The Synthesis of Alkylphenanthrenes Relevant to the Investigations on Methyl Vinhaticoate.

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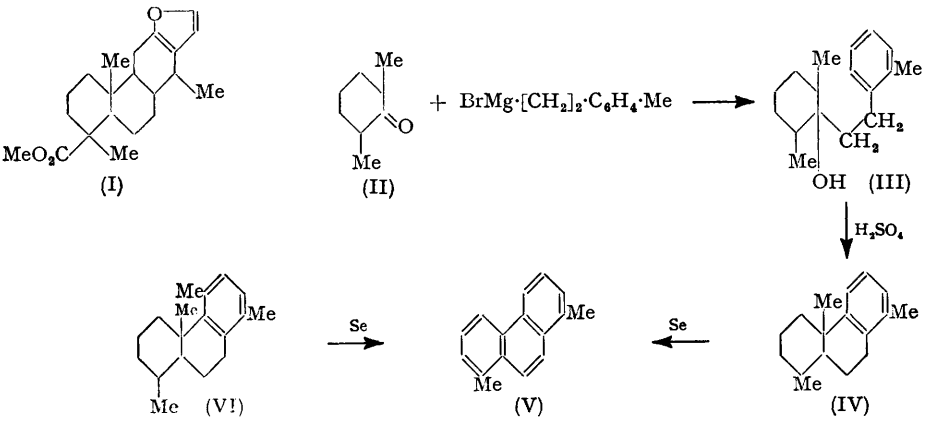
Syntheses have been effected by the Bogert–Cook method of 1 : 8-dimethyl-, 1-ethyl-8-methyl-, and 1 : 3 : 8-, 1 : 4 : 8-, and 2 : 4 : 8-trimethylphenanthrene. The appropriate phenylethylmagnesium bromides were condensed with 2 : 6-dimethyl*cyclo*hexanone and the products cyclised with sulphuric acid to octahydrophenanthrenes with an angular methyl group. These were aromatised with selenium except in the case of the 1 : 4 : 8 : 13-tetramethyl compound which gave 1 : 8-dimethylphenanthrene and required sulphur dehydrogenation for the isolation of the 1 : 4 : 8-trimethyl derivative.

A description is also included of the synthesis of 2-ethyl-1 : 5-dimethylnaphthalene.

The structure assigned to methyl vinhaticoate (I) (King and King, *J.*, 1953, 4158) rests on information obtained by the aromatisation of various derivatives of the diterpene to alkylphenanthrenes, including 1 : 8-dimethyl- (Haworth, Mavin, and Sheldrick, *J.*, 1943, 454), 1-ethyl-8-methyl-, and 1 : 3 : 8-trimethyl-phenanthrene. The orientations of these end products of (I) were ascertained, as already described (King and King, *loc. cit.*), by comparison with authentic specimens which together with two other relevant compounds, namely, 1 : 4 : 8- and 2 : 4 : 8-trimethylphenanthrene, were synthesised for this purpose by the Bogert–Cook procedure. The following is an account of the synthetical operations.

The conditions employed are substantially those described by Papa, Perlman, and Bogert (*J. Amer. Chem. Soc.*, 1938, **60**, 319) and by Huebner and Jacobs (*J. Biol. Chem.*, 1947, **170**, 203), and the reaction sequence (II)—(V) relating to the preparation of 1 : 8-dimethylphenanthrene illustrates the general method.



The choice of 2 : 6-dimethyl*cyclo*hexanone (II) rather than of the isomeric 2 : 2-dimethyl compound as a component of the synthesis depends on its considerably superior reactivity in the Grignard reaction. An improved preparation of the required ketone (II) from 2-methyl*cyclo*hexanone was based on the method of Sen and Mondal (*J. Indian Chem. Soc.*, 1928, **5**, 609; see also Cornforth and Robinson, *J.*, 1949, 1859). Formylation and *C*-methylation at the 6-position followed by removal of the formyl group gave 2 : 6-dimethyl*cyclo*hexanone (over-all yield 45%), unchanged 2-methyl*cyclo*hexanone being removed by extraction as the bisulphite compound. The 2-arylethyl bromides were obtained by a standard procedure from the appropriately substituted arylmagnesium bromides and ethylene oxide, phosphorus tribromide being used to esterify the resulting arylethyl alcohols. Ring-closure of the alcohols, *e.g.*, (III), was carried out with 85% sulphuric acid, and the octahydrophenanthrenes, for example, (IV), were aromatised with the loss of the angular substituent by heating them with selenium. The intermediate tertiary alcohols and the octahydrophenanthrenes were, with one exception, liquids consisting presumably of mixed stereoisomers; unsatisfactory analytical results were sometimes obtained owing to the presence of varying amounts of diarylbutanes formed by self-condensation of the halides in the Grignard reaction. All the phenanthrenes were crystalline solids characterised by picrates and trinitrobenzene and trinitrofluorenone derivatives. Where quantities allowed, the phenanthraquinones and derived quinoxalines were also prepared.

