the post fire damage which can be caused to electrical components from chemical corrosion. There are several tests which directly measure the corrosion of a metal target caused by combustion gases, e.g. in the ASTM "Test Methods for Heat-Shrinkable Tubing for Electrical Use" (D2671) and the ISO "Plastics - Smoke generation - Determination of the corrosivity of fire effluents" (ISO 11907), but a simpler and more popular indirect test is the IEC "Test on gases evolved during combustion of electric cables: Part 2: Determination of degree of acidity of gases evolved during the combustion of materials taken from electric cables by measuring pH and conductivity" (IEC 754-2) which entails the solution of fire gases in water and then the pH and conductivity of the resulting solution is measured.

Toxicity tests - Modern toxicity tests are based on chemical analysis of fire gases to measure the concentration of a number of known common toxic gases e.g. carbon dioxide, carbon monoxide, halogen acids, sulphur dioxide, nitrogen dioxide, nitric oxide and hydrogen cyanide. The concentration of each gas is normalised according to the relative toxicity of the gas and then the individual values are summed to give an estimate of the total toxicity. To put this information in context it is necessary to consider the size of the burning specimen, the volume into which the toxic gases are accumulated, and the likely exposure time. Typical test methods are the French Norme, "Fire tests - Analysis of pyrolysis and combustion gases - Tube furnace method" (NF X 70-100) which involves combustion of a small sample in a tube furnace and collection of the gases in inflatable bags, and the IMO Fire Test Procedure which uses the ISO 5659 smoke test to generate the gases.

Halogen content - Halogen based flame retardants are widely used as combustion inhibitors and they are very effective at suppressing ignition. However, if materials which contain these flame retardants are exposed to fire conditions which are severe or prolonged enough to cause them to burn, then corrosive and irritant halogen acids are produced together with high levels of smoke and carbon monoxide. For these reasons most rolling stock specifications require a "zero" halogen content. This is defined, for example, as chlorine plus bromine plus iodine shall be less than 0.2% and fluorine shall be less than 0.1% as measured according to clause 45 of the IEC "Specification for flexible insulating sleeving: Part 2: Methods of test" (IEC 60684-2).

Design Options

Modern thin wall wires have evolved into two types, both of which are halogen free.

Type 1 - First Generation Thin Wall Wires

Engineering polymers such as polyimides or polyetherketone (PEEK) were adopted to provide the toughness needed for thin wall wires. These inherently flame retarded materials are good insulators and at first sight appear ideal. However, used alone they are costly and produce a stiff wire that can be hard to handle. In an attempt to combat these drawbacks dual wall wires comprising PEEK extruded in a thin layer over a cheaper polyolefin layer have been produced. This improves the flexibility to some extent, but the mismatch in modulus between the inner and outer walls often results in wrinkling when the wire is bent. Wrinkles act as a stress point for premature failure in service so the use of such systems has been limited.

Type 2 - Today's Modern Thin Wall Wire

The best balance of fire safety and in-service properties has been achieved by compounding inherently flame retarded compounds together with halogen-free flame additives at lower levels. These products are dual wall to optimise the properties of each layer and are specially formulated for improved flexibility. They also meet the demands of the most rigorous fire hazard specifications, e.g. the French Norme, "Railway rolling stock - Halogen free, electrical conductors and cables with thin insulation and protective layers" (NF F 63 808), while retaining toughness, good handling characteristics and economical manufacture.

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Marcelo M. Hirschler[1]

Fire Testing of Electrical Materials

Reference: Hirschler, M. M., "**Fire Testing of Electrical Materials,**" *Electrical Insulating Materials: International Issues, ASTM STP 1376,* M. M. Hirschler, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

Abstract

In recent years electrical wire or cable insulation has been, once more, identified by NFPA statistics as a major material first ignited in residential fires (representing 7.6% of fires and 3.9% of fire fatalities in 1991-95) and the cause of 13% of catastrophic fires (1993-96). This highlights the need for renewed emphasis on fire testing of wires, cables and electrical materials. Cable fire tests can be subdivided into 5 categories:

(a) Old fashioned small scale tests, which generally address only ignitability or flame spread, but the results of which are rarely meaningful in terms of real fire performance;

(b) Vertical cable tray tests, of which there are a large variety, ranging in heat input from 20 kW up to 154 kW (in the case of riser cables), which address flame spread, and sometimes also smoke and heat release;

(c) The Steiner tunnel NFPA 262 (UL 910) test, which determines wind aided horizontal flame spread, and smoke release, under a very high heat input (ca. 90 kW), with a relatively small mass load of cables; and

(d) Small scale cable tests, often originally designed for materials, directed at measuring fundamental fire properties, such as heat release or critical fluxes for ignition or flame spread and thermal heating properties.

(e) Tests for other cable fire properties, mainly smoke (obscuration, toxicity, corrosivity) and circuit integrity.

Keywords: cables, electrical materials, fire, fire hazard, fire test, flame spread, heat release, insulation, jacket, smoke obscuration

Introduction

In the 1950's and early 1960's, in the USA, there were several fires with serious losses propagated by electrical cables. A 1966 NFPA "fire hazard study" *[1]* described

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24 such fires, the most serious at the Philadelphia Electric Co. Atomic Power Station, Peach Bottom, Florida. The study concluded that the cable fire tests available at the time (involving single cables only) were inappropriate. They specifically mentioned the lack of radiation to and from adjacent burning conductors, and recommended cables be wrapped with asbestos tape and painted with sodium silicate solution. Alternatively, NFPA suggested active fire protection measures: smoke detectors or automatic sprinklers. They also suggested putting cables in non combustible rooms, without other combustibles. The most important aspect of the NFPA study, however, is that it pointed out, probably for the first time, that "grouped" cables can spread flame much faster than single cables. Therefore, even if single cables appear adequately fire retarded, they may not be safe when put in groups, due, at least partly, to radiation effects. Concerns about flammability of cables in cable trays became an issue in Europe following the June 1967 cable fire at the La Spezia (Italy) substation of the Italian utility ENEL, which caused serious damage *[2]*. Electrical utility cable fires also occurred in other countries, including the UK (Battersea), Germany (Pleinting), Sweden (Gothenborg), Switzerland (Mühleberg), Finland and Japan (Tokai Mura). The most notable European utility cable fire was probably the 1979 fire at the British Central Electricity Generating Board (CEGB) in Tilbury. There have been only a few prominent major electrical cable fires since the 1970's in the US; interestingly, 3 of those few are telephone central offices.

None of the fire tests in use in the 1960's were sufficiently adequate to predict fire hazard in a real full scale fire environment. This is not surprising, however, since fire testing had still not developed as a science. Thus, most fire tests tended to be quite generic and had not been developed with a specific fire safety objective in mind.

Initial Fire Tests for Cables

The earliest approach, at committee ASTM D9 (Electrical and Electronic Insulating Materials), to develop fire test requirements for electrical cables was to issue "omnibus" test standards for individual materials or wires, involving a variety of test methods: ASTM D 229[2], ASTM D 350, ASTM D 470 and ASTM D 876 (first issued between 1925 and 1946). All these tests are intended to assess the properties of the plastic materials used as coatings. All the tests also involve small scale specimens and low intensity simple laboratory burners, and are intended to address individual types of materials. These standards are designed primarily to give a degree of quality assurance in terms of physical and electrical properties rather than to ensure fire safety. Subsequent omnibus tests have also been designed and added to the standards lists, including ASTM D 1000, ASTM D 2633, ASTM D 2671 and ASTM D 3032. ASTM D9 did not develop an individual fire test until 1988: ASTM D 3874, for hot wire ignition, based on the international standard.

At around that time, D9 formed subcommittee D9.21, specifically dedicated to developing fire standards, for electrotechnical products. In less than 10 years, the subcommittee generated 5 standards, including 4 test methods: ASTM D 5424 (vertical cable tray test for measuring smoke), ASTM D 5537 (vertical cable tray test for

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[2] The bibliography fully describes all standards discussed in the text or tables.

measuring heat release), ASTM D 5485 (Cone Corrosimeter: application of the cone calorimeter to assessing corrosion due to electrical cables or plaques), ASTM D 6113 (Cone Calorimeter Application Standard, to electrical cables or plaques) and ASTM D 5425 (Fire Hazard Assessment Guide). In the meantime, ASTM D20, committee on Plastics, created from committee D9 in 1937, produced generic fire standards for plastic plaques. The objective of the D9 split was that D20 could generate standards of broader interest, for materials, than just the electrical world. Moreover, another ASTM committee, E5, on fire standards, is responsible for development, of fire test standards intended to measure the response of materials, products and assemblies (i.e. not only materials and not only electrical) to heat and/or flame. Finally, it is worth mentioning that committees also exist dealing with individual occupancies, such as aircraft or ships. Thus F7, committee on aircraft, developed Federal Aviation Administration, FAA, mandated electrical materials tests, e.g. the 60° angle test, ASTM F 777. Table 1 shows ASTM tests that are either fire tests or are omnibus tests with a fire test included, applicable to the electrical materials industry.

