

2-XS (HCO_3^-) ion-exchange resin. The neutralized solutions were filtered free of resin, the resin was washed with a small amount of methanol, and the combined filtrate and wash were concentrated *in vacuo* (35° bath) to a residual solid. The residue was dissolved in CH_3OH (50 ml) and diluted with ether (75 ml) and hexane (100 ml). The solution was seeded and stored at 5° for 16 hr to give the product as a crystalline solid (3.4 g, 0.0123 mole, 24.6%), mp 170–171.5°. Continued storage afforded a second crop (1.7 g, 34% total). The material was homogeneous by tlc (silica gel-benzene-*n*-butylamine-water, 15:5:1). An analytical sample was prepared by recrystallization from a methanol-ether mixture containing a trace of hexane: mp 172–173°, $[\alpha]^{25}_D +178.1^\circ$ (*c* 1.0, CH_3OH), $\lambda_{\text{max}}^{\text{EtOH}}$ 290 m μ (ϵ 6500), $\lambda_{\text{min}}^{\text{EtOH}}$ 244 m μ (ϵ 845).

Anal. Calcd for $\text{C}_{10}\text{H}_{13}\text{FN}_2\text{O}_5$: C, 43.48; H, 4.75; N, 10.14; F, 6.88. Found: C, 43.24; H, 4.76; N, 9.88; F, 7.09.

A coupling run on the same scale as above (5 days) without the addition of molecular sieves yielded only 2.4 g (17%) of **3**.

1- β -D-Arabinofuranosyl-5-fluorocytosine (1).—A solution of **3** (2.76 g, 0.01 mole) in a 5% solution of anhydrous NH_3 in CH_3OH (200 ml) was sealed in a glass-lined bomb which was heated in an oil bath at $\sim 125^\circ$ for 16 hr. The bomb was cooled and opened and the contents was evaporated to dryness *in vacuo*. The residue was triturated with a small amount of CH_3OH , filtered, washed, and dried *in vacuo* to give **1** (2.3 g, 88%), mp 234–235° dec. The compound moved as a single spot on tlc (silica gel-benzene-*n*-butylamine-water, 15:5:1) and was free of starting material. The material was recrystallized once from a hot CH_3OH - H_2O mixture to give pure **1**, mp 230–232° dec, $[\alpha]^{25}_D +165.2^\circ$ (*c* 0.2, H_2O) [lit.² mp 237–238°, $[\alpha]^{25}_D +163 \pm 2^\circ$ (*c* 0.18, H_2O)]. The ultraviolet and infrared spectra were identical with those of a sample prepared from **2** by the method of Fox, *et al.*²

1- β -D-Arabinofuranosyl-5-fluorouracil (2).—The 4-alkoxy derivative **3** (0.6 g, 2.07×10^{-3} mole) was dissolved in 1 N HCl in methanol (20 ml), and the tightly stoppered solution was stored for 72 hr at ambient temperature. The solution was evaporated to dryness *in vacuo* and the residue was dissolved in a minimum of absolute EtOH, seeded, and stored at $\sim 5^\circ$ to give **2** (0.35 g, 65%) in two crops: mp 187–189, 184–185°. The combined crops were recrystallized once from hot absolute EtOH to give pure **2**, mp 186–188°, $[\alpha]^{25}_D +116.7^\circ$ (*c* 0.2, H_2O) [lit.³ mp 187–188°, $[\alpha]^{25}_D +128^\circ$ (*c* 0.21, H_2O)]. The ultraviolet and infrared spectra were in good agreement with those of an authentic sample.

Acknowledgment.—The authors express their appreciation to Hoffmann-LaRoche, Inc., for a generous gift of 5-fluorouracil for use in this study. We wish to thank Mr. H. I. Znak of the Riker Analytical Department for obtaining the spectral data.

Terpene Compounds as Drugs. III. Terpenylketoximes

GIANFRANCO PALA, ANTONIO MANTEGANI, AND
GERMANO COPPI

Research Laboratories of Istituto De Angeli S.p.A., Milan, Italy

Received March 20, 1967

The report¹ that some oximes of aliphatic ketones are endowed with interesting hypnotic and anticonvulsant properties and our interest in the terpene field have led us to synthesize the oximes of geranylacetone, nerylacetone, and farnesylacetone and to study their pharmacological properties. However, none of the three compounds displayed hypnotic and anticonvulsant activity of any interest. By contrast, geranylacetone oxime and nerylacetone oxime revealed a marked and unexpected hyperglycemic activity in rats and rabbits.