No difficulties were encountered in the synthesis of 1 : 8-dimethyl-, 1-ethyl-8-methyl-, and 1 : 3 : 8-trimethyl-phenanthrene, but selenium dehydrogenation of the tetramethyl derivative (VI) yielded primarily the 1 : 8-dimethyl compound (V) and although some of the expected 1 : 4 : 8-trimethylphenanthrene was undoubtedly present its isolation was impracticable. Even after most careful purification of the initial bromo-*p*-xylene, which from the production of (V) was at first supposed to contain *o*-bromotoluene, the formation of 1 : 8-dimethylphenanthrene continued. However, with the substitution of sulphur as dehydrogenating agent 1 : 4 : 8-trimethylphenanthrene was readily obtained at 240°; loss of the 4-methyl group can therefore be attributed to the higher temperature (300°) required in the selenium reaction.

The expulsion of methyl from an already aromatised ring during dehydrogenation with selenium has not previously been reported, even in the closely comparable synthesis of 1 : 4-dimethylphenanthrene (Papa *et al., loc. cit.*). In order to determine whether elimination of the 4-methyl group occurs in other trimethylphenanthrenes the preparation of the 2 : 4 : 8-trimethyl derivative was undertaken, but not only was the trimethylphenanthrene readily obtained from the selenium dehydrogenation of the requisite intermediate, but no trace of the alternative well-characterised dimethyl compound (pimanthrene) was detected in the reaction mixture.

The synthesis of 2-ethyl-1 : 5-dimethylnaphthalene was also included in this project since it was of interest as a potential dehydrogenation product of a dicarboxylic acid obtained in the oxidative degradation of methyl vinhaticoate. 2-Ethyl-5-methyl-1-tetralone (Harvey, Heilbron, and Wilkinson, *J.*, 1930, 423), prepared by a slight modification of the original method, was treated with methylmagnesium iodide. The resulting mixture of ethyldimethyltetralol and ethyl 3 : 4-dihydrodimethylnaphthalene when heated with selenium gave the liquid 2-ethyl-1 : 5-dimethylnaphthalene which was characterised by crystalline derivatives.

EXPERIMENTAL

2 : 6-*Dimethyl*cyclo*hexanone* (II).—2-Hydroxymethylene-6-methyl*cyclo*hexanone, prepared by the method of Johnson and Posvic (*J. Amer. Chem. Soc.*, 1947, **69**, 1361) and once distilled, was methylated with excess of methyl iodide–potassium carbonate in boiling acetone for 12 hr. The acetone was then distilled off, the residue treated with a large excess of 20% aqueous sodium hydroxide, and the solution distilled in steam. The distillate was extracted with ether, and the ethereal solution repeatedly shaken with saturated aqueous sodium hydrogen sulphite until no more ketone was extracted. The ethereal solution was then dried and distilled, pure 2 : 6-dimethyl*cyclo*hexanone (yield 45%) being collected between 172—173° (lit., b. p. 170—171°). The product formed a 2 : 4-dinitrophenylhydrazone, m. p. 149—150° (Birch, *J.*, 1947, 1642, gives m. p. 149—150°).

2 : 6-*Dimethyl*-1-(2-o-*tolylethyl*)cyclo*hexanol* (III).—An ethereal solution of 2 : 6-dimethyl-*cyclo*hexanone (10 g., 1 mol.) was added to one of Grignard reagent prepared from 2-*o*-tolylethyl bromide (16 g.) (cf. Shoesmith and Connor, *J.*, 1927, 1768), and the mixture set aside overnight. After 6 hr.' boiling, the solution was treated with ice and dilute hydrochloric acid, and the dried ethereal layer was distilled. The *alcohol* (III), b. p. 150—160° (bath)/1 mm., consisted of a pale yellow viscous oil (11 g., 58%) which became partly crystalline. The solid (3 g.) crystallised from a very small volume of methanol in colourless prisms, m. p. 55—57° (Found: C, 83·3; H, 10·3. $C_{17}H_{26}O$ requires C, 82·9; H, 10·6%). Redistillation of the oil from which the solid had separated gave a similar product (Found: C, 82·9; H, 10·0%) but which did not crystallise.

