

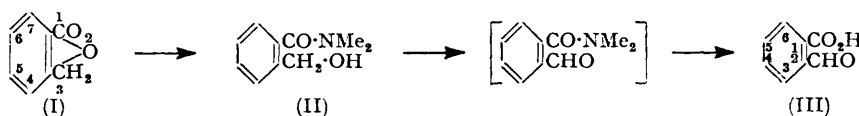
Lactones. Part I. A Novel Method for the Conversion of Phthalides into Phthalaldehydic Acids.

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Treatment of phthalides with dimethylamine gave the corresponding *o*-hydroxymethyl-*NN*-dimethylbenzamides, oxidation of which with chromium trioxide followed by hydrolysis with dilute hydrochloric acid furnished the phthalaldehydic acids.

THE experiments described in this Paper were carried out in an endeavour to find an efficient method employing mild conditions for the conversion of a phthalide (as I) into the corresponding phthalaldehydic acid (as III). The use of manganese dioxide in dilute sulphuric acid, *e.g.*, for the oxidation of meconin (6 : 7-dimethoxyphthalide) to opianic acid (5 : 6-dimethoxyphthalaldehydic acid) (Edwards, Perkin, and Stoyle, *J.*, 1925, 127, 195), in our hands led to inconsistent results; it is dependent on the quality of the reagent and is difficult to stop at the required stage, with attendant formation of hemipinic acid. The use of lead dioxide in sulphuric or acetic acids or chromium trioxide in acetic anhydride appears unsatisfactory, *e.g.*, in attempts to oxidise 5 : 6-methylenedioxyphthalide to 4 : 5-methylenedioxyphthalaldehydic acid (Stevens and Robertson, *J.*, 1927, 2790). A useful method was developed (Hirshberg, Lavie, and Bergmann, *J.*, 1951, 1030; Brown and Newbold, *J.*, 1952, 4397) in which the phthalide was brominated by *N*-bromosuccinimide and the product hydrolysed to the phthalaldehydic acid; by use of this technique, phthalaldehydic acid, *m*-opianic (4 : 5-dimethoxyphthalaldehydic acid), and 4 : 5-methylenedioxyphthalaldehydic acid were efficiently prepared. This method may be more difficult to apply when an easily substituted nuclear position is present, *e.g.*, in meconin, though this oxidation has been accomplished (Blair and Newbold, forthcoming publication).



The oxidation of a ring-opened phthalide bearing a free hydroxymethyl group was desired; while such derivatives are formed in strongly alkaline solution the use of such a medium was undesirable in view of the Cannizzaro reaction of phthalaldehydic acids; *e.g.*, opianic acid gives meconin and hemipinic (3 : 4-dimethoxyphthalic) acid (Beckett and Wright, *J.*, 1876, 29, 281). A feasible route seemed to effect the ring opening by the action of a secondary amine and to oxidise and then hydrolyse the amide under acid conditions. Though the ring opening of phthalides with hydrazine to give the *o*-hydroxymethyl-carboxyhydrazide has been studied by Teppema (*Rec. Trav. chim.*, 1923, 42, 30) little attention has been shown to the reaction of phthalide with ammonia or primary and secondary amines. While phthalide is reported to yield ammonium 2-hydroxymethylbenzoate when kept in aqueous ammonia (Hessert, *Ber.*, 1877, 10, 1445), heating the lactone in an ammonia stream gave phthalimidine (Graebe, *Annalen*, 1888, 247, 288). Theilacker and Kalenda recently (*ibid.*, 1953, 584, 87) showed that phthalide gave 2-hydroxymethyl-*N*-methylbenzamide with aqueous methylamine.

Treatment of meconin with ethanolic dimethylamine at room temperature gave 6-hydroxymethyl-2 : 3-dimethoxy-*NN*-dimethylbenzamide (as II). The dimethylamide was readily hydrolysed by hot dilute hydrochloric acid or sodium hydroxide to meconin; when heated alone the dimethylamide decomposed at *ca.* 170°, evolving dimethylamine and giving back meconin. When the dimethylamide was oxidised for a short time with chromium trioxide in acetic acid in the cold and the oxidation product, not isolated in the solid state, was then heated with dilute hydrochloric acid, opianic acid was formed in 41% overall yield from meconin; under identical oxidation conditions meconin was unaffected.

