Microwave– and Photoirradiation–Induced Staudinger Reactions of Cyclic Imines and Ketenes Generated from *α*–Diazoketones. A Further Investigation into the Stereochemical Process

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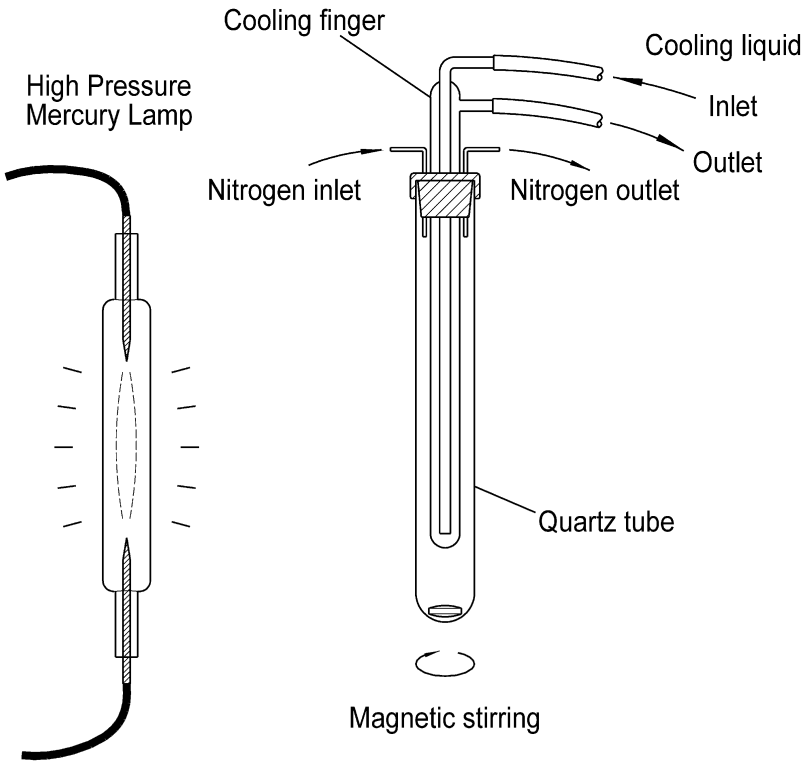
Supplemental Materials

Content S1

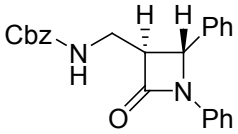
Experimental Details

General. α−Diazoketone **1**,[1] imines **2a**,[2] **2b-c**,[3] **3a−e**,[4] and **4a−b**[5] were prepared according to published procedures. Dichloromethane and 1,2−dichlorobenzene were refluxed with CaH_2 and freshly distilled prior to use. All reactions were performed under a nitrogen atmosphere. For microwave assisted reactions, a domestic microwave oven was employed and the power of microwave irradiation was set to about 500 W (Medium). Nitrogen inlet and outlet were connected via a reflux condenser with the flask in the microwave oven. Photochemical reactions were carried out in a quartz tube equipped with a cooling finger, nitrogen inlet and outlet. The reaction mixture was cooled by a circulation of −20 ˚C liquid though the cooling finger. A 500 W high−pressure mercury lamp was used as UV source, with a distance of 10 cm to the quartz tube (shown in the following figure).



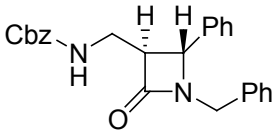
[1] Podlech, J.; Seebach, D. *Liebigs Ann.*, **1995**, 1217−1228.
[2] Bigelow, L. A.; Eatough, H. *Org. Syn.*, *Coll. Vol. 1*, 80.
[3] Linder, M. R.; Frey, W. U.; Podlech, J. *J. Chem. Soc.*, *Perkin Trans. 1* **2001**, 2566-2577.
[4] Wenker, H. *J. Am. Chem. Soc.*, **1935**, *57*, 6, 1079−1080.
[5] Wardrop, A. W. H.; Sainsbury, G. L.; Harrison, J. M.; Inch, T. D. *J. Chem. Soc.*, *Perkin Trans. 1*, **1976**, 1279−1285.

(±)−*trans*−3−Benzyloxycarbonylaminomethyl−1,4−diphenylazetidin−2−one (5a)



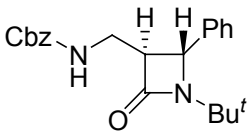
White solid; m.p. 138−139 °C; ^{1}H NMR (300 MHz): δ 3.21 (dt, J = 2.4, 5.7 Hz, 1H), 3.72 (t, J = 5.7 Hz, 2H), 4.93 (s, 1H), 5.10 (d, J = 12.3 Hz, 1H), 5.15 (d, J = 12.3 Hz, 1H), 5.31 (s, br, 1H), 7.06 (m, 1H), 7.23−7.32 (m, 14H). ^{13}C NMR (75.5 MHz): δ 38.7, 58.5, 60.5, 66.9, 117.1, 124.0, 125.8, 128.0, 128.1, 128.4, 128.5, 129.0, 129.1, 136.2, 137.2, 156.8, 165.4. MS (EI) m/z: 386 (M^+, 4.5), 358 (M^+−CO, 8.0), 295 (M^+−Bn, 12), 278 (M^+−BnOH, 5.5), 222 (M^+−CbzNHCH$_2$, 16), 91 (Bn^+, 100); IR ν (cm^{-1}): 1748, 1720. Calcd for $C_{24}H_{22}N_2O_3$: 386.1630. Found: 386.1640.

(±)−*trans*−3−Benzyloxycarbonylaminomethyl−1−benzyl−4−phenylazetidin−2−one (5b)



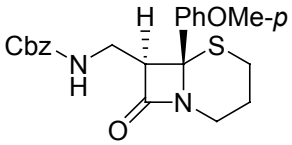
White solid; m.p. 82−83 °C; ^{1}H NMR (300 MHz): δ 3.15 (m, 1H), 3.61 (m, 2H), 3.74 (d, J = 15.0 Hz, 1H), 4.29 (d, J = 1.5 Hz, 1H), 4.82 (d, J = 15.0 Hz, 1H), 4.97 (d, J = 12.3 Hz, 1H), 5.10 (d, J = 12.3 Hz, 1H), 5.34 (s, br, 1H), 7.10−7.38 (m, 15H). ^{13}C NMR (75.5 MHz): δ 38.5, 44.4, 57.6, 60.3, 66.7, 126.4, 127.6, 127.9, 128.0, 128.2, 128.4, 128.7, 128.9, 135.2, 136.2, 136.9, 156.6, 168.1. MS (EI) m/z: 400 (M^+, 0.3), 372 (M^+−CO, 1.2), 309 (M^+−Bn, 5.3), 236 (M^+−CbzNHCH$_2$, 0.9), 91 (Bn^+, 100); IR ν (cm^{-1}): 1727, 1711. Calcd for $C_{25}H_{24}N_2O_3$: 400.1787. Found: 400.1783.

(±)−*trans*−3−Benzyloxycarbonylaminomethyl−1−tertbutyl−4−phenylazetidin−2−one (5c)



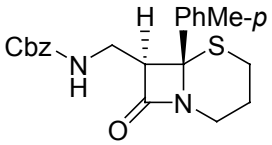
White solid; m.p. 146−147 °C; ^{1}H NMR (300 MHz): δ 1.20 (s, 9H), 2.90 (dt, J = 1.8, 5.6 Hz, 1H), 3.60 (m, 2H), 4.44 (d, J = 1.8 Hz, 1H), 5.12 (d, J = 12.3 Hz, 1H), 5.16 (d, J = 12.3 Hz, 1H), 5.21 (s, br, 1H), 7.31−7.36 (m, 10H). ^{13}C NMR (75.5 MHz): δ 28.1, 38.6, 54.7, 57.4, 58.9, 66.8, 126.2, 128.1, 128.5, 128.7, 136.4, 140.2, 156.7, 168.5. MS (EI) m/z: 366 (M^+, 1.1), 338 (M^+−CO, 6.6), 275 (M^+−Bn, 3.9), 91 (Bn^+, 73); IR ν (cm^{-1}): 1721, 1706. Calcd for $C_{22}H_{26}N_2O_3$: 366.1943. Found: 366.1935.

(±)−*cis*−8−Benzyloxycarbonylaminomethyl−1−(4−methoxyphenyl)−2−thia−6−aza−bicyclo[4.2.0]octan−7−one (6b)



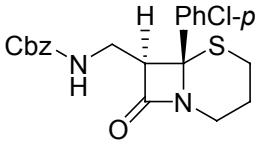
Colorless oil; ^{1}H NMR (300 MHz): δ 1.81 (m, 2H), 2.62 (m, 2H), 3.05 (m, 3H), 3.68 (t, J = 7.8 Hz, 1H), 3.82 (s, 3H), 4.09 (m, 1H), 4.74 (s, br, 1H), 5.02 (s, 2H), 6.92 (d, J = 8.5 Hz, 2H), 7.32−7.35 (m, 5H), 7.46 (d, J = 8.5 Hz, 2H). ^{13}C NMR (75.5 MHz): δ 23.8, 25.4, 37.4, 37.9, 55.3, 64.3, 65.4, 66.6, 114.0, 128.0, 128.4, 128.9, 136.3, 155.8, 159.5, 166.4. MS (EI) m/z: 412 (M^+, 0.70), 321 (M^+−Bn, 1.1), 304 (M^+−BnOH, 3.1), 248 (M^+−$CbzNHCH_2O$, 100); IR ν (cm^{-1}): 1750, 1717. Calcd for $C_{22}H_{24}N_2O_4S$: 412.1457. Found: 412.1439.

(±)−*cis*−8−Benzyloxycarbonylaminomethyl−1−(4−methylphenyl)−2−thia−6−aza−bicyclo[4.2.0]octan−7−one (6c)



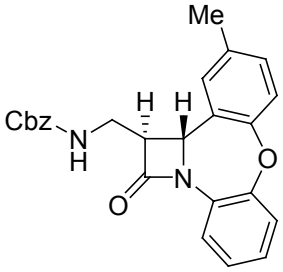
Colorless oil; ^{1}H NMR (300 MHz): δ 1.81 (m, 2H), 2.36 (s, 3H), 2.61 (m, 2H), 3.04 (m, 3H), 3.69 (t, J = 8.0 Hz, 1H), 4.10 (m, 1H), 4.76 (s, br, 1H), 5.02 (s, 2H), 7.19−7.43 (m, 9H). ^{13}C NMR (75.5 MHz): δ 21.0, 23.7, 25.3, 37.5, 37.8, 64.1, 65.5, 66.6, 127.4, 128.0, 128.4, 129.4, 133.7, 136.3, 138.3, 155.7, 166.3. MS (EI) m/z: 396 (M^+, 0.64), 368 (M^+−CO, 0.76), 305 (M^+−Bn, 0.5), 288 (M^+−BnOH, 5.3), 232 (M^+−$CbzNHCH_2$, 100); IR ν (cm^{-1}): 1756, 1717 Calcd for $C_{22}H_{24}N_2O_3S$: 396.1508. Found: 396.1491.

(±)−*cis*−8−Benzyloxycarbonylaminomethyl−1−(4−chlorophenyl)−2−thia−6−aza−bicyclo[4.2.0]octan−7−one (6d)



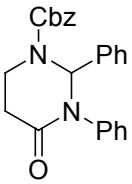
Colorless oil; ^{1}H NMR (300 MHz): δ 1.81 (m, 2H), 2.59 (m, 2H), 3.00 (m, 3H), 3.71 (t, J = 7.8 Hz, 1H), 4.10 (m, 1H), 4.90 (s, br, 1H), 5.01 (s, 2H), 7.28−7.49 (m, 9H). ^{13}C NMR (75.5 MHz): δ 23.5, 25.3, 37.5, 37.8, 64.1, 65.0, 66.7, 128.0, 128.4, 128.8, 129.0, 134.4, 135.6, 136.2, 155.7, 166.2. MS (EI) m/z: 416 (M^+, 0.62), 388 (M^+−CO, 1.1), 325 (M^+−Bn, 3.8), 308 (M^+−BnOH, 4.2), 252 (M^+−$CbzNHCH_2$, 75), 91 (Bn^+, 100); IR ν (cm^{-1}): 1752, 1717. Calcd for $C_{21}H_{21}ClN_2O_3S$: 416.0961. Found: 416.0958.

(±)−*trans*−2−Benzyloxycarbonylaminomethyl−4−methyl−azeto[1,2−*d*]−dibenzo[*b*,*f*] oxazepin−1−one (7b)



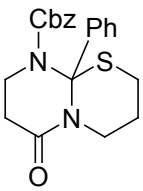
Pale yellow oil; ^{1}H NMR (400 MHz): δ 2.32 (s, 3H), 3.83 (m, 3H), 5.13 (s, 2H), 5.36 (s, br, 1H), 5.49 (s, 1H), 6.97−7.07 (m, 3H), 7.11 (m, 2H), 7.19−7.22 (m, 1H), 7.29−7.33 (m, 5H), 7.94−7.96 (m, 1H). ^{13}C NMR (75.5 MHz): δ 20.8, 38.8, 53.8, 55.9, 66.8, 119.8, 121.0, 121.4, 124.3, 124.9, 126.5, 127.9, 128.0, 128.4, 129.5, 129.7, 130.4, 134.8, 136.1, 144.0, 155.9, 156.6, 163.8. MS (EI) m/z: 414 (M^+, 2.3), 323 (M^+−Bn, 18), 306 (M^+−BnOH, 26), 250 (M^+−$CbzNHCH_2$, 21); IR ν (cm^{-1}): 1747, 1716. Calcd for $C_{25}H_{22}N_2O_4$: 414.1580. Found: 414.1587.

(±)−1−Benzyloxycarbonyl−2,3−diphenyl−tetrahydropyrimidin−4(1*H*)−one (8)



Colorless oil; ^{1}H NMR (300 MHz): δ 2.58 (m, 1H), 2.68 (m, 1H), 3.44 (m, 1H), 4.04 (m, 1H), 5.28 (s, 2H), 7.15 (s, 1H), 7.23−7.40 (m, 15H). ^{13}C NMR (75.5 MHz): δ 31.4, 37.7, 68.0, 72.4, 125.6, 126.0, 126.7, 128.0, 128.3, 128.5, 128.8, 129.0, 135.7, 138.0, 140.4, 155.2, 168.2. MS (EI) m/z: 386 (M^+, 3.8), 295 (M^+−Bn, 61), 91 (Bn^+, 100); IR ν (cm^{-1}): 1702, 1686. Calcd for $C_{24}H_{22}N_2O_3$: 386.1630. Found: 386.1635.

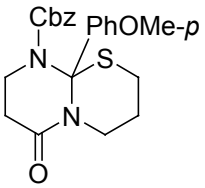
(±)−5−Benzyloxycarbonyl−6−phenyl−1,5−diaza−7−thia−bicyclo[4.4.0]decan−2−one (9a)



Colorless oil; ^{1}H NMR (300 MHz): δ 1.70 (m, 1H), 1.85 (m, 1H), 2.20 (m, 1H), 2.80 (m, 3H), 3.20 (ddd, J = 3.7, 12.1, 13.2 Hz, 1H), 3.88 (ddd, J = 3.7, 10.4, 13.2 Hz, 1H), 4.27 (dt, J = 13.2, 4.8 Hz, 1H), 4.53 (m, 1H), 4.86 (s, 2H), 7.07−7.10 (m, 2H), 7.27−7.39 (m, 6H), 7.65−7.68 (m, 2H). ^{13}C NMR (75.5 MHz): δ 24.5, 29.8, 33.1, 39.9, 40.0, 67.8, 85.6, 128.1, 128.2, 128.3, 128.4, 135.3, 140.4, 153.3, 167.3. MS (EI) m/z: 382 (M^+, 12), 291 (M^+−Bn, 4.8),

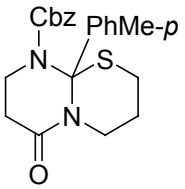
247 (9.6), 173 (4.2), 91 (Bn^+, 100); IR ν (cm^{-1}): 1700, 1651. Calcd for $C_{21}H_{22}N_2O_3S$: 382.1351. Found: 382.1352.

(±)−5−Benzyloxycarbonyl−6−(4−methoxyphenyl)−1,5−diaza−7−thia−bicyclo[4.4.0]decan−2−one (9b)



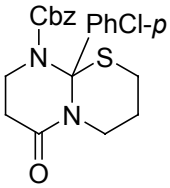
Colorless oil; ^{1}H NMR (300 MHz): δ 1.68 (m, 1H), 1.84 (m, 1H), 2.27 (m, 1H), 2.77 (m, 3H), 3.17 (ddd, J = 3.6, 12.3, 13.2 Hz, 1H), 3.81 (s, 3H), 3.88 (m, 1H), 4.25 (dt, J = 13.2, 4.8 Hz, 1H), 4.54 (dm, J = 13.8 Hz, 1H), 4.86 (d, J = 12.0 Hz, 1H), 4.92 (d, J = 12.0 Hz, 1H), 6.80 (d, J = 8.8 Hz, 2H), 7.11−7.13 (m, 2H), 7.27−7.31 (m, 3H), 7.56 (d, J = 8.8 Hz, 2H). ^{13}C NMR (75.5 MHz): δ 24.6, 29.8, 33.2, 39.9, 40.0, 55.2, 67.8, 85.4, 113.3, 128.1, 128.2, 128.3, 129.6, 132.3, 135.4, 153.3, 159.4, 167.3. MS (EI) m/z: 412 (M^+, 33), 367 (12), 321 (M^+−Bn, 20), 277 (27), 203 (6.0), 91 (100); IR ν (cm^{-1}): 1702, 1659. Calcd for $C_{22}H_{24}N_2O_4S$: 412.1457. Found: 412.1445.

(±)−5−Benzyloxycarbonyl−6−(4−methylphenyl)−1,5−diaza−7−thia−bicyclo[4.4.0]decan−2−one (9c)



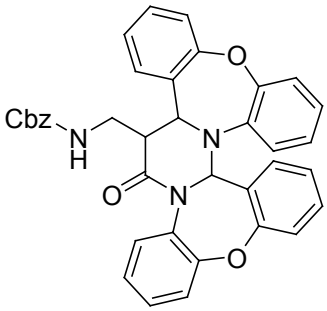
Colorless oil; ^{1}H NMR (300 MHz): δ 1.69 (m, 1H), 1.83 (m, 1H), 2.24 (m, 1H), 2.34 (s, 3H), 2.79 (m, 3H), 3.17 (ddd, J = 3.7, 12.2, 13.4 Hz, 1H), 3.87 (ddd, J = 3.7, 10.4, 13.4 Hz, 1H), 4.26 (dt, J = 13.2, 4.8 Hz, 1H), 4.54 (m, 1H), 4.86 (d, J = 12.0 Hz, 1H), 4.92 (d, J = 12.0 Hz, 1H), 7.07−7.12(m, 4H), 7.27−7.30 (m, 3H), 7.53 (d, J = 8.4 Hz, 2H). ^{13}C NMR (75.5 MHz): δ 21.0, 24.6, 29.8, 33.1, 39.8, 40.0, 67.8, 85.5, 128.0, 128.1, 128.2, 128.9, 135.3, 137.3, 138.2, 153.3, 167.3. MS (EI) m/z: 396 (M^+, 15), 351 (4.8), 305 (M^+−Bn, 6.8), 261 (11), 187 (4.7); IR ν (cm^{-1}): 1703, 1662. Calcd for $C_{22}H_{24}N_2O_3S$: 396.1508. Found: 396.1509.

