perhaps be the stable one in solution or in the liquid state; for the intermolecular forces operating during the process of crystallization may be sufficient to turn all the CH_2OH groups in the same direction and so transform the tetrahedral arrangement into the one actually found. It is of interest, however, that recent investigations[7] on thin films of the tetrapalmitate of penta-erythritol also indicate a square arrangement rather than a tetrahedral one of the CH_2OR groups around the central carbon atom of each molecule.

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Summary

Additional data confirming the crystal structure of penta-erythritol, $C(CH_2OH)_4$, as previously reported by Mark and Weissenberg, have been obtained. These data verify the non-tetrahedral arrangement of atoms about the central carbon atom of the molecule.

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NOTES

The Hydrolysis of Sulfur Monochloride.—In the discussion of the products resulting from the hydrolytic decomposition of sulfur monochloride the current standard textbooks of chemistry give the reaction[1] as

$$2S_2Cl_2 + 3H_2O = 2S + 4HCl + H_2S_2O_3 \qquad (1)$$

A simple qualitative examination, however, shows immediately the presence of hydrogen sulfide in the vapors issuing from the reaction flask and in the alkaline solution in which those vapors are fixed. In view of this fact, therefore, the primary reaction must be represented by the equation

$$S_2Cl_2 + 2H_2O = 2HCl + H_2S + SO_2 \qquad (2)$$

which obviously is followed by the reactions usually given but which are, nevertheless, distinctly secondary.

A search of the literature shows that Carius[2] was apparently the first and only investigator to note the presence of hydrogen sulfide. Using the notation of his day he wrote "the action of 'half sulfurchloride' on water is analogous to that on alcohol.

$$(\ominus\ H)_2 + Cl_2SS = (ClH)_2 + S\ \ominus\text{''} \qquad (3)$$

His results have evidently been overlooked for the period of nearly three-quarters of a century since they were published.

[7] Adams and Dyer, *Proc. Roy. Soc.* (*London*), **106A**, 706 (1924).

[1] For example, see Mellor, "Modern Inorganic Chemistry," Longmans and Co., **1925**, p. 500.

[2] Carius, *Ann.*, **106**, 291–336 (1858); *Pharm. Centrallbl.*, **1858**, 545.

Aside from its historical aspect the reaction is interesting in its bearing on the structure of the substance which is probably $S{=}S < Cl_2$ and not Cl—S—S—Cl as given by Gooch and Walker.[3]

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A New Confirmatory Test for Aluminum.—When solutions of aluminum salts are made alkaline with ammonium hydroxide in the presence of tincture of alkanet root,[1] the precipitated lake is royal purple in color and rises rapidly to form a compact mass at the surface of the liquid, leaving the solution colorless. It has been found that the volume in cubic centimeters of precipitate obtained may be taken as numerically equal to the milligrams of aluminum present, with an error not exceeding 10%. These facts have been employed in working out a confirmatory test for aluminum for use in qualitative analysis.

The test is made by dissolving the supposed precipitate of aluminum hydroxide, or a small portion of it if it is large, on the filter with 6 *N* sulfuric acid, and receiving the filtrate in a graduate. Five to ten cc. of water is next added and enough alkanet tincture[2] to make the solution ruby red. Next, 6 *N* ammonium hydroxide solution is added gradually during strong shaking until the red color changes to blue, indicating alkalinity; 3–5 cc. of ammonium hydroxide is then added in excess, and the graduate set aside for five minutes. The rise to the surface of the brilliantly colored precipitate which affords a means of semi-quantitative estimation, the function of the dye principle as an indicator, and the decolorization of the solution make this a striking and instructive test; 0.1 mg. of aluminum may be readily detected.

Under the conditions of the test, silicic acid and quadrivalent titanium give gray-blue precipitates and stannic tin forms a red-brown precipitate. Comparison with a test aluminum precipitation makes confusion impossible in these cases. Silicic acid may be further differentiated from aluminum by its failure to redissolve on acidification after precipitation. Chromium, iron, stannous tin, mercury, bismuth and lead give precipitates which offer no possibility of confusion with that of aluminum. Zinc, silver, copper, cadmium, cobalt, nickel, arsenic, antimony and manganese remain in solution.

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[3] Gooch and Walker, "Outlines of Inorganic Chemistry," MacMillan Co., **1905**, p. 270.

[1] "Dispensatory of the United States," 20th ed., p. 1235. Formánek, *Z. anal. Chem.*, **39**, 416 (1900).

[2] Alkanet root is obtainable from standard dealers. The tincture is prepared by extraction of the pulverized root with 95% alcohol.

A confirmatory test for an element need not necessarily be applicable in the presence of other elements. The nature of this procedure indicates that all substances giving flocculent precipitates in ammoniacal solution would interfere with the true aluminum color and with the semi-quantitative estimation when present in sufficient amounts. In general, three equivalents of aluminum in the presence of one equivalent of such a substance give a good aluminum color. It is interesting to note that precipitation of aluminum under the conditions of the test in the presence of the blue nickel-ammonia ion gives a deep blue rather than the characteristic purple precipitate, whereas in the presence of the blue cupric-ammonia ion, the precipitate has its normal color.

This test has been used successfully in the course in qualitative analysis at the University of Arizona. The writers feel it to be superior to the cobalt aluminate test with regard to definiteness, semi-quantitative aspect, and ease of application. Tests have been made with the ammonium salt of aurin-tricarboxylic acid, proposed as a new reagent for aluminum,[3] and results as recorded for this substance have been obtained. However, in no case could the precipitate so formed be made to rise to the surface and afford the opportunity for semi-quantitative estimation as herein described for the alkanet lake.

CONTRIBUTION FROM THE
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THE BECKMANN REARRANGEMENT INVOLVING OPTICALLY ACTIVE RADICALS[1]

BY LAUDER W. JONES AND EVERETT S. WALLIS

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Introduction

Extensive investigations of certain halogen amides, acid azides and hydroxamic acids which undergo rearrangement led Stieglitz[2] to propose that the rearrangements take place in the following manner.

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[3] Hammett and Sottery, THIS JOURNAL, **47**, 142 (1925).

[1] This paper is based upon a thesis submitted by Everett S. Wallis to the Faculty of the Graduate School of Princeton University in partial fulfilment of the requirements for the degree of Doctor of Philosophy.

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[2] Stieglitz, *Am. Chem. J.*, **18**, 751 (1896); *ibid.*, **29**, 49 (1903). Stieglitz and Earle, *ibid.*, **30**, 399, 412 (1903). Stieglitz and Slossen, *Ber.*, **28**, 3265 (1895); *Ber.*, **34**, 1613 (1901). Stieglitz and Leech, THIS JOURNAL, **36**, 272 (1914).