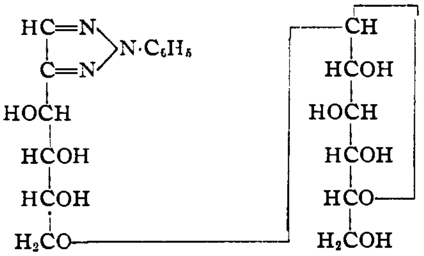
vacuum but is eliminated *in vacuo* at elevated temperatures. It is also unique in that its heptaacetate and heptabenzoate crystallize readily whereas, among the other disaccharide phenylosotriazoles investigated, only one crystalline acyl derivative has been obtained (cellobiose phenylosotriazole heptaacetate). Acid hydrolysis of gentiobiose phenylosotriazole produces a high yield (93%) of D-glucose phenylosotriazole and D-glucose (65%) as expected from the accompanying formula.



Gentiobiose Phenylosotriazole

We are indebted to Mr. Charles A. Kinser and Mrs. Betty Mount for the microchemical analyses.

Experimental

Gentiobiose Phenylosotriazole.—To a suspension of 10 g. of gentiobiose phenylosazone[3] in 900 ml. of boiling water was added a hot solution of 5.3 g. (1.1 molecular equivalents) of copper sulfate pentahydrate in 100 ml. of water. Solution of the phenylosazone took place rapidly and after refluxing for thirty minutes the solution was cooled, filtered, and the excess copper removed from the filtrate as the sulfide; the copper-free solution was neutralized with 10 g. of barium carbonate and following filtration, concentrated *in vacuo* to a thick reddish sirup. The sirup was dried by successive evaporations with three 25-ml. portions of absolute alcohol, and dissolved in 25 ml. of warm absolute alcohol, filtered to remove a small amount of inorganic contaminant and diluted with 35 ml. of ether; upon scratching, the product separated as somewhat gelatinous flocs which were recovered by filtration and washed with cold absolute alcohol and ether; yield 5.3 g. An additional 1.2 g. of product was obtained by concentration of the mother liquor; total yield 6.5 g. (71%). The material was recrystallized from 5 parts of absolute alcohol forming minute needles which melted at 91–93°[4] with foaming and rotated −34.3° in aqueous solution (*c*, 0.83). Analyses showed that the material contained one molecular equivalent of ethanol of crystallization which was retained tenaciously *in vacuo* at temperatures below the sintering point; at 97° *in vacuo* the alcohol was removed leaving an amorphous glassy material of no definite melting point; its analysis corresponded closely to that of an unsolvated disaccharide phenylosotriazole. The crystalline alcoholate was readily soluble in water, pyridine and hot alcohol and sparingly soluble in ether, acetone, ethyl acetate and cold alcohol. Upon separating from solution in impure form it shows a marked tendency to form gelatinous precipitates rather than discrete crystals.

Anal. Calcd. for $C_{18}H_{25}N_3O_9 \cdot C_2H_5OH$: C, 50.73; H, 6.60; loss on drying, 9.7. Found: C, 50.94; H, 6.39; loss on drying, 9.5.

Anal. (of the alcohol-free amorphous material). Calcd. for $C_{18}H_{25}N_3O_9$: C, 50.58; H, 5.90. Found: C, 50.56; H, 5.99.

On refluxing 1.2 g. of the crystalline alcoholate with 50 ml. of 0.5 *N* hydrochloric acid for six hours and chilling the solution, a 93% yield of D-glucose phenylosotriazole (m. p. 195–196°) was obtained and the concentrated aqueous mother liquor, after neutralization with silver carbonate, yielded 65% of D-glucose ($[\alpha]^{20}_D$ +52.5°).

Gentiobiose Phenylosotriazole Heptaacetate.—A solution of 1.0 g. of gentiobiose phenylosotriazole monoalcoholate in a mixture of 8 ml. of acetic anhydride and 0.25 g. of fused sodium acetate was heated on the steam bath for two hours, cooled, and poured into ice water; the crystalline heptaacetate (1.6 g., quantitative) was recrystallized from 10 parts of alcohol and formed clumps of very fine needles melting at 144–146° and rotating −28.1° in chloroform solution (*c*, 0.84). It was readily soluble in chloroform, acetone–ether and warm alcohol and nearly insoluble in water and hexane.

