

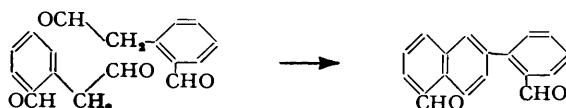
The Self-condensation of Homophthalaldehyde.

By K. T. POTTS and SIR ROBERT ROBINSON.

[Reprint Order No. 6204.]

A new view of the constitution of the product obtained by the action of hot aqueous sodium hydroxide on *isoquinoline*-sulphur trioxide is proposed and validated by the synthesis of the methyl-*o*-tolylphthalene of which the substance is the dioxo-derivative.

It is well known that hydrolytic fission of a pyridine nucleus provides derivatives of glutcondialdehyde (cf. "Heterocyclic Compounds," Ed. by Elderfield, Vol. I, p. 423, John Wiley and Sons Inc., New York). Thus the action of alkali on *isoquinoline*-sulphur trioxide should give homophthalaldehyde but Baumgarten and Olshausen (*Ber.*, 1931, **64**, 925) found that in fact the product was a phenylphthalenedialdehyde. Later (*Ber.*, 1935, **68**, 1316) Baumgarten showed that the same compound, $C_8H_{12}O_3$, could also be obtained in a similar manner from homophthalaldehyde prepared by the method of Blount and Robinson (*J.*, 1933, 555). The self-condensation thus disclosed was formulated as follows :



This is fully analogous to the condensation of phenylacetaldehyde to 2-phenylphthalene which, however, occurs under the influence of *acids*.

Although there are certain examples of ring-closure to a benzene ring which can be brought about by drastic methods using basic catalysts (*e.g.*, indoxyl from phenylglycine), it seemed to us that a more probable course of the reaction is that represented in the scheme :



On reduction by the Wolff-Kishner process, the dialdehyde gives a dimethylphenyl-naphthalene and the synthesis of this substance has been effected with the result that the correctness of our view has been demonstrated.

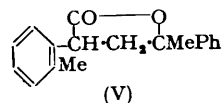
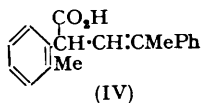
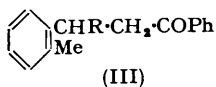
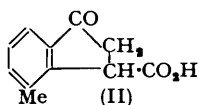
The first route attempted started with the Friedel-Crafts condensation of *o*-tolylsuccinic anhydride with benzene. Had this proceeded normally and similarly to the condensation of phenylsuccinic anhydride and veratrole (Robinson and Young, *J.*, 1935, 1414; cf. Rice, *J. Amer. Chem. Soc.*, 1931, **53**, 3159; Ali, Desai, Hunter, and Muhammed, *J.*, 1937, 1013) the later stages of the projected synthesis would probably have presented little difficulty. The product, under many conditions tried, was always 4-methylindan-1-one-3-carboxylic acid (II). This was decarboxylated in hot quinoline in the presence of copper bronze, to 4-methylindan-1-one (Young, *Ber.*, 1892, **25**, 2104).

The ultraviolet spectrum of the acid (II) was normal for the type, and the infrared spectrum showed absorption bands at 5.81 (unconjugated carboxyl), 5.94 (carbonyl in five-membered ring), and 12.75 μ (1 : 2 : 3-trisubstituted benzene).

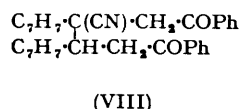
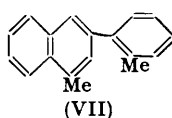
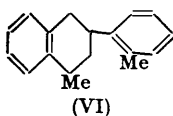
The reaction between *o*-tolylsuccinic anhydride and phenylmagnesium bromide offered an alternative but this approach was unsuccessful.

