

General

Unless otherwise noted all reagents were obtained from commercial suppliers and used without further purification. Chromatography columns were prepared using Fisher Chemicals 60A 35–70 micron silica gel. Nuclear magnetic resonance spectra were recorded using Bruker DPX300 and DPX500 MHz spectrometers. Chemical shifts are reported in parts per million (δ) downfield relative to the internal reference tetramethylsilane. Unless otherwise specified NMR spectra were recorded in deuteriochloroform at room temperature. Abbreviations used: Ar = aromatic, d = doublet, dd = doublet of doublets, dt = doublet of triplets, m = multiplet, q = quartet, s = singlet, t = triplet. Mass spectra were recorded using a micromass ZMD 2000 spectrometer employing the electrospray (ES+) ionisation technique. Accurate molecular masses were obtained from Walters LCT, GCT or Bruker MicroTof spectrometers. Infra-red spectra were recorded using a Perkin-Elmer FT-IR spectrometer. IR spectra of liquids were recorded as thin films on sodium chloride plates. IR spectra of solids were recorded using dichloromethane as solvent on sodium chloride plates. Melting points are uncorrected.

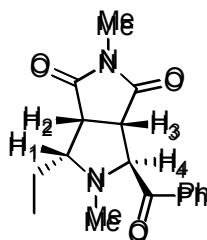
General procedure for the cycloaddition (A)

A mixture of a bromide (1 mmol) with N-methyl allyamine (1 mmol) or 3-pyrroline (1 mmol) and triethylamine (1 mmol) was dissolved in toluene (15mL), this was allowed to magnetically stirred at room temperature until alkylation was completed, as shown by TLC. To this mixture a dipolarophile (1 mmol) and Wilkinson's catalyst (0.1 mmol, 10mol %) were added and the mixture allowed to reflux in toluene (4 h-16 h) until completed, as shown by TLC. This mixture was then evaporated under vacuum before being washed with water (15ml) and extracted with ethyl acetate (3x15mL) to afford the crude product.. This was then purified via column chromatography to obtain the final product.

General Procedure for pyrrole formation (B)

As above however there is now no longer a dipolarophile added.

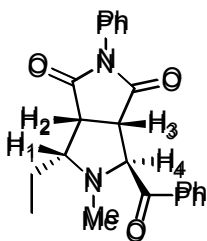
4-Benzoyl-6-ethyl-2,5-dimethyl-octahydropyrrolo[3,4-c]pyrrole-1,3-dione (3)



Prepared by general procedure A on a 1 mmol scale, crude mixture was purified using column chromatography, eluting 1:3 v/v ethyl acetate/hexane to give the product **3** (0.22g, 73% yield) as pale yellow prisms m.p. 120-122 °C. δ_{H} (500 MHz, CDCl_3); 8.14(d, 2H, J 7.3 Hz, ArH), 7.62 (t, 1H, J 7.3 Hz, ArH), 7.50 (t, 2H, J 7.3 Hz, ArH), 5.10 (s, 1H, H₄), 3.50 (m, 1H, H₁), 3.38 (t, 1H, J 7.8 Hz, H₂), 3.0 (d, 1H, J 7.8 Hz, H₃), 2.45 (s, 3H, N-CH₃), 1.84 (m, 1H, CHH), 1.48 (m, 1H, CHH), 1.13 (t, 3H, J 7.3 Hz, CH₃); δ_{C} (125 MHz, CDCl_3); 199, 178.3, 177.7, 135.6, 133.8, 129, 128.6, 68, 65.5, 47.4, 47.3, 35.2, 25.2, 22.2, 10.7; $\nu_{\text{max}}/\text{cm}^{-1}$ 2984, 2855, 1702, 1686; H.R.M.S. [ES+] found $M\text{Na}$ 323.137 1 $\text{C}_{17}\text{H}_{20}\text{N}_2\text{O}_3$ requires $M\text{Na}$ 323.1366

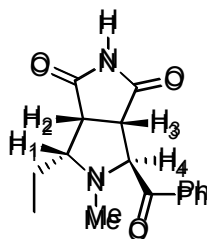
		n.O.e %						
Protons Irradiated		H ₁	H ₂	H ₃	H ₄	CHH	CHH	N-Me
	H ₁		8.23			3.16	1.24	2.33
	H ₂	6.47		5.24				
	H ₃		3.42		3.30			
	H ₄			3.78				2.72

4-Benzoyl-6-ethyl-5-methyl-2-phenyl-octahydropyrrolo[3,4-c]pyrrole-1,3-dione (4)



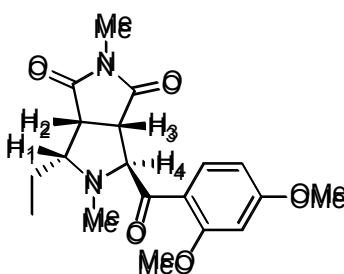
Prepared by general procedure A on a 1 mmol scale, crude mixture was purified using column chromatography, eluting 1:3 v/v ethyl acetate/hexane to give the product **4** (0.18g, 50% yield) as yellow solid; m.p. 160-162 °C; δ_{H} (500 MHz, CDCl₃); 8.16 (d, 2H, J 7.3 Hz, ArH), 7.62 (t, 1H, J 7.3 Hz, ArH), 7.52 (t, 2H, J 7.3 Hz, ArH), 7.48 (t, 2H, J 7.2 Hz, ArH), 7.39 (t, 1H, J 7.3 Hz, ArH), 7.32 (d, 2H, J 7.3 Hz, ArH), 5.23 (s, 1H, H₄), 3.59 (m, 1H, H₁), 3.53 (t, 1H, J 7.8 Hz, H₂), 3.47 (d, 1H, J 7.8 Hz, H₃), 2.45 (s, 3H, N-CH₃), 1.84 (m, 1H, CHH), 1.48 (m, 1H, CHH), 1.13 (t, 3H, J 7.3 Hz, CH₃); δ_{C} (125 MHz, CDCl₃); 199.1, 177.5, 175.8, 135.6, 135.5, 132, 130, 129, 128.9, 128.8, 126.4, 68.6, 65.9, 47.6, 47.4, 35.4, 22.3, 10.7; ν_{max} /cm⁻¹ 2942, 2855, 1713, 1688; H.R.M.S. [ES⁺] found MH⁺ 363.1715 C₂₂H₂₃N₂O₃ requires MH 363.1703.

