

Application Note

Determination of Carboxy groups of ion exchange resin

Industry	Chemicals
Instrument	Automatic potentiometric titrator
Measurement method	Conductometric titration/ Neutralization titration
Standards	

1. Scope

Ion exchange resins are widely used for water purification, ion exchange, isolation, and so on. This Application Note introduces an example of the determination of carboxy groups on the surface of an ion exchange resin (Latex particles with carboxy groups introduced on the surface) (7. Notes). The measurement method utilized was conductometric titration. The endpoint of the titration is the point at which the conductivity rate undergoes a drastic change.

2. Apparatus

Equipment	Automatic potentiometric titrator (Preamplifier for conductometric titration CMT)
Electrode	Electric conductivity cell

3. Reagents

Titrant	0.02 mol/L Sulfuric acid aqueous solution
Additive reagent	0.1 mol/L Sodium hydroxide solution

4. Procedure

- 1) Pour or pipette the sample into a beaker and measure its mass.
- 2) Add 50 mL of pure water.
- 3) Adjust the pH to between 10 and 11 by adding an appropriate amount of 0.1 mol / L sodium hydroxide aqueous solution.
- 4) Titrate with 0.02 mol / L sulfuric acid aqueous solution and identify the endpoint as being the point at which the titration curve starts to significantly bend.

5. Calculation

$$\text{Surface carboxy groups } (\mu\text{mol/g}) = (\text{EP2}-\text{EP1}) \times \text{TF} \times \text{C1} / \text{S} / \text{K1}$$

EP2	Second endpoint titration (mL)	
EP1	First endpoint titration (mL)	
TF	Factor of Titrant	= 0.9767
C1	Concentration conversion coefficient	= 4000
S	Sample size (g)	
K1	Latex concentration in the sample (%)	

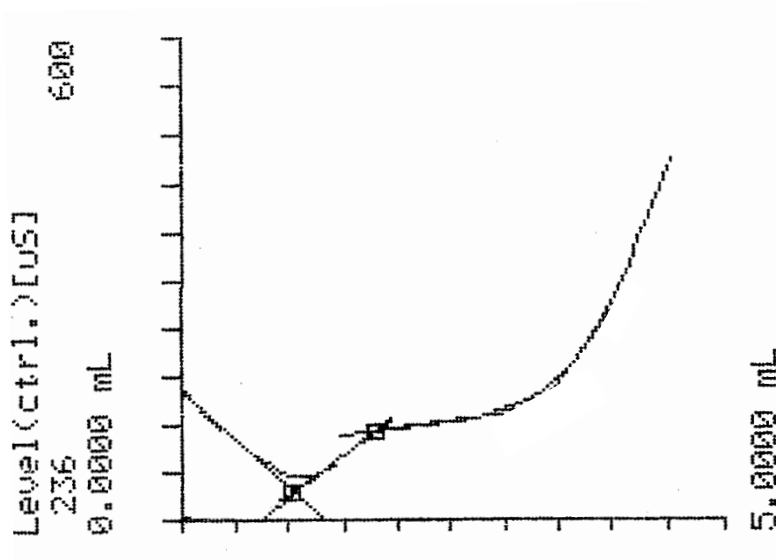
6. Example

— Parameter —

<p><Titr. Mode> Auto Intermit</p> <p><Titr. Form> Intersect</p> <p><Titr. Para.></p> <p> Max Volume 20 (mL), Sample</p> <p> Channel/Unit(Ctrl.) Ch3, 1000μS</p> <p> Wait Time 5 (s)</p> <p> Dose Mode None</p>	<p><Ctrl. Para.></p> <p> End Point No. 3</p> <p> End Sense (dE/dmL) Set</p> <p> EP Stop Mode Both</p> <p> End Sense (Angle) 15$^{\circ}$</p> <p> Gain 4</p> <p> Data sampling Set</p> <p> Data Samp. Pot. 999mV</p> <p> Data Samp. Vol. 0.1mV</p> <p> Ctrl. Speed Set</p> <p> Cut off time 5s</p> <p> Unit Volume 0.1mL</p> <p> Disp. Speed 1s/mL</p> <p> Other Control Standard</p> <p> Stirrer Speed 4</p>
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(Listed above are example settings. Availability of settings may vary by instrument model.)

— Example of titration curve —



— Measurement results —

Table 1 Measurement results

	Sample (g)	First endpoint titration (mL)	Second endpoint titration (mL)	Surface carboxy groups ($\mu\text{mol/g}$)
1	7.5686	1.3230	2.0900	38.44
2	8.0252	1.2618	2.0766	38.51
3	7.5074	1.0050	1.7569	37.99
Mean	-	-	-	38.31
SD	-	-	-	0.28
RSD (%)	-	-	-	0.73

7. Notes

In this measurement, "Intersect" was applied as the endpoint detection method. In this function, two tangential lines are drawn at the bends of the titration curve, and their point of intersection is deemed to be the endpoint. Excess sodium hydroxide solution was added to the sample to generate sodium salts that were in turn titrated with the sulfuric acid solution.

The observed bends each show an endpoint. The first endpoint corresponds to the presence of excess sodium hydroxide and the second to the carboxy groups on the surface of the particles. Therefore, the determination of carboxy groups on the surface of the latex particles is possible by calculating the difference between the titration volumes at the second and first endpoints.

8. Reference

John Hen, Determination of surface carboxy groups in styrene/itaconic acid copolymer latexes, *Journal of colloid and interface science*, **1974**, 49 (3), 425-432.