Table 2 contains a description of small scale tests; it becomes clear from the Table that many of the tests are very similar. In particular some of them are versions of the famous UL 94 series of tests for plastic plaques. An effort has been underway, at ASTM, IEC and ISO, to coordinate the profusion of these standards, administered by different bodies. Thus, ASTM D 635, ASTM D 3801, ASTM D 4804 and ASTM D 5048, like UL 94, are all basic flammability/dripping tests, ASTM D 5025 is a specification of the corresponding burner (of the Bunsen or Tirrill type) and ASTM D 5207 describes how to calibrate the burner. There have been projects to develop more repeatable burners, with work still in progress.

Internationally, responsibility is divided in a similar fashion to the way it is within ASTM. IEC addresses the electrical world while ISO is responsible for both plastics and building product fire standards. The committee responsible for the technical input to IEC in the US is ASTM D9, with subcommittee D9.89 the TAG (Technical Advisory Group) for IEC TC89 (developing material and small scale test methods and guidance documents), and subcommittee D9.20 the TAG for IEC SC20C (supervising cable fire tests: IEC 60331, IEC 60332, IEC 60754 and IEC 61034). The TAG for ISO TC61 (committee on plastics) lies within ASTM D20, and that for ISO TC92 (committee on fire and building products) within ASTM E5.

ISO has three material fire standards relevant to the electrical industry: ISO 1210 (for horizontal and vertical plastics flammability, with a 20 mm flame; combining ASTM D 635 and ASTM D 3801, i.e UL 94 HB and V-0 tests), ISO 9773 (the corresponding vertical test for flexible plastics; ASTM D 4804) and ISO 10351 (the test for the 125 mm flame; i.e. UL 94 5V or ASTM D 5048). ISO 1210 and ISO 10351 have been withdrawn now and the subjects are covered in IEC 60695-11-10 and IEC 60695-11-20. All the tests use the premixed burner specified in ASTM D 5025, which is also one of the standard sources of ignition described in ISO 10093 (P/PF2). Table 3 describes these tests, as well as some other generic (ISO TC92) tests useable for electrical materials or products, while Table 4 shows IEC standards.

Tests corresponding to the ISO ones also exist at IEC. The new proposed set of standards will be: IEC 60695-11-3 and IEC 60695-11-4, representing the specification and calibration of the 500 W and 50 W premixed burners respectively (even if the same

Table 1. ASTM Fire Tests Relevant to Electrical Materials

Designation	Properties	Committee	Application
D 229	Flaming Duration Flame Resistance	D9	Insulation Materials (Plaques)
D 350	Ignition Flame Spread (2 tests)	D9	Sleeving for Insulation (on wire or as sleeving)
D 470	Horizontal Flammability	D9	Crosslinked (Cable) Insulations & Jackets
D 635	Horizontal Flammability	D20	Plastic Materials (Plaque)
D 876	Ignition Flame Spread	D9	Non Rigid PVC (wire) Tubing for Insulation
D 1000	Burning Time	D9	Adhesive Tapes
D 1929	Ignition Temperature	D20	Plastic Materials
D 1989	Calorific Value	D5	All Materials
D 2633	Vertical Flammability	D9	Thermoplastic (wire) Insulations & Jackets
D 2671	Ignition Flame Spread	D9	Heat Shrinkable Tubing
D 2843	Smoke Obscuration (Rohm & Haas)	D20	Plastics (Plaques)
D 2863	Oxygen Index	D20	Plastics
D 3014	Flame Height Burning Time	D20	Thermoset Cellular Plastics
D 3032	Flame Spread Vertical	D9	Hookup Wire Insulation (on Wire)
D 3286	Calorific Value	D5	All Materials
D 3713	Vertical Ignition Response Index	D20	Solid Plastics

Table 1 (continued)

Designation	Properties	Committee	Application
D 3801	Vertical Flammab.	D20	Solid Plastics
D 3874	Hot Wire Ignition	D9	Insulating Materials
D 4100	Gravimetric Smoke (Arapahoe)	D20	Plastics
D 4804	Flammability (Vertical & Horizontal)	D20	Non Rigid Plastics
D 5025	Small UL 94 Burner Specification	D20	Plastics, Cables
D 5048	UL 94 5 V Burn Through	D20	Plastics
D 5207	Small UL 94 Burner Calibration	D20	Plastics
D 5424	Vertical Cable Tray Smoke Obscuration	D9	Cables - 20 kW
D 5485	Smoke Corrosivity (Cone Calorimeter)	D9	Electrical Materials, Cables
D 5537	Vertical Cable Tray Heat Release & Flame Spread	D9	Cables - 20 kW
D 6113	Cone Calorimeter (Electrical Applications; see ASTM E1354)	D9	Electrical Materials & Cables
E 136	Non Combustibility	E5	All Materials
E 662	Smoke Obscuration (NBS Chamber)	E5	All Materials
E 906	Heat & Smoke Release (OSU Calorimeter)	E5	All Materials
E 1354	Heat & Smoke Release (Cone Calorimeter)	E5	All Materials
F 777	Flammability @ 60° for FAA	D9	Insulated Wire & Cable (on Wire)

Table 2. Details of ASTM Small Scale Electrical Material Fire Tests

Number	Property	Sect.	Burner	Intens.	Orient.	Specimen	Measures
D 229	Flaming Duration	61-66	Tirrill Burner	50 W 20 mm	Vert.	5"x0.5" plaque	Flaming, dripping
D 229	Flame Resistance	67-75	Arc from NiCr Coil	55 A 120 s	Horiz.	10"x0.5"x0.5" plaque	Ignition & Burning Times
D 350	Flammability	22 - D876	Lab Burner	500 W 125 mm	70° angle	22" long wire	Burning time, lgth
D 350	Burning Rate	23-28	Bunsen Burner	Undefined	Vert.	> 1" long sleeving	Burning Time
D 470	Flammability	91-95	Tirrill Burner	500 W 125 mm	Horiz.	6" long Cable	Burning Time
D 635	Flammability	All	Lab Burner	50 W 20 mm	Horiz.	5"x0.5" plaque	Burning Rate
D 876	Flammability	14-20	Lab Burner	500 W 125 mm	70° angle	22" long wire	Burning time, lgth
D 1000	Burning Time	104-109	Bunsen Burner	500 W 125 mm	Wound around	0.75"x 15" tape	Burning Time
D 1929	Ignition Temp.	All	Furnace Assembly	400 - 750°C	Small pieces	3 g	Ign. Temp.
D 2633	Flammability	38	Tirrill Burner	500 W - 125 mm	Vert.	6" long wire	Flame propag.
D 2671	Flammability	68-69 70	Lab Burner	500 W 125 mm	70° angle	22" long wire	Burning time, lgth
D 2671	Flame Travel	68-69 71	Lab Burner	500 W 125 mm	Vert.	18" long tubing	Flame lgth
D 2671	Flame Travel	68-69 72	Lab Burner	500 W 125 mm	Vert. (Difer.)	18" long tubing	Flame lgth

Table 2 (continued)