4-Benzoyl-6-ethyl-5-methyl-octahydropyrrolo[3,4-c]pyrrole-1,3-dione (5)



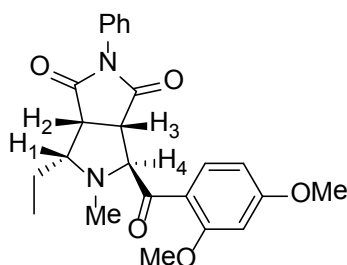
Prepared by general procedure A on a 1 mmol scale, crude mixture was purified using column chromatography, eluting 1:3 v/v ethyl acetate/hexane to give the product **5** (0.156g, 55% yield) as thick yellow oil; δ_{H} (500 MHz, CDCl_3); 8.54 (s, 1H, NH), 8.12 (d, 2H, J 7.3 Hz, ArH), 7.62 (t, 1H, J 7.3 Hz, ArH), 7.53 (t, 2H, J 7.3 Hz, ArH), 5.14 (s, 1H, H_4), 3.51 (m, 1H, H_1), 3.41 (t, 1H, J 7.8 Hz, H_2), 3.35 (d, 1H, J 7.8 Hz, H_3), 2.43 (s, 3H, N- CH_3), 1.80 (m, 1H, CHH), 1.44 (m, 1H, CHH), 1.12 (t, 3H, J 6.9 Hz, CH_3); δ_{C} (125 MHz, CDCl_3); 199.1, 178.4, 176.7, 136.5, 133.8, 129.1, 128.8, 68, 65.7, 48.6, 48.5, 3.25, 22.2, 10.8; $\nu_{\text{max}}/\text{cm}^{-1}$ 2926, 2855, 1726, 1597; H.R.M.S. [ES⁺] found MH^+ 287.1392 $\text{C}_{16}\text{H}_{19}\text{N}_2\text{O}_3$ requires MH 287.1390.

4-[(2,4-Dimethoxyphenyl)carbonyl]-6-ethyl-2,5-dimethyl-octahydropyrrolo[3,4-c]pyrrole-1,3-dione (6)



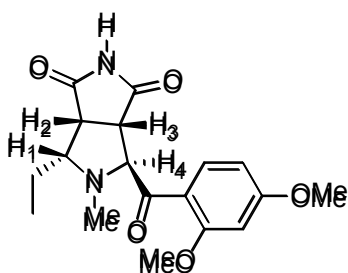
Prepared by general procedure A on a 1 mmol scale, crude mixture was purified using column chromatography, eluting 3:7 v/v ethyl acetate/hexane to give the product **6** (0.172g, 50% yield) as yellow prisms; m.p. 106-107 °C; δ_{H} (500 MHz, CDCl_3); 7.65 (d, 1H, J 8.7 Hz, ArH), 6.48 (d, 1H, J 8.7 Hz, ArH), 6.41 (s, 1H, ArH), 5.08 (s, 1H, H_4), 3.93 (s, 3H, O- CH_3), 3.80 (s, 3H, O- CH_3), 3.45 (m, 1H, H_1), 3.31 (t, 1H, J 8.3 Hz, H_2), 3.17 (d, 1H, J 8.3, H_3), 2.92 (s, 3H, N- CH_3), 2.26 (s, 3H, N- CH_3), 1.66 (m, 1H, CHH), 1.23 (m, 1H, CHH), 0.99 (t, 3H, J 7.3 Hz, CH_3); δ_{C} (125 MHz, CDCl_3); 200.9, 178.3, 177.2, 165.1, 161, 133.1, 121, 105.8, 98.3, 71.4, 65.9, 55.6, 55.5, 47.8, 47.5, 35.1, 24.9, 22.3, 10.6; $\nu_{\text{max}}/\text{cm}^{-1}$ 2970, 2943, 1702, 1660, 1599; H.R.M.S. [ES⁺] found MH^+ 361.1773 $\text{C}_{19}\text{H}_{25}\text{N}_2\text{O}_5$ requires MH 361.1758.

4-[(2,4-Dimethoxyphenyl)carbonyl]-6-ethyl-5-methyl-2-phenyl-octahydropyrrolo[3,4-c]pyrrole-1,3-dione (7) (7)



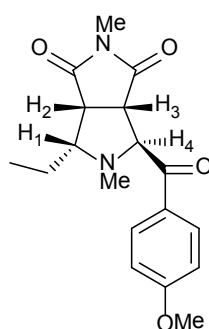
Prepared by general procedure A on a 1 mmol scale, crude mixture was purified using column chromatography, eluting 2:3 v/v ethyl acetate/hexane to give the product **7** (0.251g, 60% yield) as yellow solid; m.p. 126-127 °C; δ_{H} (500 MHz, CDCl_3); 7.76 (d, 1H, J 8.7 Hz, ArH), 7.47 (t, 2H, J 7.8 Hz, ArH), 7.38 (t, 1H, J 7.8 Hz, ArH), 7.31 (d, 2H, J 7.8 Hz, ArH), 6.57 (dd, 1H, J 8.7, 2.3 Hz, ArH), 6.48 (d, 1H, J 2.3 Hz, ArH), 5.25 (s, 1H, H_4), 3.99 (s, 3H, O- CH_3), 3.87 (s, 3H, O- CH_3), 3.64 (td, 1H, J 8.3, 2.8 Hz, H_1), 3.54 (t, 1H, J 8.3, H_2), 3.43 (d, 1H, J 8.3, H_3), 2.39 (s, 3H, N- CH_3), 1.80 (m, 1H, CHH), 1.39 (m, 1H, CHH), 1.08 (t, 3H, J 7.3, CH_3); δ_{C} (125 MHz, CDCl_3); 200.8, 177.4, 176.3, 165.2, 161.1, 132.6, 132.3, 129.1, 128.5, 126.6, 120.9, 105.8, 98.3, 72, 66.2, 55.6, 55.5, 47.9, 47.7, 35.3, 22.5, 10.5; $\nu_{\text{max}}/\text{cm}^{-1}$ 2944, 2841, 1713, 1665, 1599; H.R.M.S. [ES⁺] found MH^+ 423.1926 $\text{C}_{24}\text{H}_{27}\text{N}_2\text{O}_5$ requires MH 423.1914.