1 : 8 : 12-*Trimethyl*-1 : 2 : 3 : 4 : 9 : 10 : 11 : 12-*octahydrophenanthrene* (IV).—The crude alcohol (III) (10 g.) was slowly added to ice-cold sulphuric acid (85%; 30 c.c.) with constant shaking.

After 15 min. the mixture was extracted with light petroleum (b. p. 60—80°), and the petroleum solution thoroughly washed, first with 85% sulphuric acid, and then with water. The dried liquid was distilled, giving the *hydrophenanthrene* (IV) (7·7 g., 83%), b. p. after redistillation from sodium 204—206°/30 mm. (Found : C, 89·3; H, 10·1. $C_{17}H_{24}$ requires C, 89·4; H, 10·6%).

1 : 8-*Dimethylphenanthrene* (V).—The hydrophenanthrene (IV) (2 g.) was heated with selenium (4 g.) for 40 hr. at 320—340° (metal-bath). The product was extracted with chloroform and isolated by evaporation of the filtered solution (charcoal) as a light brown crystalline solid (1·1 g.) which by crystallisation from benzene or acetic acid gave 1 : 8-dimethylphenanthrene (V) (1 g., 55%) as colourless shining plates, m. p. 191—192° (Found : C, 92·9; H, 6·7. Calc. for $C_{16}H_{14}$: C, 93·2; H, 6·8%); it gave a picrate, m. p. 152° (lit., m. p. 151—152°), 1 : 8-dimethylphenanthraquinone, m. p. 189—190° (lit., m. p. 190°), and a quinoxaline derivative, m. p. 190—191° (sintering at 187°) (lit., m. p. 178°) (Found : C, 85·3; H, 4·9. Calc. for $C_{22}H_{16}N_2$: C, 85·7; H, 5·2%). The *trinitrobenzene* derivative, bright yellow needles from ethanol, had m. p. 173—174° (Found : C, 63·5; H, 3·7. $C_{16}H_{14},C_6H_3O_6N_3$ requires C, 63·0; H, 4·1%), and the *trinitrofluorenenone* derivative, stout orange needles from xylene–ethanol, had m. p. 193—194° (Found : C, 66·8; H, 3·9. $C_{16}H_{14},C_{13}H_5O_7N_3$ requires C, 66·8; H, 3·7%).

1-[2-(2 : 4-*Dimethylphenyl*)*ethyl*]-2 : 6-*dimethyl*cyclo*hexanol*.—2 : 6-Dimethyl*cyclo*hexanone (15 g., 1·2 mol.) and the Grignard reagent from 2-(2 : 4-dimethylphenyl)ethyl bromide (21·3 g.) (cf. Ruzicka, Ehmann, and Mörgeli, *Helv. Chim. Acta*, 1933, **16**, 314) gave, by the method previously described, the *alcohol* (13·0 g., 50%) as a colourless viscous oil, b. p. 156—157°/1 mm. (Found : C, 83·2; H, 10·6. $C_{18}H_{28}O$ requires C, 83·0; H, 10·8%).

1 : 2 : 3 : 4 : 9 : 10 : 11 : 12-*Octahydro*-1 : 6 : 8 : 12-*tetramethylphenanthrene*.—The above alcohol (12·4 g.), treated with sulphuric acid as previously described, gave the *hydrophenanthrene* (10 g., 86%) as a colourless oil, b. p. 130—140°/1 mm. After 3 days at room temperature, crystalline solid had separated, and the residue from this, when redistilled from sodium, had b. p. 130—135°/1 mm. (Found : C, 89·5; H, 10·7. $C_{18}H_{26}$ requires C, 89·2; H, 10·8%). The separated solid (0·5 g.) crystallised from ethanol in long rods, m. p. 70—71° (Found : C, 89·8; H, 10·0. $C_{20}H_{26}$ requires C, 90·2; H, 9·8%), presumably consisting of 1 : 4-*di*-(2 : 4-*dimethylphenyl*)-*butane*.

