Cyclic Acylimines and Cyclic Carbinolamides III. Piperidone and Isoindolone

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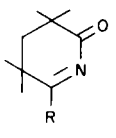
6-Phenyl-2,3,4,5-tetrahydro-3,3,5,5-tetramethyl-2-pyridone (**6**), 1-phenyl-3-isoindolone, and their methanol (**5**) and butanol (**19**) addition products, as well as 6-methoxy-3,3,5,5-tetramethyl-2-piperidone (**4**) and 1-methoxy-3-isoindolinone (**11**) were prepared and their chemical properties studied. The cyclic acylimines and their methanol addition products were found to react with weak nucleophiles (amides, alcohols), active methylene compounds (acetone, dimedone) or dienes (1,2,3,4-tetramethyl and 2,3-dimethylbutadiene) to give addition products.

In two recent publications we have described the synthesis of cyclic acylimines and cyclic carbinolamides (α-methoxylactams) in the azaphenalone (1) and isoquinolone (2) series. The cyclic acylimines were found to be reactive compounds, readily adding weak nucleophiles, dienes and active methylene compounds across the carbon-nitrogen double bond, to give addition products.

The reactivity of the acylimine system was found to depend on the degree of conjugation involving the carbon-nitrogen double bond. The more conjugated the acylimine the less active it was in addition reactions. The methanol addition products (α-methoxylactams) can substitute, in the presence of an acid catalyst, the unsaturated compound in the various reactions. The more reactive the acylimine was, the less reactive was the corresponding α-methoxylactam. The driving force in the addition reactions is probably the gain in resonance energy of the amide group. The hybridization of the lone pair of electrons on the nitrogen changes from sp^2 in the acylimine to more p-like in the addition products which enables the overlap with the carbonyl π bond.

The present investigation was aimed at the preparation of the tetrahydropyridone **1**, a six membered monocyclic acylimine and the isoindolone **2**, a five membered acylimine and the study of their chemical and physical properties.



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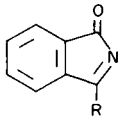
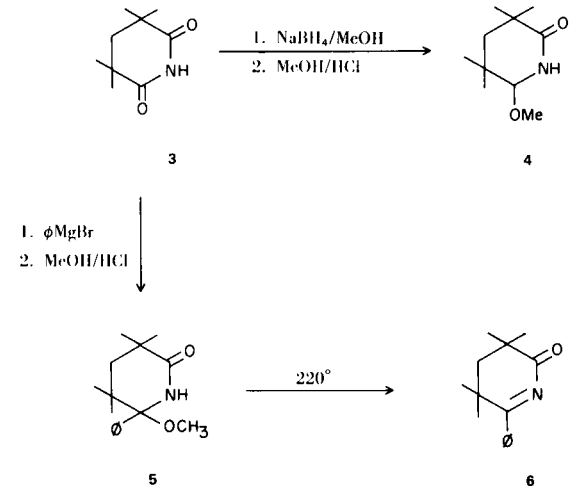


2

The *gem*-dimethyl groups in **1** should prevent the migration of the double bond away from its conjugation with the carbonyl group and also eliminate the possible enolization of the carbonyl group.

2,2,4,4-Tetramethylglutarimide (**3**) was the starting material for the preparation of the piperidone derivatives. It gave on reduction with sodium borohydride in methanol and subsequent treatment of the solution with methanolic hydrogen chloride, 2,2,5,5-tetramethyl-6-methoxy-2-piperidone (**4**).

Treatment of the glutarimide (**3**) with phenylmagnesium bromide and subsequent treatment of the reaction mixture with methanolic hydrogen chloride afforded the 6-methoxy-6-phenyl-3,3,5,5-tetramethyl-2-piperidone (**5**) which was further converted to the unsaturated piperidone **6** on heating to 220°. Compound **6** was obtained as a yellow hygroscopic oil which was reduced with sodium borohydride in dry glyme to 6-phenyl-3,3,5,5-tetramethyl-2-piperidone.



