des Stickstoffes schon vorgebildet ist (Nitro- und Nitrosoverbin-
dungen), werden bis zu 50% des Stickstoffes zu Stickstoffdioxyd
oxydiert. Bei anderen Stickstoffverbindungen dagegen nur 5—30%.

2. Die Menge des gebildeten Stickstoffdioxydes ist, abgesehen
von der Struktur der organischen Verbindung, weitgehend abhän-
gig von der Art der Verbrennungsmethode, zum geringen Teil auch
vom angewandten Katalysator.

3. Die günstigsten Bedingungen für die Mikro-Elementaranalyse
liegen in der Anwendung von 0,2 bis 0,3 g trockenen Bleidioxydpulver im Schiffchen. Bei Überlastung des Präparates zeigt sich
die Verminderung seiner Wirksamkeit zuerst in einem allmählichen
Ansteigen der Wasserstoffwerte, später, bei stärkerem Versagen
des Präparates, durch stark erhöhte Wasserstoffwerte und auch
stark erhöhte Kohlenstoffwerte.

STUDIES ON THE APPLICATION OF SALTS OF COM-
PLEX CATIONS TO THE MICROSCOPIC DETECTION
OF ANIONS.

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

By

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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 paper as “differential
reagents for certain anions which offer difficulties in separation
during their quantitative determinations”. Croceocobaltic chloride,
belonging to the 1,6- or trans class, contains two nitrito

1 W. A. HYNES and L. K. YANOWSKI, Mikrochemie, in press.
2 H. T. YAGODA, private communication.
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 prepared the croceo salts, studied their reactions, and even suggested the use of croceocobaltic nitrate, \( \text{Co(NO}_2\text{)}_2(\text{NH}_3)_4\text{NO}_3 \), 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, \( \text{Co(NO}_2\text{)}_2(\text{NH}_3)_4\text{Cl} \), was prepared according to Biltz, Hall and Blanchard. 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 \( \text{Na}_2\text{C}_2\text{O}_4\text{}/\text{ml.} \)) 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 \( \text{Na}_3\text{VO}_4\text{}/\text{ml.} \)) gave small cigar-shaped crystals toward