PHYSICAL CONSTANTS

Molecular weight	336.85
Crystalline form and color	Amorphous white powder; slightly hygroscopic
Melting point	Caramelization begins at 270°
Solubility (g./100 ml.)	
Water, 20°	8.9
Water, 100°	21.5
95% ethyl alcohol, 20°	1.5
Ether (anhydrous), 20°	0.015
Acetone (anhydrous), 20°	0.004

As a saturated water solution cools the lactate does not crystallize. On evaporation the solution is concentrated to a semiviscous fluid containing about 100 g. lactate per 100 ml. at which time the salt begins to separate as a white amorphous floc.

In the presence of small amounts of lactic acid, gallium lactate crystallizes as thin sheets or plates when a water solution is evaporated. These crystals contain water of hydration which is lost on vacuum desiccation at 20°.

When gallium lactate is dissolved in water (25 mg./ml.) the resulting solution has a *p*H of 2.7. A solution suitable for physiological studies is prepared by dissolving the gallium lactate in boiling water, cooling and adding dilute ammonium hydroxide slowly with constant stirring to prevent any localized increase in the *p*H of the solution above 7.6. The *p*H is adjusted to 7.0–7.4 by this means. When the *p*H exceeds 8 the lactate is decomposed and if the *p*H is then lowered to 6 or 7 a gelatinous precipitate of $Ga(OH)_3$ separates.

The lactate causes no precipitation of proteins from fresh horse serum or from egg albumen solutions at *p*H 6–7. Injection subcutaneously or intravenously is tolerated by rats and rabbits with little or no localized reaction if the concentration is less than 25 mg./ml. The acutely toxic dose (LD50, 10 days) for rabbit on subcutaneous injection is 480 mg./kg. body weight. Solutions at *p*H of 7.6 and lower are not decomposed during autoclaving and sterilization although a slight increase in acidity does occur. These solutions must be stored in the cold to avoid possible mold formation.

NAVAL MEDICAL RESEARCH INSTITUTE
NATIONAL NAVAL CENTER
BETHESDA, MARYLAND

RECEIVED JULY 17, 1948

Crystalline 1,4-Anhydro-D-glucitol Tetraacetate

BY HEWITT G. FLETCHER, JR. AND CATHERINE M. SPONABLE

In a recent publication Bashford and Wiggins[1] have reported the preparation of 1,4-anhydro-D-glucitol tetraacetate as a pure sirup. Several years ago using the following procedure we obtained the same substance in crystalline form.

Eighty-four grams of powdered 1,4-anhydro-D-glucitol,[2] m. p. 113–115° (cor.), was added to a mixture of 200 ml. of acetic anhydride and 200 ml. of pyridine at 0°. After one-half hour at 5° and twenty-four hours at room temperature the reaction mixture was poured on ice and extracted with chloroform. The chloroform solution was washed with aqueous sodium bicarbonate and then with water. Removal of the solvent *in vacuo* gave a sirup which on standing in aqueous alcoholic solution at 5° eventually gave crystalline material. The product was, however, more readily purified by distillation at 2 mm. pressure and a bath temperature of 175–185°, the colorless distillate crystallizing spontaneously. Recrystallization from aqueous alcohol and from a mixture of benzene and heptane afforded large, clear rectangular prisms melting at 52–54° (cor.) and rotating in chloroform $[\alpha]^{20}_{D} + 47.5°$ (*c*, 4.08).

Anal. Calcd. for $C_{14}H_{20}O_9$: C, 50.60; H, 6.07. Found: C, 50.62; H, 6.07.

Dr. Wiggins informs us that nucleation with this material caused his sirupy 1,4-anhydro-D-glucitol tetraacetate to crystallize completely.

DEPARTMENT OF CHEMISTRY
MASSACHUSETTS INSTITUTE OF TECHNOLOGY
CAMBRIDGE 39, MASSACHUSETTS

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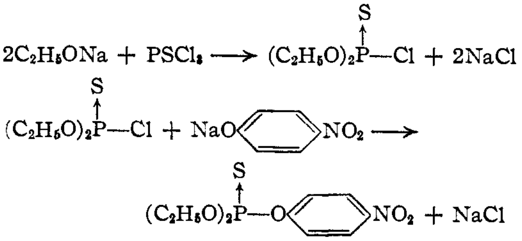
Preparation of O,O-Diethyl O-*p*-Nitrophenyl Thiophosphate (Parathion)

BY JOHN H. FLETCHER, J. C. HAMILTON, I. HECHENBLEIKNER, E. I. HOEGBERG, B. J. SERTL AND J. T. CASSADAY

O,O-Diethyl O-*p*-nitrophenyl thiophosphate is reported by Martin and Shaw[1] and by Thurston[2] to possess interesting insecticidal properties, but these authors do not give detailed directions for its preparation, nor have we found this information elsewhere in the literature. We have synthesized this compound according to the general scheme proposed by German chemists[2] and have found certain modifications to be advantageous. The reactions involved are



$$2C_2H_5ONa + PSCl_3 \longrightarrow (C_2H_5O)_2\overset{\overset{S}{\uparrow}}{P}-Cl + 2NaCl \qquad (1)$$

(2)

Sodium ethoxide (from 46 g. of sodium) in ethanol (1200 cc.) was added during three and one-half hours to a stirred solution of thiophosphoryl chloride (169.5 g.) in benzene (450 cc.), the reaction temperature being held at 5–10°. After standing several hours, the mixture was concentrated *in vacuo* to a thick slurry, and benzene (200 cc.) and water (450 cc.) were added. After shaking and separating, the aqueous layer was again extracted with benzene; the combined benzene extracts were washed with water, dried over Drierite, and concentrated *in vacuo*. Distillation of the residue using a Vigreux column gave 94 g. (50% yield) of colorless O,O-diethyl chlorothiophosphate, b. p. 71.5–72° (7 mm.), n^{25}_{D} 1.4684. Mastin and co-workers[3] give the boiling point as 96–99° (25 mm.).

Anal. Calcd. for $C_4H_{10}ClO_2PS$: Cl, 18.8; P, 16.4; S, 17.0. Found: Cl, 18.3; P, 16.2; S, 17.1.

An experiment in which the second reaction above was run at 125° in chlorobenzene, as proposed by the Germans, gave a 79% yield of parathion after fifty-one hours. We

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[1] V. G. Bashford and L. F. Wiggins, *J. Chem. Soc.*, 299 (1948).

[2] We are indebted to the Atlas Powder Company for a sample of this material for which the trivial name arlitan has been proposed. *Cf.* S. Soltzberg, R. M. Goepp, Jr., and W. Freudenberg, THIS JOURNAL, **68**, 919 (1946); R. C. Hockett, M. Conley, M. Yusem and R. I. Mason, *ibid.*, 922.

[1] Martin and Shaw, BIOS Final Report No. 1095, Item 22, May–June 1946 (PB-78244).

[2] Thurston, FIAT Final Report No. 949, October 14, 1946 (PB-60890).

[3] Mastin, Norman and Weilmuenster, THIS JOURNAL, **67**, 1662 (1945).