4-[(2,4-Dimethoxyphenyl)carbonyl]-6-ethyl-5-methyl-octahydropyrrolo[3,4-c]pyrrole-1,3-dione (8)



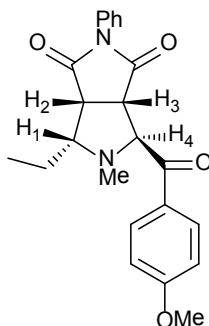
Prepared by general procedure A on a 1 mmol scale, crude mixture was purified using column chromatography, eluting 3:7 v/v ethyl acetate/hexane to give the product **8** (0.181g, 52% yield) as yellow solid; m.p. 168-169 °C; δ_{H} (500 MHz, CDCl_3); 8.36 (s, 1H, N-H), 7.65 (d, 1H, J 8.7 Hz, ArH), 6.48 (d, 1H, J 8.7 Hz, ArH), 6.41 (s, 1H, ArH), 5.09 (s, 1H, H_4), 3.91 (s, 3H, O- CH_3), 3.80 (s, 3H, O- CH_3), 3.45 (m, 1H, H_1), 3.34 (t, 1H, J 8.3 Hz, H_2), 3.24 (d, 1H, J 8.3 Hz, H_3), 2.30 (s, 3H, NCH_3), 1.66 (m, 1H, CHH), 1.28 (m, 1H, CHH), 1.01 (t, 3H, J 7.3 Hz, CH_3); δ_{C} (125 MHz, CDCl_3); 200.7, 178.4, 177.2, 16.2, 161, 132.6, 120.9, 105.8, 98.3, 7.4, 66, 55.6, 55.5, 49.6, 48.9, 35.1, 22.4, 110.7; $\nu_{\text{max}}/\text{cm}^{-1}$ 2943, 2843, 1724, 1660; H.R.M.S. [ES⁺] found MH^+ 347.2 $\text{C}_{18}\text{H}_{23}\text{N}_2\text{O}_5$ requires MH 347.2

4-Ethyl-6-[(4-methoxyphenyl)carbonyl]-2,5-dimethyl-octahydropyrrolo[3,4-c]pyrrole-1,3-dione (**9**)



Prepared by general procedure A on a 1 mmol scale, crude mixture was purified using column chromatography, eluting 3:7 v/v ethyl acetate/hexane to give the product **9** (0.166g, 50% yield) as a yellow solid; m.p. 97.3-99.1 °C; δ_{H} (500 MHz, CDCl_3); 8.12 (d, 2H, J 8.5 Hz, ArH), 6.99 (d, 2H, J 8.5 Hz, ArH), 5.06 (s, 1H, H_4), 3.89 (s, 3H, O- CH_3), 3.51 (m, 1H, H_1), 3.38 (t, 1H, J 7.8 Hz, H_2), 3.26 (d, 1H, J 7.8 Hz, H_3), 3.01 (s, 3H, N- CH_3), 2.38 (s, 3H, N- CH_3), 1.76 (m, 1H, CHH), 1.36 (m, 1H, CHH), 1.11 (t, 3H, J 7.3 Hz, CH_3); δ_{C} (125 MHz, CDCl_3); 198.6, 178.5, 176.8, 164, 130.9, 128.7, 114.1, 67.3, 66.5, 47.5, 47.4, 35.2, 34.8, 25.1, 22.2, 10.7; $\nu_{\text{max}}/\text{cm}^{-1}$; 2685, 1703, 1600; H.R.M.S. [ES⁺] found MH^+ 331.1658 $\text{C}_{18}\text{H}_{23}\text{N}_2\text{O}_4$ requires MH 331.1652.

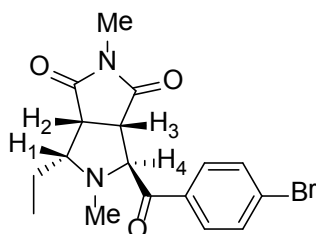
4-Ethyl-6-[(4-methoxyphenyl)carbonyl]-5-methyl-2-phenyl-octahydropyrrolo[3,4-c]pyrrole-1,3-dione (**10**)



Prepared by general procedure A on a 1 mmol scale, crude mixture was purified using column chromatography, eluting 1:1 v/v ethyl acetate/hexane to give the product **10** (0.31g, 80% yield) as yellow oil; δ_{H} (500 MHz, CDCl_3); 8.15 (d, 2H, J 8.7 Hz, ArH), 7.48 (t, 2H, J 7.6 Hz, ArH), 7.40 (t, 1H, J 7.3 Hz, ArH), 7.32 (d, 2H, J 7.8 Hz, ArH), 6.99 (d, 2H, J 8.7 Hz, ArH), 5.18 (s, 1H, H_4), 3.89 (s, 3H, O- CH_3), 3.59 (m, 1H, H_1), 3.54 (t, 1H, J 7.8 Hz, H_2), 3.45 (d, 1H, J 7.8 Hz, H_3), 2.44 (s, 3H, N- CH_3), 1.82 (m, 1H, CHH), 1.47 (m, 1H, CHH), 1.12 (t, 3H, J 7.3 Hz, CH_3); δ_{C} (125 MHz, CDCl_3); 198, 177.6, 175.9,

164.1, 131, 129.1, 128.8, 128.6, 126.4, 114.2, 68.2, 65.9, 55.6, 47.8, 47.5, 35.4, 22.3, 10.7 ; $\nu_{\max}/\text{cm}^{-1}$; 2882, 1714, 1600, ;
 H.R.M.S. [ES⁺] found MH^+ 393.1819 $\text{C}_{23}\text{H}_{25}\text{N}_2\text{O}_4$ requires MH 393.11809.

