An Efficient and General Approach to β-Functionalized Ketones

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Supporting Information

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Materials and general methods

All solvents were distilled before use. All cyclopropyl alcohols were prepared using the Kulinkovich reaction.[1] All the other compounds were purchased from Aldrich or Acros, and were used without further purification.

^{1}H and ^{13}C NMR spectra were recorded on a Bruker 500 MHz spectrometer. Mass spectra were obtained using a HP 5890 series GC-MS instrument. GC-MS method: initial temp.: 50 ^{o}C (hold for 3 min), rate: 15 ^{o}C/min, final temp.: 280 ^{o}C (hold for 7 min). Column chromatography was performed using 65-250 mesh silica gel.

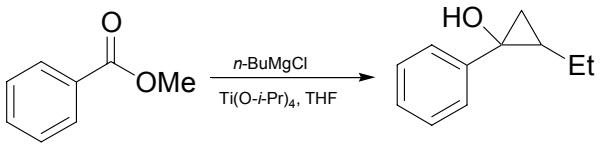
Yields reported in the supporting information refer to a single experiment. Known compounds were characterized by comparing their ^{1}H NMR spectra to the previously reported data. Their purity was confirmed by NMR analysis with a copy of ^{1}H NMR spectrum included. Previously unknown compounds were synthesized, purified, analyzed from a single run. They were characterized by ^{1}H NMR, ^{13}C NMR, MS, and HRMS. HRMS were performed by the Mass Spectrometry Facility at the University of Notre Dame, IN. For all of the unknown compounds, a copy of their ^{1}H NMR and ^{13}C NMR spectra is included.

Representative procedure for the synthesis of cyclopropanols via the Kulinkovich reaction

In a fume hood, 14 mmol of ethylmagnesium chloride in THF was added dropwise over 30 minutes at room temperature to a solution of 5 mmol of ester and 7 mmol of titanium tetraisopropoxide in THF. The reaction was allowed to stand while stirring overnight. Then the solution was quenched with distilled water and stirred while still in the fume hood. A precipitate was formed and the solution was filtered. After filtration, the solution was extracted with 3 x 30 mL of diethyl ether, washed with

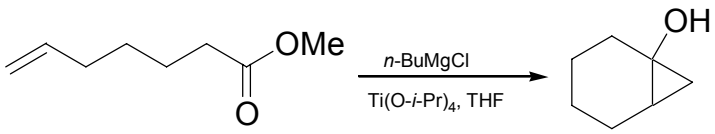
distilled water, and dried over $MgSO_4$ followed by filtration and concentration. The organic residue was subjected to column chromatography to afford the cyclopropanols **1**-**4**.

General procedure for the synthesis of 2-Ethyl-1-phenylcyclopropanol (5)



In a fume hood, 14 mmol of *n*-butylmagnesium chloride in THF was added dropwise over 30 minutes at room temperature to a solution of 5 mmol of ester and 7 mmol of titanium tetraisopropoxide in THF. The reaction was allowed to stand while stirring overnight. Then the solution was quenched with distilled water and stirred while still in the fume hood. A precipitate was formed and the solution was then filtered. After the filtration, the solution was extracted with 3 x 30 mL of diethyl ether, washed with distilled water, and dried over $MgSO_4$ followed by filtration and concentration. Then the organic residue was subjected to column chromatography to afford compound **5**.

General procedure for the synthesis of Bicyclo[4.1.0]heptan-1-ol (8)



To a solution of methyl 6-heptenoate (10 mmol) in 10 mL of THF was added Ti(O-*i*-Pr)$_4$ (10 mmol). *n*-Butylmagnesium chloride (50 mmol, 25 mL of 2.0 M solution in THF) was added at room temperature over a period of 1 h. The reaction mixture was stirred for overnight and quenched with distilled water. A precipitate was formed and the solution was then filtered. After the filtration, the solution was extracted with 3 x 30 mL of diethyl ether, washed with distilled water, and dried over $MgSO_4$ followed by filtration

and concentration. Then the organic residue was subjected to column chromatography to afford compound **8**.

General procedure for the preparation of (Bicyclo[4.1.0]hept-1-yloxy)-trimethylsilane (10)

To a stirred solution of trimethyl silyl enol ether (5 mmol) in toluene (12 mL) at 0°C was added diethyl zinc (10.2 mL, 1 M, 10.2 mmol) and then diiodomethane (0.82 mL, 10.2 mmol). The white suspension was allowed to warm to room temperature and stirred for 16 hours before the addition of pyridine (1.65 mL, 20.4 mmol). After 25 minutes the reaction mixture was poured onto petroleum ether (100 mL). The aqueous layer was extracted with petroleum ether (2x 50 mL). The combined organic extract was washed with brine and dried with anhydrous $MgSO_4$, followed by filtration and removal of solvent with rotary evaporation. The crude products were purified with column chromatography using silica gel as stationary phase and 9:1 Hexane/Et_2O as mobile phase.

Representative procedure for the synthesis of β-functionalized ketones using CAN

Organic solvent (MeOH, MeCN, or CH_2Cl_2) was added to 1.0 mmol of cyclopropanol (or **10**) in a round bottom flask containing a magnetic stir bar. Next, a solution of 1.0 mmol of the ionic substrate that contains the desired anion was made using water or methanol depending on the reaction conditions needed. This ionic solution was poured into the flask. In a separate reaction vessel, a solution of 2.0 mmol of CAN in an appropriate solvent (MeOH, MeCN, or H_2O) was prepared. Under nitrogen atmosphere and constant stirring of the cyclopropanol and ionic solution mixture, the CAN solution was added dropwise. The reaction was run for 30 minutes, and was worked up by removing the organic solvent via rotary evaporation. Distilled water (30 mL) was poured into the flask and the solution was extracted with 3 x 30 mL of diethyl

ether, washed with distilled water, and dried over $MgSO_4$ followed by filtration and concentration. The organic residue was subjected to column chromatography to afford the products. All of the β-substituted ketones are prone to decomposition in neat form, so they are kept in $CDCl_3$ as a solution.

Spectral data for the starting materials and products

1-Phenyl-cyclopropanol (**1**)[2]: The representative procedure was followed using methyl benzoate (0.68 g, 5 mmol), titanium tetraisopropoxide (2 g, 7 mmol), and EtMgCl (7 mL of 2 M solution, 14 mmol) employing THF as solvent. The reaction was run overnight at room temperature. Chromatographic purification (Hexane/ethyl acetate) provided 0.52 g of compound **1** as a yellow liquid (78%). ^{1}H NMR (500MHz, $CDCl_3$): δ 7.34-7.30 (m, 5H); 2.29 (s, 1H); 1.25 (m, 2H); 1.03 (m, 2H). A copy of the ^{1}H NMR spectrum can be found at the end of this document to confirm the compound's purity.

1-(4-Methoxy-phenyl)-cyclopropanol (**2**): The representative procedure was followed using methyl 4-methoxybenzoate (0.83 g, 5 mmol), titanium tetraisopropoxide (2 g, 7 mmol), and EtMgCl (7 mL of 2 M solution, 14 mmol) employing THF as solvent. The reaction was run overnight at room temperature. Chromatographic purification (Hexane/ethyl acetate) provided 0.62 g of compound **2** as a yellow liquid (75%). ^{1}H NMR (500MHz, $CDCl_3$): δ 7.25-6.83 (m, 4H); 3.77 (s, 3H); 2.51 (s, 1H); 1.17 (m, 2H); 0.94 (m, 2H). Registry number: 15973-65-6. A copy of the ^{1}H NMR spectrum can be found at the end of this document to confirm the compound's purity.

1-Cyclohexylcyclopropanol (**3**)[3]: The representative procedure was followed using methyl cyclohexanecarboxylate (0.71 g, 5 mmol), titanium tetraisopropoxide (2 g, 7 mmol), and EtMgCl (7 mL of 2 M solution, 14 mmol) employing THF as solvent. The

reaction was run overnight at room temperature. Chromatographic purification (Hexane/ethyl acetate) provided 0.57 g of compound **3** as a yellow liquid (82%). ^{1}H NMR (500MHz, $CDCl_3$): δ 1.93 (s, 1H); 1.76-1.11 (m, 11H); 0.65 (m, 2H); 0.40 (m, 2H). A copy of the ^{1}H NMR spectrum can be found at the end of this document to confirm the compound's purity.

1-Methyl-cyclopropanol (**4**): The representative procedure was followed using ethyl acetate (0.44 g, 5 mmol), titanium tetraisopropoxide (2 g, 7 mmol), and EtMgCl (7 mL of 2 M solution, 14 mmol) employing THF as solvent. The reaction was run overnight at room temperature. Chromatographic purification (Hexane/ethyl acetate) provided 0.23 g of compound **4** as a yellow liquid (63%). ^{1}H NMR (500MHz, $CDCl_3$): δ 1.82 (s, 1H); 1.21 (s, 3H); 0.62 (m, 2H); 0.41 (m, 2H). Registry number: 29526-99-6. A copy of the ^{1}H NMR spectrum can be found at the end of this document to confirm the compound's purity.

2-Ethyl-1-phenylcyclopropanol (**5**)[4]: The representative procedure was followed using methyl benzoate (0.68 g, 5 mmol), titanium tetraisopropoxide (2 g, 7 mmol), and *n*-BuMgCl (7 mL of 2 M solution, 14 mmol) employing THF as solvent. The reaction was run overnight at room temperature. Chromatographic purification (Hexane/ethyl acetate) provided 0.57 g of compound **5** as a yellow liquid (70%, diastereomeric mixture). ^{1}H NMR (500MHz, $CDCl_3$): (*trans* isomer) δ 7.40–7.20 (m, 5H); 2.56 (bs, 1H); 1.38–1.26 (m, 1H); 1.15–1.03 (m, 1H); 1.05 (dd, J = 5.7, 10.2 Hz, 1H); 0.86 (dd, J = 5.7, 12.3 Hz, 1H); 0.82–0.72 (m, 4H). A copy of the ^{1}H NMR spectrum can be found at the end of this document to confirm the compound's purity.

Bicyclo[4.1.0]heptan-1-ol (**8**)[5]: The general procedure was followed using methyl 6-heptenoate (1.42 g, 10 mmol), titanium tetraisopropoxide (2.9 g, 10 mmol), and *n*-BuMgCl (25 mL of 2.0 M solution in THF, 50 mmol) employing THF as solvent. The reaction was run overnight at room temperature. Chromatographic purification (Hexane/ethyl acetate) provided 0.81 g of compound **8** as a yellow oil (72%). ^{1}H NMR (500MHz, $CDCl_3$): δ 2.18-1.07 (m, 10H); 0.85 (m, 1H); 0.37 (m, 1H). A copy of the ^{1}H NMR spectrum can be found at the end of this document to confirm the compound's purity.

(Bicyclo[4.1.0]hept-1-yloxy)-trimethylsilane (**10**)[6]: The general procedure was followed using (cyclohexenyloxy)trimethylsilane (0.85 g, 5 mmol), diethyl zinc (10.2 mL of 1 M solution, 10.2 mmol), diiodomethane (0.82 mL, 10.2 mmol), and pyridine (1.65 mL, 20.4 mmol) with toluene as solvent. The reaction was run for 16 hours at 0 ^{o}C with warming to room temperature. Chromatographic purification (Hexane/diethyl ether) provided 0.83 g of compound **10** as a yellow oil (69%). ^{1}H NMR (500MHz, $CDCl_3$) δ 2.14-2.08 (m, 1H); 1.98-1.86 (m, 2H); 1.45-1.34 (m, 2H); 1.20-1.02 (m, 4H); 0.74-0.20 (m, 2H); 0.09 (s, 9H). A copy of the ^{1}H NMR spectrum can be found at the end of this document to confirm the compound's purity.

3-Azido-1-phenyl-1-propanone (**1a**)[7]: The representative procedure was followed using **1** (134 mg, 1 mmol), NaN_3 (65 mg, 1 mmol), and CAN (1.1 g, 2 mmol) with CH_3CN-H_2O(20%) as solvent. The reaction was run for 30 minutes at room temperature. Chromatographic purification (Hexane/ethyl acetate) provided 135 mg of compound **1a** as a yellow liquid (77%). ^{1}H NMR (500MHz, $CDCl_3$): δ 7.98-7.47 (m, 5H);

3.72 (t, J = 6.5 Hz, 2H); 3.23 (t, J = 6.5 Hz, 2H). A copy of the ^{1}H NMR spectrum can be found at the end of this document to confirm the compound's purity.

3-Azido-1-(4-methoxy-phenyl)-1-propanone (**2a**)[7]: The representative procedure was followed using **2** (164 mg, 1 mmol), NaN_3 (65 mg, 1 mmol), and CAN (1.1 g, 2 mmol) with CH_3CN-H_2O(20%) as solvent. The reaction was run for 30 minutes at room temperature. Chromatographic purification (Hexane/ethyl acetate) provided 170 mg of compound **2a** as a yellow liquid (83%). ^{1}H NMR (500MHz, $CDCl_3$): δ 7.92-6.90 (m, 4H); 3.84 (s, 3H); 3.68 (t, J = 6.5 Hz, 2H); 3.14 (t, J = 6.5 Hz, 2H). A copy of the ^{1}H NMR spectrum can be found at the end of this document to confirm the compound's purity.

3-Azido-1-cyclohexyl-1-propanone (**3a**): The representative procedure was followed using **3** (140 mg, 1 mmol), NaN_3 (65 mg, 1 mmol), and CAN (1.1 g, 2 mmol) with CH_3CN-H_2O(20%) as solvent. The reaction was run for 30 minutes at room temperature. Chromatographic purification (Hexane/ethyl acetate) provided 129 mg of compound **3a** as a yellow liquid (71%). ^{1}H NMR (500MHz, $CDCl_3$): δ 3.50 (t, J = 6.4 Hz, 2H); 2.67 (t, J = 6.4 Hz, 2H); 2.32 (m, 1H); 1.81-1.22 (m, 10H). ^{13}C NMR (125MHz, $CDCl_3$): δ 210.6; 59.9; 50.4; 45.4; 39.9; 38.8; 28.0; 27.8; 25.4. GC-MS: *m/z* (rel int) 181 (M^+, 1); 126 (8); 111 (16); 83 (100); 55 (91). FAB-HRMS calcd for $C_9H_{16}ON_3$ $(M+H)^+$: 182.1293, found: 182.1281.

4-Azido-2-butanone (**4a**): The representative procedure was followed using **4** (72 mg, 1 mmol), NaN_3 (65 mg, 1 mmol), and CAN (1.1 g, 2 mmol) with CH_3CN-H_2O(20%) as solvent. The reaction was run for 30 minutes at room temperature. Chromatographic purification (Hexane/ethyl acetate) provided 79 mg of compound **4a** as a yellow liquid

(70%). ^{1}H NMR (500MHz, $CDCl_3$): δ 3.48 (t, J = 6.4 Hz, 2H); 2.64 (t, J = 6.4 Hz, 2H); 2.01 (s, 3H). Registry number: 193401-62-6. A copy of the ^{1}H NMR spectrum can be found at the end of this document to confirm the compound's purity.

1-Phenyl-3-thiocyanato-1-propanone (**1b**): The representative procedure was followed using **1** (134 mg, 1 mmol), NH_4SCN (76 mg, 1 mmol), and CAN (1.1 g, 2 mmol) with CH_3CN as solvent. The reaction was run for 30 minutes at room temperature. Chromatographic purification (Hexane/ethyl acetate) provided 166 mg of compound **1b** as a yellow liquid (87%). ^{1}H NMR (500MHz, $CDCl_3$): δ 7.96-7.47 (m, 5H); 3.53 (t, J = 6.5 Hz, 2H); 3.31 (t, J = 6.5 Hz, 2H). Registry number: 16006-57-8. A copy of the ^{1}H NMR spectrum can be found at the end of this document to confirm the compound's purity.

1-(4-Methoxy-phenyl)-3-thiocyanato-1-propanone (**2b**): The representative procedure was followed using **2** (164 mg, 1 mmol), NH_4SCN (76 mg, 1 mmol), and CAN (1.1 g, 2 mmol) with CH_3CN as solvent. The reaction was run for 30 minutes at room temperature. Chromatographic purification (Hexane/ethyl acetate) provided 197 mg of compound **2b** as a yellow liquid (89%). ^{1}H NMR (500MHz, $CDCl_3$): δ 7.93-6.89 (m, 4H); 3.85 (s, 3H); 3.46 (t, J = 6.5 Hz, 2H); 3.30 (t, J = 6.5 Hz, 2H). Registry number: 70018-94-9. A copy of the ^{1}H NMR spectrum can be found at the end of this document to confirm the compound's purity.

1-Cyclohexyl-3-thiocyanato-1-propanone (**3b**): The representative procedure was followed using **3** (140 mg, 1 mmol), NH_4SCN (76 mg, 1 mmol), and CAN (1.1 g, 2 mmol) with CH_3CN as solvent. The reaction was run for 30 minutes at room temperature. Chromatographic purification (Hexane/ethyl acetate) provided 177 mg of compound **3b**

as a yellow liquid (90%). ^{1}H NMR (500MHz, $CDCl_3$): δ 3.12 (t, J = 6.5 Hz, 2H); 2.98 (t, J = 6.5 Hz, 2H); 2.36 (m, 1H); 1.87-1.18 (m, 10H). ^{13}C NMR (125MHz, $CDCl_3$): δ 209.8; 50.3; 39.4; 30.7; 29.2; 27.8; 27.5; 25.2; 25.0. GC-MS: *m/z* (rel int) 197 (M^+, 1); 111 (35); 82 (100); 54 (70). FAB-HRMS calcd for $C_{10}H_{16}ONS$ $(M+H)^+$: 198.0953, found: 198.0932.

4-Thiocyanato-2-butanone (**4b**): The representative procedure was followed using **4** (72 mg, 1 mmol), NH_4SCN (76 mg, 1 mmol), and CAN (1.1 g, 2 mmol) with CH_3CN as solvent. The reaction was run for 30 minutes at room temperature. Chromatographic purification (Hexane/ethyl acetate) provided 110 mg of compound **4b** as a yellow liquid (85%). ^{1}H NMR (500MHz, $CDCl_3$): δ 3.10 (t, J = 6.5 Hz, 2H); 2.97 (t, J = 6.5 Hz, 2H); 2.16 (s, 3H). Registry number: 57308-66-4. A copy of the ^{1}H NMR spectrum can be found at the end of this document to confirm the compound's purity.

3-Iodo-1-phenyl-1-propanone (**1c**)[8]: The representative procedure was followed using **1** (134 mg, 1 mmol), NaI (150 mg, 1 mmol), and CAN (1.1 g, 2 mmol) with CH_3CN-H_2O(20%) as solvent. The reaction was run for 30 minutes at room temperature. Chromatographic purification (Hexane/ethyl acetate) provided 234 mg of compound **1c** as a yellow liquid (90%). ^{1}H NMR (500MHz, $CDCl_3$): δ 7.97-7.42 (m, 5H); 3.58 (t, J = 7.1 Hz, 2H); 3.45 (t, J = 7.1 Hz, 2H). A copy of the ^{1}H NMR spectrum can be found at the end of this document to confirm the compound's purity.