Number	Property	Sect.	Burner	Intens.	Orient.	Specimen	Measures
D 3014	Flame Height	All	Lab Burner	960°C	Vert.	10"x0.75"x 0.75"	Flame Height
D 3032	Flame Spread	18	Tirrill Burner	500 W 125 mm	Vert.	21" long wire	Burn time, lgth
D 3713	Ignitability	All	Lab Burner	50 W 20 mm	Vert.	5"x0.5" plaque	Flaming, dripping
D 3801	Flammability	All	Lab Burner	50 W 20 mm	Vert.	5"x0.5" plaque	Burning time
D 3874	Hot Wire Ignition	All	NiCr Hot Wire	6.5 W/in	Wrap around	5"x0.5" plaque	Time to ignition
D 4804	Flammability	1-5 6-13	Lab Burner	50 W 20 mm	Vert.	8"x2" plaque	Burning time
D 4804	Flammability	1-5 14-19	Lab Burner	50 W 20 mm	Horiz.	5"x0.5" plaque	Burning time
D 5025	Burner Spec.	All	Lab Burner	50 W & 500 W	-	-	-
D 5048	UL 94 5V Burn Through	10.1	Tirrill or Lab Burner	500 W 125 mm	Vert.	5"x0.5" plaque	Burn through time
D 5048	UL 94 5V Burn Through	10.2	Tirrill or Lab Burner	500 W 125 mm	Horiz.	6"x6" plaque	Burn through time
D 5207	UL 94 Burner Calibr.	All	Lab Burner	50 W & 500 W	-	-	Calibr.
F 777	Flammability	All	Lab Burner	76 mm	60° angle	30" wire	Burn time, lgth

Table 3. International (ISO) Standard Test Methods

Number	Property	Burner	Intensity	Orient.	Specimen	Measures	ASTM Eq.
ISO 871	Ignition Temp.	Furnace Assembly	400-750°C	Small pieces	3 g	Ign. temp.	D1929
ISO 1182	Non combust.					Classif.	E136
ISO 1210 (WD)	Flammab., Ignition	Lab Burner	50 W 20 mm	Horiz. & Vert.	Plaques	Burning Rate	D635 + D3801
ISO 1716	Calorific Value	Oxygen Bomb				Calorific Value	D3286
ISO 4589-2	Oxygen Index	Special Apparatus	O_2 - N_2 mix	Vert.	Plaques	LOI	D2863
ISO 4589-3	Temper. Index	Special Apparatus	O_2 - N_2 mix	Vert.	Plaques	LOI-TI	None
ISO 5657	Ignitab.	Cone heater	Variable	Horiz.	Plaques	Ign. Flux	None
ISO 5659-2	Smoke Obscur.	Closed Chamber	25, 50 kW/m^2	Horiz.	Plaques	Smoke	None (in draft)
ISO 5660-1	Heat & Ign.	Cone Calorim.	10-100 kW/m^2	Horiz.	Plaques	Heat, ign.	E1354
ISO TR 5924	Smoke obscur.	Dual chamber	50 kW/m^2	Horiz.	Plaques	Smoke	None
ISO 9773	Flammab., Ignition	Lab Burner	50 W 20 mm	Vert.	Plaques (Flexible)	Burning Rate	D4804
ISO 10093	Standard Ignitions	Spec for Burners	Includes Tirrill				None (ASTM D5025)
ISO 10351 (WD)	Flammab., Ignition	Lab Burner	500 W 125 mm	Horiz. & Vert.	Plaques	Burning Rate	D5048

Table 4. International (IEC) Standards

Number	Property	Burner/Intensity	Orientation	ASTM Equiv.
IEC 60331-1	Circuit Integrity	Special apparatus	Horiz.	None
IEC 60332-1	Flammability	1 kW Burner	Vert./1 wire	None
IEC 60332-2	Flammability	500 W Burner	Vert./1 thin wire	D3032
IEC 60332-3	Flame Spread	20 kW Burner	Cable Tray	None
IEC 60695-1	Fire hazard assessment guidance			D5425
IEC 60695-2-1	Ignition (Flamm.)	Glow Wire	Horiz.	None (in draft)
IEC 60695-2-2	Ignition (Flamm.)	Needle Flame	Vert.	None
IEC 60695-2-4	Flammability	Lab Burner 50 W & 500 W	Both	None
IEC 60695-3	Fire hazard assessment guidance on properties			None
IEC 60695-4	Terminology			E176
IEC 60695-5	Smoke corrosivity guidance			
IEC 60695-6-30	Smoke	Smoke Chamber	Vert.	E662
IEC 60695-7	Smoke Toxicity Guidance			
IEC 60695-8	Heat Release Guidance			
IEC 60695-9	Flame Spread Guidance			
IEC 60695-10	Abnormal Heat	Ball Pressure		None
IEC 60695-11-10	Flammability	Lab Burner 50 W (UL 94 HB, V-0)	Horiz. & Vert.	D635 + D3801
IEC 60695-11-20	Flammability	500 W Burner (UL 94 5V)	Horiz. & Vert.	D5048
IEC 60695-11-3 Draft	Burner Spec.	500 W Burner	Horiz. & Vert.	None
IEC 60695-11-4 Draft	Burner Spec.	50 W Burner	Horiz. & Vert.	None
IEC 60695-11-30 Draft	Flammability	History UL 94	Horiz. & Vert.	None
IEC 60707	Flammability	UL 94 Classes	Vert. & Horiz.	None
IEC 60754-1	Halogen Acid Gas	Tube Furnace	Pieces	None
IEC 60754-2	pH & Conductiv.	Tube Furnace	Pieces	None
IEC 61034	Smoke obsc.	3 m cube	Horiz./ cable tray	None

burner can be used for both 50 W and 500 W tests). They are likely to differ from the ASTM and ISO burner specifications, because there are more options. IEC 60695-11-10 is the test method corresponding to UL 94 HB & V-0 (or ISO 1210, or ASTM D 635 and ASTM D 3801) and IEC 60695-11-20 is the one corresponding to UL 94 5-V (or to ISO 10351 or ASTM D 5048). A joint ISO/IEC task group has successfully combined ISO 1210 and ISO 10351 with the pertinent parts of IEC 60707 to create IEC 60695-11-10 and IEC 60695-11-20, with responsibility being assigned to IEC. IEC 60707 includes the classification system, being harmonized with the UL one.

The IEC 60695 series of standards are mostly guidance documents, except for ignition tests (IEC 60695-2-1: glow wire, with four parts, addressing the apparatus and test procedure, a test method for end-products and a test method for materials, and IEC 60695-2-2: needle flame) and a smoke obscuration test (the NBS smoke chamber; IEC 60695-6-30, apparatus, and IEC 60695-6-31, materials). Some of these guidance documents will be described to a small extent later on in this paper.

IEC also has a number of cable fire tests. They are: IEC 60331, for circuit integrity/fire resistance of cables (with no US equivalent), IEC 60332-1, for single cable flammability (1 kW flame applied onto a 600 mm long cable), IEC 60332-2, also for single cable flammability (500 W flame applied to a "thin" cable, 600 mm long), IEC 60332-3, for flame spread of bunched cables in trays (20 kW, or 70,000 BTU/h vertical 3.5 m cable tray), IEC 60754-1, for halogen acid gases emitted from cable materials (with a tube furnace), IEC 60754-2, for pH and conductivity of solutions of cable material emissions (also using a tube furnace) and IEC 61034, for smoke obscuration of bunched cables in a horizontal tray (using the 3 m cube).

UL and CSA also have cable and electrical material fire tests, most of them used for certification and regulation. The most notable small scale materials test is, of course, UL 94, with HB, V-0 and 5V parts. The Canadian equivalent, CSA C22.2 No. 0.17, has the same parts. UL 1581-1080, contains the UL VW1 cable test, a vertical test with a 500 W flame, on a single cable, similar (but not identical) to IEC 60332-2 and to CAN CSA FT1. A similar test is described in UL 1581-1060. Both UL and CSA also have a horizontal 500 W test (UL 1581-1100 and CSA FT2) and CSA has a test for portable cables (FT5, mostly for mining applications), which includes a 500 W flame application. It is essential to emphasize that the UL, CSA and IEC small scale cable tests are <u>not</u> identical, in spite of what superficial descriptions suggest; there is substantial interest in retaining differences by some affected parties. The medium or large scale tests at both UL and CSA are: UL 1581-1160 (vertical cable tray test, 2.4 m high sample, ca. 20 kW-70,000 BTU/h flame), CSA FT4 or UL 1581-1164 (vertical cable tray test, differing in that the burner is at a 20° angle and the cable loading is tougher), UL 1666 (riser cable test, 145 kW, 495,000 BTU/h, in a two story facility) and NFPA 262/CSA FT6 (plenum cable test, in the Steiner tunnel, horizontal ceiling 25 ft cable tray, ca. 90 kW, 300,000 BTU/h, measuring flame spread and smoke release, for communications cables).