Anal. Calcd. for $C_{32}H_{39}N_3O_{16}$: C, 53.26; H, 5.45; CH_3CO, 41.8. Found: C, 53.53; H, 5.62; CH_3CO, 41.6.

Gentiobiose Phenylosotriazole Heptabenzoate.—To a solution of 0.5 g. of gentiobiose phenylosotriazole monoalcoholate in 5 ml. of pyridine was added 2 ml. of benzoyl chloride; after standing at 25° for twenty-four hours the mixture was poured into ice water and the gummy precipitate washed by decantation with dilute sodium bicarbonate solution and water. The damp precipitate was digested on the steam-bath with 25 ml. of alcohol when spontaneous crystallization occurred; the cooled mixture was filtered and washed with alcohol yielding 1.2 g. (quantitative) of the heptabenzoate. The compound was recrystallized by dissolving it in 10 parts of chloroform and adding 20 parts of hexane or 50 parts of absolute alcohol; it crystallized as fine, short needles melting at 122–123° and rotating +1.5° in chloroform solution (*c*, 0.88). It is soluble in chloroform, ether, acetone and pyridine and sparingly soluble in water, hexane and hot alcohol.

Anal. Calcd. for $C_{67}H_{53}N_3O_{16}$: C, 69.60; H, 4.62; C_6H_5CO, 63.6. Found: C, 69.40; H, 4.57; C_6H_5CO, 63.3.

CONTRIBUTION FROM THE
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The Separation of Iodine-131 from Tellurium

BY MILTON LEVY, ALBERT S. KESTON AND SIDNEY UDENFRIEND

The irradiated unit[1] supplied by the U. S. Atomic Energy Commission as a source of I^{131} contains 50 g. of tellurium. The chromic or nitric acid oxidations such as have been used in working up cyclotron targets proved cumbersome in the case of the pile units because of the large amount of tellurium. The following method based on fusion with sodium hydroxide has proven rapid and reliable.

The tellurium powder is transferred to a 100-ml. Pyrex kjeldahl flask (through a powder funnel) containing 5 g. of sodium hydroxide pellets. The

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[3] Berlin, THIS JOURNAL, 48, 1107 (1926).

[4] The melting points were made with the stem of the thermometer fully immersed in the heated bath. The rotations are specific rotations $[\alpha]^{20}_D$; the tube length was 4 dm. and *c* is the concentration in grams in 100 ml. of solution. All the crystalline compounds were recrystallized to constant m.p. and rotation.

[1] Catalog item No. 37, "Radioisotopes," Catalog No. 2, revised September, 1947. U. S. Atomic Energy Commission, Oak Ridge, Tenn.

mixture is shaken and heated gently to drive out water and then to a dull red (above 452°, the m. p. of tellurium). The mixture is held at this high temperature for about five minutes with occasional shaking during the progress of heating. The flask is allowed to cool somewhat and then plunged into 100 ml. of water in a large mortar. The flask breaks and both it and the solid contents are coarsely powdered with a pestle. The aqueous layer with suspended tellurium is transferred to a liter erlenmeyer flask with suction and the residue in the mortar extracted with several 50-ml. portions of hot water until only minor amounts of radioactive materials are being transferred. The extracts and washings are heated to boiling to coagulate the precipitate. The solution is cooled and filtered to remove tellurium.

