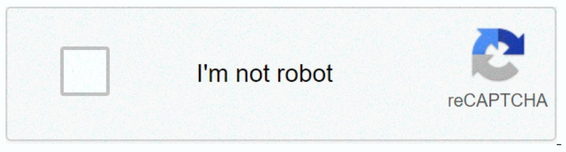

I am not robot!

Mechanism of benzimidazole from o-phenylenediamine and formic acid. Synthesis of ibuprofen from benzene.

[ps2id id='background' target="/]BACKGROUNDPrinciple:The two Carbon-nitrogen bonds in benzimidazole when disconnected give o-phenylenediamine and formic acid. Therefore, synthesis of benziemidazole is affected by simply heating the o-phenylenediamine and formic acid together (condensation type of reaction).1Aim: To prepare benzimidazole from o-phenylenediamine.Reaction:Mechanism:Use:Antitumor, antifungal, antiparasitic, analgesics, antiviral, antihistamine, as well as used in cardiovascular disease, neurology, endocrinology, and ophthalmology.[ps2id id='requirements' target="/]REQUIREMENTSChemicals: o-phenylenediamine Formic acid (90%) NaOH (10%)Apparatus: Round bottomed flask (250 ml) Beaker Buchner funnel Measuring cylinder Filter paper[ps2id id='procedure' target="/]PROCEDUREPlace 27 g (0.25 mol) of o-phenylenediamine in a round bottomed flask of 250 ml and add 17.5 g (16 ml, 0.34 mol) of 90% formic acid. Heat the mixture on a water bath at 100 °C for 2 h. Cool and add 10% sodium hydroxide solution slowly, with constant rotation of the flask, until the mixture is just alkaline to litmus. Filter off the synthesized crude benzimidazole by using the pump, wash with ice cold water, drain well and wash again with 25 ml of cold water.Recrystallisation: Dissolve the synthesized product in 400 ml of boiling water, add 2 g of decolourising carbon and digest for 15 min. Filter rapidly through a preheated Buchner funnel and a flask at the pump. Cool the filtrate to about 10 °C, filter off the benzimidazole, wash with 25 ml of cold water and dry at 100 °C. The yield of pure benzimidazole, m.p. 171-172 °C, is 25 g (85%).Calculation:Here limiting reagent is o-phenylenediamine; hence yield should be calculated from its amount taken.Molecular formula of o-phenylenediamine = $C_6H_8N_2$Molecular formula of benzimidazole = $C_7H_6N_2$Molecular weight of o-phenylenediamine = 108 g/moleMolecular weight of benzimidazole = 118 g/moleTheoretical yield:108 g o-phenylenediamine forms 118 g benzimidazoleTherefore, 27 g o-phenylenediamine will form .....? (X) g benzimidazoleX =(118 × 27)/108 = 29.5 gTheoretical yield = 29.5 gPractical yield =————- g% Yield = (Practical Yield)/(Theoretical Yield) × 100[ps2id id='conclusion' target="/]CONCLUSIONBenzimidazole was synthesized and the percentage yield was found to be.....%.[ps2id id='references' target="/][ps2id id='1' target="/]REFERENCESVogel's Textbook of Practical Organic Chemistry by Brian S. Furniss, Antony J. Hannaford, Peter W. G.
Smith & Austin R. Tatchell; Fifth Edition; Page No.- 1162Practical in organic chemistry, by Hitesh G. Raval, Sunil L. Baldania and Dimal A. Shah, Nirav Prakashan, Page No.- 301.

