Micro Determination of Magnesium with Naphthol Black

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Naphthol black or wool black ([naphthalene disulphonic (1, 3)-<7 azo 1>-naphthalene<4 azo 1>[Naphthol-(2)-disulphonic-(3, 6)]), an acidic disazo dye, has been employed for the micro-determination of magnesium. The blue lake is formed instantaneously in highly alkaline medium (pH>12) and found to be sufficiently stable in presence of 1.0% starch and 50% glycerol solutions for the measurements to be carried out. Among the other stabilizers tried, the mixture of the above two was found to be most satisfactory. The maximum difference in absorbance between the lake and the dye solutions lies at 570 mμ as indicated in Fig. 1. All the measurements were, therefore, carried out at this particular wavelength. Beer's law is followed within the range of 8—26 μg of magnesium per m*l* of solution. The mole ratio studies do not reveal the presence of any definite complex. The recommended procedure is as follows:

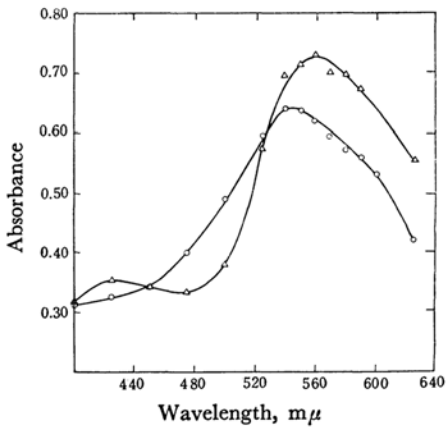

Fig. 1. Absorption curves of naphthol black and its magnesium lake at pH>12 and 30°C.
A (-△-) 2 m*l* of 0.1% napthol black, 1 m*l* of 0.1% Mg(II) and 2 m*l* each of 1% starch, 50% glycerol and 5 N sodium hydroxide in 25 m*l*, against water.
B (-○-) As in A except magnesium solution, against water.

A suitable volume of the test solution containing 0.2 to 0.65 mg of magnesium is taken in a 25 m*l* measuring flask and 1 m*l* of 0.1% dye (Gurr's, C. I. 315, recrystallised from water) solution and 2 m*l* of each of starch, glycerol and 5 N sodium hydroxide solutions are added. The volume is then raised up to the mark with double distilled water and the solution shaken thoroughly. The absorbance of the solution is then measured by Beckman DU 2400 spectrophotometer against the water blank at 570 mμ. The absorbance can also be measured against the reagent blank provided it is used within a period of one hour of its preparation. The concentration of magnesium in the test solution is then found from a calibration curve obtained by using solutions containing 8, 10, 12, 16, 20, 24, 26 μg of magnesium per m*l*, all the measurements being carried out under the same conditions. It is preferable to run the standard and unknown side by side. The alkali should, however, be added in the last and in sufficient quantity, so that the pH of the resulting solution is>12. The sequence for other materials *i. e.*, the test solution, stabilizers and the dye solution is rather immaterial.

The influence of the diverse ions on the determination of magnesium is observed by noting the absorbance of the solutions containing 0.5 mg of magnesium and 0.5 and 2.0 mg of each ion in 25 m*l* of the solution. Al^{3+}, Mn^{2+}, Fe^{2+}, Fe^{3+}, Zn^{2+} interfere, whereas Na^{+}, K^{+}, NH_4^{+}, Sr^{2+}, Ba^{2+}, F^{-}, SO_4^{2-}, Cl^{-}, tartrate and oxalate do not. Calcium does not interfere when present in amount almost equivalent to that of magnesium, whereas in presence of higher concentrations the interference is noted.