This sequence of reactions has been successfully used for the preparation of phthalaldehydic acid, *m*-opianic acid, and 3-methoxy-, 4 : 5 : 6-trimethoxy-, and 5-methoxy-4-methylphthalaldehydic acid from the corresponding phthalides without isolation of the dimethylamides. The reaction of the phthalide with dimethylamine was carried out in ethanol in the cold and was followed by the oxidation and hydrolysis; if the phthalide was recovered unchanged the initial dimethylamine treatment was carried out at an elevated temperature in an autoclave and the process repeated.

6-Methoxyphthalaldehydic acid has been reported as a degradation product of mellein (ochracin), a compound produced by *Aspergillus melleus* Yukawa and *Aspergillus ochraceus* (Yabuta and Sumiki, *J. Agric. Chem. Soc. Japan*, 1934, 10, 703). It has been synthesised by treatment of 7-methoxyphthalide with dimethylamine, to give 6-hydroxymethyl-2-methoxy-*NN*-dimethylbenzamide, followed by oxidation and hydrolysis. A convenient route to 7-methoxyphthalide has been developed starting from 3-methoxy-2-nitrobenzoic acid (Ewins, *J.*, 1912, 101, 544) which was reduced to 2-amino-3-methoxybenzoic acid with sodium dithionite, the amino-acid being transformed by a Sandmeyer reaction into the nitrile which was then hydrolysed to 3-methoxyphthalic acid. 3-Methoxyphthalic anhydride was then converted into 7-methoxyphthalide by a modification of the procedure of Duncanson, Grove, and Zealley (*J.*, 1953, 1331). An unambiguous synthesis of 7-methoxyphthalide, albeit in small yield, has been achieved from 2-amino-3-methoxybenzoic acid by the reactions used by Brown and Newbold (*J.*, 1953, 1285) for the synthesis of 7-methoxy-6-methylphthalide.

The action of ethanolic ammonia in the cold on phthalide and meconin has been examined; *o*-hydroxymethylbenzamide and 2 : 3-dimethoxy-6-hydroxymethylbenzamide were formed respectively in good yield. It is interesting that on oxidation with chromium trioxide by the standard procedure each amide gave the corresponding imide of the dicarboxylic acid.

EXPERIMENTAL

Ultra-violet spectra were determined in EtOH unless otherwise stated, with a Unicam S.P. 500 spectrophotometer. pK_a values (classical) were determined as described in *J.*, 1954, 3935.

Standard Chromium Trioxide Oxidation Procedure.—A solution of the amide derivative (1 part) or, if the amide was not isolated as a solid, that derived from the phthalide (1 part) in glacial acetic acid (20 parts) was stirred at room temperature and treated with a solution of chromic acid (1 part) in water (1 part) and glacial acetic acid (20 parts) added in one portion and stirred for a further 5 min. Water (100 parts) was then added and the mixture extracted with chloroform (3 × 50 parts), and the combined extracts were washed with water, 10% aqueous sodium hydrogen carbonate, and water, and dried (Na_2SO_4).

*6-Hydroxymethyl-2 : 3-dimethoxy-*NN*-dimethylbenzamide.*—Dimethylamine (50 c.c.) was added to a solution of meconin (2 g.) in ethanol (120 c.c.), and the mixture kept at room temperature for 2 days. After removal of solvent under reduced pressure below 35° the residual yellow gum slowly solidified; crystallisation from ethyl acetate–light petroleum (b. p. 60–80°) gave the *dimethylamide* (2.2 g., 89%) as plates, m. p. 93–95° (Found: C, 60.2; H, 6.8. $\text{C}_{12}\text{H}_{17}\text{O}_4\text{N}$ requires C, 60.2; H, 7.2%). The compound gave a 20° depression on admixture with meconin. Light absorption: Max. at 2080 (ϵ 29,400) and 2800 (ϵ 2300) and inflexion at 2260 Å (ϵ 11,500). The *acetate*, prepared in good yield by the action of acetic anhydride–pyridine at room temperature overnight, separated from light petroleum (b. p. 40–60°) as prisms, m. p. 63–64° (Found: C, 60.2; H, 6.6. $\text{C}_{14}\text{H}_{19}\text{O}_5\text{N}$ requires C, 59.8; H, 6.8%). Light absorption: Max. at 2080 (ϵ 32,600) and 2800 (ϵ 2400), and inflexion at 2260 Å (ϵ 12,500). The amide began to decompose at 170° (bath-temp.); after $\frac{1}{2}$ hr. at 170–180° the residue was cooled and crystallised from ethanol, to give meconin (85%) as needles, m. p. and mixed m. p. 101–102°. Hydrolysis of the dimethylamide was slow in boiling water; boiling with 3*N*-hydrochloric acid for $\frac{1}{2}$ hr. gave an almost quantitative yield of meconin. Boiling with 3*N*-sodium hydroxide also gave meconin in good yield.

