Microwave-Assisted One-Pot Synthesis of 1-Indanones from Arenes and α,β -Unsaturated Acyl Chlorides

Wei Yin^a, Yuan Ma^{a,*}, Jiayi Xu^b, and Yufen Zhao^a

^aKey Laboratory of Bioorganic Phosphorus Chemistry and Chemical Biology, Ministry of Education, Department of Chemistry, Tsinghua University, Beijing 100084, People's Republic of China. ^bKey Laboratory of Bioorganic Chemistry and Molecular Engineering of Ministry of Education, College of Chemistry and Molecular Engineering, Peking University, Beijing 100871, People's Republic of China

mayuan@mail.tsinghua.edu.cn

Supplemental Materials

Content	S1
Experimental details: General.....	S2
The analytic data of 1-indanones 4 and 5	S2
Copies of ¹ H NMR and ¹³ C NMR spectra of the unknown compounds 4 and 5	S8
Copy of ¹ H NMR NOE spectrum of unknown compound 4k'	S17

Experimental Details

General. *o*-Dichlorobenzene was dried over anhydrous sodium sulfate and distilled prior to use. All reactions were performed in a commercial microwave reactor (XH-100A, 100 - 1000 W, Beijing Xianghu Science and Technology Development Co. Ltd, Beijing, P. R. China). The temperature of the reaction mixture was measured by an immersed platinum resistance thermometer. Melting points were measured on a melting point apparatus and are uncorrected. ^1H NMR (200 or 300 MHz) and ^{13}C NMR (50 or 75 MHz) spectra were recorded in CDCl_3 with TMS as an internal standard. Mass spectra and HRMS data were obtained on an EI mass spectrometer. IR spectra were recorded on a FT-IR spectrometer with an OMNI sampler. TLC separations were performed on silica gel GF₂₅₄ plates, and the plates were visualized with UV light.

3-Methyl-1-indanone (4a)

Colorless oil, ^1H NMR (300 MHz, CDCl_3) δ 7.73-7.34 (m, 4H), 3.43 (ddq, $J = 7.5, 3.4, 6.9$ Hz, 1H), 2.93 (dd, $J = 19.2, 7.5$ Hz, 1H), 2.27 (dd, $J = 19.2, 3.4$ Hz, 1H), 1.40 (d, $J = 6.9$ Hz, 3H); ^{13}C NMR (75.5 MHz, CDCl_3) δ 21.2, 32.6, 45.2, 123.3, 125.2, 127.3, 134.6, 136.3, 159.8, 206.3. Known compound.¹

3,5-Dimethyl-1-indanone (4b)

Colorless oil, ^1H NMR (200 MHz, CDCl_3) δ 7.61 (d, $J = 8.0$ Hz, 1H), 7.29 (s, 1H), 7.17 (d, $J = 8.0$ Hz, 1H), 3.38 (ddq, $J = 7.6, 3.5, 7.4$ Hz, 1H), 2.91 (dd, $J = 19.0, 7.6$ Hz, 1H), 2.45 (s, 3H), 2.24 (dd, $J = 19.0, 3.5$ Hz, 1H), 1.38 (d, $J = 7.4$ Hz, 3H); ^{13}C NMR (50.3 MHz, CDCl_3) δ 21.2, 22.0, 32.5, 45.4, 123.2, 125.5, 128.6, 134.1, 145.8, 160.4, 205.6. Known compound.²

5-Fluoro-3-methyl-1-indanone (4c)

Colorless oil, ^1H NMR (200 MHz, CDCl_3) δ 7.76-7.69 (m, 1H), 7.18-7.02 (m, 2H), 3.43 (ddq, $J = 7.4, 3.4, 7.0$ Hz, 1H), 2.96 (dd, $J = 18.9, 7.4$ Hz, 1H), 2.30 (dd, $J = 18.9, 3.4$ Hz, 1H), 1.41 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (50.3 MHz, CDCl_3) δ 21.1, 32.6, 45.4, 124.9, 125.6, 127.4, 132.8, 155.0, 162.9, 204.3. Known compound.⁴

