

FLAME EMISSION EXPERIMENTS

21.25 DETERMINATION OF ALKALI METALS BY FLAME PHOTOMETRY

Although flame emission measurements can be made by using an atomic absorption spectrometer in the emission mode, the following account refers to the use of a simple flame photometer (the Corning Model 410 flame photometer).

Before attempting to use the instrument read the instruction manual supplied by the manufacturers.

Preparation of standard solutions for calibration curves. The following concentrations are suitable:

(a) Sodium. Dissolve 2.542 g sodium chloride in 1 L de-ionised water in a graduated flask. This solution contains the equivalent of 1.000 mg Na per mL (i.e. 1000 ppm). Dilute this stock solution to give four solutions containing 10, 5, 2.5, and 1 ppm of sodium ions.

(b) Potassium. Dissolve 1.909 g potassium chloride in 1 L de-ionised water. This solution contains the equivalent of 1.000 mg K per mL (i.e. 1000 ppm).

Dilute this stock solution to give four solutions containing 20, 10, 5, and

2 ppm of potassium ions.

(c) Calcium. Dissolve 2.497 g calcium carbonate in a little dilute hydrochloric acid, and dilute to 1 L with de-ionised water. This stock solution contains

the equivalent of 1.000 mg Ca per mL. Dilute this solution to give solutions containing 100, 50, 25, and 10 ppbn of calcium ions.

(d) Lithium. Dissolve 5.324 g pure lithium carbonate in a little dilute hydrochloric acid and dilute to 1 L with de-ionised water. This solution contains

1.000 mg Li per mL (i.e. 1000 ppbn). Dilute the stock solution to give solutions containing 20, 10, 5, and 2 ppm, of lithium ions.

Prepare calibration curves for each of the above four elements. With the aid of these calibration curves, carry out the following simple determinations.

1 . Potassium in potassium sulphate. Weigh out accurately about 0.20 g potassium

sulphate and dissolve it in 1 L de-ionised water. Dilute 10.0 mL of this solution to 100 mL, and determine the potassium with the flame photometer using the potassium filter.

2. ***Potassium and sodium in a mixture.*** Mix suitable volumes of the above stock solutions so that the resulting solution contains, Say, 4- 10 pprn Na and 10-15 pprn K. Determine the Na and K with the aid of the appropriate filters. Compare the results obtained with the true values.

3. ***Sodium, potassium, and calcium in a mixture.*** Mix appropriate volumes of the above stock solutions so that the test solution contains, Say, 5 ppmNa, 10 pprn K, and 40 pprn Ca. Determine the Na, K, and Ca with the aid of the appropriate filters. Compare the results obtained with the true values.

4. ***Calcium in calcium carbonate.*** Determine the calcium in an analysed sample

of dolomite. Dissolve about 0.38 g, accurately weighed in 1: 1 hydrochloric acid, warm gently, filter through a quantitative filter paper, wash, dilute the combined filtrate and washings to 1 L. Measure the calcium content of the resulting solution: use a calcium filter. Compare the value for Ca thus obtained with the known Ca content.

Prepare a calibration curve for each element (see Section 21.16) and use this to evaluate the concentration of any unknown sample (see Note).

Note. The unknown solution may require dilution to give a reading on the calibration curve.

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