4-[(4-Bromophenyl)carbonyl]-6-ethyl-2,5-dimethyl-octahydropyrrolo[3,4-c]pyrrole-1,3-dione (11)

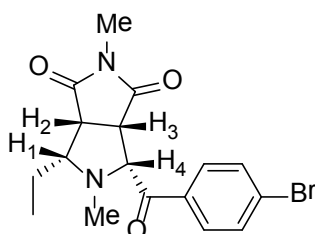


Prepared by general procedure A on a 1 mmol scale, crude mixture was purified using column chromatography, eluting 2:3 v/v ethyl acetate/hexane to give the product **11** (0.182g, 48% yield) as yellow gum; δ_{H} (500 MHz, CDCl_3); 8.01 (d, 2H, J 8.7 Hz, ArH), 7.67 (d, 2H, J 8.7 Hz, ArH), 5.02 (s, 1H, H_4), 3.48 (m, 1H, H_1), 3.36 (t, 1H, J 8.3 Hz, H_2), 3.26 (d, 1H, J 8.3 Hz, H_3), 3.02 (s, 3H, N- CH_3), 2.39 (s, 3H, N- CH_3), 1.77 (m, 1H, CHH), 1.38 (m, 1H, CHH), 1.11 (t, 3H, J 7.3 Hz, CH_3); δ_{C} (125 MHz, CDCl_3); 198, 178.2, 176.5, 134.2, 131.1, 130.6, , 129.5, 68.1, 6.5.5, 48, 35.1, 25.4, 22.2, 10.7 ; $\nu_{\max}/\text{cm}^{-1}$ 2880, 1703, 1656, 1585; H.R.M.S. [ES⁺] found MH^+ 379.0656 $\text{C}_{17}\text{H}_{20}\text{BrN}_2\text{O}_3$ requires MH 379.0651.(Br⁷⁹)

		n.O.e %						
Protons Irradiated		H_1	H_2	H_3	H_4	CHH	CHH	N-Me
	H_1		7.65			3.72	1.25	2.51
	H_2	5.77		7.85				
	H_3		6.49		2.89			
	H_4			4.60				2.68

and

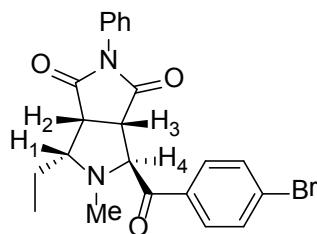
4-[(4-Bromophenyl)carbonyl]-6-ethyl-2,5-dimethyl-octahydropyrrolo[3,4-c]pyrrole-1,3-dione (12)



Product **12** (0.061g, 18% yield) as a yellow gum; δ_{H} (500 MHz, CDCl_3); 7.91 (d, 2H, J 8.3 Hz, ArH), 7.64 (d, 2H, J 8.3 Hz, ArH), 3.92 (d, 1H, J 7.8 Hz, H_4), 3.59 (t, 1H, J 7.8, H_1), 3.35 (t, 1H, J 7.8 Hz, H_2), 2.89 (s, 3H, N- CH_3), 2.42 (m, 1H, H_3), 2.27 (s, 3H, N- CH_3), 1.84 (m, 1H, CHH), 1.70 (m, 1H, CHH), 1.20 (t, 3H, J 7.3 Hz, CH_3); δ_{C} (125 MHz, CDCl_3); 195, 175.3, 175.2, 135.8, 13.4, 131, 130.1, 129.5, 71, 47.2, 45.9, 39.2, 25.1, 22.2, 10.5; $\nu_{\text{max}}/\text{cm}^{-1}$ 2880, 1776, 1703, 1656; H.R.M.S. [ES⁺] found MH^+ 379.0654 $\text{C}_{17}\text{H}_{20}\text{BrN}_2\text{O}_3$ requires MH 379.0651 (Br^{79})

		n.O.e %							
Protons Irradiated		H_1	H_2	H_3	H_4	CHH	CHH	N-Me	
	H_1		9.12		5.66				
	H_2	7.51		5.23					
	H_3	0	7.00		7.51				
	H_4	7.01		7.83				3.31	

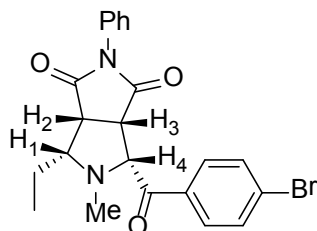
4-[(4-Bromophenyl)carbonyl]-6-ethyl-5-methyl-2-phenyl-octahydropyrrolo[3,4-c]pyrrole-1,3-dione (13)



Prepared by general procedure **A** on a 1 mmol scale, crude mixture was purified using column chromatography, eluting 2:3 v/v ethyl acetate/hexane to give the product **13** (0.28g, 64% yield) as a thick yellow gum; δ_{H} (500 MHz, CDCl_3); 8.03 (d, 2H, J 8.7 Hz, ArH), 7.67 (d, 2H, J 8.7 Hz, ArH), 7.49 (t, 2H, J 7.8 Hz, ArH), 7.41 (t, 1H, J 7.8 Hz, ArH), 7.31 (d, 2H, J 7.8 Hz, ArH), 5.15 (s, 1H, H_4), 3.55 (m, 1H, H_1), 3.52 (t, 1H, J 8.3 Hz, H_2), 3.45 (d, 1H, J 8.3 Hz, H_3), 2.45 (s, 3H, N- CH_3), 1.84 (m, 1H, CHH), 1.50 (m, 1H, CHH), 1.20 (t, 3H, J 7.3 Hz, CH_3); δ_{C} (125 MHz, CDCl_3); 198, 177.4, 175.6, 134.2, 132.4, 132.3, 130.1, 129.2, 129.1, 126.6, 126.4, 68.7, 65.8, 47.4, 47.3, 35.3, 22.2, 10.7; $\nu_{\text{max}}/\text{cm}^{-1}$ 2880, 2798, 1704; H.R.M.S. [ES⁺] found MH^+ 441.0811 $\text{C}_{22}\text{H}_{22}\text{BrN}_2\text{O}_3$ requires MH 441.0808 (Br^{79})

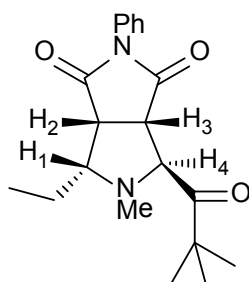
and

4-[(4-Bromophenyl)carbonyl]-6-ethyl-5-methyl-2-phenyl-octahydropyrrolo[3,4-c]pyrrole-1,3-dione (14)