(±)−5−Benzyloxycarbonyl−6−(4−chlorophenyl)−1,5−diaza−7−thia−bicyclo[4.4.0]decan−2−one (9d)



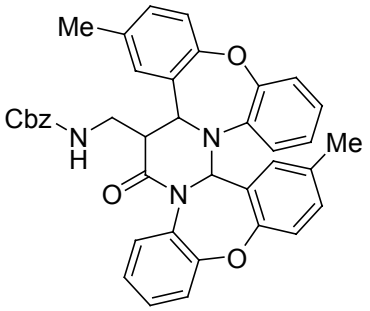
Colorless oil; ^{1}H NMR (400 MHz): δ 1.71 (m, 1H), 1.83 (m, 1H), 2.20 (m, 1H), 2.72 (m, 1H), 2.84 (m, 2H), 3.14 (ddd, J = 3.7, 12.1, 13.6 Hz, 1H), 3.87 (ddd, J = 3.7, 10.8, 13.3 Hz, 1H), 4.27 (dt, J = 13.0, 4.8 Hz, 1H), 4.53 (m, 1H), 4.83 (d, J = 12.0 Hz, 1H), 4.91 (d, J = 12.0 Hz, 1H), 7.07−7.09 (m, 2H), 7.21−7.32 (m, 5H), 7.55 (m, 2H). ^{13}C NMR (100 MHz): δ 24.4, 29.8, 33.1, 39.9, 40.0, 68.1, 85.1, 128.35, 128.40, 128.42, 129.65, 129.74, 134.3, 135.0, 139.1, 153.3, 167.2. MS (EI) m/z: 416 (M^+, 12), 371 (3.2), 281 (7.5), 211 (4.9); IR ν (cm^{-1}): 1700, 1650. Calcd for $C_{21}H_{21}ClN_2O_3S$: 416.0961. Found: 416.0962.

(±)−3−Benzyloxycarbonylaminomethyltetrabenzo[*e,h,l,o*]−7,14−dioxa−1,10−diaza−tricyclo[9.5.0.0^{4,10}]hexadecan−2−one (10a)



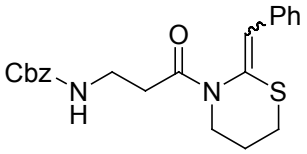
White solid; mp 250−251 ˚C; ^{1}H NMR (300 MHz): δ 3.22 (m, 1H), 3.40 (dt, J = 14.1, 4.8 Hz, 1H), 3.78 (dt, J = 11.3, 3.9 Hz, 1H), 4.60 (d, J = 11.3 Hz, 1H), 4.76 (d, J = 12.5 Hz, 1H), 4.96 (d, J = 12.5 Hz, 1H), 5.38 (t, J = 5.9 Hz, 1H), 6.44 (s, 1H), 6.86−7.35 (m, 20H), 7.55 (d, J = 7.8 Hz, 1H). ^{13}C NMR (75.5 MHz): δ 39.6, 48.3, 56.5, 66.4, 78.1, 119.7, 120.8, 121.6, 121.9, 122.6, 124.4, 125.3, 125.8, 126.1, 126.9, 127.5, 127.7, 127.8, 128.2, 128.4, 129.0, 129.6, 130.1, 130.6, 131.8, 132.8, 136.5, 139.2, 148.3, 153.8, 156.4, 156.6, 158.6, 170.4. MS (EI) m/z: 595 (M^+, 1.0), 487 (5.9), 431 (29), 309 (16), 292 (46), 195 (100); IR ν (cm^{-1}): 1716, 1651. Calcd for $C_{37}H_{29}N_3O_5$: 595.2107. Found: 595.2097.

(±)−3−Benzyloxycarbonylaminomethyl−2',2'''−dimethyl−tetrabenzo[*e,h,l,o*]−7,14−dioxa−1,10−diaza−tricyclo[9.5.0.0^{4,10}]hexadecan−2−one (10b)



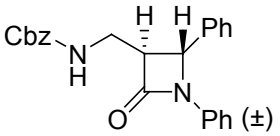
White solid; mp 135−137 ˚C; ^{1}H NMR (300 MHz): δ 2.29 (s, 6H), 3.21 (m, 1H), 3.40 (m, 1H), 3.80 (dt, J = 11.4, 4.5 Hz, 1H), 4.53 (d, J = 11.4 Hz, 1H), 4.84 (d, J = 12.6 Hz, 1H), 4.97 (d, J = 12.6 Hz, 1H), 5.51 (s, br, 1H), 6.40 (s, 1H), 6.85−7.32 (m, 19H). ^{13}C NMR (75.5 MHz): δ 20.7, 20.8, 39.7, 47.9, 56.7, 66.3, 78.1, 119.7, 120.5, 121.5, 121.7, 121.8, 122.6, 125.6, 126.0, 126.6, 127.5, 127.6, 127.7, 127.9, 128.3, 129.0, 129.5, 130.8, 131.0, 131.9, 132.9, 134.0, 134.9, 136.7, 139.2, 148.5, 154.1, 154.6, 156.3, 156.5, 170.6. MS (EI) m/z: 623 (M^+, 0.3), 515 (4.7), 459 (8.8), 306 (36), 250 (12); IR ν (cm^{-1}): 1717, 1656. Calcd for $C_{39}H_{33}N_3O_5$: 623.2420. Found: 623.2432.

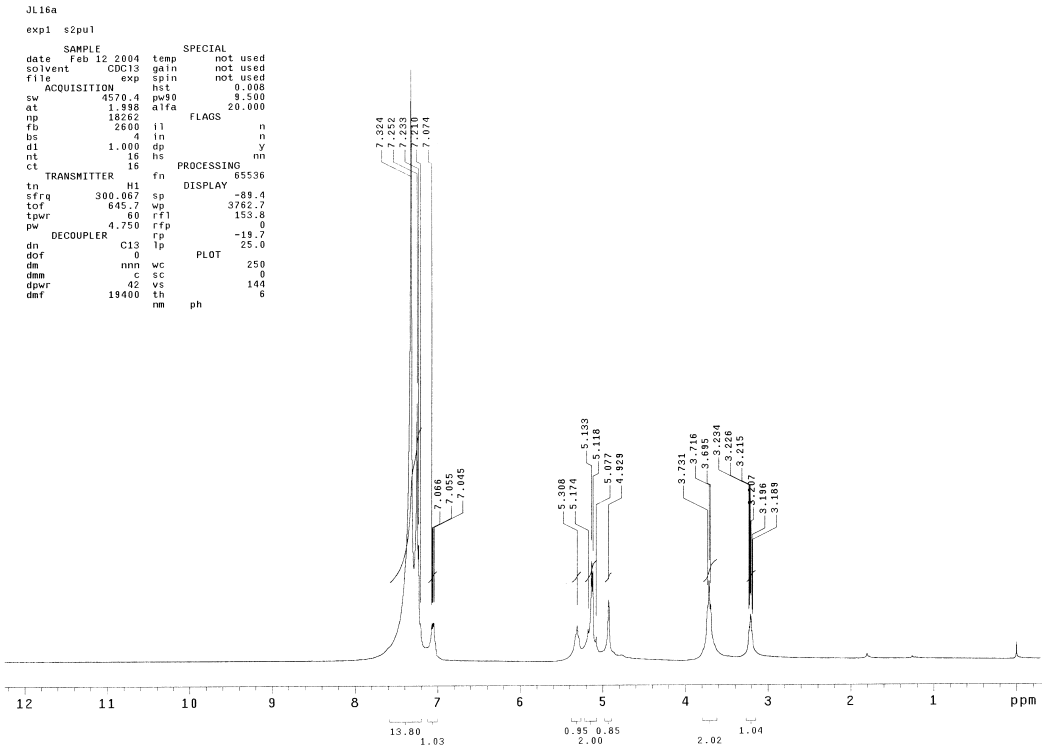
(±)−3−(3−Benzyloxycarbonylaminopropionyl)−2−benzylidene−1,3−thiazacyclohexane (11)

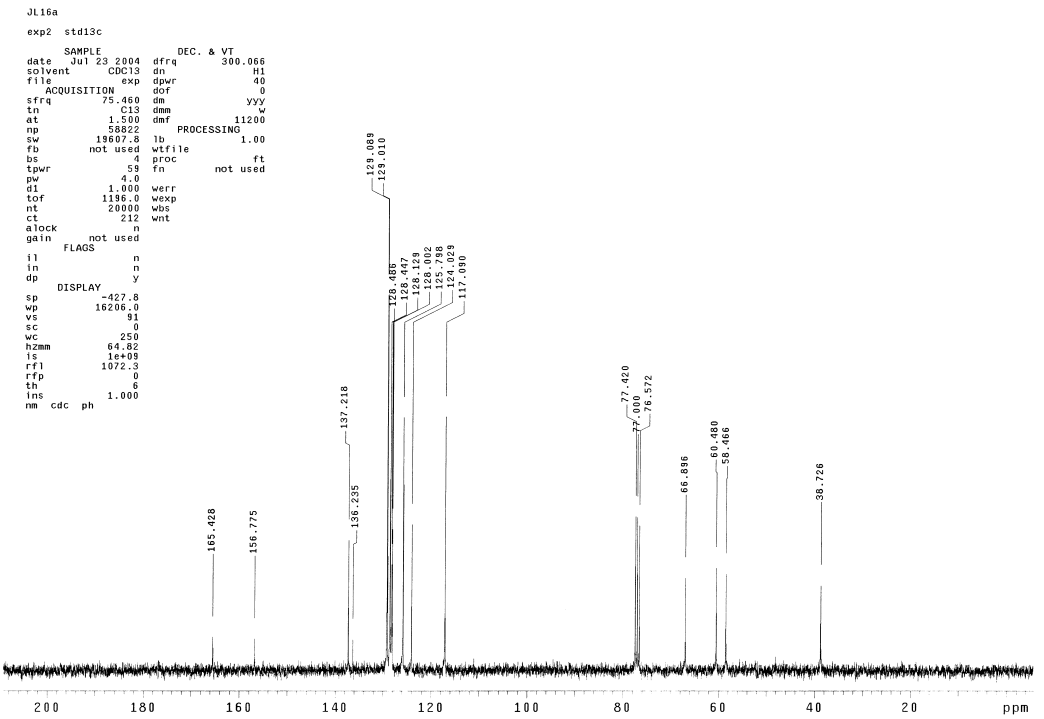


Colorless oil; ^{1}H NMR (300 MHz): δ 2.02 (quintet, J = 5.7 Hz, 2H), 2.73 (t, J = 5.7 Hz, 2H), 2.80 (t, J = 5.7 Hz, 2H), 3.50 (m, 2H), 3.85 (m, 2H), 5.08 (s, 2H), 5.57 (s, br, 1H), 6.61 (s, 1H), 7.25−7.47 (m, 10H). ^{13}C NMR (75.5 MHz): δ 24.6, 28.5, 33.8, 37.0, 44.4, 66.5, 127.9, 128.0, 128.3, 128.4, 129.1, 131.0, 133.4, 134.0, 136.6, 156.3, 171.4. MS (EI) m/z: 396 (M^+, 4.7), 305 (M^+−Bn, 3.6), 288 (17), 197 (22), 191 (100); IR ν (cm^{-1}): 1717, 1652. Calcd for $C_{22}H_{24}N_2O_3S$: 396.1508. Found: 396.1506.

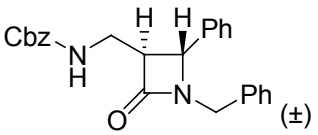
5a

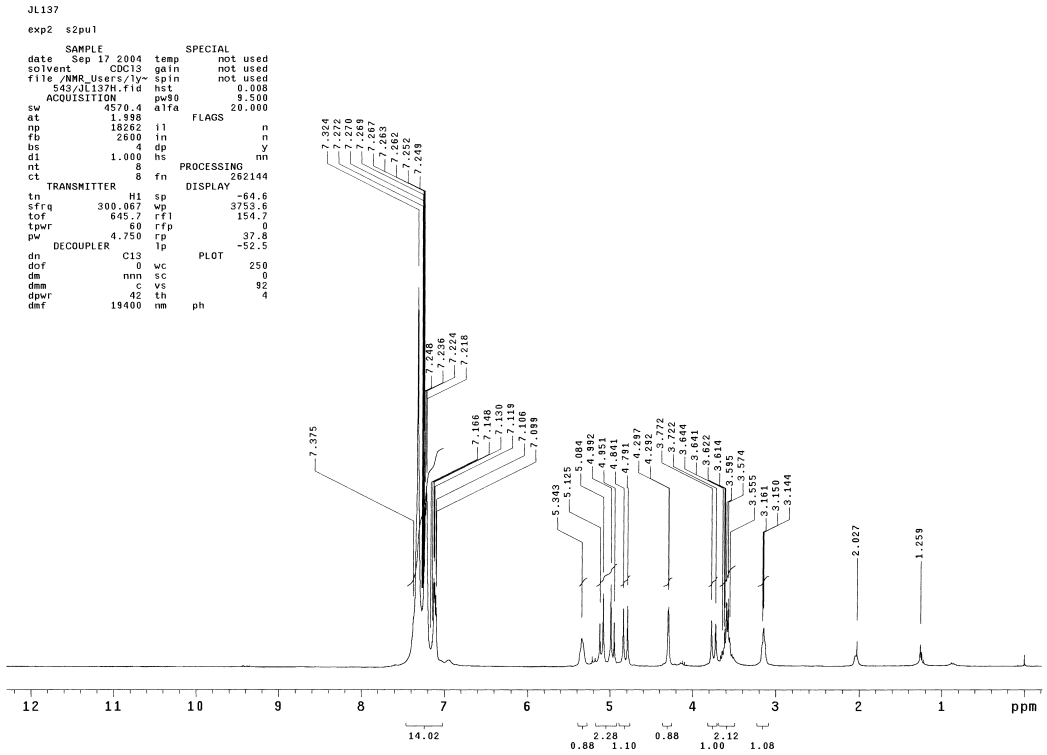

Cbz
N
H
H
H
Ph
N
O
Ph (±)

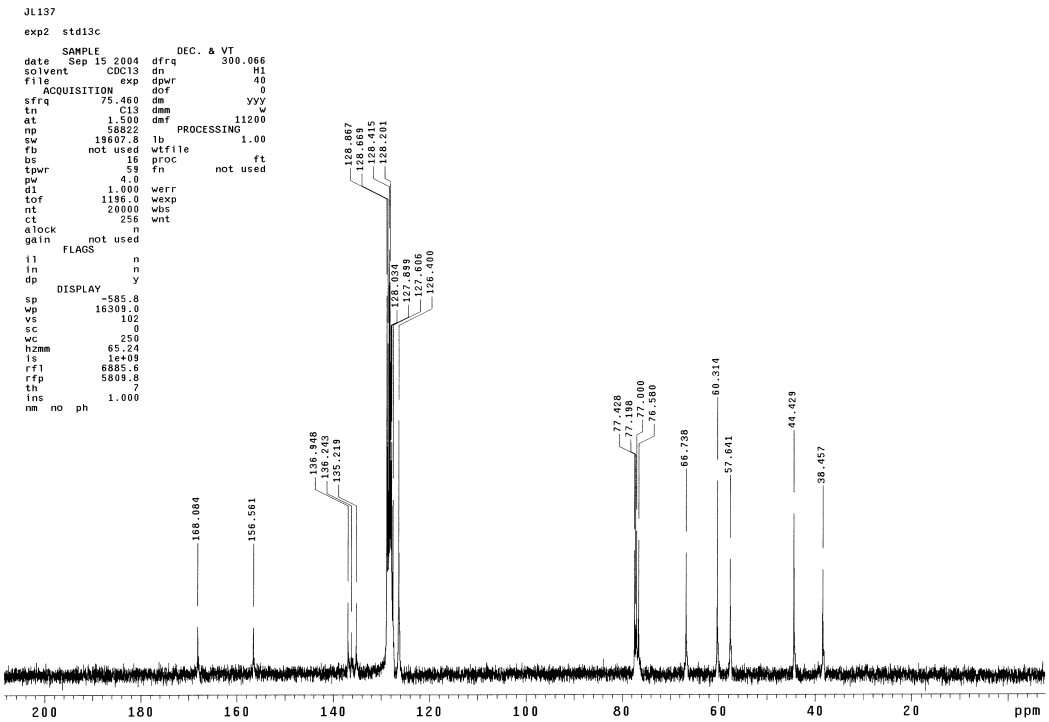

JL16a
exp1 s2pul
SAMPLE
date Feb 12 2004
solvent CDCl3
file exp
ACQUISITION
sw 4570.4
at 1.998
np 18262
fb 2600
bs 4
d1 1.000
nt 16
ct 16
TRANSMITTER
tn H1
sfrq 300.067
tof 645.7
tpwr 60
pw 4.750
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 42
dmf 19400
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 9.500
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn 65536
DISPLAY
sp -89.4
wp 3762.7
rf1 153.8
rfp 0
rp -19.7
lp 25.0
PLOT
wc 250
sc 0
vs 144
th 6
nm ph
7.324
7.252
7.233
7.210
7.074
7.066
7.055
7.045
5.308
5.174
5.133
5.118
5.077
4.928
3.731
3.716
3.695
3.294
3.226
3.215
3.207
3.196
3.189
12
11
10
9
8
7
6
5
4
3
2
1
ppm
13.80
1.03
0.95 0.85
2.00
2.02
1.04


JL16a
exp2 std13c
SAMPLE
date Jul 23 2004
solvent CDCl3
file exp
ACQUISITION
sfrq 75.460
tn C13
at 1.500
np 58822
sw 19607.8
fb not used
bs 4
tpwr 59
pw 4.0
d1 1.000
tof 1196.0
nt 20000
ct 212
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -427.8
wp 16206.0
vs 91
sc 0
wc 250
hzmm 64.82
is 1e+09
rf1 1072.3
rfp 0
th 6
ins 1.000
nm cdc ph
DEC. & VT
dfrq 300.066
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 11200
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
129.089
129.010
128.486
128.447
128.129
128.002
127.788
125.208
124.029
117.090
137.218
136.235
165.428
156.775
77.420
77.000
76.572
66.836
60.480
58.466
38.726
200
180
160
140
120
100
80
60
40
20
ppm

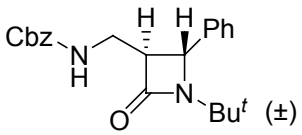
5b

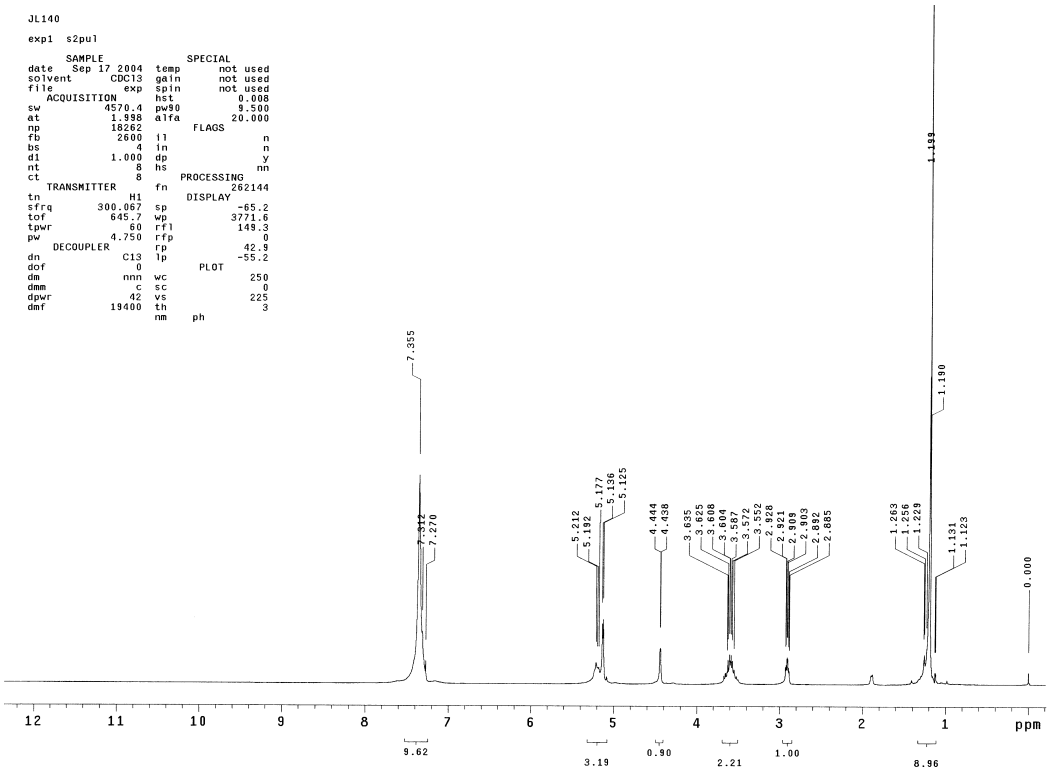

Cbz
N
H
H
H
Ph
N
Ph
O
(±)