Some military cable specifications exist, most based on standard tests; one notable exception is MIL-C-915, pt. 18 (measuring the ignitability of a cable to a spark plug). Also, British Naval Engineering Standards (NES series 700) are often called for in specifications for wires or cables. They include the oxygen index test, the temperature index, smoke obscuration and the famous smoke toxicity test (NES 713), which is run in a closed chamber, with a small burner, and is not met by halogenated materials.

Internationally, it is also worth mentioning that, in 1967, the year of the La Spezia plant fire, and as its consequence, an Italian cable standard (CEI 20-22) was issued to assess the fire performance of cables in trays. The test could be performed at only one institution, Italian Experimental Electrotechnical Center (CESI), in Milan. In most cases, the response by both cable manufacturers and users was to commission testing at CESI and subsequently refuse to accept cables for use in power plants if they did not pass the CESI test. A further response, after the 1979 UK utility fire mentioned, was to develop an international cable tray test, which eventually became IEC 60332-3, and to conduct UK studies to check for correlation between the two tests. The studies were conducted at the Central Electricity Research Laboratory *[3]* and at Queen Mary & Westfield College, University of London *[4]*, and were successful.

Grouped Cable Fire Testing

Having described traditional laboratory scale tests, both on materials and on burners, the fact remains, as explained in above, that testing of individual cables cannot predict the potential fire hazard inherent in the use of grouped (or bunched) cables. This is critical, since cables are only rarely isolated from other cables, but rather normally present in trays or just simply laid out in concealed spaces (such as air handling spaces, overheads, underfloors, computer rooms, cable cabinets, etc.). This issue becomes a more severe problem as a facility becomes older, since excess cables, once installed, are rarely removed ("mined") when replaced, and serve as an additional source of combustible mass. This is the reason why fire testing of grouped cables has now been shown to be essential.

This resulted in the development of all the now common vertical cable tray tests. The first one is the most famous one: IEEE 383, later to become UL 1581-1160, first standardized in 1974, using the 70,000 BTU/h (20.3 kW) gas ribbon burner proposed by McAvoy *[5]*. Its development was reviewed in a 1977 symposium. Work also took place, simultaneously and in parallel, in Canada, particularly at Ontario Hydro (e.g. Stonkus, 1972 *[6]*). The test adopted by the Canadian Standards Association (CSA) (CSA FT4) was based on the one issued by Ontario Hydro (OH L-891SM) and not on the one issued by IEEE. Therefore, the Canadian vertical tray test has always been different from IEEE 383 (more details later). The IEEE 383 test requires, basically, that cables don't burn up completely under the exposure conditions (20 min, at 70,000 BTU/h; actual requirements are a char length of less than 8 ft (ca. 2.44 m)). IEEE also then came out with its own version of the CSA FT4 test, which it standardized as IEEE 1202. The IEEE 383 test became the first tray cable test adopted as a North American standard for measuring flammability of grouped cables. Specifications for shipboard cable and nuclear power industry power cable also refer to the IEEE 383 test for flame propagation. Table 5 presents the differences between the various standard vertical cable tray tests and Table 6 shows the magnitudes that are actually measured in each test (and the criteria, if applicable). There are some aspects that are worth highlighting, though, as follows.

The Canadian FT4 standard differs from UL 1581-1160 in a few main aspects, as follows (which is the reason that it eventually became a part of UL 1581: section 1164):

Table 5. Comparison of Some Features of Vertical Cable Tray Tests

	IEEE 383 UL 1581-1160	ICEA T-29-520	CSA FT4	IEEE 1202	UL 1685/ UL1581[a]	UL 1685/ CSAFT4[b]	IEC 60332-3	CEI 20.22 Pt 2
Burner power (kW) (ca.)	21	62	20	20	21	21	20	30[c]
Time of flame (min)	20	20	20	20	20	20	20, 40 [d]	60
Alternate source	Yes, oily rag [e]	no	no	no	no	no	no	no
Burner placement[f]	600 mm [g] 75 mm in back	300 mm 200 mm in back	300 mm 75 mm in front	300 mm 75 mm in front	457 mm 75 mm in back	457 mm 75 mm in front	600 mm 75 mm in front	200 mm 50 mm[h] front/back
Angle of burner	horiz.	horiz.	20° up	20° up	horiz.	20° up	horiz.	horiz.
Tray length (m)	2.4	2.4	3.0	2.4	2.4	2.4	3.5	4.5
Tray width (m)	0.3	0.3	0.3	0.3	0.3	0.3	0.5	0.5
Sample length (m)	2.4	2.4	2.3	2.3	2.4	2.4	3.5 m	4.5
Width of tray used for cables (m)	0.15 front only	0.15	0.25 front only	full front only	0.15 front only	full front only	0.30 front or front + back [i]	0.20 front + back
Thin-size cables to be bundled	no	no	if D < 13 mm	if D < 13 mm	no	if D < 13 mm	mounted flush, with no spaces	mounted flush, with no spaces
Test enclosure specified	no	no	yes	yes	yes	yes	yes	yes
Required air flow rate	N.A.	N.A.	> 0.17 m^3/s	0.65 m^3/s	5 m^3/s	5 m^3/s	0.08 m^3/s	10 m^3/s
Test runs needed	3	2	2	2 x 2 [j]	1	1	1	1
Max. char length (m, from bottom)	2.4	2.4	1.786 [k]	1.786 [k]	2.4	1.786 [k]	3.1	4.1
Peak smoke release rate ($m^2 s^{-1}$)	N.A.	N.A.	N.A.	N.A.	0.25	0.40	N.A.	N.A.
Tot. smoke released (m^2)	N.A.	N.A.	N.A.	N.A.	95	150	N.A.	N.A.

a Version run with UL 1581 exposure. Equivalent to ASTM D5424/D5537, except ASTM has no failure criteria.
b Version with CSA FT4 exposure. Equivalent to ASTM D5424/D5537, except ASTM has no failure criteria.
c Electrical oven, two radiant plates facing cables (500 x 500 mm)
d Time is 20 min for Category C, 40 min for Categories A and B.
e Valid only for the IEEE 383 and not the UL 1581 version.
f Height above bottom, followed by distance from specimen surface.
g This dimension is 457 mm in the UL 1581 version.
h Minimum distance from cable surface.
i Depends on amount of cable loading.
j Two each on two different sizes of specimens.
k Char length of 1.5 m is measured from horizontal height line of burner.

Table 6. Measurements Required in Cable Tray Tests

	Char Length	RHR	RSR	TSR	Mass Loss
IEEE 383 UL 1581-1160	Mandatory	No	No	No	Optional
CSA FT4	Mandatory	No	No	No	Optional
IEEE 1202	Mandatory	No	No	No	Optional
UL 1685 (UL 1581)	Mandatory	Optional	Mandatory	Mandatory	Optional
UL 1685 (CSA FT4)	Mandatory	Optional	Mandatory	Mandatory	Optional
IEC 60332-3	Mandatory	No	No	No	Optional
ASTM D 5424	Optional	Optional	Mandatory	Mandatory	Optional
ASTM D 5537	Mandatory	Mandatory	Optional	Optional	Mandatory
ICEA T-29-520	Mandatory	No	No	No	Optional
CEI 20-22 Pt 2	Mandatory	No	No	No	Optional

Table 7. Cable Loading & Flame Application in IEC 60332-3

Cable Category	Packing Density	Burner Flame Application
	L of combustion products per m of tray	min
A	7.0	40
B	3.5	40
C	1.5	20

- The burner is at an angle of 20° from the horizontal, rather than vertical, and it is sited 1 ft from the floor rather than 18 in from the floor;
- The burner is sited in front of cable tray rather than at the back;
- Cable loadings are different, the CSA FT4 test having significantly more cable, particularly for smaller diameters;
- Tray length is 3 m (10 ft) and not 8 ft (2.4 m); minimum cable length is 2.3 m;
- Failure criterion is a char length of 1.5 m, rather than 8 ft (2.4 m);
- In summary, the CSA FT4 test is substantially more severe than UL 1581-1160.

Internationally, the IEC 60332-3 standard is substantially similar to the UL 1581 test, but it has some significant differences. The differences include:

- The cable tray is mounted against a wall, rather than free standing, so that mass loss cannot be measured continuously;
- The tray and cable lengths are both 3.5 m;
- The cable loading and flame duration (20 or 40 min) depends on the category of cables to be tested, with unspecified category criteria (see Table 7);
- The burner, horizontal, is at a height of 2 ft from the floor;
- Failure criterion is char length of 2.50 m from the bottom edge of the burner;
- The air inlet and outlet are not symmetrical within the chamber.
- In summary, the IEC 60332-3 test is less severe than the CSA FT4 test.

