Solubility of Strychnine Acid Sulfate in Sulfuric Acid

BY BENJ. M. G. ZWICKER[1] AND REX J. ROBINSON

Information concerning the solubility of strychnine acid sulfate in sulfuric acid was of interest to the authors in the separation of strychnine from some of its reduction products in a 60% (by weight) sulfuric acid medium. Since this information was not available in the literature, it became necessary to determine it experimentally.

Merck U. S. P. XI Strychnine Sulfate was dissolved in 64% sulfuric acid to the extent of 10.5 g. of base per 100 g. of solvent. Portions of this solution were weighed into glass-stoppered bottles. Appropriate quantities of water were added to these aliquots, carefully mixed, warmed and then allowed to attain equilibria at 20°. After several hours, the bottles were reweighed and the crystalline strychnine acid sulfate was separated by decanting through a weighed Jena sintered-glass micro-crucible of porosity II. The strychnine acid sulfate was converted to strychnine by macerating it with 6 *N* ammonium hydroxide. The insoluble base was then transferred to the filter, washed with a little dilute ammonium hydroxide, dried *in vacuo* at 60° and then weighed.

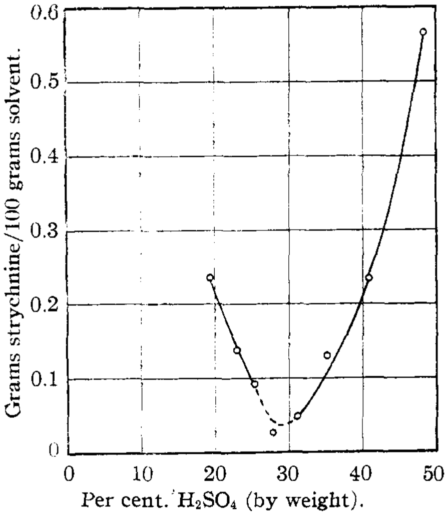

Fig. 1.—Solubility of strychnine acid sulfate in sulfuric acid.

The solubilities, determined by difference, are shown in Fig. 1 as grams of strychnine per 100 g. of sulfuric acid solution. These data indicate a minimum solubility of strychnine at an acid concentration of about 28.5%. At 20° the solubility was 0.029 g. per 100 g. of 28.5% acid. Also it was found that by lowering the temperature to 6° the solubility was decreased to 0.020 g. per 100 g. of 28.5% acid.

TABLE I

SOLUBILITY OF STRYCHNINE ACID SULFATE IN SULFURIC ACID AT 20°

% H_2SO_4	Strychnine, g.	Strychnine per 100 g. solvent, g.
59.7	0.4016	10.29[a]
48.3	.0280	0.569
40.9	.0142	.237
35.3	.0092	.133
31.1	.0040	.051
27.8	.0026	.029
25.4	.0093	.095
23.0	.0150	.139
19.4	.0298	.237

[a] Not a completely saturated solution.

CHEMICAL AND OCEANOGRAPHIC LABORATORIES
UNIVERSITY OF WASHINGTON
SEATTLE, WASHINGTON
RECEIVED AUGUST 18, 1941

---

[1] Present address: The Hydrocarbon Chemical and Rubber Co., The B. F. Goodrich Co., Akron, Ohio.

NEW COMPOUNDS

Oxime of Furfuralacetone

This compound was prepared in very poor yield (5%) by the method of Harries and DeOsa[1] for the oxime of benzalacetone. It was recrystallized several times from dilute ethanol and formed small tan-colored leaflets; melting point, 88–90°.

Anal. Calcd. for $C_8H_9O_2N$: N, 9.27. Found: N, 9.30. The analysis was performed by Dr. T. S. Ma.

---

(1) Harries and DeOsa, *Ber.*, **36**, 2998 (1903).

260 ADDISON ROAD
RIVERSIDE, ILL.
RICHARD D. KLEENE
RECEIVED SEPTEMBER 8, 1941

Derivatives of 1-Phenylcycloparaffin-1-carboxylic Acids

For purposes of identification the following derivatives of 1-phenylcyclobutane-, 1-phenylcyclopentane- and 1-phenylcyclohexane-1-carboxylic acids have been prepared. The acids were prepared by conventional methods,[1] and their physical constants agreed with those given in the literature.

The acids were refluxed with a slight excess of thionyl chloride for ten to twenty minutes and then, after cooling, a benzene solution of the appropriate amine was added and the entire mixture refluxed for a few minutes. The warm solution was suction-filtered to remove the precipitated amine hydrochloride, and the filtrate was washed succes-

---

(1) Case, THIS JOURNAL, **56**, 715 (1934).