Opianic Acid.—6-Hydroxymethyl-2 : 3-dimethoxy-*NN*-dimethylbenzamide (1 g.) was oxidised according to the standard procedure and the product isolated by using chloroform. Removal of the chloroform under reduced pressure gave a gum (700 mg.) which was refluxed with hydrochloric acid (10 c.c.; 3*N*) for 2 hr. On cooling, opianic acid (400 mg., 46%) separated;

it crystallised from water as needles, m. p. and mixed m. p. 145—146° (Found : C, 57.0; H, 5.0. Calc. for $C_{10}H_{10}O_5$: C, 57.1; H, 4.8%). Light absorption : Max. at 2150 (ϵ 20,700) and 2840 Å (ϵ 6500). Edwards, Perkin, and Stoye (*loc. cit.*) give m. p. 146°.

Phthalaldehydic Acid.—Phthalide (2 g.) in ethanol (10 c.c.) was heated with dimethylamine (20 c.c.) in an autoclave at 130° for 4 hr. and then oxidised, and the product was hydrolysed as in the previous cases. Phthalaldehydic acid (20% yield) separated from benzene–light petroleum (b. p. 60—80°) as rosettes of needles, m. p. and mixed m. p. 97°.

m-Opianic Acid.—*m*-Meconin (500 mg.) and ethanolic dimethylamine (40 c.c.; 33%) were heated in an autoclave for 5 hr. at 140—145° and then oxidised and the product hydrolysed as in the previous cases. *m*-Opianic acid (40 mg.) was obtained from water as needles, m. p. 185—187° alone or mixed with an authentic specimen (m. p. 187—188°) (Brown and Newbold, *J.*, 1952, 4397).

3-Methoxyphthalaldehydic Acid.—4-Methoxyphthalide (0.5 g.) (Buehler, Powers, and Michels, *J. Amer. Chem. Soc.*, 1944, 66, 417) was heated at 140—150° with ethanol (10 c.c.) and dimethylamine (35 c.c.) for 4 hr. The solution was evaporated under reduced pressure below 35° (bath-temp.), the residual gum oxidised according to the standard procedure, and the product hydrolysed to give *3-methoxyphthalaldehydic acid* (210 mg.) crystallising from benzene–light petroleum (b. p. 60—80°) as needles, m. p. 155—157°, pK_a 5.57 (Found : C, 59.9; H, 4.8%; equiv., 181. $C_9H_8O_4$ requires C, 60.0; H, 4.5%; equiv., 180). Light absorption in water : Max. at 2140 (ϵ 39,300), 2680 (ϵ 3100), and 3010 (ϵ 3900) and inflexion at 2350—2450 Å (ϵ 6800).

5-Methoxy-4-methylphthalaldehydic Acid.—6-Methoxy-5-methylphthalide (Charlesworth, Rennie, Sinder, and Yan, *Canad. J. Res.*, 1945, 23, B, 17) (500 mg.) was heated with a 30% ethanolic solution of dimethylamine (70 c.c.) in an autoclave at 140° for 4 hr. The mixture was evaporated under reduced pressure below 40° to a gum, which was oxidised by the standard procedure, and the product was isolated by using chloroform. The chloroform extract was evaporated and the residual gum refluxed for 1 hr. with hydrochloric acid (20 c.c.; 3*N*), then cooled and the acid fraction isolated by way of chloroform. Crystallisation from benzene gave *5-methoxy-4-methylphthalaldehydic acid* (200 mg.) as needles, m. p. 162—163° (Found : C, 62.1; H, 5.4%; equiv., 198. $C_{10}H_{10}O_4$ requires C, 61.85; H, 5.2%; equiv., 194). Light absorption in water : Max. at 2180 (ϵ 22,000) and 2900 (ϵ 9700) and inflexion at 2300—2340 Å (ϵ 12,900).

