

Paint, Adhesive

## Amino Group in Dyestuff

Redox titration by  
Automatic Potentiometric Titrator

**Standard**

### 1. Abstract

In this application, we show how to titrate aromatic primary amine in dyestuff with nitrous acid and quantify it by generating diazonium salt. After adding concentrated hydrochloric acid to the sample, titrate it to the equivalence point with 0.1 mol / L sodium nitrite solution while keeping it at 15 ° C or lower. The equivalence point is the inflection point on the titration curve and the content of nitrogen present as amino group is calculated from the titration amount of sodium nitrite solution.

### 2. Reference

- 1) Seiji Takagi “Experiment and Calculation in Quantitative Analysis ” –Vol.2 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 )

(nitrogen(N) mg equivalent to 1mL of 0.1mol/L NaNO<sub>2</sub>)

K1 : Coefficient ( 0.1 )

SIZE : Sample size ( g )

## 9. Example of measurement

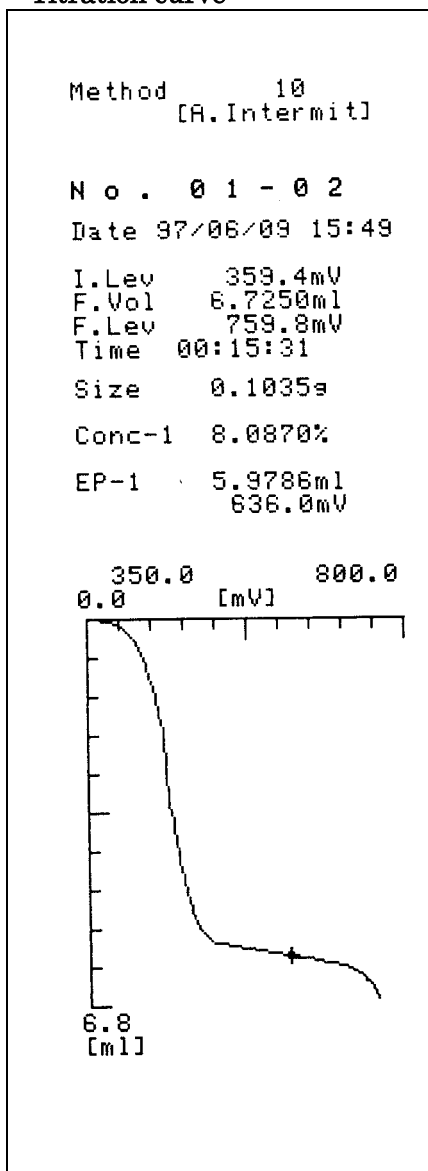
— Ambient condition —

Room temperature : 23 °C	Humidity : 59 %	Weather : Cloudy
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### - 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 (%)	Statistical calculation	
				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	0.1035	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

Functional groups (-NH<sub>2</sub>, -NHR, -NRR') obtained by removing hydrogen from ammonia and amine are called amino groups. The amino group on the aromatic ring shows the property as an electron pair donating group, and when cold nitrous acid acts, the diazonium salt is formed. This reaction is called a diazotization reaction, and titration using the reaction is called diazotization titration.

In this test, good repeatability was obtained with a relative standard deviation of 1.1%. The use of an automatic potentiometric titrator improves the reliability of the measurement.