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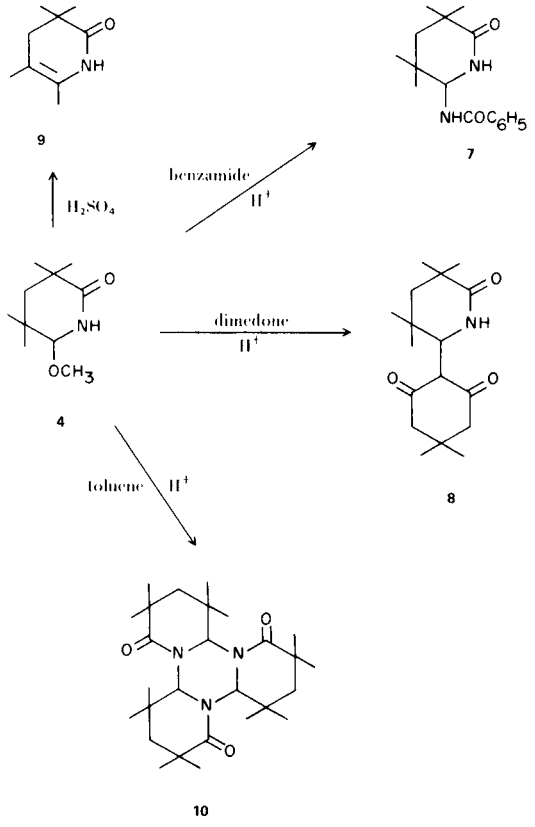
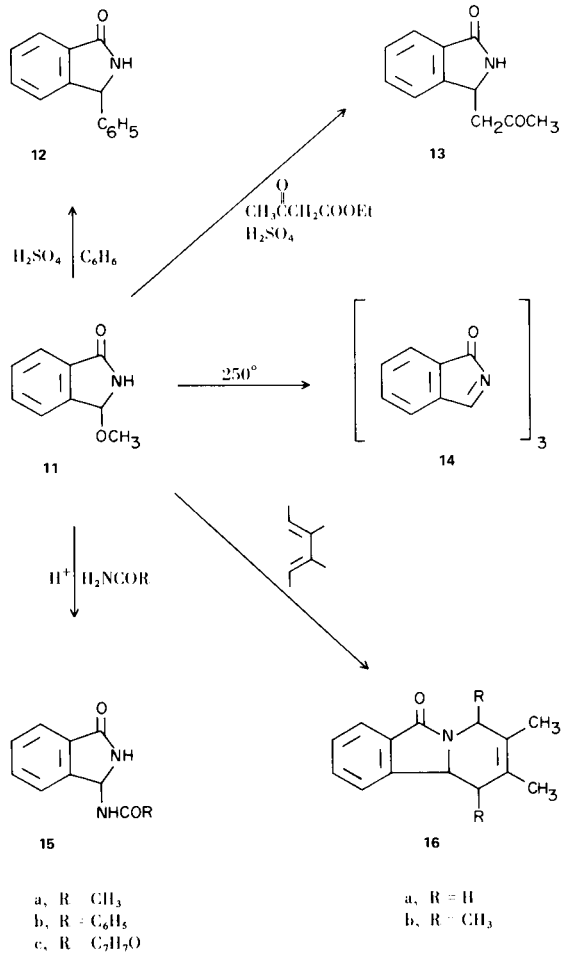
6-Methoxy-6-phenylpiperidone (**5**) reacted with benzamide in the presence of an acid catalyst to give 6-

benzamino-6-phenyl-3,3,5,5-tetramethyl-2-piperidone but was not reduced with sodium borohydried in dry glyme after 16 hours at room temperature.

We did not succeed in obtaining 2,3,4,5-tetrahydro-3,3,5,5-tetramethyl-2-pyridone (**1**, R = H). The 6-methoxy derivative **4**, which does not bear the additional phenyl group, was found to be rather stable. It was not reduced with sodium borohydride in glyme after 60 hours and did not react with acetone at reflux in the presence of an acid catalyst. It did however react with dimedone or benzamide to give the corresponding 6-piperidone derivatives (**7**,**8**) and in concentrated sulfuric acid it rearranged to 1,2,3,4-tetrahydro-3,3,5,6-tetramethyl-2-pyridone (**9**). Refluxing a toluene solution of the methoxy **4** for 20 hours in the presence of naphthalenesulfonic acid afforded a triazine trimer **10**.



