

Paint · Adhesive

## Amino Group in Dyestuff

Redox titration by  
Automatic Potentiometric Titrator

**Standard**

### 1. Abstract

Measurement of amino group in dyestuff is performed by potentiometric titration with 0.1mol/L sodium nitrite while maintaining temperature below 15°C after concentrated hydrochloric acid is added.

The amino group can be calculated from titration volume up to the endpoint.

### 2. Reference

- 1) “Experiment and Calculation in Quantitative Analysis” –Vol.2 by Seiji Takagi from Kyoritsu Publishing Company

### 3. Cautions in measurement

- 1) In order to avoid evaporation and decomposition of sodium nitrite, the sample temperature must be maintained below 15°C during titration.

## 4. Post-measurement care

Clean the electrode with pure water, and keep it dipped in water for use in next measurement.

## 5. Test equipment

Main unit : Automatic potentiometric titrator (Standard preamplifier STD-)  
Electrode :  Platinum electrode  
 Ceramic type reference electrode

## 6. Reagent

Titrant : 0.1mol/L Sodium nitrite (f=1.00)  
Additive : Pure water, Concentrate hydrochloric acid

## 7. Measurement procedure

—Measurement—

- 1) Add 10mL concentrate HCl to the sample in a 100mL beaker, and dissolve.
- 2) Add 40mL pure water, and cool the liquid down to below 15°C in thermal bath.
- 3) Titrate with 0.1mol/L sodium nitrite to obtain amino group in dyestuff.

## 8. Formula

Concentration of Amino group ( % ) = ( EP1 - BL1 ) × FA1 × C1 × K1 / SIZE

EP1 : Titration volume ( mL )  
BL1 : Blank level ( 0.00mL )  
FA1 : Factor of titrant ( 1.00 )  
C1 : Concentration conversion coefficient ( 1.4 mg/mL )  
(NH<sub>2</sub> in mg equivalent to 1mL of 0.1mol/L NaNO<sub>2</sub>)  
K1 : Coefficient ( 0.1 )  
SIZE : Sample size ( g )

## 9. Example of measurement

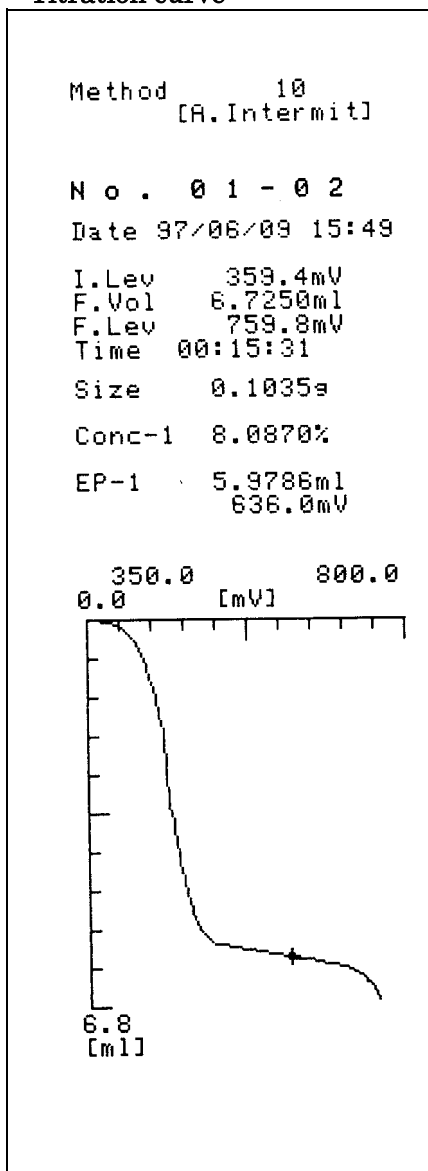
— Ambient condition —

|                          |                 |                  |
|--------------------------|-----------------|------------------|
| Room temperature : 23 °C | Humidity : 59 % | Weather : Cloudy |
|--------------------------|-----------------|------------------|