o-Tolylideneacetophenone, prepared by an application of Weygand and Schächer's method (*Ber.*, 1935, **68**, 227), added hydrogen cyanide in the normal manner in the presence of acetic acid (cf. *Org. Synth.*, Coll. Vol., II, p. 498), yielding the nitrile (III; R = CN) which was then converted into the related amide (III; R = CO·NH₂) and methyl ester (III; R = CO₂Me). The nitrile gave very poor yields of alcohol on reaction with methylmagnesium iodide, but the ester afforded a mixture of 4-phenyl-2-*o*-tolylpent-3-enoic acid

(IV) and the related γ -lactone (V); prolonged refluxing of the reaction mixture favoured the formation of the acid (cf. Kloetzel, *J. Amer. Chem. Soc.*, 1940, **62**, 1708). Both products were utilised; the acid (IV) was reduced catalytically (Adams), then cyclised to an



α -tetralone (anhydrous hydrofluoric acid) which was reduced to a tetralol (lithium aluminium hydride) and dehydrogenated (palladised charcoal) to the desired 1-methyl-3-*o*-tolynaphthalene (VII). The lactone (V) was reduced, cyclised, and further reduced in one operation by heating with hydriodic acid, syrupy phosphoric acid, and red phosphorus (cf. Miescher and Billeter, *Helv. Chim. Acta*, 1939, **22**, 605; Dev, *J. Indian Chem. Soc.*, 1948, **25**, 323). The product was 1-methyl-3-*o*-tolyltetralin (VI) which was dehydrogenated to the naphthalene (VII) by means of palladised charcoal.



When *o*-tolylideneacetophenone was treated with potassium cyanide, the solution being alkaline, 3-cyano-1 : 6-diphenyl-3 : 4-di-*o*-tolylhexane-1 : 6-dione (VIII) was produced. It is assumed that initial formation of the normal adduct (III; R = CN) is followed by a Michael-type reaction with a second molecule of the unsaturated ketone; this process appears to be reversed when the substance is heated above its melting point.

The substance (VIII) shows no infrared absorption band which can be attributed to a cyano-group, whereas the nitrile (III; R = CN) showed both nitrile and carbonyl bands.

Dr. A. S. Bailey has kindly informed us that cyanodihydrosanguinarine also exhibits no nitrile absorption in the infrared region.

EXPERIMENTAL

Ultraviolet spectra were recorded in methanol, and evaporations were carried out under reduced pressure on the steam-bath.

*iso*Quinoline-Sulphur Trioxide.—The method of Baumgarten and Olshausen (*loc. cit.*) was used, the only variation being that the sulphonic acid was collected after about 5 min. to prevent any contamination by *iso*quinoline hydrochloride, which tended to separate when the solution was cooled for longer periods. The m. p. of an analytical specimen was 254—255° (lit., 250—254°) and tended to vary with the rate of heating (Found: C, 51.6; H, 3.4%). Calc. for $C_9H_7O_3NS$: C, 51.6; H, 3.4%. Light absorption: λ_{max} , 2300, 2650, 2750, 3320 ($\log \epsilon$ 4.31, 3.29, 3.29, 3.64), λ_{min} , 2500, 2700, 2950 ($\log \epsilon$ 3.01, 3.28, 2.84).

2-Phenyl-naphthalene-2' : 4-dialdehyde (I).—*iso*Quinoline-sulphur trioxide (4.2 g.) was heated with an aqueous solution of sodium hydroxide (4.8 g. in 50 c.c.). The yellow solution soon became turbid and after 5 minutes' heating was cooled. 2-Phenyl-naphthalene-2' : 4-dialdehyde separated as needles (1.2 g.; m. p. 125—126°). It crystallised from aqueous methanol as needles, m. p. 126° (Baumgarten recorded m. p. 126.5° (Found: C, 82.7; H, 4.8. Calc. for $C_{18}H_{12}O_2$: C, 83.1; H, 4.7%). With concentrated sulphuric acid the aldehyde gave a yellow solution which became red when heated. It was insoluble in cold, concentrated nitric acid, and, on warming, a clear yellow solution was obtained. The di(phenylhydrazone), red needles from pentyl alcohol, had m. p. 198—200°, and the dioxime, white needles from aqueous alcohol, had m. p. 199°, in agreement with the values reported by Baumgarten.

