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ACCESSORY MATERIAL

2-Phenyl-2-[2-(2-pyridyl)ethyl]4,5,6,7-tetrahydroindan-1,3-dione ***9***

2-Vinylpyridine (15 mL) was added to a solution of 2-phenyl-4,5,6,7-tetrahydroindan-1,3-dione (30g, 113mmol) in ethanol (200 mL) and the reaction mixture heated under reflux for 24h. The solvent was reduced in volume and allowed to cool. The solid which separated out was filtered off and recrystallised from ethanol to give *2-Phenyl-2-[2-(2-pyridyl)ethyl]4,5,6,7-tetrahydroindan-1,3-dione* **9** as yellow crystals (37g, 84%). m.p. 69.5-70^{o}C, ν_{max}($CHCl_3$) 1740, 1700, 1660 and 1600 cm^{-1}, δ($CDCl_3$) 8.4-8.6 (1H, m, aromatic), 6.9-7.7 (8H, m, aromatic) 2.2-2.8 (8H, m, CO((CH_2))$_4$CO), and 1.5-2.0 (4H, m, C(Ph)CH_2CH_2). (Found: C 79.5, H 6.3, N 4.2. $C_{22}H_{21}NO_2$ requires C 79.9, H 6.4, N 4.2%).

2-Phenyl-2-[2-(2-pyridyl)methyl]4,5,6,7-tetrahydroindan-1,3-dione ***58***

2-Phenyl-4,5,6,7-tetrahydroindan-1,3-dione (10g) was added to a solution of sodium (1.01g) in dried propanol (100 mL) whereupon the solution turned purple. Sodium iodide (7g) was added and the resulting mixture heated and stirred. 2-Picolyl chloride (5.64g) was added slowly over 10 minutes and the mixture then stirred and heated under reflux for 7 hours during which time the mixture darkened. The solvent was removed under reduced pressure to give a thick dark oil. Water was added, then chloroform, and the mixture thoroughly shaken. The chloroform layer was run off, and the procedure repeated twice more. The combined chloroform extracts were dried (Na_2SO_4) and the solvent removed under reduced pressure. The resulting dark oil was dissolved in a little ethanol and warmed with decolourising charcoal (5g). The mixture was filtered and the volume reduced before being cooled overnight during which time a dark solid was formed. Repeated recrystallisation from ethanol gave *2-phenyl-2-[2-(2-pyridyl)methyl]4,5,6,7-tetrahydroindan-1,3-dione* **58** as an off-yellow crystalline solid, (5.1g, 36%), m.p. 129-130^{o}C, ν_{max}(Nujol) 1760, 1710, 1625 and 750 cm^{-1}; δ($CDCl_3$) 7-8.3 (9H, ArH), 3.7 (2H, s, CH_2), 2.3 (4H, m, CCH_2) and 1.7 (4H, m, CCH_2CH_2); M/e 317 (M^+). (Found: C 79.5, H 6.1, N 4.4. $C_{21}H_{19}NO_2$ requires C 79.5, H 6.0,N 4.4%).