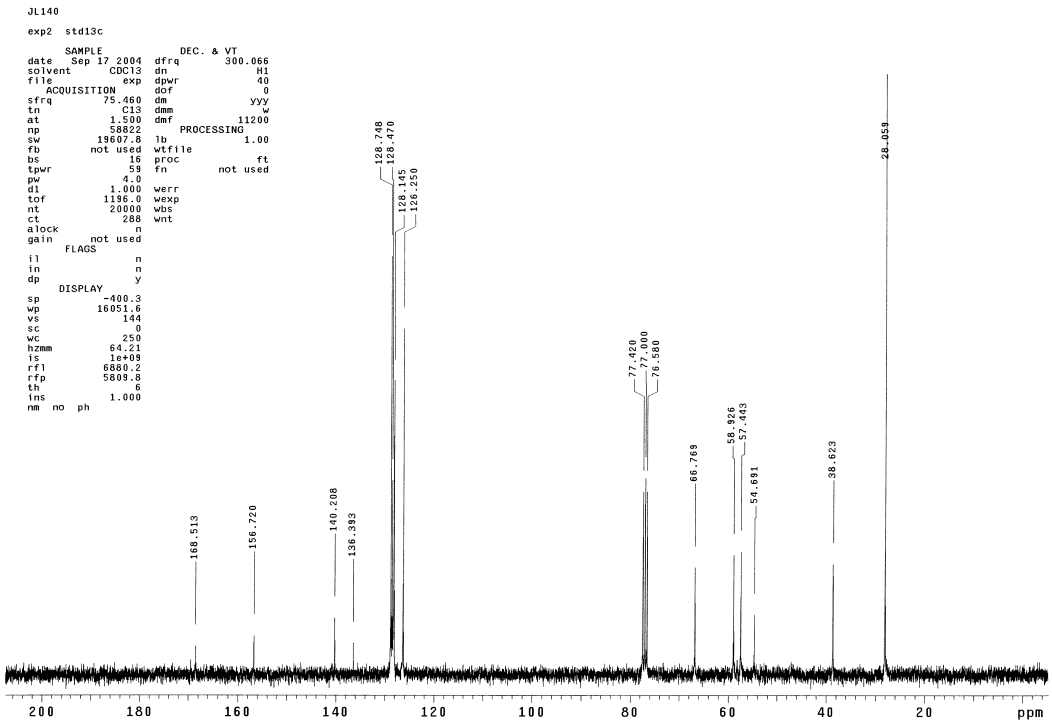

JL137
exp2 s2pul
SAMPLE
date Sep 17 2004
solvent CDCl3
file /NMR_Users/ly~543/JL137H.fid
ACQUISITION
sw 4570.4
at 1.998
np 18262
fb 2600
bs 4
d1 1.000
nt 8
ct 8
TRANSMITTER
tn H1
sfrq 300.067
tof 645.7
tpwr 60
pw 4.750
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 42
dmf 19400
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 9.500
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn 262144
DISPLAY
sp -64.6
wp 3753.6
rfl 154.7
rfp 0
rp 37.8
lp -52.5
PLOT
wc 250
sc 0
vs 92
th 4
nm ph
7.324
7.322
7.270
7.269
7.267
7.263
7.262
7.252
7.249
7.248
7.236
7.224
7.218
7.166
7.148
7.130
7.119
7.106
7.089
7.375
5.343
5.125
5.084
4.892
4.851
4.841
4.791
4.297
4.232
3.772
3.722
3.644
3.641
3.622
3.614
3.595
3.574
3.555
3.161
3.150
3.144
2.027
1.259
12 11 10 9 8 7 6 5 4 3 2 1 ppm
14.02
2.28
0.88 1.10
0.88
2.12
1.00 1.08


JL137
exp2 std13c
SAMPLE
date Sep 15 2004
solvent CDCl3
file exp
ACQUISITION
sfrq 75.460
tn C13
at 1.500
np 58822
sw 19607.8
fb not used
bs 16
tpwr 59
pw 4.0
d1 1.000
tof 1196.0
nt 20000
ct 256
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -585.8
wp 16309.0
vs 102
sc 0
wc 250
hzmm 65.24
is 1e+09
rfl 6885.6
rfp 5809.8
th 7
ins 1.000
nm no ph
DEC. & VT
dfrq 300.066
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 11200
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
128.867
128.669
128.415
128.201
128.034
127.899
127.606
126.400
136.848
136.243
135.219
168.084
156.561
77.428
77.198
77.000
76.580
66.738
60.314
57.641
44.429
38.457
200 180 160 140 120 100 80 60 40 20 ppm

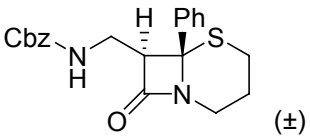
5c

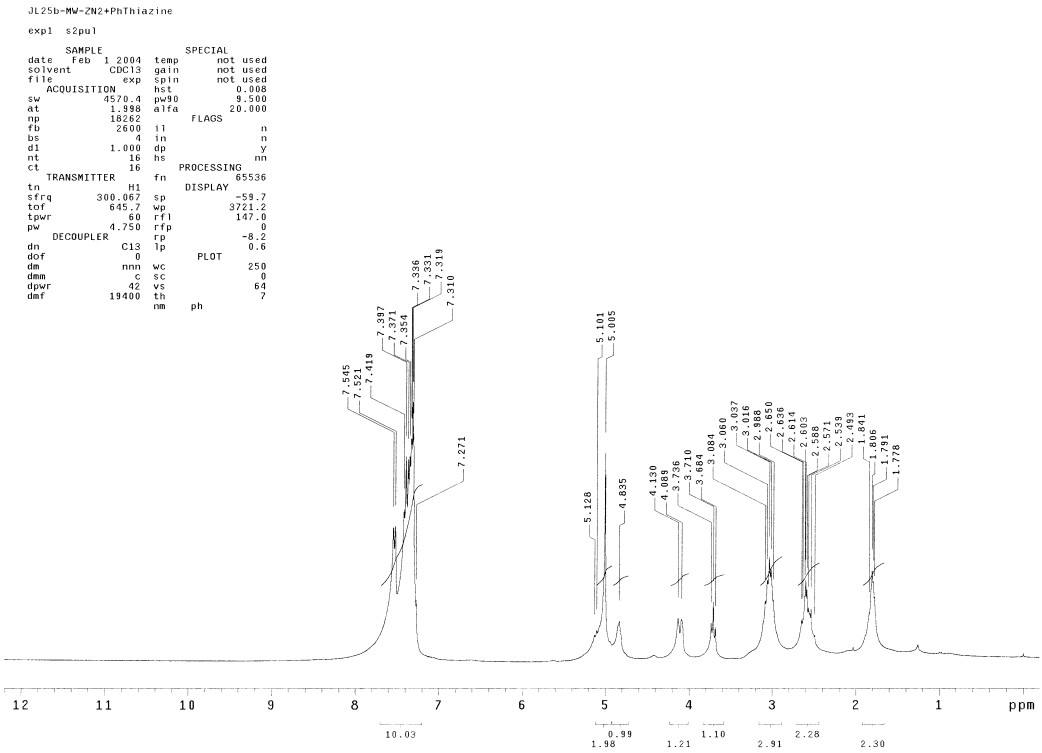

Cbz
N
H
H
H
Ph
N
Bu^t
O
(±)

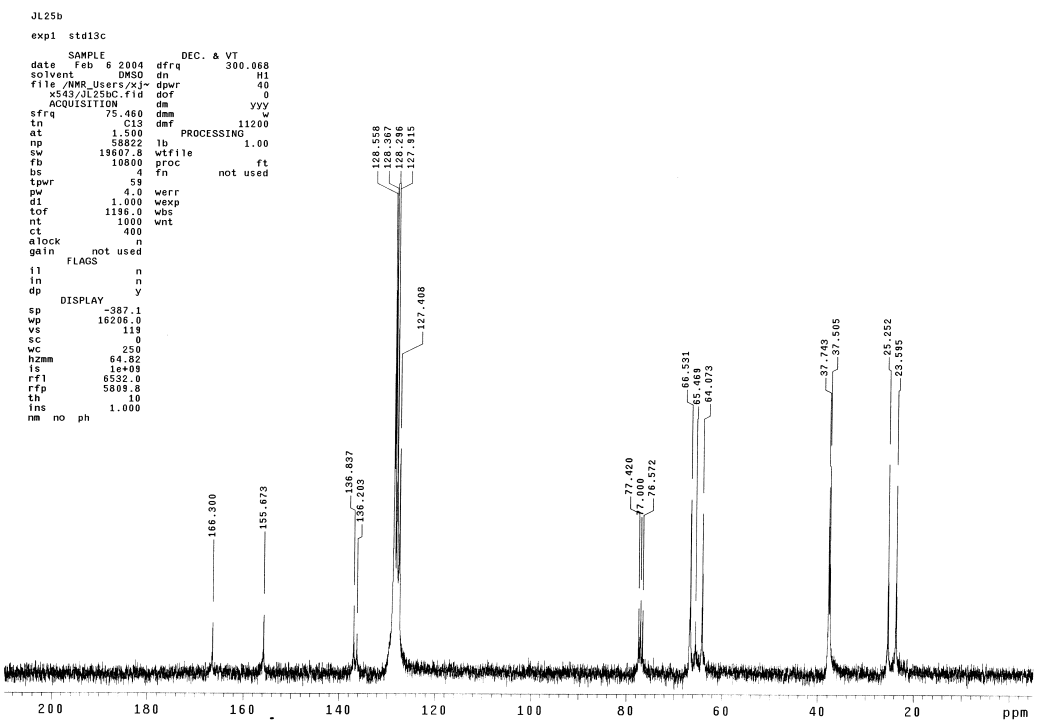

JL140
exp1 s2pul
SAMPLE
date Sep 17 2004
solvent CDCl3
file exp
ACQUISITION
sw 4570.4
at 1.998
np 18262
fb 2600
bs 4
d1 1.000
nt 8
ct 8
TRANSMITTER
tn H1
sfrq 300.067
tof 645.7
tpwr 60
pw 4.750
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 42
dmf 19400
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 9.500
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn 262144
DISPLAY
sp -65.2
wp 3771.6
rf1 149.3
rfp 0
rp 42.9
lp -55.2
PLOT
wc 250
sc 0
vs 225
th 3
nm ph
7.355
7.312
7.270
5.212
5.192
5.177
5.136
5.125
4.444
4.438
3.635
3.625
3.608
3.604
3.587
3.572
3.552
2.928
2.921
2.909
2.903
2.892
2.885
1.263
1.256
1.229
1.199
1.190
1.131
1.123
0.000
12 11 10 9 8 7 6 5 4 3 2 1 ppm
9.62
3.19
0.90
2.21
1.00
8.96


JL140
exp2 std13c
SAMPLE
date Sep 17 2004
solvent CDCl3
file exp
ACQUISITION
sfrq 75.460
tn C13
at 1.500
np 58822
sw 19607.8
fb not used
bs 16
tpwr 59
pw 4.0
d1 1.000
tof 1196.0
nt 20000
ct 288
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -400.3
wp 16051.6
vs 144
sc 0
wc 250
hzmm 64.21
is 1e+09
rf1 6880.2
rfp 5809.8
th 6
ins 1.000
nm no ph
DEC. & VT
dfrq 300.066
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 11200
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
128.748
128.470
128.145
126.250
77.420
77.000
76.580
66.769
58.926
57.443
54.691
38.623
28.059
168.513
156.720
140.208
136.393
200 180 160 140 120 100 80 60 40 20 ppm

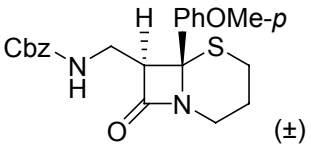
6a

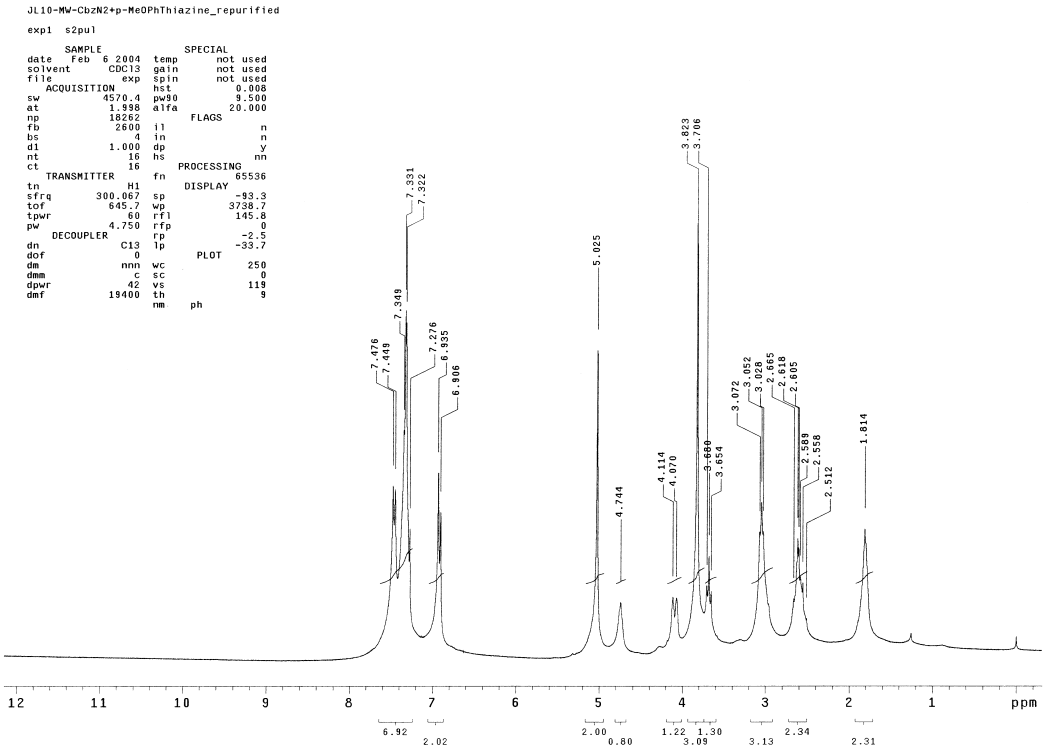

Cbz
N
H
H
Ph
S
N
O
(±)

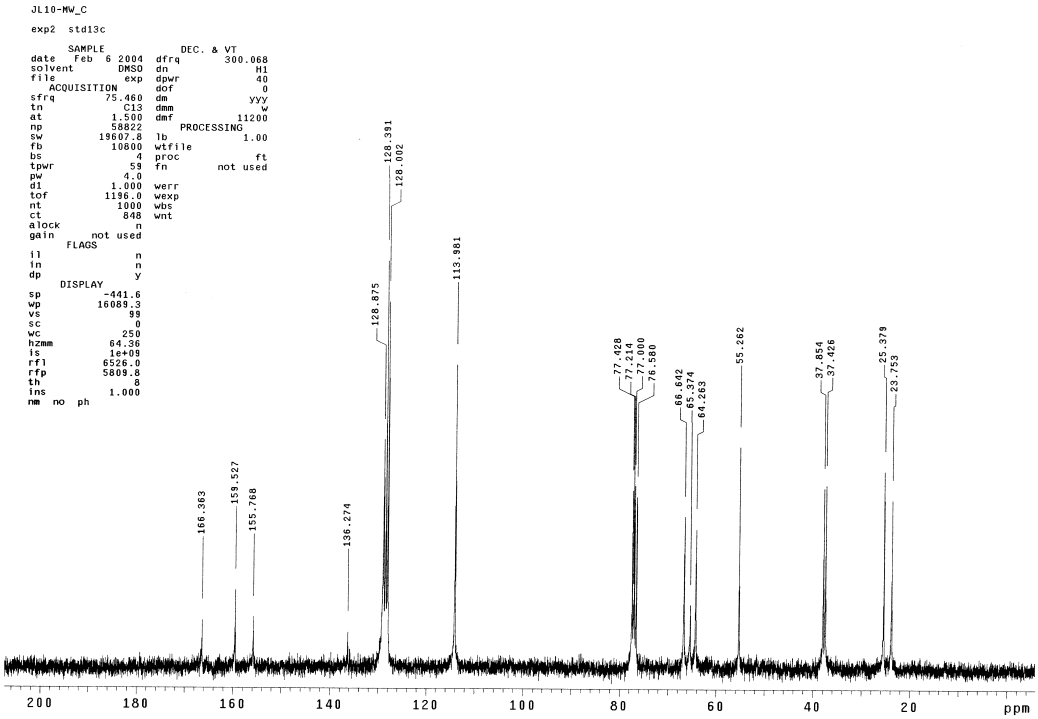

JL25b-MW-ZN2+PhThiazine
exp1 s2pul
SAMPLE
date Feb 1 2004
solvent CDCl3
file exp
ACQUISITION
sw 4570.4
at 1.998
np 18262
fb 2600
bs 4
d1 1.000
nt 16
ct 16
TRANSMITTER
tn H1
sfrq 300.067
tof 645.7
tpwr 60
pw 4.750
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 42
dmf 19400
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 9.500
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn 65536
DISPLAY
sp -59.7
wp 3721.2
rfl 147.0
rfp 0
rp -8.2
lp 0.6
PLOT
wc 250
sc 0
vs 64
th 7
nm ph
7.336
7.331
7.319
7.310
7.397
7.371
7.354
7.545
7.521
7.419
7.271
5.101
5.005
5.128
4.835
4.130
4.093
4.056
3.736
3.710
3.684
3.084
3.060
3.037
3.016
2.988
2.650
2.636
2.614
2.603
2.588
2.571
2.539
2.493
1.841
1.806
1.791
1.778
12
11
10
9
8
7
6
5
4
3
2
1
ppm
10.03
0.99
1.98
1.21
1.10
2.91
2.28
2.30


JL25b
exp1 std13c
SAMPLE
date Feb 6 2004
solvent DMSO
file /NMR_Users/xj~
x543/JL25bC.fid
ACQUISITION
sfrq 75.460
tn C13
at 1.500
np 58822
sw 18607.8
fb 10800
bs 4
tpwr 59
pw 4.0
d1 1.000
tof 1196.0
nt 1000
ct 400
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -387.1
wp 16206.0
vs 119
sc 0
wc 250
hzmm 64.82
is 1e+09
rfl 6532.0
rfp 5809.8
th 10
ins 1.000
nm no ph
DEC. & VT
dfrq 300.068
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 11200
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
128.558
128.387
128.216
127.915
127.408
166.300
155.673
136.837
136.203
77.420
77.000
76.572
66.531
65.469
64.073
37.743
37.505
25.252
23.595
200
180
160
140
120
100
80
60
40
20
ppm