3-Iodo-1-(4-methoxy-phenyl)-1-propanone (**2c**): The representative procedure was followed using **2** (164 mg, 1 mmol), NaI (150 mg, 1 mmol), and CAN (1.1 g, 2 mmol) with CH_3CN-H_2O(20%) as solvent. The reaction was run for 30 minutes at room temperature. Chromatographic purification (Hexane/ethyl acetate) provided 248 mg of

compound **2c** as a yellow liquid (92%). ^{1}H NMR (500MHz, $CDCl_3$): δ 7.90-6.83 (m, 4H); 3.81 (s, 3H); 3.82 (t, J = 7.1 Hz, 2H); 3.43 (t, J = 7.1 Hz, 2H). Registry number: 130543-58-7. A copy of the ^{1}H NMR spectrum can be found at the end of this document to confirm the compound's purity.

1-Cyclohexyl-3-iodo-1-propanone (**3c**)[9]: The representative procedure was followed using **3** (140 mg, 1 mmol), NaI (150 mg, 1 mmol), and CAN (1.1 g, 2 mmol) with CH_3CN-H_2O(20%) as solvent. The reaction was run for 30 minutes at room temperature. Chromatographic purification (Hexane/ethyl acetate) provided 255 mg of compound **3c** as a yellow liquid (96%). ^{1}H NMR (500MHz, $CDCl_3$): δ 3.27 (t, J = 7.0 Hz, 2H); 3.08 (t, J = 7.0 Hz, 2H); 2.30 (m, 1H); 1.84-1.24 (m, 10H). A copy of the ^{1}H NMR spectrum can be found at the end of this document to confirm the compound's purity.

4-Iodo-2-butanone (**4c**)[10]: The representative procedure was followed using **4** (72 mg, 1 mmol), NaI (150 mg, 1 mmol), and CAN (1.1 g, 2 mmol) with CH_3CN-H_2O(20%) as solvent. The reaction was run for 30 minutes at room temperature. Chromatographic purification (Hexane/ethyl acetate) provided 170 mg of compound **4c** as a yellow liquid (86%). ^{1}H NMR (500MHz, $CDCl_3$): δ 3.25 (t, J = 7.0 Hz, 2H); 3.06 (t, J = 7.0 Hz, 2H); 2.16 (s, 3H). A copy of the ^{1}H NMR spectrum can be found at the end of this document to confirm the compound's purity.

3-Bromo-1-phenyl-1-propanone (**1d**)[11]: The representative procedure was followed using **1** (134 mg, 1 mmol), KBr (118 mg, 1 mmol), and CAN (1.1 g, 2 mmol) with CH_2Cl_2-H_2O(50%) as solvent. The reaction was run for 30 minutes at room temperature. Chromatographic purification (Hexane/ethyl acetate) provided 151 mg of compound **1d** as a yellow liquid (71%). ^{1}H NMR (500MHz, $CDCl_3$): δ 7.95-7.34 (m, 5H);

3.72 (t, J = 6.8 Hz, 2H); 3.56 (t, J = 6.8 Hz, 2H). A copy of the ^{1}H NMR spectrum can be found at the end of this document to confirm the compound's purity.

3-Bromo-1-(4-methoxy-phenyl)-1-propanone (**2d**): The representative procedure was followed using **2** (164 mg, 1 mmol), KBr (118 mg, 1 mmol), and CAN (1.1 g, 2 mmol) with CH_2Cl_2-H_2O(50%) as solvent. The reaction was run for 30 minutes at room temperature. Chromatographic purification (Hexane/ethyl acetate) provided 182 mg of compound **2d** as a yellow liquid (75%). ^{1}H NMR (500MHz, $CDCl_3$): δ 7.93-6.87 (m, 4H); 3.85 (s, 3H); 3.71 (t, J = 6.9 Hz, 2H); 3.49 (t, J = 6.9 Hz, 2H). Registry number: 33994-11-5. A copy of the ^{1}H NMR spectrum can be found at the end of this document to confirm the compound's purity.

3-Bromo-1-cyclohexyl-1-propanone (**3d**): The representative procedure was followed using **3** (140 mg, 1 mmol), KBr (118 mg, 1 mmol), and CAN (1.1 g, 2 mmol) with CH_2Cl_2-H_2O(50%) as solvent. The reaction was run for 30 minutes at room temperature. Chromatographic purification (Hexane/ethyl acetate) provided 201 mg of compound **3d** as a yellow liquid (92%). ^{1}H NMR (500MHz, $CDCl_3$): δ 3.53 (t, J = 6.8 Hz, 2H); 3.01 (t, J = 6.8 Hz, 2H); 2.31 (m, 1H); 1.84-1.15 (m, 10H). Registry number: 78864-62-7. A copy of the ^{1}H NMR spectrum can be found at the end of this document to confirm the compound's purity.

4-Bromo-2-butanone (**4d**)[11]: The representative procedure was followed using **4** (72 mg, 1 mmol), KBr (118 mg, 1 mmol), and CAN (1.1 g, 2 mmol) with CH_2Cl_2-H_2O(50%) as solvent. The reaction was run for 30 minutes at room temperature. Chromatographic purification (Hexane/ethyl acetate) provided 125 mg of compound **4d** as a yellow liquid (83%). ^{1}H NMR (500MHz, $CDCl_3$): δ 3.53 (t, J = 6.8 Hz, 2 H); 3.04 (t,

J = 6.8 Hz, 2 H); 2.13 (s, 3 H). A copy of the ^{1}H NMR spectrum can be found at the end of this document to confirm the compound's purity.

2-Azidomethyl-1-phenyl-1-butanone (**6a**): The representative procedure was followed using **5** (162 mg, 1 mmol), NaN_3 (65 mg, 1 mmol), and CAN (1.1 g, 2 mmol) with CH_3CN-H_2O(20%) as solvent. The reaction was run for 30 minutes at room temperature. GC analysis suggested 48% yield of **6a** was obtained. Pure product was obtained from silica gel chromatography as a yellow oil. ^{1}H NMR (500MHz, $CDCl_3$): δ 7.95-7.44 (m, 5H); 5.65 (m, 1H); 3.42 (dd, *J* = 6.6, 17.4 Hz, 1H); 3.17 (dd, *J* = 5.9, 17.4 Hz, 1H); 2.36-1.05 (m, 2H); 1.02 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (125MHz, $CDCl_3$): δ 201.5; 128.4; 128.0; 127.6; 125.4; 40.3; 37.9; 22.2; 13.5. GC-MS: *m/z* (rel int) 203 (M^+, 5); 161 (65); 105 (100); 77 (50); 51 (27). FAB-HRMS calcd for $C_{11}H_{14}ON_3$ $(M+H)^+$: 204.1137, found: 204.1142.

2-Iodomethyl-1-phenyl-1-butanone (**6b**): The representative procedure was followed using **5** (162 mg, 1 mmol), NaI (150 mg, 1 mmol), and CAN (1.1 g, 2 mmol) with CH_3CN-H_2O(20%) as solvent. The reaction was run for 30 minutes at room temperature. Chromatographic purification (Hexane/ethyl acetate) provided 190 mg of compound **6b** as a yellow liquid (66%). ^{1}H NMR (500MHz, $CDCl_3$): δ 7.94-7.32 (m, 5H); 4.58 (m, 1H); 3.81 (dd, *J* = 7.5, 17.4 Hz, 1H); 3.52 (dd, *J* = 6.4, 17.4 Hz, 1H); 1.83 (m, 1H); 1.27 (m, 1H); 1.06 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (125MHz, $CDCl_3$): δ 202.8; 133.0; 132.1; 128.3; 127.6; 48.7; 29.8; 25.4; 13.8. GC-MS: *m/z* (rel int) 288 (M^+, 1); 161 (28); 105 (100); 77 (57); 51 (21). FAB-HRMS calcd for $C_{11}H_{14}OI$ $(M+H)^+$: 289.0089, found: 289.0084.

2-Bromomethyl-1-phenyl-1-butanone (**6c**): The representative procedure was followed using **5** (162 mg, 1 mmol), KBr (118 mg, 1 mmol), and CAN (1.1 g, 2 mmol) with CH_2Cl_2-H_2O(50%) as solvent. The reaction was run for 30 minutes at room temperature. Chromatographic purification (Hexane/ethyl acetate) provided 142 mg of compound **6c** as a yellow oil (59%). ^{1}H NMR (500MHz, $CDCl_3$): δ 7.96-7.45 (m, 5H); 3.83 (m, 1H); 3.75 (dd, J = 8.3, 9.7 Hz, 1H); 3.49 (dd, J = 5.5, 9.8 Hz, 1H); 1.85 (m, 1H); 1.39 (m, 1H); 0.88 (t, J = 7.5 Hz, 3H). ^{13}C NMR (125MHz, $CDCl_3$): δ 201.9; 132.9; 132.4; 128.4; 127.6; 49.5; 31.7; 26.1; 12.7. GC-MS: *m/z* (rel int) 240 (M^+, 2); 149 (41); 104 (100); 76 (32); 50 (9). FAB-HRMS calcd for $C_{11}H_{14}OBr$ $(M+H)^+$: 241.0228, found: 241.0247.

3-Azido-cycloheptanone (**9a**): The representative procedure was followed using **8** (112 mg, 1 mmol), NaN_3 (65 mg, 1 mmol), and CAN (1.1 g, 2 mmol) with CH_3CN-H_2O(20%) as solvent. The reaction was run for 30 minutes at room temperature. GC analysis suggested 56% yield of **9a** was obtained. Pure product was obtained from silica gel chromatography as a yellow oil. ^{1}H NMR (500MHz, $CDCl_3$): δ 5.23 (m, 1H); 2.88 (m, 2H); 2.53-2.00 (m, 4H); 1.80-1.53 (m, 4H). ^{13}C NMR (125MHz, $CDCl_3$): δ 207.8; 51.7; 46.3; 43.6; 33.2; 24.1; 23.2. GC-MS: *m/z* (rel int) 153 (M^+, 12); 111 (76); 83 (25); 54 (100). FAB-HRMS calcd for $C_7H_{12}ON_3$ $(M+H)^+$: 154.0980, found: 154.0987.

3-Iodo-cycloheptanone (**9b**)[9]: The representative procedure was followed using **8** (112 mg, 1 mmol), NaI (150 mg, 1 mmol), and CAN (1.1 g, 2 mmol) with CH_3CN-H_2O(20%) as solvent. The reaction was run for 30 minutes at room temperature. Chromatographic purification (Hexane/ethyl acetate) provided 162 mg of compound **9b** as a yellow liquid (68%). ^{1}H NMR (500MHz, $CDCl_3$): δ 4.49 (m, 1H); 3.24 (dd, J = 2.9,

15.1 Hz, 1H); 3.14 (dd, J = 9.4, 15.0 Hz, 1H); 2.60-1.73 (m, 8H). The isolated yield of compound **9b** was improved to 87% using compound **10** at 0 ^{o}C following the same procedure. A copy of the ^{1}H NMR spectrum can be found at the end of this document to confirm the compound's purity.

3-Bromo-cycloheptanone (**9c**): The representative procedure was followed using **8** (112 mg, 1 mmol), KBr (118 mg, 1 mmol), and CAN (1.1 g, 2 mmol) with CH_2Cl_2-H_2O(50%) as solvent. The reaction was run for 30 minutes at room temperature. Chromatographic purification (Hexane/ethyl acetate) provided 114 mg of compound **6c** as a yellow oil (60%). ^{1}H NMR (500MHz, $CDCl_3$): δ 4.40 (m, 1H); 3.15 (dd, J = 2.9, 15.1 Hz, 1H); 3.07 (dd, J = 8.7, 15.1 Hz, 1H); 2.59-2.40 (m, 2H); 2.23-2.21 (m, 2H); 1.77-1.74 (m, 4H). ^{13}C NMR (125MHz, $CDCl_3$): δ 204.6; 52.7; 47.1; 43.4; 40.6; 26.8; 23.0. GC-MS: *m/z* (rel int) 190 (M^+, 10); 111 (60); 83 (28); 54 (100). FAB-HRMS calcd for $C_7H_{12}OBr$ $(M+H)^+$: 191.0072, found: 191.0045.

References:

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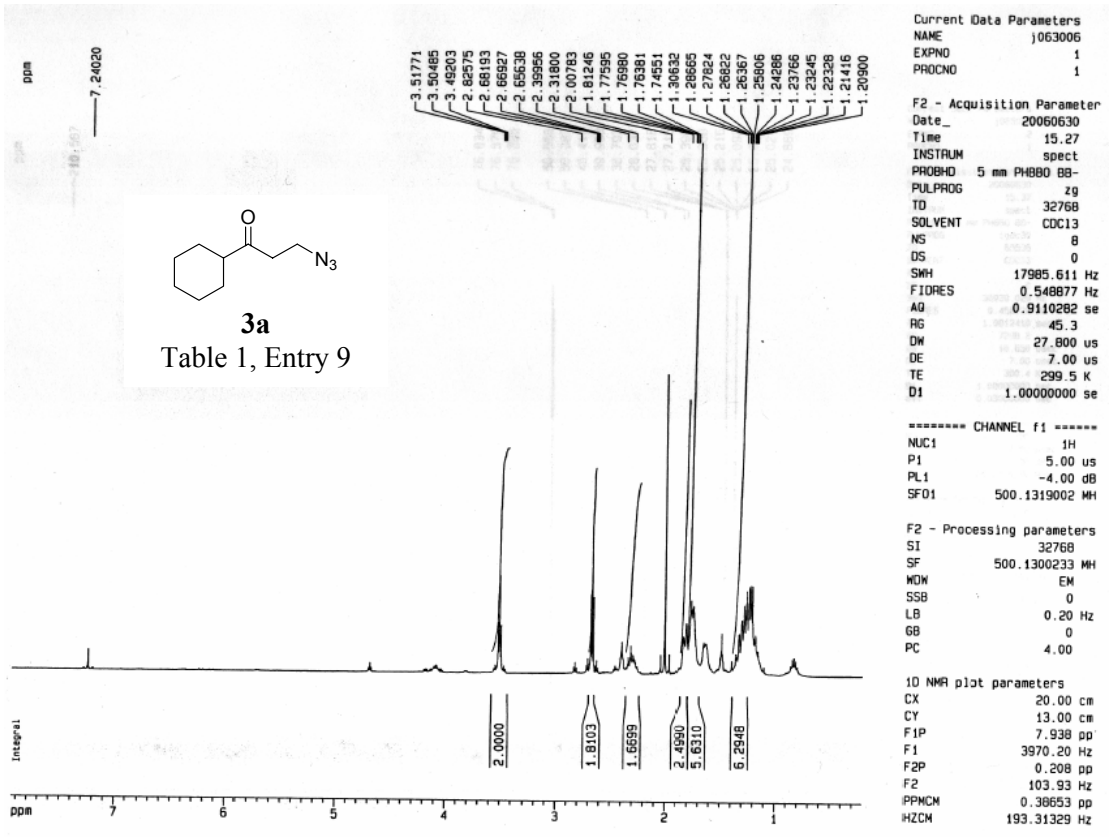
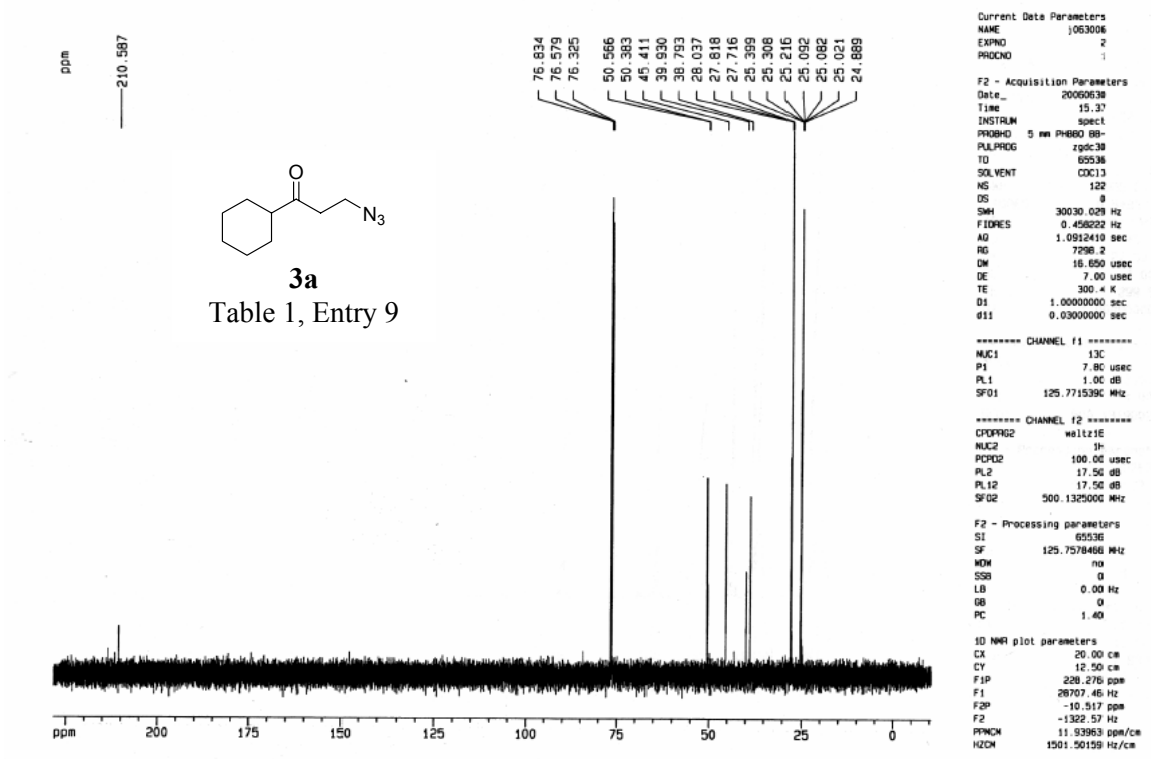


