Soloehrome Violet R.S. as a New Colorimetric Reagent

Part II*

The Microdetermination of Vanadium

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With 5 Figures in the Text

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Fast Grey R.A. (1-azometahydroxynaphthyl-2-hydroxy-3-nitrobenzene-5-sulphonic acid) among a number of solochrome dyes has approximately the same structural formula as two other solochrome dyes namely solochrome violet R.S. (1-azometahydroxynaphthyl-2-hydroxybenzene-5-sulphonic acid) and solochrome black P.V. (1-azoparahydroxynaphthyl-2-hydroxybenzene-5-sulphonic acid).

The fact that Fast Grey R.A. has proved to be a suitable colorimetric reagent for the microdetermination of vanadium⁴, molybdenum⁵, zirconium⁶, bismuth⁹ and copper² created the interest for testing the behaviour of solochrome violet R.S. towards chelation with a number of metal ions with the object of investigating the properties of this dye as a colorimetric reagent and comparing them with those of Fast Grey R.A., in order to trace the influence of the presence or absence of the nitro group in both compounds. The results of the preliminary experiments led to the conclusion that the present dye holds a good promise as a new colorimetric reagent for the microdetermination of vanadium and molybdenum and consequently, it was the aim of this investigation to study the behaviour of the vanadium-dye complex for the above purpose.

The present method has some advantages in comparison with others; namely the quantitative estimation of 0.05 ppm (and hence the high sensitivity), the use of a Universal Coleman spectrophotometer for determining low vanadium concentrations in the range of 0.05 to 6 ppm, the rapid and simple procedure, and the high specificity of the reagent.

Experimental

All the chemicals used in this investigation were of the highest purity. The water used was always twice distilled.

Solutions. The vanadium solution was prepared by dissolving 0.2296 g of the A.R. ammonium vanadate in one litre of water. The solution was standardized following the recommended procedures by Sandell and Snell and both gave concordant results within the calculated amount. Further dilutions were prepared from this stock solution. Solutions of the interfering cations containing 10 to 100 fold the vanadium content were prepared either by dilution from 0.02 M nitrate solutions or by dissolving the calculated amounts of the solid compounds in the appropriate volumes of water.

Reagent Solution. 0.05 or 0.1 g of the dye is dissolved in 100 ml of water.

Effect of pH. Since solochrome violet R.S. is a sulphonic acid azodye, i.e. a weak acid, its orange red aqueous solution is easily converted to a faint orange solution in a fairly acid medium. By developing the violet color of the vanadium-solochrome violet compound in different acidities using acidic buffer solutions or 0.1 M nitric, hydrochloric or sulphuric acid solutions, it was concluded that the best color is developed in 10 ml of nitric acid per 25 ml.

Effect of Alcohol and Acetone. By developing the colored vanadium-solochrome violet compound in solutions made 30 per cent in respect to ethyl alcohol or acetone it was concluded that both solvents are without influence on the developed color.

Selection of the Suitable Wave Length. On measuring the absorbance of a blank and of a colored solution containing 25 µg vanadium per 25 ml against pure water and against each other (difference curve) with a Universal Coleman spectrophotometer model 14 at the different wave lengths within the range of 400—700 nm, a peak was observed in the latter curve (Fig. 1) corresponding to a maximum absorbance at a wave length of 580 nm in comparison with that of 560 nm exhibiting the maximum absorbance of a East Grey-vanadium complex of the same concentration. On the other hand, the solutions containing each 1 ppm of vanadium exhibited the minimum transmittancy of 63% in presence of solochrome violet and 55.5% in presence of Fast Grey, indicating that the nitro group in Fast Grey has a remarkable influence on the depth of the color of the vanadium Fast Grey complex.

Molecular Structure of the Vanadium-Solochrome Violet Compound. Solochrome violet is an azo-dye with the constitutional formula C_{16}H_{11}O_{5}N_{2}S. In order to investigate the molecular structure of its compound with vanadium, measurements were carried out with a Universal Coleman spectrophotometer and cuvettes of 13.06 optical thickness at a wave length of 580 nm, using 50 µg cation per 25 ml. The sequence of addition followed was 50 µg vanadium + 10 ml 0.1 M nitric acid + 1 ml 10%, hydroxylamine-hydrochloride solution + increasing volumes of freshly