

in an atmosphere saturated with water vapor at room temperature, until weight equilibrium was reached. It then melted sharply at  $81.0^{\circ}$  corr. when heated at the usual rate of  $1-2^{\circ}$  per minute. The value  $85$  or  $86^{\circ}$  probably corresponds to a partially dehydrated product; for, if the temperature of the bath be raised very slowly, about  $1^{\circ}$  in three minutes, the hydrated substance will begin to melt at  $81^{\circ}$ , but at the same time will gradually become dehydrated; with cautious heating it is possible to reach  $111^{\circ}$ , 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  $111^{\circ}$ .

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  $70^{\circ}$ : weight of sample, 4.395 mg.:  $H_2O$  found, 0.395 mg. or 8.99%; calcd. for  $C_{10}H_{12}O_3 \cdot H_2O$ , 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 micro-analysis was made by Mr. F. C. Silbert of this Laboratory.

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## Rotenone in a Species of *Spatholobus*

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Some time ago the Insecticide Division received a sample of roots<sup>1</sup> 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<sup>2</sup> 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,<sup>3</sup> the author found that the acetone extract of *S. roxburghii* gave a deep blue color, estimated to be

(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.<sup>4</sup> 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

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### 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<sub>2</sub>O<sub>3</sub> and 1.59% K<sub>2</sub>O, pure iron catalyst 973, and singly promoted catalyst 921 containing 1.31% Al<sub>2</sub>O<sub>3</sub> adsorbed