3a

Table 1, Entry 9

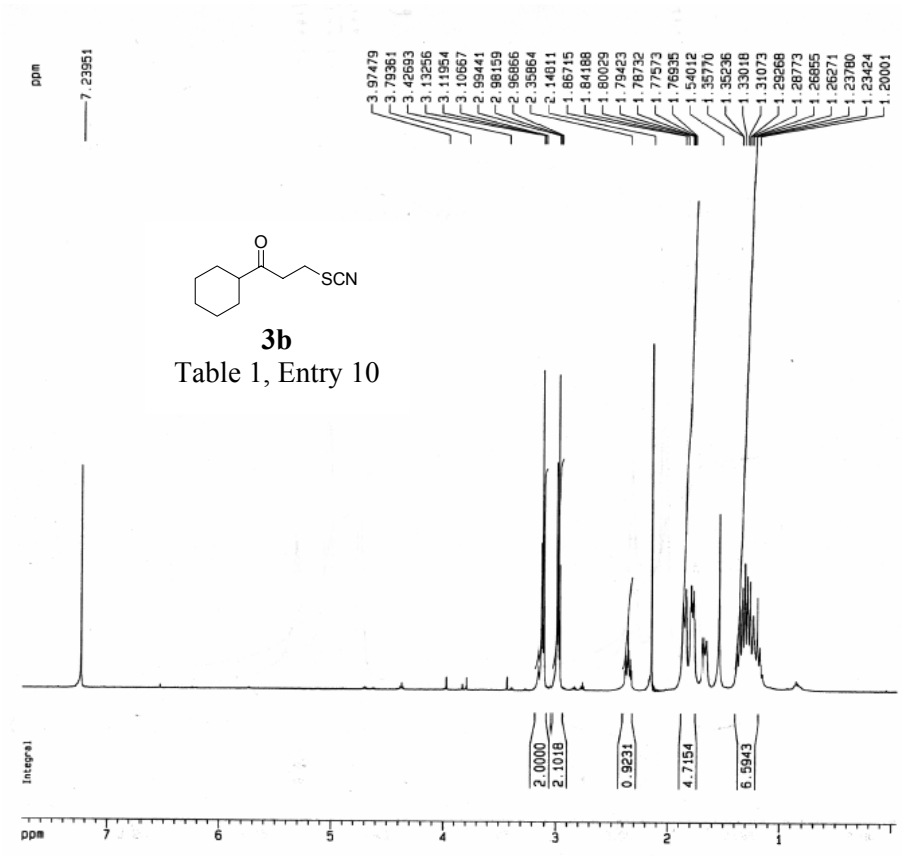
```
Current Data Parameters
NAME                 j063006
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameter
Date_               20060630
Time                   15.27
INSTRUM                spect
PROBHD   5 mm PH8BO BB-
PULPROG                   zg
TD                     32768
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                17985.611 Hz
FIDRES              0.548877 Hz
AQ                  0.9110282 se
RG                      45.3
DW                    27.800 us
DE                     7.00 us
TE                    299.5 K
D1                 1.00000000 se

======== CHANNEL f1 ======
NUC1                       1H
P1                     5.00 us
PL1                   -4.00 dB
SFO1            500.1319002 MH

F2 - Processing parameters
SI                     32768
SF              500.1300233 MH
WDW                       EM
SSB                        0
LB                    0.20 Hz
GB                         0
PC                      4.00

1D NMR plot parameters
CX                   20.00 cm
CY                   13.00 cm
F1P                   7.938 pp
F1                 3970.20 Hz
F2P                   0.208 pp
F2                  103.93 Hz
PPMCM              0.38653 pp
HZCM             193.31329 Hz
```



3a

Table 1, Entry 9

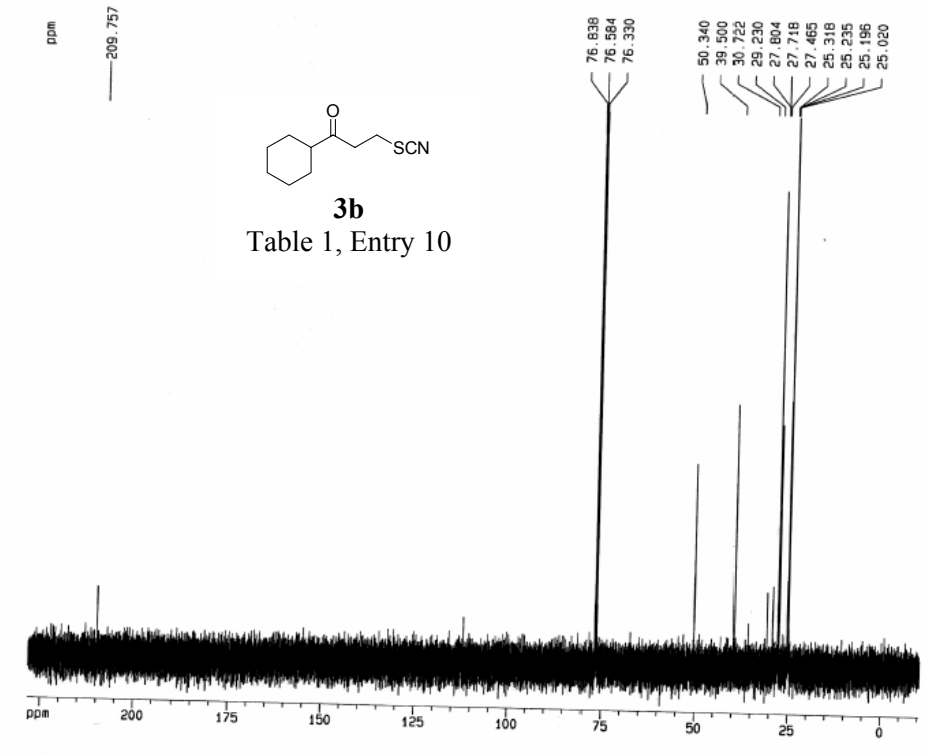
```
Current Data Parameters
NAME                 j063006
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_               20060630
Time                   15.37
INSTRUM                spect
PROBHD   5 mm PH8BO BB-
PULPROG             zgdc30
TD                     65536
SOLVENT                CDCl3
NS                       122
DS                         0
SWH                30030.029 Hz
FIDRES              0.458222 Hz
AQ                  1.0912410 sec
RG                     7298.2
DW                   16.650 usec
DE                     7.00 usec
TE                    300.4 K
D1                 1.00000000 sec
d11                0.03000000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                     7.80 usec
PL1                    1.00 dB
SFO1            125.7715390 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 100.00 usec
PL2                    17.50 dB
PL12                   17.50 dB
SFO2            500.1325000 MHz

F2 - Processing parameters
SI                     65536
SF              125.7578466 MHz
WDW                       no
SSB                        0
LB                     0.00 Hz
GB                         0
PC                      1.40

1D NMR plot parameters
CX                   20.00 cm
CY                   12.50 cm
F1P                 228.276 ppm
F1                28707.46 Hz
F2P                 -10.517 ppm
F2                 -1322.57 Hz
PPMCM              11.93963 ppm/cm
HZCM             1501.50159 Hz/cm
```



3b

Table 1, Entry 10

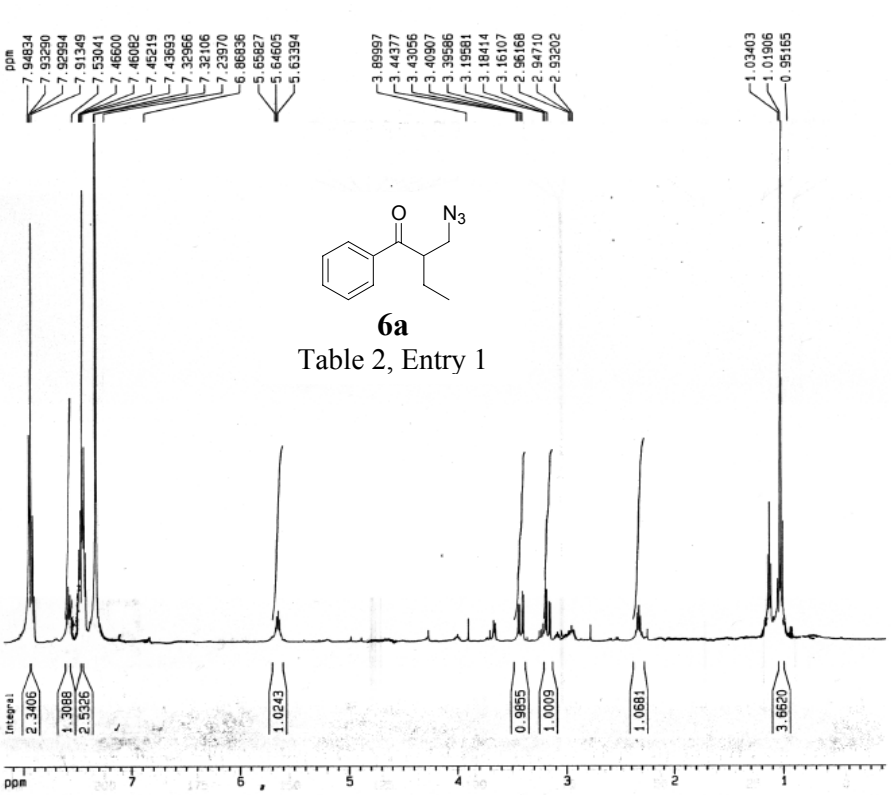
```
Current Data Parameters
NAME                  j073106
EXPNO                       5
PROCNO                      1

F2 - Acquisition Parameter
Date_                20060731
Time                    16.47
INSTRUM                 spect
PROBHD   5 mm PHBBO BB-
PULPROG                    zg
TD                      32768
SOLVENT                 CDCl3
NS                          8
DS                          0
SWH                17985.611 Hz
FIDRES              0.548877 Hz
AQ                  0.9110282 se
RG                      228.1
DW                     27.800 us
DE                      7.00 us
TE                     299.8 K
D1                 1.00000000 se

======== CHANNEL f1 ======
NUC1                       1H
P1                      5.00 us
PL1                     -4.00 dB
SFO1              500.1319002 MH

F2 - Processing parameters
SI                      32768
SF                500.1300233 MH
WDW                        EM
SSB                         0
LB                      0.20 Hz
GB                          0
PC                       4.00

1D NMR plot parameters
CX                     20.00 cm
CY                      8.00 cm
F1P                     7.767 pp
F1                   3884.76 Hz
F2P                    -0.048 pp
F2                    -24.23 Hz
PPMCM                0.39080 pp
HZCM               195.44934 Hz
```



3b

Table 1, Entry 10

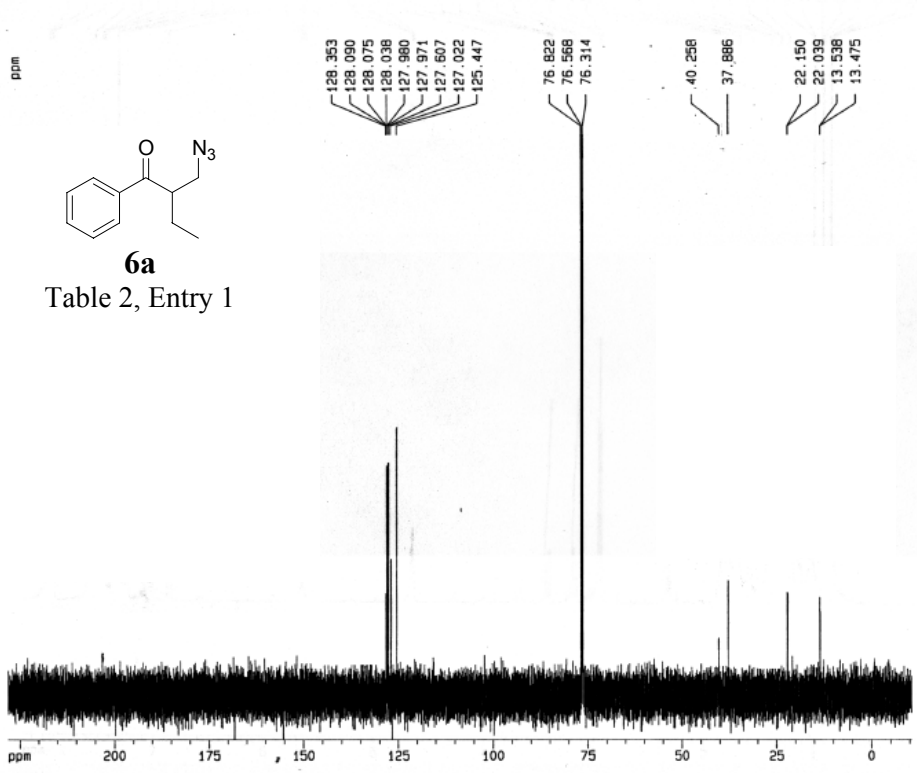
```
Current Data Parameters
NAME                  j072806
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_                20060728
Time                    16.06
INSTRUM                 spect
PROBHD   5 mm PHBBO BB-
PULPROG                 zgpc30
TD                      65536
SOLVENT                 CDCl3
NS                        201
DS                          0
SWH                30030.029 Hz
FIDRES              0.458222 Hz
AQ                  1.0912410 sec
RG                       3251
DW                    16.650 usec
DE                     7.00 usec
TE                     300.6 K
D1                 1.00000000 sec
d11                0.03000000 sec

======== CHANNEL f1 ========
NUC1                       13C
P1                     7.80 usec
PL1                     1.00 dB
SFO1             125.7715390 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                100.00 usec
PL2                    17.50 dB
PL12                   17.50 dB
SFO2             500.1325000 MHz

F2 - Processing parameters
SI                      65536
SF               125.7578466 MHz
WDW                        no
SSB                         0
LB                      0.00 Hz
GB                          0
PC                       1.40

1D NMR plot parameters
CX                     20.00 cm
CY                     12.50 cm
F1P                  228.276 ppm
F1                  28707.46 Hz
F2P                  -10.517 ppm
F2                  -1322.57 Hz
PPMCM               11.93963 ppm/cm
HZCM              1501.50159 Hz/cm
```



6a

Table 2, Entry 1

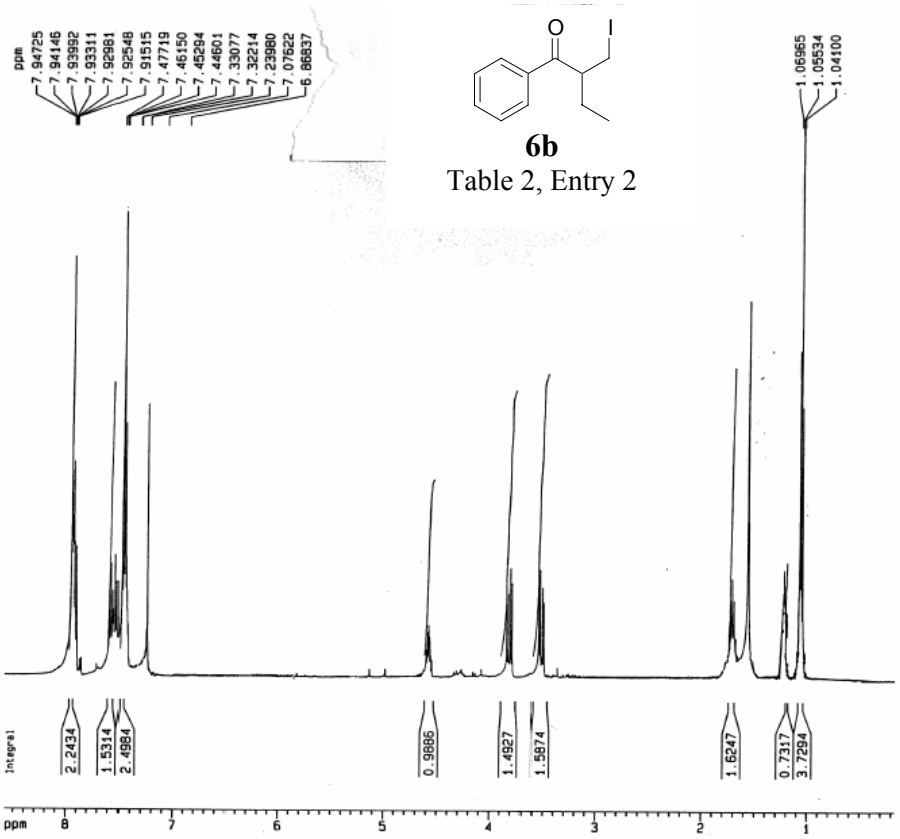
```
NAME                 j101106
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameter
Date_               20061011
Time                   14.38
INSTRUM                spect
PROBHD   5 mm PHBBO BB-
PULPROG                   zg
TD                     32768
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                17985.611 Hz
FIDRES             0.548877 Hz
AQ                 0.9110282 se
RG                     101.6
DW                    27.800 us
DE                     7.00 us
TE                     301.1 K
D1                1.00000000 se

======== CHANNEL f1 ======
NUC1                       1H
P1                     5.00 us
PL1                   -4.00 dB
SFO1           500.1319002 MH

F2 - Processing parameters
SI                     32768
SF           500.1300233 MH
WDW                       EM
SSB                        0
LB                  0.20 Hz
GB                         0
PC                      4.00

1D NMR plot parameters
CX                    20.00 cm
CY                    13.00 cm
F1P                   8.186 pp
F1                 4094.10 Hz
F2P                   0.065 pp
F2                 32.42 Hz
PPMCM              0.40606 pp
HZCM              203.08412 Hz
```



6a

Table 2, Entry 1

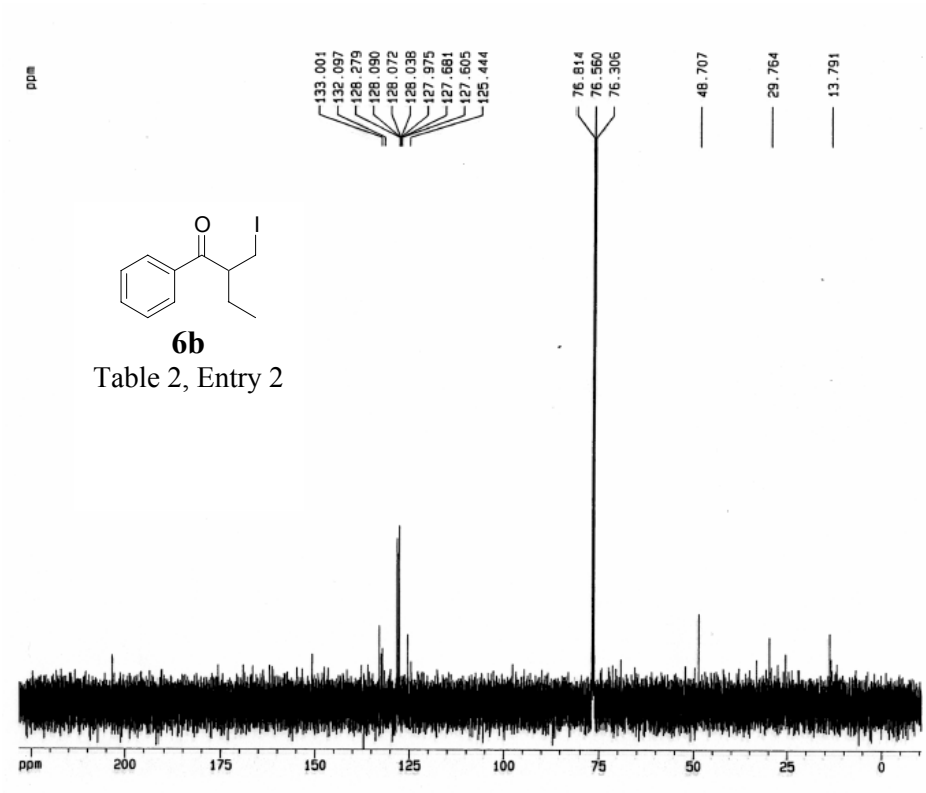
```
Current Data Parameters
NAME                 j101106
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_               20061011
Time                   15.00
INSTRUM                spect
PROBHD   5 mm PHBBO BB-
PULPROG             zgdc30
TD                     65536
SOLVENT                CDCl3
NS                       155
DS                         0
SWH                30030.029 Hz
FIDRES             0.458222 Hz
AQ                 1.0912410 sec
RG                    4597.6
DW                   16.650 usec
DE                    7.00 usec
TE                     301.8 K
D1                1.00000000 sec
d11               0.03000000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                    7.80 usec
PL1                   1.00 dB
SFO1          125.7715390 MHz

======== CHANNEL f2 ========
CPDPRG2               waltz16
NUC2                       1H
PCPD2                100.00 usec
PL2                   17.50 dB
PL12                  17.50 dB
SFO2          500.1325000 MHz

F2 - Processing parameters
SI                     65536
SF           125.7578466 MHz
WDW                       no
SSB                        0
LB                   0.00 Hz
GB                         0
PC                      1.40

1D NMR plot parameters
CX                    20.00 cm
CY                     6.00 cm
F1P                  228.276 ppm
F1                 28707.46 Hz
F2P                  -10.517 ppm
F2                -1322.57 Hz
PPMCM             11.93963 ppm/cm
HZCM              1501.50159 Hz/cm
```



