

Boos and associates for microanalytical data, to Messrs. A. B. White and R. W. Walker for phase solubility analysis and infrared spectra, respectively, and to

Drs. C. C. Porter and J. J. Wittick for pharmacological testing and optical rotatory measurements, respectively.

New Compounds

Some Substituted γ,γ -Pentamethyleneparaconamides

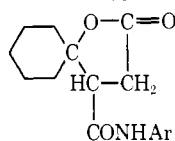
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In connection with our interest in pharmacological properties of paraconamide derivatives, we have synthesized some derivatives of γ,γ -pentamethyleneparaconamide. However, none of the compounds described here (Table I) was active when screened for

TABLE I
N-ARYLPARACONAMIDE



No.	Ar	Mp, °C	Recrystn solvent	Yield, %	Formula ^a
1	C ₆ H ₅	195-197	MeOH-H ₂ O	87.6	C ₁₆ H ₁₉ NO ₃
2	2-ClC ₆ H ₄	118-120	EtOH-H ₂ O	79.6	C ₁₆ H ₁₇ ClNO ₃
3	4-ClC ₆ H ₄	207-210	MeOH	74.5	C ₁₆ H ₁₇ ClNO ₃
4	α -C ₁₀ H ₇	210-211	MeOH	92.9	C ₂₀ H ₂₁ NO ₃
5	β -C ₁₀ H ₇	175-176	EtOH	91.6	C ₂₀ H ₂₁ NO ₃

^a All compds were analyzed for C, H, N.

insecticide, fungicide, and herbicide activity. The methods of preparation are adaptations of known procedures.

Experimental Section¹

γ,γ -Pentamethyleneparaconyl Chloride.—A mixture of 9.9 g (0.05 mole) of γ,γ -pentamethyleneparaconic acid² and 10 ml of SOCl₂ was refluxed for 6 hr. Excess SOCl₂ was removed under diminished pressure, then PhH was added and evapd to dryness. Recrystn of the residue from hexane gave 10.1 g (93.5%) of product, mp 86-87°. *Anal.* (C₁₀H₁₃ClO₃) C, H.

General Procedure for Compounds Listed in Table I.—To a soln of the appropriate amine in 10-40 ml of PhH was added a soln of 0.05 mole of acid chloride in 90 ml of PhH at room temp and stirred for an additional hr. The sepd cryst were collected, washed with H₂O, and recrystd to give the pure paraconamides listed in Table I.

(1) All melting points are uncorrected. Microanalyses were performed by Miss Teruko Nisi. The analytical results obtained for the indicated elements are within $\pm 0.3\%$ of the theoretical values.

(2) W. S. Johnson, C. E. Davis, R. H. Hunt, and G. Stork, *J. Amer. Chem. Soc.*, **70**, 3021 (1948).

Synthesis of 2,3,6-Trimethoxy- β -phenethylamine

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In a recent paper, Matsuhiro and Furst¹ have questioned the identity of 2,3,6-trimethoxy- β -phenethylamine, which was first reported by Merchant and Mountwalla² and later by us.³ The present paper concerns an unequivocal synthesis of this amine which had not previously been reported by us in our investigation of the deamination of polymethoxy- β -phenethylamines by liver monamine oxidase.^{3,4}

Experimental Section

2,3,6-Trimethoxyphenylacetoneitrile.—A slurry of 100 g (0.47 mole) of 2,3,6-trimethoxybenzoic acid, mp 148-149° (reported,⁵ 148-149°), obtained in 61% yield from 1,2,4-trimethoxybenzene by the procedure of Gilman and Thirtle,⁵ in 1 l. of dry C₆H₆ was added gradually to a stirred mixt of 38 g (1 mole) of LAH in 1 l. of anhyd Et₂O. The mixt was stirred and heated under reflux for 4 hr, cooled, and decompd with H₂O and dil H₂SO₄. The Et₂O-C₆H₆ layer was sepd, washed with H₂O, dil Na₂CO₃, and H₂O, dried (MgSO₄), and filtered. The filtrate was treated with 5 ml of pyridine and 75 ml of SOCl₂ was added slowly. The mixt was stirred at room temp for 2 hr and poured into ice-H₂O; the org layer was sepd, washed (H₂O, dil Na₂CO₃, H₂O), dried (MgSO₄), and filtered and the solvents were evapd. The residual oily crude chloride was dissolved in 700 ml of Me₂CO and stirred for 28 hr with a soln of KCN in 300 ml of H₂O. The Me₂CO was evapd, and the residue was extd with Et₂O; this ext was washed (H₂O) and dried (MgSO₄), the Et₂O was evapd, and the residue distd; bp 128-133° (0.25 mm); yield, 24 g (25%). *Anal.* (C₁₁H₁₃NO₃) C, H, N.

2,3,6-Trimethoxy- β -phenethylamine.—A soln of 16 g (0.077 mole) of 2,3,6-trimethoxyphenylacetoneitrile in 60 ml of MeOH contg 8.3 g of NH₃ and 10 ml of Raney Ni catalyst slurry were placed in a 300-ml stirring autoclave, which was sealed and pressured to 105 kg/cm² with H₂. The mixt was stirred and heated at 125° for 2 hr and filtered, the MeOH was evapd, and the residue was distd; bp 110-115° (0.4 mm); yield, 13.9 g (86%). A soln of the free base in Et₂O treated with dry HCl gave the HCl salt, mp 122-123° (reported¹ 134-135°), after one crystn from EtOH-EtOAc-Et₂O. After 2 more recrystns from *i*-PrOH-EtOAc (1:3), the HCl salt melted at 131-132° (Fisher block). The tlc (on silica gel (Chroma-Plate 7 G), developed with 1-Bu-

(1) B. Matsuhiro and A. Furst, *J. Med. Chem.*, **13**, 973 (1970).

(2) J. R. Merchant and A. J. Mountwalla, *J. Org. Chem.*, **23**, 1774 (1958).

(3) L. C. Clark, F. Benington, and R. D. Morin, *J. Med. Chem.*, **8**, 353 (1965).

(4) L. C. Clark, F. Benington, and R. D. Morin, *Alabama J. Med. Sci.*, **1**, 417 (1964).

(5) H. Gilman and J. R. Thirtle, *J. Amer. Chem. Soc.*, **66**, 858 (1944).