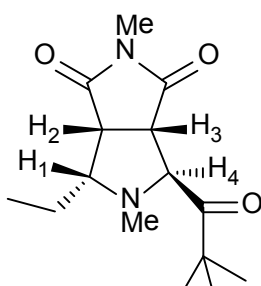
Prepared by general procedure **A** on a 1 mmol scale, crude mixture was purified using column chromatography, eluting 2:3 v/v ethyl acetate/hexane to give the product **14** (0.062g, 14% yield) as a yellow gum; δ_{H} (500 MHz, CDCl_3); 7.93 (d, 2H, J 8.7 Hz, ArH), 7.62 (d, 2H, J 8.7 Hz, ArH), 7.40 (t, 2H, J 7.8 Hz, ArH), 7.33 (t, 1H, J 7.8, ArH), 7.21 (d, 2H, J 7.8 Hz, ArH), 4.02 (d, 1H, J 8.3 Hz H_4), 3.72 (t, 1H, J 8.3 Hz, H_3), 3.50 (t, 1H, J 8.3 Hz, H_2), 2.53 (m, 1H, H_1), 2.31 (s, 3H, N- CH_3), 1.90 (m, 1H, CHH), 1.77 (m, 1H, CHH), 1.21 (t, 3H, J 7.3 Hz, CH_3); δ_{C} (125 MHz, CDCl_3); 198.1, 175.4, 175.6, 134.2, 132.4, 132, 130.1, 129.3, 129.1, 126.7, 126.4, 68.7, 65.8, 47.4, 47.3, 45.1, 39, 35.3, 22.2, 10.7; $\nu_{\text{max}}/\text{cm}^{-1}$ 2977, 1716, 1681; H.R.M.S. [ES+] found MH^+ 441.0800 $\text{C}_{22}\text{H}_{22}\text{BrN}_2\text{O}_3$ requires MH 441.0808 (Br^{79})

4-(2,2-Dimethylpropanoyl)-6-ethyl-5-methyl-2-phenyl-octahydropyrrolo[3,4-c]pyrrole-1,3-dione (16)



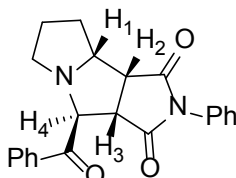
Prepared by general procedure **A** on a 1 mmol scale, crude mixture was purified using column chromatography, eluting 1:1 v/v ethyl acetate/hexane to give the product **15** (0.15g, 45% yield) as thick yellow oil; δ_{H} (500 MHz, CDCl_3); 7.46 (t, 2H, J 7.6 Hz, ArH), 7.38 (t, 1H, J 7.3 Hz, ArH), 7.28 (d, 2H, J 7.6 Hz, ArH), 4.71 (s, 1H, H_4), 3.78 (m, 1H, H_1), 3.50 (t, 1H, J 8.3 Hz, H_2), 3.19 (d, 1H, J 8.3 Hz, H_3), 2.32 (s, 3H, N- CH_3), 1.77 (m, 1H, CHH), 1.43 (m, 1H, CHH), 1.26 (s, 9H, C-(CH_3) $_3$), 1.08 (t, 3H, J 7.3 Hz, CH_3); δ_{C} (125 MHz, CDCl_3); 217, 176.9, 175.8, 131.9, 129.1, 128.6, 126.4, 67.3, 66.4, 48, 47.4, 45.1, 35.3, 26, 22.3, 10.6; $\nu_{\text{max}}/\text{cm}^{-1}$; 2685, 1714, 1598, ; H.R.M.S. [ES+] found MH^+ 343.2026 $\text{C}_{20}\text{H}_{26}\text{N}_2\text{O}_3$ requires MH 343.2016

4-(2,2-Dimethylpropanoyl)-6-ethyl-2,5-dimethyl-octahydropyrrolo[3,4-c]pyrrole-1,3-dione (15)



Prepared by general procedure **A** on a 1mmol scale, crude mixture was purified using column chromatography, eluting 3:7 v/v ethyl acetate/hexane to give the product **16** (0.11g, 40% yield) as a yellow solid; m.p. 108.5-111.3°C; δ_{H} (500 MHz, CDCl_3); 4.60 (s, 1H, H_4), 3.71 (m, 1H, H_1), 3.35 (t, 1H, J 8.0Hz, H_2), 2.99 (d, 1H, J 8.0Hz, H_3), 2.98 (s, 3H, N- CH_3), 2.26 (s, 3H, N- CH_3), 1.71 (m, 1H, CHH), 1.31 (m, 1H, CHH), 1.24 (s, 9H, C-(CH_3)₃), 1.06 (t, 3H, J 7.3Hz, CH_3); δ_{C} (125 MHz, CDCl_3); 217, 177.8, 176.7, 67.7, 66, 47.8, 47.3, 35, 25.9, 25.1, 22.3, 10.7; $\nu_{\text{max}}/\text{cm}^{-1}$; 2876, 2865 1702, 1608; H.R.M.S. [ES⁺] found MH^+ 281.1866 $\text{C}_{15}\text{H}_{25}\text{N}_2\text{O}_3$ requires MH 281.1859.

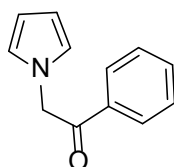
4-Benzoyl-2-phenyl-decahydropyrrolo[3,4-a]pyrrolizine-1,3-dione (17)



Prepared by general procedure **A** on a 1mmol scale, crude mixture was purified using column chromatography, eluting 3:7 v/v ethyl acetate/hexane to give the product **17** (0.191g, 53% yield) as a pale yellow solid; m.p. 109-110°C; δ_{H} (500 MHz, CDCl_3); 8.11 (d, 2H, J 7.3 Hz, ArH), 7.60 (t, 1H, J 7.3 Hz, ArH), 7.51 (dt, 4H, J 7.3, 1.8 Hz, ArH), 7.42 (t, 1H, J 7.3, ArH), 7.28 (d, 2H, J 7.3 Hz, ArH), 5.28 (s, 1H, H_4), 4.12 (d, 1H, J 7.3 Hz, H_3), 3.92 (m, 1H, H_1), 3.55 (t, 1H, J 7.3 Hz, H_2), 3.46 (m, 1H, NCHH), 2.75 (m, 1H, NCHH), 2.36 (m, 1H, CHH), 2.12 (m, 1H, CHH), 1.92 (m, 1H, CHH), 1.80 (m, 1H, CHH); δ_{C} (125 MHz, CDCl_3); 195, 178.6, 176.8, 134.7, 133.5, 131.9, 129.3, 129.2, 128.8, 128.7, 126, 71.3, 67.1, 54.3, 49.7, 49.5, 25.8, 24.7; $\nu_{\text{max}}/\text{cm}^{-1}$ 2984, 2902, 1713, 1645; H.R.M.S. [ES⁺] found MH^+ 361.1561 $\text{C}_{22}\text{H}_{21}\text{N}_2\text{O}_3$ requires MH 361.1546.