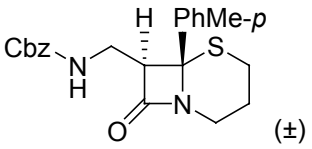
6b

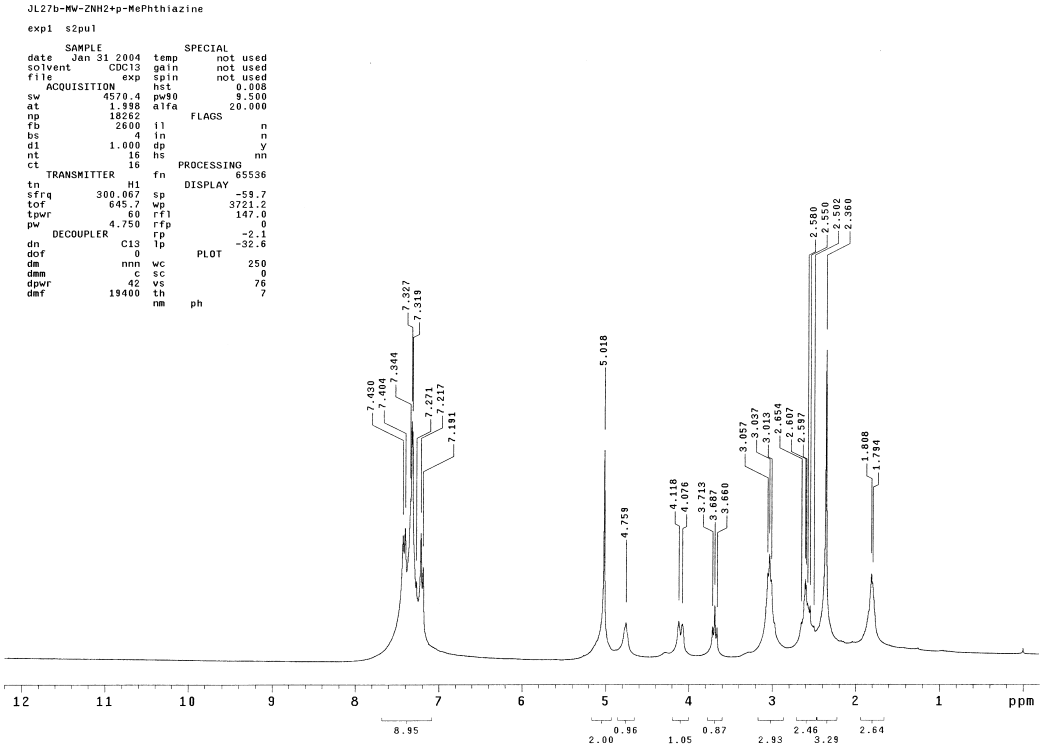

H
PhOMe-*p*
Cbz
N
H
S
N
O
(±)

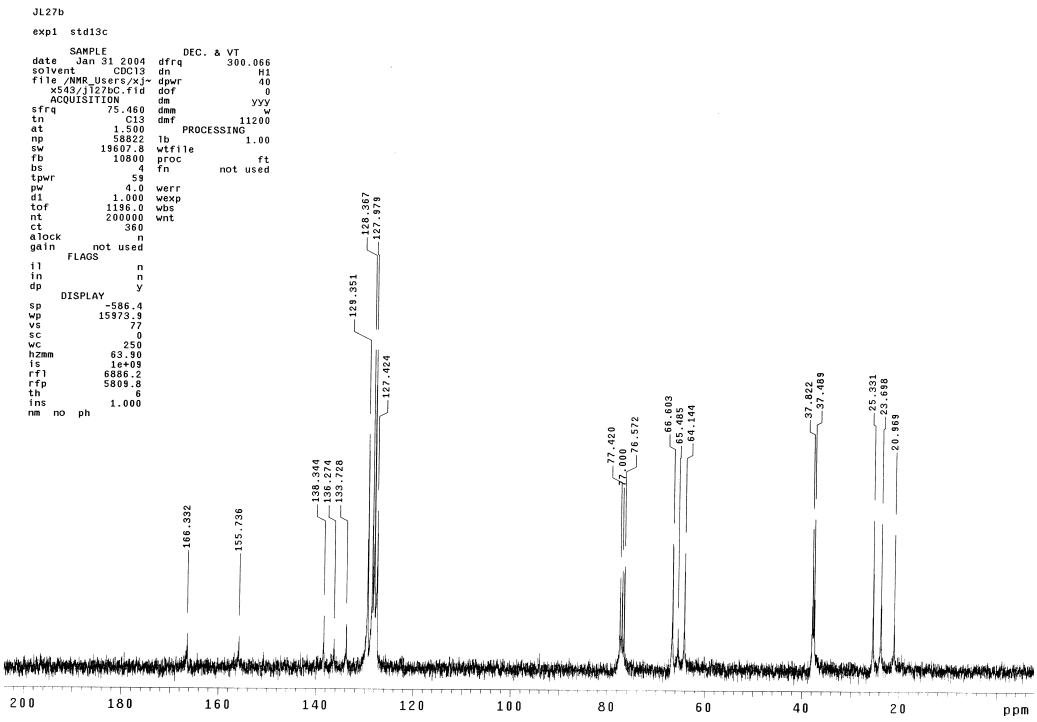

JL10-MW-CbzN2+p-MeOPhThiazine_repurified
exp1 s2pul
SAMPLE
date Feb 6 2004
solvent CDCl3
file exp
ACQUISITION
sw 4570.4
at 1.998
np 18262
fb 2600
bs 4
d1 1.000
nt 16
ct 16
TRANSMITTER
tn H1
sfrq 300.067
tof 645.7
tpwr 60
pw 4.750
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 42
dmf 19400
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 9.500
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn 65536
DISPLAY
sp -93.3
wp 3738.7
rfl 145.8
rfp 0
rp -2.5
lp -33.7
PLOT
wc 250
sc 0
vs 119
th 9
nm ph
7.476
7.449
7.349
7.331
7.322
7.276
6.935
6.906
5.025
4.744
4.114
4.070
3.823
3.706
3.660
3.654
3.072
3.052
3.028
2.665
2.618
2.605
2.589
2.558
2.512
1.814
12 11 10 9 8 7 6 5 4 3 2 1 ppm
6.92 2.02 2.00 0.80 1.22 1.30 3.09 3.13 2.34 2.31


JL10-MW_C
exp2 std13c
SAMPLE
date Feb 6 2004
solvent DMSO
file exp
ACQUISITION
sfrq 75.460
tn C13
at 1.500
np 58822
sw 19607.8
fb 10800
bs 4
tpwr 59
pw 4.0
d1 1.000
tof 1196.0
nt 1000
ct 648
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -441.6
wp 16089.3
vs 99
sc 0
wc 250
hzmm 64.36
is 1e+09
rfl 6526.0
rfp 5809.8
th 8
ins 1.000
nm no ph
DEC. & VT
dfrq 300.068
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 11200
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
128.391
128.002
128.875
113.981
77.428
77.214
77.000
76.580
66.642
65.374
64.263
55.262
37.854
37.426
25.379
23.753
166.363
159.527
155.768
136.274
200 180 160 140 120 100 80 60 40 20 ppm

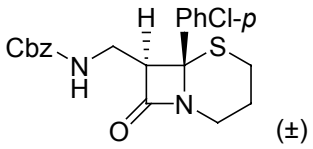
6c

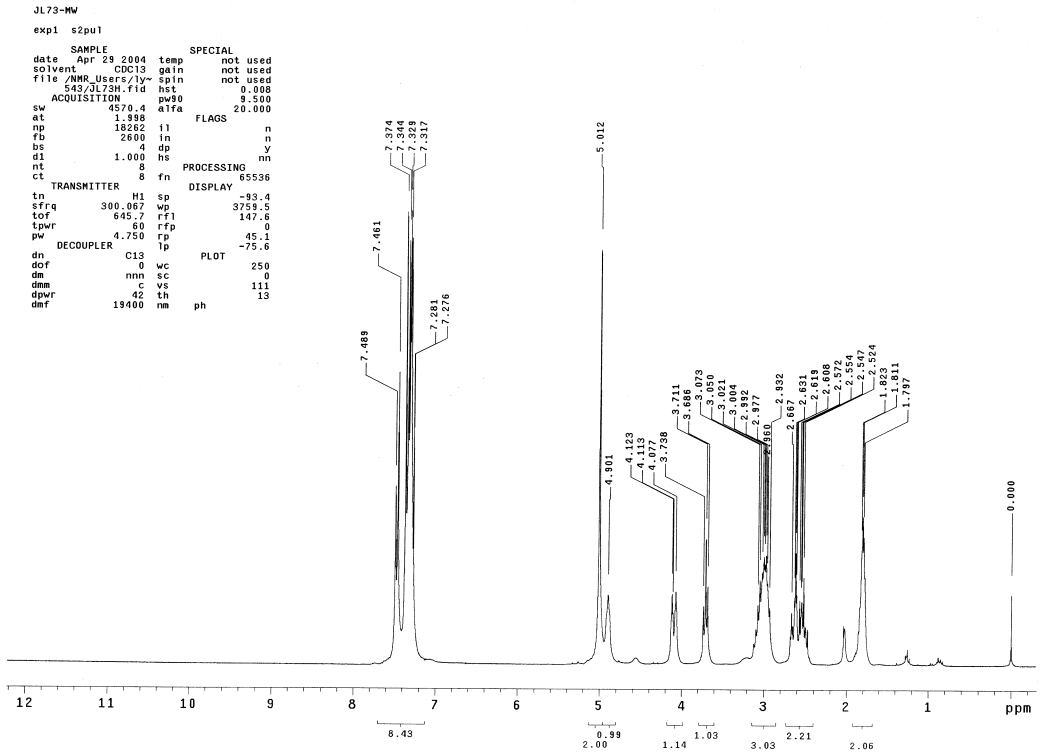

H
PhMe-p
Cbz
N
H
S
N
O
(±)


JL27b-MW-ZNH2+p-MePhthiazine
exp1 s2pul
SAMPLE
date Jan 31 2004
solvent CDCl3
file exp
ACQUISITION
sw 4570.4
at 1.998
np 18262
fb 2600
bs 4
d1 1.000
nt 16
ct 16
TRANSMITTER
tn H1
sfrq 300.067
tof 645.7
tpwr 60
pw 4.750
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 42
dmf 19400
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 9.500
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn 65536
DISPLAY
sp -59.7
wp 3721.2
rfl 147.0
rfp 0
rp -2.1
lp -32.6
PLOT
wc 250
sc 0
vs 76
th 7
nm ph
7.430
7.400
7.344
7.327
7.319
7.271
7.217
7.191
5.018
4.759
4.118
4.076
3.713
3.687
3.660
3.057
3.037
3.013
2.654
2.607
2.597
2.580
2.550
2.502
2.360
1.808
1.784
12
11
10
9
8
7
6
5
4
3
2
1
ppm
8.95
2.00
0.96
1.05
0.87
2.93
2.46
3.29
2.64


JL27b
exp1 std13c
SAMPLE
date Jan 31 2004
solvent CDCl3
file /NMR_Users/xj~
x543/jl27bC.fid
ACQUISITION
sfrq 75.460
tn C13
at 1.500
np 58822
sw 19607.8
fb 10800
bs 4
tpwr 59
pw 4.0
d1 1.000
tof 1196.0
nt 200000
ct 360
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -586.4
wp 15973.9
vs 77
sc 0
wc 250
hzmm 63.90
is 1e+09
rfl 6886.2
rfp 5809.8
th 6
ins 1.000
nm no ph
DEC. & VT
dfrq 300.066
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 11200
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
128.367
127.979
128.351
127.424
138.344
136.274
133.728
166.332
155.736
77.420
77.000
76.572
68.603
65.546
64.144
37.822
37.488
25.331
23.698
20.969
200
180
160
140
120
100
80
60
40
20
ppm

6d


H
PhCl-p
Cbz
N
H
S
N
O
(±)


JL73-MW
exp1 s2pul
SAMPLE
date Apr 29 2004
solvent CDCl3
file /NMR_Users/ly~
543/JL73H.fid
ACQUISITION
sw 4570.4
at 1.998
np 18262
fb 2600
bs 4
d1 1.000
nt 8
ct 8
TRANSMITTER
tn H1
sfrq 300.067
tof 645.7
tpwr 60
pw 4.750
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 42
dmf 19400
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 9.500
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn 65536
DISPLAY
sp -93.4
wp 3759.5
rf1 147.6
rfp 0
rp 45.1
lp -75.6
PLOT
wc 250
sc 0
vs 111
th 13
nm ph
7.374
7.344
7.329
7.317
7.461
7.489
7.281
7.276
5.012
4.901
4.123
4.119
4.077
3.738
3.711
3.686
3.073
3.050
3.021
3.004
2.992
2.977
2.960
2.932
2.667
2.631
2.619
2.608
2.575
2.564
2.547
2.524
1.823
1.811
1.797
0.000
12
11
10
9
8
7
6
5
4
3
2
1
ppm
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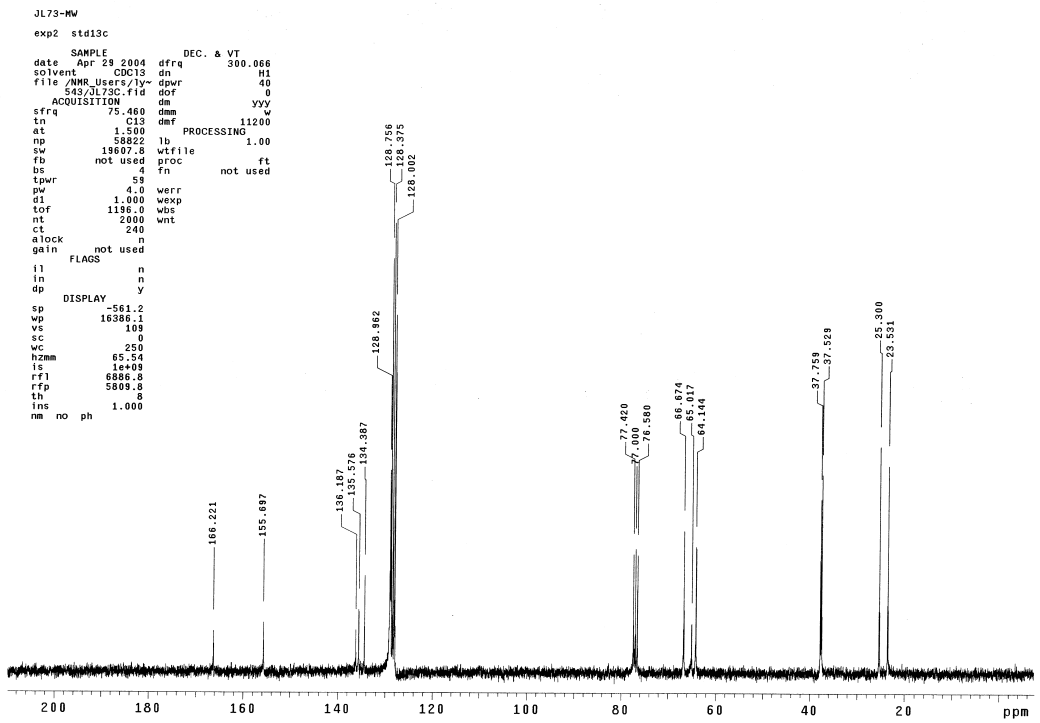
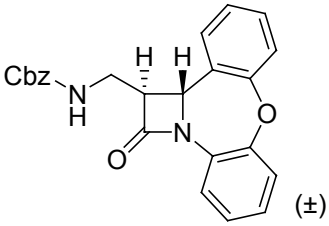
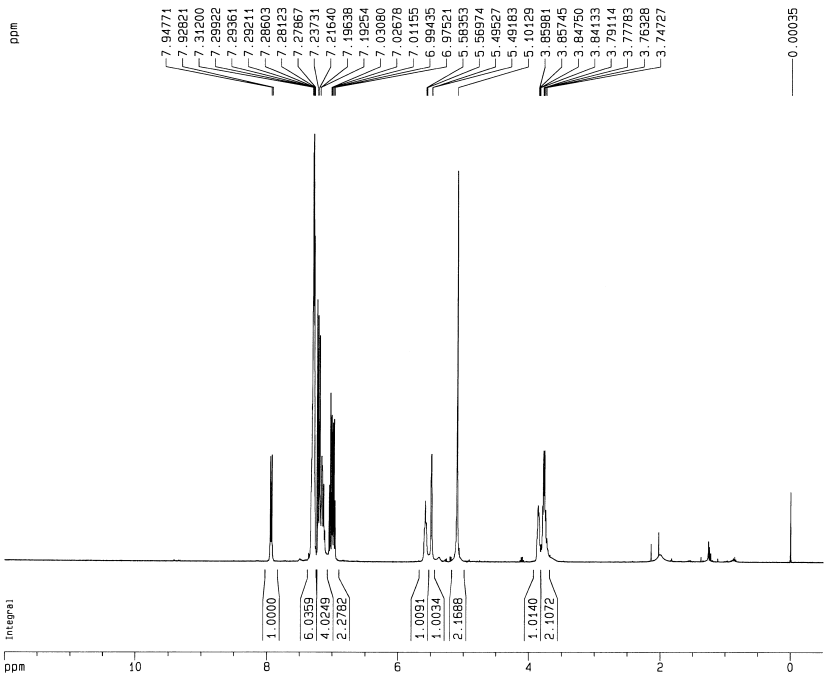
0.99
2.00
1.14
1.03
3.03
2.21
2.06


JL73-MW
exp2 std13c
SAMPLE
date Apr 29 2004
solvent CDCl3
file /NMR_Users/ly~
543/JL73C.fid
ACQUISITION
sfrq 75.460
tn C13
at 1.500
np 58822
sw 18607.8
fb not used
bs 4
tpwr 59
pw 4.0
d1 1.000
tof 1196.0
nt 2000
ct 240
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -561.2
wp 16386.1
vs 109
sc 0
wc 250
hzmm 65.54
is 1e+09
rf1 6886.8
rfp 5809.8
th 8
ins 1.000
nm no ph
DEC. & VT
dfrq 300.066
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 11200
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
128.756
128.375
128.002
128.962
166.221
155.687
136.187
135.576
134.387
77.420
77.000
76.580
66.674
65.017
64.144
37.759
37.529
25.300
23.531
200
180
160
140
120
100
80
60
40
20
ppm

7a



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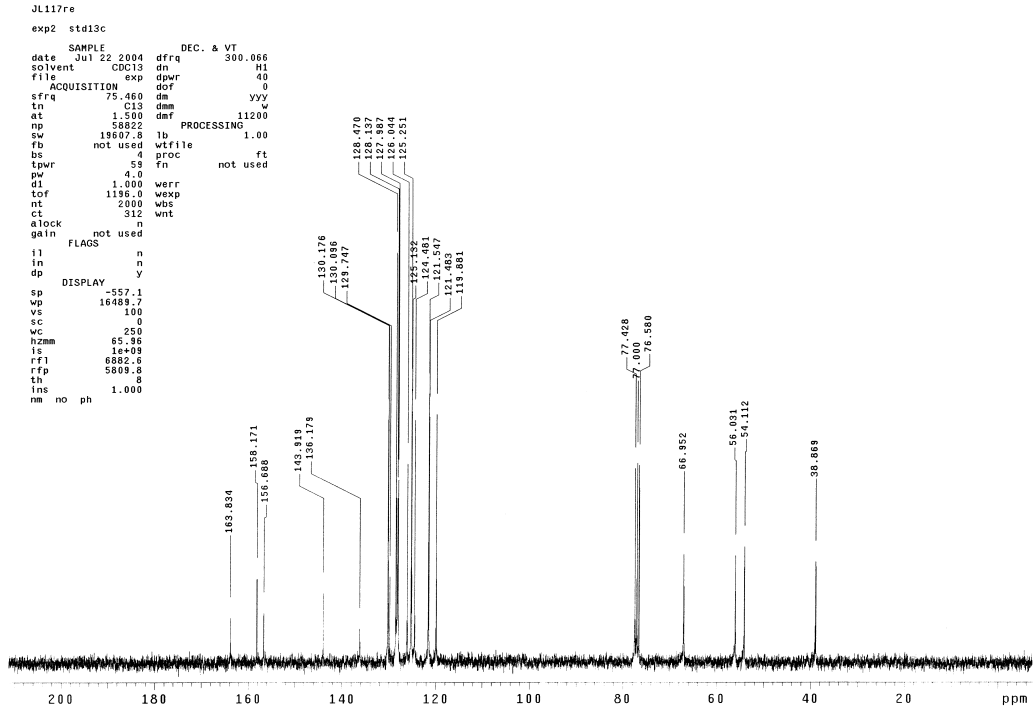
Current Data Parameters
NAME              H22441
EXPNO                  1
PROCNO                 1