Several studies have compared the severity of the various cable tray tests. The ones worth mentioning include: Coaker et al. *[7-12]* and Barnes et al. *[13-14]*. In 1991, an ad hoc group of cable manufacturers and (mainly) cable materials manufacturers worked with UL to develop a standard "limited smoke" (/LS) marking for cables. The resulting standard, UL 1685, had two versions: UL 1581-1160 and CSA FT4. The test included pass/fail criteria, which required the char length criterion of the original test (i.e. 8 ft for UL 1581-1160 and 1.5 m for CSA FT4), as well as requirements for maximum rate of smoke release (Pk RSR) and total smoke release (TSR) (Table 6). It must be pointed out that UL 1685 Pk RSR and TSR criteria are based on optical density rather than the more technically correct extinction coëfficient (differing by a factor of 2.3). The corresponding pass-fail criteria for CSA FT4 thus become, by simple arithmetic: TSR $\leq$ 345 m^2 (compared to $\leq$ 150 m^2 in UL 1685) and Pk RSR $\leq$ 0.92 m^2/s (versus $\leq$ 0.40 m^2/s in UL 1685) and are used in that way in the corresponding CSA requirements, where cable are marked FT4-ST1 (flame tested level 4, smoke tested level 1).

UL standards are often written in a condensed way, and this can provide insufficient information for a laboratory to build the equipment, conduct the test properly, and ensure it obtains results comparable to those from UL. Thus, ASTM committee D9 developed a pair of standards based on UL 1685, with all the needed information: they are ASTM D5424 (with smoke obscuration as the mandatory measurement, and heat release, mass loss, toxic gases and char length as optional measurements) and ASTM D5537 (with heat release, mass loss and char length as requirements and smoke and toxic gas release as optional measurements). Both standards can be satisfied with a single test (or burn) and can be conducted in 2 options: UL 1581-1160 or CSA FT4. Thus, ASTM D5537 is the only large scale cable fire test requiring heat release rate measurements.

Another tray cable test worth mentioning has been extensively used in the US and that is considerably more stringent than the UL 1581: ICEA T-29-520. It is virtually identical to the UL 1581 test, except that the burner input is 210,000 BTU/hr (ca. 62 kW).

Vertical cable tray tests are not the only ones addressing fire performance of grouped cables. There are 2 other tests: the riser and the plenum cable fire tests. Riser cables have the second most severe requirement, they must pass ANSI/UL 1666. Cables are mounted in a vertical tray arrangement, within a 19 ft high concrete shaft divided into two compartments at the 12 ft level, with a 1 ft x 2 ft opening between the compartments. The ignition source is a gas flame of 495,000 BTU/h (145 kW), on for 30 min. Cables pass the test if there is no "flame" at the top of the bottom compartment during the test; char length and smoke obscuration, mass loss or heat release are not measured. Results are based on flame height, although some temperatures are also measured. This test is considerably less severe than the plenum cable test, although the incident heat is much higher. Plenum cables must pass the severest cable fire test: NFPA 262 (also known as UL 910). It involves loading a horizontal 25 ft long, 1 ft wide tunnel (Steiner Tunnel) with cables and exposing them to a 300,000 BTU/h (87.9 kW) gas flame for 20 min, under an 240 ft/min air flow rate. In order to "pass", cables need to spread flame a distance of less than 5 ft, beyond the gas flame itself, have a peak smoke optical density not exceeding 0.5 and an average optical density not exceeding 0.15, both measured in the exhaust duct. Such cables are then described as "having adequate fire-resistant and low-smoke-producing characteristics" for use in "ducts, plenums and other environmental air-handling spaces", or in any "compartment or chamber to which one or more air ducts are connected and which forms part of the air distribution systems". A plenum is an area located above false ceilings and where the heating, ventilating or air conditioning ducts are located, as well as communications cables and other utilities. Details of the history and controversies surrounding this test have been discussed elsewhere *[15-20]*.

Small Scale Heat Release Tests

It is now clear that heat release is the single most important fire property, since its peak value will determine the peak intensity of a fire *[21-25]*. The fire science community has understood this concept only somewhat recently. The exponential growth in the interest in the use of heat release rate happened when it became possible to measure this magnitude directly and accurately. The main small scale calorimeter tests measuring heat release *[26]* are the cone (ASTM E 1354) *[27]*, the OSU (ASTM E 906) *[28]* and the Factory Mutual apparatus *[29-32]*. The cone calorimeter is the instrument most recently developed of them; thus, most cone work on cables has been relatively recent. On the other hand, work on cables using both older instruments started in the mid 1970's.

E.E. Smith and co-workers published several papers *[33-37]* showing how the OSU apparatus can be used to predict fire performance of cables. The first work published involved showing how to test materials (plaques), wires and cables with the OSU, at a 30 kW/m^2 incident flux. The work showed the similarities and the differences between the results of both types of testing: wire or cable construction can have a very significant effect on heat and smoke release. When the work was extended to communications cables for use in plenum applications *[35]*, the data from the OSU apparatus was used, in conjunction with the OSU fire model *[38]*, to attempt to predict NFPA 262 results. The prediction was not fully successful, particularly in terms of smoke release, the most critical aspect of tunnel cable tests. In this work the cables were placed in the sample holder by bending them to fill the area without cutting them, while the earlier work used 6" (0.15 m) lengths and laid them side by side. However, even with the various flaws found, this work showed that heat release rate data could be used to predict full scale fire performance. Improvements have been made since, especially on smoke release. In fact, later work *[39]* showed that the OSU apparatus could also be used, at incident heat fluxes of ≤ 30 kW/m^2, to predict length of char and flame spread in a vertical cable tray test [CSA FT4].

Some other work, independently done at the David Taylor Naval Research Center, also found it possible to predict vertical cable tray data (in this case the IEEE 383 test) from OSU data, the preferred incident flux being 20 kW/m^2 *[40-41]*. This work represents a fundamental effort, unfortunately finally truncated. Pocock's objective was to develop a correlation between the data on heat release of electrical cables (using two types of cables: one with high flame spread and one with low flame spread) in the OSU apparatus and the IEEE 383 cable tray flame spread test. Pocock also studied several effects on the burning characteristics of selected cables:

- cable diameter
- ratio of copper conductor cross-sectional area to overall cable cross-sectional area
- cable spacing
- incident energy level.

Pocock found that incident energy and cable diameter had the greatest effect on heat release rate, while copper ratio and cable spacing (in the OSU test) had very little significant effect on test results. He also found that the OSU results were reasonably reproducible, particularly on heat release, although somewhat less on smoke release. He felt the OSU was a more adequate bench scale fire test than some alternatives investigated (FM calorimeter and smoke obscuration tests: ASTM E 662, ASTM D 4100, ASTM E 84). Moreover, cables passing the IEEE 383 test had very little heat release per square meter of cable surface during the first 5 min of the test, at an incident flux of 20 kW/m^2. On the other hand, cables that failed the IEEE 383 test released substantial amount of heat over that same initial period. Another possible screening parameter is the peak heat release rate (Pk RHR): most cables passing the IEEE 383 test had values $<$ 100 kW/m^2 while failing cables had values $\gg$ 100 kW/m^2. However, a few passing cables had Peak RHR only slightly $>$ 100 kW/m^2. More recent work used the OSU to compare the fire performance of various types of cable materials: traditional vinyls, vinyl thermoplastic elastomers, traditional polyolefins and highly fire retarded polyolefins *[7]*.

The Factory Mutual apparatus was used extensively, albeit almost exclusively by the FM laboratory, to carry out cable fire tests, with very little testing of cables using this technique outside of FM. A notable example is CEGB utility research laboratory work *[42]*, where cross-over of cable rankings was found when testing at 21 and at 42% oxygen. The most important conclusion, however, is that heat release tests could be used to predict full scale cable fire performance. Another important aspect of the Factory

Mutual calorimeter, which should not be forgotten, is that it is actually used for specification of cables in non combustible industrial occupancies, as discussed earlier.