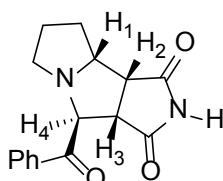
and

(1-phenyl-2-(1H-pyrrol-1-yl)ethan-1-one) (18)



Product **18** (0.048g, 26% yield) as a brown solid; m.p. 68-69°C; δ_{H} (500 MHz, CDCl_3); 7.96 (d, 2H, J 7.3 Hz, ArH), 7.63 (t, 1H, J 7.3 Hz, ArH), 7.51 (t, 2H, J 7.3 Hz, ArH), 6.68 (s, 2H, PyrroleH), 6.25 (s, 2H, PyrroleH), 5.32 (s, 2H, CH_2); δ_{C} (125 MHz, CDCl_3); 194, 134.7, 133.9, 128.9, 128.6, 121, 110, 55.4 $\nu_{\text{max}}/\text{cm}^{-1}$ 2965, 1705, 1597,; H.R.M.S. [ES⁺] found MH^+ 186.0914 $\text{C}_{12}\text{H}_{12}\text{NO}$ requires MH 186.09134.

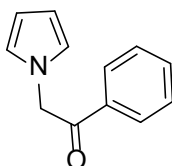
4-Benzoyl-decahydropyrrolo[3,4-a]pyrrolizine-1,3-dione(19)



Prepared by general procedure A on a 1 mmol scale. Crude mixture was purified using column chromatography, eluting with 2:3 v/v ethyl-acetate/hexane to give the product **19** (0.127 g, 45%) as pale yellow plates, m.p. 209-210 °C; δ_{H} (500 MHz, CDCl_3); 8.07 (d, 2H, J 7.3 Hz, ArH), 7.68 (s, 1H, N-H), 7.60 (t, 1H, J 7.3 Hz, ArH), 7.50 (t, 2H, J 7.3 Hz, ArH), 5.17 (s, 1H, H_4), 3.98 (d, 1H, J 8.7 Hz, H_3), 3.82 (m, 1H, H_1), 3.42 (t, 1H, J 8.7, H_2), 3.40 (m, 1H, NCHH), 2.75 (m, 1H, NCHH), 2.28 (m, 1H, CHH), 2.10 (m, 1H, CHH), 1.90 (m, 1H, CHH), 1.83 (m, 1H, CHH); δ_{C} (125 MHz, CDCl_3); 195, 179, 178.1, 134.6, 133.5, 129.1, 128.7, 70.5, 67.8, 54.2, 51, 50.6, 25.9, 24.8; $\nu_{\text{max}}/\text{cm}^{-1}$ 3154, 2901, 2974, 1714, 1648; H.R.M.S. [ES⁺] found MH^+ 285.1238. $\text{C}_{16}\text{H}_{17}\text{N}_2\text{O}_3$ requires MH 285.1233.

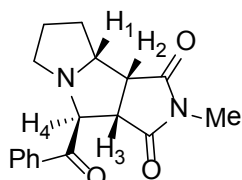
and

(1-Phenyl-2-(1H-pyrrol-1-yl)ethan-1-one) (18)



Product **18** (0.079g, 43% yield) as a brown solid; m.p. 68-69°C

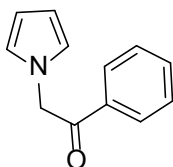
4-Benzoyl-2-methyl-decahydropyrrolo[3,4-a]pyrrolizine-1,3-dione (20)



Prepared by general procedure A on a 1 mmol scale, Crude mixture was purified using column chromatography, eluting with 2:3 v/v ethyl-acetate/hexane to give the product **20** (0.12 g, 40%) as a yellow gum; δ_{H} (500 MHz, CDCl_3); 8.08 (d, 2H, J 7.3 Hz, ArH), 7.59 (t, 1H, J 7.3 Hz, ArH), 7.50 (t, 2H, J 7.3 Hz, ArH), 5.15 (s, 1H, H_4), 3.92 (d, 1H, J 8.3 Hz, H_3), 3.81 (m, 1H, H_1), 3.38-3.34 (m, 2H, H_2 , NCHH), 2.99 (s, 3H, N- CH_3), 2.54 (m, 1H, NCHH), 2.25 (m, 1H, CHH), 2.05 (m, 1H, CHH), 1.84 (m, 1H, CHH), 1.60 (m, 1H, CHH); δ_{C} (125 MHz, CDCl_3); 195, 179.5, 177.9, 134.7, 133.5, 129.2, 128.7 01.7, 66.6, 54.2, 49.6, 49.2, 25.8, 25.2, 24.6; $\nu_{\text{max}}/\text{cm}^{-1}$ 2984, 1698, 1599; H.R.M.S. [ES⁺] found MH^+ 299.1393. $\text{C}_{17}\text{H}_{19}\text{N}_2\text{O}_3$ requires MH 299.1390.

