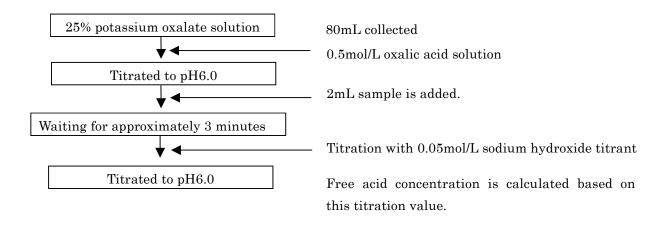
| AQUACOUNTER Application Sheet |                           | COM series |    | D/   | DATA No. F8 |    | 1 st edition |       |
|-------------------------------|---------------------------|------------|----|------|-------------|----|--------------|-------|
| I Electronics                 | Quan                      | tification | of | free | acid        | in | lead         | frame |
|                               | treatment solution for IC |            |    |      |             |    |              |       |

#### 1. Measurement outline

The etching treatment solution for IC lead frames comprises of free acids, whose main component is hydrochloric acid, and ferric chloride solution. While copper ion is gradually dissolved, the concentrations of hydrochloric acid and ferric chloride also change when etching treatment is conducted on lead frames.

Various methods have been contrived as the measurement method on free acids including ferric chloride. It is difficult to fractionate only free acids using neutralization titration since ferric chloride is also titrated. One method for masking ferric chloride is the use of adjuvant complexing agent. This section introduces an example in which it is quantified by neutralization titration after masking ferric chloride with potassium oxalate as the adjuvant complexing agent.



#### 2. Reagents and Electrodes

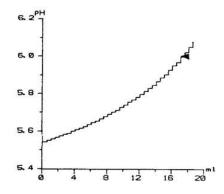
| (1) Reagents          | Titrant             | 0.5mol/L oxalic acid titrant<br>0.05mol/L sodium hydroxide titrant |  |  |
|-----------------------|---------------------|--|--|--|
|                       | Loading buffer      | 25% potassium oxalate solution                                     |  |  |
| (2) Electrodes        | Indicator electrode | *Glass electrode GE-101B to IE jack                                |  |  |
| *standard accessories | Reference electrode | *Reference electrode RE-201 to RE jack                             |  |  |

### AQUA COUNTER

## 3. Measurement conditions example (for COM1600S + 1 unit of Buret B-2000-20)

| Master file          | 1                                       |               |                      |            |       |  |
|----------------------|---|---------------|----------------------|------------|-------|--|
| Condition file 1 + 2 |   |               |                      |            |       |  |
| Parameters fo        | r condition file 1                      | Parameters fo | or condition file 2  |            |       |  |
| (For the titrat      | (For the titration with oxalic acid) (F |               | ment)                | Mode No. 5 |       |  |
| Method               | DISP                                    | Method        | AUTO/SET             | Pre Int    | 0 sec |  |
| Amp No.              | 1                                       | Amp No.       | 1                    | Del K      | 5     |  |
| Buret No.            | 2                                       | Buret No.     | 1                    | Del Sens   | 0 mV  |  |
| Meas Unit            | pН                                      | Meas Unit     | pН                   | Int Time   | 3 sec |  |
| S Timer              | 0 sec                                   | S Timer       | 240 sec              | Int Sens   | 3 mV  |  |
| Direction            | DOWN                                    | CP mL         | 0 mL                 | Brt Speed  | 2     |  |
| End Point            | 6.00 pH                                 | Direction     | UP                   | Pulse      | 40    |  |
| Max Vol.             | 20 mL                                   | DP mL         | 1 mL                 |            |       |  |
|                      |   | End Sens      | 500                  |            |       |  |
|                      |   | End Point     | 6.00 pH              |            |       |  |
|                      |   | Over mL       | 1 mL                 |            |       |  |
|                      |   | Max. Vol.     | 20 mL                |            |       |  |
|                      |   | Unit          | %                    |            |       |  |
|                      |   | Blank         | 0                    |            |       |  |
|                      |   | Factor        | Titer of the titrant |            |       |  |
|                      |   | Molarity      | 0.05                 |            |       |  |
|                      |   | K             | 36.46                |            |       |  |
|                      |   | L             | 1.476                |            |       |  |
|                      |   | Formula       | (D-B)×K×F×M/(S×L×10) |            |       |  |
|                      |   | Mode No.      | 5                    |            |       |  |

## 4. Measurement example



### Measurement results on hydrochloric acid and free acid

| Sample<br>No. | Sample<br>volume<br>(mL) | Titration<br>value<br>(mL) | Concentra-<br>tion<br>(%) |
|---------------|--------------------------|----------------------------|---------------------------|
| 1             | 2.5                      | 17.220                     | 0.8510                    |
| 2             | 2.5                      | 15.011                     | 0.7461                    |
| 3             | 2.5                      | 13.985                     | 0.6951                    |
|               | Avg                      |                            | 0.760 %                   |
|               | Std. Dev.                |                            | 0.08 %                    |
|               | C.V.                     |                            | 10.4 %                    |

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#### 5. Outline

(1) About masking method with complexing agent

In this method, measurement was taken according to the analysis method in Analytical Chemistry (Vol.17 1273 1968), results could not be obtained with good repeatability. Though its cause is not clear, it is assumed that it may depend on the amount of complexing agent added, complexing period and the concentration of ferric chloride.

(2) About other measurement methods

As a neutralization method other than this method, a neutralization method in which ferric chloride is changed to ferrous chloride by adding sodium thiosulfate has been proposed (patented).

#### **Key words**

Hydrochloric acid, ferric chloride, neutralization titration, potassium oxalate

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