Experimental Section

Geranylacetone Oxime.—Geranylacetone² (4.6 g, 0.0237 mole), hydroxylamine hydrochloride (2.47 g, 0.0355 mole), and NaHCO_3

(2.98 g, 0.0355 mole) were poured into 10 ml of water, and the mixture was stirred for 24 hr at room temperature. An emulsion formed which was then extracted with ether, the ethereal solution was washed with water and dried (Na_2SO_4), and the solvent was removed. The residue was distilled *in vacuo* to yield a colorless oil (4.1 g, 83%), bp 107–108° (0.05 mm), n^{20}_D 1.4897, lit.³ n^{20}_D 1.4894.

Anal. Calcd for $\text{C}_{13}\text{H}_{23}\text{NO}$: C, 74.58; H, 11.07; N, 6.69. Found: C, 74.74; H, 11.06; N, 6.51.

Nerylacetone oxime was similarly prepared from nerylacetone² with an 84% yield, bp 112–114° (0.12 mm), n^{20}_D 1.4890.

Anal. Calcd for $\text{C}_{13}\text{H}_{23}\text{NO}$: C, 74.58; H, 11.07; N, 6.69. Found: C, 74.55; H, 11.18; N, 6.49.

Farnesylacetone oxime was similarly prepared from farnesylacetone² in an 81% yield, bp 142–143° (0.07 mm), n^{20}_D 1.4974.

Anal. Calcd for $\text{C}_{15}\text{H}_{25}\text{NO}$: C, 77.92; H, 11.26; N, 5.05. Found: C, 77.75; H, 11.05; N, 4.90.

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Formation of

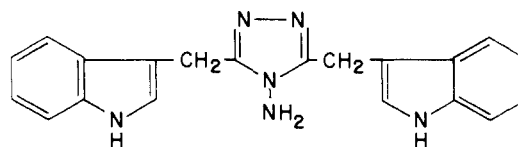
4-Amino-3,5-di(3-indolylmethyl)-s-triazole from Indole-3-acetonitrile and Hydrazine

ROBERT G. TABORSKY¹

Research Division, Cleveland Clinic Foundation,
Cleveland, Ohio 44106

Received May 17, 1967

During investigations of the chemistry of indolic compounds and possible routes to tryptamines, indole-3-acetonitrile was treated with anhydrous hydrazine. Analytical data obtained together with consideration of reactions reported in the Experimental Section led to the structural assignment as 4-amino-



3,5-di(3-indolylmethyl)-s-triazole for the compound obtained.

Experimental Section

Mass spectroscopy was performed by the Morgan Schaffer Corp., Montreal 26, Quebec, Canada. Nmr spectra was done by Nuclear Magnetic Resonance Specialties, Inc., New Kensington, Pa.

Indole-3-acetonitrile (5.0 g, 0.032 mole) was refluxed with 25.0 ml of anhydrous hydrazine for 18 hr. Most of the hydrazine was removed under vacuum and the residual solution was poured into water resulting in the precipitation of 6.1 g (56% yield) of light yellow product, mp 224–226°. Three crystallizations from ethanol-water gave a cream-colored compound, mp 227–228° (cor).

Anal. Calcd for $\text{C}_{23}\text{H}_{25}\text{N}_6$: C, 70.15; H, 5.30; N, 24.55. Found: C, 70.42; H, 5.52; N, 24.22.

Chromatography on thin layer silica on glass in 9:1 CHCl_3 - CH_3OH produced one spot at R_f 0.1 giving a positive xanthidol reaction for indoles, negative ninhydrin reaction, and weak fluorescence under uv light. The compound was insoluble in water, but soluble in dilute HCl. Mass spectroscopic analysis gave 342 as the parent peak and therefore molecular weight. The infrared absorption spectrum (KBr) showed the presence of the N-H stretching band at 2.95 μ . The uv spectrum (in

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(2) Veterans Administration Hospital, Cleveland, Ohio 44106.