The trimer showed a molecular peak in the mass spectrum, one carbonyl absorption at 1650 cm^{-1} in the infrared (no NH absorption) and three one hydrogen singlets at δ 3.93, 5.65 and 5.93 ppm in the n.m.r. spectrum.

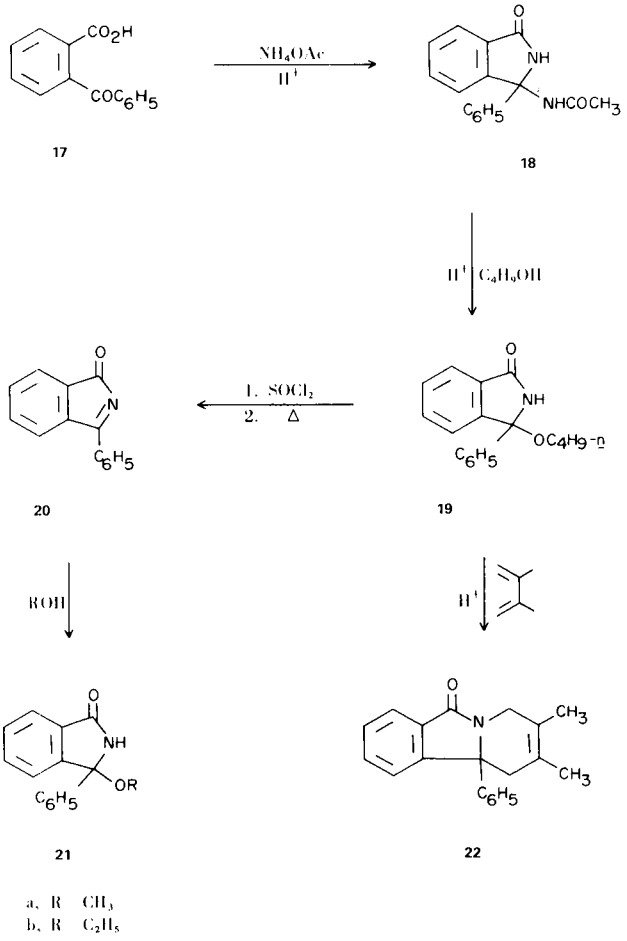


The 3-methoxy-1-isoindolinone (**11**) was prepared by the reduction of phthalimide according to Reissert (3) and treatment of the hydroxy intermediate with methanol and sulfuric acid (4). Attempts to obtain the unsaturated isoindolone led, in this case, also to the formation of a triazine trimer (**14**). The methoxyisoindolinone (**11**) reacted with benzene and ethyl acetoacetate in concentrated sulfuric acid to give the 3-phenylisoindolinone (**12**) and the 3-acetonyl derivative (**13**), respectively. It reacted in boiling benzene and in the presence of β-naphthalenesulfonic acid with benzamide and benzyl carbamate to give 3-substituted isoindolinone (**15**). It also reacted in the presence of an acid catalyst with 1,2,3,4-tetramethyl and 2,3-dimethylbutadiene to give Diels-Alder type products (**16**).

The trimer in this case also showed a molecular peak in the mass spectrum, one carbonyl absorption in the infrared (no NH absorption) at 1720 cm^{-1} and three one hydrogen singlets at δ 7.18, 6.0 and 5.9 ppm in the n.m.r.