4 : 5 : 6-Trimethoxyphthalaldehydic Acid.—5 : 6 : 7-Trimethoxyphthalide (McRae, Van Order, Griffiths, and Habgood, *Canad. J. Chem.*, 1951, 29, 482) (300 mg.) in ethanolic dimethylamine (50 c.c.; 33%) was heated in a sealed tube at 60° for 4 hr. and kept at room temperature for 2 days. The product, not isolated as a solid, was oxidised by the standard procedure, and the oxidation product hydrolysed to give *4 : 5 : 6-trimethoxyphthalaldehydic acid* (50 mg.) which separated from benzene–light petroleum (b. p. 60—80°) as needles, m. p. 137—138°, depressed on mixing with starting material (Found : C, 54.9; H, 5.3. $C_{11}H_{12}O_6$ requires C, 55.0; H, 5.0%). Light absorption in water : Max. at 2230 (ϵ 20,500) and 2720 (ϵ 9600) and inflexion at 3120—3220 Å (ϵ 2800).

2-Amino-3-methoxybenzoic Acid.—Sodium dithionite (8 g.) was added in portions, with stirring, to 3-methoxy-2-nitrobenzoic acid (2 g.; Ewins, *J.*, 1912, 101, 544) in a solution of potassium hydroxide (2 g.) in water (20 c.c.) at <40° (water-cooling). After $\frac{1}{2}$ hr. at room temperature the solution was made acid (Congo-red) with hydrochloric acid (*d* 1.16) and heated on the steam-bath (fume-cupboard) for 3 hr. to remove sulphur dioxide. On cooling, 2-amino-3-methoxybenzoic acid (1.4 g.) separated. It had m. p. 164—166° (lit., m. p. 162—163°).

3-Methoxyphthalic Acid.—A suspension of 2-amino-3-methoxybenzoic acid (5 g.) in hydrochloric acid (7.5 c.c.; *d* 1.16) and water (25 c.c.) was diazotised at 0—5° with sodium nitrite (2 g.) in water (10 c.c.). After addition of urea the filtered diazonium solution was poured into a hot solution of potassium cyanide (8 g.) and copper sulphate (7 g.) in water (100 c.c.). The mixture was heated on the steam-bath for $1\frac{1}{2}$ hr., cooled and acidified (Congo-red) with hydrochloric acid (*d* 1.16). The crude cyano-acid (4 g.) which separated was refluxed with aqueous potassium hydroxide (100 c.c.; 10%) for 4 hr. The solution was acidified (Congo-red) and evaporated to dryness under reduced pressure. The dark red residue was extracted with boiling ethyl acetate (3 × 100 c.c.); concentration of the extract to small bulk and cooling gave 3-methoxyphthalic acid (3.5 g.) as a brown solid, m. p. 162—165°. Crystallisation from ethyl acetate–light petroleum (b. p. 60—80°) gave the acid as prisms, m. p. 169—171° (Bentley, Robinson, and Weizmann, *J.*, 1907, 91, 104, give m. p. 173—174°) (Found : C, 54.9; H, 4.3. Calc. for $C_9H_8O_5$: C, 55.1; H, 4.1%). Light absorption : Max. at 2110 (ϵ 22,500) and 3000 Å (ϵ 3400).

The crude acid (2 g.) was heated at 180—185° for 10 min. at atmospheric pressure and at

160—170°/10⁻³ mm.; 3-methoxyphthalic anhydride (1.8 g.) sublimed (m. p. 155—157°). Crystallisation from ethyl acetate—light petroleum (b. p. 60—80°) gave blades, m. p. 159—160° (Bentley *et al.*, *loc. cit.*, give m. p. 160—161°) (Found: C, 60.6; H, 3.5. Calc. for C₉H₆O₄: C, 60.7; H, 3.4%).