1-Methyl-3-*o*-tolyl-naphthalene (VII).—A mixture of 2-phenyl-naphthalene-2' : 4-dialdehyde (1.6 g.), ethylene glycol (30 c.c.), 90% hydrazine hydrate (6 c.c.), and sodium hydroxide (3 g.) was refluxed for 1 hr. and the temperature then raised to *ca.* 200° for a further hour. Water (50 c.c.) was added to the cooled solution and the product isolated by ether-extraction in the usual manner. 1-Methyl-3-*o*-tolyl-naphthalene distilled as a viscous oil, b. p. 118—120°/0.05 mm. (Found: C, 92.9; H, 6.8. $C_{18}H_{16}$ requires C, 93.1; H, 6.9%). The 1 : 3 : 5-trinitrobenzene

complex was prepared in alcohol and it crystallised from methanol as yellow needles, m. p. 108—109° (Found : C, 54.8; H, 3.4; N, 12.8. $C_{18}H_{16}, 2C_6H_3O_6N_3$ requires C, 54.7; H, 3.4; N, 12.8%). The picrate was not suitable for purposes of characterisation.

o-Tolylsuccinic Acid.—In the preparation of α -cyano-*o*-methylcinnamic acid (cf. *Org. Synth.*, Coll. Vol. I, 2nd edn., pp. 181, 451) the yield was raised to about 74% when the amount of sodium hydroxide employed in the condensation was increased. The acid crystallised from water as hexagonal plates, m. p. 204—205° (Fiquet, *Ann. Chim.*, 1893, 29, 484, gives m. p. 202°) (Found : C, 70.4; H, 4.8. Calc. for $C_{11}H_9O_2N$: C, 70.6; H, 4.8%). The ethyl ester was obtained in the usual way and crystallised from aqueous alcohol as large needles, m. p. 60° (*idem, loc. cit.*, reports m. p. 60°).

Ethyl $\alpha\beta$ -dicyano- β -o-methyltolylpropionate can be hydrolysed to *o*-tolylsuccinic acid without further purification but a small amount was dissolved in ether and dried. It distilled as a slightly viscous oil, b. p. 155°/0.5 mm. (Found : C, 69.5; H, 6.0. $C_{14}H_{14}O_2N_2$ requires C, 69.4; H, 5.8%). *o*-Tolylsuccinic acid crystallised from water as small, irregular prisms, m. p. 181—182°, in agreement with Alder and Schmitz (*Annalen*, 1949, 565, 99).

o-Tolylsuccinic Anhydride.—*o*-Tolylsuccinic acid (25 g.) was refluxed with pure, redistilled acetyl chloride (175 c.c.) for 2 hr., most of it had dissolved after 30 min. Excess of acetyl chloride and acetic acid was removed and the residue distilled as a very viscous oil (20.4 g., 90%), b. p. 145°/0.6 mm. On redistillation, *o*-tolylsuccinic anhydride was obtained as a viscous oil, b. p. 141°/0.5 mm. The anhydride crystallised after several months and was obtained as waxy prisms, m. p. 45—46°, by recrystallisation from ether (Found : C, 69.2; H, 5.3. $C_{11}H_{10}O_3$ requires C, 69.5; H, 5.3%). The *anilide*, readily obtained from the anhydride (300 mg.) and aniline (20 mg.) in benzene 8 c.c.), crystallised from aqueous methanol as needles, m. p. 167—168° (Found : C, 72.3; H, 6.0. $C_{17}H_{17}O_3N$ requires C, 72.1; H, 6.1%).