View PDFVolume 21, Issue 2, February 2017, Pages 229-237Author links open overlay panel rights and contentUnder a Creative Commons licenseopen accessBenzimidazole nucleuso-PhenylenediaminePharmacological activityTherapeutic compound© 2016 King Saud University. Production and hosting by Elsevier B.V. Volume 73, Issue 25, 22 June 2017, Pages 3458-3462Author links open overlay panel, , , , rights and contentBenzimidazoles are ubiquitous motifs, which have found practical applications in a number of fields such as synthesis of natural products and biologically active molecules.1 Also, benzimidazoles are important intermediates in the synthesis of pharmaceutical compounds such as antimicrobial compounds, anthelmintic and antipsychotic drugs, antiulcer and anticancer agents (Fig. 1).2, 3, 4, 5, 6, 7, 8, 9 Many synthetic procedures for the synthesis of benzimidazoles from o-phenylenediamines were reported. For example, a condensation reaction between o-phenylenediamine and carboxylic acid or their derivatives to form benzimidazoles is the most popular method.10 Many kinds of aldehydes, alcohols or orthoesters are utilized to generate benzimidazoles in the presence of various catalysts in oxidative conditions.11 Using CO_2 as C1 block for the synthesis of organic compounds is still a long-standing goal, and many cyclization of o-phenylenediamines by CO_2 to construct benzimidazoles was reported.12 N,N-dimethylformamide (DMF) can be easily synthesized from CO_2 with dimethylamine in the presence of H_2 and suitable catalyst.13 Also, DMF or its derivatives are efficient reagents for the synthesis of benzimidazoles with 1,2-diaminobenzene (Scheme 1, a).14 The synthesis of benzimidazoles from DMF and o-phenylenediamines attracted our attention because using DMF as C1 source could be considered as the indirect utilizing of CO_2.As one of the most effective polar solvents for various chemical reactions, N,N-dimethylformamide has been employed as a widely utilized reactant in organic transformations such as formylation, amination, and cyanation reactions.15 A few approaches have been reported to form benzimidazole from DMF and 1,2-dimethylamine. Treatment of o-phenylenediamine with DMF and 2.5 equivalents of $SiCl_4$ in refluxing CH_2Cl_2 to provide benzimidazole was reported by Bourguignon, but there was only one example in moderate yield (Scheme 1, eq. (1)).14c Kamble and co-workers reported an flexible method to form benzimidazole from o-phenylenediamine and DMF, but a large amount of concentrated hydrochloric acid (70%) was used in this process (Scheme 1, eq. (2)).14b Recently, Bhanage and Liu also reported the preparation of benzimidazole derivatives from DMF and o-phenylenediamines in the presence of hydrosilicon, but in their report, metal catalyst $Zn(OAc)_2{\bullet}2H_2O$ or additive $B(C_6F_5)_3$ and CO_2 were necessary, respectively (Scheme 1, eqs. (3) and (4)).14(a), 16 In a continuation of our ongoing research on the synthesis of valuable benzimidazole compounds,12e we fortunately found an efficient protocol for the synthesis of benzimidazoles from o-phenylenediamines and DMF derivatives employing $PhSiH_3$ as the only promoter without any other catalysts or additives under metal-free conditions (Scheme 1, b).Section snippetsInitially, we began our studies by investigating the condensation reaction of commercially available o-phenylenediamines 1a with DMF 2a. To our delight, when $PhSiH_3$ was employed, the reaction afforded the desired benzimidazole 3a in 95% yield at 120 °C after 12 h (Table 1, entry 1). With this preliminary and intriguing result in hand, we turned to extensively screen a series of hydrosilicons. Various hydrosilicons were tested under the same conditions such as Ph_2SiH_2, Ph_3SiH, $(CH_3)_2PhSiH$, $(CH_3CH$In summary, a simple method for the synthesis of benzimidazoles from o-phenylenediamines and DMF using $PhSiH_3$ as the only promotor is reported. Azabenzimidazole, benzothiazoles and 2-substituted benzimidazoles are also performed in good yields. This work presents a simple system to synthesize benzimidazoles under mild condition. This method has wide substrate scope, providing moderate to high yields. Besides, NMR characterization and HRMS also gave some hints for proposed mechanism. FutureAll reagents and reactants were obtained from commercial sources and used without further purification. Anhydrous solvents were stored in the desiccator. All reactions were monitored by TLC with GF254 silica gel coated plates. Flash column chromatography was carried out by using 200–300 mesh silica gel. 1H NMR and 13C NMR spectra were recorded on a Bruker AvanceⅢ NMR spectrometer (400 MHz) in $CDCl_3$ or DMSO-d6 internally referenced to tetramethylsilane (TMS) or $CDCl_3$ (DMSO-d6) signals.
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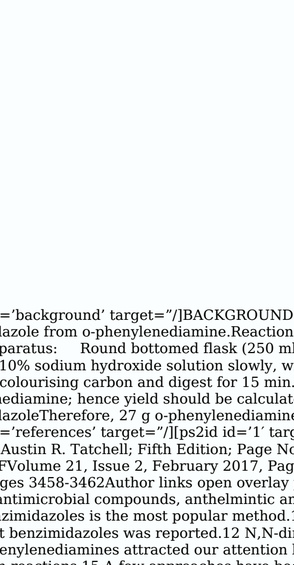
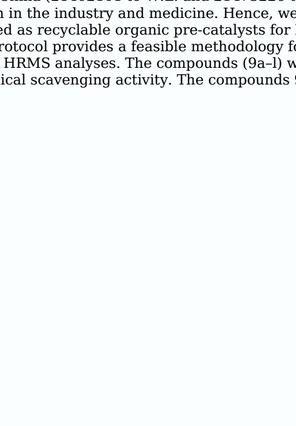