6b

Table 2, Entry 2

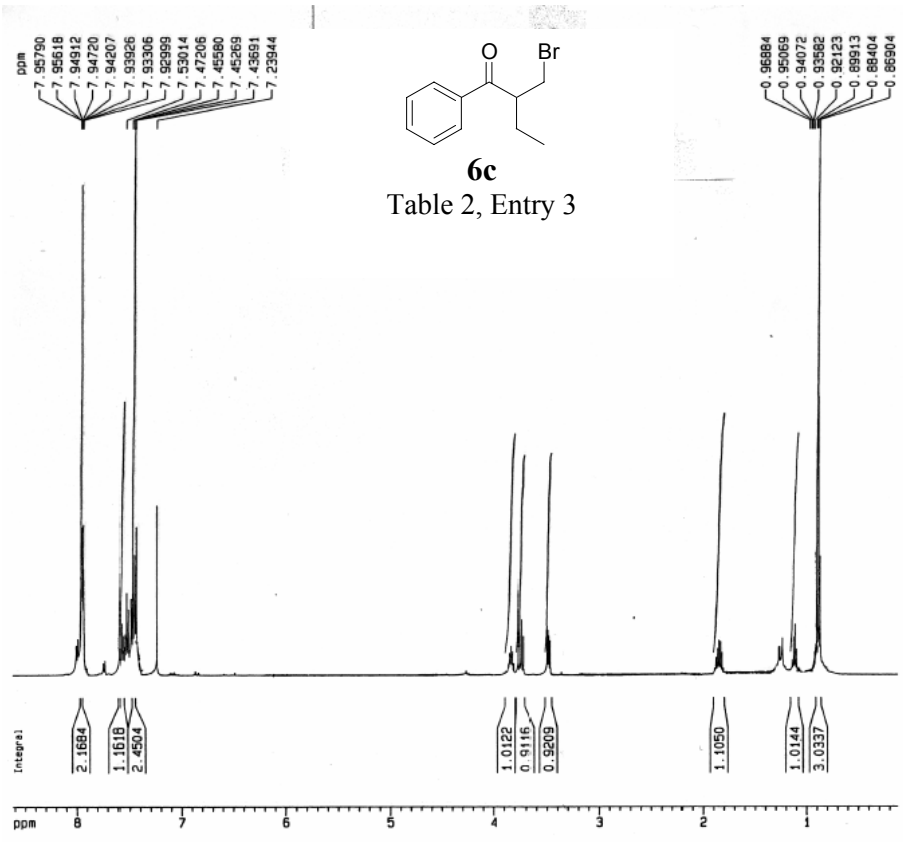
```
                    j100406
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameter
Date_              20061004
Time                  11.16
INSTRUM               spect
PROBHD    5 mm PHBBO BB-
PULPROG                 zg
TD                   32768
SOLVENT              CDCl3
NS                       8
DS                       0
SWH           17985.611 Hz
FIDRES         0.548877 Hz
AQ             0.9110282 se
RG                   161.3
DW                27.800 us
DE                 7.00 us
TE                301.1 K
D1            1.00000000 se

======== CHANNEL f1 ======
NUC1                    1H
P1                 5.00 us
PL1               -4.00 dB
SFO1        500.1319002 MH

F2 - Processing parameters
SI                   32768
SF          500.1300233 MH
WDW                     EM
SSB                      0
LB                 0.20 Hz
GB                       0
PC                   4.00

1D NMR plot parameters
CX                20.00 cm
CY                13.00 cm
F1P               8.579 pp
F1              4290.61 Hz
F2P               0.165 pp
F2                82.57 Hz
PPMCM             0.42069 pp
HZCM            210.40176 Hz
```



6b

Table 2, Entry 2

```
Current Data Parameters
NAME                 j100406
EXPNO                      3
PROCNO                     1

F2 - Acquisition Parameters
Date_               20061004
Time                   11.27
INSTRUM                spect
PROBHD    5 mm PHBBO BB-
PULPROG              zgdc30
TD                    65536
SOLVENT               CDCl3
NS                      245
DS                        0
SWH           30030.029 Hz
FIDRES         0.458222 Hz
AQ             1.0912410 sec
RG                  2896.3
DW                 16.650 usec
DE                  7.00 usec
TE                 302.0 K
D1            1.00000000 sec
d11           0.03000000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                  7.80 usec
PL1                 1.00 dB
SFO1        125.7715390 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2              100.00 usec
PL2                 17.50 dB
PL12                17.50 dB
SFO2        500.1325000 MHz

F2 - Processing parameters
SI                    65536
SF          125.7578466 MHz
WDW                      no
SSB                       0
LB                 0.00 Hz
GB                        0
PC                    1.40

1D NMR plot parameters
CX                 20.00 cm
CY                  4.00 cm
F1P               228.275 ppm
F1              28707.46 Hz
F2P               -10.517 ppm
F2              -1322.57 Hz
PPMCM          11.93963 ppm/cm
HZCM         1501.50159 Hz/cm
```



6c

Table 2, Entry 3

```
NAME                 j100206
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameter
Date_               20061002
Time                   15.25
INSTRUM                spect
PROBHD   5 mm PHBBO BB-
PULPROG                   zg
TD                     32768
SOLVENT                CDCl3
NS                         8
DS                         0
SWH              17985.611 Hz
FIDRES           0.548877 Hz
AQ               0.9110282 se
RG                       181
DW                  27.800 us
DE                   7.00 us
TE                   301.2 K
D1              1.00000000 se

======== CHANNEL f1 ======
NUC1                      1H
P1                    5.00 us
PL1                  -4.00 dB
SFO1          500.1319002 MH

F2 - Processing parameters
SI                     32768
SF              500.1300233 MH
WDW                       EM
SSB                        0
LB                   0.20 Hz
GB                         0
PC                      4.00

1D NMR plot parameters
CX                   20.00 cm
CY                   10.00 cm
F1P                   8.622 pp
F1                 4311.97 Hz
F2P                   0.122 pp
F2                   61.21 Hz
PPMCM             0.42497 pp
HZCM             212.53781 Hz
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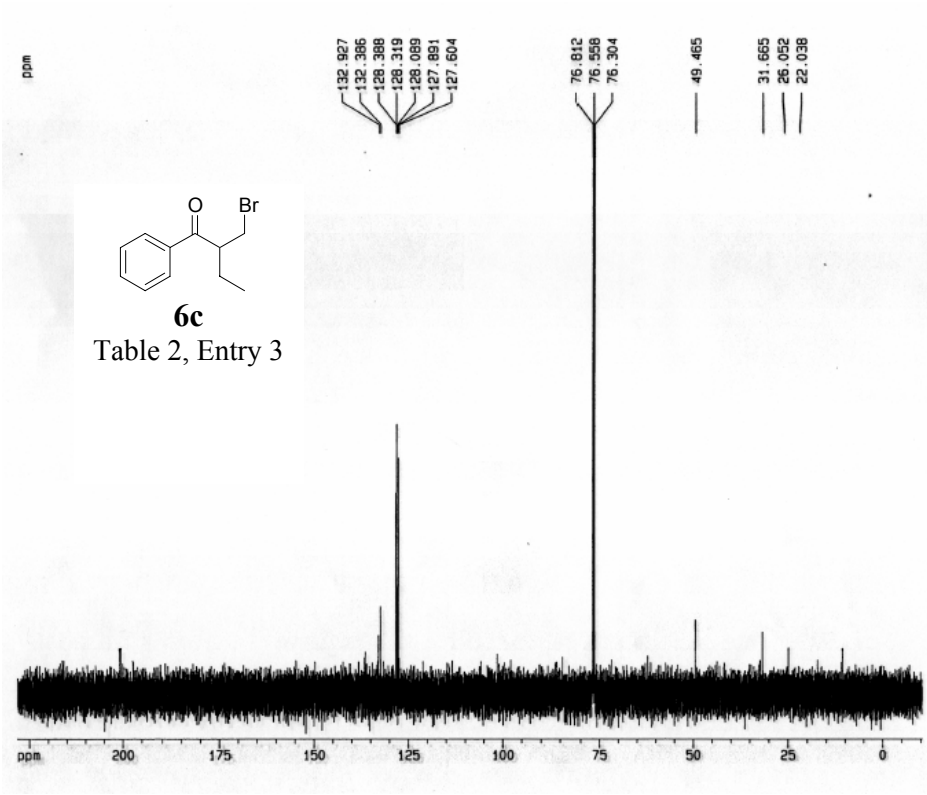


6c

Table 2, Entry 3

```
Current Data Parameters
NAME                 j100206
EXPNO                      2
PROCNO                     1

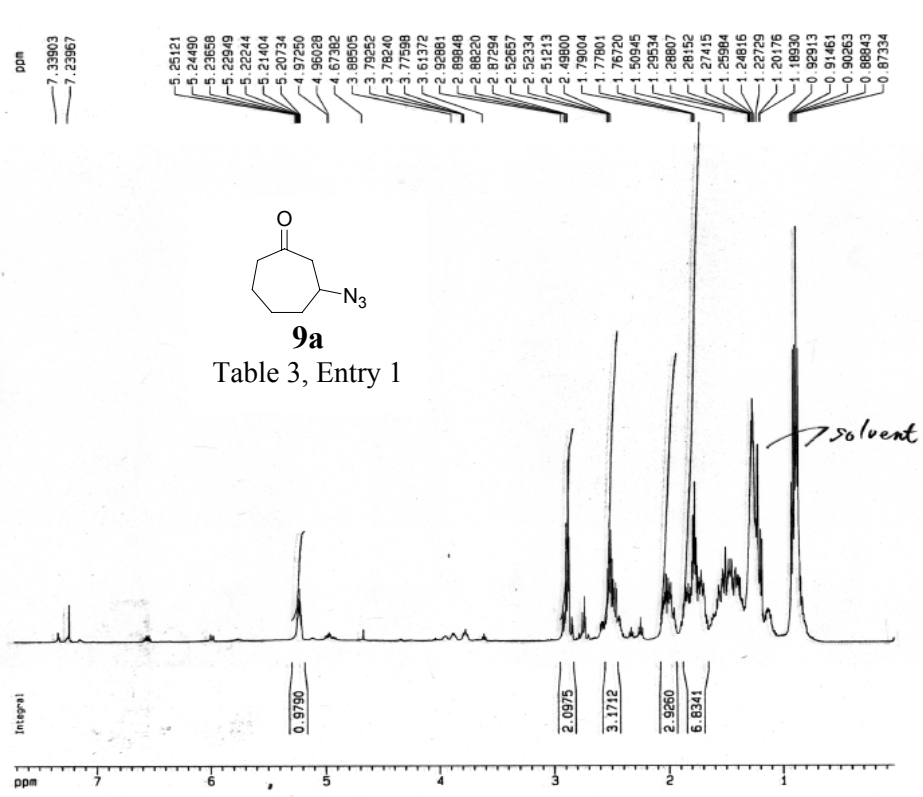
F2 - Acquisition Parameters
Date_               20061002
Time                   15.38
INSTRUM                spect
PROBHD   5 mm PHBBO BB-
PULPROG             zgdc30
TD                     65536
SOLVENT                CDCl3
NS                       217
DS                         0
SWH              30030.029 Hz
FIDRES           0.458222 Hz
AQ               1.0912410 sec
RG                      4096
DW                  16.650 usec
DE                   7.00 usec
TE                   301.9 K
D1              1.00000000 sec
d11             0.03000000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                   7.80 usec
PL1                  1.00 dB
SFO1          125.7715390 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2              100.00 usec
PL2                  17.50 dB
PL12                 17.50 dB
SFO2          500.1325000 MHz

F2 - Processing parameters
SI                     65536
SF              125.7578466 MHz
WDW                       no
SSB                        0
LB                    0.00 Hz
GB                         0
PC                      1.40

1D NMR plot parameters
CX                   20.00 cm
CY                    6.00 cm
F1P                 228.276 ppm
F1                28707.46 Hz
F2P                -10.517 ppm
F2                -1322.57 Hz
PPMCM            11.93963 ppm/cm
HZCM             1501.50159 Hz/cm
```



9a

Table 3, Entry 1

```
NAME                j092706
EXPNO                     4
PROCNO                    1

F2 - Acquisition Parameter
Date_              20061027
Time                 16.09
INSTRUM              spect
PROBHD   5 mm PHBBO BB-
PULPROG                 zg
TD                   32768
SOLVENT              CDCl3
NS                       8
DS                       0
SWH              17985.611 Hz
FIDRES           0.548877 Hz
AQ               0.9110282 se
RG                    50.8
DW                27.800 us
DE                 7.00 us
TE                300.9 K
D1             1.00000000 se

======== CHANNEL f1 ======
NUC1                    1H
P1                 5.00 us
PL1               -4.00 dB
SF01        500.1319002 MH

F2 - Processing parameters
SI                   32768
SF          500.1300233 MH
WDW                     EM
SSB                      0
LB                0.20 Hz
GB                       0
PC                4.00

1D NMR plot parameters
CX                20.00 cm
CY                10.00 cm
F1P               7.725 pp
F1              3863.39 Hz
F2P               0.037 pp
F2                18.49 Hz
PPMCM             0.38439 pp
HZCM            192.24525 Hz
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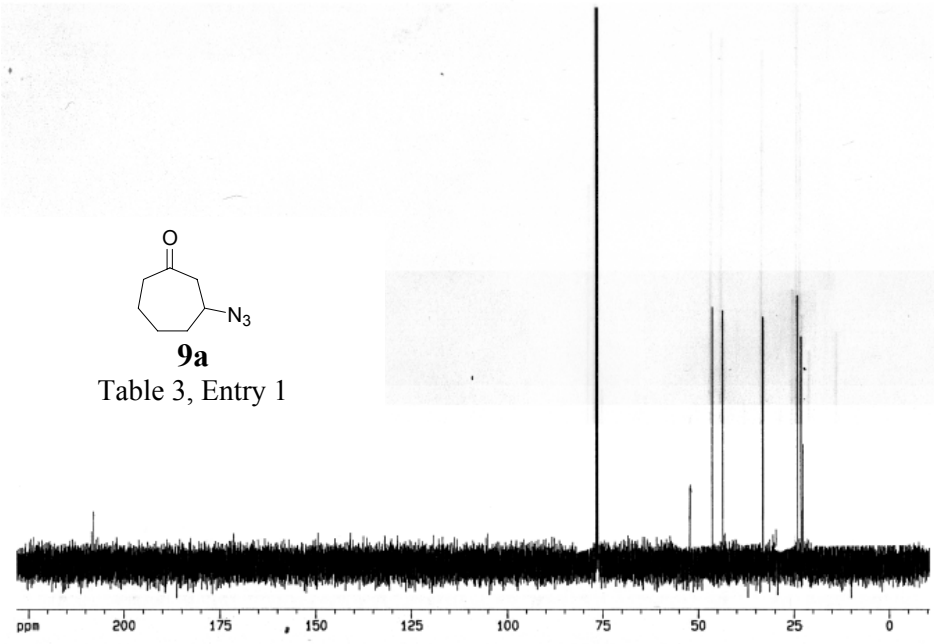
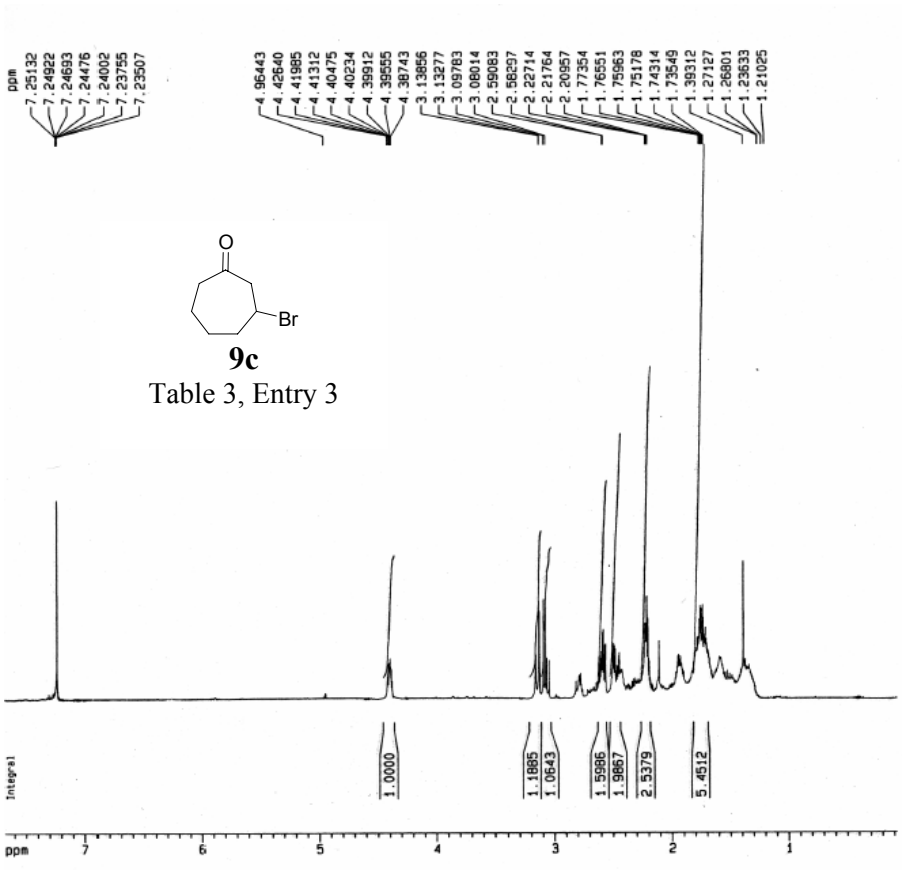