7-Methyl-3-oxoindane-1-carboxylic Acid (II).—A mixture of *o*-tolylsuccinic anhydride (4.0 g.) and thiophen-free benzene (6.0 g.) was added to a solution of freshly sublimed aluminium chloride (7.5 g.) in nitrobenzene (20 c.c.) and kept in a stoppered flask for 48 hr. The complex was decomposed by ice and dilute hydrochloric acid, and the nitrobenzene and excess of benzene removed by steam-distillation. The remaining oil solidified; this was dissolved in sodium hydrogen carbonate solution and the solution extracted with ether. Acidification with dilute hydrochloric acid gave a partly crystalline precipitate (2.1 g., 50%; m. p. 154°). *7-Methyl-3-oxoindane-1-carboxylic acid* crystallised from benzene as irregular prisms, m. p. 157—158° (Found : C, 69.8, 69.6; H, 5.4, 5.5. $C_{11}H_{10}O_3$ requires C, 69.5; H, 5.3%). Light absorption : λ_{\max} . 2500, 2950 (log ϵ 4.04, 3.34), λ_{\min} . 2300, 2750 (log ϵ 3.62, 3.16). The acid gave positive reactions in the sodium nitroprusside-alkali and *m*-dinitrobenzene-alkali colour tests. With concentrated sulphuric acid it gave a yellow colour, becoming orange-red on warming and fading on dilution of the solution. The 2 : 4-dinitrophenylhydrazone crystallised from methanol as clusters of bright red needles, m. p. 259—260° (decomp.) (Found : C, 55.4; H, 3.7; N, 14.6. $C_{17}H_{14}O_6N_4$ requires C, 55.1; H, 3.8; N, 15.1%).

7-Methylindane-1-carboxylic Acid.—Amalgamated zinc was prepared by shaking zinc wool (1 g.), mercuric chloride (0.5 g.), concentrated hydrochloric acid (1 c.c.), and water (5 c.c.) for 5 min. and then decanting the solution. *7-Methyl-3-oxoindane-1-carboxylic acid* (500 mg.), concentrated hydrochloric acid (2 c.c.), water (2 c.c.), and the amalgamated zinc were refluxed together for 8 hr. Concentrated hydrochloric acid (1 c.c.) was added at intervals of 3 hr. Overnight the oily product crystallised. *7-Methylindane-1-carboxylic acid* crystallised from water as glistening needles (450 mg.), m. p. 107° (Found : C, 75.2; H, 6.8. $C_{11}H_{12}O_2$ requires C, 75.0; H, 6.9%). The acid was soluble in most organic solvents and insoluble in water. It sublimed readily at atmospheric pressure and was slightly volatile in steam. Light absorption : λ_{\max} . 2100, 2650, 3200 (log ϵ 4.11, 2.68, 2.27), λ_{\min} . 2500, 2850 (log ϵ 2.49, 2.01).

4-Methylindan-1-one.—A mixture of *7-methyl-3-oxoindane-1-carboxylic acid* (140 mg.), copper bronze (200 mg.), and quinoline (5 c.c.) was refluxed for 30 min. Ether was added to the cooled solution which was extracted with 10% hydrochloric acid, then with 2*N*-sodium hydroxide, washed with water, and dried (Na_2SO_4). Evaporation of the ether gave an oily residue which rapidly crystallised. *4-Methylindan-1-one* separated from light petroleum as irregular prisms, m. p. 95—96° (Young, *Ber.*, 1892, 25, 2104, reports m. p. 95°). It gave a positive reaction in the *m*-dinitrobenzene-alkali colour test. The phenylhydrazone crystallised from aqueous methanol as golden needles, m. p. 133° (*idem, loc. cit.*, reports m. p. 132°).