5-Chloro-3-methyl-1-indanone (4d)

Colorless oil, ^1H NMR (200 MHz, CDCl_3) δ 7.63 (d, $J = 8.4$ Hz, 1H), 7.49 (s, 1H), 7.34 (d, $J = 8.4$ Hz, 1H), 3.42 (ddq, $J = 7.6, 3.6, 7.0$ Hz, 1H), 2.95 (dd, $J = 19.2, 7.6$ Hz, 1H), 2.28 (dd, $J = 19.2, 3.6$ Hz, 1H), 1.40 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (50.3 MHz, CDCl_3) δ 21.0, 32.6, 45.2, 124.6, 125.5, 127.4, 128.1, 141.2, 161.3, 204.8. Known compound.⁵

5-Bromo-3-methyl-1-indanone (4e)

Colorless oil, ^1H NMR (300 MHz, CDCl_3) δ 7.64 (d, $J = 8.2$ Hz, 1H), 7.56 (s, 1H), 7.51 (d, $J = 8.2$ Hz, 1H), 3.43 (ddq, $J = 7.7, 3.6, 7.2$ Hz, 1H), 2.93 (dd, $J = 19.2, 7.7$ Hz, 1H), 2.27 (dd, $J = 19.2, 3.6$ Hz, 1H), 1.40 (d, $J = 7.2$ Hz, 3H); ^{13}C NMR (75.5 MHz, CDCl_3) δ 21.0, 32.5, 45.1, 124.5, 128.6, 129.9, 130.9, 135.1, 161.3, 204.7. Known compound.⁶

3-Methyl-5-phenyl-1-indanone (4f)

Colorless crystals, m.p. 79.5-80.5 $^{\circ}\text{C}$; Lit⁷ m.p. 74-75.5 $^{\circ}\text{C}$; ^1H NMR (300 MHz, CDCl_3) δ 7.82-7.42 (m, 8H), 3.50 (ddq, $J = 7.6, 3.4, 7.2$ Hz, 1H), 2.99 (dd, $J = 19.0, 7.6$ Hz, 1H), 2.34 (dd, $J = 19.0, 3.4$ Hz, 1H), 1.46 (d, $J = 7.2$ Hz, 3H); ^{13}C NMR (75.5 MHz, CDCl_3) δ 21.3, 32.8, 45.6, 123.77, 123.80, 126.9, 127.5, 128.3, 128.9, 135.2, 140.3, 147.9, 160.6, 206.1.

5-Isopropyl-3-methyl-1-indanone (4g)

Colorless oil, $R_f = 0.38$ [ethyl acetate: petroleum ether (60-90 $^{\circ}\text{C}$) 1:15, v/v, silica gel plate]; IR (KBr) ν (cm^{-1}): 1711.8 (C=O); ^1H NMR (200 MHz, CDCl_3) δ 7.65 (d, $J = 8.0$ Hz, 1H), 7.34 (s, 1H), 7.25 (d, $J = 8.0$ Hz, 1H), 3.40 (ddq, $J = 7.4, 3.4, 7.0$ Hz, 1H), 2.98 (m, $J = 7.0$ Hz, 1H), 2.92 (dd, $J = 19.0, 7.4$ Hz, 1H), 2.26 (dd, $J = 19.0, 3.4$ Hz, 1H), 1.40 (d, $J = 7.0$ Hz, 3H), 1.29 (d, $J = 7.0$ Hz, 6H); ^{13}C NMR (50.3 MHz, CDCl_3) δ 21.2, 23.6, 32.6, 34.6, 45.5, 122.8, 123.3, 126.1, 134.4, 156.7, 160.5, 205.9. MS (EI) m/z (rel. intensity, %): 188 (M^+ , 44), 173 ($\text{M}^+ - \text{CH}_3$, 100), 145 ($\text{M}^+ - \text{Pr}$, 29), 131 (22), 115 (21), 77 (11); HRMS Calcd. for $\text{C}_{13}\text{H}_{16}\text{O}$ (M^+) 188.1201; Found 188.1201.

