

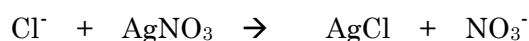
AQUACOUNTER Application Sheet	COM series	DATA No. F1	1st edition
Electronics	Quantification of trace chlorine ion in IC molding resin		

1. Measurement outline

Chlorine ion in IC molding resin causes parts failure in which the circuit on IC board is corroded. Therefore, analysis on chlorine ion contained in material is an important item on which the product performance depends.

The method for quantifying chlorine ion in resin applies potentiometric titration with silver nitrate titrant by dissolving the sample in solvent (acetone, cyclohexane, methyl ethyl ketone, methanol, tetrahydrofuran, etc.) which is acidified with acetic acid or nitric acid. If the sample does not dissolve under room temperature, it needs to be dissolved by heating.

This section introduces an example in which 5g of resin powder was weighed precisely to be heated and dissolved in 40mL methanol and acidified with nitric acid for potentiometric titration with silver nitrate.



2. Reagents and Electrodes

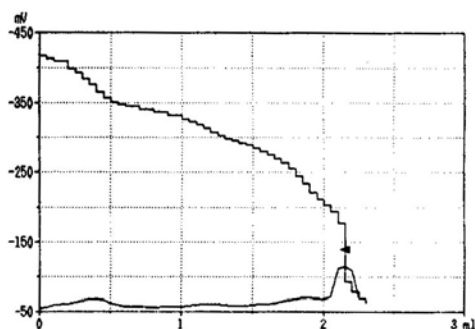
(1) Reagents	Titrant	0.005mol/L AgNO ₃ titrant
	Titration solvent	40mL methanol for one measurement
	Loading buffer	0.1mL 1+1 nitric acid
(2) Electrodes	Indicator electrode	Silver indicator electrode AG-312 to IE jack (P/N D231259-A)
	Reference electrode	Silver reference electrode MS-231 to RE jack (P/N 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	5 sec
CP	0 mL
DP	0 mL
End Sens	500
Over mL	0 mL
Max Vol	40 mL
Mode No.	5
Unit	ppm
Blank	0
Factor	Titer of the titrant
Molarity	0.005
K	35.453
Formula	$(D-B) \times F \times K \times M \times 1000 / S$

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 on chlorine ion in resin

Sample No.	Sample volume (g)	Titration value (mL)	Concentration (ppm)
1	6.0263	1.553	45.05
2	8.9661	2.425	47.23
3	7.7833	2.120	47.61
Avg. (Average value)			46.63 ppm
Std. Dev. (Standard deviation)			1.38 ppm
C.V. (Coefficient of variation)			3.0 %

5. Outline

(1) About blank test on titration solvent

When quantifying trace chlorine in resin, the trace chlorine contained in the solvent will cause measurement error. Thus it is necessary that the blank test is always conducted on the solvent to make correction.

(2) About indicator electrode

Since this measurement is a non-aqueous titration which uses organic solvent, the indicator electrode needs to be selected with consideration of its chemical resistance. AG-312 in which silver electrode is fixed to Teflon is recommended as the indicator electrode for this measurement (AG-311 uses epoxy resin and may become dissolved).

(3) About acidic loading buffer

The acidic loading buffer used in this measurement is acetic acid or nitric acid. Caution is required when nitric acid is used in measurement of trace chlorine ion since the silver electrode may be dissolved and cause error if the nitric acid concentration becomes too high.

(4) About reference electrode

Since this measurement is a non-aqueous titration, the liquid junction block for the reference electrode may be dehydrated and increase electrical resistance. Thus it is necessary that the crystal of inner solution be removed regularly by soaking it in purified water or by loosening the sleeve for the liquid junction block.

(5) About standard addition method

The inflection point for titration curve may become unclear when chlorine ion is quantified in the trace amount of several ppm. Favorable results may be obtained when the standard addition method is used as the titration method in such cases. In this method, solution containing chlorine ion (about several tens of ppm) is prepared in advance to titrate a certain volume of this reagent and calculate the blank test value. Then a certain volume of the sample and this reagent is titrated. The measurement result is calculated by subtracting the blank test value from the measurement value.

Key words

Resin, IC, chlorine ion, precipitation titration, standard addition method

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