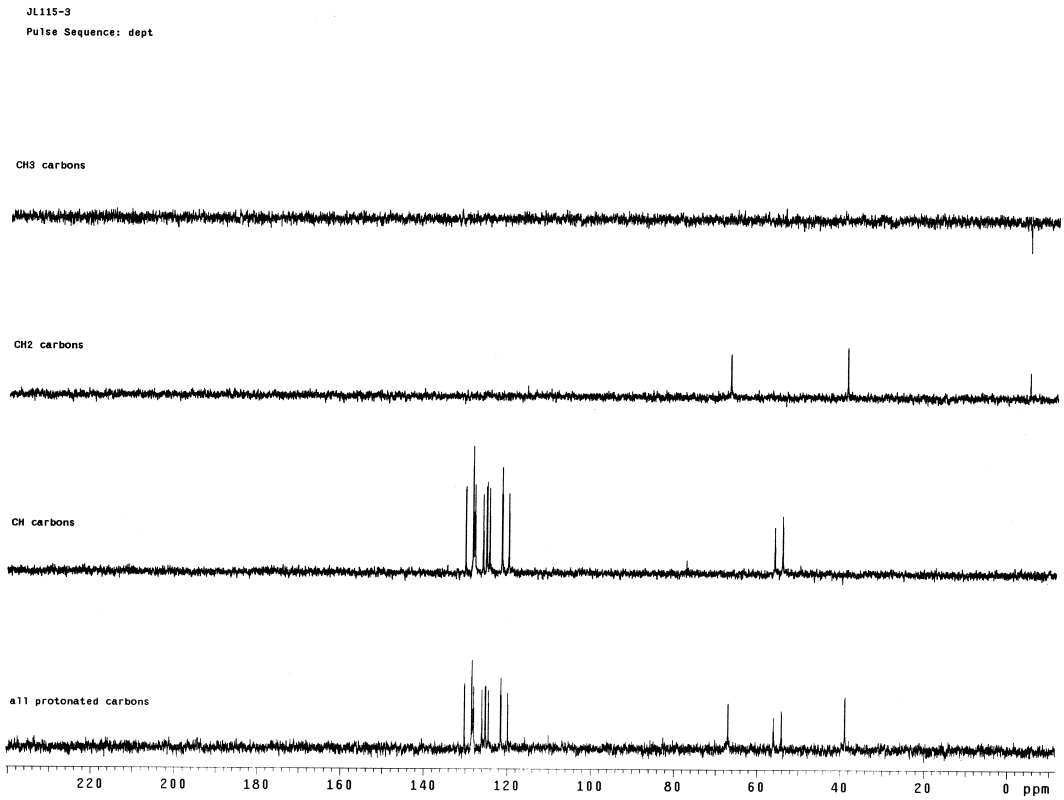
F2 - Acquisition Parameters
Date_           20040727
Time                8.34
INSTRUM           ARX400
PROBHD     5 mm Multinu
PULPROG               zg
TD                 32768
SOLVENT            CDCl3
NS                    54
DS                     0
SWH            6410.256 Hz
FIDRES         0.195625 Hz
AQ            2.5559540 sec
RG                   128
DW               78.000 usec
DE               111.43 usec
TE                300.0 K
D1           2.00000000 sec
P1                 2.00 usec
DE               111.43 usec
SFO1         400.1320000 MHz
NUCLEUS               1H

F2 - Processing parameters
SI                 32768
SF           400.1300183 MHz
WDW                   EM
SSB                    0
LB                 -0.30 Hz
GB                     0
PC                  0.50

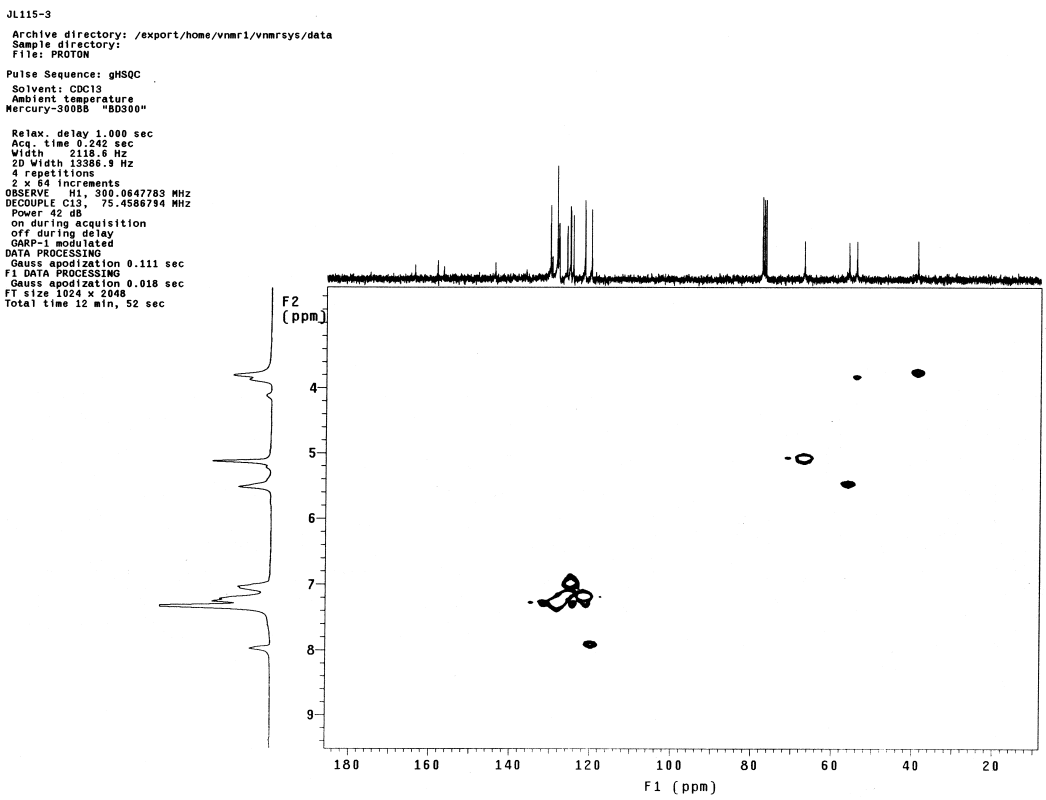
1D NMR plot parameters
CX                 20.00 cm
F1P               12.000 ppm
F1               4801.56 Hz
F2P               -0.500 ppm
F2               -200.07 Hz
PPMCM            0.62500 ppm/cm
HZCM           250.08125 Hz/cm
```



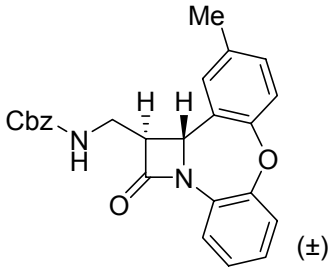
DEPT of **7a**

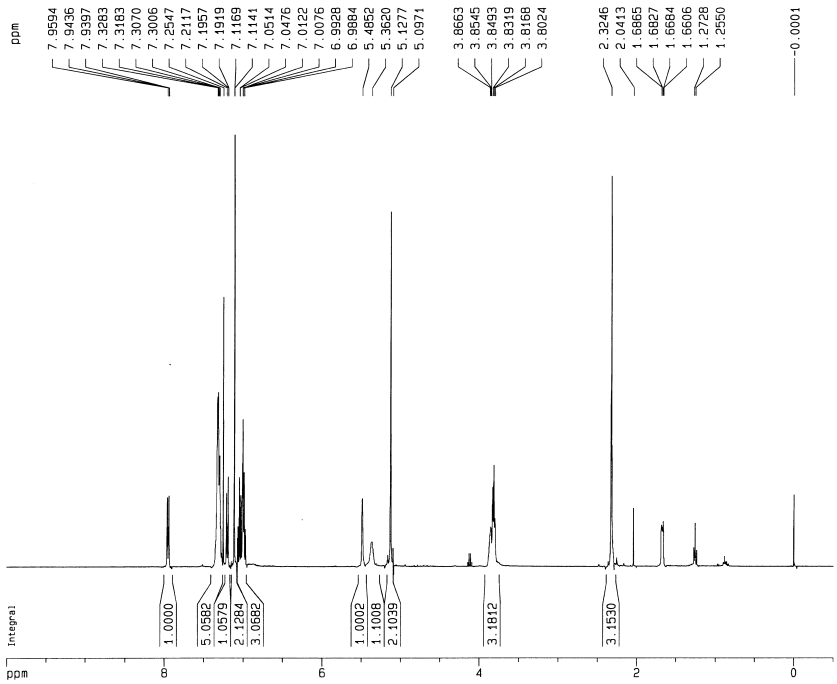


gHSQC of **7a**



7b



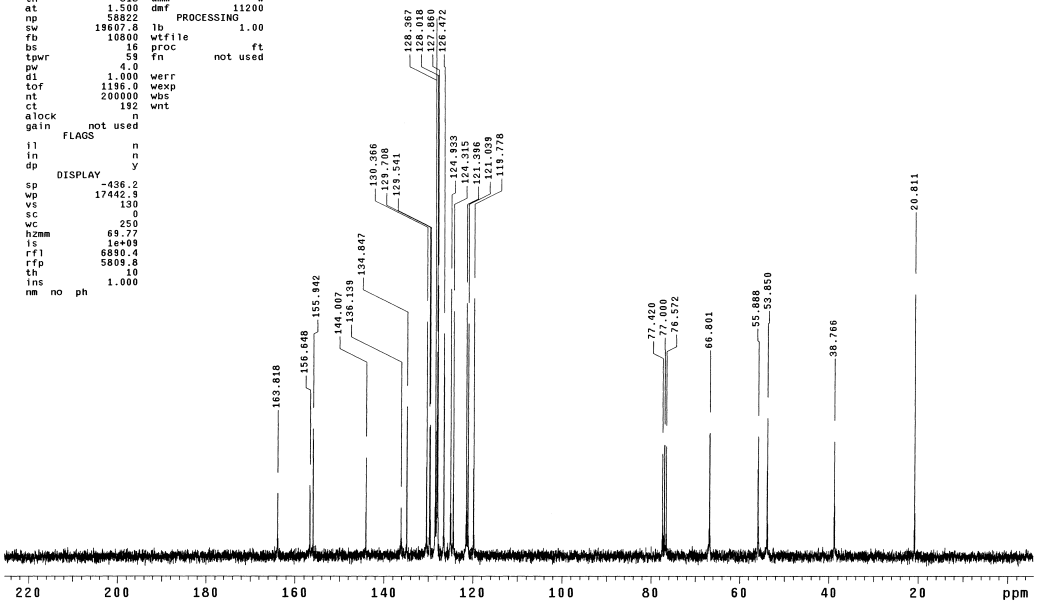



```
Current Data Parameters
NAME              H23712
EXPNO                  1
PROCNO                 1

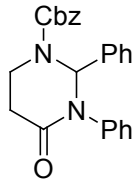
F2 - Acquisition Parameters
Date_           20040906
Time                8.16
INSTRUM           ARX400
PROBHD     5 mm Multinu
PULPROG              zg
TD                32768
SOLVENT           CDCl3
NS                   64
DS                    0
SWH            6410.256 Hz
FIDRES         0.195625 Hz
AQ           2.5559540 sec
RG                  715
DW               78.000 usec
DE               111.43 usec
TE                300.0 K
D1           2.00000000 sec
P1                2.00 usec
DE               111.43 usec
SFO1        400.1320000 MHz
NUCLEUS              1H

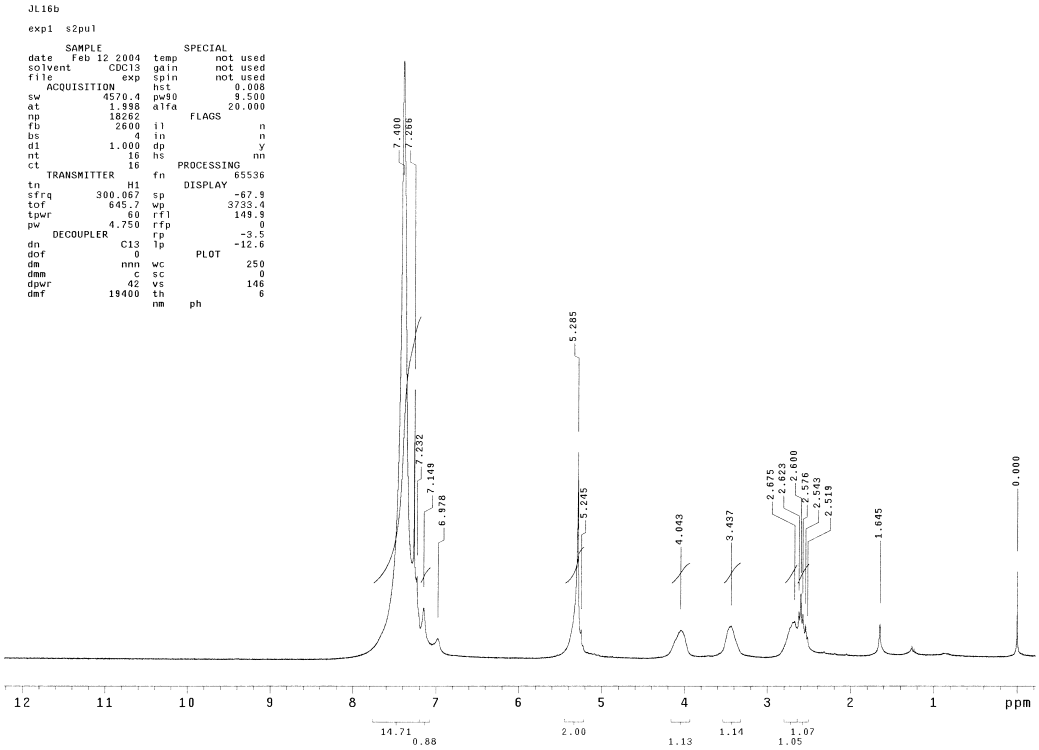
F2 - Processing parameters
SI                32768
SF          400.1300117 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 0.50