1 : 3 : 8-*Trimethylphenanthrene*.—The foregoing hydrophenanthrene (2 g.) was heated with selenium (4 g.) at 320—340° for 24 hr., and the product extracted with ether and distilled. The fraction of b. p. 120—160° (bath)/0·2 mm. crystallised from methanol, giving 1 : 3 : 8-*trimethylphenanthrene* (0·9 g., 50%) as colourless needles, m. p. 116° (Found : C, 92·3; H, 7·2. $C_{17}H_{16}$ requires C, 92·7; H, 7·3%); the *picrate*, orange needles from ethanol, had m. p. 174—175° (Found: C, 61·8; H, 4·2. $C_{17}H_{16},C_6H_3O_7N_3$ requires C, 61·5; H, 4·3%); the *trinitrobenzene* derivative, yellow needles from ethanol, had m. p. 188° (Found : C, 63·3; H, 4·3. $C_{17}H_{16},C_6H_3O_6N_3$ requires C, 63·7; H, 4·4%); and the *trinitrofluorenone* derivative, orange needles from xylene–ethanol, had m. p. 199° (Found : C, 67·6; H, 4·25. $C_{17}H_{16},C_{13}H_5O_7N_3$ requires C, 67·3; H, 4·0%). 1 : 3 : 8-*Trimethylphenanthraquinone* crystallised from ethanol or acetic acid in orange needles, m. p. 180° (Found : C, 81·3; H, 6·0. $C_{17}H_{14}O_2$ requires C, 81·6; H, 5·6%); the derived *quinoxaline*, pale yellow long needles from ethanol or acetic acid, had m. p. 146—147° (Found : C, 85·2; H, 5·2. $C_{23}H_{18}N_2$ requires C, 85·7; H, 5·6%).

2-o-*Ethylphenylethyl Bromide*.—2-*o*-Ethylphenylethyl alcohol (Kutz, Nickels, McGovern, and Corson, *J. Org. Chem.*, 1951, **16**, 699), b. p. 76—78°/0·1 mm. (lit., b. p. 127·5°/20 mm.) [α-*naphthylurethane*, colourless needles from light petroleum (b. p. 60—80°), m. p. 104° (Found : C, 78·6; H, 6·6. $C_{21}H_{21}O_2N$ requires C, 79·0; H, 6·6%)], was converted with phosphorus tribromide in the usual way into the *bromide* (65%), b. p. 68°/0·1 mm., n_D^{20} 1·5562 (Found : Br, 37·0. $C_{10}H_{13}Br$ requires Br, 37·5%).

1-(2-o-*Ethylphenylethyl*)-2 : 6-*dimethyl*cyclo*hexanol*.—2 : 6-Dimethyl*cyclo*hexanone (16 g., 1·2 mol.) and Grignard reagent from the above bromide (23 g.) gave the cyclo*hexanol* (14·4 g., 51%) as a colourless oil, b. p. 122—124°/0·05 mm. (Found : C, 83·3; H, 10·9. $C_{18}H_{28}O$ requires C, 83·0; H, 10·8%).

8-*Ethyl*-1 : 2 : 3 : 4 : 9 : 10 : 11 : 12-*octahydro*-1 : 12-*dimethylphenanthrene*.—The foregoing alcohol (13·8 g.) when treated with sulphuric acid in the usual way gave the *hydrophenanthrene* as a colourless oil (11·3 g., 88%), b. p. 106—110°/0·05 mm. (Found : C, 89·0; H, 10·6. $C_{18}H_{26}$ requires C, 89·2; H, 10·8%).

1-*Ethyl*-8-*methylphenanthrene*.—The above hydrophenanthrene (2 g.) was heated with selenium (4 g.) at 320—340° for 48 hr. Distillation of the product followed by crystallisation from methanol gave 1-*ethyl*-8-*methylphenanthrene* (1·0 g., 55%) as glistening plates, m. p. 106° (Found : C, 92·6; H, 7·5. $C_{17}H_{16}$ requires C, 92·7; H, 7·3%); the *picrate*, orange needles from ethanol, had m. p. 124—125° (Found: C, 61·0; H, 4·4. $C_{17}H_{16},C_6H_3O_7N_3$ requires C, 61·5; H, 4·3%); the *trinitrobenzene* derivative, yellow needles from ethanol, had m. p. 145—146° (Found: C, 63·6; H, 4·2. $C_{17}H_{16},C_6H_3O_6N_3$ requires C, 63·7; H, 4·4%); and the *trinitrofluorenone* derivative, orange needles from xylene–ethanol, had m. p. 158—159° (Found: C, 67·0; H, 3·8. $C_{17}H_{16},C_{13}H_5O_3N_3$ requires C, 67·3; H, 4·0%). 1-*Ethyl*-8-*methylphenanthraquinone*, orange plates from ethanol, had m. p. 154—155° (Found: C, 81·9; H, 5·9. $C_{17}H_{14}O_2$ requires C, 81·6; H, 5·6%); and the derived *quinoxaline*, pale yellow leaflets from acetic acid, or felted needles from ethanol, had m. p. 130° (Found: C, 85·8; H, 5·9. $C_{23}H_{18}N_2$ requires C, 85·7; H, 5·6%).

