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

BY ALAN R. ALBRIGHT

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).

in an atmosphere saturated with water vapor at room temperature, until weight equilibrium was reached. It then melted sharply at 81.0° corr. when heated at the usual rate of 1–2° per minute. The value 85 or 86° probably corresponds to a partially dehydrated product; for, if the temperature of the bath be raised very slowly, about 1° in three minutes, the hydrated substance will begin to melt at 81°, but at the same time will gradually become dehydrated; with cautious heating it is possible to reach 111°, the melting point of the anhydrous material, before fusion is complete; but it is also possible with irregular heating to observe melting at almost any point intermediate between 81 and 111°.

The solubility of the anhydrous substance in boiling water is about 1 g. in 100 cc.; in benzene, somewhat more than 1 g. in 10 cc.

Another property of 1,3,5-xylenoxyacetic acid not mentioned in the literature is its volatility in a current of steam. We found that 100 g. of steam in the course of one hour carried over 0.0066 g. weighed as anhydride.

Analysis.—A determination of water of crystallization was made in a micro vacuum drying tube over phosphorus pentoxide at about 70°: weight of sample, 4.395 mg.: H_2O found, 0.395 mg. or 8.99%; calcd. for $C_{10}H_{12}O_3.H_2O$, 9.09%. Micro combustion analysis of the anhydrous material remaining after the water determination gave the following results: weight of sample, 4.000 mg.: CO_2, 9.81 mg.; H_2O, 2.44 mg. C found, 66.88%; calcd., 66.63%; H found, 6.82%; calcd., 6.72%. This microanalysis was made by Mr. F. C. Silbert of this Laboratory.

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Rotenone in a Species of Spatholobus

BY HOWARD A. JONES

Some time ago the Insecticide Division received a sample of roots[1] of a fish-poisoning plant from Burma, which the Economic Botanist of that country considered to be *Spatholobus roxburghii* Benth., a large, woody climber. In the course of tests of numerous plant extracts, Drake and Spies[2] found that an acetone extract of this root material possessed a high toxicity to goldfish. Recent tests by F. L. Campbell of the Bureau of Entomology also indicate that the extract is highly toxic to mosquito larvae.

While testing a number of plant extracts of high fish and insect toxicity for rotenone by the blue color test recently outlined,[3] the author found that the acetone extract of *S. roxburghii* gave a deep blue color, estimated to be

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(1) The material received consisted mainly of the bark of the root.

(2) Drake and Spies, *J. Econ. Entomol.*, **25**, 129 (1932).

(3) Jones and Smith, *Ind. Eng. Chem., Anal. Ed.*, **5**, 75 (1933).

equivalent to at least 1% rotenone or deguelin (both give the test). No further plant material was available. The remainder of the acetone extract, representing 6.3 g. of original material, was evaporated to dryness in a vacuum and the rotenone determined by crystallization from carbon tetrachloride.[4] The amount of material so obtained corresponded to about 1% of rotenone in the original root. This material recrystallized from absolute alcohol gave large, hexagonal plates melting at 163°, with indices of refraction identical with those of pure rotenone.

There are numerous species of *Spatholobus*, most of them native to India, the Malay Peninsula or the Philippine Islands. Since rotenone has now been found in *S. roxburghii* it would be well for investigators in these countries to study the rotenone content of plants of this genus.

[4] Jones, *Ind. Eng. Chem.*, *Anal. Ed.*, **5**, 23 (1933).

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COMMUNICATIONS TO THE EDITOR

THE ADSORPTION OF NITROGEN BY IRON SYNTHETIC AMMONIA CATALYSTS

Sir:

The experimental work of recent years upon the mechanism of ammonia synthesis over iron catalysts has culminated in the idea that the synthesis occurs through nitrogen molecules reacting with the surface atoms of the iron catalyst to form a layer of nitrogen held to the surface by activated adsorption [see Taylor, THIS JOURNAL **53**, 578 (1931)] or a surface iron nitride [see Frankenburger, Ullmann's "Enzyklopadie der technischen Chemie," 1928; Emmett, *J. Chem. Ed.*, **7**, 2571 (1930)] which in turn can be reduced by hydrogen to form ammonia. Various indirect lines of evidence have been advanced in support of this hypothesis. However, as yet no data have been published to show whether the adsorption of nitrogen by the surface of an iron catalyst at synthesis temperatures proceeds at a sufficient rate to permit it to be an essential step in the synthesis of ammonia. The present letter presents briefly the results that have been obtained recently relative to the rate and extent of nitrogen adsorption by various types of iron ammonia catalysts.

At 400° and one atmosphere pressure 10-cc. samples of doubly promoted iron catalyst 931 containing 1.3% Al_2O_3 and 1.59% K_2O, pure iron catalyst 973, and singly promoted catalyst 921 containing 1.31% Al_2O_3 adsorbed