MASTER TEST METHODS

MTM 0265: CATION SALT SPLITTING CAPACITY AND PERCENT REGENERATION : H form resins

Objective

To determine the total exchange capacity of cation exchange resins on a weight (eq/kg) and volume (eq/L) basis. Also determine the percent regeneration.

Area of Application

This procedure is applicable to strong acid cation resins with sulfonic acid functional groups in the hydrogen form.

Principle

A sample of resin is pretreated as for the Moisture Holding Capacity. A known weight and volume of resin is eluted with $NaNO_3$ and the quantity of H determined by titration with NaOH. This provides the initial quantity of H present. The resin is then fully converted to the H form, the H is eluted with $NaNO_3$, and the quantity of H is determined by titration with NaOH. This provides the strong acid, or salt splitting, capacity. This capacity is assumed equivalent to the total capacity for cation resins with sulphonic acid functional groups.

Equipment

Dewatering apparatus.
Source of vacuum, 40 ± 2 torr below atmospheric pressure.
Analytical balance, ± 0.001 g precision or better.
Ventilated oven, 105 ± 2 °C.
Desiccator.
Tared weighing dishes.
25 ml graduated cylinder, Kimble 20022-25 or equivalent.
Fritted glass filter tube.
1 L volumetric flask.
100 ml pipet.
Equipment for NaOH titration.

Reagents

1.0 N HCl.
0.5 N $NaNO_3$.
0.1000 N NaOH, standardized.
Deionized water.
Phenolphthalein indicator solution (5 g/L), used only for manual titrations.

Master Test Method

1. Prepare resin by the Master Test Method procedure for MHC.
2. Measure and record the **MHC** (to nearest 0.1%).
3. Weigh out a 15.0 ± 0.5 g sample of the prepared resin. Record the resin weight as $\mathbf{W_{moist}}$ (to nearest 0.01 g).
4. Quantitatively transfer the sample to a 25 ml graduated cylinder.
5. Cover the resin with deionized water to the 25 ml mark.
6. Shake the sample gently for 30 sec.
7. Allow the resin to settle, then tap the base of the graduated cylinder until no further settling is observed. Record the resin volume as $\mathbf{V_{moist}}$ (to nearest 0.25 ml).
8. Quantitatively transfer the sample to a fritted glass filter tube.
9. Pass 1 L of 0.5 N $NaNO_3$ through the sample at a rate of 25 ml/min. Collect exactly 1 L of effluent in a clean 1 L volumetric flask.
10. Stopper the flask and mix the contents thoroughly by inverting at least 5 times.
11. Pipet a 100 ml sample of the $NaNO_3$ effluent into a titration beaker.
12. Titrate the sample for H using 0.1000 N NaOH. Record the titration volume as $\mathbf{V_{As\ received,\ NaOH}}$ (to nearest 0.01 ml) and the NaOH normality as $\mathbf{N_{NaOH}}$ (to nearest 0.0001 eq/L).
13. Rinse the same resin sample with 200 ml of deionized water. Discard the rinsate.
14. Pass 1 L of 1.0 N HCl through the sample at a rate of 25 ml/min.
15. Rinse with 1 L of deionized water at a flow rate of 25 ml/min. At the end of the rinse, verify that the effluent is near neutral using indicator paper.
16. Pass 1 L of 0.5 N $NaNO_3$ through the sample at a rate of 25 ml/min. Collect exactly 1 L of effluent in a clean 1 L volumetric flask.
17. Stopper the flask and mix the contents thoroughly by inverting at least 5 times.
18. Pipet a 100 ml sample of the $NaNO_3$ effluent into a titration beaker.
19. Titrate the sample for H using 0.1000 N NaOH. Record the titration volume as $\mathbf{V_{Regen,\ NaOH}}$ (to nearest 0.01 ml).
20. Pipet a 100 ml sample of the $NaNO_3$ influent blank into a titration beaker.
21. Titrate the blank for H using 0.1000 N NaOH. Record the titration volume as $\mathbf{V_{Blank,\ NaOH}}$ (to nearest 0.01 ml).
22. Report results to 1 decimal place, i.e., 95.0 %.
23. Calculate

$$\textbf{Wt.Cap.}\ (\text{eq/kg}) = \frac{10 \times (\mathbf{V_{Regen,\ NaOH}}\ (\text{ml}) - \mathbf{V_{Blank,\ NaOH}}\ (\text{ml})) \times \mathbf{N_{NaOH}}\ (\text{eq/L})}{\mathbf{W_{moist}}\ (\text{g}) \times (1 - \mathbf{MHC}\ (\%) / 100)}$$

$$\textbf{Vol Cap.}\ (\text{eq/L}) = \frac{10 \times (\mathbf{V_{Regen,\ NaOH}}(\text{ml}) - \mathbf{V_{Blank,\ NaOH}}\ (\text{ml})) \times \mathbf{N_{NaOH}}\ (\text{eq/L})}{\mathbf{W_{moist}}\ (\text{ml})}$$

$$\textbf{PERCENT}_{\mathbf{H}}\ (\%) = \frac{100 \times (\mathbf{V_{As\ received,\ NaOH}}\ (\text{ml}) - \mathbf{V_{Blank,\ NaOH}}\ (\text{ml}))}{\mathbf{V_{Regenerated,\ NaOH}}\ (\text{ml}) - \mathbf{V_{Blank,\ NaOH}}\ (\text{ml})}$$

24. Report capacity results to 2 decimal places, *i.e.*, 5.00 eq/kg or 2.00 eq/L. Report regeneration to 1 decimal place, *i.e.*, 95.0%.

Notes

1. This test is run on new ion exchange resins "as received." New resins receive no pretreatment.
2. This procedure measures the salt splitting capacity of the cation resin. It is generally assumed that this is the total capacity for cation exchange resins with sulphonic acid groups.
3. This procedure is a combination of MTM 0230 and MTM 0260.
4. The titration can be done manually or with an automatic titrator. For a manual titration, use 2 drops of phenolphthalein indicator solution, and the endpoint occurs when the color changes from clear to pink and persists for 15 s.
5. A blank determination is not required for every sample if it can be shown by good laboratory practice that the blank is negligible. For example, this can be accomplished by routine monitoring: once per shift, once per day, once per week, once per batch of reagents, *etc.*