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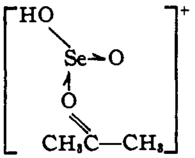

where the acetone has displaced a hydroxyl ion from the selenium. In favor of this structure, one may cite the complete agreement with the kinetic evidence; but also, the subsequent disproportionation of the intermediate to pyruvaldehyde and selenium is most reasonable. The coördination of the acetone through its oxygen necessarily results in a redistribution of charge within the acetone molecule which would result in enhanced reactivity of the α-hydrogens; and it is not unlikely that the bond distances and angles in the complex are such that the selenium oxygens are placed in a favorable steric position for reaction with α-carbon.

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Iodometric Analysis of *o*-Nitrobenzeneseleninic Acid

BY OLAV FOSS

o-Nitrobenzeneseleninic acid has been found to react with thiosulfuric acid as follows

$$o\text{-}O_2NC_6H_4Se(O)OH + 3S_2O_3^{--} + 3H^+ = o\text{-}O_2NC_6H_4SeS_2O_3^{-} + S_4O_6^{--} + 2H_2O$$

The reaction is rapid and quantitative. The reaction product, *o*-nitrobenzeneselenenyl thiosulfate, has previously been isolated as a potassium salt by the reaction of *o*-nitrobenzeneselenenyl bromide with potassium thiosulfate,[1] and is also formed in reactions of various *o*-nitrobenzeneselenenyl sulfur compounds with thiosulfate.[1] It forms yellowish green, stable solutions. In neutral and acid solutions it is indifferent to iodine and does not interfere with iodine–thiosulfate titrations carried out in the same solutions. Hence the above reaction may be utilized for the iodometric analysis of *o*-nitrobenzeneseleninic acid, excess of thiosulfate being back-titrated with iodine. The following procedure may be employed.

To 12–20 mg. of *o*-nitrobenzeneseleninic acid dissolved in 25 ml. of water are added 5 ml. of 0.1 *N* sulfuric acid and then 20–30 ml. of 0.01 *N* sodium thiosulfate (5 ml. excess). After standing for two to three minutes at room temperature, starch is added, and the excess of thiosulfate is back-titrated with 0.01 *N* iodine.

Notice may be made to the analogous method of Norris and Fay[2] for the iodometric analysis of selenious acid, by means of the change

$$SeO_2 + 4S_2O_3^{--} + 4H^+ = Se(S_2O_3)_2^{--} + S_4O_6^{--} + 2H_2O$$

Experimental

The *o*-nitrobenzeneseleninic acid employed was prepared from the trichloride by hydrolysis,[3] and recrystallized from water until colorless.

0.1589 g. of the acid was dissolved to 250 ml. in water in a volumetric flask. Twenty-five ml. was pipetted out and 5 ml. 0.1 *N* sulfuric acid and then 25 ml. 0.01 *N* sodium thiosulfate (consuming 24.79 ml. of 0.01078 *N* iodine) were added. The time of standing was 2, 2, 5, 5, 8, 8 minutes. Amounts of 0.01078 *N* iodine then consumed: 5.95, 5.92, 5.92, 5.93, 5.92, 5.93 ml., respectively. Average: 5.93 ml., which gives 0.1585 g. of *o*-nitrobenzeneseleninic acid, *i.e.*, 99.7%.

Indifference of *o*-nitrobenzeneselenenyl thiosulfate to iodine. To 25 ml. of 0.01 *N* sodium thiosulfate were added 5 ml. 10% acetic acid, 5 ml. of starch solution, and (a) 20 ml. of water, (b) 20 ml. of 0.01 *M* potassium *o*-nitrobenzeneselenenyl thiosulfate. The solutions were then titrated with 0.01 *N* iodine. Amounts consumed: (a) 24.49, 24.50 ml. (b) 24.50, 24.50, 24.49 ml.

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[1] Foss, THIS JOURNAL, **69**, 2236 (1947).

[2] Norris and Fay, *Am. Chem. J.*, **18**, 703 (1896); **23**, 119 (1900).

[3] Behaghel and Seibert, *Ber.*, **66**, 708 (1933).

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Mixed Crystal Formation in Linear Copolyesters

BY C. S. FULLER

Slagle and Ott[1] showed that solidified mixes of normal aliphatic acids frequently exhibit a single X-ray spacing along the direction of the molecule axes in the mixed crystal. The purpose of this note is to point out that an analogous behavior occurs in copolymers in which the constituents are ethylene glycol dibasic acid residues of different chain lengths and, presumably, are randomly distributed along the long chain molecules.

A copolyester capable of being cold drawn into strong fibers was prepared by treating equal molar quantities of pure 10-, 11- and 12-membered aliphatic dicarboxylic acids with ethylene glycol. The X-ray fiber diagram of the copolymer (Fig. 1 (a)) is similar to that exhibited by many simple linear polyesters.[2] A mixed crystal is therefore indicated.

The average fiber period for the copolyester measures 17.83 Å. This value was obtained from the three strongest layer-line reflections (17.78, 18.00, 17.70). The fiber periods of the constituent ethylene polyesters prepared from the individual pure dibasic acids according to the same procedure are as follows

Ethylene decane 1-10 polyester	16.86 Å.
Ethylene undecane 1-11 polyester	36.32 (18.16 Å.)
Ethylene dodecane 1-12 polyester	19.40 Å.

The odd number of chain atoms in the 1-11 polyester causes a doubling of the fiber period. In addition, as the fiber pattern of Fig. 1b shows, the polar layers are perpendicular to the fiber axis unlike the even polyesters in which these planes are inclined to the axis.[3] It is noteworthy that (insofar as we can speak of polar layers in the copolymer) the pattern of the copolyester exhibits

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[1] Slagle and Ott, THIS JOURNAL, **55**, 4396 (1933).

[2] Carothers and Hill, *ibid.*, **54**, 1579 (1932).

[3] Fuller and Frosch, *J. Phys. Chem.*, **43**, 323 (1939).