

the solution alkaline with dilute sodium hydroxide, the characteristic sky-blue precipitate of the magnesium lake appears. If the amount of magnesium is very small, a drop of the stock reagent which has been diluted 4 to 5 times with water gives better results. In this case also the lake is rather slow in forming and shaking for two to three minutes is necessary to coagulate the precipitate.

Professor D. P. Smith of this University found that an excess of ammonium salts destroys the sensitivity of the reagent; hence in the regular course of analysis, the ammonium salts are "smoked off" in a casserole before applying the test.

Nickel and cobalt also give somewhat similar blue lakes which may lead to confusion. After the removal of the heavy metals by precipitation as sulfides, however, the test is absolutely characteristic and the confusion attending the use of the classical magnesium ammonium phosphate precipitate as a test, especially in elementary courses of analysis, is obviated.

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**The Preparation of Selenic Acid and its Salts.**—A number of methods for the preparation of selenic acid have been reported in the literature.<sup>1</sup> As a rule the salts of this acid have been prepared by the oxidation of the selenites.

Meyer and Heider<sup>1a</sup> treated selenium dioxide with 30% hydrogen peroxide, warming the mixture on the water-bath and upon analysis found that 48% of the oxide had been oxidized to selenic acid. In another experiment using a larger amount of hydrogen peroxide an analysis showed an oxidation of 70%.

The experiments of Meyer and Heider suggested that it might be worth while to try to find the best conditions for preparing selenic acid, and possibly the selenates, by the hydrogen peroxide method.

A number of experiments were carried out, treating weighed amounts of selenium dioxide with varying amounts of hydrogen peroxide. The time and the temperature of the treatment were varied. Oxidation values all the way from 20 to 70% were obtained. Small amounts of the oxide

<sup>1</sup> The methods for the preparation of selenic acid and its salts are found in the following references: (a) Meyer and Heider, *Ber.*, **48**, 1154 (1915); (b) Mitscherlich, *Pogg. Ann.*, **9**, 623 (1827); (c) Wohlwill, *Ann.*, **114**, 169 (1860); (d) *Wien Akad. Ber.*, **39**, 299 (1860); (e) Thomsen, *Ber.*, **2**, 598 (1869); (f) Diemer and Lenher, *J. Phys. Chem.*, **13**, 505 (1909); (g) Müller, *Ber.*, **36**, 4262 (1903); (h) *Chem.-Ztg.*, **31**, 630 (1907); (i) Meyer and Moldenhauer, *Z. anorg. Chem.*, **116**, 193 (1921); (j) Worsley and Baker, *J. Chem. Soc.*, **123**, 2870 (1923).

were then refluxed with 30% hydrogen peroxide, with the result that the oxidation values were much higher. These results were reproducible as is shown in the following table.

TABLE I  
RESULTS OF EXPERIMENTS

Selenium dioxide, g.	0.2019	0.2000	0.2029
Hydrogen peroxide, 30%, cc.	10	10	10
Time of refluxing, hours	2	2	2
Oxidation, %	89.5	90.7	91.9 *

A quantity of selenic acid was prepared, adhering to the conditions shown in Table I. The small amount of selenious acid was reduced to selenium, filtered off and the filtrate concentrated, following the procedure of Cameron and Macallan.<sup>2</sup> In this manner selenic acid of high purity with a concentration of 84% was readily prepared.

Since hydrogen peroxide proved so effective in oxidizing selenious acid, it seemed desirable to try its use for the oxidation of selenites to selenates. Copper selenite was prepared by treating copper carbonate with selenious acid. The green, insoluble copper selenite remained. This salt was covered with 30% hydrogen peroxide and refluxed for three hours, during which time it was oxidized to the blue, soluble selenate. A small amount of the insoluble selenite was filtered off, leaving a clear blue filtrate from which copper selenate crystals formed, identical in appearance to those of copper sulfate. Using the same procedure selenates of nickel, cobalt, aluminum and magnesium were prepared. All of these salts were obtained in a well defined, crystalline condition, similar to the corresponding sulfates.

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**Use of Cresol Red in Acid Solutions.**—As has been pointed out by Clark<sup>1</sup> and Cohen,<sup>2</sup> the sulfonephthalein indicator dyes undergo two distinct color changes at different points on the  $P_H$  scale. However, of this series only thymol blue and meta cresol purple have been applied as indicators in both acid and alkaline solutions and the fact that the sulfonic acid dissociation of cresol red is sufficiently repressed so that it can be used in acid solutions has not been emphasized. This indicator is really

<sup>2</sup> Cameron and Macallan, *Chem. News*, **59**, 219 (1899).

<sup>1</sup> Clark, "The Determination of Hydrogen Ions," Williams and Wilkins Co., Baltimore, Md., 1922, p. 93.

<sup>2</sup> Cohen, "U. S. Public Health Reports," **41**, 3051-3074 (1926).