7-Hydroxyphthalide.—3-Methoxyphthalic anhydride (4.4 g.), glacial acetic acid (13 c.c.), hydrochloric acid (13 c.c.; *d* 1.16), and zinc dust (11 g.) were heated under reflux for 6 hr. The mixture was kept at room temperature overnight and the filtered solution was almost neutralised with aqueous sodium carbonate, and extracted with chloroform (3 × 30 c.c.), and after being washed with water (20 c.c.) the dried (Na₂SO₄) extract was evaporated under reduced pressure to a gum (3.8 g.) which rapidly solidified. Separation of 4- and 7-methoxyphthalides by fractional crystallisation (cf. Duncanson, Grove, and Zealley, *loc. cit.*) proved impracticable. The solid was refluxed for 2½ hr. with aqueous hydrobromic acid (100 c.c.; *d* 1.46—1.49) in a coal-gas atmosphere. The solution was evaporated under reduced pressure to a red gum which was dissolved in water (50 c.c.) and treated with charcoal. The solution was evaporated to dryness under reduced pressure. The resulting solid (2.0 g.) was heated at 130—140°/10⁻³ mm., and the sublimate (1.4 g.), m. p. 125—127°, was collected. Crystallisation from ethyl acetate—light petroleum (b. p. 60—80°) gave 7-hydroxyphthalide as prisms, m. p. 134—136° (Found: C, 64.25; H, 4.3. Calc. for C₈H₆O₃: C, 64.0; H, 4.0%). Light absorption: Max. at 2120 (ε 26,000), 2320 (ε 8700), and 2990 Å (ε 4600). Eliel, Rivard, and Burgstahler (*J. Org. Chem.*, 1953, 18, 1679) give m. p. 135—136.5°. The compound gave a purple colour with ferric chloride in aqueous ethanol.

7-Methoxyphthalide.—(a) 2-Amino-3-methoxybenzoic acid (5 g.) was refluxed for 3 hr. with lithium aluminium hydride (8 g.) in ether (250 c.c.). The mixture was worked up as given for the corresponding methyl derivative (Brown and Newbold, *J.*, 1953, 1285). Removal of the ether gave a light yellow, partially solid material (3.5 g.) which was used without further treatment for the next stage. A solution of the crude amino-alcohol in hydrochloric acid (27 c.c.; *d* 1.16) and water (90 c.c.) was diazotized at 0° with a solution of sodium nitrite (2.7 g.) in water (20 c.c.). After addition of urea the filtered solution was added to a hot solution of potassium cyanide (14.4 g.) and copper sulphate (12.6 g.) in water (150 c.c.). After being heated on the steam-bath for 30 min. the cooled mixture was extracted with ether (3 × 50 c.c.), and the combined extracts were washed with water, dried (Na₂SO₄), and evaporated to a red oil (700 mg.). The oil was heated under reflux with aqueous potassium hydroxide (20 c.c.; 10%) for 2 hr. The solution was decanted from tar, acidified (Congo-red), and extracted with chloroform (3 × 20 c.c.), the combined chloroform extracts were washed with water (20 c.c.) and dried (Na₂SO₄). Removal of the solvent gave an orange gum which slowly solidified. Crystallisation from ethyl acetate—light petroleum (b. p. 60—80°) (charcoal) gave 7-methoxyphthalide (100 mg.) as needles, m. p. 107—109° (Found: C, 65.6; H, 5.1. Calc. for C₉H₈O₃: C, 65.85; H, 4.9%). Light absorption: Max. at 2090 (ε 33,000), 2340 (ε 8200), and 2960 Å (ε 4700). Duncanson, Grove, and Zealley (*loc. cit.*) give m. p. 96°; that this low m. p. was due to some residual 4-methoxyphthalide in the specimen has been shown by direct comparison of the infra-red spectra of the two substances. (We are indebted to Mr. J. F. Grove for this information.)

(b) 7-Hydroxyphthalide (650 mg.) was suspended in dry ether (5 c.c.) and treated with an ethereal solution of diazomethane (from 6 g. of nitrosomethylurea). Reaction and dissolution were rapid; after 6 hr. the solution was evaporated and the residue crystallised from ethyl acetate—light petroleum (b. p. 60—80°), giving 7-methoxyphthalide (570 mg.) as needles, m. p. 107—109° alone or mixed with preparation (a) (Found: C, 66.1; H, 5.1%). A specimen of 7-methoxyphthalide was demethylated under reflux with aqueous hydrobromic acid as given for the mixed phthalides, furnishing 7-hydroxyphthalide as prisms [from ethyl acetate—light petroleum (b. p. 60—80°)], m. p. and mixed m. p. 132—134°.

*6-Hydroxymethyl-2-methoxy-*NN*-dimethylbenzamide*.—7-Methoxyphthalide (120 mg.) in ethanol (10 c.c.) was cooled to 0° and treated with dimethylamine (10 c.c.) and kept at room temperature for 3 days. Evaporation of the solution under reduced pressure below 35° and crystallisation of the residue from ethyl acetate—light petroleum (b. p. 60—80°) gave the *dimethylamide* (140 mg.) as blades, m. p. 120—121° (Found: C, 63.1; H, 6.8. C₁₁H₁₅O₃N requires C, 63.1; H, 7.2%).