The 3-phenyl-1-isoindolone (**20**) was prepared from *o*-benzoylbenzoic acid (**17**) which was converted to the

3-butoxy-3-phenyl-1-isoindolinone (**19**) *via* the 3-acetamido derivative (**18**). Treatment of the butoxy derivative with thionyl chloride and distillation of the reaction product afforded a yellow oil (**20**) which solidified on standing at room temperature. It showed a carbonyl absorption at 1755 cm^{-1} in the infrared and had only aromatic protons in the n.m.r. spectrum. This compound was reactive in addition reactions and the disappearance of the yellow colour could be used as an indication for the termination of the addition reactions. The 3-phenyl-1-isoindolone (**20**) reacted, within seconds, with methanol and ethanol to give colourless addition products, the 3-alkoxy derivatives (**21**). The carbon-nitrogen double bond was reduced with sodium borohydride in dry glyme to give 3-phenyl-1-isoindolinone identical with (**12**) described above. The 3-butoxy derivative (**19**) was not reduced by sodium borohydride under the same experimental conditions. The unsaturated compound (**20**) or its butanol addition product (**19**) reacted with 2,3-dimethyl butadiene to give a Diels-Alder type product (**22**).



EXPERIMENTAL

Melting points are corrected, infrared spectra were measured in chloroform solutions and n.m.r. spectra in deuteriochloroform (unless otherwise indicated).

3,3,5,5-Tetramethyl-6-methoxy-2-piperidone (**4**).

2,2,4,4-Tetramethylglutarimide (5.0 g., 0.03 mole) in methanol (50 ml.) was reduced with sodium borohydride (2.5 g., 0.066 mole) at 0°. After the addition of the hydride the solution was stirred for an additional 0.5 hours and methanolic hydrogen chloride was added dropwise (20 ml., 21%). The acid solution was left at room temperature for 0.5 hours and then poured onto a cold solution of sodium carbonate (50 ml., 10%). The aqueous suspension was extracted with ethyl acetate. The ethyl acetate solution was washed with water, dried and evaporated. The solid residue was crystallized from hexane, m.p. 109-110°, yield 5.2 g. (90%); i.r.: 3400, 2820, 1660 and 1090 cm^{-1}; n.m.r.: δ 7.75 (s, 1H); 3.95 (d, 1H, J = 5 cps); 3.35 (s, 3H); 1.98 (d, 1H, J = 13 cps); 1.35 (d, 1H, J = 13 cps); 1.25 (s, 3H); 1.19 (s, 3H); 1.07 (s, 3H); 1.00 (s, 3H).

Anal. Calcd. for $C_{10}H_{19}NO_2$: C, 64.83; H, 10.34; N, 7.56. Found: C, 64.59; H, 10.25; N, 7.59.

6-Methoxy-6-phenyl-3,3,5,5-tetramethyl-2-piperidone (**5**).

To a solution of phenylmagnesium bromide (0.1 mole) in dry ether (225 ml.) there was added 2,2,4,4-tetramethylglytarimide (3.4 g., 0.020 mole). The reaction mixture was refluxed and stirred for 2 hours. Dry methanol (100 ml.) was added to the cooled solution (0°) followed by methanolic hydrogen chloride (50 ml., 20%). After stirring at room temperature for an hour, the solution was poured onto aqueous sodium carbonate (200 ml., 10%). The ethereal solution was washed with water, dried and evaporated. The residue (6.6 g.) was chromatographed over deactivated neutral alumina (75 g. + 10 ml. methanol). The product was eluted with hexane, m.p. 137-138°, yield 4.2 g. (79%); i.r.: 3410, 2850, 1660, 1090, 1060 cm^{-1}; n.m.r.: δ 7.38 (s, 5H); 6.62 (s, 1H); 3.08 (s, 3H); 2.37 (d, 1H, J = 14); 1.40 (d, 1H, J = 14); 1.32 (s, 3H); 1.25 (s, 3H); 0.88 (s, 3H); 0.82 (s, 3H); m/e = 261.

Anal. Calcd. for $C_{16}H_{23}NO_2$: C, 73.53; H, 8.87; N, 5.36. Found: C, 73.41; H, 8.83; N, 5.27.

6-Phenyl-2,3,4,5-tetrahydro-3,3,5,5-tetramethyl-2-pyridone (**6**).

