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HIRANUMA APPLICATION DATA		Automatic Titrator	Data No.	F10	Apr. 5,2019
Electric & Electronics	Determination of alkaline component in photoresist remover solution (Determination of carbonates with barium chloride addition method)				

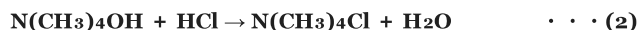
1. Abstract

Strong alkaline solution is used as remover for residual photoresist on the substrate after the development of the substrate for liquid crystal display instrument etc. The composition depends on the intended use but sodium hydroxide, potassium hydroxide, and tetramethylammonium hydroxide (TMAH) are used as remover.

TMAH can be determined by potentiometric titration with hydrochloric acid standard solution. TMAH could absorb carbon dioxide gas and generate carbonates, it indicates another inflection point at pH around 4 on the titration with hydrochloric acid standard solution. This report introduces an example of fractional determination for TMAH and carbonates. Barium chloride is added to determine carbonates; the precipitate of barium carbonate is generated on this method (formula (1)).



This solution is titrated with hydrochloric acid. Only TMAH is determined at the first inflection point, and barium carbonate is determined at the second inflection point on the titration.



The reactions between TMAH and hydrochloric acid is described in formula (2), barium carbonate and hydrochloric acid is shown in formula (3). It reaches the endpoint at pH around 9 and 4 on the reaction (2) and (3), respectively. The titrant volume to determine carbonates gets twice on this method compared with the method without the addition of barium chloride. This method is useful especially for a sample containing less amount of carbonate.

2. Configuration of instruments and Reagents

(1) Configuration of instruments

Main unit : Hiranuma Automatic Titrator COM series

Electrodes : Glass electrode GE-101B

Reference electrode RE-201Z

*The following electrodes are usable instead of the above electrodes.

· Glass reference combination electrode GR-501BZ...Fixed sleeve type

· Glass reference combination electrode GR-511BZ...Moveable sleeve type

(2) Reagents

Titration : 0.5 mol/L Hydrochloric acid standard solution

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ilute to 100 mL

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3. Measurement procedure

- (1) Take about 25 g of sample into a 50 mL beaker and weigh accurately.
- (2) Add 10 mL of 10 % barium chloride solution.
- (3) Immerse electrodes and start titration with 0.5 mol/L hydrochloric acid standard solution.

4. Measurement conditions and results

Examples of titration conditions

(1) Titration of TMAH

Cndt No	1	Constant No.	1	Mode No.	22
Method	Auto	Size	25.1046 g	Pre Int	0 sec
Buret No.	1	Blank	0 mL	Del K	9
Amp No.	1	Molarity	0.5 mol/L	Del Sens	0 mV
D. Unit	pH	Factor	1.005	Int Time	2 sec
S-Timer	5 sec	K	91.15	Int Sens	3 mV
C.P. mL	11.5 mL	L	0.000	BrT Speed	2
T Timer	5 sec	Unit	%	Pulse	20
D.P. mL	0 mL	Formula	$(D-B)*F*M*K/(S*10)$		
End Sens	3000	Decimal Places	4		
Over mL	0 mL	Auto In Pram.	None		
Max Vol.	20 mL				

(2) Titration of carbonates

Cndt No	2	Constant No.	2	Mode No.	23
Method	Auto	Size	25.1046 g	Pre Int	0 sec
Buret No.	1	Blank	0 mL	Del K	0
Amp No.	1	Molarity	0.5 mol/L	Del Sens	0 mV
D. Unit	pH	Factor	1.005	Int Time	2 sec
S-Timer	0 sec	K	104.15	Int Sens	3 mV
C.P. mL	0 mL	L	0	BrT Speed	2
T Timer	0 sec	Unit	%	Pulse	20
D.P. mL	0 mL	Formula	$(D-B)*K*F*M/(S*10)$		
End Sens	5000	Decimal Places	4		
Over mL	0.1 mL	Auto In Pram.	None		
Max Vol.	20 mL				

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RESULTS OF TMAH

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Number of measurement	Size (g)	Titrant Volume(mL)	Concentration (%)
1	25.1046	12.514	2.2832
2	25.1382	12.513	2.2799
3	25.1796	12.534	2.2800
Statistic calculation		Avg.	2.281 %
		SD	0.0019 %
		RSD	0.08 %

Measurement results of carbonates

Number of measurement	Size (g)	Titrant volume (mL)	Concentration (%)
1	25.1046	0.605	0.1261
2	25.1382	0.621	0.1293
3	25.1796	0.624	0.1297
Statistic calculation		Average	0.128 %
		Standard deviation	0.0020 %
		Coefficient of variation	1.54 %

Expanded figure

Examples of titration curves

5.Note

- (1) Measurement method
- TMAH is strong alkaline reagent which absorbs carbon dioxide gas in air and generates carbonates. Purging air on sample solution with nitrogen gas can reduce the influence of carbon dioxide on the titration. Using air passed through soda lime absorption column instead of nitrogen gas also has a positive effect. Water wasn't added to sample; non-diluted sample was titrated directly in this measurement. Please pay attention to carbonic acid in the water when it is added for the sample dilution before the titration.
- (2) Sample measurement without addition of barium chloride
- Barium chloride was added to the sample solution to determine the carbonates. The measurement is possible without barium chloride when the sample contains a certain amount of carbonate. (Please refer to application note F9 regarding the method without the addition of barium chloride.)

Keywords: Tetramethylammonium hydroxide, TMAH, Remover, Carbonates, Neutralization titration, Barium chloride, Barium carbonate

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