9a

Table 3, Entry 1

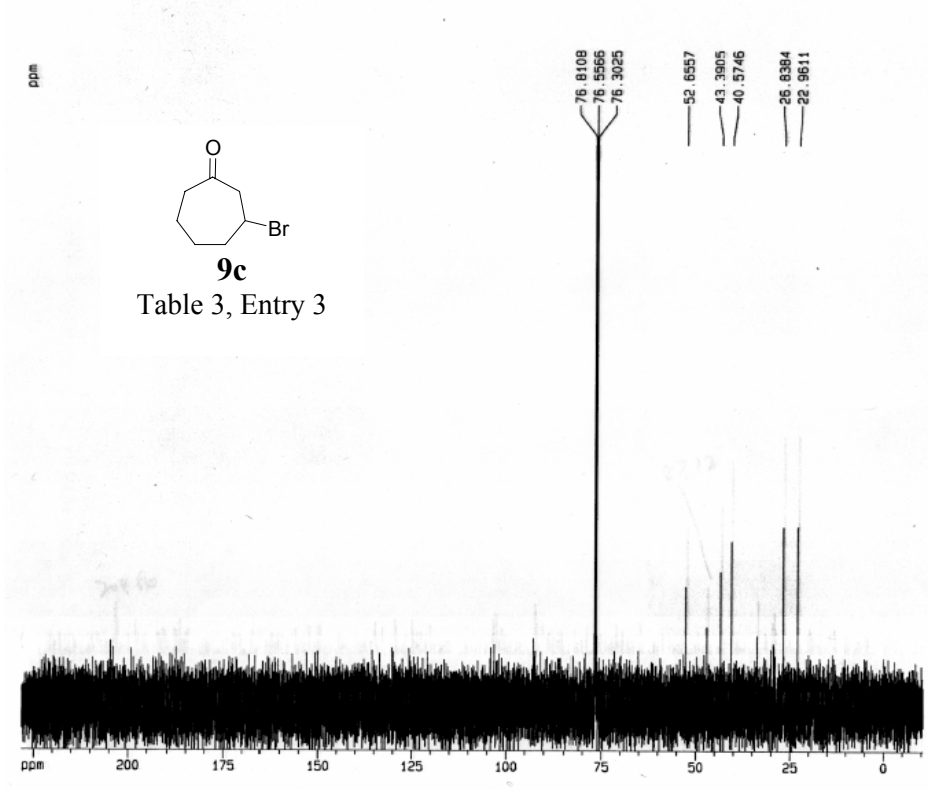
```
F2 - Acquisition Parameters
Date_              20061027
Time                 16.17
INSTRUM              spect
PROBHD   5 mm PHBBO BB-
PULPROG           zgdc30
TD                   65536
SOLVENT              CDCl3
NS                     109
DS                       0
SWH              30030.029 Hz
FIDRES           0.458222 Hz
AQ               1.0912410 sec
RG                    8502
DW                16.650 usec
DE                 7.00 usec
TE                301.8 K
D1             1.00000000 sec
d11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                 7.80 usec
PL1                1.00 dB
SF01        125.7715390 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2            100.00 usec
PL2                17.50 dB
PL12               17.50 dB
SF02        500.1325000 MHz

F2 - Processing parameters
SI                   65536
SF          125.7578466 MHz
WDW                     no
SSB                      0
LB                0.00 Hz
GB                       0
PC                1.40

1D NMR plot parameters
CX                20.00 cm
CY                 6.00 cm
F1P              228.276 ppm
F1             28707.46 Hz
F2P             -10.517 ppm
F2             -1322.57 Hz
PPMCM          11.93963 ppm/cm
HZCM         1501.50159 Hz/cm
```



9c

Table 3, Entry 3

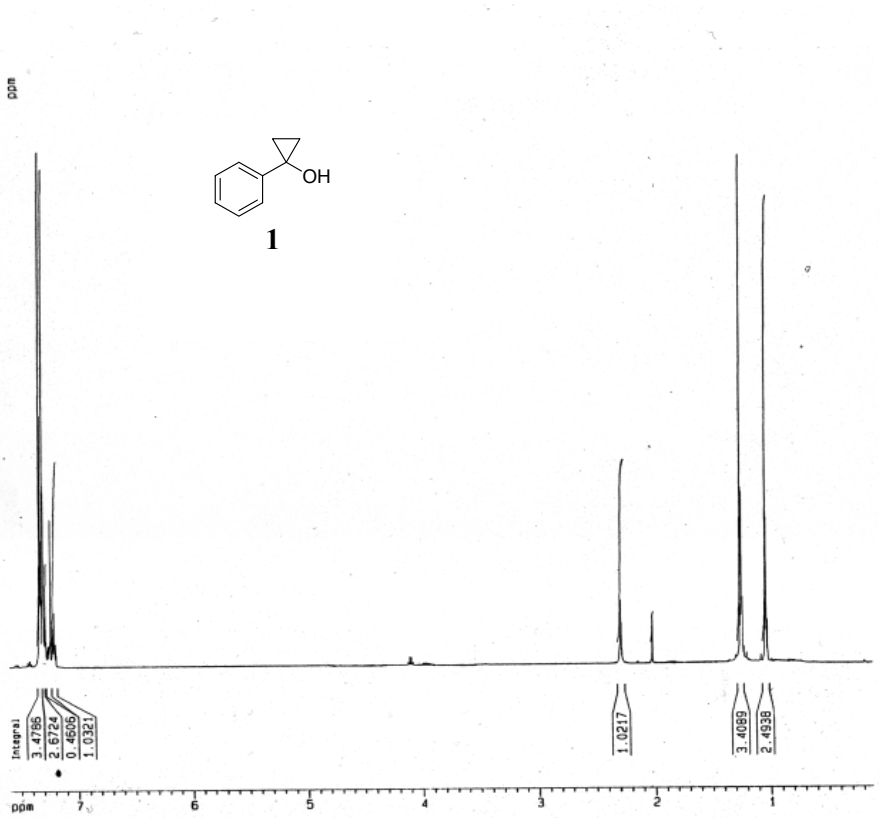
```
Current Data Parameters
NAME                  j092706
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameter
Date_              20061027
Time                   15.50
INSTRUM                spect
PROBHD   5 mm PHBBO BB-
PULPROG                   zg
TD                     32768
SOLVENT                CDCl3
NS                         8
DS                         0
SWH               17985.611 Hz
FIDRES             0.548877 Hz
AQ                 0.9110282 sec
RG                       181
DW                    27.800 usec
DE                     7.00 usec
TE                    301.0 K
D1                1.00000000 sec

======== CHANNEL f1 ======
NUC1                      1H
P1                     5.00 usec
PL1                   -4.00 dB
SFO1             500.1319002 MHz

F2 - Processing parameters
SI                     32768
SF               500.1300233 MHz
WDW                       EM
SSB                        0
LB                      0.20 Hz
GB                         0
PC                      4.00

1D NMR plot parameters
CX                     20.00 cm
CY                     10.00 cm
F1P                    7.682 ppm
F1                  3842.03 Hz
F2P                    0.080 ppm
F2                     39.85 Hz
PPMCM                 0.38012 ppm/cm
HZCM                190.10921 Hz
```



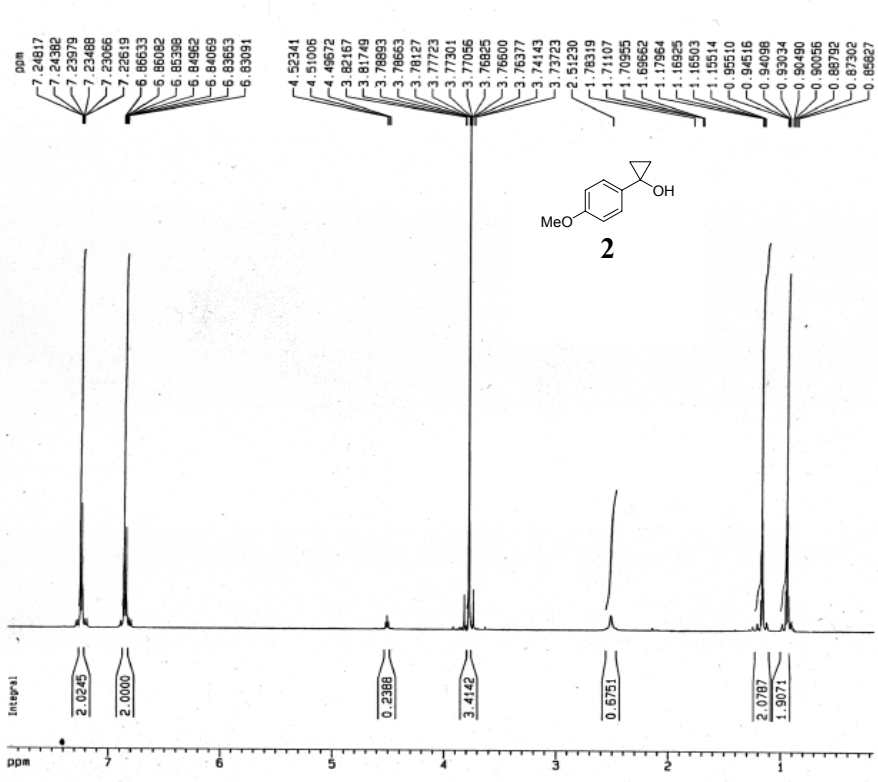
9c

Table 3, Entry 3

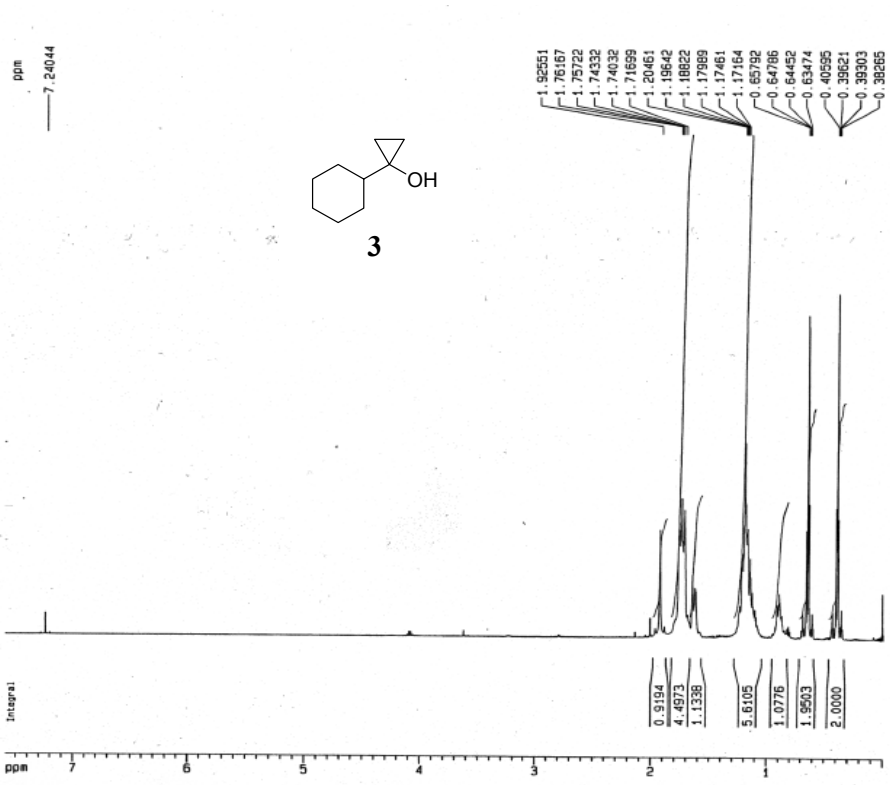
```
Current Data Parameters
NAME                  j092706
EXPNO                       3
PROCNO                      1

F2 - Acquisition Parameters
Date_              20061027
Time                   15.58
INSTRUM                spect
PROBHD   5 mm PHBBO BB-
PULPROG                zgdc30
TD                     65536
SOLVENT                CDCl3
NS                       208
DS                         0
SWH               30030.029 Hz
FIDRES             0.458222 Hz
AQ                 1.0912410 sec
RG                    14596.5
DW                   16.650 usec
DE                    7.00 usec
TE                    301.7 K
D1                1.00000000 sec
d11               0.03000000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                    7.80 usec
PL1                   1.00 dB
SFO1             125.7715390 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                      1H
PCPD2                100.00 usec
PL2                   17.50 dB
PL12                  17.50 dB
SFO2             500.1325000 MHz

F2 - Processing parameters
SI                     65536
SF               125.7578466 MHz
WDW                       no
SSB                        0
LB                      0.00 Hz
GB                         0
PC                      1.40

1D NMR plot parameters
CX                     20.00 cm
CY                      4.00 cm
F1P                  228.276 ppm
F1                 28707.46 Hz
F2P                  -10.517 ppm
F2                 -1322.57 Hz
PPMCM               11.93963 ppm/cm
HZCM             1501.50159 Hz/cm
```




```
Current Data Parameters
NAME                  j041306
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameter
Date_              20060413
Time                   10.47
INSTRUM                spect
PROBHD   5 mm PHBBO BB-
PULPROG                   zg
TD                     32768
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                5000.000 Hz
FIDRES             0.152588 Hz
AQ                 3.2769499 se
RG                       128
DW                 100.000 us
DE                   7.00 us
TE                   300.4 K
D1                1.00000000 se

======== CHANNEL f1 ======
NUC1                       1H
P1                   5.00 us
PL1                  -4.00 dB
SFO1            500.1319002 MH

F2 - Processing parameters
SI                     32768
SF                500.1300233 MH
WDW                       EM
SSB                        0
LB                   0.20 Hz
GB                         0
PC                      4.00

1D NMR plot parameters
CX                   20.00 cm
CY                  150.00 cm
F1P                   7.599 pp
F1                 3800.54 Hz
F2P                   0.124 pp
F2                  62.22 Hz
PPMCM                0.37373 pp
HZCM               186.91589 Hz
```

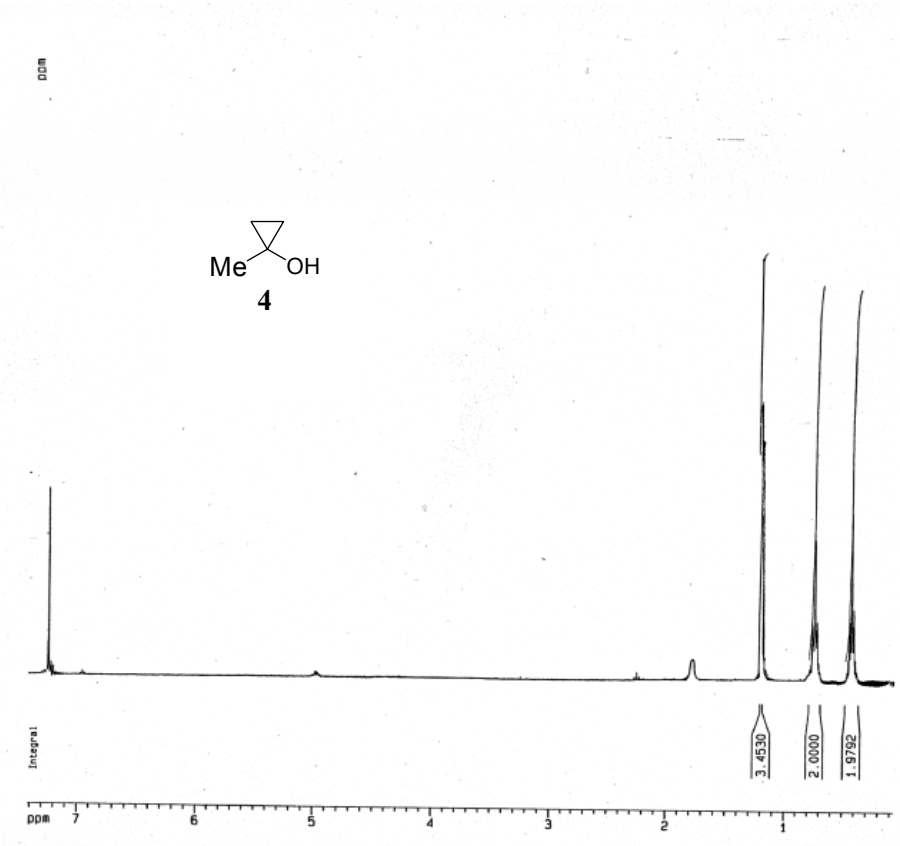



```
Current Data Parameters
NAME                  j060906
EXPNO                       3
PROCNO                      1

F2 - Acquisition Parameter
Date_              20060609
Time                   14.41
INSTRUM                spect
PROBHD   5 mm PHBBO BB-
PULPROG                   zg
TD                     32768
SOLVENT                CDCl3
NS                         8
DS                         0
SWH               17985.611 Hz
FIDRES             0.548877 Hz
AQ                 0.9110282 se
RG                      50.8
DW                  27.800 us
DE                   7.00 us
TE                   299.5 K
D1                1.00000000 se

======== CHANNEL f1 ======
NUC1                       1H
P1                   5.00 us
PL1                  -4.00 dB
SFO1            500.1319002 MH

F2 - Processing parameters
SI                     32768
SF                500.1300233 MH
WDW                       EM
SSB                        0
LB                   0.20 Hz
GB                         0
PC                      4.00

1D NMR plot parameters
CX                   20.00 cm
CY                   13.00 cm
F1P                   7.896 pp
F1                 3948.84 Hz
F2P                   0.165 pp
F2                  82.57 Hz
PPMCM                0.38653 pp
HZCM               193.31329 Hz
```



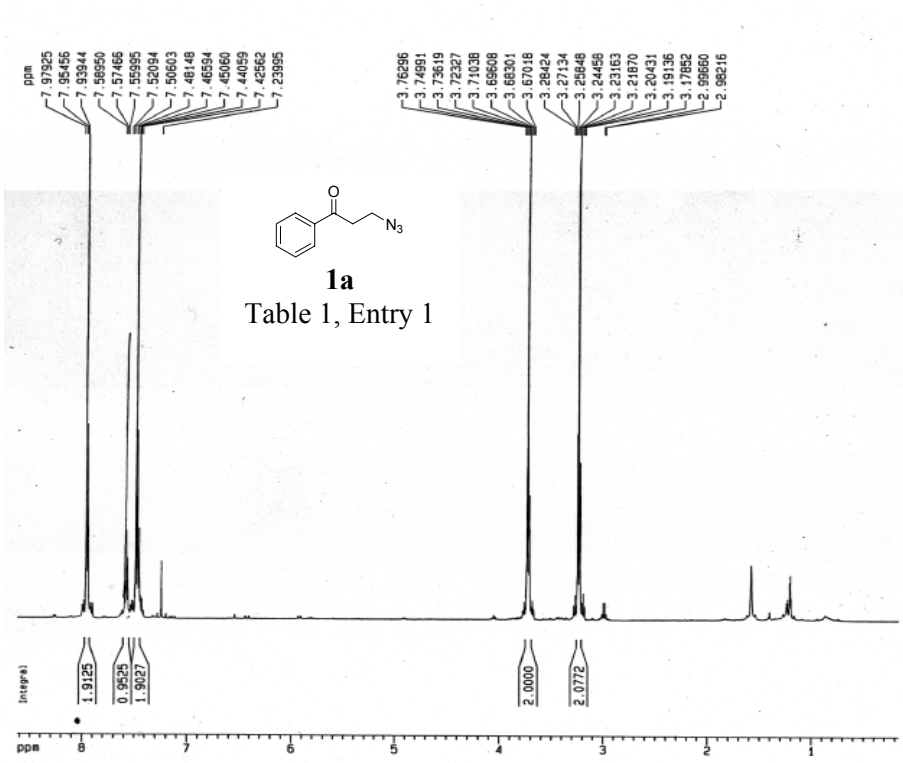
3

```
Current Data Parameters
NAME                  j062606
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameter
Date_                20060626
Time                    11.36
INSTRUM                 spect
PROBHD   5 mm PHBBO BB-
PULPROG                    zg
TD                      32768
SOLVENT                 CDCl3
NS                          8
DS                          0
SWH                17985.611 Hz
FIDRES              0.548877 Hz
AQ                  0.9110282 se
RG                       28.5
DW                     27.800 us
DE                      7.00 us
TE                      299.7 K
D1                 1.00000000 se

======== CHANNEL f1 ======
NUC1                       1H
P1                      5.00 us
PL1                    -4.00 dB
SFO1              500.1319002 MH

F2 - Processing parameters
SI                      32768
SF                500.1300233 MH
WDW                        EM
SSB                         0
LB                   0.20 Hz
GB                          0
PC                       4.00

1D NMR plot parameters
CX                      20.00 cm
CY                      13.00 cm
F1P                     7.586 pp
F1                   3794.14 Hz
F2P                    -0.001 pp
F2                     -0.54 Hz
PPMCM                0.37937 pp
HZCM               189.73425 Hz
```



