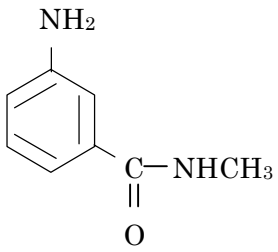
AQUACOUNTER Application Sheet	COM series	DATA No. B12	1st edition
Pharmaceuticals	Measurement of sulfamethizole purity by diazotization titration		

1. Measurement outline

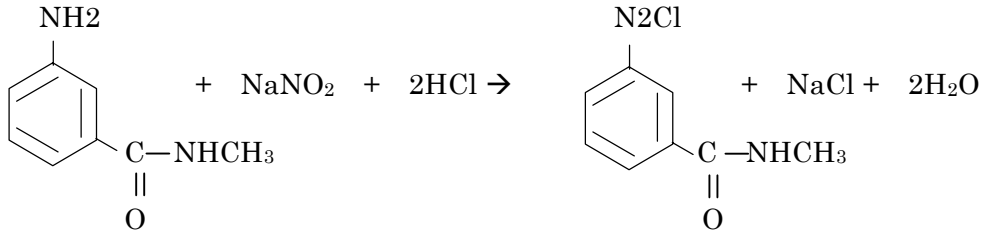
This section introduces an example of purity measurement on AMB, an intermediate for medical products, by diazotization titration conforming to Japanese Pharmacopoeia (13th edition).

0.5g sample was weighed and added with 10mL hydrochloric acid and 40mL purified water, and then was added with 10mL potassium bromide solution as the reaction catalyst. Amperometric titration was conducted with sodium nitrite titrant on the sample solution which was cooled to 10°C or lower.



3-amino-N-methylbenzamid

$C_8H_{10}N_2O$



2. Reagents and Electrodes

(1) Reagents	Titrant	0.1mol/L sodium nitrite titrant
	Hydrochloric acid 10mL for 1 measurement	
	Potassium bromide solution(3→10) 10mL for 1 measurement	
(2) Electrodes	Twin platinum electrode TPT-351 *P-2000 standard accessory	

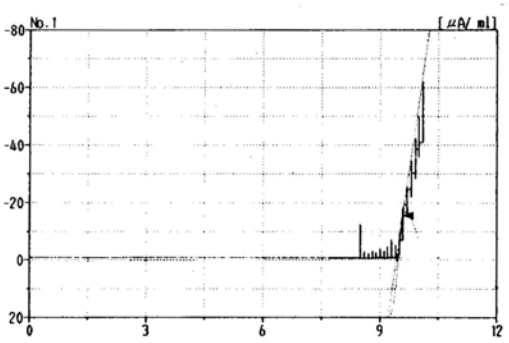
3. Measurement conditions example (for COM-1600P w/ Polarization titration unit)

Master File No.1 Condition file: 1	
Method	F Cross
Amp No.	2
Buret No.	1
Meas Unit	μA
S-Timer	0 sec
CP	0 mL
T-Timer	0 sec
DP	1.00 mL
End Sens	60
Over mL	0 mL
Max Vol	16 mL
Mode No.	21
Unit	%
Blank	0
Factor	Titer of the titrant
Molarity	0.1
K	270.32
Formula	(D-B)×K×F×M/(S×10)

Mode No.21	
Pre Int	0 sec
Del K	2
Del Sens	5 mV
Int Time	10 sec
Int Sens	3 mV
Brt Speed	2
Pulse	40

Polarization voltage (VPOL): 200mV

4. Measurement example



Sample measurement results

Sample No.	Sample volume (g)	Titration value (mL)	Titer (%)
1	0.1503	9.590	99.15
2	0.1487	9.504	99.32
3	0.1480	9.440	99.11
	Avg.		99.19 %
	Std. Dev.		0.11 %
	C.V.		0.11 %

5. Outline

(1) In this section, favorable results were obtained using the current titration (constant-voltage current titration) method as the end point detection method. Though constant-current potentiometric titration was also tried, current titration method was better.

(2) Since diazotization titration requires time for measurement in general, titrant was successively added until it was near titration end point in advance to conduct titration by fixed-volume dropping and a certain waiting period after reaction period (2 minutes on reaction timer) in order to shorten the measurement period in this section.

(3) Since the concentration of hydrochloric acid affects the reaction speed in diazotization reaction, it is important that the concentration of hydrochloric acid at titration is adjusted to 1 – 3mol/L

Key words

Medical product, diazotization titration, sodium nitrite, dual platinum electrode, current titration

Hitachi High-Technologies Corporation
Head Office 1-24-14, Nishishinbashi,Minato-Ku,Tokyo 105-8717, Japan
Tel : 81-3-3504-7239 Fax : 81-3-3835-7302
http://www.hitachi-hitech.com

Hiranuma Sangyo Co.,Ltd.
1739, Motoyoshidacho, Mito-City, Ibaraki 310-0836, Japan
Tel : 81-29-247-6411 Fax : 81-29-247-6942
http://www.hiranuma.com