STUDIES ON THE APPLICATION OF SALTS OF COMPLEX CATIONS TO THE MICROSCOPIC DETECTION OF ANIONS.

II. 1,6-DINITRITOTETRAMMINOCOBALTIC CHLORIDE (CROCEOCOBALTIC CHLORIDE).

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It was decided to make the croceo type of cobalt complexes the second in this series of studies, because this was the class of compounds to which we referred in our first paper1 as “differential reagents for certain anions which offer difficulties in separation during their quantitative determinations”2. Croceocobaltic chloride, belonging to the 1,6- or trans class, contains two nitrito...
groups instead of two of the co-ordinated ammonia molecules. Later papers shall deal with the reactions of its spatial isomer, i.e., the flavo, cis or 1,2-series of salts, followed by a consideration of the monosubstitution products.

S. M. Joergensen\(^3\) prepared the croceo salts, studied their reactions, and even suggested the use of croceocobaltic nitrate, \(\text{Co(NO}_2\text{)}_3(\text{NH}_3)_4\text{NO}_3\), as a reagent for the determination of the chromate and dichromate ions. Up to the present, however, we have been unable to find any actual utilization of this complex cation as a reagent.

**Experimental Part.**

The reagent, croceo- or 1,6-dinitritotetramminocobaltic chloride, \(\text{Co(NO}_2\text{)}_3(\text{NH}_3)_4\)\(\text{Cl}\), was prepared according to Biltz, Hall and Blanchard.\(^4\) A saturated aqueous solution (approximately 0.1 M), was used for the reactions. The chloride was selected because the luteo chloride had been previously employed. For purposes of comparison, a drop of the reagent solution was evaporated on the slide. Crystals forming in the drop appeared slightly different from those found after complete evaporation as shown in the composite figure 1.

The sample substances and the technique were the same as those employed in the previous work. Use of cover slips was found to be unnecessary. As in the previous case, crystal formations were checked against those obtained on evaporation of the solutions of the test substances alone, and only those which differed characteristically from the crystals of the reagent and of the test substance were considered.

**Oxalate ion:** A 1% solution of the anion (15.2 mg. of \(\text{Na}_2\text{C}_2\text{O}_4/\text{ml.}\)) reacted with the reagent to form twinned prisms immediately (Fig. 2). These prisms formed in 8 minutes in a 0.1% solution. L. I.: 20 \(\gamma\) of \(\text{C}_2\text{O}_4^-=\).

**Orthovanadate ion:** A 1% solution of the anion (16 mg. of \(\text{Na}_3\text{VO}_4/\text{ml.}\)) gave small cigar-shaped crystals toward

\(^3\) Z. anorg. allg. Chem., 5, 147 (1894).