[a]Reaction conditions: o-phenylenediamine (1 mmol), benzaldehyde (1 mmol), $Zn(OTf)_2$ (10 mol%) were stirred for 6h under reflux in Ethanol [b], isolated yields.

Therefore, synthesis of benziemidazole is affected by simply heating the o-phenylenediamine and formic acid together (condensation type of reaction).1Aim: To prepare benzimidazole from o-phenylenediamine.Reaction:Mechanism:Use:Antitumor, antifungal, antiparasitic, analgesics, antiviral, antihistamine, as well as used in cardiovascular disease, neurology, endocrinology, and ophthalmology.[ps2id id='requirements' target="/]REQUIREMENTSChemicals: o-phenylenediamine Formic acid (90%) NaOH (10%)Apparatus: Round bottomed flask (250 ml) Beaker Buchner funnel Measuring cylinder Filter paper[ps2id id='procedure' target="/]PROCEDUREPlace 27 g (0.25 mol) of o-phenylenediamine in a round bottomed flask of 250 ml and add 17.5 g (16 ml, 0.34 mol) of 90% formic acid. Heat the mixture on a water bath at 100 °C for 2 h. Cool and add 10% sodium hydroxide solution slowly, with constant rotation of the flask, until the mixture is just alkaline to litmus. Filter off the synthesized crude benzimidazole by using the pump, wash with ice cold water, drain well and wash again with 25 ml of cold water.Recrystallisation: Dissolve the synthesized product in 400 ml of boiling water, add 2 g of decolourising carbon and digest for 15 min.



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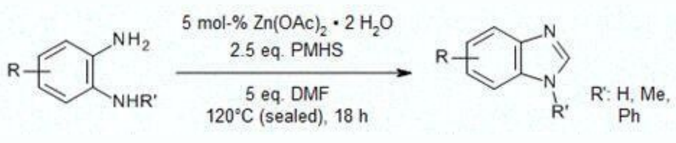

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Synthesis and Characterization of Benzimidazole by Using o-Phenylenediamine with Different Aldehydes and Carboxylic Acids in the Presence of ρ-T_sOH as a Catalyst

ABDULLAH JAWAD KADHIM[1]* and ALI CHAZI KAZIM[2]

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ABSTRACT

This research paper deals with the synthesis and diagnose of Benzimidazole rings which were have been prepared by using two different methods in which used starting material o-phenylenediamine with different compounds. The first method is with aldehydes such as 4-Chlorobenzaldehyde, 4-N, N-Dimethylbenzaldehyde, and Formaldehyde. The second is with carboxylic acids such as salicylic acid, acetic acid, and butamoic acid. ρ-T_sOH has been using as a catalyst in the synthesis methods above and used F.T.I.R and HNMR spectroscopy are used for diagnosing the prepared rings in addition to the physical properties.

Keywords: Aldehydes, Benzimidazole, Carboxylic Acids, ρ-(Toluric sulphonic acid).

INTRODUCTION

Benzimidazole is one of the heterocyclic compounds that shows different biological qualities such as antibacterial and antifungal[1]. Also, some Benzimidazoles have an effect on human viruses such as cytomegalovirus[2]. There are two procedures for the synthesis of 2-substituted Benzimidazoles. The first is the reaction of phenylenediamines and carboxylic acids or its derivatives by heating[3] in strong drying conditions[4]. The second includes a two-step procedure that includes the oxidative cyclodehydrogenation of Schiff bases, which are often generated from the condensation of phenylenediamines and aldehydes[3] and with aryl-aldehydes by using an acidic agent and also with silica gel at room temperature[4]. ρ-TsOH has been used as a neutral acid catalyst to synthesize a number of benzimidazoles[5]. It is considered as an important, effective, available and inexpensive incentive[6]. Also, there are many ways to synthesize benzimidazole by using different catalysts such as Nanocrystallinie oxides with iodine[7], H_2O_2/HCl and $Cu(OTf)_2$[8]. HCl[9]. In this work, the aim was the synthesis of benzimidazole rings by using one catalyst is ρ-TsOH as in previous studies above, which have used one catalyst in different circumstances.