3,5,7-Trimethyl-1-indanone (4i)

Colorless oil, ^1H NMR (300 MHz, CDCl_3) δ 7.11 (s, 1H), 6.92 (s, 1H), 3.31 (ddq, $J = 7.8, 3.6, 7.2$ Hz, 1H), 2.88 (dd, $J = 18.9, 7.8$ Hz, 1H), 2.59 (s, 3H), 2.39 (s, 3H), 2.23 (dd, $J = 18.9, 3.6$ Hz, 1H), 1.35 (d, $J = 7.2$ Hz, 3H); ^{13}C NMR (75.5 MHz, CDCl_3) δ 18.2, 21.4, 21.8, 31.9, 46.0, 123.0, 130.3, 131.6, 138.2, 145.0, 161.3, 206.7. Known compound.²

3,4,7-Trimethyl-1-indanone (4j)

Colorless oil, ^1H NMR (300 MHz, CDCl_3) δ 7.23 (d, $J = 7.5$ Hz, 1H), 7.01 (d, $J = 7.5$ Hz, 1H), 3.43 (ddq, $J = 7.8, 1.6, 6.9$ Hz, 1H), 2.88 (dd, $J = 18.7, 7.8$ Hz, 1H), 2.59 (s, 3H), 2.36 (s, 3H), 2.29 (dd, $J = 18.7, 1.6$ Hz, 1H), 1.29 (d, $J = 6.9$ Hz, 3H); ^{13}C NMR (75.5 MHz, CDCl_3) δ 17.6, 18.0, 21.3, 31.4, 46.3, 129.4, 132.3, 133.2, 135.3, 135.8, 158.8, 207.8. Known compound.⁸

5,6-Dichloro-3-methyl-1-indanone (4k)

Yellowish solid, m.p. 67.5-68.5 °C; $R_f = 0.26$ [ethyl acetate: petroleum ether (60-90 °C) 1:15, v/v, silica gel plate]; IR (KBr) ν (cm^{-1}): 1720.2 (C=O); ^1H NMR (200 MHz, CDCl_3) δ 7.79 (s, 1H), 7.62 (s, 1H), 3.42 (ddq, $J = 7.6, 3.2, 7.0$ Hz, 1H), 2.98 (dd, $J = 19.2, 7.6$ Hz, 1H), 2.32 (dd, $J = 19.2, 3.2$ Hz, 1H), 1.41 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (50.3 MHz, CDCl_3) δ 21.1, 32.4, 45.3, 116.0, 124.9, 127.4, 136.0, 139.2, 158.6, 203.7. MS (EI) m/z (rel. intensity, %): 214 (M^+ , 56), 199 ($\text{M}^+ - \text{CH}_3$, 100), 179 ($\text{M}^+ - \text{Cl}$, 15), 136 (20), 115 (30), 83 (16); HRMS Calcd. for $\text{C}_{10}\text{H}_8\text{Cl}_2\text{O}$ (M^+) 213.9952; Found 213.9952.

