

Table 1. Factor 3-activity of 6-selenoctic acid

Amount in diet Com- pound $\mu\text{g } \%$	Sele- nium $\mu\text{g } \%$	No. of animals	No. of dead animals *	Protect- ion %	ED <sub>50</sub> $\mu\text{g Se}/$ 100 g diet
0	0	30	27		
3.8	2	20	17	16	
5.7	3	10	6	43	3.8
7.6	4	10	5	57	
11.4	6	10	2	83	
19	10	10	0	100	

\* The experiments were terminated on the 30th day.

of the Factor 3 assay using a vitamin E and Factor 3-free 30 % *Torula* yeast ration<sup>10</sup>, and weaning, inbred Fischer 344 rats<sup>11</sup> have been specified elsewhere. For evaluation of the results, the average velocity of death from liver necrosis ( $V_{st}$ ) was calculated from individual survival times<sup>12</sup>. The effects were expressed in "% protection", i.e., in per cent reduction of the velocity as compared to that of the control groups (% protection =  $100 - 100 \times V_{st} \text{ exp.} / V_{st} \text{ contr.}$ ). Weighted averages were used, and the ED<sub>50</sub> was computed from those results which fell on the ascending part of the dose response curve (between 15 and 85 % protection). The assay method, set up twice weekly with 10–15 groups of 10 rats each, yields very consistent ED<sub>50</sub>'s for selenite, Factor 3 and other compounds tested.

- Schwarz, K. *Proc. Soc. Exptl. Biol. Med.* **78** (1951) 852.
- Eggert, R. G., Patterson, E., Akers, W. T. and Stokstad, E. L. R. *J. Animal Science* **16** (1957) 1037.
- DeWitt, W. B. and Schwarz, K. *Experientia* **14** (1958) 28.
- Schwarz, K., Bieri, J. G., Briggs, G. M. and Scott, M. L. *Proc. Soc. Exptl. Biol. Med.* **95** (1957) 621.
- Patterson, E. L., Milatroy, R. and Stokstad, E. L. R. *Proc. Soc. Exp. Biol. and Med.* **95** (1957) 621.
- Schwarz, K. and Foltz, C. M. *J. Am. Chem. Soc.* **79** (1957) 3292.
- Bergson, G. *Acta Chem. Scand.* **11** (1957) 1607.
- Schwarz, K. and Foltz, C. M. *Federation Proc.* **17** (1958) 492.
- Schwarz, K. and Foltz, C. M. *J. Biol. Chem. In press.*
- Schwarz, K. *Proc. Soc. Exptl. Biol. Med.* **77** (1951) 818.

*Acta Chem. Scand.* **12** (1958) No. 6

- Jay, G. E., Jr., National Vitamin Foundation: *Rat Quality, A Consideration of Heredity, Diet and Disease*, New York, (1953) p. 98.
- Schwarz, K. *Proc. Soc. Exptl. Biol. Med.* **80** (1952) 319.

Received June 13, 1958.

## Preparation of Monoperphthalic Acid

S. LINHOLTER and P. SØRENSEN

*Department of Organic Chemistry, University of Technology, Copenhagen, Denmark*

The method for preparing monoperphthalic acid by oxidising phthalic anhydride with an alkaline solution of hydrogen peroxide, first suggested by Böhme<sup>1</sup>, gives according to *Organic Syntheses*<sup>2</sup> normally 65–70 % yield. We found, however, this reaction to be somewhat capricious, sometimes giving only 40–50 % yield. By slightly modifying the procedure we succeeded in eliminating the capriciousness, and at the same time the yield was raised to 90–95 %.

The essential problem is to keep the solution sufficiently cool during the reaction. Operating the same charge as indicated in *Organic Syntheses* we cooled the solution in a carbon dioxide-acetone mixture and maintained a temperature below  $-5^\circ$  during the whole reaction. At this temperature a crystal-layer will cover the walls of the reaction vessel, thus preventing the transmission of the heat of reaction to the cooling mixture. It is, therefore, necessary constantly to scrape down the crystals from the walls, which may easily be effected when using a beaker as reaction vessel instead of a flask. Using this procedure no foaming is observed and practically no phthalic acid was formed.

After the addition of sulphuric acid the temperature is allowed to rise to  $0^\circ$  and enough water is added to dissolve the inorganic material. The solution is extracted 5 times with ether, the ether-solution washed 3 times with ammoniumsulphate solution and dried over magnesiumsulphate.

The yield, ranging from 90 to 95 %, was estimated iodometrically as indicated in *Organic Syntheses*.

- Böhme, H. *Ber.* **70** (1937) 379.
- Böhme, H. *Org. Syntheses* **20** (1940) 70; Coll. Vol. **3** (1955) 619.

Received July 2, 1958.