1-[2-(2 : 5-*Dimethylphenyl*)*ethyl*]-2 : 6-*dimethyl*cyclo*hexanol*.—2 : 6-Dimethyl*cyclo*hexanone (6 g., 1·7 mol.) and Grignard reagent from 2-(2 : 5-dimethylphenyl)ethyl bromide (9 g.) (Papa *et al., loc. cit.*), made from rigorously purified bromo-*p*-xylene, gave the cyclo*hexanol* as a colourless oil (5·9 g., 54%), b. p. 158—161°/1 mm. (Found: C, 83·5; H, 10·1. $C_{18}H_{28}O$ requires C, 83·0; H, 10·8%).

1 : 2 : 3 : 4 : 9 : 10 : 11 : 12-*Octahydro*-1 : 5 : 8 : 12-*tetramethylphenanthrene*.—Ring closure of the above alcohol (5·5 g.) with sulphuric acid gave the *hydrophenanthrene* as a colourless oil (4·2 g., 82%), b. p. 130—135°/1 mm. (Found: C, 88·9; H, 10·3. $C_{18}H_{26}$ requires C, 89·2; H, 10·8%).

1 : 4 : 8-*Trimethylphenanthrene*.—The relevant hydrophenanthrene (2 g.) was heated with sulphur (1·05 g.) at 220—240° for 3 hr. The product was distilled and the yellow oily fraction, b. p. 110—150° (bath)/0·05 mm., converted into the orange *trinitrobenzene* derivative which after four crystallisation from ethanol had m. p. 160—161° (Found: C, 63·9; H, 4·6. $C_{17}H_{16},C_6H_3O_6N_3$ requires C, 63·7; H, 4·4%), and when pure was of the usual bright yellow colour. The trinitrobenzene derivative was decomposed by passage of its solution in benzene down an alumina column followed by elution with light petroleum (b. p. 60—80°), the eluate when evaporated giving 1 : 4 : 8-*trimethylphenanthrene* (0·35 g., 19%). After three crystallisations from methanol the pure hydrocarbon formed colourless needles, m. p. 64° (Found: C, 92·8; H, 7·5. $C_{17}H_{16}$ requires C, 92·7; H, 7·3%); the *picrate*, orange needles from ethanol, had m. p. 136—137° (Found: C, 62·0; H, 4·5. $C_{17}H_{16},C_6H_3O_7N_3$ requires C, 61·5; H, 4·3%); and the *trinitrofluorenone* derivative, orange needles from ethanol, had m. p. 165—166° (Found: C, 67·0; H, 3·7. $C_{17}H_{16},C_{13}H_5O_7N_3$ requires C, 67·3; H, 4·0%).

When the dehydrogenation was carried out with selenium in the usual way the distillate deposited crystalline material (0·15 g.) which was identified as 1 : 8-dimethylphenanthrene. The mother-liquors from the crystallisation of the dimethyl compound were worked up through the trinitrobenzene derivative but, although a product of the correct m. p. was obtained, regeneration gave a hydrocarbon still appreciably contaminated with the dimethylphenanthrene, which, owing to its low solubility, could not be removed by crystallisation.

2-(3 : 5-*Dimethylphenyl*)*ethyl Bromide*.—2-(3 : 5-Dimethylphenyl)ethyl alcohol (cf. Matsui, *J. Soc. Chem. Ind. Japan*, 1942, **45**, 300), b. p. 130°/15 mm. (lit., b. p. 123—125°/9 mm.), with phosphorus tribromide in the usual way gave the *bromide* (50%), b. p. 75—78°/0·5 mm., n_D^{20} 1·5452 (Found: C, 56·4; H, 6·1. $C_{10}H_{13}Br$ requires C, 56·3; H, 6·1%).

1-[2-(3 : 5-*Dimethylphenyl*)*ethyl*]-2 : 6-*dimethyl*cyclo*hexanone*.—2 : 6-Dimethyl*cyclo*hexanone (10 g., 1·2 mol.) and Grignard reagent from the foregoing bromide (14·9 g.) gave the cyclo*hexanol* (4·8 g., 26%) as a colourless oil, b. p. 160—162°/1·5 mm. (Found: C, 83·4; H, 10·6. $C_{18}H_{28}O$ requires C, 83·0; H, 10·8%).