4,5-Dichloro-3-methyl-1-indanone (4k')

Yellowish oil, $R_f = 0.34$ [ethyl acetate: petroleum ether (60-90 °C) 1:15, v/v, silica gel plate]; IR (KBr) ν (cm^{-1}): 1721.0 (C=O); ^1H NMR (200 MHz, CDCl_3) δ 7.56 (d, $J = 8.1$ Hz, 1H), 7.48 (d, $J = 8.1$ Hz, 1H), 3.60 (ddq, $J = 7.6, 1.2, 6.8$ Hz, 1H), 2.99 (dd, $J = 19.2, 7.6$ Hz, 1H), 2.40 (dd, $J = 19.2, 1.2$ Hz, 1H), 1.44 (d, $J = 6.8$ Hz, 3H); ^{13}C NMR (50.3 MHz, CDCl_3) δ 20.2, 33.0, 45.6, 122.2, 130.1, 130.7, 136.2, 139.5, 158.1, 204.1. MS (EI) m/z (rel. intensity, %): 214 (M^+ , 46), 199 ($\text{M}^+ - \text{CH}_3$, 100), 179 ($\text{M}^+ - \text{Cl}$, 9), 136 (14), 115 (22), 83 (19); HRMS Calcd. for $\text{C}_{10}\text{H}_8\text{Cl}_2\text{O}$ (M^+) 214.9952; Found 214.9949.

3-Phenyl-1-indanone (4l)

Colorless crystals, m.p. 77-78.5 °C; Lit¹ m.p. 78 °C; ¹H NMR (200 MHz, CDCl₃) δ 7.62-7.11 (m, 9H), 4.58 (dd, J = 8.1, 3.8 Hz, 1H), 3.24 (dd, J = 19.2, 8.1 Hz, 1H) 2.69 (dd, J = 19.2, 3.8 Hz, 1H); ¹³C NMR (50.3 MHz, CDCl₃) δ 44.4, 46.8, 123.4, 126.9, 127.0, 127.7, 127.9, 128.9, 135.1, 136.8, 143.7, 158.0, 206.2.

5-Fluoro-3-phenyl-1-indanone (4m)

Colorless crystals, m.p. 107.5-108.5 °C; R_f = 0.30 [ethyl acetate: petroleum ether (60-90 °C) 1:15, v/v, silica gel plate]; IR (KBr) ν (cm⁻¹): 1713.1 (C=O); ¹H NMR (300 MHz, CDCl₃): δ 7.83-6.95 (m, 8H), 4.57 (dd, J = 8.1, 3.8 Hz, 1H), 3.23 (dd, J = 19.3, 8.1 Hz, 1H), 2.63 (dd, J = 19.3, 3.8 Hz, 1H); ¹³C NMR (50 MHz, CDCl₃): δ 43.7, 46.9, 115.5, 115.9, 123.5, 126.7, 128.0, 129.0, 129.2, 135.1, 157.6, 164.3, 205.4. MS (EI) m/z (rel. intensity, %): 226 (M⁺, 100), 208 (M⁺-PhH, 31), 207 (M⁺-F, 11), 197 (39), 183 (31), 77 (24); HRMS Calcd. for C₁₅H₁₁FO (M⁺) 226.0794; Found 226.0788.

5-Chloro-3-phenyl-1-indanone (4n)

Colorless oil; ¹H NMR (200 MHz, CDCl₃) δ 7.03-7.83 (m, 8H), 4.56 (dd, J = 8.0, 4.0 Hz, 1H), 3.22 (dd, J = 19.2, 8.0 Hz, 1H) 2.62 (dd, J = 19.2, 4.0 Hz, 1H); ¹³C NMR (50.3 MHz, CDCl₃) δ 43.8, 46.7, 123.5, 126.7, 128.1, 128.9, 129.0, 130.9, 132.8, 135.1, 142.2, 155.0, 205.2. Known compound.^{3,9}

3,7-Dimethyl-1-indanone (5b)

Colorless oil, ¹H NMR (200 MHz, CDCl₃) δ 7.44 (t, J = 7.8, 7.4 Hz, 1H), 7.25 (d, J = 7.8 Hz, 1H), 7.09 (d, J = 7.4 Hz, 1H), 3.36 (ddq, J = 7.7, 3.6, 7.0 Hz, 1H), 2.89 (dd, J = 18.8, 7.7 Hz, 1H), 2.63 (s, 3H), 2.24 (dd, J = 18.8, 3.6 Hz, 1H), 1.37 (d, J = 7.0 Hz, 3H); ¹³C NMR (50.3 MHz, CDCl₃) δ 18.2, 21.4, 32.1, 45.8, 122.5, 128.3, 129.1, 133.9, 138.5, 160.7, 205.6. Known compound.^{2,3}