### - Titration parameter -

|                     |                     |
|---------------------|---------------------|
| Model : AT-400      |                     |
| Method No. : 10     |                     |
| Titr.mode : Auto    |                     |
| Intermit            |                     |
| Titr.form : EP Stop |                     |
| [TITR. PARA]        |                     |
| Form : EP Stop      | [CALCU. PARA]       |
| Buret No. : 1       | Sample Measurement  |
| Preamp : STD        | Conc1 CalcuNo. 2    |
| Detector No. : 2    | Conc1 Dim. [%]      |
| Dimension : mV      | Conc1 EP Position 1 |
| Max.Vol : 20.0mL    | Data [f(EP2-Blank)] |
| W.Time : 0s         | T.Type [Normal]     |
| Direction : Auto    | Data [f(T.Vol)]     |
|                     | Local Blank         |
|                     | Blank 0.00mL        |
|                     | Common T.Factor     |
|                     | K1 0.1              |
|                     | C1 1.4mg/mL         |
|                     | Temp.Comp. [Off]    |
| [CTRL. PARA]        |                     |
| End Point No. : 1   |                     |
| S(dE) : 50          |                     |
| S(E/mL) : 50        |                     |
| O.Titr : 0mL        |                     |
| Gain : 1            |                     |
| S.Pot : 4.0mV       |                     |
| Stab. : 0.5mV/s     |                     |
| Delay Time : 20s    |                     |
| L.Time : 60s        |                     |
| M.Unit : 0.5mL      |                     |
| Separation : Off    |                     |
| A.Simulation : Off  |                     |

### - Titration curve -



(The above printout data are obtained from titration by AT-400 unit)

#### « TITR. PARA: Titration parameter »

Form: of titration / Buret No.: the burette used in titration / Detector No.: the detector used in titration  
Dimension: potential unit/ Max Volume: of titration / Wait Time: before titration starts / Direction.: of titration

#### « CTRL. PARA: Control parameter »

End Point No. number of EPs detected / S(dE): EP potential (difference) / S(E/mL): EP potential (differential)  
Over Titr.: over-titration volume / Gain: sensitivity of detection signal/ S.Pot.: potential changes of sampling signal  
Stab.: stability sense potential/ L.Time: time for stability check  
M.Unit.: titration for sampling signal / Separation: of potential/ A.Simulation: redetection of EP  
Delay Time: before stability check

#### « CALCU. PARA Result parameter »

Calc.No.: of formula / Conc.1: formula 1 / Conc1Dim: unit of concentration/Conc1EP Position: EP at concentration 1  
K1: unit conversion coefficient/ Factor: of reagent / Blank: blank level / Data: titration form used in calculation  
T.Type: titration type (normal or back) / Common T.Factor: factor of titrant registered  
C1(mg/mL): concentraionat EP 1/Data: calculation of titration / Temp.Comp: temperature compensation

–Measurement results–

| n | Sample (g)    | Titration (mL) | Amino group (%) | Batch processed amino group |          |
|---|---------------|----------------|-----------------|-----------------------------|----------|
|   |               |                |                 | Mean                        |          |
| 1 | 0.1075        | 6.3061         | 8.2126          | Mean                        | 8.1900 % |
| 2 | <u>0.1035</u> | <u>5.9786</u>  | <u>8.0870</u>   | SD                          | 0.0938 % |
| 3 | <u>0.1035</u> | 6.1142         | 8.2704          | RSD                         | 1.1449 % |

\*The above results were obtained by 3 tests of the same sample.

\* Red underline shows the data from page 3/4.

## 10.Summary

Dyestuff is colored substance, which is used to dye fabrics or paper usually by dissolving in solvent (typically water). Those not dissolved in solvent and dispersed in medium are called colorant or pigment.

Mono functional group (-NH<sub>2</sub>, -NHR, -NRR') where hydrogen is removed from ammonia, primary or secondary amine is called Amino group. When substituted on aromatic ring, it shows nature of electron releasing group.

The test result shows a good repeatability with less than 1.1% relative standard deviation.

Precise and reliable measurement is assured by the automated potentiometry.

The analysis of amino group in dyestuff can be perfectly made by any of the following titration systems manufactured by Kyoto Electronics (KEM).

### 【AT-610】



#### Awarded Product of Supreme Technology from Kyoto City

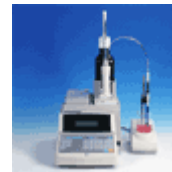
- Easy key entry by touch panel of large color LCD (8-inch wide)
- Simultaneous titration in parallel
- Both potentiometric and Karl Fischer moisture titration (coulometric + volumetric) can be performed at a time.

### 【AT-510】



- Compact and cost performance model
- PC card expands data memory for convenience and versatility.

### 【AT-500N-1】



- Low cost and high performance
- Easy view with back light LCD
- GLP/GMP conformed model

# KEM KYOTO ELECTRONICS MANUFACTURING CO.,LTD.

Overseas Division: Yamawaki Bldg 9F,4-8-21 Kudan-minami,Chiyoda-ku,  
Tokyo 102-0074 JAPAN

Phone: +81-3-3239-7333, Fax: +81-3-3237-0537

Head Office: 56-2 Ninodan-cho,Shinden,Kisshoin,Minami-ku,  
Kyoto 601-8317 JAPAN

Phone: +81-75-691-4122, Fax: +81-75-691-9961

URL: <http://www.kyoto-kem.com>