4

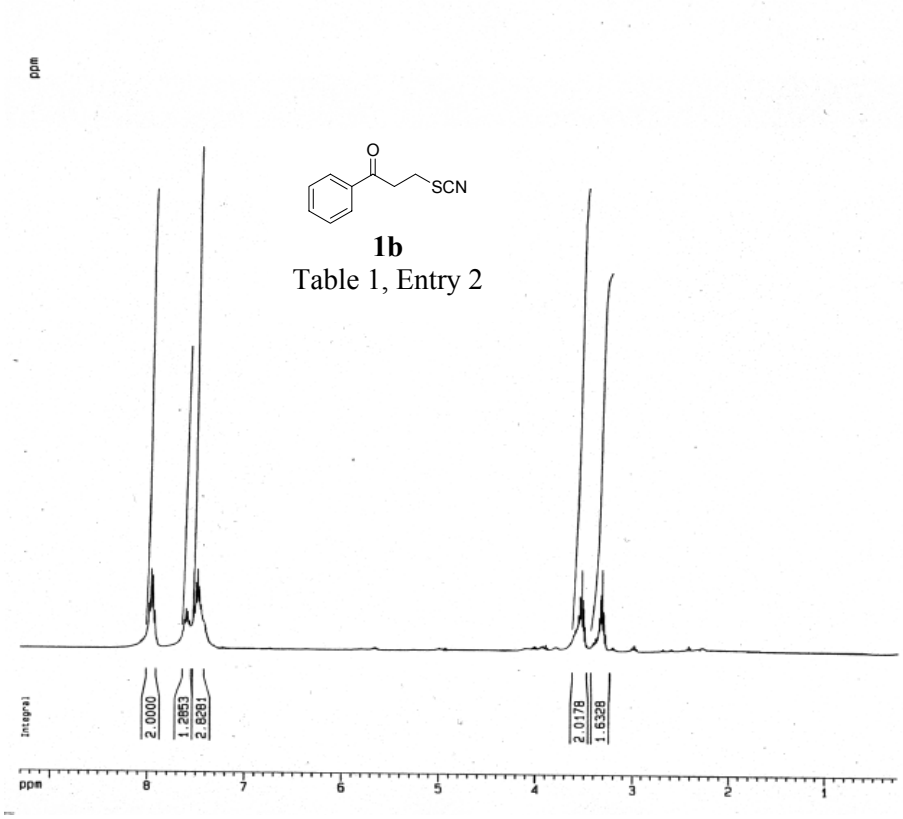
```
Current Data Parameters
NAME                  j072406
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameter
Date_                20060724
Time                    15.17
INSTRUM                 spect
PROBHD   5 mm PHBBO BB-
PULPROG                    zg
TD                      32768
SOLVENT                 CDCl3
NS                          8
DS                          0
SWH                17985.611 Hz
FIDRES              0.548877 Hz
AQ                  0.9110282 se
RG                        114
DW                     27.800 us
DE                      7.00 us
TE                      299.7 K
D1                 1.00000000 se

======== CHANNEL f1 ======
NUC1                       1H
P1                      5.00 us
PL1                    -4.00 dB
SFO1              500.1319002 MH

F2 - Processing parameters
SI                      32768
SF                500.1300233 MH
WDW                        EM
SSB                         0
LB                   0.20 Hz
GB                          0
PC                       4.00

1D NMR plot parameters
CX                      20.00 cm
CY                     200.00 cm
F1P                     7.410 pp
F1                   3706.21 Hz
F2P                     0.066 pp
F2                     32.92 Hz
PPMCM                0.36723 pp
HZCM               183.66426 Hz
```



1a

Table 1, Entry 1

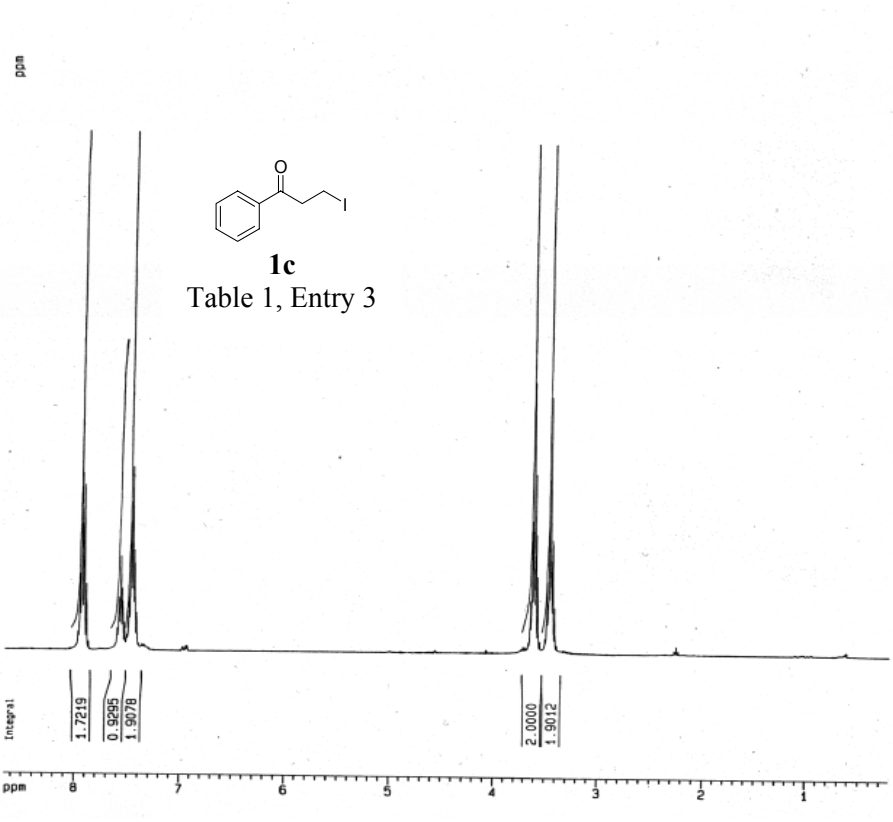
```
Current Data Parameters
NAME                  j061506
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameter
Date_              20060615
Time                  10.22
INSTRUM               spect
PROBHD    5 mm PHBBO BB-
PULPROG                 zg
TD                   32768
SOLVENT              CDCl3
NS                       8
DS                       0
SWH              17985.611 Hz
FIDRES               0.548877 Hz
AQ                 0.9110282 sec
RG                     181
DW                   27.800 usec
DE                    7.00 usec
TE                   299.3 K
D1                1.00000000 sec

======== CHANNEL f1 ======
NUC1                    1H
P1                    5.00 usec
PL1                  -4.00 dB
SFO1           500.1319002 MHz

F2 - Processing parameters
SI                   32768
SF              500.1300233 MHz
WDW                     EM
SSB                      0
LB                    0.20 Hz
GB                       0
PC                    4.00

1D NMR plot parameters
CX                   20.00 cm
CY                   40.00 cm
F1P                  8.622 ppm
F1                 4311.97 Hz
F2P                  0.165 ppm
F2                  82.57 Hz
PPMCM                0.42283 ppm
HZCM               211.46980 Hz
```



1b

Table 1, Entry 2

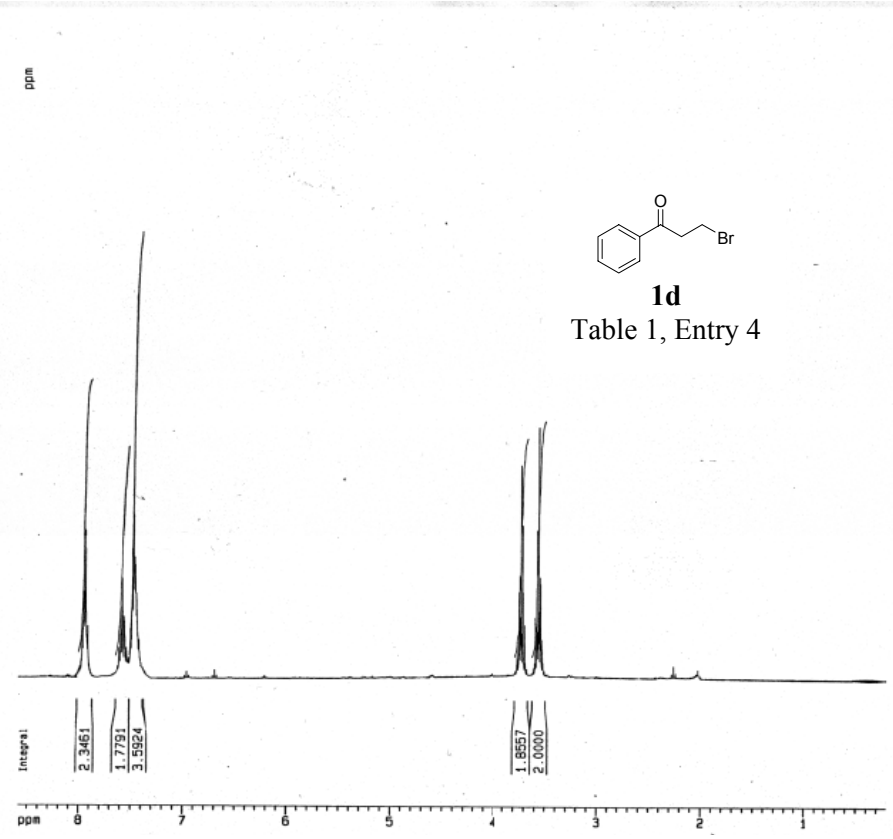
```
Current Data Parameters
NAME                  j072606
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameter
Date_              20060726
Time                  14.25
INSTRUM               spect
PROBHD    5 mm PHBBO BB-
PULPROG                 zg
TD                   32768
SOLVENT              CDCl3
NS                       8
DS                       0
SWH              17985.611 Hz
FIDRES               0.548877 Hz
AQ                 0.9110282 sec
RG                      64
DW                   27.800 usec
DE                    7.00 usec
TE                   297.4 K
D1                1.00000000 sec

======== CHANNEL f1 ======
NUC1                    1H
P1                    5.00 usec
PL1                  -4.00 dB
SFO1           500.1319002 MHz

F2 - Processing parameters
SI                   32768
SF              500.1300233 MHz
WDW                     EM
SSB                      0
LB                    0.20 Hz
GB                       0
PC                    4.00

1D NMR plot parameters
CX                   20.00 cm
CY                   13.00 cm
F1P                  9.305 ppm
F1                 4653.74 Hz
F2P                  0.251 ppm
F2                  125.29 Hz
PPMCM                0.45273 ppm
HZCM               226.42220 Hz
```



1c

Table 1, Entry 3

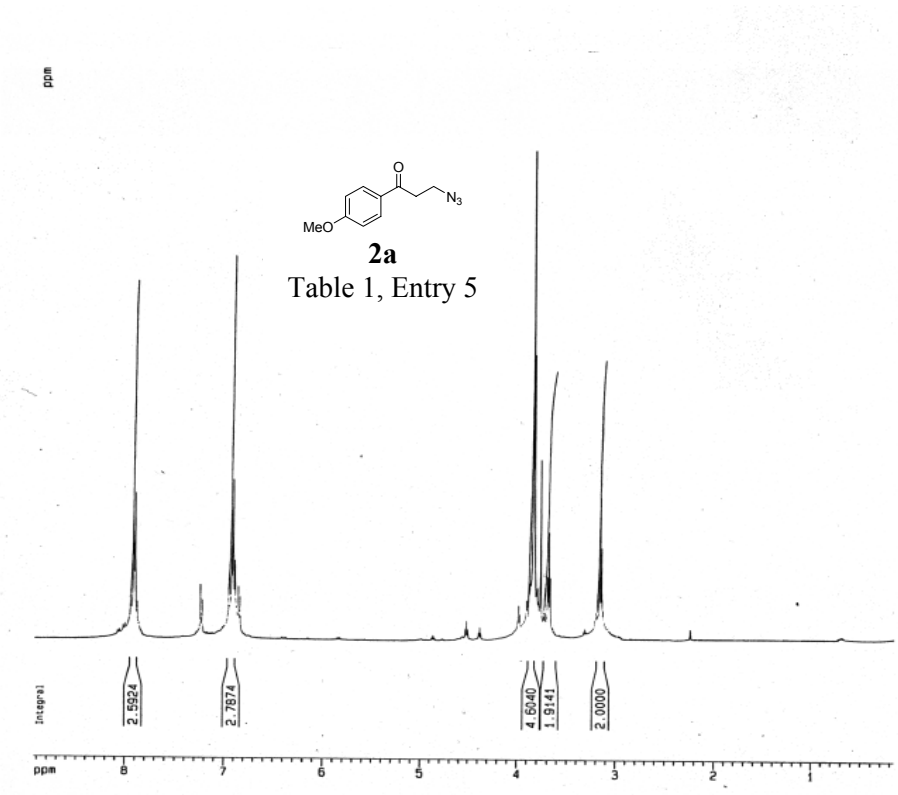
```
Current Data Parameters
NAME                  j079006
EXPNO                       3
PROCNO                      1

F2 - Acquisition Parameter
Date_                20060710
Time                    16.26
INSTRUM                 spect
PROBHD   5 mm PHBBO BB-
PULPROG                    zg
TD                      32768
SOLVENT                 CDCl3
NS                          8
DS                          0
SWH                17985.611 Hz
FIDRES              0.548877 Hz
AQ                  0.9110282 se
RG                       50.8
DW                    27.800 us
DE                     7.00 us
TE                     299.4 K
D1                1.00000000 se

======== CHANNEL f1 ======
NUC1                       1H
P1                     5.00 us
PL1                   -4.00 dB
SFO1           500.1319002 MH

F2 - Processing parameters
SI                      32768
SF             500.1300233 MH
WDW                        EM
SSB                         0
LB                   0.20 Hz
GB                          0
PC                       4.00

1D NMR plot parameters
CX                    20.00 cm
CY                    13.00 cm
F1P                    8.664 pp
F1                  4333.33 Hz
F2P                    0.208 pp
F2                    103.93 Hz
PPMCM                 0.42283 pp
HZCM                211.46980 Hz
```



1d

Table 1, Entry 4

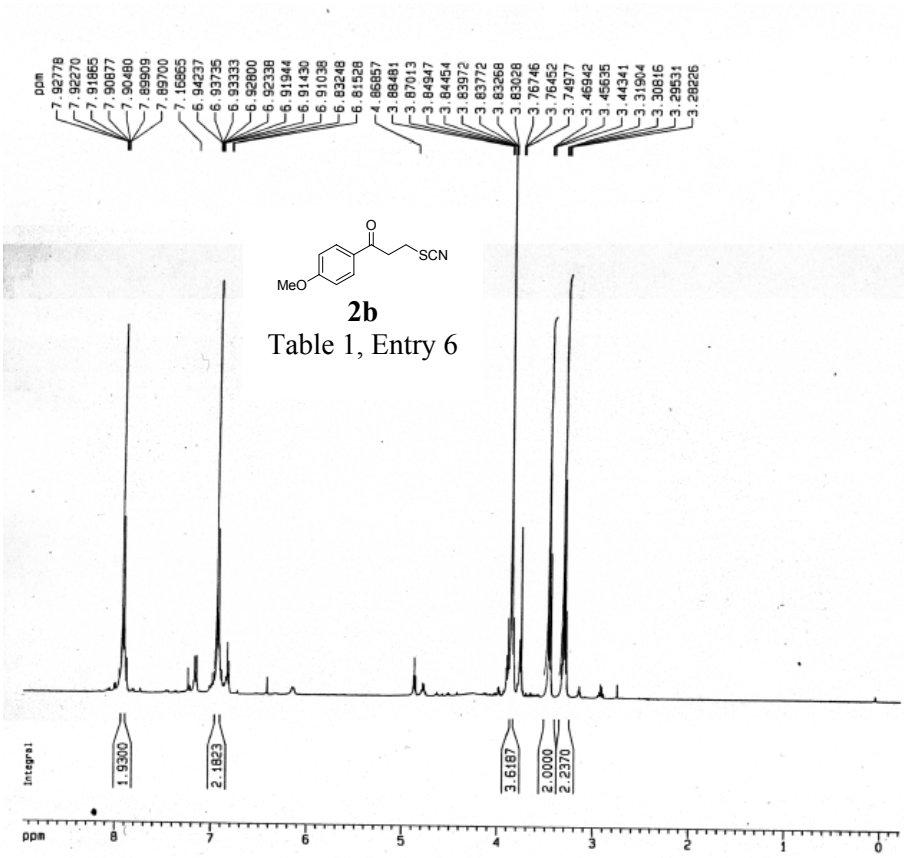
```
Current Data Parameters
NAME                  j072406
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameter
Date_                20060724
Time                    15.27
INSTRUM                 spect
PROBHD   5 mm PHBBO BB-
PULPROG                    zg
TD                      32768
SOLVENT                 CDCl3
NS                          8
DS                          0
SWH                17985.611 Hz
FIDRES              0.548877 Hz
AQ                  0.9110282 se
RG                       50.8
DW                    27.800 us
DE                     7.00 us
TE                     299.5 K
D1                1.00000000 se

======== CHANNEL f1 ======
NUC1                       1H
P1                     5.00 us
PL1                   -4.00 dB
SFO1           500.1319002 MH

F2 - Processing parameters
SI                      32768
SF             500.1300233 MH
WDW                        EM
SSB                         0
LB                   0.20 Hz
GB                          0
PC                       4.00

1D NMR plot parameters
CX                    20.00 cm
CY                    40.00 cm
F1P                    8.579 pp
F1                  4290.61 Hz
F2P                    0.208 pp
F2                    103.93 Hz
PPMCM                 0.41856 pp
HZCM                209.33372 Hz
```