Many tests were carried out using the cone calorimeter, most often using plaques of materials to be used for making cable insulations or cable jackets. It is worth highlighting studies made by British workers *[43-44]* and by US workers *[7-12, 45-48]* and later work *[13-14, 49-54]*. In the first case a variety of cable materials with different chemistries were being compared, to understand the advantages of each material, with a variety of tests, including the cone calorimeter. The work showed that no material is universally adequate for making cables, and each material has different advantages and disadvantages. The first stage of the US work was designed as a fundamental series of research projects, trying to understand the way to develop new compounds with better fire performance characteristics. Materials were tested in the cone calorimeter and in the OSU apparatus, and very good correlations were found between the results in both apparatuses. Two other studies followed, the first one *[25]* comparing the fire properties of many materials (a total of 35, of which ca. 12 were designed for wire and cable coating applications) and a series of cable fire tests designed to predict full scale results from RHR fire tests *[10-11, 46, 51]*. The work involved cone calorimeter cable burns and some full scale cable burns, carried out in two facilities. Both facilities were instrumented to be able to measure heat release, smoke release, mass loss and gas release for each test. The conclusions were that cables can be burnt adequately in the cone calorimeter and that the results can then be used to predict the outcome of cable tray tests: UL 1581-1160, CSA FT4 (or IEEE 1202) or ICEA T29-520. In the full scale tests it was found that peak flame height correlated well with extent of char length. In both cases, when the cable fails the test, and burns to the top, there is a plateau, since, obviously, a further increase in peak heat release rate can no longer increase char length or flame height. The results indicate a number of reasonable correlations can be found, for example, (a) between extent of char length and peak heat release rate in the full scale test, and (b) between peak heat release rate in the full scale test and in the cone calorimeter. In order for the cone calorimeter to be a good predictor of full scale fire performance, it must be run at an incident flux of 20-30 kW/m^2. The addition of results from burns at 40-50 kW/m^2 improves the correlation even more. The only exception is the ICEA T29-520 test, which is more severe and requires a combination of results at two fluxes, a low of ca. 20 and a high of 40-50 kW/m^2 in the cone calorimeter. An especially interesting set of results from this work were the conclusions on smoke obscuration. Many of the cables that gave off higher heat release also gave off higher releases of smoke (albeit with some exceptions) and of combustion products. It is interesting to note that this is consistent with the real life results from a cable fire in a Japanese underground tunnel. The cables involved in the tunnel fire contained insulation and jacketing materials made with compounds releasing very little smoke in the small scale smoke test (NBS smoke chamber, ASTM E 662) in the specification; however, the cables released large quantities of black smoke in the real fire *[55]*. It is not unusual to find poor direct correlations between smoke obscuration results in small scale tests and real scale fires. Thus, calculations are often required before predicting smoke results. However, there are also some clear-cut cases, where relatively high smoke can be released from relatively low heat release cables. These are the cases requiring special attention.

During the course of their cable work these researchers went a step further. They tested the compounds in the cables, both in the cone calorimeter and in the OSU apparatus *[8]*. This showed a reasonable correlation between rate of heat release results of jacket compounds and cable tray test rate of heat release results of cables, in either RHR calorimeter *[10-11]*. Furthermore, smoke factor appeared to be a fairly effective first approximation parameter to measure *[56-58]*, since the cone calorimeter results of tests at 20 and at 40 kW/m^2 correlated well both among themselves and with the OSU apparatus results. Another set of industrial cable burn tests involves a large number of different chemistries in the polymers used, but maintaining a constant cable construction, to eliminate this variable *[51]*. The set of 21 cables was tested in the cone calorimeter at 20, 40 and 70 kW/m^2 and in the ASTM D 5537 test, additionally measuring smoke release *[14, 52-54]*. This work confirms most of the results predicted earlier, but goes further in indicating, as the previous British work on cable compounds *[43-44]* had suggested, that there are more than one reason for picking a particular set of compounds for building cables. The main conclusions that were drawn from these cable tray tests were:

- Cable tray tests are well suited to measure many essential fire hazard assessment parameters, and not only flame spread.
- Peak and average heat release rate values are excellent indicators of overall cable fire performance in tray tests, and are much better discriminators than char length or peak flame height.
- Cables that pass tray tests will release $\leq$ 50% of their combustible mass, while those that fail release much more.
- Full scale heat release rate is a reasonable predictor of total smoke release, albeit mostly for passing cables.
- Large improvements in smoke obscuration in full scale fires can be achieved by improving cable fire performance, without necessarily decreasing the specific (per unit mass) amount of smoke emitted by the products.
- Trends found by the cone calorimeter are similar to trends found in full scale tests.
- Cone calorimeter heat release rate is a reasonable predictor of full scale rate of heat release and char length, particularly for passing cables.
- Cone calorimeter smoke factor correlates reasonably well with full scale total smoke release, at least in the first instance.
- The cone calorimeter, with the large number of parameters it measures appears to be a very promising instrument for testing cable fire performance and predicting full scale results.
- For all correlations investigated, it appears that results for passing cables can be analyzed more adequately than those for failing cables.
- In the only case investigated where sheath and insulation materials were the same, the overall fire performance of the cables appeared similar. However, ignitability and propensity to flashover, in the cone calorimeter, still suggest that the sheath may be more important for overall fire performance than the primary insulation.

Work by UL *[59]* shows an independent confirmation of the previous industrial work. Unfortunately, this work involved very few cables and shows little information about the type of cables involved. The range of fluxes found best for predictions is 20-30 kW/m^2. This work is an affirmation that small scale calorimetry can be used to predict results of full scale cable tray tests, as found previously from the calorimeter work (Factory Mutual, OSU calorimeter and cone) described before.

ASTM Committee D9 developed an applications standard for the cone calorimeter to be used with electrical cables (and other electrotechnical materials or products), ASTM D 6113, and completed a round robin for precision evaluation. The test method requires laying the cables side by side, cut to 100 mm lengths, in the cone tray. Other mounting methods have been proposed, and some are being discussed by the committee in 1999. A guidance standard, developed by IEC TC89, on summary and relevance of heat release test methods, IEC 60695-8-2 is due for publication in 2000.

Predictions of Cable Fire Performance

A significant amount of research has shown that cable construction strongly affects cable fire performance: a set of cable coating materials that can produce a plenum-rated cable can fail the test for a different construction. This results in details of listed cable constructions for plenum-rated cable being closely-guarded secrets. Very little work has been published on predicting the effect of cable construction on cable fire performance, but this is clearly an area where there is significant potential for new applied work.

Smoke Obscuration

Smoke obscuration is mostly assessed in materials tests, such as those mentioned above, particularly the NBS smoke chamber, with a number of designations, e.g. ASTM E 662, BS 6401, NFPA 258, NES 711, Boeing BSS 7238, Airbus ATS 1000, NF C 20-902-1. Smoke obscuration can be measured, mostly for quality control, in the Arapahoe smoke test (ASTM D 4100) or the Rohm and Haas chamber (ASTM D 2843). A variant of the NBS smoke chamber has been developed, mainly for the maritime industry: ISO 5659-2 (also NFPA 270 and ASTM E 1995), mandatory for naval use. Research and development measurements of smoke obscuration are made with one of the heat release tests mentioned above: cone calorimeter (ASTM D 6113, for cables), OSU (ASTM E 906) or FM apparatuses. The cone was shown to give reasonable correlations with larger scale smoke tests, but only if derived parameters are used *[54]*; by extension, it was proposed that the OSU is also adequate for the purpose (e.g. Hirschler 1991 *[57]*). The lack of correlation of the NBS smoke chamber (ASTM E 662) with full scale dynamic smoke environments has already been mentioned and has also long been documented (e.g. Hirschler 1993 *[60]*). On the other hand, little information has been published on correlations of results for cables or cable materials between ASTM D 2843 or ISO 5659-2 and full scale fire situations. It is important to stress, however, that little work has been done on predicting data from fire scenarios for which such static tests were developed: closed compartments (or concealed spaces) where there is the potential for oxygen vitiation. In larger scale, vertical cable tray tests offer the opportunity for measuring smoke obscuration (see Table 6), as does the NFPA 262 plenum cable test.

