Anal. Calcd. for $C_6H_5SO_4K$: S, 15.1. Found: S (from quinoline), 15.1, 15.05; (from dimethylaniline), 15.0, 15.0.

CHEMICAL DEPARTMENT OF THE CANCER RESEARCH INSTITUTE OF THE UNIVERSITY OF JERUSALEM, AND THE DEPARTMENT OF CHEMISTRY, NEW YORK UNIVERSITY
NEW YORK, N. Y.
RECEIVED OCTOBER 7, 1941

The Change in Magnetic Susceptibility of Nickel Disalicylaldehyde on Hydrogenation

BY M. A. FOBES AND GEORGE N. TYSON, JR.

A mechanism that accounts for the uptake of hydrogen in copper disalicylaldehyde has been reported previously.[1] In the course of further magnetic susceptibility measurements of hydrogenated disalicylaldehydes it appears reasonable to extend the concept to the hydrogenated nickel compound. Green nickel disalicylaldehyde becomes yellow when hydrogenated, and both forms of the compound exhibit two unpaired electrons as shown in the experimental data.

Solutions and Hydrogenations.—The divalent nickel disalicylaldehyde used in this investigation has been described previously.[2] 1.9332 g. was diluted to 100 ml. using J. T. Baker and Co. C. P. grade pyridine. The solution so obtained was measured magnetically within a few minutes of mixing. The solution was then hydrogenated by placing approximately 50 ml. in a 250-ml. Florence flask, cooling to about 5° and evacuating to 15 mm. pressure, with subsequent introduction of hydrogen into the evacuated flask, to a pressure slightly greater than atmospheric. When the flask containing the hydrogen and the solution was heated to 90° for approximately 200 hours, hydrogenation occurred. The original solution was a light green, while the hydrogenated compound formed a yellow liquid with a brownish cast, which readily reverted to the green color when exposed to oxygen.

Magnetic Measurements.—The magnetic susceptibility measurements were made by the Gouy method. The susceptibilities due to the nickel disalicylaldehyde in the freshly prepared green solution (I) (measured at 24°) and the yellow brown hydrogenated nickel disalicylaldehyde (II) (measured at 26°) are shown in Table I. In the column listing the Bohr magnetons, the calculations have been made assuming complete quenching of the orbital contribution to the magnetic moment. In the conversion of the experimental data into Bohr magnetons, a correction has been made for the diamagnetism of the nonmetal portion of the molecule.

TABLE I

Soln.	$\chi_s \times 10^6$	$\chi_m \times 10^6$	Bohr magnetons Exptl.	Calcd.	Unpaired electrons
I	12.32	3706	3.0	2.83	2
II	12.08	3625	3.0	2.83	2

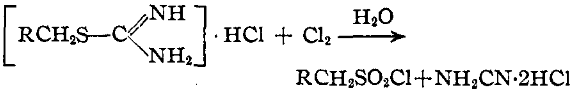
CHEMISTRY DEPARTMENT
POMONA COLLEGE
CLAREMONT, CALIFORNIA
RECEIVED JUNE 30, 1941

[CONTRIBUTION FROM MERCK AND CO., AND UNIVERSITY OF NORTH CAROLINA]

Explosion Hazard in the Chlorination of Alkyl Isothioureas to Prepare Alkyl Sulfonyl Chlorides

BY KARL FOLKERS, ALFRED RUSSELL AND R. W. BOST

In a series of papers, Sprague and Johnson[1] recently described a new and excellent method for the preparation of a considerable number of alkyl sulfonyl chlorides. The method consisted in the reaction of chlorine upon S-alkyl-isothiourea salts in aqueous solution according to the scheme



$$\left[RCH_2S\text{—}C\begin{matrix}\nearrow NH\\ \searrow NH_2\end{matrix}\right]\cdot HCl + Cl_2 \xrightarrow{H_2O} RCH_2SO_2Cl + NH_2CN\cdot 2HCl$$

It was stated that when R was the carboxyl group, the reaction did not produce the sulfonyl chloride derivative. In connection with one of the problems in the Merck Research Laboratory, one of the research chemists investigated the reaction of chlorine upon formamidine thiolacetic acid hydrochloride ($R{=}CO_2H$) since the corresponding sulfonyl chloride was desired. The first experiment gave a very hygroscopic product melting at 78–81° which was apparently sulfoacetic acid.[2] This result suggested that the reaction actually did go, but that the sulfonyl chloride was hydrolyzed to the sulfonic acid. In the next experiment, greater care was taken to have all the materials and apparatus more carefully cooled throughout, but the solution was refrigerated over the week-end. After further brief passage of chlorine, the oily globules were collected in a vial from a separatory funnel; they exploded violently. The chemist suffered severe

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(1) Tyson and Vivian, THIS JOURNAL, **63**, 1403 (1941).

(2) Tyson and Adams, *ibid.*, **62**, 1228 (1940).

(1) Sprague and Johnson, THIS JOURNAL, **58**, 1348 (1936); **59**, 1837 (1937); **59**, 2439 (1937); **61**, 176 (1939); "Sulfonic Halides," U. S. Patent 2,147,346 by T. B. Johnson, assignor to Röhm and Haas Company, Philadelphia, Pa.; "Process of Preparing Sulfonyl Halides and Sulfonic Acid from Pseudothioureas," U. S. Patent 2,146,744 by T. B. Johnson, assignor to Röhm and Haas Company, Philadelphia, Pa.

(2) Sulfoacetic acid monohydrate was hygroscopic and melted at 84–86° according to O. Stillich, *J. prakt. Chem.*, **73**, 538 (1906).