1D NMR plot parameters
CX                20.00 cm
F1P               10.000 ppm
F1               4001.30 Hz
F2P               -0.500 ppm
F2              -200.07 Hz
PPMCM            0.52500 ppm/cm
HZCM           210.06825 Hz/cm
```



8


Cbz
N
Ph
N
Ph
O


JL16b
exp1 s2pul
SAMPLE
date Feb 12 2004
solvent CDCl3
file exp
ACQUISITION
sw 4570.4
at 1.998
np 18262
fb 2600
bs 4
d1 1.000
nt 16
ct 16
TRANSMITTER
tn H1
sfrq 300.067
tof 645.7
tpwr 60
pw 4.750
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 42
dmf 19400
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 9.500
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn 65536
DISPLAY
sp -67.9
wp 3733.4
rfl 149.9
rfp 0
rp -3.5
lp -12.6
PLOT
wc 250
sc 0
vs 146
th 6
nm ph
7.400
7.266
7.232
7.149
6.978
5.285
5.245
4.043
3.437
2.675
2.623
2.600
2.575
2.543
2.519
1.645
0.000
ppm
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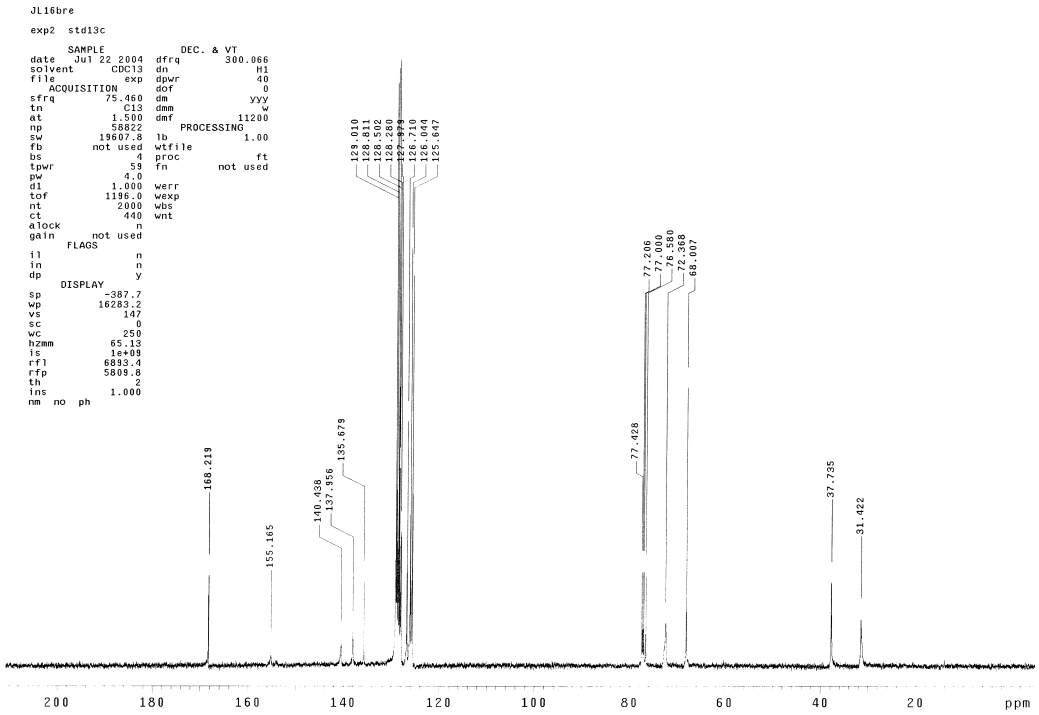
0.88
2.00
1.13
1.14
1.07
1.05


JL16bre
exp2 std13c
SAMPLE
date Jul 22 2004
solvent CDCl3
file exp
ACQUISITION
sfrq 75.460
tn C13
at 1.500
np 58822
sw 19607.8
fb not used
bs 4
tpwr 59
pw 4.0
d1 1.000
tof 1196.0
nt 2000
ct 440
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -387.7
wp 16283.2
vs 147
sc 0
wc 250
hzmm 65.13
is 1e+09
rfl 6893.4
rfp 5809.8
th 2
ins 1.000
nm no ph
DEC. & VT
dfrq 300.066
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 11200
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
129.010
128.811
128.502
128.280
128.279
128.119
126.711
126.044
125.647
168.219
155.165
140.438
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77.000
76.588
72.388
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77.428
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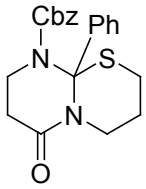
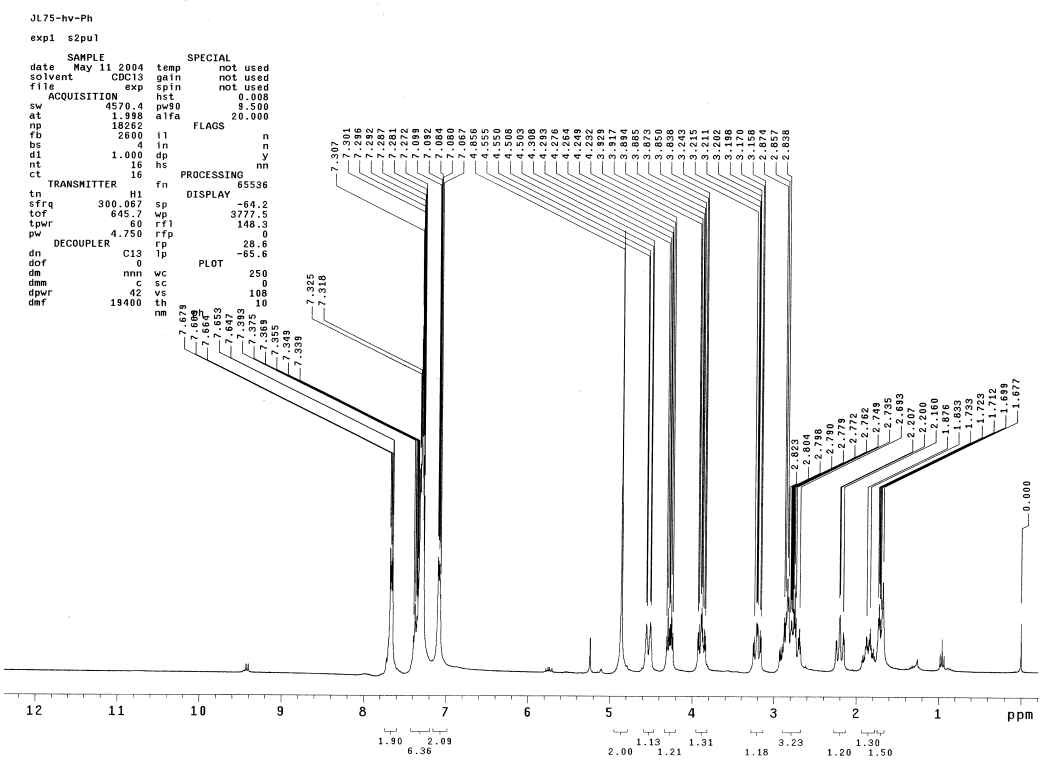
31.422
ppm

9a


Cbz
Ph
N
S
N
O


JL75-hv-Ph
exp1 s2pul
SAMPLE
date May 11 2004
solvent CDCl3
file exp
ACQUISITION
sw 4570.4
at 1.998
np 18262
fb 2600
bs 4
d1 1.000
nt 16
ct 16
TRANSMITTER
tn H1
sfrq 300.067
tof 645.7
tpwr 60
pw 4.750
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 42
dmf 19400
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 9.500
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn 65536
DISPLAY
sp -64.2
wp 3777.5
rfl 148.3
rfp 0
rp 28.6
lp -65.6
PLOT
wc 250
sc 0
vs 108
th 10
nm
7.307
7.301
7.296
7.292
7.287
7.281
7.272
7.099
7.092
7.084
7.080
7.067
4.856
4.555
4.550
4.506
4.503
4.308
4.293
4.276
4.264
4.249
4.232
3.929
3.917
3.884
3.865
3.873
3.850
3.838
3.243
3.215
3.211
3.202
3.198
3.170
3.158
2.874
2.857
2.838
7.325
7.318
7.679
7.674
7.664
7.653
7.647
7.393
7.375
7.369
7.355
7.349
7.339
2.823
2.804
2.798
2.790
2.779
2.772
2.762
2.749
2.735
2.693
2.207
2.200
2.160
1.878
1.853
1.733
1.723
1.712
1.699
1.677
0.000
12
11
10
9
8
7
6
5
4
3
2
1
ppm
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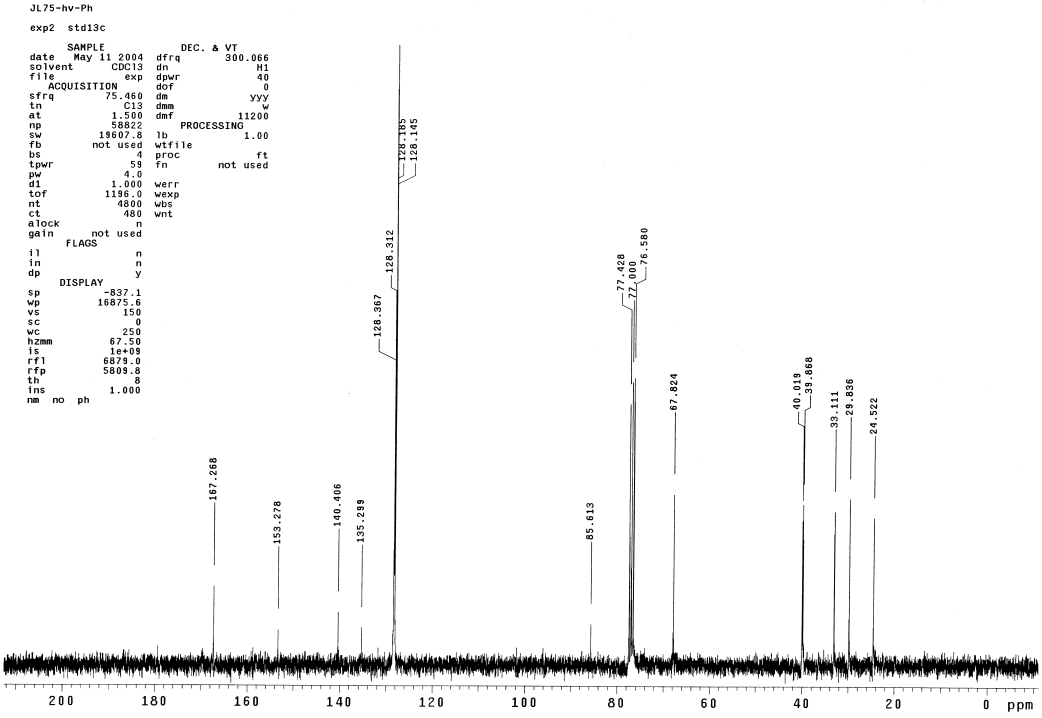
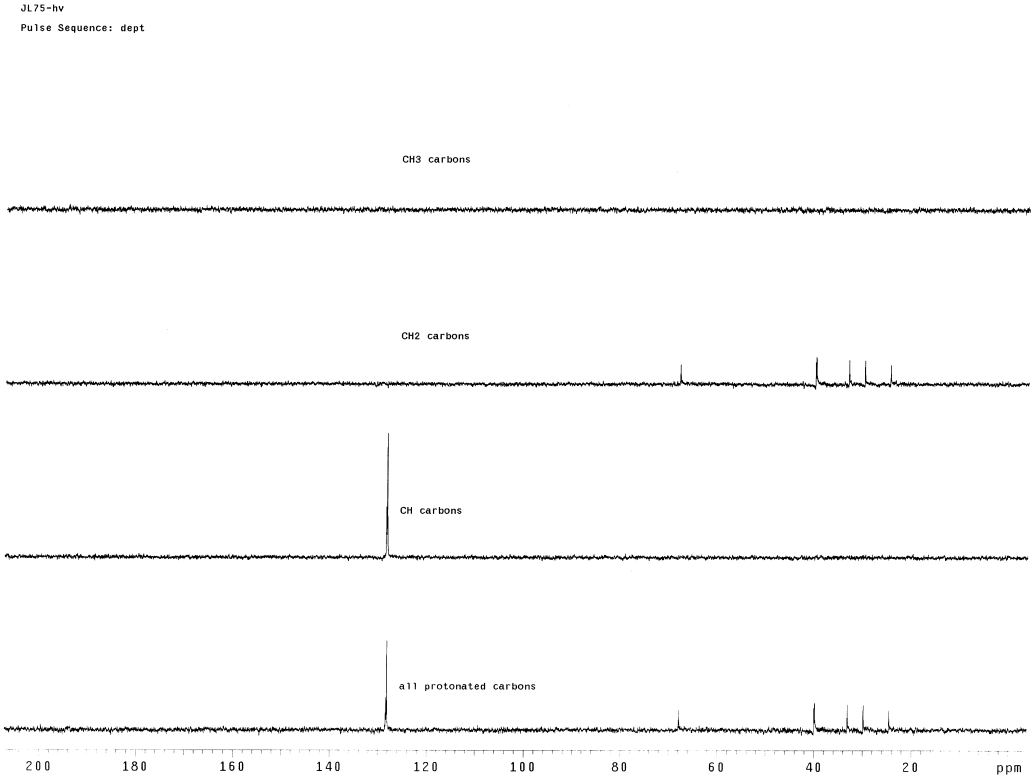
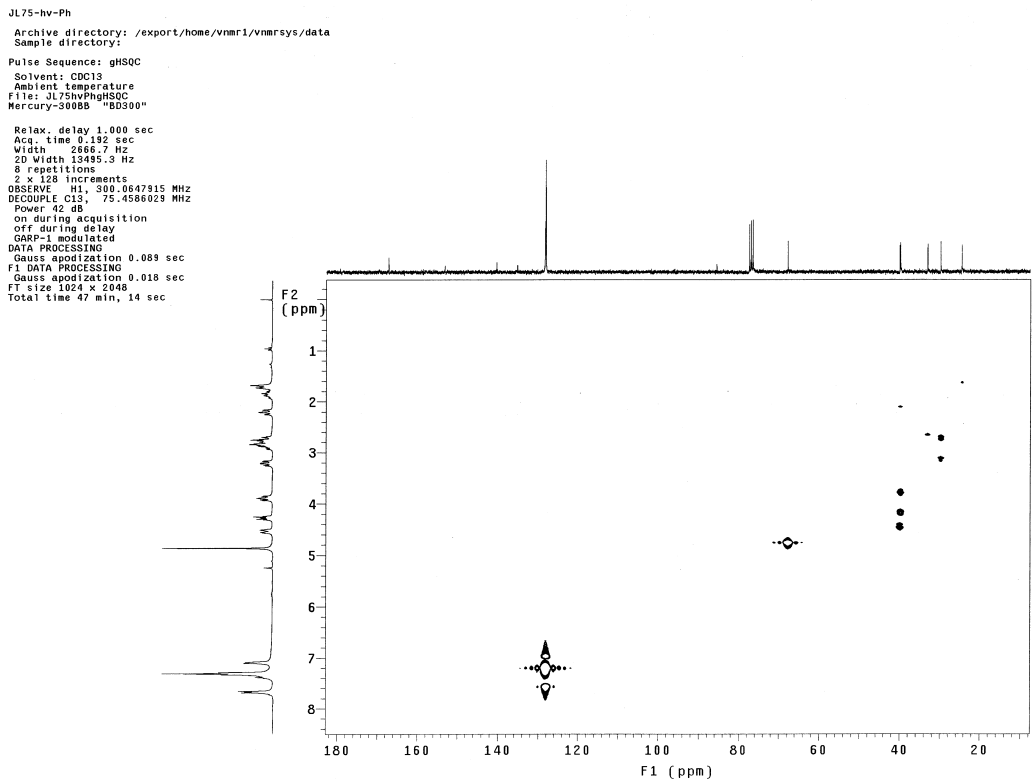
1.18
3.23
1.20
1.30
1.50


JL75-hv-Ph
exp2 std13c
SAMPLE
date May 11 2004
solvent CDCl3
file exp
ACQUISITION
sfrq 75.460
tn C13
at 1.500
np 58822
sw 19607.8
fb not used
bs 4
tpwr 59
pw 4.0
d1 1.000
tof 1196.0
nt 4800
ct 480
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -837.1
wp 16875.6
vs 150
sc 0
wc 250
hzmm 67.50
is 1e+09
rfl 6879.0
rfp 5809.8
th 8
ins 1.000
nm no ph
DEC. & VT
dfrq 300.066
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 11200
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
128.785
128.145
128.312
128.367
77.428
77.000
76.580
67.824
40.019
39.868
33.111
29.836
24.522
167.268
153.278
140.406
135.299
85.613
200
180
160
140
120
100
80
60
40
20
0
ppm

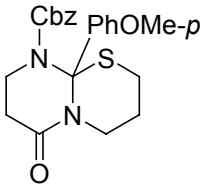
DEPT of **9a**

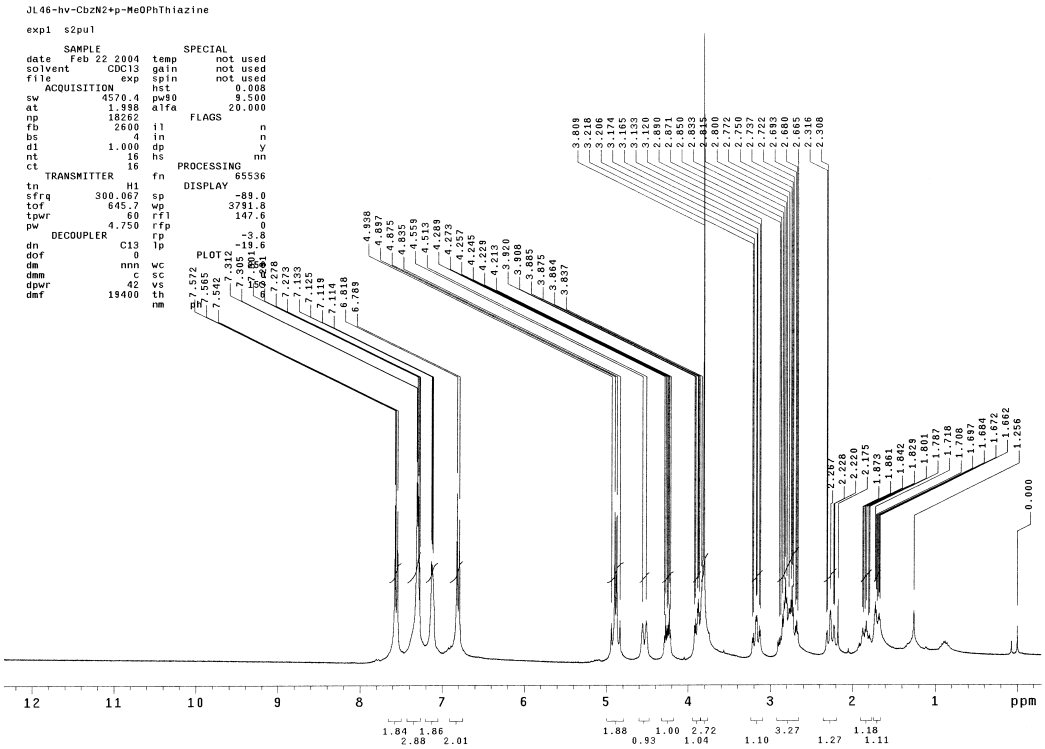


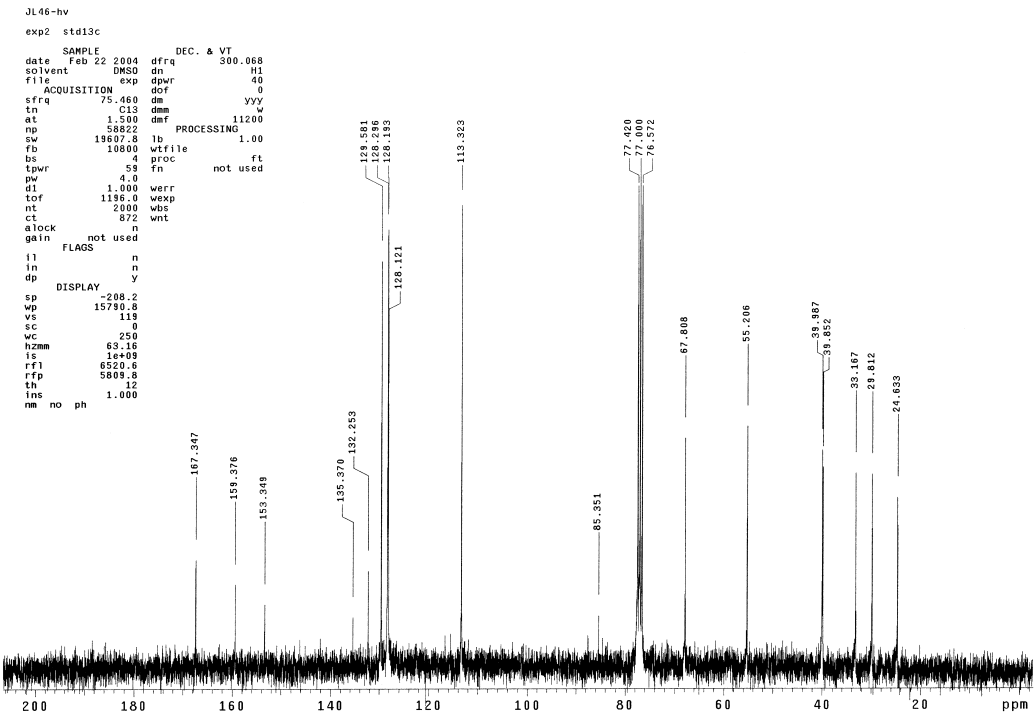
gHSQC of **9a**



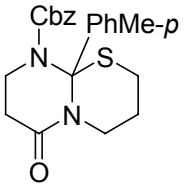
9b

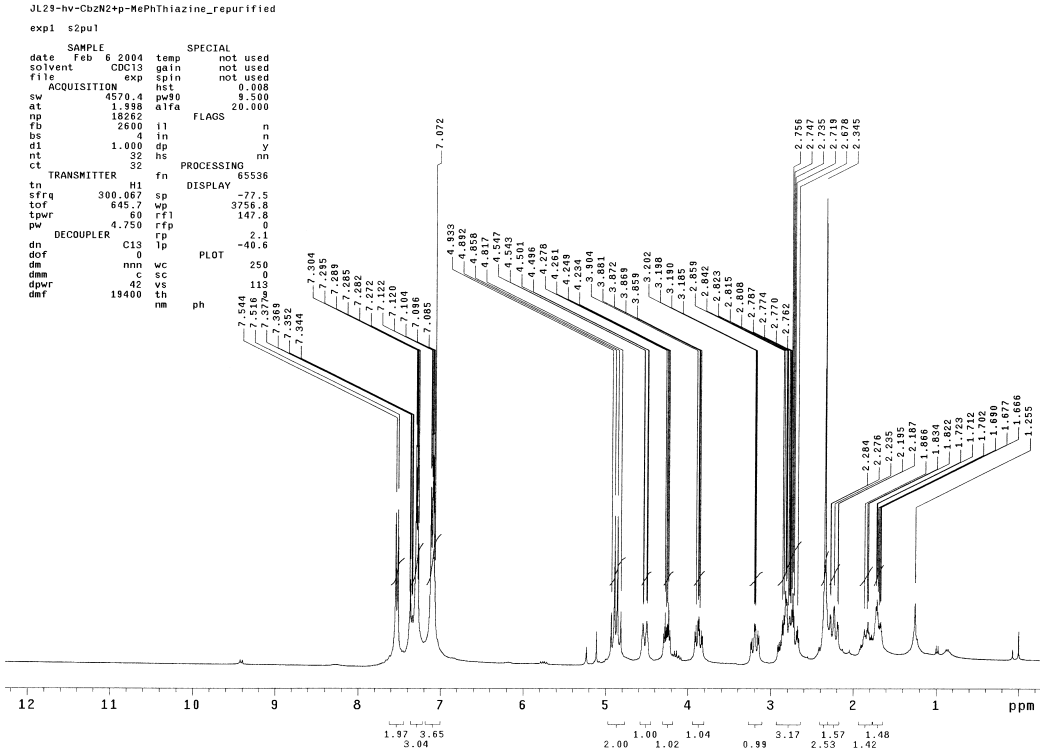

Cbz
PhOMe-p
N
S
N
O


JL46-hv-CbzN2+p-MeOPhThiazine
exp1 s2pul
SAMPLE
date Feb 22 2004
solvent CDCl3
file exp
ACQUISITION
sw 4570.4
at 1.998
np 18282
fb 2600
bs 4
d1 1.000
nt 16
ct 16
TRANSMITTER
tn H1
sfrq 300.067
tof 645.7
tpwr 60
pw 4.750
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 42
dmf 19400
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 9.500
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn 65536
DISPLAY
sp -89.0
wp 3791.8
rf1 147.6
rfp 0
rp -3.8
lp -19.6
PLOT
wc
sc
vs
th
nm
ppm


JL46-hv
exp2 std13c
SAMPLE
date Feb 22 2004
solvent DMSO
file exp
ACQUISITION
sfrq 75.460
tn C13
at 1.500
np 58822
sw 19607.8
fb 10800
bs 4
tpwr 59
pw 4.0
d1 1.000
tof 1196.0
nt 2000
ct 872
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -208.2
wp 15790.8
vs 119
sc 0
wc 250
hzmm 63.16
is 1e+09
rf1 6520.6
rfp 5809.8
th 12
ins 1.000
nm no ph
DEC. & VT
dfrq 300.068
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 11200
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
ppm

9c


Cbz
PhMe-p
N
S
N
O


JL29-hv-CbzN2+p-MePhThiazine_repurified
exp1 s2pul
SAMPLE
date Feb 6 2004
solvent CDCl3
file exp
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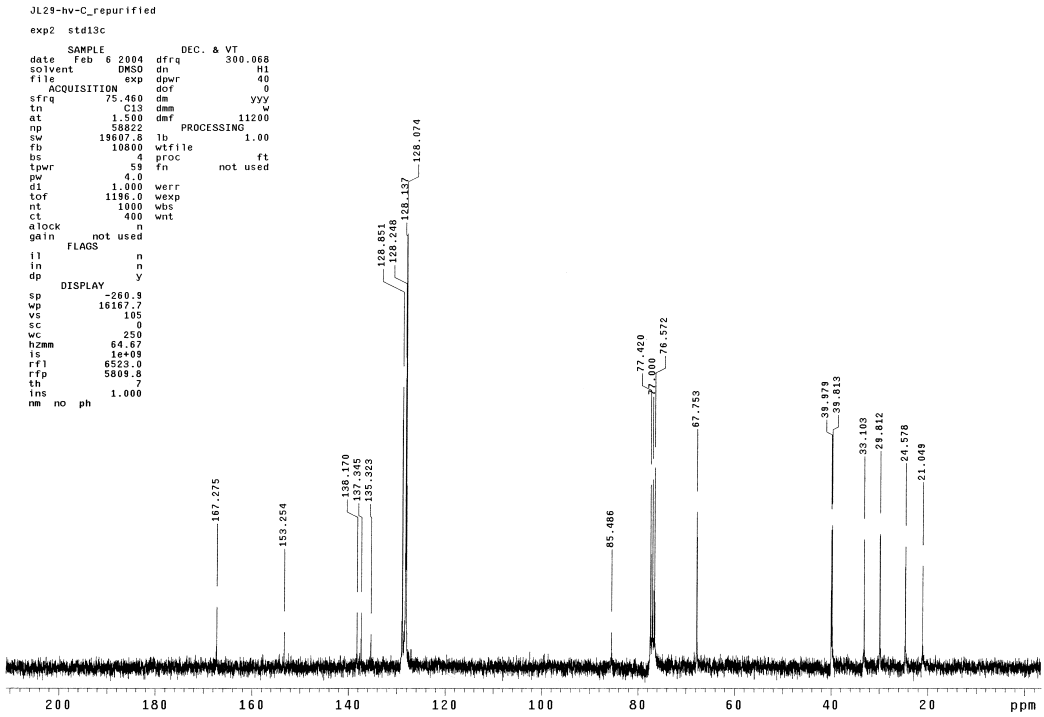
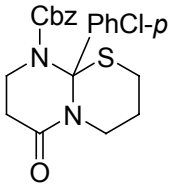
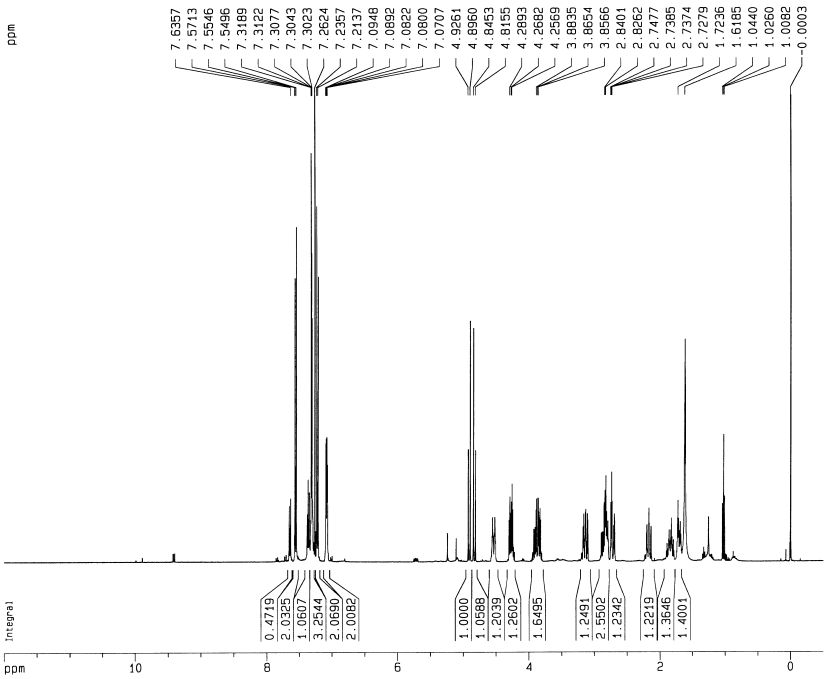
sw 4570.4
at 1.998
np 18262
fb 2600
bs 4
d1 1.000
nt 32
ct 32
TRANSMITTER
tn H1
sfrq 300.067
tof 645.7
tpwr 60
pw 4.750
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 42
dmf 19400
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 9.500
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn 65536
DISPLAY
sp -77.5
wp 3756.8
rf1 147.8
rfp 0
rp 2.1
lp -40.6
PLOT
wc 250
sc 0
vs 113
th 7
nm ph
7.072
4.933
4.892
4.858
4.817
4.547
4.543
4.501
4.496
4.278
4.261
4.248
4.234
3.904
3.881
3.872
3.865
3.859
3.202
3.198
3.190
3.185
3.159
3.159
2.842
2.823
2.815
2.808
2.787
2.774
2.770
2.762
2.756
2.747
2.735
2.729
2.678
2.345
7.544
7.516
7.377
7.369
7.352
7.344
7.304
7.295
7.289
7.285
7.282
7.272
7.122
7.120
7.104
7.096
7.085
2.284
2.276
2.235
2.195
2.187
1.866
1.834
1.822
1.723
1.712
1.702
1.690
1.677
1.666
1.255
12 11 10 9 8 7 6 5 4 3 2 1 ppm
1.97 3.65
3.04
2.00 1.00 1.02 1.04
0.99 3.17 1.57 1.48
2.53 1.42


JL29-hv-C_repurified
exp2 std13c
SAMPLE
date Feb 6 2004
solvent DMSO
file exp
ACQUISITION
sfrq 75.460
tn C13
at 1.500
np 58822
sw 19607.8
fb 10800
bs 4
tpwr 59
pw 4.0
d1 1.000
tof 1196.0
nt 1000
ct 400
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -260.9
wp 16167.7
vs 105
sc 0
wc 250
hzmm 64.67
is 1e+09
rf1 6523.0
rfp 5809.8
th 7
ins 1.000
nm no ph
DEC. & VT
dfrq 300.068
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 11200
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
128.074
128.137
128.851
128.248
77.420
77.000
76.572
67.753
39.979
39.813
33.103
29.812
24.578
21.049
167.275
153.254
130.170
132.345
135.323
85.486
200 180 160 140 120 100 80 60 40 20 ppm

9d



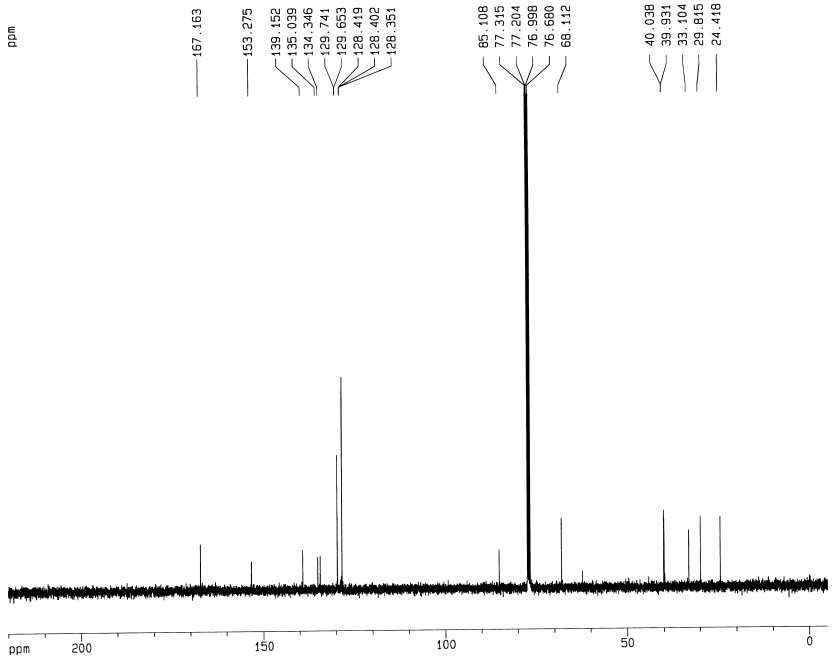