7-Fluoro-3-methyl-1-indanone (5c)

Colorless oil, $R_f = 0.45$ [ethyl acetate: petroleum ether (60-90 °C) 1:15, v/v, silica gel plate]; IR (KBr) ν (cm^{-1}): 1719.1 (C=O); ^1H NMR (200 MHz, CDCl_3) δ 7.59-7.09 (m, 3H), 3.60 (ddq, $J = 7.8, 1.6, 7.0$ Hz, 1H), 2.99 (dd, $J = 19.2, 7.8$ Hz, 1H), 2.40 (dd, $J = 19.2, 1.6$ Hz, 1H), 1.44 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (50.3 MHz, CDCl_3) δ 20.3, 33.1, 45.7, 122.3, 130.2, 130.9, 136.3, 139.6, 158.2, 204.2. MS (EI) m/z (rel. intensity, %): 164 (M^+ , 70), 163 ($\text{M}^+ - \text{H}$, 100), 149 ($\text{M}^+ - \text{CH}_3$, 16), 145 ($\text{M}^+ - \text{F}$, 6); HRMS Calcd. for $\text{C}_{10}\text{H}_9\text{FO}$ (M^+) 164.0637; Found 164.0637.

7-Chloro-3-methyl-1-indanone (5d)

Colorless oil, $R_f = 0.45$ [ethyl acetate: petroleum ether (60-90 °C) 1:15, v/v, silica gel plate]; IR (KBr) ν (cm^{-1}): 1720.0 (C=O); ^1H NMR (200 MHz, CDCl_3) δ 7.62-7.41 (m, 3H), 3.60 (ddq, $J = 7.6, 1.8, 7.0$ Hz, 1H), 2.99 (dd, $J = 19.2, 7.6$ Hz, 1H), 2.40 (dd, $J = 19.2, 1.8$ Hz, 1H), 1.44 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (50.3 MHz, CDCl_3) δ 20.3, 33.1, 45.8, 122.3, 129.2, 130.2, 136.4, 139.6, 158.2, 204.1. MS (EI) m/z (rel. intensity, %): 180 (M^+ , 20), 165 ($\text{M}^+ - \text{CH}_3$, 30), 145 ($\text{M}^+ - \text{Cl}$, 13), 115 (100), 91 (7); HRMS Calcd. for $\text{C}_{10}\text{H}_9\text{ClO}$ (M^+) 180.0342; Found 180.0344.

7-Bromo-3-methyl-1-indanone (5e)

Colorless oil, $R_f = 0.45$ [ethyl acetate: petroleum ether (60-90 °C) 1:15, v/v, silica gel plate]; IR (KBr) ν (cm^{-1}): 1719.7 (C=O); ^1H NMR (200 MHz, CDCl_3) δ 7.84-7.27 (m, 3H), 3.61 (ddq, $J = 7.8, 1.8, 7.0$ Hz, 1H), 2.99 (dd, $J = 19.2, 7.8$ Hz, 1H), 2.40 (dd, $J = 19.2, 1.8$ Hz, 1H), 1.44 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (50.3 MHz, CDCl_3) δ 20.3, 33.2, 45.8, 122.3, 129.8, 130.3, 131.9, 138.3, 158.3, 204.2. MS (EI) m/z (rel. intensity, %): 224 (M^+ , 19), 209 ($\text{M}^+ - \text{CH}_3$, 33), 183 ($\text{M}^+ - \text{CH}_3\text{CHCH}_2$, 100), 145 ($\text{M}^+ - \text{Br}$, 13); HRMS Calcd. for $\text{C}_{10}\text{H}_9\text{BrO}$ (M^+) 223.9837; Found 223.9835.