6-Methoxy-6-phenyl-3,3,5,5-tetramethyl-2-piperidone (**5**) was heated to 220° for 10 minutes. The pale yellow oily product, which was obtained in quantitative yield, was too unstable (hygroscopic) to be submitted for analysis; i.r. (carbon tetrachloride): 1720 and 1680 cm^{-1}; n.m.r. (carbon tetrachloride): δ 7.5-7.3 (m, 5H); 1.69 (s, 2H); 1.25 (s, 12H) p.p.m.; λ max (carbon tetrachloride) = 270 mμ (ε = 1.5 x 10^3); m/e = 229.

6-Phenyl-3,3,5,5-tetramethyl-2-piperidone.

The pyridone **6** described above (0.23 g.) in dry glyme (2 ml.) was reduced with excess sodium borohydride. The yellow colour disappeared on the addition of the hydride. The solution was distributed between ethyl acetate and aqueous hydrochloric acid. The ethyl acetate was separated, dried and evaporated to give a solid residue (0.18 g.) which was chromatographed over neutral alumina (10 g.). The product was eluted with methylene chloride to give 0.09 g. (38%), m.p. 150-151°; i.r.: 3390, 1660 and 1650 cm^{-1}; n.m.r.: δ 7.3 (m, 5H); 5.7 (s, 1H); 4.29 (s, 1H); 1.71 (s, 1H); 1.63 (s, 1H); 1.35 (s, 3H); 1.28 (s, 3H); 0.90 (s, 3H); 0.84 (s, 3H) p.p.m.; m/e = 231.

Anal. Calcd. for $C_{15}H_{21}NO$: C, 77.88; H, 9.15; N, 6.05. Found: C, 77.77; H, 9.31; N, 6.05.

6-Benzamido-6-phenyl-3,3,5,5-tetramethyl-2-piperidone.

A solution of the methoxy derivative **5** (0.62 g.) and benzamide

(0.25 g.) in benzene (20 ml.) containing β-naphthalenesulfonic acid (0.050 g.) was refluxed for 40 hours. The benzene solution was diluted with more benzene (50 ml.) and was washed with water, dried and evaporated. The residue was crystallized from ethyl acetate hexane, m.p. 193-194°, yield 0.60 g. (80%); i.r.: 3460, 3380, 1665 cm^{-1}; n.m.r.: δ 7.8-7.2 (m, 5H); 7.35 (s, 5H); 1.90 (s, 1H); 1.80 (s, 1H); 1.40 (s, 3H); 1.27 (s, 3H); 1.12 (s, 3H); 0.92 (s, 3H) p.p.m.

Anal. Calcd. for $C_{22}H_{26}N_2O_2$: C, 75.40; H, 7.48; N, 7.99. Found: C, 74.99; H, 7.83; N, 7.73.

6-Benzamido-3,3,5,5-tetramethyl-2-piperidone (**7**).

A solution of the methoxylactam **4** (0.185 g., 0.001 mole), benzamide (0.121 g., 0.001 mole) in benzene (5 ml.) containing β-naphthalenesulfonic acid (0.020 g.) was refluxed for 3 hours. The solution was diluted with ethyl acetate (25 ml.), washed with aqueous bicarbonate and water, dried and evaporated. The residue was crystallized from ethyl acetate-hexane, m.p. 169-170°, yield 0.22 g. (81%); i.r.: 3440, 1665 cm^{-1}; n.m.r.: δ 8.0-7.8 (m, 2H); 7.6-7.4 (m, 3H); 7.1 (d, 1H, J = 9); 6.55 (s, 1H); 5.39 (q, 1H); 1.67 (s, 2H); 1.21 (s, 6H); 1.09 (s, 3H); 1.03 (s, 3H) p.p.m.

Anal. Calcd. for $C_{16}H_{22}N_2O_2$: C, 70.04; H, 8.08; N, 10.21. Found: C, 69.82; H, 8.17; N, 10.23; m/e = 274.

6-Dimedonyl-3,3,5,5-tetramethyl-2-piperidone (**8**).

