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 $\gamma,\gamma$ -Pentamethyleneparaconamides

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



<sup>*a*</sup> All compds were analyzed for C, H, N.

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

#### Experimental Section<sup>1</sup>

 $\gamma$ , $\gamma$ -Pentamethyleneparaconyl Chloride.—A mixture of 9.9 g (0.05 mole) of  $\gamma$ , $\gamma$ -pentamethyleneparaconic acid<sup>2</sup> and 10 ml of SOCl<sub>2</sub> was refluxed for 6 hr. Excess SOCl<sub>2</sub> 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. (C<sub>10</sub>H<sub>13</sub>ClO<sub>3</sub>) 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  $H_2O$ , and recrystd to give the pure paraconamides listed in Table I.

### Synthesis of 2.3,6-Trimethoxy-β-phenethylamine

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In a recent paper, Matsuhiro and Furst<sup>1</sup> have questioned the identity of 2,3,6-trimethoxy- $\beta$ -phenethylamine, which was first reported by Merchant and Mountwalla<sup>2</sup> and later by us.<sup>3</sup> 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- $\beta$ -phenethylamines by liver monamine oxidase.<sup>3,4</sup>

#### **Experimental Section**

2,3,6-Trimethoxyphenylacetonitrile.—A shurry of 100 g (0.47 mole) of 2,3,6-trimethoxybenzoic acid, mp 148-149° (reported,<sup>5</sup> 148-149°), obtained in 61% yield from 1,2,4-trimethoxybenzene by the procedure of Gilman and Thirtle,<sup>6</sup> in 1 l. of dry C<sub>6</sub>H<sub>6</sub> was added gradually to a stirred mixt of 38 g (1 mole) of LAH in 1 l. of anhyd Et<sub>2</sub>O. The mixt was stirred and heated under reflux for 4 hr, cooled, and decompd with  $H_2O$  and dil  $H_2SO_4$ . The Et<sub>2</sub>O-C<sub>6</sub>H<sub>6</sub> layer was sepd, washed with H<sub>2</sub>O, dil Na<sub>2</sub>CO<sub>3</sub>, and H<sub>2</sub>O, dried (MgSO<sub>4</sub>), and filtered. The filtrate was treated with 5 ml of pyridine and 75 ml of SOCl<sub>2</sub> was added slowly. The mixt was stirred at room temp for 2 hr and poured into ice-H<sub>2</sub>O; the org layer was sepd, washed  $(H_2O, dil Na_2CO_3, H_2O)$ , dried  $(MgSO_4)$ , and filtered and the solvents were evapd. The residual oily crude chloride was dissolved in 700 ml of  $Me_2CO$  and stirred for 28 hr with a soln of KCN in 300 ml of  $H_2O$ . The  $Me_2CO$  was evapd, and the residue was extd with  $Et_2O$ ; this ext was washed (H<sub>2</sub>O) and dried (MgSO<sub>4</sub>), the Et<sub>2</sub>O was evapd, and the residue distd; bp 128-133° (0.25 mm); yield, 24 g (25%). Anal. ( $C_{11}H_{13}NO_3$ ) C, H, N

**2,3,6-Trimethoxy-\beta-phenethylamine.**—A soln of 16 g (0.077 mole) of 2,3,6-trimethoxyphenylacetonitrile in 60 ml of MeOH contg 8.3 g of NH<sub>3</sub> and 10 ml of Raney Ni catalyst slurry were placed in a 300-ml stirring autoclave, which was sealed and pressured to 105 kg/cm<sup>2</sup> with H<sub>2</sub>. 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 Et<sub>2</sub>O treated with dry HCl gave the HCl salt, mp 122–123° (reported' 134–135°), after one crystn from EtOH-EtOAc-Et<sub>2</sub>O. 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-

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<sup>(1)</sup> 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.

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