2-Phenyl-2-[2-(3-pyridyl)methyl]4,5,6,7-tetrahydroindan-1,3-dione ***60***

To a solution of sodium (1.26, 55 mmol) in dry methanol (100 mL) containing 2-phenyl-4,5,6,7-tetrahydroindan-1,3-dione (6.2g, 27 mmol) was added 3-picolylchloride hydrochloride (4.5 g, 27 mmol) portionwise over one hour. The resulting mixture was heated under reflux for a further hour and after this period the methanol was removed under reduced pressure to yield a thick oil. Water (100 mL) was added and the mixture extracted with ether. The combined extracts were dried (Na_2SO_4) and the ether removed under reduce pressure to yield a yellow oil. Chromatography of the oil over silica using diethyl ether/petroleum ether (b.p. 40-60^{o}C) 20:80 gave *2-phenyl-2-[2-(3-pyridyl)methyl]4,5,6,7-tetrahydroindan-1,3-dione* **60** (0.33g, 4%) as shining yellow plates, m.p. 129-130^{o}C, ν_{max}(liquid film) 1730, 1685, 1630, 1600 cm^{-1}; δ($CDCl_3$) 9.6 (1H, s, N^+H_a), 8.1 (1H, s, N^+H_b), 7.3 (5H, s, ArH), 3.5 (1H, d, N^+CH_{eq}), 2.9 (1H, m, $N^+CH(CH_2)_2$) and 2.8 (1H, m, N^+CH_{ax})CH_2). (Found: C, 68.4; H, 7.2; N, 3.7. $C_{21}H_{26}NO_2Cl$. 0.5 H_2O requires C, 68.4; H, 7.1, 3.7%).
A further fraction eluted with ether gave a white crystalline solid. One recrystallisation from diethyl ether/ petroleum ether (b.p. 40-60^{o}C) 20:80 gave *3-methoxy-hexahydro-2-phenyl-2-[2-(3-pyridyl)methyl]4,5,6,7-tetrahydroindan-1,3-dione* **14** as shining white needles (0.29 g, 3%), m.p. 98-99^{o}C, ν_{max}($CDCl_3$) 1760, 1600 cm^{-1}; δ($CDCl_3$) 7-8.4 (9H, m, ArH), 3.7 (1H, dd, COCH), 3.2 (3H, s, OMe), 3.1 (2H, AB quartet, C(Ph)CH_2(Py)) and 1.2-2.2 (8H, m, aliphatic), M/e 349 (M^+), 318 (M^+- OMe). (Found: C 76, H 6.6, N 3.9. $C_{22}H_{23}NO_3$ requires C 75.8, H 6.59, N 4.0%).

2-Phenyl-2-(2-(2-quinolyl)ethyl)indan-1,3-dione ***15***

2-Phenyl-indan-1,3-dione (10g, 45mmol) with a slight molar excess of 2-vinyl-quinoline (8.5g, 55mmol) in absolute ethanol (250 mL) was refluxed for 48h. The bright red solution was reduced in volume and left to stand whereupon a pink solid formed. The solid was filtered off and washed with cold methylated spirits. One recrystalisation from absolute ethanol gave *2-phenyl-2-(2-(2-quinolyl)ethyl)indan-1,3-dione* **15** as a slightly

pink crystalline solid (12.7g, 75%), m.p. 161-2°C , ν_{max}(Nujol)1740, 1700, 1600 and 750 cm^{-1}; δ($CDCl_3$) 7.2-8.1 (15H, ArH) and 2.7-3.0 (4H, m, CH_2CH_2), M/e 375 (M^+), (Found: C 82.7, H 5.1, N 3.7. $C_{26}H_{19}NO_2$ requires C 82.2, H 5.1, N 3.7%).

2-Phenyl-2-(2-phthalimidoethyl)-4,5,6,7-tetrahydroindan-1,3-dione ***19***

2-Phenyl-4,5,6,7-tetrahydroindan-1,3-dione (4.2g), *N*-(2-bromoethyl)phthalimide (50.8g) and sodium iodide (30g) were added to a solution of sodium (4.6g) in *n*-propanol (240ml), and the resulting reaction mixture stirred and heated under reflux for 16h. The cooled solution was poured into water and the propanol distilled off. Extraction with dichloromethane, followed by solvent evaporation, gave a yellow solid. Repeated recrystallisation from ethanol gave *2-phenyl-2-(2-phthalimidoethyl)-4,5,6,7-tetrahydroindan-1,3-dione* **19** as yellow crystals (30g, 38%), m.p. 197-198°C. (Found: C 75.1, H 5.4, N 3.5. $C_{25}H_{21}NO_4$ requires C 75.2, H 5.3, N 3.5%).

2-Phenyl-2-(3-phthalimidopropyl)-4,5,6,7-tetrahydroindan-1,3-dione ***21***

2-Phenyl-4,5,6,7-tetrahydroindan-1,3-dione (22.6g), *N*-(3-bromopropylyl)phthalimide (25.7g) and sodium iodide (13g) were added to a solution of sodium (2.3g) in *n*-propanol (100 mL), and the resulting reaction mixture stirred and heated under reflux for 9h, during which time the colour of the reaction mixture changed from deep purple to yellow. The cooled reaction mixture was poured into water, and the precipitate that formed, filtered off, dried, and recrystallised from ethanol to afford *2-phenyl-2-(3-phthalimidopropyl)-4,5,6,7-tetrahydroindan-1,3-dione* **21** as a crystalline yellow solid (30g, 75%), m.p. 154-155°C. ν_{max} ($CHCl_3$) 1780, 1729, 1690, 1640 and 1600 cm^{-1}, λ_{max} (EtOH) 218, 232 and 240 nm, δ($CDCl_3$) 7.7-8.0 (4H, m, ArH), 7.2-7.6 (5H, m, ArH), 3.6 (2H, t, $CH_2N(CH_2)_2$) and 1.4-2.6 (12H, m, aliphatic). (Found: C 75.7, H 5.7, N 3.4. $C_{26}H_{23}NO_4$ requires C 75.5, H 5.6, N 3.4%).

