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Note

A device for regeneration of cation-exchange resins with hydrochloric acid

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Cation-exchange resins are usually regenerated with 5–6 *M* hydrochloric acid. A mixture of water and hydrochloric acid distils as a 5.54 *M* azeotrope ("constant-boiling hydrochloric acid"), the maximum boiling point of which is at 108.6° (refs. 1 and 2). Laboratories, however, do not seem to utilize this fact, although the volume of hydrochloric acid needed for regeneration is usually 4–6 times the volume of the resin, and the used hydrochloric acid generally contains non-volatile solutes, from which the azeotrope can be distilled.

I have found it practical to use simple distillation apparatus in a circulating way to regenerate resins with hydrochloric acid. The system has several advantages, *viz*: (a) the acid content in the distillate is accurately known, (b) use of large amounts of strong acids is avoided, (c) disposal of hydrochloric acid into sewers is minimized, (d) the system is economical, (e) solutes washed from the resin can be regained in concentrated form (important with, *e.g.* radioactive materials), and (f) the system is practical because it operates independently and the resin does not drain.

APPARATUS

Fig. 1 shows a simple version of the device, which consists of an all-glass distillation assembly, a column for the resin and a PTFE siphon-tube to permit circulation of the acid in the system. A contact thermometer is used to control the distillation so that current to the heating mantle is switched off if the temperature drops, *e.g.*, 4–5°, from 108.6°.

OPERATION

A suspension of the moist resin (*e.g.*, 1 l) in 5 *M* hydrochloric acid is poured into the column, which should be about half full in order to ensure adequate hydrostatic-pressure limits. If the resin flows sluggishly, the column must be large enough to ensure that it does not fill when distillation is carried out at the desired rate. The PTFE tube must be carefully tightened so that it does not become loose during operation.

When the resin has settled, the levels of the top of the resin bed and the end of the PTFE tube in the flask are adjusted to be about equal, and about 1 l of 5 *M* hydrochloric acid is applied to the resin. After most of the acid has flowed into the flask, the

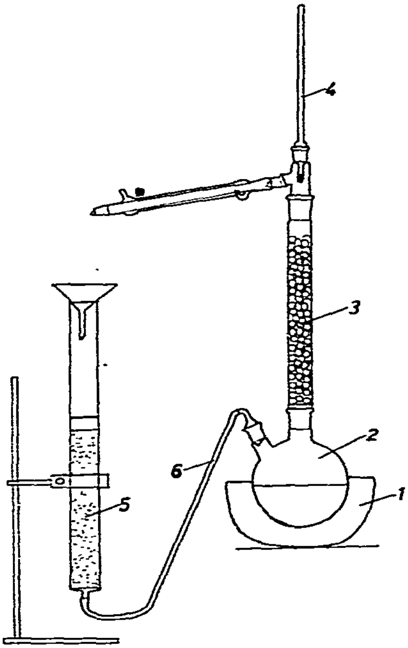

Fig. 1. Device for regeneration of cation-exchange resins: 1 = heating mantle; 2 = round-bottomed flask; 3 = column filled with glass beads (*ca.* 5 mm in diameter); 4 = thermometer (50–150°); 5 = column of resin; 6 = PTFE siphon. The operation is described in the text.

heating mantle and water supply to the condenser are turned on. Adequate boiling is adjusted by controlling the current to the heating mantle, and anti-bumping granules are used to ensure smooth boiling. The distillate is collected in a separate vessel until the boiling point of the azeotrope is reached; it is then allowed to enter the resin column. After equilibrium conditions have been attained, the system operates automatically, and the time needed for regeneration of the resin is estimated from the rate of distillation.

When the regeneration is complete, hydrochloric acid remaining in the column is rinsed into the round-bottomed flask with a small amount of distilled water; the column is then removed from the system, and hydrochloric acid is distilled at about 108.6° for collection and storage.

The apparatus can also be used to wash the resin with distilled water; in this instance, a suitable amount of sodium hydroxide must be placed in the flask to neutralize incoming acid.

REFERENCES

1 R. C. Weast (Editor), *Handbook of Chemistry and Physics*, 55th ed., The Chemical Rubber Co., Cleveland, Ohio, 1974, p. D-27.
2 E. Heftmann (Editor), *Chromatography*, Reinhold Publishing Co., New York, 1961, p. 320.