1 : 2 : 3 : 4 : 9 : 10 : 11 : 12-*Octahydro*-1 : 5 : 7 : 12-*tetramethylphenanthrene*.—Treatment of the requisite alcohol (4·6 g.) with sulphuric acid gave the *hydrophenanthrene* as a colourless oil (3·1 g., 72%), b. p. 100—102°/0·04 mm. (Found: C, 89·1; H, 10·4. $C_{18}H_{26}$ requires C, 89·2; H, 10·8%).

2 : 4 : 8-*Trimethylphenanthrene*.—The octahydrotetramethylphenanthrene (2 g.) was dehydrogenated with selenium (4 g.) at 320—340° for 24 hr. The product distilled as an uncrystallisable oil which was converted into the *trinitrobenzene* derivative; after three crystallisations from ethanol this separated as yellow needles, m. p. 150—151° (Found: C, 63·6; H, 4·2. $C_{17}H_{16},C_6H_3O_6N_3$ requires C, 63·7; H, 4·4%). The hydrocarbon was regenerated from this derivative on an alumina column in the usual way, and the 2 : 4 : 8-*trimethylphenanthrene* (0·7 g., 39%) so obtained separated from methanol in large prisms, m. p. 50° (Found: C, 92·2; H, 7·6. $C_{17}H_{16}$ requires C, 92·7; H, 7·3%); the *picrate*, orange needles from ethanol, had m. p. 135° (Found: C, 61·6; H, 4·2. $C_{17}H_{16},C_6H_3O_7N_3$ requires C, 61·5; H, 4·3%); and the *trinitrofluorenone* derivative, orange needles or slender red prisms from xylene–ethanol, had m. p. 178—179° (Found: C, 67·6; H, 4·0. $C_{17}H_{16},C_{13}H_5O_7N_3$ requires C, 67·3; H, 4·0%).

2-*Ethyl*-5-*methyl*-1-*tetralone*.—2-*o*-Tolylethyl bromide and the sodio-derivative of diethyl ethylmalonate, condensed in the usual way, gave diethyl α-ethyl-α-(2-*o*-tolylethyl)malonate (55%) as a colourless oil, b. p. 192—194°/11 mm., n_D^{17} 1·4917, which was converted by the usual methods into the corresponding monocarboxylic acid and thence into the tetralone (Harvey *et al.*, *loc. cit.*); the 2 : 4-*dinitrophenylhydrazone*, bright orange prisms from ethanol, had m. p. 172—173° (Found : C, 61·8; H, 5·1. $C_{19}H_{20}O_4N_4$ requires C, 61·9; H, 5·5%).

2-*Ethyl*-1 : 5-*dimethylnaphthalene*.—2-Ethyl-5-methyl-1-tetralone (5 g.) was treated in boiling ether for 6 hr. with excess of methylmagnesium iodide. The product (3·8 g.) boiled from 140° to 152°/12 mm., presumably owing to partial dehydration (cf. Ruzicka and Ehmann, *Helv. Chim. Acta*, 1932, **15**, 140), and without further treatment was dehydrogenated with selenium (3 g.) at 300—320° for 24 hr. The *naphthalene* (1·5 g., 30%) isolated by distillation, b. p. 135—137°/12 mm., n_D^{20} 1·6023 (Found : C, 91·9; H, 8·7. $C_{14}H_{16}$ requires C, 91·3; H, 8·7%), was purified by regeneration from the *trinitrobenzene* derivative, yellow leaflets (from ethanol), m. p. 112—113° (Found : C, 60·7; H, 4·5. $C_{14}H_{16},C_6H_3O_6N_3$ requires C, 60·5; H, 4·8%), but it failed to crystallise. The very soluble *picrate* crystallised in orange needles as the alcoholate from a small volume of ethanol (removal of the solvent entailing decomposition), and had m. p. 82—84° (Found : C, 57·1; H, 5·2. $C_{14}H_{16},C_6H_3O_7N_3,C_2H_6O$ requires C, 57·5; H, 5·5%). The *trinitrofluorenone* derivative, prepared in ethanol, separated as orange needles, m. p. 104—105°, decomposed by recrystallisation (Found : C, 64·7; H, 4·2. $C_{14}H_{16},C_{13}H_5O_7N_3$ requires C, 64·9; H, 4·2%).

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