2-Methylstyryl Phenyl Ketone.—*o*-Tolualdehyde (2.0 g.; *Org. Synth.*, 1950, 30, 99) was added to a mixture of acetophenone (2.0 g.), sodium hydroxide (0.5 g.), water (5 c.c.), and 95% ethanol (5 c.c.), and the whole shaken for 2 hr., during which an oil separated. After

24 hr. at 0°, an equal volume of water was added and the product isolated by means of ether. After a small fore-run of acetophenone, 2-methylstyryl phenyl ketone distilled as a pale yellow, slightly viscous oil (3.4 g., 92%), b. p. 152°/0.5 mm. (Weygand and Schächer, *Ber.*, 1935, 68, 227, give b. p. 218—219°/12 mm.). The 2:4-dinitrophenylhydrazone crystallised from methanol as orange needles, m. p. 167° (Found: C, 65.9; H, 4.4. $C_{23}H_{18}O_4N_4$ requires C, 65.7; H, 4.5%). The semicarbazone separated from aqueous methanol (charcoal) as white needles, m. p. 168° (Found: C, 72.9; H, 6.2. $C_{17}H_{17}ON_3$ requires C, 73.2; H, 6.1%).

β -Benzoyl- α -o-tolylpropionitrile (III; R = CN).—A solution of 2-methylstyryl phenyl ketone (4.4 g.) in 95% ethanol (70 c.c.) containing acetic acid (1.2 g.) was heated to 35° and then, with stirring, potassium cyanide (2.6 g.) in water (10 c.c.) was added. The stirring was continued for 3 hr. and, after standing overnight at 0°, the product (3.5 g., 73%; m. p. 84—85°) crystallised. The nitrile crystallised from 90% ethanol as needles, m. p. 89° (Found: C, 81.5; H, 6.3. $C_{17}H_{15}ON$ requires C, 81.9; H, 6.1%). The 2:4-dinitrophenylhydrazone crystallised from methanol as orange, irregular prisms, m. p. 163—164° (Found: C, 63.9; H, 4.4. $C_{23}H_{18}O_4N_5$ requires C, 64.3; H, 4.5%).

3-Cyano-1:6-diphenyl-3:4-di-o-tolylhexane-1:6-dione (VIII).—2-Methylstyryl phenyl ketone (3.3 g.) was treated with 50% ethanol (10 c.c.) and powdered sodium cyanide (1.5 g.). The oil did not completely dissolve and, after 30 min. on the steam-bath, became very viscous and partly solid. An equal volume of water was added, and, with cooling, an excess of concentrated hydrochloric acid. After decantation of the mother-liquor the resultant gum was dissolved in hot glacial acetic acid, and the product (0.7 g., m. p. 241—242°) crystallised from the cooled solution. It separated from dilute acetic acid as rectangular plates, m. p. 246° (Found: C, 83.9; H, 6.4; N, 3.5. $C_{33}H_{29}O_2N$ requires C, 84.1; H, 6.2; N, 3.0%). It dissolved in concentrated sulphuric acid to an intense red solution. With concentrated nitric acid a cherry-red colour was obtained.

β -Benzoyl- α -o-tolylpropionamide.—Concentrated sulphuric acid (10 c.c.) was added during 15 min. to a stirred suspension of β -benzoyl- α -o-tolylpropionitrile (10 g.) in glacial acetic acid (50 c.c.). After an hour at the room temperature, the deep red solution was poured on ice and next day the crystalline material (8.7 g., 82%; m. p. 159°) was collected. The amide crystallised from benzene as needles, m. p. 161° (Found: C, 76.2; H, 6.5. $C_{17}H_{17}NO_2$ requires C, 76.4; H, 6.4%).

Methyl β -Benzoyl- α -o-tolylpropionate (III; R = CO₂Me).—A mixture of β -benzoyl- α -o-tolylpropionamide (8.7 g.), ethanol (80 c.c.), and 2N-sodium hydroxide (80 c.c.) was refluxed until the evolution of ammonia ceased (10 hr.). Most of the alcohol was distilled off and water (50 c.c.) and excess of 3N-hydrochloric acid were added to the filtered liquid. The resultant gum was separated and dissolved in the minimum volume of methanol, and this solution treated with an excess of ethereal diazomethane. After 2 hr. at room temperature the solvent was removed and the residue crystallised from aqueous methanol. The methyl ester (4.5 g., 50%) separated from aqueous methanol as prisms, m. p. 90° (Found: C, 76.2; H, 6.4. $C_{18}H_{18}O_3$ requires C, 76.6; H, 6.4%). When the gum which separated after acidification of the reaction mixture was crystallised from glacial acetic acid needles of 4-hydroxy-4-phenyl-2-o-tolylbut-3-enoic lactone [2:3-dihydro-2-oxo-5-phenyl-3-o-tolylfuran], m. p. 248° (Found: C, 81.1; H, 5.8. $C_{17}H_{14}O_2$ requires C, 81.6; H, 5.6%), were obtained.