Internationally, low smoke cables are qualified by the 3 metre cube test, (IEC 61034). In this test the vertical cable tray and the propane burner are replaced by a horizontal tray and an alcohol pan fire (formerly a baker's tray). The cable samples are only 2 m long and the packing density is 5 kg combustible/m. The test method chamber is a 3 metre cubical room (total volume 27 m^3). A section of cable is mounted horizontally over a 1 L alcohol burn, which will burn for ca. 25 min, and the smoke is mixed with a small fan. Smoke obscuration is measured photometrically, horizontally. An attenuation value, A_0 is defined as

$$A_0 = \frac{V}{L}\log\left(\frac{I_0}{I_t}\right) \tag{1}$$

(V is the chamber volume, L the light path length, I_0 the initial transmittance, and T_t the measured transmittance). UK recommended values for A_0, based on cable diameter, are:

Cable Diameter (mm)	Number of Cable Sections	A_0 (m^{-1})
10-15	4	0.7
15-25	3	0.8
25-40	2	1.0
40+	1	1.5

Few attempts have been made to predict correlation between larger scale smoke obscuration tests: a study found poor correlation between ASTM D 5424 and IEC 61034.

The IEC documents regarding the NBS smoke chamber test described above should also be noted: IEC 60695-6-30 and IEC 60695-6-31.

Smoke Toxicity

This has been one of the most emotional issues in the fire area for > 20 years. It has been shown that about 2/3 of people US fire fatalities die of smoke inhalation, but a similar fraction of the fatalities die in "flashover" fires, where the fire has progressed beyond the room of fire origin. In such fires, the carbon monoxide yield is sufficient (and virtually independent of the material burning) to cause lethal atmospheres. Thus, it is now clear that in the majority of fires, particularly large fires, the smoke toxicity of individual materials or products has little effect on the overall toxic hazard *[60-71]*. Furthermore, very small fires, where the toxic hazard can be heavily dependent on the individual material burning, rarely generate sufficient smoke to cause lethal concentrations. However, rare situations can be envisaged where toxic potency can make a difference. In particular, the toxic potency of fluoropolymers burning in isolation (something very unlikely to happen in practice) is significantly higher than that of most other materials. In this case, the individual toxicants causing high toxic potencies are complex materials (probably aerosols) which are not routinely found by chemical analysis. Thus, if bioassay is not used, the toxic potency of fluoropolymers appears similar to that of any other combustible material, since chemical analysis searches only for common gases: carbon monoxide, carbon dioxide, hydrogen cyanide, nitrogen oxides, hydrogen chloride, hydrogen bromide, hydrogen fluoride, formaldehyde and acrolein, typically. This is correct in most fires, unless fluoropolymers burn alone.

Both ASTM and NFPA have developed a test method (ASTM E 1678, NFPA 269), based on the NIST radiant method: radiant exposure to quartz lamps (50 kW/m^2 heat flux) and a ca. 200 L exposure chamber, for 6 rats (nose exposure only), in a closed system. Animals are exposed to smoke for 30 min, and then followed for a 14 day post-exposure period. Fatalities are counted and toxic potencies calculated (as LC_{50}: lethal concentration killing 50% of the animals: as the LC_{50} decreases the toxic potency increase, because it means that less material is needed for lethality). For post-flashover fires, values of $LC_{50} > 8$ mg/L are considered of "normal toxicity", since that level corresponds to the toxicity of the carbon monoxide inevitably present at flashover. Moreover, a material containing only carbon, hydrogen, nitrogen, chlorine, bromine, aluminum and silicon is expected to be of normal toxicity, and need not be retested, but there is no consensus on this.

New York State and New York City use the University of Pittsburgh test *[72]* which is a flow through system exposing 4 mice. This test method has been the subject of much controversy. The test has been broadly attacked for technical deficiencies, including the fact that multiple toxic potencies can be obtained with the same material, depending on initial mass loaded, and the excessive sensitivity of mice to irritants. However, it has been used to obtain a very large data base, particularly of electrical materials and products (as administered by the National Electrical Manufacturers Association, NEMA), which showed little differences between materials: 96% of all toxic potencies were statistically indistinguishable *[73-74]*, including polyolefins, fluoropolymers and PVC compounds. New York State has no pass-fail criterion but simply requires testing and filing the information, but New York City has a pass-fail criterion of 19.7 g (of mass charged to cause 50% mice lethality).

The UK Navy (and some other military and transport specifiers) requires cable materials to meet NES 713: a small burner is used on cable materials, and concentrations of a set of 12 combustion gases are measured with Draeger tubes. Concentrations are then divided by arbitrary NES 713 toxicity indices to obtain an overall index, which is best if it is very low. The method has no pass-fail criteria, but specifications do. Data in Table 8, comparing NES 713 indices with experimental toxicity values *[70]*, show that the indices produce high values for materials containing halogens, which rarely "pass" this arbitrary test.

Internationally, animal bioassays are not used, and all testing involves chemical analysis. No international standard toxicity test exists, although work is in progress, at IEC TC89, to develop a test based on the DIN tube furnace. The most recent activity has been the development of the concept of "Life Threat", calculating separately the effects of asphyxiant (or narcotic) gases (such as the carbon oxides) and of irritant gases (such as nitrogen oxides or hydrogen halides) and adding a multiplicative factor of 10 to account for differences between lethality and incapacitation. This effort would boost the importance of toxicity in terms of hazard, and would also be favorable to materials with no heteroatoms, which don't release any of the common irritants. A potential pitfall for polyolefins is that animal bioassays indicate they can be more irritant than PVC *[75]*, but this is unlikely to be taken into account because it requires the use of animals, which will not happen. Furthermore, this concept may, in fact, limit the total amount of combustible materials allowed in a compartment. The concept of Life Threat is being developed in ISO TC92 (ISO CD 13571) and in ASTM E5 and has been severely criticized as illogical *[76]*. An alternative concept is also being considered at ASTM E5: toxic hazard for post-flashover fires.

Table 8. Toxicity Assumptions in NES 713 (ppm)

Gas	LC_{50}	NES 713 Index	Ratio
CO	4,600	4,000	1.2
HCN	150	150	1.0
HBr	3,700	150	24.7
HCl	3,700	500	7.4
HF	500	100	5.0

It should also be mentioned that smoke toxicity information has been used for assessment of fire hazard (and fire risk) in scenarios involving electrical cables, indicating the small degree of importance of this parameter *[77-79]*.

Work is in progress, within IEC TC89, on a tube furnace-based smoke toxicity test. The smoke toxic potency apparatus and test method will be IEC 60695-7-50 and the calculation and interpretation of test results will be IEC 60695-7-51. There is not yet any established date of issuance. Guidance documents issued by IEC TC89 on smoke toxicity exist: IEC 60695-7-1 (general guidance), IEC 60695-7-3 (use and interpretation of test results) and IEC 60695-7-4 (unusual toxic effects)

Smoke Corrosivity

Smoke corrosivity has been a subject of intense debate in recent years. The issue is, primarily, of commercial or marketing interest, while most other fire issues, are safety concerns. Thus, smoke corrosivity control decisions are unlikely to appear in codes and regulations, but should surface in standards and specifications by powerful users. Three types of corrosive effects of smoke on electrical or electronic circuitry can be identified: metal loss, bridging of conductor circuits and formation of non-conducting surfaces on contacts *[80]*. Metal loss results in an increase in resistance of the circuitry, so that electrical conduction is impaired. Bridging (or leakage current) has the opposite effect: decrease in resistance by creating alternative simple paths for current flow. Deposit formation can cause, like metal loss, a loss of electrical conductivity and, thus, make an electrical contact unusable. It can also, mechanically, render parts, such as ball bearings, ineffective as they are not able to turn adequately. The performance tests proposed initially addressed purely metal loss, although bridging can mask their results.

Acid gases combine with water to causes metal corrosion. Originally acid gases were believed to be the **only** entities capable of causing corrosion. Thus, corrosive potential of smoke was determined based only on acid gas emission rankings following material combustion in a hot tube furnace, under an air flow. Water soluble effluents were captured and the solutions titrated for acid gas content (HCl, HBr, HF), acidity (pH) and/or conductivity. In practice, decisions were often taken based purely on chemical composition: i.e. halogen content (or simply whether or not halogens are present). It has since been found that smoke corrosivity can occur with halogen-free smoke, and that it can, under certain conditions, be larger than that due to halogens *[80-82]*. Post-exposure treatments, such as cleaning, can retard (or even fully stop) the corrosion process and save the equipment *[80, 82-83]*. The most common surrogate corrosion testing methods (hot tube furnace tests) include: IEC 60754-1, IEC 60754-2 (international), CSA C22.2 No. 0.3, Clause 4.31 (Canadian), DIN 57 472-Pt 813 (German), or CEGB E/TSS/EX5/8056, Pt. 3 (UK) *[84-88]*.