A mixture of the methoxylactam **4** (0.185 g., 0.001 mole), dimedone (0.140 g., 0.001 mole) in benzene (10 ml.) containing β-naphthalenesulfonic acid was refluxed for 6 hours. The crystalline material which separated from the hot solution was filtered and recrystallized from ethanol, m.p. 235°, yield 0.3 g. (100%); i.r. (potassium bromide): 3260 and 1625 cm^{-1}.

Anal. Calcd. for $C_{17}H_{27}NO_3$: C, 69.54; H, 9 28; N, 4.77. Found: C, 69.11; H, 9.04; N, 4.80.

1,2,3,4-Tetrahydro-3,3,5,6-tetramethyl-2-pyridone (**9**).

A solution of the methoxylactam **4** in concentrated sulfuric acid (3 ml.) was left at room temperatures overnight (16 hours). The solution was then poured onto crushed ice and extracted into ethyl acetate. The ethyl acetate solution was washed with water, dried and evaporated. The residue was crystallized from hexane, m.p. 107-109°, yield 0.17 g. (64%); i.r.: 3410 and 1670; n.m.r.: δ 7.5 (s, 1H); 2.08 (s, 2H); 1.80 (s, 3H); 1.70 (s, 3H); 1.13 (s, 6H) p.p.m.; m/e = 153.

Anal. Calcd. for $C_9H_{15}NO$: C, 70.55; H, 9.87; N, 9.14. Found: C, 70.19; H, 9.79; N, 8.93.

Tris(3,3,5,5-tetramethyl-2-piperidone) (**10**).

A solution of the methoxylactam **4** (0.20 g.) in toluene (5 ml.) containing β-naphthalenesulfonic acid was refluxed for 20 hours. The solution was diluted with benzene (25 ml.) washed with water, dried and evaporated. The residue was triturated with methanol, filtered and crystallized from hexane, m.p. 160° (d); i.r.: 1650 cm^{-1}; n.m.r.: δ 5.93 (s, 1H); 5.65 (s, 1H); 3.93 (s, 1H); 2.81 (d, 1H, J = 12); 1.9-0.8 (singlets 41H); m/e = 459.

Anal. Calcd. for $C_{27}H_{45}N_3O_3$: C, 70.55; H, 9.87; N, 9.14. Found: C, 70.25; H, 9.99; N, 9.10.

1-Methoxy-3-isoindolinone (**11**).

This compound was prepared by the reduction of phthalimide according to Reissert and subsequent treatment of the 1-hydroxy-3-isoindolone with methanolic hydrogen chloride. The product melted at 99° (lit. (4) m.p. 98°) after crystallization from ethyl acetate-hexane; i.r.: 3440, 1725, 1090 and 1030 cm^{-1}; n.m.r.: δ 8.42 (s, 1H); 8.0-7.6 (m, 4H); 6.04 (s, 1H); 3.30 (s, 3H).

1-Phenyl-3-isoindolinone (**12**).

A suspension of the methoxy **11** (1.0 g.) described above in concentrated sulfuric acid containing benzene (2 ml.) was stirred for 16 hours. The acid solution was poured on crushed ice and extracted with ethyl acetate. The residue obtained after the removal of the solvent was crystallized from ethyl acetate-hexane, m.p. 216-218° (lit. (5) m.p. 218°), yield 0.65 g. (51%).

1-Acetonyl-3-isoindolinone (**13**).

A mixture of the methoxyisoindolone **11** (1.0 g.), excess ethyl acetoacetate (1.0 ml.) in concentrated sulfuric acid (5.0 ml.) was stirred at room temperature for 16 hours. The solution was poured onto crushed ice and extracted with ethyl acetate. The residue obtained after the removal of the solvent was crystallized from ethyl-hexane, m.p. 141-142°, yield 0.83 g. (65%); i.r.: 3460 and 1715 cm^{-1}; n.m.r.: δ 7.9-7.4 (m, 5H); 5.0 (q, 1H); 3.0 (2q, 2H); 2.23 (s, 3H); m/e = 189.

Anal. Calcd. for $C_{11}H_{11}NO_2$: C, 69.82; H, 5.86; N, 7.40. Found: C, 69.52; H, 5.97; N, 7.20.

