Category

AUTOMATIC TITRATOR COM Series

Electrical/Electronics technologies

Trace Cl^- in IC molding resin

by Potentiometric titration method (non-aqueous)

Referenced methods

Key words; Resin, IC, chlorine ion, precipitation titration, non-aqueous

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 factor for the performance of products.

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

This application introduces an example in which weighted 5g of resin powder heated and dissolved in 40mL methanol and acidified with nitric acid for potentiometric titration with silver nitrate.

$$Cl^- + AgNO_3 \rightarrow AgCl + NO_3^-$$

Reagents

Titrant	:	0.005 mol/L silver nitrate solution
Titration solvent	:	Methanol … 40mL per measurement
Buffer	:	Nitric acid (1+1) … 0.1mL per measurement

Instruments & Electrodes

Recommended automatic titrator	**COM-1700S / COM-300A**
・P/N D231259-A	**AG-312** Silver electrode for organic solvent use
・P/N D231243-A	**MS-231** Reference electrode

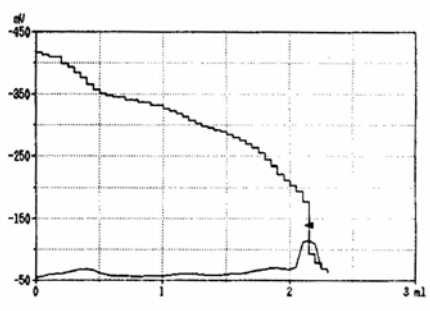
Model: **COM-1700S**

Model: **COM-300A**

Condition parameters (example)

Master File 1 **Condition 1** (for BLANK)				**Master File 2** **Condition 2** (for measurement)			
Method	Auto			Method	Auto		
Buret No.	1	Mode No.	22	BURET No.	1	Mode No.	5
Amp. No.	2 or 3	Pre Int	0 sec	Amp. No.	2 or 3	Pre Int	0 sec
Meas Unit	mV	Del K	0	Meas Unit	mV	Del K	5
S-Timer	10 sec	Del Sens	0 mV	S-Timer	10 sec	Del Sens	0 mV
CP mL	0 mL	Int Time	5 sec	CP mL	0 mL	Int Time	3 sec
DP mL	0 mL	Int Sens	3 mV	DP mL	0 mL	Int Sens	3 mV
End Sens	500	Brt Speed	2	End Sens	500	Brt Speed	2
Over mL	0 mL	Pulse	8	Over mL	0 mL	Pulse	40
Max. volume	1 mL			Max Volume	40		
Unit	mL			Unit	ppm		
Size	0 g			Size	--- g		
Blank	0 mL			Blank	Result value from blank titration		
Factor	Titer of the titrant			Factor	Titer of the titrant		
Molarity	0.005 mol/L			Molarity	0.005 mol/L		
K	35.453 (as Cl^-)			K	35.453 (as Cl^-)		
L	-			L	-		
Formula	D			Formula	(D-B)xFxKxMx1000/S		

Measurement result example



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 %**

Information plus

1. Maintenance of electrode performance

Since this measurement is a non-aqueous titration, the liquid junction 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 DI water or by loosening the sleeve for the liquid junction block.

2. NaCl standard solution addition method

The inflection point for titration curve may be unclear when chlorine ion is quantified in the trace amount (few ppm). Favorable results may be obtained when the standard addition method is used as the titration method in such cases. A known-concentration standard solution containing chlorine ion (about 10~20 ppm) is prepared and added to the sample solution prior to titration. The measurement result is calculated by subtracting the blank test value from the measurement value.

For more information, please feel free to contact:

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