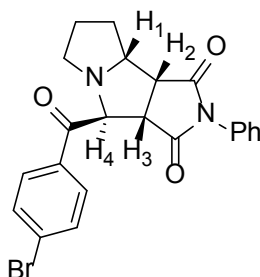
and

(1-Phenyl-2-(1H-pyrrol-1-yl)ethan-1-one) (18)



Product **18** (0.08g, 45% yield) as a brown solid; m.p. 68-69 °C

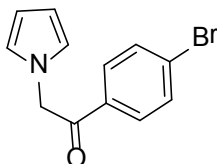
7-[(4-Bromophenyl)carbonyl]-4-phenyl-4,7-diazatricyclo[6.3.0.0^{2,6}]undecane-3,5-dione (21)



Prepared by general procedure **A** on a 1 mmol scale, Crude mixture was purified using column chromatography, eluting with 2:3 v/v ethyl-acetate/hexane to give the product **21** (0.14 g, 32%) as a pale yellow solid; m.p. 168-169°C ; δ_{H} (500 MHz, CDCl_3); 7.96 (d, 2H, J 8.7 Hz, ArH), 7.65 (d, 2H, J 8.7 Hz, ArH), 7.51 (d, 2H, J 7.8 Hz, ArH), 7.42 (t, 2H, J 7.8 Hz, ArH), 7.35 (t, 1H, J 7.8 Hz, ArH), 5.19 (s, 1H, H_4), 4.11 (d, 1H, J 7.3 Hz, H_3), 3.87 (m, 1H, H_1), 3.53 (t, 1H, J 7.3, H_2), 3.44 (m, 1H, NCHH), 2.73 (m, 1H, NCHH), 2.35 (m, 1H, CHH), 2.11 (m, 1H, CHH), 1.91 (m, 1H, CHH), 1.79 (m, 1H, CHH); δ_{C} (125MHz, CDCl_3); 194, 178.5, 176.6, 134.4, 133.6, 132, 131.8, 131.6, 129.4, 129.2, 126, 71.4, 67, 54.3, 50, 49.6, 49.3, 25.8, 24.7; $\nu_{\text{max}}/\text{cm}^{-1}$ 2988, 1713, 1655, 1586; H.R.M.S. [ES⁺] found MH^+ 439.0661. $\text{C}_{22}\text{H}_{20}\text{BrN}_2\text{O}_3$ requires MH 439.0651.(Br⁷⁹)

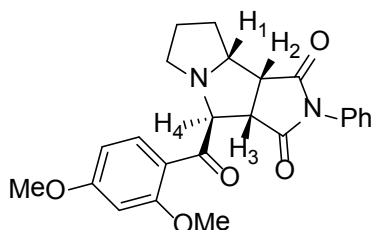
and

1-(4-Bromophenyl)-2-(1H-pyrrol-1-yl)ethan-1-one (22)



Product **232** (0.12g, 48% yield) as a brown solid; m.p. 157-158 °C; δ_{H} (500 MHz, CDCl_3); 7.81 (d, 2H, J 8.7 Hz, ArH), 7.65 (d, 2H, J 8.7 Hz, ArH), 6.66 (t, 2H, J 2.3 Hz, PyrroleH), 6.26 (t, 2H, J 2.3 Hz, PyrroleH), 5.28 (s, 2H, CH_2); δ_{C} (125 MHz, CDCl_3); 193.5, 133.4, 132.3, 129.5, 129.2, 122.8, 109.3, 55.4; $\nu_{\text{max}}/\text{cm}^{-1}$ 2685, 1705, 1586, 1072; H.R.M.S. [ES⁺] found MH^+ 264.0016 $\text{C}_{12}\text{H}_{11}\text{BrNO}$ requires MH 264.0018 (Br⁷⁹)

4-[(2,4-Dimethoxyphenyl)carbonyl]-2-phenyl-decahydropyrrolo[3,4-a]pyrrolizine-1,3-dione (23)



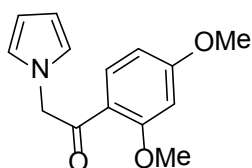
Prepared by general procedure **A** on a 1 mmol scale, Crude mixture was purified using column chromatography, eluting with 3:7 v/v ethyl-acetate/hexane to give the product **23** (0.14 g, 34%) as a dark yellow gum; δ_{H} (500 MHz, CDCl_3); 7.90

(d, 1H, *J* 8.9 Hz, ArH), 7.51-7.46 (m, 3H, ArH), 7.43-7.38 (m, 2H, ArH), 6.57 (dd, 1H, *J* 2.6, 8.9 Hz, ArH), 6.48 (d, 1H, *J* 2.6 Hz, ArH), 5.42 (s, 1H, H₄), 4.04 (d, 1H, *J* 8.6 Hz, H₃), 3.96 (s, 3H, O-CH₃), 3.92 (m, 1H, H₁), 3.87 (s, 3H, O-CH₃), 3.50 (t, 1H, *J* 8.6 Hz, H₂), 3.36 (m, 1H, NCHH), 2.71 (m, 1H, NCHH), 2.28 (m, 1H, CHH), 2.11 (m, 1H, CHH), 1.87 (m, 1H, CHH), 1.72 (m, 1H, CHH); δ_C (125 MHz, CDCl₃); 195, 178, 172.2, 165.1, 161, 134, 132.1, 129.3, 128.6, 126.5, 126, 106, 98, 74.8, 66.9, 55.6, 54.9, 49.9, 49.7, 25.9, 24.6; ν_{max}/cm⁻¹ 2973, 2943, 1718, 1680; H.R.M.S. [ES⁺] found MH⁺ 421.1764. C₂₄H₂₅N₂O₅ requires *MH* 421.1757.

		n.O.e %			
Protons Irradiated		H ₁	H ₂	H ₃	H ₄
	H ₁				
	H ₂	11.71		10.58	
	H ₃		7.87		3.63
	H ₄			5.27	

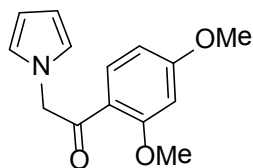
and

1-(2,4-Dimethoxyphenyl)-2-(1H-pyrrol-1-yl)ethan-1-one (24)



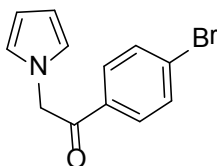
Product **25** (0.1g, 43% yield) as brown needles; m.p. 75-76 °C; δ_H (500 MHz, CDCl₃); 7.94 (d, 1H, *J* 8.7 Hz, ArH), 6.64 (s, 2H, PyrroleH), 6.57 (dd, 1H, *J* 2.3, 8.7 Hz, ArH), 6.49 (d, 1H, *J* 2.29 Hz, ArH), 6.22 (s, 2H, PyrroleH), 5.22 (s, 2H, CH₂), 3.96 (s, 3H, O-CH₃), 3.87 (s, 3H, O-CH₃); H.R.M.S. [ES⁺] found MH⁺ 246.11248 C₁₄H₁₆NO₃ requires *MH* 246.1

