ethanol, 5% sodium hydroxide; slightly soluble in water, cold ethanol, cold acetic acid, diluted hydrochloric acid; insoluble ether. The adduct decomposed at temperatures above the melting point to give the original reagents; the addition was also somewhat reversible in cold sodium hydroxide solutions.

Anal. Calcd. for $C_9H_9O_2N$: N, 8.58. Found: N, 8.51.

3,6-Endomethylene-1,2,3,6-tetrahydro-*o*-phthalic Acid.—Two-tenths gram of 3,6-endomethylene-1,2,3,6-tetrahydro-*o*-phthalimide were placed on a steam-bath with 5 ml. of 10% potassium hydroxide and heated for three hours, at the end of which time no ammonia was detectable in the vapors above the flask. Concentrated hydrochloric acid was added in excess, and the mixture was then evaporated to dryness. The residue was then triturated in 20 ml. of ether to remove the maleic acid formed by the alkali-catalyzed reversal of the addition. The remaining organic constituents were taken up in 10 ml. of boiling acetone, and petroleum ether was added to the solution until it became cloudy, whereupon the mixture was left at 0° to crystallize. After one recrystallization from alcohol-ether (1:2) the melting point was observed to be 177°, which was not lowered by 3,6-endomethylene-1,2,3,6-tetrahydro-*o*-phthalic acid from another source.

DEPARTMENT OF CHEMISTRY
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N,N-Dimethyl-N′-(2-pyridyl)-N′-(2-furfuryl)-ethylenediamine

BY L. P. KYRIDES AND M. F. ZIENTY

In a recent publication Vaughan and Anderson[1] described the preparation of N,N-dimethyl-N′-(2-pyridyl)-N′-(2-furfuryl)-ethylenediamine (I) originally synthesized by Viaud.[2]

These authors reported that (I) is very unstable at temperatures even as low as −80° and that they were unable to prepare its mineral acid salts.[3]

Prior to the disclosure of Vaughan, *et al.*, the monohydrochloride of (I) had been prepared in this Laboratory for pharmacological evaluation. Since our findings do not support the observations reported, we are prompted to record our results.

Furfuryl alcohol was converted to the chloride in 75% yield by a modification of a synthesis described by Kirner,[4] with thionyl chloride in ether solution in the presence of pyridine at −15-20°. Distilled furfuryl chloride[5] was then treated with the lithium salt of N,N-dimethyl-N′-(2-pyridyl)-ethylenediamine in benzene solution at room temperature. After removal of the solvent and unreacted N,N-dimethyl-N′-(2-pyridyl)-ethylenediamine the desired product distilled as a yellow oil, stable at room temperature.

The monohydrochloride was prepared as well as the dihydrogen citrate salt.

The synthesis of (I) is described in detail.

Experimental[6]

N,N-Dimethyl-N′-(2-pyridyl)-N′-(furfuryl)-ethylenediamine.—To a suspension of 8.3 g. (0.36 mole) of lithium amide in 500 cc. of dry benzene was added 59.4 g. (0.36 mole) of N,N-dimethyl-N′-(2-pyridyl)-ethylenediamine,[7,8] and the mixture refluxed for three hours with stirring. The reaction was cooled to 25° and maintained at this temperature while a solution of 42 g. (0.36 mole) of furfuryl chloride in 200 cc. of dry benzene was added dropwise during one hour, then refluxed for one hour. On cooling, the solid material was removed by suction filtration and the solvent distilled under reduced pressure. After a forerun of 17.7 g. (28.7% recovery) of the starting ethylenediamine (b. p. 100–105° (0.4 mm.)), 45.9 g. (52%) of the desired tertiary amine was collected as a light yellow oil, b. p. 117.5–118° (0.2 mm.). The dihydrogen citrate was prepared in 88% yield by treating a methanol solution of 4.9 g. (0.026 mole) anhydrous citric acid with a methanol solution of 10 g. (0.04 mole) of (I) and precipitation with dry ether. Recrystallization of this salt from methanol–ether gave colorless crystals, m. p. 95–97°.[1a]

The monohydrochloride was prepared by treating an ethyl acetate solution of 36.2 g. (0.14 mole) (I) with 5 g. (0.14 mole) dry hydrochloric acid in methanol. On cooling, 37.8 g. (92%) of monohydrochloride was obtained. Recrystallization from ethyl acetate yielded colorless needles, m. p. 117–119°.

Anal. Calcd. for $C_{14}H_{19}N_3O \cdot HCl$: Cl, 12.61. Found: Cl, 12.80.

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[1] Vaughan and Anderson, THIS JOURNAL, **70**, 2607 (1948). reported m. p. 95–97°.

[2] Viaud, *Technologie Produits Pharmaceutiques*, **2**, 53 (1947).

[3] In a personal communication, Dr. Anderson, after receiving the manuscript of this article, has written us as follows: "We today converted a sample of our stable citrate to the base and then to the hydrochloride in solution. In contrast to our previous experience, the solution remained colorless. Our only explanation is that an impurity in the original base caused its instability to temperature and mineral acids, and in the process of preparing the citrate this impurity was removed. We found also that the 5-bromofurfuryl analog hydrochloride appears to be stable when made from the citrate," and authorizes us to publish this statement as a footnote.

[4] Kirner, THIS JOURNAL, **50**, 1958 (1928).

[5] Gilman and Vernon, *ibid.*, **46**, 2576 (1924), reported furfuryl chloride as extremely unstable.

[6] All melting points are corrected.

[7] Whitmore, Goldsmith and Rytina, THIS JOURNAL, **67**, 393 (1945).

[8] Huttrer, Djerassi, Beears, Mayer and Scholz, *ibid.*, **68**, 1999 (1946).

RESEARCH LABORATORIES OF
SUMNER CHEMICAL COMPANY, INC.
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The Influence of Crystal Face in the Catalytic Deposition of Cobalt on a Single Crystal of Copper[1]

BY HENRY LEIDHEISER, JR., AND RICHARD MEELHEIM

It has previously been shown in two different cases[2,3] that the rate of gaseous, catalytic reactions on the surface of a metal differs with the crystal face exposed at the surface. It has also been shown by Beeck, Smith and Wheeler[4] that the rate of the reaction of hydrogen and ethylene on a nickel film with a (110) orientation parallel to the surface is greater than that on an unoriented film. Results reported herein show that the rate of another type of catalytic reaction, the

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[1] This work was supported by a grant from the Research Corporation.

[2] Reaction of hydrogen and oxygen on copper: Leidheiser and Gwathmey, THIS JOURNAL, **70**, 1200 (1948).

[3] Decomposition of carbon monoxide on nickel: Leidheiser and Gwathmey, *ibid.*, **70**, 1206 (1948).

[4] Beeck, Smith and Wheeler, *Proc. Roy. Soc. (London)*, **A177**, 62 (1940).