Reaction of Methyl β -Benzoyl- α -o-tolylpropionate with Methylmagnesium Iodide.—Methylmagnesium iodide (magnesium, 3.4 g.; methyl iodide, 19.6 g.) in ether (65 c.c.) was added with stirring to a solution of methyl β -benzoyl- α -o-tolylpropionate (28.2 g.) in ether (300 c.c.) at 0°. After being stirred for a further 30 min. at 0°, the mixture was allowed to reach the room temperature and then heated at 50—60° for 3 hr. During this period the precipitate formed a viscous, semi-solid mass. The complex was decomposed with ice and dilute hydrochloric acid, the ethereal layer was immediately separated, and the mother-liquor was extracted with a further quantity of ether (3 × 30 c.c.). After being shaken with sodium carbonate solution (3 × 20 c.c.) the extract was dried (Na₂SO₄) and evaporated, and the residual oil crystallised on the addition of a little methanol. δ -Hydroxy- δ -phenyl- α -o-tolylvaleric lactone (V) (5.0 g.) separated from methanol or light petroleum as beautiful needles, m. p. 120° (Found: C, 81.4; H, 6.9. $C_{18}H_{16}O_2$ requires C, 81.2; H, 6.8%). A well-developed infrared band due to the carbonyl of the γ -lactone occurred at 5.64 μ . The sodium carbonate extract was decolorised by boiling with charcoal and, after cooling, was acidified with dilute hydrochloric acid. The oil which separated was isolated by means of ether in the usual manner and distilled as a pale yellow, very viscous oil (7.2 g.), b. p. ca. 200°/1 mm. 4-Phenyl-2-o-tolylpent-3-enoic acid (IV) crystallised from methanol as white needles, m. p. 117° (Found: C, 81.2; H, 6.9. $C_{18}H_{16}O_2$

requires C, 81.2; H, 6.7%). Esterification (diazomethane) gave *methyl 4-phenyl-2-o-tolylpent-3-enoate*, b. p. 145—147°/0.5 mm., n_D^{23} 1.6610 (Found: C, 81.5; H, 7.2. $C_{19}H_{20}O_2$ requires C, 81.4; H, 7.2%). A carbonyl band was present at 5.78 μ .

δ -Phenyl- α -o-tolylvaleric Acid.—A solution of the above unsaturated acid (5.0 g.) in methanol (30 c.c.) was shaken with hydrogen in the presence of Adams catalyst (0.8 g.) at room temperature and pressure. The theoretical volume of hydrogen (580 c.c.) was absorbed in 15 min. After isolation, the saturated *acid* distilled as a very viscous oil (4.4 g., 88%), b. p. 195°/1.5 mm. It crystallised from light petroleum as needles, m. p. 69—70° (Found: C, 80.5; H, 7.4. $C_{18}H_{20}O_2$ requires C, 80.5; H, 7.5%). With ethereal diazomethane it gave a *methyl ester*, b. p. 145° (bath-temp.)/1.5 mm., n_D^{23} 1.4260 (Found: C, 80.6; H, 7.9. $C_{19}H_{22}O_2$ requires C, 80.8; H, 7.9%).