2a

Table 1, Entry 5

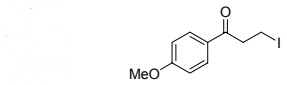
```
Current Data Parameters
NAME                 j061906
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameter
Date_               20060619
Time                   16.00
INSTRUM                spect
PROBHD   5 mm PHBBO BB-
PULPROG                  zg
TD                    32768
SOLVENT               CDCl3
NS                         8
DS                         0
SWH               17985.611 Hz
FIDRES             0.548877 Hz
AQ                 0.9110282 se
RG                      28.5
DW                    27.800 us
DE                     7.00 us
TE                    298.9 K
D1                1.00000000 se

======== CHANNEL f1 ======
NUC1                      1H
P1                     5.00 us
PL1                   -4.00 dB
SFO1           500.1319002 MH

F2 - Processing parameters
SI                        32768
SF                500.1300233 MH
WDW                       EM
SSB                        0
LB                      0.20 Hz
GB                         0
PC                      4.00

1D NMR plot parameters
CX                     20.00 cm
CY                     13.00 cm
F1P                     8.921 pp
F1                   4461.49 Hz
F2P                     0.165 pp
F2                     82.57 Hz
PPMCM                 0.43778 pp
HZCM                218.94600 Hz
```



2b

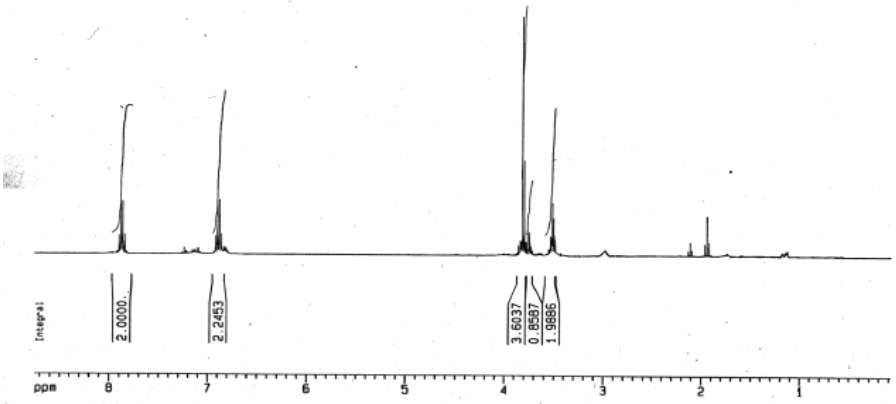
Table 1, Entry 6

```
Current Data Parameters
NAME                 j073106
EXPNO                      3
PROCNO                     1

F2 - Acquisition Parameter
Date_               20060731
Time                   15.54
INSTRUM                spect
PROBHD   5 mm PHBBO BB-
PULPROG                  zg
TD                    32768
SOLVENT               CDCl3
NS                         8
DS                         0
SWH               17985.611 Hz
FIDRES             0.548877 Hz
AQ                 0.9110282 se
RG                      45.3
DW                    27.800 us
DE                     7.00 us
TE                    299.7 K
D1                1.00000000 se

======== CHANNEL f1 ======
NUC1                      1H
P1                     5.00 us
PL1                   -4.00 dB
SFO1           500.1319002 MH

F2 - Processing parameters
SI                        32768
SF                500.1300233 MH
WDW                       EM
SSB                        0
LB                      0.20 Hz
GB                         0
PC                      4.00

1D NMR plot parameters
CX                     20.00 cm
CY                     20.00 cm
F1P                     8.963 pp
F1                   4482.85 Hz
F2P                    -0.219 pp
F2                   -109.67 Hz
PPMCM                 0.45913 pp
HZCM                229.62628 Hz
```

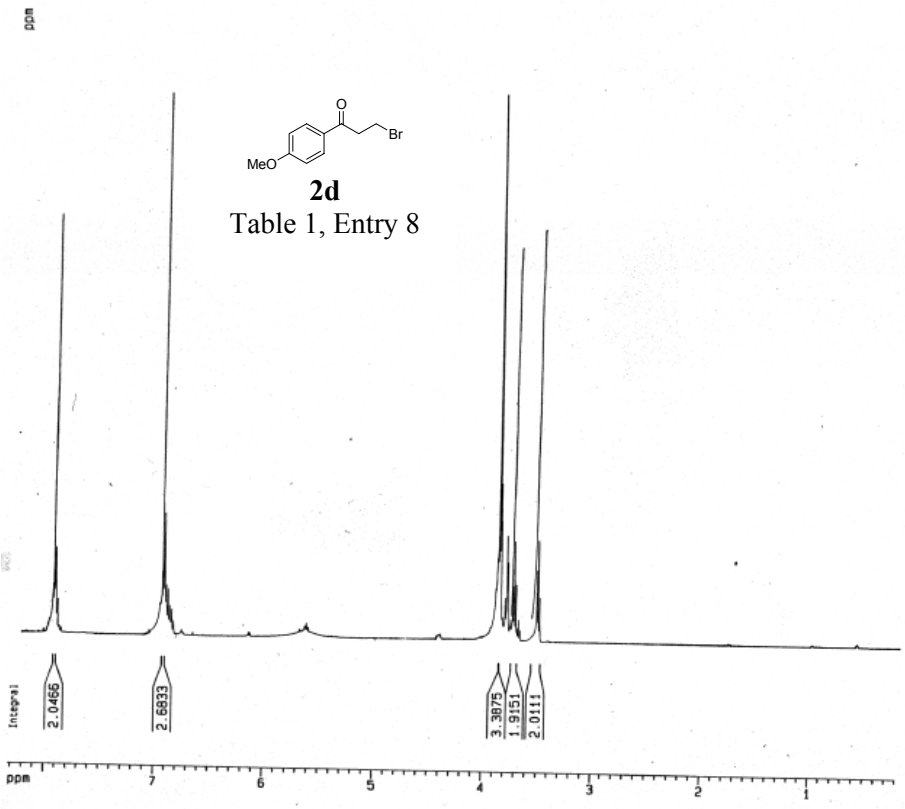


2c

Table 1, Entry 7



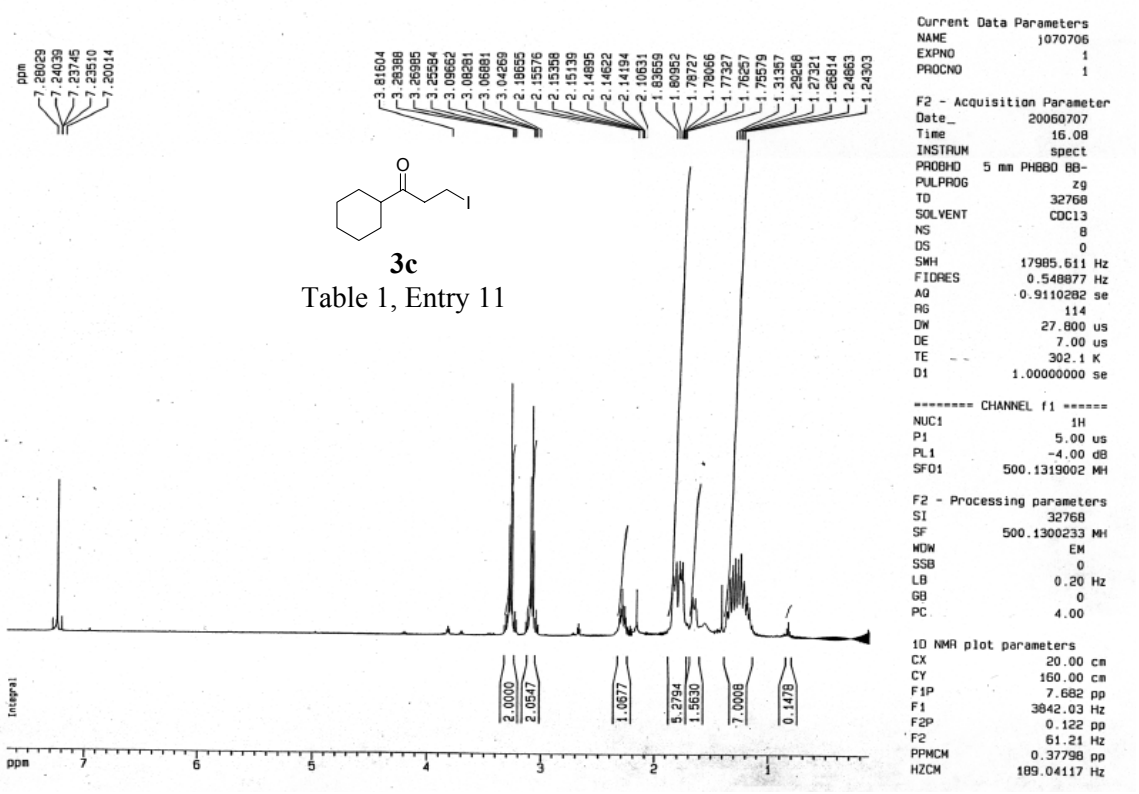

```
Current Data Parameters
NAME                  j071306
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameter
Date_                20060713
Time                    17.03
INSTRUM                 spect
PROBHD   5 mm PHBBO BB-
PULPROG                    zg
TD                      32768
SOLVENT                 CDCl3
NS                          8
DS                          0
SWH                17985.611 Hz
FIDRES              0.548877 Hz
AQ                  0.9110282 se
RG                        28.5
DW                    27.800 us
DE                     7.00 us
TE                     300.0 K
D1                1.00000000 se

======== CHANNEL f1 ======
NUC1                       1H
P1                      5.00 us
PL1                    -4.00 dB
SFO1              500.1319002 MH

F2 - Processing parameters
SI                      32768
SF                500.1300233 MH
WDW                        EM
SSB                         0
LB                     0.20 Hz
GB                          0
PC                       4.00

1D NMR plot parameters
CX                     20.00 cm
CY                     13.00 cm
F1P                     8.750 pp
F1                   4376.05 Hz
F2P                     0.080 pp
F2                     39.85 Hz
PPMCM                0.43351 pp
HZCM               216.80992 Hz
```



2d

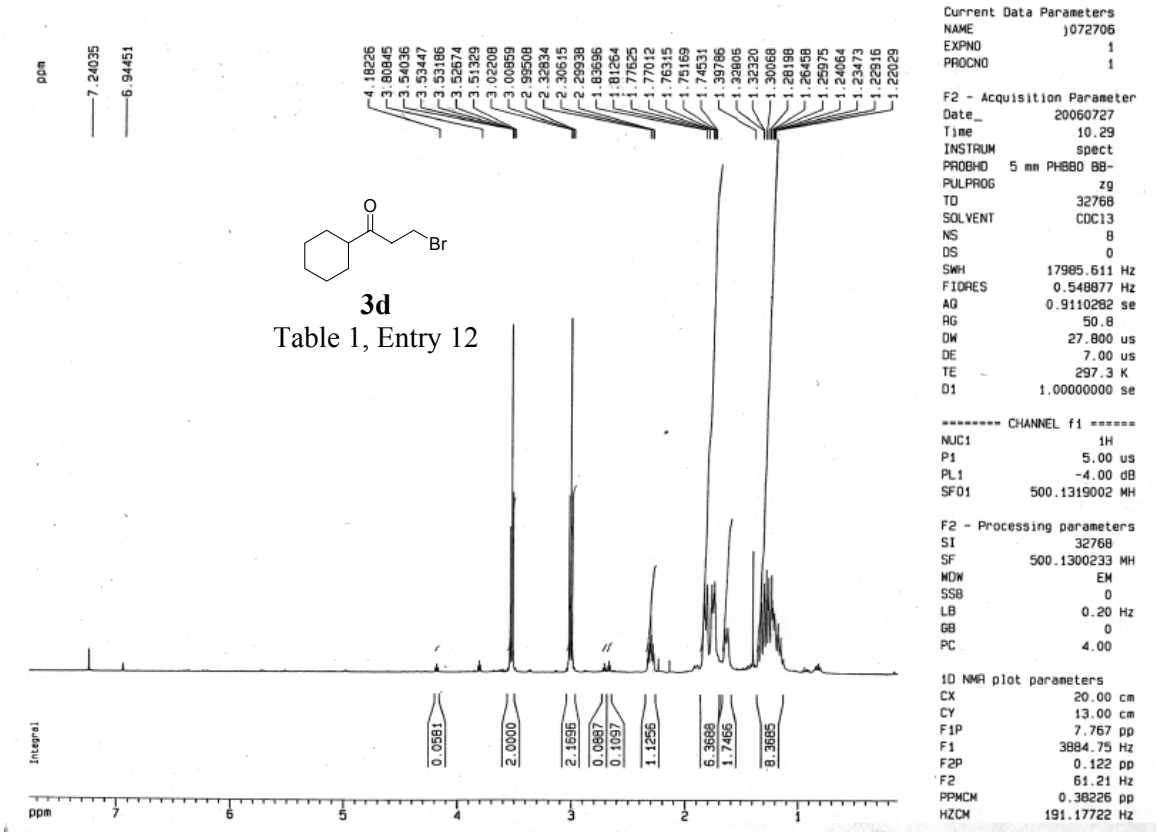
Table 1, Entry 8

```
Current Data Parameters
NAME                  j072806
EXPNO                       3
PROCNO                      1

F2 - Acquisition Parameter
Date_                20060728
Time                    16.16
INSTRUM                 spect
PROBHD   5 mm PHBBO BB-
PULPROG                    zg
TD                      32768
SOLVENT                 CDCl3
NS                          8
DS                          0
SWH                17985.611 Hz
FIDRES              0.548877 Hz
AQ                  0.9110282 se
RG                        50.8
DW                    27.800 us
DE                     7.00 us
TE                     299.7 K
D1                1.00000000 se

======== CHANNEL f1 ======
NUC1                       1H
P1                      5.00 us
PL1                    -4.00 dB
SFO1              500.1319002 MH

F2 - Processing parameters
SI                      32768
SF                500.1300233 MH
WDW                        EM
SSB                         0
LB                     0.20 Hz
GB                          0
PC                       4.00

1D NMR plot parameters
CX                     20.00 cm
CY                     13.00 cm
F1P                     8.320 pp
F1                   4161.10 Hz
F2P                     0.200 pp
F2                     99.82 Hz
PPMCM                0.40602 pp
HZCM               203.06383 Hz
```