Tris(isoindolone) (**14**).

3-Hydroxy-1-isoindolinone was heated to 250° for 10 minutes. The residue was triturated with methanol, filtered and crystallized from methanol, m.p. 310-311°, yield 35%; i.r.: 1720 cm^{-1}; n.m.r.: δ 8.4-7.5 (m, 12H); 7.18 (s, 1H); 6.0 (s, 1H); 5.9 (s, 1H); m/e = 393.

Anal. Calcd. for $C_{24}H_{15}N_3O_3$: C, 73.27; H, 3.85; N, 10.68. Found: C, 73.51; H, 3.91; N, 10.17.

1-Acetamido-3-isoindolinone (**15a**).

A mixture of the methoxyisoindolinone **11** (0.163 g., 0.001 mole), acetamide (0.06 g., 0.001 mole) and β-naphthalenesulfonic acid (0.010 g.) in benzene (10 ml.) was refluxed for 2 hours. The product which precipitated was filtered and crystallized from ethyl acetate, m.p. 253-254°, yield 76%; i.r. (potassium bromide): 3300, 1700 and 1675 cm^{-1}; m/e = 190.

Anal. Calcd. for $C_{10}H_{10}N_2O_2$: C, 63.15; H, 5.30; N, 14.73. Found: C, 63.26; H, 5.21; N, 14.86.

The methoxy **11** reacted similarly with benzamide and benzyl carbamate to give crystalline products which melted at 231° (65%) and 208° (76%), respectively.

Anal. Calcd. for $C_{15}H_{12}N_2O_2$: C, 71.41; H, 4.80; N, 11.11. Found: C, 71.31; H, 4.80; N, 11.26.

Anal. Calcd. for $C_{16}H_{14}N_2O_3$: C, 68.07; H, 5.00; N, 9.92. Found: C, 68.29; H, 5.26; N, 9.97.

The Reaction of **11** with 2,3-Dimethylbutadiene.

A solution of the methoxy **11** (2.0 g.), 2,3-dimethylbutadiene (2 ml. excess), trifluoroacetic acid (2 ml.) in benzene (50 ml.) was refluxed for 24 hours. The benzene solution was diluted with more benzene (50 ml.), washed with water and aqueous bicarbonate, dried and evaporated. The residue was triturated with methanol to remove the trimer formed (0.3 g.). The methanol was removed and the residue was chromatographed over basic alumina (80 g.), methylene chloride eluted the product (**16a**) which was crystallized from hexane, m.p. 120-121°, yield 0.86 g. (33%); i.r.: 1700 cm^{-1}; n.m.r. (carbon tetrachloride): 7.85-7.30 (m, 4H); 4.37 (d, 1H); 4.36 (q, 1H); 2.8-1.4 (m, 2H); 1.70 (s, 6H) p.p.m.; m/e = 213.

Anal. Calcd. for $C_{14}H_{15}NO$: C, 78.84; H, 7.09; N, 6.57. Found: C, 78.49; H, 7.06; N, 6.45.

The Reaction of **11** with 1,2,3,4-Tetramethylbutadiene.

The above procedure was repeated using β-naphthalenesulfonic

acid (0.4 g.) instead of trifluoroacetic acid. The product (**16b**) was crystallized from hexane, m.p. 107°, yield 1.2 g. (41%); i.r.: 1685 cm^{-1}; n.m.r.: (carbon tetrachloride): δ 7.9-7.2 (m, 4H); 4.43 (d, 1H, J = 4 cps); 4.10 (q, 1H); 3.8-1.9 (m, 1H); 1.78 (s, 3H); 1.67 (s, 3H); 1.62 (d, 3H, J = 7 cps); 0.56 (d, 3H, J = 7 cps); m/e = 241.

Anal. Calcd. for $C_{16}H_{19}NO$: C, 79.63; H, 7.94; N, 5.80. Found: C, 79.49; H, 8.08; N, 5.52.

1-Acetamido-1-phenyl-3-isoindolinone (**18**).