1-(2,4-Dimethoxyphenyl)-2-(1H-pyrrol-1-yl)ethan-1-one (24)



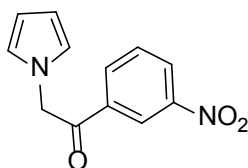
Prepared by general procedure **B** on a 1mmol scale, crude mixture was purified using column chromatography, eluting 3:7 v/v ethyl acetate/hexane to give the product **25** (0.1195g, 57% yield) as a brown solid; m.p. 75-76°C; δ_{H} (500 MHz, CDCl_3); 7.94 (d, 1H, J 8.7 Hz, ArH), 6.64 (s, 2H, PyrroleH), 6.57 (dd, 1H, J 2.3, 8.7 Hz, ArH), 6.49 (d, 1H, J 2.29 Hz, ArH), 6.22 (s, 2H, PyrroleH), 5.22 (s, 2H, CH_2), 3.96 (s, 3H, O- CH_3), 3.87 (s, 3H, O- CH_3); H.R.M.S. [ES⁺] found MH^+ 246.11248 $\text{C}_{14}\text{H}_{16}\text{NO}_3$ requires MH 246.1.

1-(4-Bromophenyl)-2-(1H-pyrrol-1-yl)ethan-1-one (22)



Prepared by general procedure **B** on a 1mmol scale, crude mixture was purified using column chromatography, eluting 3:7 v/v ethyl acetate/hexane to give the product **23** (0.1405g, 63% yield) as a brown solid; m.p. 157-58 °C; δ_{H} (500 MHz, CDCl_3); 7.81 (d, 2H, J 8.7 Hz, ArH), 7.65 (d, 2H, J 8.7 Hz, ArH), 6.66 (t, 2H, J 2.3 Hz, PyrroleH), 6.26 (t, 2H, J 2.3 Hz, PyrroleH), 5.28 (s, 2H, CH_2); δ_{C} (125 MHz, CDCl_3); 193, 133, 129, 122, 110, 56; $\nu_{\text{max}}/\text{cm}^{-1}$ 2685, 1705, 1586, 1072; H.R.M.S. [ES⁺] found MH^+ 264.0016 $\text{C}_{12}\text{H}_{11}\text{BrNO}$ requires MH 264.0018 (Br^{79})

Nitrophenyl)-2-(1H-pyrrol-1-yl)ethan-1-one (25)



Prepared by general procedure **B** on a 1mmol scale, crude mixture was purified using column chromatography, eluting 2:8 v/v ethyl acetate/hexane to give the product **26** (0.178g, 92% yield) as a brown solid; m.p. 112-113 °C; δ_{H} (500 MHz, CDCl_3); 8.75 (s, 1H, ArH), 8.48 (dd, 1H, J 7.8, 2.3 Hz, ArH), 8.26 (d, 1H, J 7.8 Hz, ArH), 7.73 (t, 1H, J 7.8 Hz, ArH), 6.69 (t, 2H, J 2.3 Hz, PyrroleH), 6.28 (t, 2H, J 2.3 Hz, PyrroleH), 5.37 (s, 2H, CH_2); δ_{C} (125 MHz, CDCl_3); 192, 148.5, 136, 133.6, 130.3, 128.1, 122.9, 121.8, 109, 109.6, 55.7; $\nu_{\text{max}}/\text{cm}^{-1}$ 2987, 1715, 1615, 1089; H.R.M.S. [ES⁺] found MNa^+ 253.0584. $\text{C}_{12}\text{H}_{10}\text{N}_2\text{O}_3$ requires MNa 253.0583.

checkCIF/PLATON report

No syntax errors found. CIF dictionary Interpreting this report

Datablock: jw19_a_manual

Bond precision: C-C = 0.0023 Å Wavelength=1.54180

Cell: a=12.5132(9) b=9.0524(4) c=14.4988(10)

alpha=90 beta=111.345(8) gamma=90

Temperature: 101 K

Calculated Reported

Volume 1529.69(19) 1529.67(17)

Space group P 21/n P 1 21/n 1

Hall group -P 2yn -P 2yn

Moiety formula C17 H20 N2 O3 C17 H20 N2 O3

Sum formula C17 H20 N2 O3 C17 H20 N2 O3

Mr 300.35 300.35

Dx,g cm-3 1.304 1.304

Z 4 4

Mu (mm-1) 0.732 0.732

F000 640.0 640.0

F000' 641.96

h,k,lmax 14,10,17 14,10,17

Nref 2705 2696

Tmin,Tmax 0.839,0.896 0.780,1.000

Tmin' 0.803

Correction method= MULTI-SCAN

Data completeness= 0.997 Theta(max)= 66.560

R(reflections)= 0.0422(2244) wR2(reflections)= 0.1111(2696)

S = 1.048 Npar= 202

The following ALERTS were generated. Each ALERT has the format

test-name ALERT alert-type alert-level.

Click on the hyperlinks for more details of the test.

Alert level G

PLAT005_ALERT_5_G No _iucr_refine_instructions_details in the CIF ? Do !

PLAT093_ALERT_1_G No su's on H-positions, refinement reported as . mixed

PLAT128_ALERT_4_G Note: Alternate Setting of Space-group P21/c . P21/n

PLAT152_ALERT_1_G The Supplied and Calc. Volume s.u. Differ by ... 2 Units

PLAT793_ALERT_4_G The Model has Chirality at C8 (Verify) S

PLAT793_ALERT_4_G The Model has Chirality at C9 (Verify) S

PLAT793_ALERT_4_G The Model has Chirality at C10 (Verify) R

PLAT793_ALERT_4_G The Model has Chirality at C11 (Verify) R

0 ALERT level A = Most likely a serious problem - resolve or explain

0 ALERT level B = A potentially serious problem, consider carefully

0 ALERT level C = Check. Ensure it is not caused by an omission or oversight

8 ALERT level G = General information/check it is not something unexpected

2 ALERT type 1 CIF construction/syntax error, inconsistent or missing data

0 ALERT type 2 Indicator that the structure model may be wrong or deficient

0 ALERT type 3 Indicator that the structure quality may be low

5 ALERT type 4 Improvement, methodology, query or suggestion

1 ALERT type 5 Informative message, check

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 01/06/2013; check.def file version of 24/05/2013

