added to an excess of methylmagnesium iodide over a period of five minutes with rapid stirring. The mixture was hydrolyzed by adding ice and dilute hydrochloric acid. The organic layer was dried and evaporated on the steam-bath. The colorless oil which remained after evaporation was crystallized from petroleum ligroin. The melting point was 75–77°, yield 1.02 g. This compound was not further investigated.

Two *meso* Forms of 2,5-Diphenyl-4-methyl-4-tetrahydropyranol.—Eighty ml. of a benzene solution containing 1.1 g. of *cis*-2,6-diphenyl-4-tetrahydropyrone[7] was added over a period of 20 min. to a solution of an excess of methylmagnesium iodide in ether which was being stirred rapidly. The reaction was allowed to proceed for 20 min. and hydrolyzed with ice and diluted HCl. The organic layer was dried and evaporated on a steam-bath and the residue chromatographed on the Magnesol–Celite column. A fraction of 20 mg. melting at 67–69°, which we believe to be the *meso*-1-compound, was followed by a larger fraction of *meso*-2-compound weighing 140 mg. which melted from 142–145°. This latter fraction proved to be identical with the material from the *t*-butyl alcohol–benzaldehyde reaction by mixed melting point.

2,6-Diphenyl-3,5-dimethyl-4-ethyl-4-tetrahydropyranol.—An excess of ethylmagnesium bromide in 180 ml. of ether was added to 11 g. of 2,6-diphenyl-3,5-dimethyl-4-tetrahydropyrone[8] dissolved in ether. After reaction, the material was hydrolyzed with NH_4Cl solution and the product recovered and crystallized from petroleum ligroin; yield, 9 g., m.p. 177–178°. This substance is identical with that from the acid condensation; the mixed melting point showed no depression.

2,6-Diphenyl-3,4,5-trimethyl-4-tetrahydropyranol.—This compound was prepared as above, substituting methyl Grignard for the ethyl compound; yield 8.5 g., m.p. 158–159°. This compound is not identical with the compound prepared by acid condensation above. The mixed melting point was 138–143°.

Infrared spectra were obtained by using a Nujol mull spread on a salt plate. A Baird model B recording spectrophotometer was used.

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The Synthesis of 6-Thioguanine

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As part of a program devoted to the investigation of antimetabolites of the purine and pyrimidine bases,[1,2] 6-thioguanine (2-amino-6-mercaptopurine) was synthesized. This compound has been found to behave as a purine antagonist similar to 6-mercaptopurine in *Lactobacillus casei*[3–5] and to exhibit activity against a number of animal tumors.[6,7]

Thioguanine was prepared in the first instance by the reaction of a suspension of guanine in tetrahydronaphthalene with phosphorus pentasulfide, a reaction which earlier had given satisfactory results with pyrimidines and quinazolines[8] and later was found fruitful in the conversion of hypoxanthine to 6-mercaptopurine.[9] However, erratic results were obtained with guanine; in many instances only unreacted guanine was isolated from the reaction mixture. This was believed to be due to the extreme insolubility of both starting material and product in the solvent, with a resultant dependence on the physical state of the starting material. This interpretation finds support in the superior results obtainable through the use of solvents, such as pyridine, in which a greater solubility is demonstrable.

Experimental

A mixture of 10 g. of finely powdered guanine and 50 g. of powdered phosphorus pentasulfide in 250 ml. of dry pyridine was heated under reflux conditions for 2.5 hours. The pyridine was removed by distillation under reduced pressure and the residue was heated with 200 ml. of water for about ten minutes. After cooling, 100 ml. of concentrated ammonium hydroxide was added and the mixture thoroughly chilled. The insoluble residue and the precipitate of ammonium phosphate was filtered off. The orange filtrate was acidified to *p*H 4 with hydrochloric acid and kept at 4° overnight. The precipitate of crude thioguanine was collected and treated with 200 ml. of 6 *N* ammonium hydroxide. The insoluble residue consisting mainly of guanine was removed by filtration. After removal of most of the excess ammonium hydroxide from the filtrate under reduced pressure, the solution was adjusted to *ca.* *p*H 4 with hydrochloric acid and chilled. Pale yellow needles of thioguanine were collected, washed with water and dried at 110°. This product (3.5 g.) was 93% pure on the basis of its ultraviolet absorption spectrum. A sample was purified for analysis by recrystallization from 1000 parts of hot water. The colorless needles thus obtained did not melt below 360°. Ultraviolet absorption spectrum: at *p*H 1, λ_{max} 258, 347 mμ (E_m 8,100, 20,900); at *p*H 11, λ_{max} 242, 270, 322 mμ (E_m 8,700, 7,200, 16,000).

Anal. Calcd. for $C_5H_5N_5S$: C, 35.9; H, 3.0; N, 41.9. Found: C, 36.0; H, 3.3; N, 41.8.

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The Occurrence of a Sulfuric Acid Ester of Choline in the Mycelium of a Strain of *Penicillium chrysogenum*

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In a recent publication Gordon, *et al.*,[1] stated, that they had found a relatively high quantity of rather loosely bound methionine in a hot water extract of the mycelium of *P. chrysogenum* (Wis 49-133). We too are investigating the sulfur metabolism of this particular strain of *P. chrysogenum* and we wish to report the occurrence of the sulfuric acid ester of choline, $(CH_3)_3N^+$-CH_2-CH_2-O-SO_3^-, in mycelial extracts of this mould. The culture filtrates did not contain this ester.

The presence of ethereal sulfates in culture filtrates of Penicillia has been reported,[2] but so far as we are aware this particular ester has only been found by Woolley and Peterson[3] in the mycelium of *Aspergillus sydowi*.

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