A mixture of *o*-benzoylbenzoic acid (**17**) (45.2 g., 0.2 mole), ammonium acetate (62 g., 0.5 mole), acetic acid (240 ml.) and toluene (750 ml.) was refluxed for 6 days and the water formed was removed by azeotropic distillation. The reaction mixture was cooled to 0° and the colourless precipitate was collected, triturated with hot water and dried at 60° under reduced pressure. It was crystallized from 2-propanol, m.p. 268°, yield 46.5 g. (88%); i.r.: 3430, 1710 and 1690 cm^{-1}; m/e = 266.

Anal. Calcd. for $C_{16}H_{14}N_2O_2$: C, 72.16; H, 5.30; N, 10.52. Found: C, 72.33; H, 5.21; N, 10.52.

1-Butoxy-1-phenyl-3-isoindolinone (**19**).

A mixture of the above described acetamidoisoindolinone (**18**) (62 g.) in 1-butanol (1500 ml.) containing β-naphthalenesulfonic acid (4.0 g.) was refluxed for 40 hours. The solid precipitate was collected and the butanol solution was evaporated. The residue was distributed between chloroform and water, the chloroform solution was separated, dried and evaporated. The residue was triturated with hexane, collected and crystallized from hexane, m.p. 98°, yield 45 g. (69%); i.r.: 3440 and 1720 cm^{-1}; n.m.r.: δ 7.9-7.2 (m, 10H); 3.7-2.8 (m, 2H), 1.8-1.2 (m, 4H).

Anal. Calcd. for $C_{18}H_{19}NO_2$: C, 76.84; H, 6.81; N, 4.98. Found: C, 77.19; H, 6.78; N, 5.05.

1-Phenyl-3-isoindolone (**20**).

A solution of 3-butoxy-3-phenyl-1-isoindolinone (**19**) (30 g.), thionyl chloride (90 ml.) in benzene (450 ml.) was refluxed for 16 hours. The solvent was removed and the residue was distilled at 172° (0.1 mm). The yellow oil which distilled solidified on cooling. It was triturated with dry ether and collected to give a yellow reactive crystalline material, 12.8 g. (58%), m.p. 144°; i.r. (carbon tetrachloride): 1755 and 1725 cm^{-1}; n.m.r. (carbon tetrachloride): δ 8.3-7.0 (m); U.V.: λ max (sulfuric acid) = 391 mμ (ε = 2.1 x 10^4), 349 mη (ε = 2.3 x 10^4), 240 mμ (ε = 1.6 x 10^4) and 217 mμ (ε = 1.7 x 10^4); m/e = 207.

Anal. Calcd. for $C_{14}H_9NO$: N, 6.76. Found: N, 6.44.

The 3-phenyl-1-isoindolone reacted very fast with methanol and ethanol to give the corresponding 3-alkoxy derivatives **21a** (m.p. 127°) and **21b** (m.p. 149°). It was also reduced in dry glyme with sodium borohydride at room temperature within seconds to give a product identical with 3-phenyl-1-isoindolinone described above.

The Reaction of **19** with 2,3-Methylbutadiene (**22**).

A solution of the butoxy **19** (0.5 g.), excess 2,3-dimethylbutadiene (1.0 ml.) and β-naphthalenesulfonic acid (0.1 g.) in benzene (10 ml.) was refluxed for 40 hours and the residue was chromatographed over neutral alumina and eluted with benzene-methylene chloride (1:1). The crystalline product was recrystallized from ethyl acetate-hexane, m.p. 131-132°, yield 0.4 g. (78%); i.r. (carbon tetrachloride): 1710 and 1680 cm^{-1}; n.m.r.: δ 8.0-7.8 (m, 1H); 7.2 (s, 5H); 4.55 (d, 1H, J = 17 cps); 3.25 (d, 2H, J = 17 cps); 2.28 (d, 1H, J = 17 cps); 1.75 (s, 3H); 1.59 (s, 3H); m/e = 289.

Anal. Calcd. for $C_{20}H_{19}NO$: C, 83.01; H, 6.61; N, 4.84. Found: C, 82.84; H, 6.36; N, 5.04.

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