2-Phenyl-2-[2-(5-ethyl-2-pyridyl)ethyl]indan-1,3-dione ***24***

2-Phenyl-indan-1,3-dione (16.67g, 73mmol) was refluxed with 5-ethyl-2-vinylpyridine (10g) in absolute ethanol (400 mL) for 40h. The solution was concentrated in volume and left overnight to *yield 2-phenyl-2-[2-(5-ethyl-2-pyridyl)ethyl]indan-1,3-dione* **24** as white needles, (23.6g, 90%), m.p. 83-84°C, ν_{max}(Nujol) 1740, 1710, 1600 and 750 cm^{-1}; δ($CDCl_3$) 6.9-8.3 (12H, ArH), 2.7 (4H, s, CH_2CH_2), 2.6 (2H, q, CH_2CH_3) and 1.2 (3H, t, CH_2CH_3), M/e 355 (M^+), (Found: C, 81.2; H, 6.0; N, 3.9. $C_{24}H_{21}NO_2$ requires C, 81.1; H, 5.9; N, 3.9%).

Cis-, and Trans- 2-Phenyl-2-(2-(5-ethyl-2-pyridyl)ethyl)indan-1,3-diol ***25***

2-phenyl-2-[2-(5-ethyl-2-pyridyl)ethyl]indan-1,3-dione **24** (5g, 14mmol) was dissolved in dry methanol (50 mL). Sodium borohydride (0.53g, 14mmol) was added portion wise and the resulting mixture heated for one hour. Dilute hydrochloric acid was added until the mixture became acidic and the methanol removed under reduced pressure. Saturated sodium bicarbonate (10 mL) was then added and the mixture extracted with chloroform. The combined extracts were dried (Na_2SO_4) and the solvent removed under reduced pressure to give *cis*-, and *trans- 2-phenyl-2-(2-(5-ethyl-2-pyridyl)ethyl)indan-1,3-diol* **25** as a gum. ν_{max}(liquid film) 3500, 1600 and 750 cm^{-1}; δ($CDCl_3$) 6.9-8.4 (11H, m, ArH), 5.5-6.6 (1H, s, CH(OH)), 2.65 (4H, m, CH_2CH_2), 2.6 (2H, q, CH_2CH_3) and 1.2 (3H, t, CH_2CH_3).

2-Methyl-2-(2-(2-pyridyl)ethyl)indan-1,3-dione ***61***

2-Methyl-indan-1,3-dione (40g, 250mmol) and 2-vinyl-pyridine (31.5g, 300mmol) were dissolved in absolute ethanol (200 mL) and the resulting mixture heated under reflux for 24h. The solvent was reduced in volume and the residue left overnight in an ice box. The solid that was deposited was broken up and filtered off.

Recrystallisation from methylated spirits gave *2-methyl-2-(2-(2-pyridyl)ethyl)indan-1,3-dione* **61** as a white crystalline solid (45g, 68%), m.p. 53°C, $\nu_{max}(CHCl_3)$1740, 1710 and 1600 cm^{-1}; $\delta(CDCl_3)$ 7.8-8.2 (4H, m, $(CO)_2ArH$), 7-8.4 (3H, m, PyH), 2-2.8 (4H, m, CH_2CH_2) and 1.4 (3H, s, Me).