REFERENCES

1. Gagnier, S. V.; Larock, R. C. *J. Am. Chem. Soc.* **2003**, *125*, 4804-4807.
2. Kemp, W.; Spanswick, J. *J. Chem. Soc. Perkin Trans. I.* **1972**, *1*, 151-155.
3. Allen, J. M.; Johnston, K. M.; Jones, J. F.; Shotter, R. G. *Tetrahedron.* **1977**, *33*, 2083-2087.
4. Pines, S. H.; Douglas, A. W. *J. Am. Chem. Soc.* **1976**, *98*, 8119-8125.
5. Kerr, C. A.; Rae, I. D. *Aust. J. Chem.* **1978**, *31*, 341-346.

6. Sircar, I.; Duell, B. L.; Cain, M. H.; Burke, S. E.; Bristol, J. A. *J. Med. Chem.* **1986**, 29, 2142-2148.
7. Smith, C. E.; Williamson, W. R. N.; Cashin, C. H.; Kitchen, E. A. *J. Med. Chem.* **1979**, 22, 1464-1469.
8. Boykin, D. W.; Hertzler, R. L.; Delphon, J. K.; Eisenbraun, E. J. *J. Org. Chem.* **1989**, 54, 1418-23.
9. Boegesoe, K. P.; Arnt, J.; Hyttel, J.; Pedersen, H. *J. Med. Chem.* **1993**, 36, 2761-2770.

5-Isopropyl-3-methyl-1-indanone (4g)

containing a little 4a

4g containing a little 4a

¹³C NMR spectrum of 4a

5,6-Dichloro-3-methyl-1-indanone (4k)

4,5-Dichloro-3-methyl-1-indanone (4k')

YV-o2C1 (1)
 expl s2pu1
 SAMPLE DEC. & VT
 date Jan 3 2006 dn H1
 solvent CDCl3 dof 1100.0
 file exp dm nm
 ACQUISITION dmm C
 sfrq 199.911 dmf 9900
 tn H1
 at 2.000 lb 0.50
 np 20000 fn not used
 sw 5000.0
 fb 2800 werr
 bs 4 wexp
 pw 5.0 wbs
 pw 5.0 wnt
 tpwr 54
 d1 1.000 sp
 tof 315.0 wp -81.6
 nt 16 vs 1938.0
 ct 16 sc 90
 alock n wc 250
 gain 2 hzmm 7.75
 FLAGS is 879.41
 il n rfl 1149.4
 in n rfp 0
 dp y th 6
 ins 1.000
 nm ph

YV-o2C1 (1)
 exp2 std13c
 SAMPLE DEC. & VT
 date Jan 3 2006 dfrq 199.911
 solvent CDCl3 dn H1
 file exp dpwr 35
 ACQUISITION dof 1100.0
 sfrq 50.273 dm yyy
 tn C13 dmm w
 at 1.500 dmf 9900
 np 375.04 PROCESSING
 sw 12500.0 lb 0.50
 fb 7000 wtfile ft
 bs 4 proc not used
 tpwr 6.0
 d1 1.000 werr
 tof 850.0 wexp
 nt 2000 wbs
 ct 36 wnt
 alock n
 gain 20
 FLAGS n
 il n
 in n
 dp y
 DISPLAY
 sp -136.1
 wp 11372.7
 vs 19
 sc 0
 wc 250
 hzmm 45.48
 is 5151.52
 rfl 4295.8
 rfp 3870.6
 th 3
 ins 77.000
 nm no ph

5-Fluoro-3-phenyl-1-indanone (4m)

5-Bromo-3-phenyl-1-indanone (4o)