Therefore, synthesis of benziemidazole is affected by simply heating the o-phenylenediamine and formic acid together (condensation type of reaction).1Aim: To prepare benzimidazole from o-phenylenediamine.Reaction:Mechanism:Use:Antitumor, antifungal, antiparasitic, analgesics, antiviral, antihistamine, as well as used in cardiovascular disease, neurology, endocrinology, and ophthalmology.[ps2id id='requirements' target="/]REQUIREMENTSChemicals: o-phenylenediamine Formic acid (90%) NaOH (10%)Apparatus: Round bottomed flask (250 ml) Beaker Buchner funnel Measuring cylinder Filter paper[ps2id id='procedure' target="/]PROCEDUREPlace 27 g (0.25 mol) of o-phenylenediamine in a round bottomed flask of 250 ml and add 17.5 g (16 ml, 0.34 mol) of 90% formic acid. Heat the mixture on a water bath at 100 °C for 2 h. Cool and add 10% sodium hydroxide solution slowly, with constant rotation of the flask, until the mixture is just alkaline to litmus. Filter off the synthesized crude benzimidazole by using the pump, wash with ice cold water, drain well and wash again with 25 ml of cold water.Recrystallisation: Dissolve the synthesized product in 400 ml of boiling water, add 2 g of decolourising carbon and digest for 15 min. Filter rapidly through a preheated Buchner funnel and a flask at the pump. Cool the filtrate to about 10 °C, filter off the benzimidazole, wash with 25 ml of cold water and dry at 100 °C. The yield of pure benzimidazole, m.p. 171-172 °C, is 25 g (85%).Calculation:Here limiting reagent is o-phenylenediamine; hence yield should be calculated from its amount taken.Molecular formula of o-phenylenediamine = C6H8N2Molecular formula of benzimidazole = C7H6N2Molecular weight of o-phenylenediamine = 108 g/moleMolecular weight of benzimidazole = 118 g/moleTheoretical yield:108 g o-phenylenediamine forms 118 g benzimidazoleTherefore, 27 g o-phenylenediamine will form .....? (X) g benzimidazoleX =(118 × 27)/108 = 29.5 gTheoretical yield = 29.5 gPractical yield =————- g% Yield = (Practical Yield)/(Theoretical Yield) × 100[ps2id id='conclusion' target="/]CONCLUSIONBenzimidazole was synthesized and the percentage yield was found to be.....%.[ps2id id='references' target="/][ps2id id='1' target="/]REFERENCESVogel's Textbook of Practical Organic Chemistry by Brian S. Furniss, Antony J. Hannaford, Peter W. G. Smith & Austin R. Tatchell; Fifth Edition; Page No.- 1162Practical in organic chemistry, by Hitesh G. Raval, Sunil L. Baldania and Dimal A.
Shah, Nirav Prakashan, Page No.- 301. View PDFVolume 21, Issue 2, February 2017, Pages 229-237Author links open overlay panel rights and contentUnder a Creative Commons licenseopen accessBenzimidazole nucleuso-PhenylenediaminePharmacological activityTherapeutic compound© 2016 King Saud University. Production and hosting by Elsevier B.V. Volume 73, Issue 25, 22 June 2017, Pages 3458-3462Author links open overlay panel, , , , rights and contentBenzimidazoles are ubiquitous motifs, which have found practical applications in a number of fields such as synthesis of natural products and biologically active molecules.1 Also, benzimidazoles are important intermediates in the synthesis of pharmaceutical compounds such as antimicrobial compounds, anthelmintic and antipsychotic drugs, antiulcer and anticancer agents (Fig. 1).2, 3, 4, 5, 6, 7, 8, 9 Many synthetic procedures for the synthesis of benzimidazoles from o-phenylenediamines were reported. For example, a condensation reaction between o-phenylenediamine and carboxylic acid or their derivatives to form benzimidazoles is the most popular method.10 Many kinds of aldehydes, alcohols or orthoesters are utilized to generate benzimidazoles in the presence of various catalysts in oxidative conditions.11 Using CO2 as C1 block for the synthesis of organic compounds is still a long-standing goal, and many cyclization of o-phenylenediamines by CO2 to construct benzimidazoles was reported.12 N,N-dimethylformamide (DMF) can be easily synthesized from CO2 with dimethylamine in the presence of H2 and suitable catalyst.13 Also, DMF or its derivatives are efficient reagents for the synthesis of benzimidazoles with 1,2-diaminobenzene (Scheme 1, a).14 The synthesis of benzimidazoles from DMF and o-phenylenediamines attracted our attention because using DMF as C1 source could be considered as the indirect utilizing of CO2.As one of the most effective polar solvents for various chemical reactions, N,N-dimethylformamide has been employed as a widely utilized reactant in organic transformations such as formylation, amination, and cyanation reactions.15 A few approaches have been reported to form benzimidazole from DMF and 1,2-dimethylamine. Treatment of o-phenylenediamine with DMF and 2.5 equivalents of SiCl4 in refluxing CH2Cl2 to provide benzimidazole was reported by Bourguignon, but there was only one example in moderate yield (Scheme 1, eq. (1)).14c Kamble and co-workers reported an flexible method to form benzimidazole from o-phenylenediamine and DMF, but a large amount of concentrated hydrochloric acid (70%) was used in this process (Scheme 1, eq. (2)).14b Recently, Bhanage and Liu also reported the preparation of benzimidazole derivatives from DMF and o-phenylenediamines in the presence of hydrosilicon, but in their report, metal catalyst Zn(OAc)2•2H2O or additive B(C6F5)3 and CO2 were necessary, respectively (Scheme 1, eqs. (3) and (4)).14(a), 16 In a continuation of our ongoing research on the synthesis of valuable benzimidazole compounds,12e we fortunately found an efficient protocol for the synthesis of benzimidazoles from o-phenylenediamines and DMF derivatives employing PhSiH3 as the only promoter without any other catalysts or additives under metal-free conditions (Scheme 1, b).Section snippetsInitially, we began our studies by investigating the condensation reaction of commercially available o-phenylenediamines 1a with DMF 2a. To our delight, when PhSiH3 was employed, the reaction afforded the desired benzimidazole 3a in 95% yield at 120 °C after 12 h (Table 1, entry 1). With this preliminary and intriguing result in hand, we turned to extensively screen a series of hydrosilicons. Various hydrosilicons were tested under the same conditions such as Ph2SiH2, Ph3SiH, (CH3)2PhSiH, (CH3CHIn summary, a simple method for the synthesis of benzimidazoles from o-phenylenediamines and DMF using PhSiH3 as the only promotor is reported. Azabenzimidazole, benzothiazoles and 2-substituted benzimidazoles are also performed in good yields. This work presents a simple system to synthesize benzimidazoles under mild condition. This method has wide substrate scope, providing moderate to high yields. Besides, NMR characterization and HRMS also gave some hints for proposed mechanism. FutureAll reagents and reactants were obtained from commercial sources and used without further purification. Anhydrous solvents were stored in the desiccator.
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Herein, we developed Z-scheme MgO/TiO2/g-C3N4 ternary heterojunction photocatalyst with surface defects and effective charge separation for reduction of recalcitrant dinitrobenzene isomers under simulated solar light irradiation. Mott-Schottky (MS) plot analysis and electron spin resonance (ESR) radical trapping experiment suggested the formation of Z-scheme heterojunction at the interface of TiO2/g-C3N4, which played a crucial role in the electron-hole separation. Incorporating MgO into the structure further enhances charge separation via Ti3+ and oxygen vacancy (OV) defects formation at the TiO2/MgO interface as confirmed by electron paramagnetic resonance (EPR) and X-ray photoelectron spectroscopy (XPS) analyses. Besides, the surface basicity of MgO enhanced conversion of dinitrobenzene (DNB) isomers through formation of nitrophenylhydroxylamine intermediate which can easily be reduced to phenylenediamines (PDAs). As confirmed by high performance liquid chromatography (HPLC) analysis, excellent selectivity for PDAs (95–98%) was achieved in 90 min with ternary MgO/TiO2/g-C3N4 composite compared to the binary MgO/TiO2 and TiO2/g-C3N4. A possible reaction pathway and photocatalytic reduction mechanism were proposed and elucidated. This work demonstrated an effective strategy to reduce recalcitrant dinitrobenzene isomers using efficient, low-cost, and