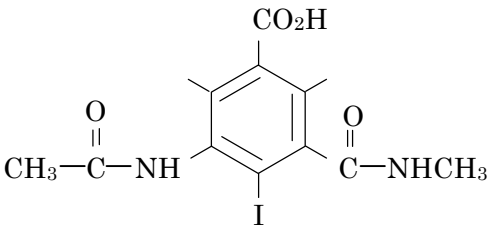
AQUACOUNTER Application Sheet	COM series	DATA No. B16	1st edition
Pharmaceuticals	Quantification of iothalamic acid by precipitation titration		

1. Measurement outline

The method for quantifying iothalamic acid which is used as a contrast medium for X ray is stipulated in Japanese Pharmacopoeia (13th revision).

In this method, the sample is dissolved in 40mL sodium hydroxide solution, and 1g zinc powder is added for 30-minute boiling with a circulating cooler to allow hydrolysis. It is filtered after cooling, and the flask and filter paper are washed with water. 5mL glacial acetic acid is added to the sample solution for titration with silver nitrate titrant, with the end point detected when the yellow color of the indicator (1mL tetrabromophenolphthalein ethyl ester) turns to green.

This section introduces an example with potentiometric titration method using silver electrode as the end point detection method.



Iothalamic acid
$C_{11}H_9I_3N_2O_4$

$$NaI + AgNO_3 \rightarrow AgI + NaNO_3$$

2. Reagents and Electrodes

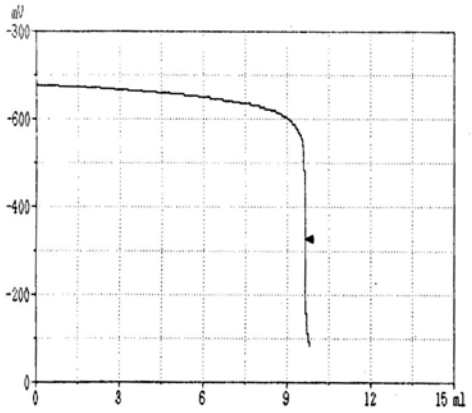
(1) Reagents	Titrant	0.1mol/L $AgNO_3$ titrant
	Loading buffer	5mL glacial acetic acid
(2) Electrodes	Indicator electrode	Silver indicator electrode AG-311 to IE jack (P/N E231245-A)
	Reference electrode	Silver reference electrode MS-231 to RE jack (P/N D231243-A)

3. Measurement conditions example (for COM-1600S)

Master File No.1 Condition file: 1	
Method	AUTO
Amp No.	2
Buret No.	1
Meas Unit	mV
S-Timer	0 sec
CP	0 mL
DP	0 mL
End Sens	500
Over mL	0.50 mL
Max Vol	40 mL
Mode No.	5
Unit	%
Blank	0
Factor	Titer of the titrant
Molarity	0.1
K	204.64
Formula	(D-B)×F×K×M/(S×10)

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

4. Measurement example



Measurement results

Sample No.	Sample volume (g)	Titration value (mL)	content (%)
1	0.2000	9.623	99.37
2	0.2000	9.637	99.51
3	0.2000	9.625	99.39
4	0.2000	9.630	99.44
5	0.2000	9.622	99.36
6	0.2000	9.642	99.56
	Avg.		99.44 %
	Std. Dev.		0.08 %
	C.V.		0.08 %

5. Outline

(1) In this experiment, potential difference detection method was used for end point detection in measurement to deliver results with high precision.
In general, potentiometric titration method is popularly used in quantification of halogen ions (Br^-, I^-, Cl^-), and one feature of this method is easy end point detection even in cases in which judgment of indicator inflection point is difficult due to coloring in sample, etc. It also has the feature to allow measurement with little individual error.

(2) When conducting potentiometric titration on high-concentration halogen ion with silver nitrate in general, generation of silver halide precipitation is feared to adhere to indicator electrode, etc. and affect the titration curve and measurement precision. As one measure against this, “silver chloride precipitate cohesion prevention agent” can be added in advance to the titrated solution to reduce precipitation of silver chloride and prevent adherence to the indicator electrode as well as absorption of halogen ion into precipitation, thus improving the measurement precision.

Key words

Medical product, iothalamic acid, precipitation titration, silver chloride precipitate cohesion prevention agent

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