YW-Cin-Br 25-27

exp1 s2pu1

SAMPLE		temp	SPECIAL
date	May 23 2005		not used
solvent	CDC13	gain	not used
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	8006.0	pw90	9.500
at	1.996	alfa	20.000
np	23970	FLAGS	
fb	not used	l1	n
bs	4	ln	n
d1	1.000	dp	y
nt	16	hs	nn
ct	16	PROCESSING	
TRANSMITTER		lb	0.50
tn	H1	fn	65536
sfrq	300.067	DISPLAY	
tof	645.7	sp	-166.2
tpwr	60	wp	3890.7
pw	4.750	rfl	868.8
DECOUPLER		rfp	0
dn	C13	rp	-39.1
dof	645.7	lp	-62.4
dm	nnm	PLOT	
dmm	c	vc	250
dpwr	42	sc	0
dmf	19400	vs	49
		th	17
		nm	ph

YW-Cin-Br 25-27

exp2 std13c

SAMPLE		DEC. & VT	
date	May 23 2005	dfrq 300.066	
solvent	CDC13	dn H1	
file	exp	dpwr 40	
ACQUISITION		dof 0	
sfrq	75.460	dm yyy	
tn	C13	dmm w	
at	1.500	dmf 11200	
np	58822	PROCESSING	
sw	19607.5	lb	0.50
fb	10800	wtfile	ft
bs	4	proc	not used
tpwr	59	fn	
pw	4.0		
d1	1.000	warr	
tof	1196.0	wexp	
nt	2000	wbs	
ct	186	wnt	
alock	n		
gain	not used		
FLAGS			
l1	n		
ln	n		
dp	y		
DISPLAY			
sp	-1066.9		
wp	19607.2		
vs	23		
sc	0		
wc	250		
h2mm	78.43		
ls	16409		
rrf1	1067.5		
rfp	2		
th	2		
ins	1.000		
nm	no ph		

7-Fluoro-3-methyl-1-indanone (5c)

7-Chloro-3-methyl-1-indanone (5d)

7-Bromo-3-methyl-1-indanone (5e)

7-Bromo-3-phenyl-1-indanone (5o)

YW-Cin-Br 11-13
exp1 s2pul

date	May 23 2005	temp	not used
solvent	CDC13	gain	not used
file	exp	spin	not used
ACQUISITION	exp	hst	0.008
sw	6006.0	pu90	8.500
at	1.586	alfa	20.000
np	23970	FLAGS	
fb	not used	il	n
bs	4	in	n
d1	1.000	dp	y
nt	18	hs	nm
ct			
TRANSMITTER	1b	fn	0.50
tn	H1	fn	65536
sfrq	300.067	sp	
tof	645.7	sp	-120.4
tpwr	60	wp	3854.7
pw	4.750	rfl	870.6
DECOUPLER	C13	rfl	0
dn		rp	-40.6
dof	645.7	lp	-54.1
dm	nmn		
dmm		vc	250
dpwr	42	sc	0
dmf	19400	vs	49
		th	9
		nm	ph

YW-Cin-Br 11-13
exp2 std13c

date	May 23 2005	dfrq	300.066
solvent	CDC13	dn	H1
file	exp	dpwr	40
ACQUISITION	exp	dof	0
sfrq	75.460	dm	yyy
tn	C13	dm	v
at	1.500	dmf	11200
np	58822	PROCESSING	0.50
sw	19607.5	lb	
fb	10800	wtfile	ft
bs	59	fn	not used
tpwr	4.0		
pw	1.000	warr	
d1	1196.0	wexp	
tof	2000	wbs	
nt	92	wnt	
ct			
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-1068.1		
vp	19607.2		
vs	15		
sc	0		
wc	250		
hzmm	78.43		
ls	1e+09		
rfl	8878.4		
rfd	5809.5		
th	3		
ins	1.000		
nm	no	ph	

NOE spectrum of 4,5-Dichloro-3-methyl-1-indanone (4k')

No obvious NOE was observed.