Smoke corrosivity performance testing started with the French telecommunications industry test (CNET test, 1983 *[81]*), which first showed that smoke corrosivity could occur with materials containing no halogens. In the test the resistance of a copper printed circuit exposed to smoke is measured before and after exposure. The original version, proposed additional consideration of other fire properties: oxygen index (ASTM D 2863) and ignitability (ASTM D 1929). In the test a small material sample (600 mg) is mixed with polyethylene (100 mg) and placed in a 20 L chamber, conditioned to high relative humidity and slightly elevated temperature. The target is a copper printed circuit board, at a total area of ca. 5.6 cm^2 and a copper thickness of 170,000 Å. Combustion results from rapid application of an intense flux, rapidly raising sample temperatures to ca. 800°C. Water soluble combustion products are condensed onto the target, water-cooled to a temperature below that of the bulk of the chamber. The test apparatus is limited to material testing and is incapable of addressing product corrosion. For chlorinated materials, CNET test results were found *[83]* to closely parallel those of acid gas tests, such as the one used in Canada. The CNET test, however, is less sensitive to smoke corrosivity by other halogenated materials, particularly fluorinated ones, which often cause corrosion only several hours after exposure. The CNET test is a standard: ISO 11907-2, even though the interlaboratory evaluation carried out showed very poor precision. The test is rarely used today, and few apparatuses are available. The main overall problems with the CNET test are solvable: (a) the use of forced condensation, which means that the effects of non water-soluble products is lost, (b) the lack of post-exposure and (c) a very intense fire module. Some minor problems associated with the CNET test include the excessive time required to clean and condition (to the desired humidity) the chamber, the small chamber volume, and the type of sample chosen, which does not allow testing of finished products, or even mock-ups of finished products. The good fire performance of a tested material does not yield any advantages in the test itself, because of the forced combustion in the presence of polyethylene. Finally, test results can be difficult to interpret because the copper "lines" in the circuit board are so close that there often is a combination of resistance increase due to metal loss and resistance decrease due to bridging. The most serious issue for the CNET test, however, was found when the results of tests on > 20 materials were compared with those from surrogate tests: no parallels with acid gas emission were found *[89-93]*, suggesting the only outliers were chlorinated materials, e.g. based on poly(vinyl chloride), since materials containing bromine, and especially fluorine, were statistically almost indistinguishable from those containing no halogen.

In the US, an ASTM E5 task group developed a test method with a closed system, which has recently been abandoned (persuasive negatives). It has a plastic exposure chamber (ca. 200 L, from the NBS cup furnace toxicity test and the NIST radiant smoke toxicity test, associated with a radiant combustion chamber, ASTM E 1678 or NFPA 269). The sample (up to 50 x 50 mm, with up to 25 mm thickness) is subjected to radiant heat, produced by a set of quartz lamps, for 15 min. The lamps are set at an incident heat flux of 50 kW/m^2. The test method contains all the smoke within the combustion/exposure chamber. However, it is not actually static, as the smoke circulates, impelled through a set of channels communicating the combustion and exposure chambers. There is a 1 h exposure in the test chamber, followed by a 23 h post-exposure, at room temperature and 75% relative humidity. The apparatus is suitable to measure corrosive potential of products, if small representative samples can be placed in the holder. The smoke corrosivity is measured with copper circuit board targets, based on the principle of a Wheatstone bridge circuit, and manufactured by Rohrback Cosasco. The Rohrback targets used differ from the printed circuit boards used for the CNET test in that they have much larger gaps between copper runs, so that bridging is extremely unlikely. There is more experience with the more sensitive targets (2,500 Å nominal span), but the less sensitive ones (45,000 Å span) show similar trends; results are, however, not interchangeable. In fact, the correlation between the two types of targets is unsatisfactory. The test apparatus can accommodate other targets, including full scale equipment, such as a small computer. This test appears to be promising, but no round robin series or other interlaboratory determination of reproducibility or reliability has yet been made.

The third performance smoke corrosivity metal loss test proposed is a dynamic tubular apparatus associated with the German smoke toxicity test (DIN 53436), in a flow-through mode, recommended by ISO TC61 as ISO 11907-3, and generally referred to as the DIN smoke corrosivity test. The sample is placed in a long combustion boat, and a 100 mm long tubular furnace, at a fixed temperature, is slowly (10 mm/min) slid over the sample, and the smoke then exposes a variety of targets. Insufficient published test information prevents discussion of relevance to full-scale smoke corrosivity or precision. One great advantage is that such tubular chambers are fairly common in Europe.

The fourth, and most advanced, smoke corrosivity metal loss test is the cone corrosimeter, ASTM D 5485, also being proposed for international standardization, as ISO 11907-4, by ISO TC61. Burning is conducted in a cone calorimeter (ASTM E 1354, ISO 5660), from which an aliquot of the smoke is diverted into an exposure chamber, where the copper corrosion target is placed. Exposure lasts 1 h and is followed by a 23 h post-exposure at room temperature and 75% relative humidity, to permit corrosion to take its toll (under the concept that recovery after a fire is extremely unlikely to start before 24 h after the start of the fire). It uses the same Rohrback targets as the draft ASTM E5 test, and has two great advantages over all other tests: (a) the fire model is acknowledged to be excellent: the cone calorimeter, and (b) many other fire properties can be assessed simultaneously with the determination of smoke corrosivity. The conditions under which the test is used can be a function of the scenario of interest; if a single scenario is investigated, the recommended heat flux is 50 kW/m^2 (which corresponds to a well developed fire) and the targets to be used are the less sensitive ones (45,000 Å span). The number of laboratories capable of conducting this test is increasing, particularly since the cone calorimeter is gaining in worldwide popularity. A preliminary round robin evaluation was conducted, by ASTM committee D9, but the results are not available.

The latest performance test to be developed for smoke corrosivity is one based on leakage current (or bridging), and it is the result of a multi-year research program by a coalition of DuPont, Lucent (ex AT&T) and UL *[94-95]*. Two fire models were examined: the DIN tube furnace and the cone calorimeter, and the tube furnace was chosen. The targets are copper "interdigitated combs", developed by Bell Labs for atmospheric "dust" testing. The targets cannot conduct electricity when clean (as there is no connection between the terminals), but "leakage current" caused by the smoke produces conductor bridging and increases in current. The test, thus, assesses the decrease in resistance of the targets by different types of smoke. This is dependent not only on the fire conditions (heat flux, in the cone calorimeter; temperature, residence time and air flow rate, in the tube furnace) but also on the voltage applied to the targets and the corresponding relative humidity. Evaluations have covered a broad range of voltages (5-200 V) and relative humidities. Early reported results indicate that materials with excellent fire performance give the best results, irrespective of halogen content. Similar results are found with cables containing such materials: plenum-rated cables perform well and others do not. The main reason seems to be that leakage current appears to be caused by additives and by particles, such as soot or smoke, and not by halogen atoms. The test has been proposed to IEC TC89 and ASTM D9.21 as a new work item.

Two documents have also issued by IEC TC89, within the IEC 60695 series of guidance documents, addressing smoke corrosivity issues: IEC 60695-5-1 (general guidance) and IEC 60695-5-2 (summary and relevance of test methods).

Circuit Integrity

The concept of circuit integrity (or fire resistance of cables) has existed in Europe for many years. The issue addresses whether a cable can continue to operate (i.e. conduct electricity) during a fire, irrespective of its own contribution (or lack of it) to the hazard inherent in the fire as fuel. These cables are used to feed emergency systems (which give power to appliances and services which must continue to operate during crises) and fire alarm and signal cables (which generate messages needed during fire incidents). Two types of tests assess the concept of circuit integrity or fire resistance (sometimes called survivability): those similar to IEC 60331 or BS 6387 (which apply a fire directly to cables, and perhaps follow up with some added physical tests, such as water or mechanical shock) and those, such as the ASTM E 119 time-temperature curve (also in draft UL 2196, ULC ORD C854-1996 and CSA C22.2 N0 0.3), which use high-intensity furnaces and assess the fire resistance of cable systems. The first edition of IEC 60331 was issued in 1970. There is no standard test in the USA for circuit integrity but CSA has incorporated IEC 60331 and draft UL 2196 into its battery of tests, contained in the omnibus CSA C22.2 No. 0.3 standard, where they will be listed with the designation "CIT" for circuit integrity. However, power cables for rail applications have long been required to meet a circuit integrity test by NFPA 130 (as part of the IEEE cable tray test).

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