2-Phenyl-2-(2-(2-pyridyl)methyl)indan-1,3-dione ***62***

2-Phenyl-indan-1,3-dione (10g, 45mmol) followed by sodium iodide (6.75g) was added to a solution of sodium (1.1g) in dried *n*-propanol (100 mL) whilst thoroughly stirring. 2-Picolyl chloride (5.74g, 45 mmol) was then added and the resulting mixture stirred and heated under reflux for 7h during which time the mixture turned brick red. Water was then added, then chloroform, and the resulting mixture thoroughly shaken. The chloroform layer was run off and the procedure repeated twice more. The combined extracts were dried (Na_2SO_4) and the solvent removed under reduced pressure. The resulting red oil was dissolved in a little ethanol and heated with decolourising charcoal (7g), the mixture was filtered and the volume reduced in vacuo. The resulting mixture was left overnight during which time a solid formed. Repeated crystallisation from absolute ethanol gave *2-phenyl-2-(2-(2-pyridyl)methyl)indan-1,3-dione* **62** as colourless crystalline solid (7.8g, 56%), m.p. 153-154°C, ν_{max}(Nujol) 1740, 1705, 1600 and 750 cm^{-1}; $\delta(CDCl_3)$ 6.9-8.0 (13H, ArH), 3.9 (2H, s, CH_2), M/e 313 (M^+), (Found: C 80.5, H 4.9, N 4.4. $C_{21}H_{15}NO_2$ requires C 80.5, H 4.8, N 4.4%).

2-Phenyl-2-[2-(2-pyridyl)ethyl]phenalene-indan-1,3-dione ***35***

2-phenylphenalene-indan-1,3-dione (10g, 37 mmol) was heated under reflux with 2-vinylpyridine (4.2 mL) and Triton B in methanol (2 mL) in dry benzene (400 mL) for one week. The benzene layer was thoroughly washed with 5N sodium hydroxide solution and then with 2N hydrochloric acid. The aqueous layer was made basic with 5N sodium hydroxide and vigorously extracted with ethylacetate. The combined extracts were dried (Na_2SO_4) and the solvent removed under reduced pressure to give a slightly yellow solid. One recrystallisation from absolute ethanol gave *2-phenyl-2-[2-(2-pyridyl)ethyl]phenalene-indan-1,3-dione* **35** as colourless needles (2.8g, 25%), m.p. 172°C, ν_{max}(Nujol) 1690, 1660 and 1580 cm^{-1}; $\delta(CDCl_3)$ 7.0-8.5 (15H, m, ArH) and 2.9 (4H, s, CH_2CH_2). (Found: C 82.8, H 5.1, N 3.55. $C_{26}H_{19}NO_2$ requires C 82.7, H 5.1, N 3.7%).

6-Phenyl-5,7-diketo-6-[2-(2-pyridyl)ethyl]dibenzo[a,c]cycloheptane ***38***

6-Phenyl-5,7-diketodibenzo[a,c]cycloheptane (1.5g, 5.0 mmol) was added to dried benzene (50 mL). 2-Vinylpyridine (0.55 mL) and Triton B in methanol (0.3 mL) were added and the mixture refluxed for 7 days. The benzene layer was concentrated under reduced pressure to give a gum. The gum was dissolved in a little absolute ethanol and the solution left to stand yielding a white crystalline solid. Recrystallisation from absolute ethanol gave *6-phenyl-5,7-diketo-6-[2-(2-pyridyl)ethyl]dibenzo[a,c]cycloheptane* **38** as colourless needles (0.89g, 44%), m.p. 153°C, ν_{max}(Nujol) 1760, 1700 and 1600 cm^{-1}; $\delta(CDCl_3)$ 7.1-8.6 (17H, m, ArH), and 2.65-2.45 (4H, m, CH_2CH_2). (Found: C 83.45, H 5.3, N 3.6. $C_{28}H_{21}NO_2$ requires C 83.35, H 5.25, N 3.5%).

2-Methyl-2-(2-phthalimodoethyl)-indan-1,3-dione ***41***

A mixture of 2-methyl-indan-1,3-dione (20.7g), sodium iodide (19.5g) and *N*-(2-bromoethyl)phthalimide (33g) was stirred and heated under reflux in a solution of sodium (3g) in *n*-propanol (200 ml) for 24h. The cooled solution was poured into water, basified with dilute NaOH, to remove unreacted 2-methylindan-1,3-dione, and extracted with dichloromethane. Solvent evaporation gave a pale yellow solid. Recrystallisation from ethylacetate afforded *2-methyl-2-(2-phthalimodoethyl)-indan-1,3-dione* **41** (30g, 68%) as a crystalline solid, m.p. 132-134°C. ν_{max} ($CDCl_3$) 1780, 1720, 1690, 1640 and 1600 cm^{-1}, $\delta(CDCl_3)$ 7.9 (4H, s, ArH) 7.7 (4H, s, aromatic), 3.75 (2H, t, CH_2N), 2.3 (2H, t, CH_2CH_2) and 1.4 (3H, s, CH_3), λ_{max} (EtOH) 225 nm. (Found: C 72.0, H 4.5, N 4.2. $C_{20}H_{15}NO_4$ requires C 72.1, H 4.5, N 4.2%).

