acetic acid the 4-aminothymine separated in needles. This pyrimidine was purified by recrystallization from boiling water and melted at 355°. The rearrangement of the acylurea III into 4-aminothymine IV can also be effected by heating in aqueous solution with magnesium oxide. For analysis the compound was dried at 100° in a vacuum.

Anal. Calcd. for $C_5H_7O_2N_3$: N, 29.79. Found: N, 29.56.

Summary

1. Methyl cyanacetyl-urea can be rearranged to 2,6-dioxy-4-amino-5-methylpyrimidine by the action of alkali.

2. Methyl cyanacetyl-urea, when reduced catalytically in the presence of platinum is converted smoothly into thymine.

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Notes

Preparation of Triethylsilicon Halides

BY E. A. FLOOD

It is commonly supposed that triethylsilicon halides cannot be prepared, readily, from di-triethylsilicon oxide. Nevertheless it has been found that triethylsilicon bromide, chloride and fluoride can be obtained very easily from the oxide by treating a mixture of concentrated sulfuric acid and di-triethylsilicon oxide with the corresponding sodium or ammonium halide. The mixture is allowed to stand for some hours and the product extracted with petroleum ether. The triethylsilicon halide is separated from the solvent by distillation. The yields are practically quantitative.

Triethylsilicon fluoride, prepared in this way, is a colorless mobile liquid having a very faint "camphor-like" odor. It boils at 110° (uncorr.) under atmospheric pressure. It is much more stable toward hydrolysis than is the corresponding bromide and may be distilled in air without appreciable decomposition.

Analyzed for fluorine by a modified Starck method, calcd. for $(C_2H_5)_3SiF$: F, 14.15. Found: F, 14.31, 14.63, mean 14.48.

It was shown to be a single substance as follows. About ten grams was allowed to distil under its own vapor pressure at room temperature into a receiver (volume 302 cc.) cooled to −33°. The vapor density of the first two or three drops of distillate collected was determined, as well as the vapor density of the last two or three drops of residual liquid remaining in the original container. The vapor densities of the two extreme fractions agreed within the experimental error and gave a value for the molecular weight in close agreement with that required by the formula $(C_2H_5)_3SiF$. The data follow: mol. wt. (vapor density). First fraction. Subs., 0.0534, 0.0501; vol.. 302.0; pressure, mm., 24.7, 22.9; *t*, 27.7°, 28.0°.

Mol. wt. 134.3, 136.1, mean, 135.2. Last fraction. Subs., 0.0383, 0.0506; vol., 302.0; pressure, mm., 17.7, 22.9; *t*, 28.1, 28.5; mol. wt. 134.6, 137.6, mean, 136.2. Calcd. for $(C_2H_5)_3SiF$: mol. wt., 134.2.

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A Hydrate of 1,3,5-Xylenoxyacetic Acid

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In a study of phenolic fractions obtained from coal tar, we have found it desirable to prepare derivatives of certain pure phenols for purposes of comparison. One of the derivatives which we prepared was the oxyacetic acid formed by interaction of symmetrical xylenol and chloroacetic acid in the presence of sodium hydroxide. We were especially interested in this compound because Schneider and Shohan,[1] working with a symmetrical xylenol prepared by synthesis, found that their specimen melted at 111° whereas Schütz and collaborators,[2] Steinkopf and Höpner,[3] and Brückner[4] state that its melting point is 85 or 86°. We find this discrepancy to be explained by the fact that the material reported melting at 86° is a monohydrate, the anhydrous substance melting at 111°.

The xylenol which we used was a coal tar product melting at 63.2°, obtained from the U. S. Bureau of Mines. The hydrate of 1,3,5-xylenoxyacetic acid, easily prepared by crystallizing the product from water, melts actually at 81.0°, but is unstable, losing its water of crystallization on standing for a few days over phosphorus pentoxide in a vacuum desiccator at room temperature. The product then melts at 111.2°. A mixture of equal parts of hydrate and anhydrous material melted indistinctly between 97 and 104°. The hydrate loses at least a part of its water when it is dissolved in warm benzene and is reprecipitated by addition of petroleum ether. Schneider and Shohan reported 86–92° as the melting range of their product before recrystallization; they then recrystallized from benzene and found the melting point to be 111°. It is probable that their first product was largely the hydrate and that it lost water when warmed with benzene.

We report the melting point of the hydrate as 81.0° on the basis of the following evidence. The crude reaction product, isolated from aqueous alkaline medium by precipitation with hydrochloric acid, melted at 81–83°. We recrystallized it from water, and allowed it to stand in a desiccator

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[1] Schneider and Shohan, *Bur. Mines, Repts. Investigations* No. **2969**, 1929, 10 pp.

[2] Schütz and co-workers, *Ber.*, **56**, 1971 (1923).

[3] Steinkopf and Höpner, *J. prakt. Chem.*, [2] **113**, 141 (1926).

[4] Brückner, *Erdöl und Teer*, **4**, 562, 580, 598 (1928); *C. A.*, **23**, 1246 (1929); *Z. Angew. Chem.*, **41**, 1043, 1062 (1928).