The remainder of the isolation requires an all-glass-standard taper distilling apparatus consisting of a 500-ml. flask, an adapter with addition tube, an adapter and condenser and a receiving adapter arranged to dip into a receiving solution, The filtered alkaline extract is run into the distilling flask, which contains a few boiling stones, and evaporated to a volume of about 50 ml. The distillate contains no radioactive material and is discarded. A concentrated solution of potassium permanganate is added in excess to oxidize all iodine in the alkaline solution to iodate. The receiving flask is changed to one containing a dilute solution of sodium sulfite and sodium carbonate (about 1 mg. of each in several ml. of water). The delivery tip is covered by this solution. Fifty ml. of concentrated sulfuric acid is added through the addition tube. Care is necessary because of the heat developed. After the sulfuric acid has been added, 1 g. of oxalic acid dissolved in a minimal amount of hot water is added. The iodate is reduced to iodine. The mixture is now distilled until substantially all of the radioactivity has collected in the receiver where the iodine is converted to iodide. The solution of iodide containing some carbonate, sulfite and sulfate is used directly in most of our work.

The tellurium residue when dissolved in nitric acid containing a small amount of chloride ion, gave, in the presence of excess dissolving silver, a silver chloride precipitate which contained relatively little radioactivity. When unirradiated tellurium to which I^{131} has been added is dissolved in this way, substantially all the radioactivity is coprecipitated with the silver chloride. Portions of the irradiated tellurium gave large amounts of I^{131} in the silver chloride precipitate. Since the tellurium residue from the pile unit contained little I^{131} and all other fractions except the final product contained not more than 10% of the radioactivity of the final product, we believe the yield to be 85–95% of the I^{131} originally present.

The exposures of the operators (two) during the working up of a unit by the method is minimized by the use of lead bricks, long handled tongs and rapid handling of materials. On pocket ionization chambers 0.04–0.06 roentgen was registered on the day of processing a unit. This is well below the allowed maximum of 0.1 roentgen per day. Film badges[2] worn during the week including the day of the preparation showed less than ten per cent. of the permitted maximum exposure for the week.

The irradiated tellurium units used in this investigation were supplied by the Clinton Laboratories and obtained on allocation from the U. S. Atomic Energy Commission.

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(2) Film Badge Service, Tracerlab, Inc., Boston, Mass.

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Preparation of *t*-Butylamine

BY D. E. PEARSON, J. F. BAXTER AND K. N. CARTER

Tertiary butylamine has been made by the hydrogenation of 2,2-dimethylethylenimine[1] and by the reaction of *t*-butylmagnesium chloride and methoxyamine.[2] Simple hydrolysis of the readily obtainable *t*-butylurea (E. K.) with aqueous alkali is too slow for satisfactory use. Smith and Emerson[3] accomplished the hydrolysis indirectly by reaction with phthalic anhydride and subsequent hydrolysis of the resulting phthalimide. It has now been found that the amine is readily prepared from *t*-butylurea by saponification in aqueous ethylene glycol solution. The procedure shown has been checked according to "Organic Syntheses" recommendations.

$$(CH_3)_3CHNHCONH_2 + 2NaOH \longrightarrow (CH_3)_3CNH_2 + NH_3 + Na_2CO_3$$

Procedure

A 1-liter round-bottomed, S. T. flask was equipped with an upright condenser, and a glass tubing was led from the top of the condenser to a small flask immersed in ice-water. The trap was unnecessary, if sufficiently cold water was used in the condenser. The flask was charged with sodium hydroxide (60 g., 1.5 moles) dissolved in 75 cc. of water, *t*-butylurea (70 g., 0.6 mole) and 225 cc. of ethylene glycol (practical grade), and the mixture refluxed for four hours. The liquid temperature was 115° when refluxing started and fell to 86° at the end. The *t*-butylurea gradually dissolved, and a gelatinous mass of sodium carbonate was formed. Shorter reflux gave lower yields; longer periods gave no increase. Lower concentrations of ethylene glycol gave proportionately lower yields. The flask was then cooled, equipped for distillation and the fraction boiling at 40–60° was collected in an ice-cooled receiver. The crude amine, including any in the trap, weighed 37–39 g. It was dried with 5–7 g. of solid sodium hydroxide. If a lower, aqueous

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(1) Karabinos and Serijan, THIS JOURNAL, **67**, 1856 (1945); Campbell, Sommers and Campbell, *ibid.*, **68**, 140 (1946).

(2) Jones, *J. Chem. Soc.*, 781 (1946).

(3) Smith and Emerson, THIS JOURNAL, **67**, 1862 (1945).