6-Methoxyphthalaldehydic Acid.—2-Hydroxy-6-hydroxymethyl-*NN*-dimethylbenzamide (100 mg.) was oxidised by the standard procedure and the product, isolated by chloroform, was refluxed for 1 hr. with hydrochloric acid (5 c.c.; 3*N*). *6-Methoxyphthalaldehydic acid hydrate* (50 mg.), isolated by chloroform, crystallised from water as needles, m. p. 151—153° (Found: equiv., 198. C₉H₈O₄.H₂O requires equiv., 198). After drying at 58°/0.5 mm. for 3 hr. the

anhydrous form, m. p. 151—153°, was obtained (Found : C, 59.7; H, 4.5%; equiv., 180.5. $C_9H_8O_4$ requires C, 60.0; H, 4.5%; equiv., 180). Light absorption in water : Max. at 2100 (ϵ 22,000), 2500 (ϵ 3900), and 3060 Å (ϵ 3200). The pK_a was 5.13. Yabuta and Sumiki (*loc. cit.*) give m. p. 152—153° for 6-methoxyphthalaldehydic acid obtained from degradation of mellein; a direct comparison of the two preparations has not been possible.

The 2 : 4-dinitrophenylhydrazone separated from ethanol as yellow needles, m. p. 250—252° (decomp.) [lit., 245—246° (decomp.)] (Found : C, 50.3; H, 3.35; N, 15.8. $C_{15}H_{12}O_7N_4$ requires C, 50.0; H, 3.4; N, 15.55%).

o-Hydroxymethylbenzamide.—Liquid ammonia (200 c.c.) was added to a solution of phthalide (5 g.) in ethanol (100 c.c.) and kept overnight at room temperature, then evaporated under reduced pressure, and the residue crystallised from ethyl acetate–light petroleum (b. p. 60—80°), to give *o*-hydroxymethylbenzamide (75%) as blades, m. p. 149—150° (Found : C, 63.1; H, 5.9. $C_8H_9O_2N$ requires C, 63.6; H, 6.0%).

Phthalimide.—*o*-Hydroxymethylbenzamide (500 mg.) was oxidised according to the standard procedure. By means of chloroform, phthalimide (70% yield) was isolated and separated from water as needles, m. p. and mixed m. p. 235°.

6-Hydroxymethyl-2 : 3-dimethoxybenzamide.—Liquid ammonia (200 c.c.) was added slowly with shaking to a solution of meconin (5 g.) in ethanol (300 c.c.), and the solution kept overnight at room temperature. The solvent was removed under reduced pressure, to give a gum which slowly solidified. The solid was triturated with hot benzene, the benzene rejected, and the residue crystallised from ethyl acetate–light petroleum (b. p. 60—80°) to give 6-hydroxymethyl-2 : 3-dimethoxybenzamide (3.5 g.) as blades, m. p. 140—142° (Found : C, 57.0; H, 5.8. $C_{10}H_{13}O_4N$ requires C, 56.9; H, 6.2%). Light absorption : Max. at 2060 (ϵ 24,700) and 2820 (ϵ 2200) and inflexion at 2260 Å (ϵ 9300). The acetate, prepared in 80% yield by the action of acetic anhydride and pyridine at room temperature overnight and isolated by use of chloroform, separated from benzene–light petroleum (b. p. 60—80°) as blades, m. p. 159—160° (Found : C, 57.2; H, 6.1. $C_{12}H_{15}O_5N$ requires C, 56.9; H, 6.0%). Light absorption : Max. at 2080 (ϵ 23,400) and 2820 (ϵ 2100) and inflexion at 2260 Å (ϵ 9250).

Hemipinimide.—Oxidation of 2 : 3-dimethoxy-6-hydroxymethylbenzamide (250 mg.) by the standard procedure, followed by isolation using chloroform and crystallisation of the product from ethanol, gave hemipinimide (100 mg.) as needles, m. p. 225—227° alone or mixed with a specimen prepared from opianic acid (Liebermann, *Ber.*, 1886, 19, 2275; lit., m. p. 228—230°) (Found : C, 58.1; H, 4.4. Calc. for $C_{10}H_9O_4N$: C, 58.0; H, 4.4%). Light absorption : Max. at 2280 (ϵ 29,600) and 3320 (ϵ 5000) and inflexion at 2450 Å (ϵ 15,600).

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