```
Current Data Parameters
NAME              H22442
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20040727
Time                8.40
INSTRUM           ARX400
PROBHD     5 mm Multinu
PULPROG               zg
TD                 32768
SOLVENT            CDCl3
NS                    64
DS                     0
SWH            6410.256 Hz
FIDRES         0.195625 Hz
AQ           2.5559540 sec
RG                  1430
DW               78.000 usec
DE               111.43 usec
TE                300.0 K
D1           2.00000000 sec
P1                 2.00 usec
DE               111.43 usec
SFO1        400.1320000 MHz
NUCLEUS               1H

F2 - Processing parameters
SI                 16384
SF          400.1300085 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  0.50

1D NMR plot parameters
CX                 20.00 cm
F1P               12.000 ppm
F1               4801.56 Hz
F2P               -0.500 ppm
F2               -200.07 Hz
PPMCM            0.62500 ppm/cm
HZCM           250.08125 Hz/cm
```



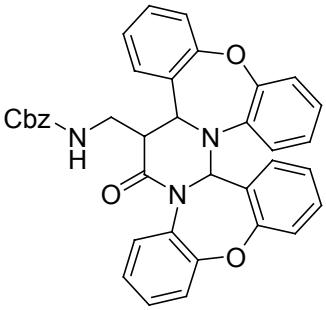

```
Current Data Parameters
NAME              C22442
EXPNO                  2
PROCNO                 1

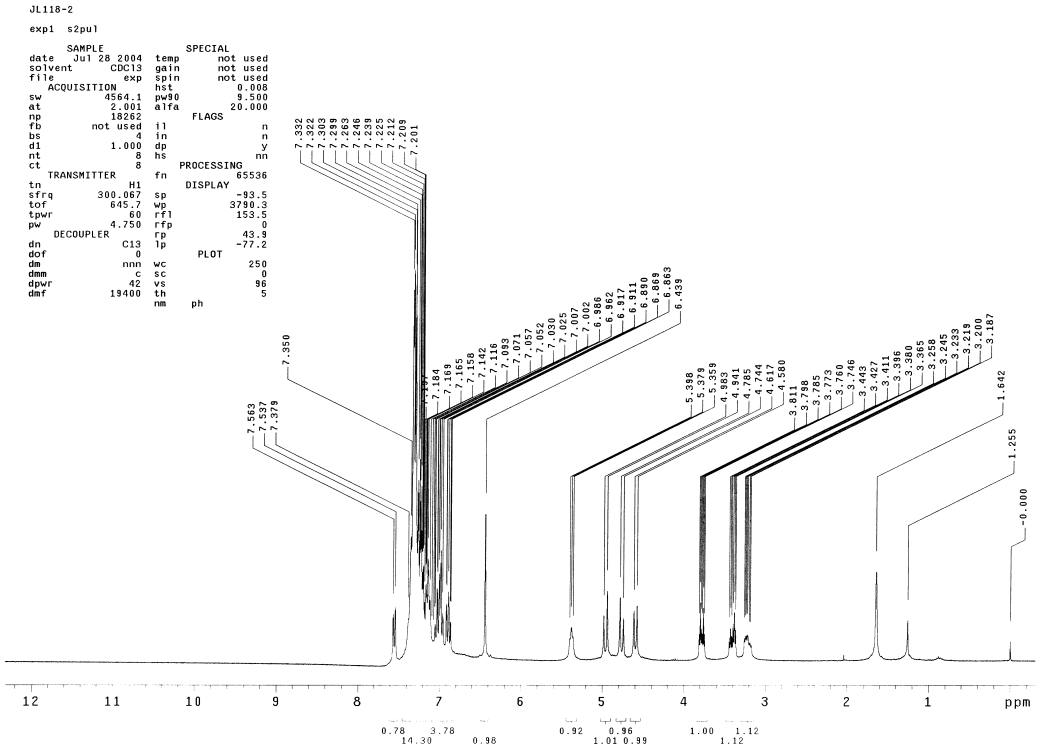
F2 - Acquisition Parameters
Date_           20040727
Time               10.49
INSTRUM           ARX400
PROBHD     5 mm Multinu
PULPROG            zgdc
TD                 32768
SOLVENT            CDCl3
NS                  4000
DS                     0
SWH           29411.766 Hz
FIDRES         0.897576 Hz
AQ           0.5571060 sec
RG                 32768
DW               17.000 usec
DE                24.29 usec
TE                300.0 K
D12          0.00002000 sec
DL5                24.00 dB
CPDPRG           waltz16
P31               100.00 usec
D1           2.00000000 sec
P1                 4.00 usec
DE                24.29 usec
SFO1        100.6240000 MHz
NUCLEUS               13C
D11          0.03000000 sec

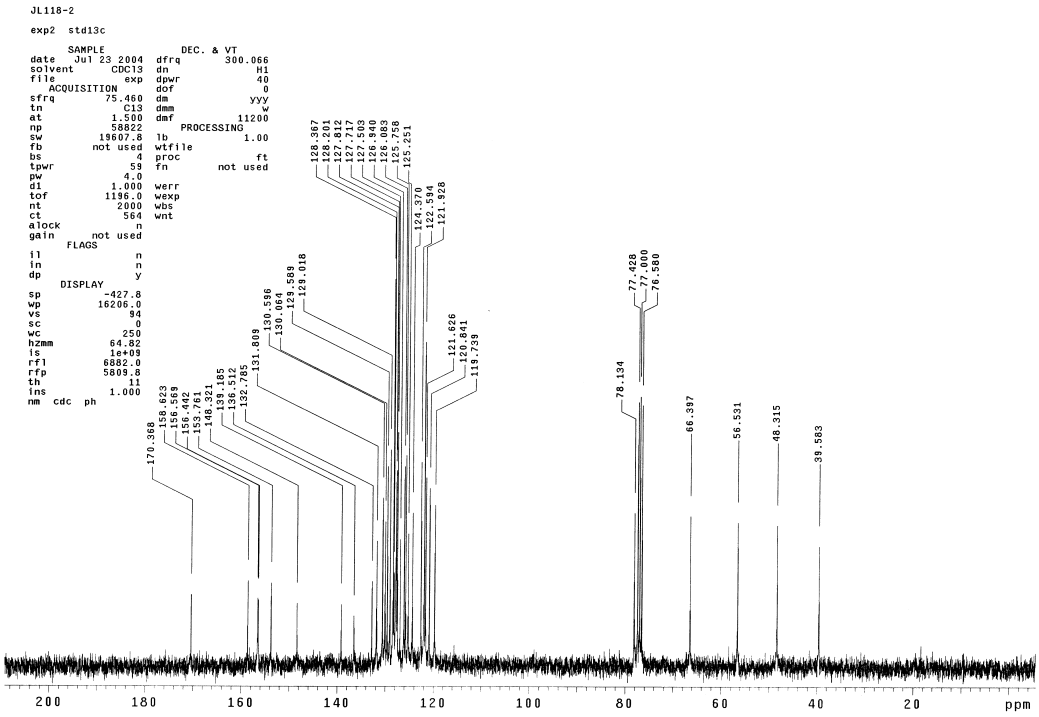
F2 - Processing parameters
SI                 32768
SF          100.6127711 MHz
WDW                   EM
SSB                    0
LB                  0.50 Hz
GB                     0
PC                  0.30