environmental benign photocatalyst with a facile identification of reaction intermediates.A simple and green protocol was developed for the reductive cyclization of o-phenylenediamine with CO2 and BH3NH3 to yield 1H-benzimidazole. The desired 1H-benzimidazole derivatives were produced under mild conditions. Mechanism investigation indicated that the coordination of o-phenylenediamine with the boron atom of BH3NH3 promoted the transfer of the formyl group to form a stable intermediate, which facilitated the intramolecular nucleophilic addition-elimination for the formation of target product. In this process, BH3NH3 served multifunctional roles, acting as a reducing agent and a formylation catalyst.Imidazoles and benzimidazoles together with their partly or fully saturated derivatives particularly as carbenes, are very important and useful heterocycles with applications in the chemical, biological, material and pharmaceutical sciences. The general trend of the previous edition has been followed with emphasis on developments regarding structure, reactions and synthesis, reported in the literature from 2007 to 2019.
A large part of the current review describes the reactivity and synthesis of conjugated and nonconjugated imidazoles and their substituents whereas theoretical and experimental structure elucidation methods, as well as applications, are also presented.The cyanation and formylation of imidazo[1,2-a]pyridines were developed under copper-mediated oxidative conditions using ammonium iodide and DMF as a nontoxic combined cyano-group source and DMF as a formylation reagent. Mechanistic studies indicate that the cyanation of imidazo[1,2-a]pyridines proceeds through a two-step sequence: initial iodination and then cyanation.
The cyanation has a broad substrate scope and high functional group tolerance, and can be safely conducted on a gram scale. A novel copper-mediated formylation using the widely available DMF as the formylation reagent and environmentally friendly molecular oxygen as the oxidant has also been developed. This protocol also provided a convenient approach for the synthesis of clinically used saripidem.View all citing articles on ScopusView full text© 2017 Elsevier Ltd. All rights reserved.
@article{Alaqeel2017SyntheticAT, title={Synthetic approaches to benzimidazoles from o-phenylenediamine: A literature review}, author={Shatha Ibrahim Alaqeel}, journal={Journal of Saudi Chemical Society}, year={2017}, volume={21}, pages={229-237}, url={ 99123372} }Hadole CdRajput JdR. BendreChemistry, Medicine2018This review summarizes pharmacological and medicinal activities of 2-substituted benzimidazole and its marketed drugs.W. AkhtarM. F. Khan M. AlamMedicine, Chemistry2017S. JohnG. Kavya Akhil SivanMaterials Science, Chemistry2020A mild and effective protocol for benzimidazolesynthesis from o-phenylenediamine and aromatic aldehydes catalysed by DBU is described. The synthesized compounds find application in the… Rushi D. TrivediS. DeR. GibbsChemistry2006R. SrinivasuluK. R. KumarP. SatyanarayanaChemistry2014We report the synthesis of benzimidazole derivatives using zinc triflate as an efficient catalyst. One-pot synthesis of 2-substituted benzimidazole derivatives from o-phynelyenediamine and… Songnian LinLihu YangChemistry2005G. HolanE. SamuelB. C. EnnisR. HindeChemistry1967Methods of preparation of 2-trihalogenomethyl-benzimidazoles, -benzothiazoles, and -benzoxazoles have been investigated.
It has been shown that the reaction of the mono-salts of o-phenylenediamines,… Ş. DemirayakA. KaraburunI. KayağilU. UçucuR. BeisChemistry, Medicine2005Some of the compounds obtained by the reaction of 2-chloroacetylamino-3-ethoxycarbonylthiophene derivatives and a suitable benzazole-2-thione or benzimidazole derivatives showed remarkable analgesic activities.The synthesis and evaluation of the novel head-to-head bisbenzimidazole compound showed potent growth inhibition and some evidence of antitumor activity, and an X-ray crystallographic study of a complex with the DNA dodecanucleotide sequence d(CGCGAATTCGCG) shows the compound bound in the A/T minor groove region of a B-DNA duplex.