1 : 2 : 3 : 4-Tetrahydro-4-methyl-1-oxo-2-o-tolyl-naphthalene.—The foregoing acid (7.6 g.) was dissolved in anhydrous hydrogen fluoride (ca. 20 c.c.), and the copper vessel closed to the atmosphere. After 72 hr. the excess of hydrogen fluoride was removed in a stream of air on the steam-bath, the residue dissolved in ether, and the extract washed first with sodium carbonate solution and then with water. After drying and evaporation the viscous residue was distilled and the *tetralone* (3.6 g., 50%) obtained as a pale lemon-coloured, viscous oil, b. p. 147—150°/0.5 mm. (Found: C, 86.5; H, 7.3. $C_{18}H_{18}O$ requires C, 86.4; H, 7.3%). A prominent band (due to CO) was present at 5.95 μ . The *2 : 4-dinitrophenylhydrazone* crystallised from acetone as orange-red needles, m. p. 279—280° (Found: C, 66.9; H, 4.8. $C_{24}H_{22}O_4N_4$ requires C, 67.0; H, 5.2%).

1 : 2 : 3 : 4-Tetrahydro-1-hydroxy-4-methyl-2-o-tolyl-naphthalene.—The above tetralone (2.3 g.) in ether (40 c.c.) was added, slowly and with stirring, to a slurry of lithium aluminium hydride (3.5 g.) in ether (40 c.c.). After 30 minutes' stirring the excess of hydride was decomposed with dilute hydrochloric acid, and the ethereal layer separated, washed with water, and dried. The solution had an intense blue fluorescence in ultraviolet light. The *tetralol* distilled as a viscous fluorescent oil (1.5 g., 65%), b. p. 162°/0.5 mm. (Found: C, 85.8; H, 7.9. $C_{18}H_{20}O$ requires C, 85.7; H, 8.0%). Light absorption: λ_{max} , 2040, 2550, 3800, 4200 (log ϵ 5.24, 4.46, 3.58, 3.56); λ_{min} , 3700, 4000, 4200 (log ϵ 3.43, 3.47, 3.56). An absorption band was present at 2.96 μ (OH).

1 : 2 : 3 : 4-Tetrahydro-1-methyl-3-o-tolyl-naphthalene (VI).— δ -Hydroxy- δ -phenyl- α -tolylvaleric lactone (4.1 g.), syrupy phosphoric acid (20 c.c.; *d*, 1.75), iodine (2.0 g.), and red phosphorus (4.0 g.) were heated together at 160—170° for 72 hr., then cooled, diluted with water, and extracted with ether. The extract was shaken with sodium carbonate solution (3 \times 20 c.c.), washed with water, and dried (Na_2SO_4). After evaporation of the ether the *tetrahydronaphthalene* remained as a pale oil and distilled as a colourless, mobile oil (2.3 g., 64%) with a characteristic odour, b. p. 155°/1.5 mm., n_D^{23} 1.5880 (Found: C, 91.4; H, 8.5. $C_{18}H_{20}$ requires C, 91.5; H, 8.5%). The specimen for analysis was distilled from sodium.

1-Methyl-3-o-tolyl-naphthalene (VII).—(a) *1 : 2 : 3 : 4-Tetrahydro-1-hydroxy-4-methyl-2-o-tolyl-naphthalene* (0.6 g.) was heated with palladised charcoal (0.5 g.) at 270—310° in a stream of nitrogen for 2 hr. The product, isolated by means of ether, was dissolved in alcohol, and *1 : 3 : 5-trinitrobenzene* in alcohol added. The trinitrobenzene complex separated as yellow needles, m. p. 108°, alone and when mixed with a specimen prepared from the hydrocarbon obtained as above from the dialdehyde of Baumgarten and Olshausen (*loc. cit.*). The infrared spectra of the trinitrobenzene complexes were identical in all respects. (b) *1 : 2 : 3 : 4-Tetrahydro-1-methyl-3-o-tolyl-naphthalene* (0.7 g.) was heated with palladised charcoal (0.6 g.) at 270—310° in a stream of nitrogen for 2 hr. The product was isolated as the trinitrobenzene complex, m. p. 106—107°, raised to 108—109° on recrystallisation from methanol. The m. p. was not depressed on admixture with an authentic specimen of *1-methyl-3-o-tolyl-naphthalene* trinitrobenzene complex.