1D NMR plot parameters
CX                 20.00 cm
F1P              220.000 ppm
F1             22134.81 Hz
F2P               -5.000 ppm
F2               -503.07 Hz
PPMCM            11.25000 ppm/cm
HZCM           1131.89368 Hz/cm
```

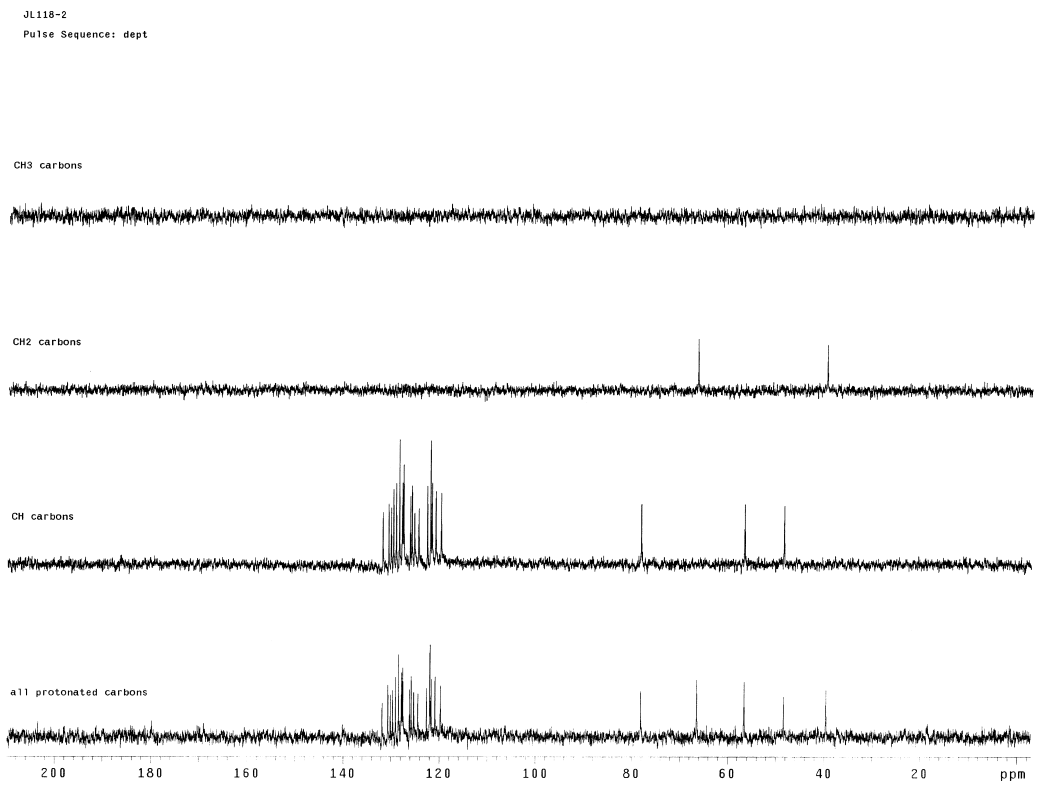
10a


Cbz
N
H
O
N
O
N
O

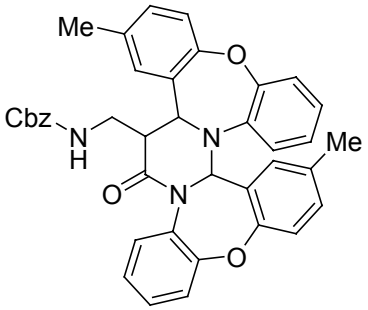

JL118-2
exp1 s2pul
SAMPLE
date Jul 28 2004
solvent CDCl3
file exp
ACQUISITION
sw 4564.1
at 2.001
np 18262
fb not used
bs 4
d1 1.000
nt 8
ct 8
TRANSMITTER
tn H1
sfrq 300.067
tof 645.7
tpwr 60
pw 4.750
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 42
dmf 19400
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 9.500
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn 65536
DISPLAY
sp -93.5
wp 3790.3
rfl 153.5
rfp 0
rp 43.9
lp -77.2
PLOT
wc 250
sc 0
vs 96
th 5
nm ph
7.332
7.322
7.303
7.289
7.263
7.246
7.239
7.225
7.212
7.209
7.201
7.350
7.563
7.537
7.379
7.184
7.169
7.165
7.158
7.155
7.142
7.116
7.093
7.071
7.057
7.052
7.030
7.025
7.007
7.002
6.986
6.942
6.917
6.911
6.890
6.869
6.863
6.488
5.388
5.379
5.359
4.983
4.941
4.785
4.744
4.617
4.580
3.811
3.798
3.785
3.773
3.760
3.746
3.445
3.427
3.411
3.396
3.380
3.365
3.258
3.245
3.233
3.219
3.200
3.187
1.642
1.255
-0.000
12 11 10 9 8 7 6 5 4 3 2 1 ppm
0.78 3.78
14.30 0.98
0.92 0.96
1.01 0.99
1.00 1.12
1.12

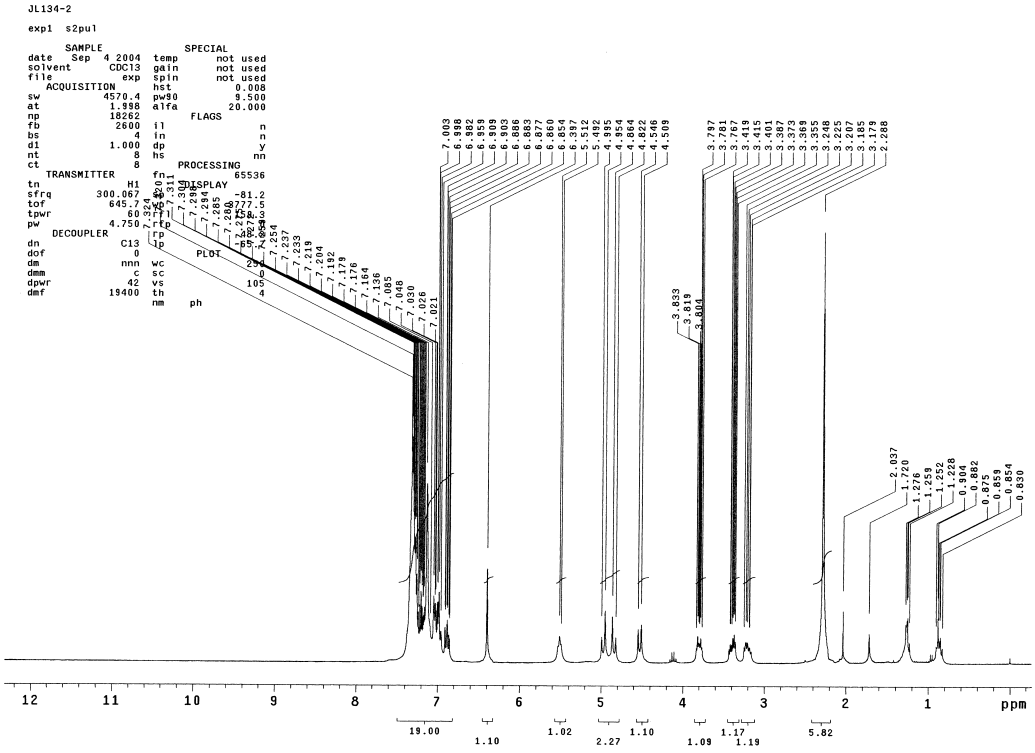

JL118-2
exp2 std13c
SAMPLE
date Jul 23 2004
solvent CDCl3
file exp
ACQUISITION
sfrq 75.460
tn C13
at 1.500
np 58822
sw 19607.8
fb not used
bs 4
tpwr 59
pw 4.0
d1 1.000
tof 1196.0
nt 2000
ct 564
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -427.8
wp 16206.0
vs 94
sc 0
wc 250
hzmm 64.82
is 1e+09
rfl 6882.0
rfp 5809.8
th 11
ins 1.000
nm cdc ph
DEC. & VT
dfrq 300.066
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 11200
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
128.367
128.201
127.812
127.717
127.503
126.840
126.085
125.758
125.251
124.370
122.594
121.928
121.626
120.841
119.739
129.589
129.018
130.586
130.064
131.809
132.785
139.185
139.572
148.321
153.761
156.442
158.569
158.623
170.368
77.428
77.000
76.580
78.134
66.397
56.531
48.315
39.583
200 180 160 140 120 100 80 60 40 20 ppm

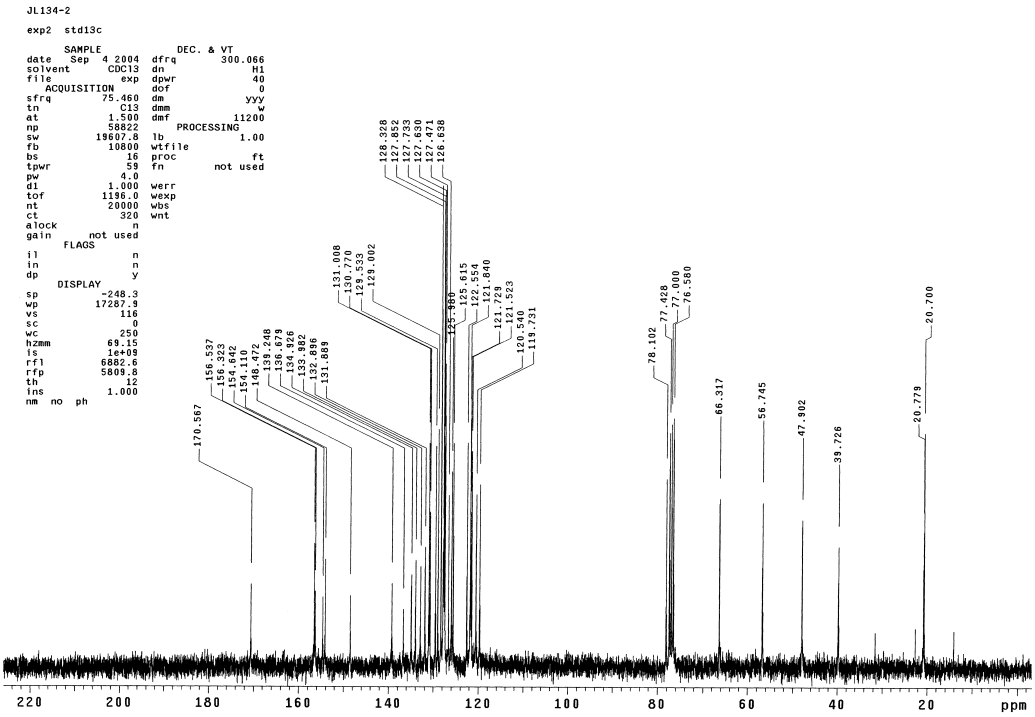
DEPT of **10a**


JL118-2
Pulse Sequence: dept
CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
ppm

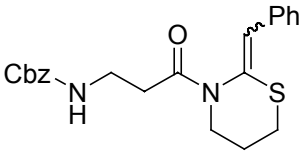
10b

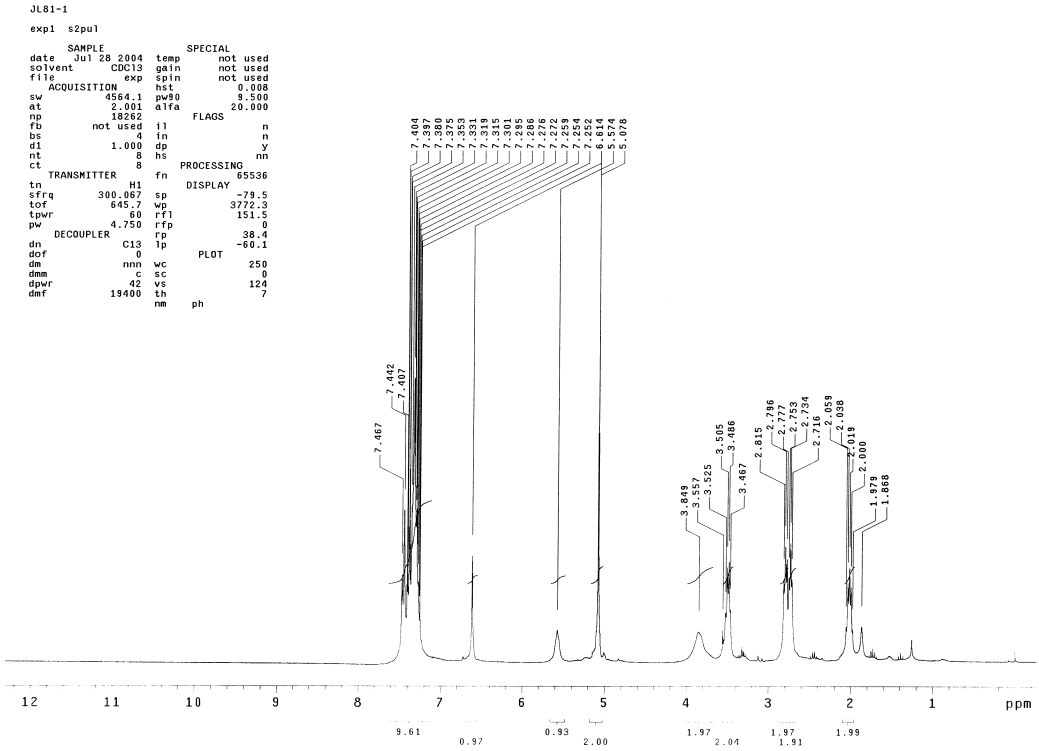

Me
Cbz
N
H
O
N
N
Me
O
O


JL134-2
exp1 s2pul
SAMPLE
date Sep 4 2004
solvent CDCl3
file exp
ACQUISITION
sw 4570.4
at 1.998
np 18262
fb 2600
bs 4
d1 1.000
nt 8
ct 8
TRANSMITTER
tn H1
sfrq 300.067
tof 645.7
tpwr 60
pw 4.750
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 42
dmf 19400
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 9.500
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn 65536
DISPLAY
sp -81.2
wp 8777.5
rf1 -158.3
rfp 7.324
PLOT
sc 0
vs 105
th 4
nm ph
ppm
19.00
1.10
1.02
2.27
1.10
1.09
1.17
1.19
5.82


JL134-2
exp2 std13c
SAMPLE
date Sep 4 2004
solvent CDCl3
file exp
ACQUISITION
sfrq 75.460
tn C13
at 1.500
np 58822
sw 19607.8
fb 10800
bs 16
tpwr 59
pw 4.0
d1 1.000
tof 1196.0
nt 20000
ct 320
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -248.3
wp 17287.9
vs 116
sc 0
wc 250
hzmm 69.15
is 1e+09
rf1 6882.6
rfp 5809.8
th 12
ins 1.000
nm no ph
DEC. & VT
dfrq 300.066
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 11200
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
ppm

11


Ph
O
Cbz
N
H
N
S


JL81-1
exp1 s2pul
SAMPLE
date Jul 28 2004
solvent CDCl3
file exp
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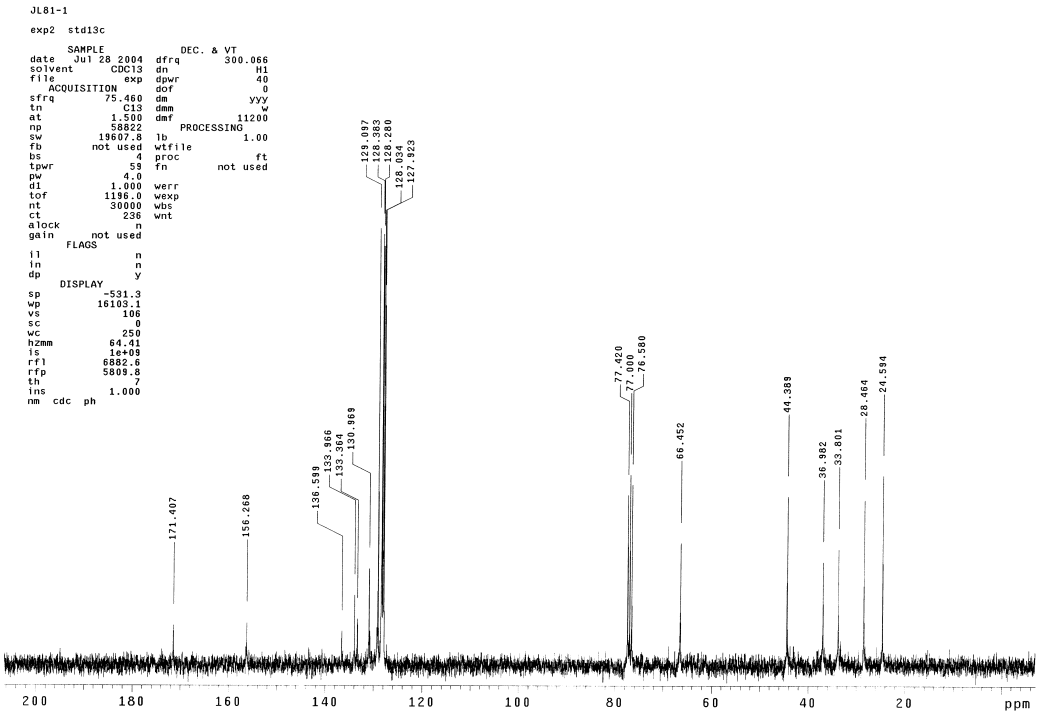
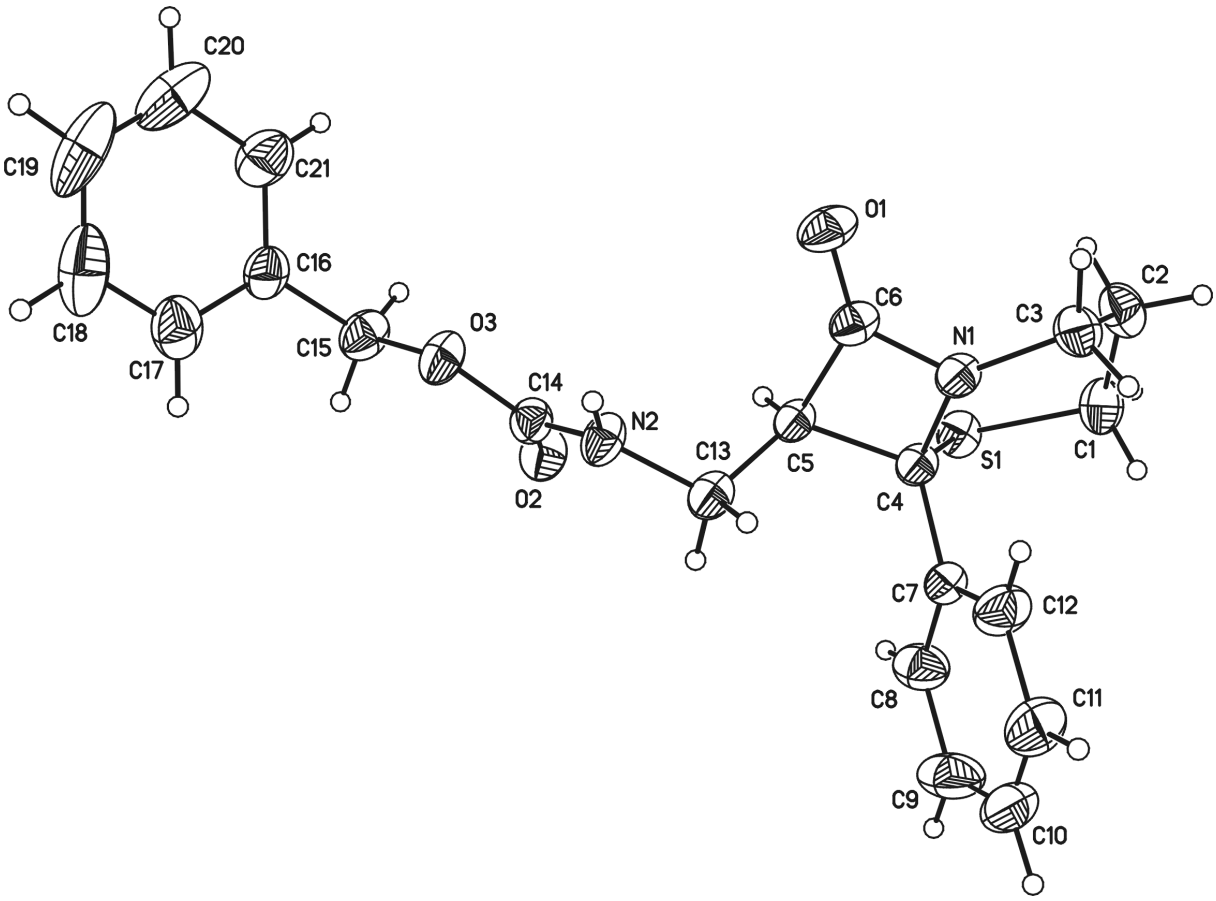
at 2.001
np 18262
fb not used
bs 4
d1 1.000
nt 8
ct 8
TRANSMITTER
tn H1
sfrq 300.067
tof 645.7
tpwr 60
pw 4.750
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 42
dmf 19400
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 9.500
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn 65536
DISPLAY
sp -79.5
wp 3772.3
rfl 151.5
rfp 0
rp 38.4
lp -60.1
PLOT
wc 250
sc 0
vs 124
th 7
nm ph
7.404
7.397
7.380
7.375
7.353
7.331
7.318
7.315
7.301
7.295
7.286
7.276
7.272
7.259
7.254
7.252
6.614
5.574
5.078
7.442
7.407
7.467
3.849
3.557
3.525
3.505
3.466
3.467
2.815
2.796
2.777
2.755
2.734
2.716
2.059
2.038
2.019
2.000
1.979
1.868
12 11 10 9 8 7 6 5 4 3 2 1 ppm
9.61 0.97 0.93 2.00 1.97 2.04 1.97 1.91 1.99


JL81-1
exp2 std13c
SAMPLE
date Jul 28 2004
solvent CDCl3
file exp
ACQUISITION
sfrq 75.460
tn C13
at 1.500
np 58822
sw 19607.8
fb not used
bs 4
tpwr 59
pw 4.0
d1 1.000
tof 1196.0
nt 30000
ct 236
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -531.3
wp 16103.1
vs 106
sc 0
wc 250
hzmm 64.41
is 1e+09
rfl 5882.6
rfp 5809.8
th 7
ins 1.000
nm cdc ph
DEC. & VT
dfrq 300.066
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 11200
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
129.097
128.383
128.280
128.094
127.923
77.420
77.000
76.580
66.452
44.389
36.982
33.801
28.464
24.594
171.407
156.268
136.598
133.466
133.364
130.969
200 180 160 140 120 100 80 60 40 20 ppm

Crystal Structure of 6a[6] (ORTEP Figure)



[6] CCDC 246471 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.