2-Methyl-2-(3-phthalimidopropyl)-indan-1,3-dione ***45***

2-Methyl-indan-1,3-dione (16g), *N*-(3-bromopropyl)phthalimide (25.7g) and sodium iodide (13g) were added to a solution of sodium (2.3g) in *n*-propanol (100 ml), and the resulting reaction mixture was stirred and heated under reflux for 9h, during which time a colour change from bright red to yellow was observed. The cooled reaction mixture was then poured into water, and the solid which separated out was filtered off and washed with ether. The ether insoluble solid was dried, and recrystallised from dichloromethane to give *2-methyl-1-oxo-3-(3-N-phthalimidopropoxy)indene* **40** as a white solid (20g, 40%), m.p. 184°C, ν_{max} ($CDCl_3$) 1770 and 1710 cm^{-1}, λ_{max} (MeOH) 223 and 240 nm, δ($CDCl_3$) 7.6-8.1 (4H, m, ArH), 6.95-7.6 (4H, m, aromatic), 4.6 (2H, t, OCH_2CH_2), 4.0 (2H, t, CH_2N), 2.3 (2H, q, $CH_2CH_2CH_2$) and 1.93 (3H, s, CH_3), (Found: C, 72.4; H, 4.6; N, 4.2. $C_{21}H_{17}NO_4$ requires C, 72.6; H, 4.9; N, 4.0%). The ethereal solution was basified with dilute sodium hydroxide solution to remove excess 2-methylindan-1,3-dione. Diethylether extraction followed by ether evaporation gave a pale yellow solid. Repeated recrystallisation of the solid with ether *afforded 2-methyl-2-(3-phthalimidopropyl)-indan-1,3-dione* **45** as a white crystalline solid (5g, 14%), ν_{max} (nujol) 1770, 1740 and 1705 cm^{-1}, δ($CDCl_3$) 7.6-8.1 (8H, m, ArH), 3.58 (2H, t, CH_2N), 1.4-2.0 (4H, m, $CH_2CH_2CH_2N$) and 1.27 (3H, s, CH_3), (Found: C 72.5, H 4.6, N 4.1. $C_{21}H_{17}NO_4$ requires C 72.6, H 4.9, N 4.0%).

2-Methyl-2-(4-phthalimidobutyl)-indan-1,3-dione ***53***

2-Methylindan-1,3-dione (11.5g), *N*-(4-bromobutyl)phthalimide (20g) and sodium iodide (10.6g) were added to a solution of sodium (1.7g) in *n*-propanol (200 ml), and the reaction mixture stirred and heated under reflux for 24h. The cooled reaction mixture was poured into water, concentrated, basified with dilute sodium hydroxide, to remove any excess 2-methylindan-1,3-dione, and extracted with dichloromethane. Solvent evaporation afforded a viscous red oil (10g). A portion, 4g, of the oil was chromatographed on a Woelm neutral alumina (grade III) column and eluted with diethylether to give *2-methyl-2-(4-phthalimidobutyl)-indan-1,3-dione* **53** as a white crystalline solid (2g), m.p. 79-79.5°C, ν_{max} ($CDCl_3$) 1770, 1740, 1705 and 1600 cm^{-1}, λ_{max} (MeOH) 220 and 242 nm, δ($CDCl_3$) 7.9 (4H, s, ArH), 7.7 (4H, s, aromatic), 3.5 (2H, t, $N\text{-}CH_2$), 1.0-2.2 (6H, m, $\text{-}CH_2\text{-}CH_2\text{-}CH_2\text{–}CH_2\text{-N}$) and 1.3 (3H, s, CH_3). (Found: C 72.9, H 5.4, N 4.01. $C_{22}H_{19}NO_4$ requires C 73.1, H 5.3, N 3.9%).