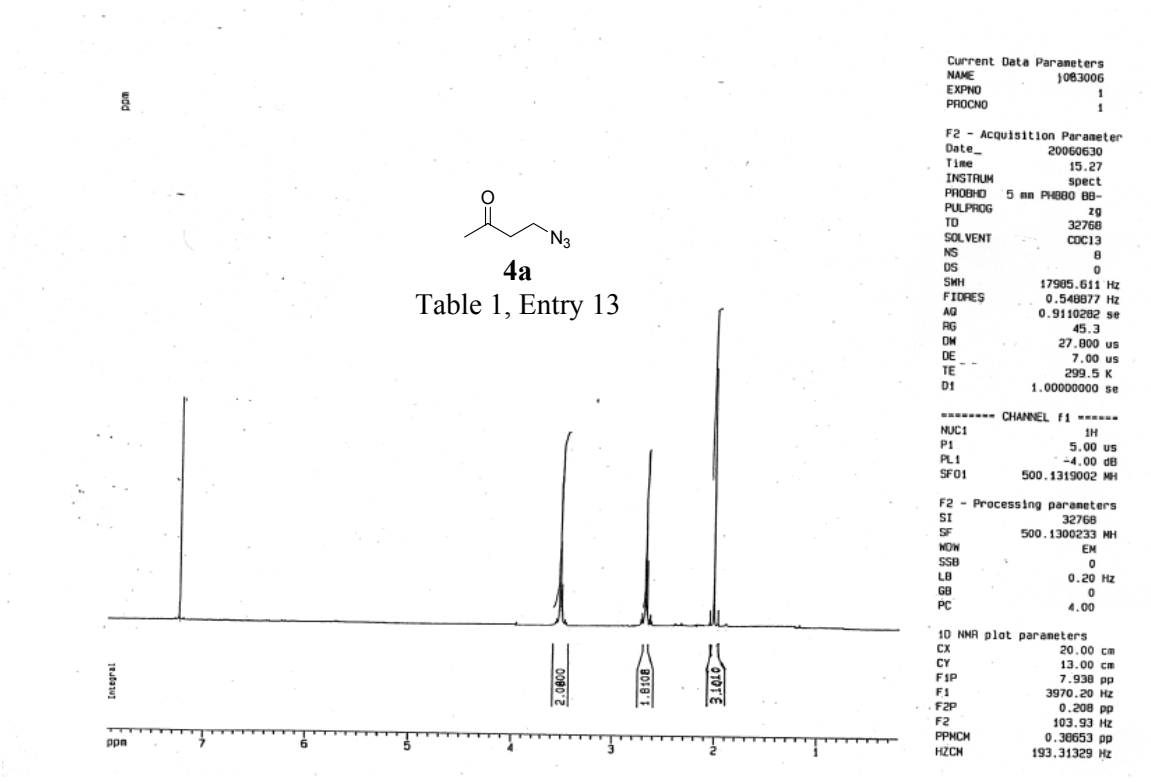
3c

Table 1, Entry 11



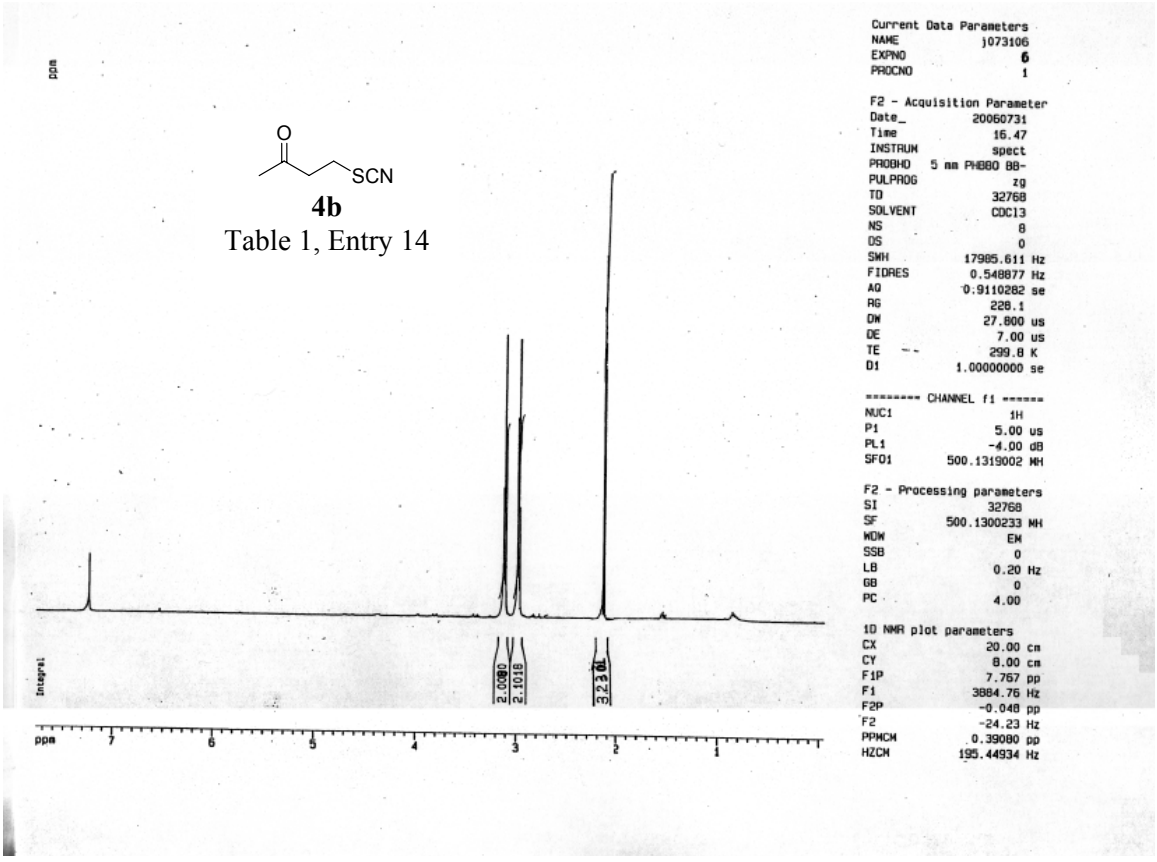
3d

Table 1, Entry 12



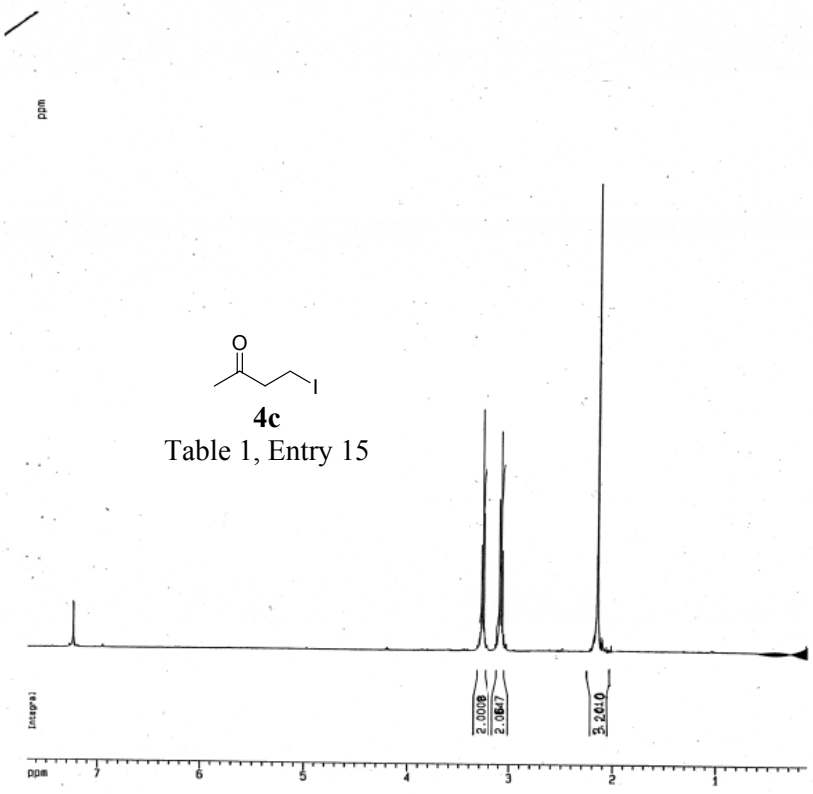
4a

Table 1, Entry 13



4b

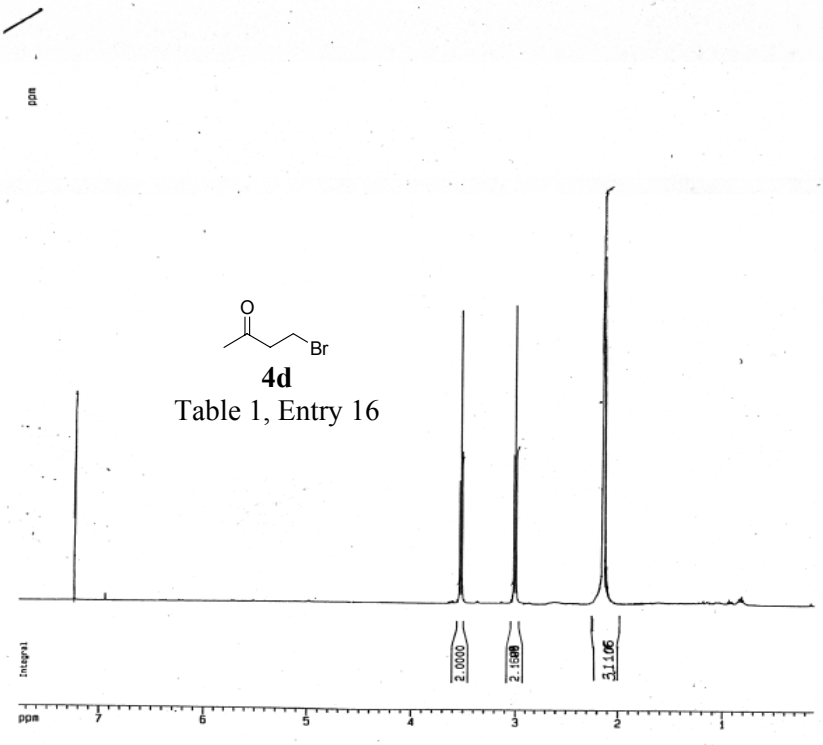
Table 1, Entry 14



4c

Table 1, Entry 15

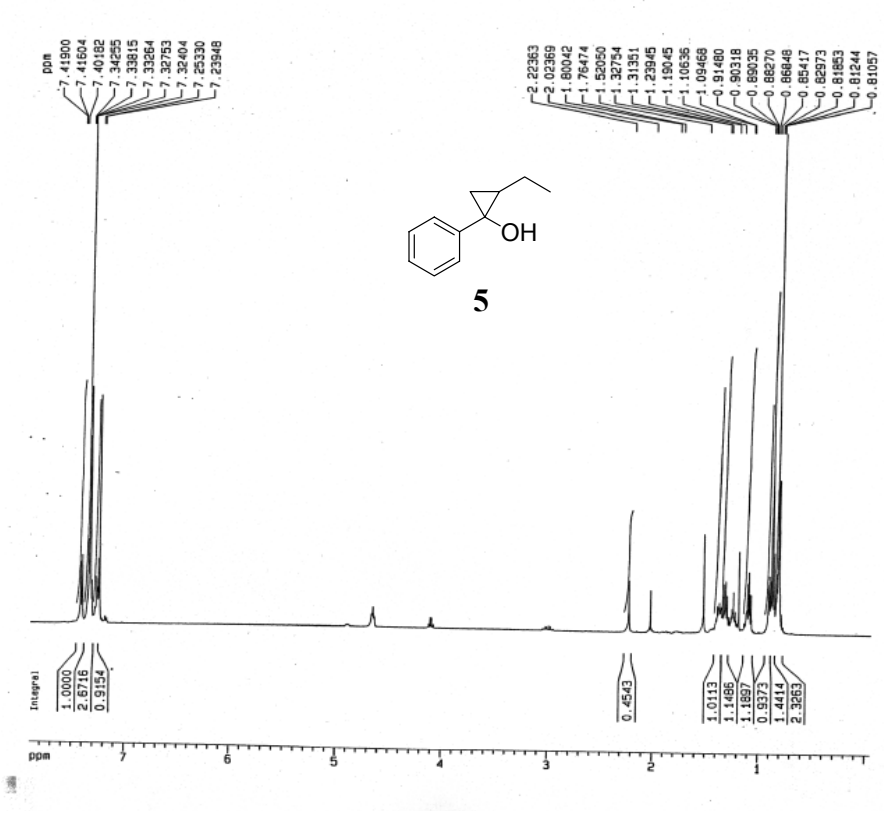
```
Current Data Parameters
NAME                 j070706
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameter
Date_              20060707
Time                  16.08
INSTRUM               spect
PROBHD   5 mm PHBBO BB-
PULPROG                   zg
TD                     32768
SOLVENT                CDCl3
NS                         8
DS                         0
SWH               17985.611 Hz
FIDRES             0.548877 Hz
AQ                 0.9110282 se
RG                       114
DW                    27.800 us
DE                     7.00 us
TE                    302.1 K
D1                 1.00000000 se

======== CHANNEL f1 ======
NUC1                      1H
P1                     5.00 us
PL1                   -4.00 dB
SFO1              500.1319002 MH

F2 - Processing parameters
SI                     32768
SF                500.1300233 MH
WDW                       EM
SSB                        0
LB                     0.20 Hz
GB                         0
PC                      4.00

1D NMR plot parameters
CX                    20.00 cm
CY                   160.00 cm
F1P                    7.682 pp
F1                  3842.03 Hz
F2P                    0.122 pp
F2                    61.21 Hz
PPMCM                 0.37798 pp
HZCM                189.04117 Hz
```



4d

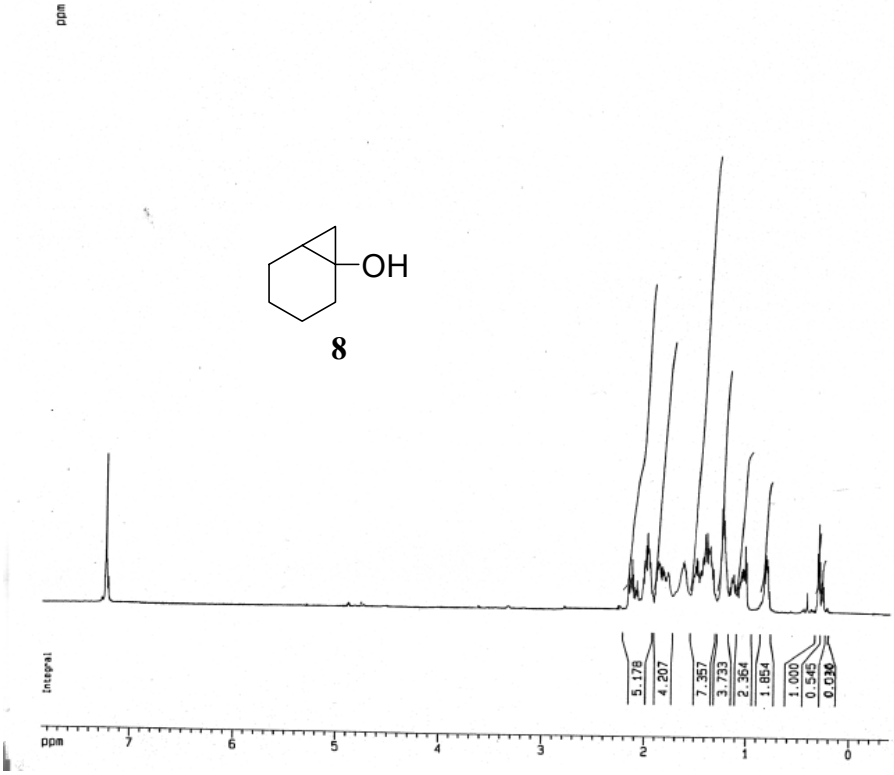
Table 1, Entry 16

```
Current Data Parameters
NAME                 j072706
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameter
Date_              20060727
Time                  10.29
INSTRUM               spect
PROBHD   5 mm PHBBO BB-
PULPROG                   zg
TD                     32768
SOLVENT                CDCl3
NS                         8
DS                         0
SWH               17985.611 Hz
FIDRES             0.548877 Hz
AQ                 0.9110282 se
RG                      50.8
DW                    27.800 us
DE                     7.00 us
TE                    297.3 K
D1                 1.00000000 se

======== CHANNEL f1 ======
NUC1                      1H
P1                     5.00 us
PL1                   -4.00 dB
SFO1              500.1319002 MH

F2 - Processing parameters
SI                     32768
SF                500.1300233 MH
WDW                       EM
SSB                        0
LB                     0.20 Hz
GB                         0
PC                      4.00

1D NMR plot parameters
CX                    20.00 cm
CY                    13.00 cm
F1P                    7.767 pp
F1                  3884.75 Hz
F2P                    0.122 pp
F2                    61.21 Hz
PPMCM                 0.38226 pp
HZCM                191.17722 Hz
```



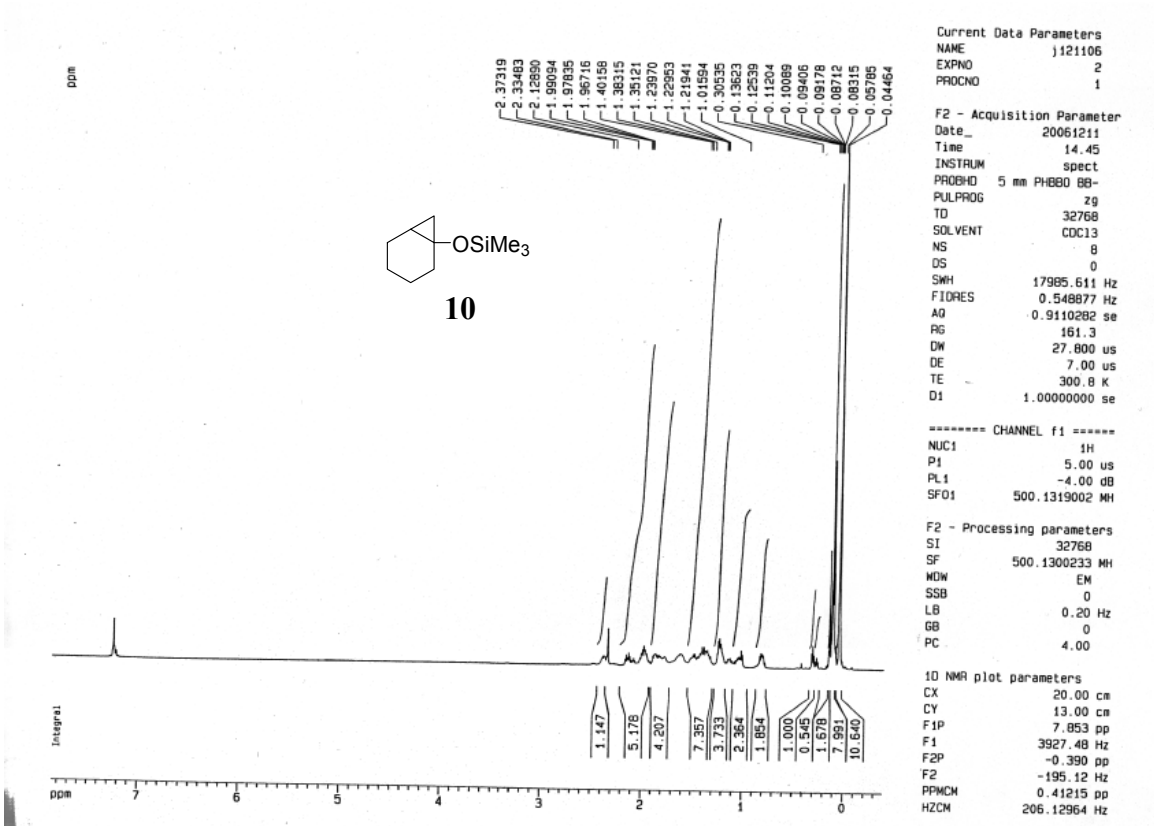
5

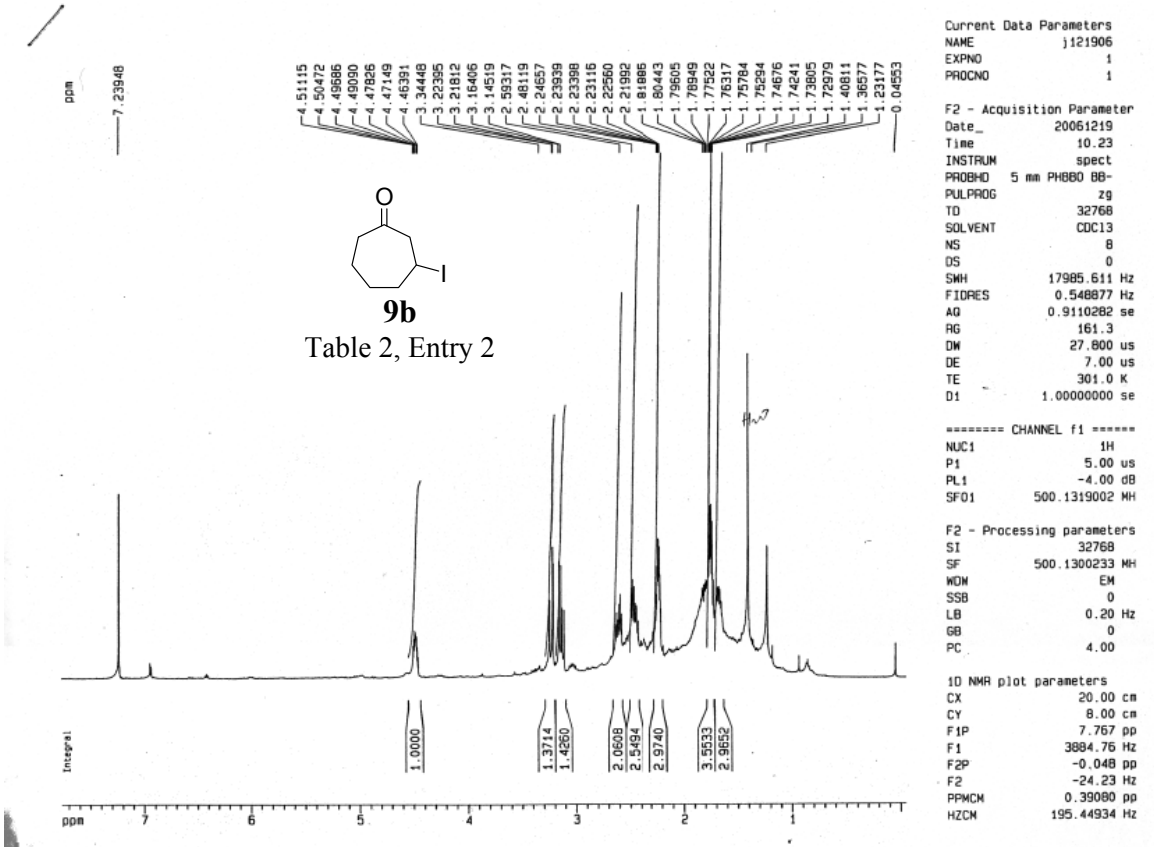
```
Current Data Parameters
NAME                  j073106
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameter
Date_                20060731
Time                    10.09
INSTRUM                 spect
PROBHD   5 mm PHBBO BB-
PULPROG                    zg
TD                      32768
SOLVENT                 CDCl3
NS                          8
DS                          0
SWH                17985.611 Hz
FIDRES              0.548877 Hz
AQ                  0.9110282 se
RG                        256
DW                   27.800 us
DE                    7.00 us
TE                   300.0 K
D1                1.00000000 se

======== CHANNEL f1 ======
NUC1                       1H
P1                    5.00 us
PL1                  -4.00 dB
SFO1           500.1319002 MH

F2 - Processing parameters
SI                      32768
SF             500.1300233 MH
WDW                        EM
SSB                         0
LB                   0.20 Hz
GB                          0
PC                    4.00

1D NMR plot parameters
CX                   20.00 cm
CY                    6.00 cm
F1P                   7.896 pp
F1                 3948.84 Hz
F2P                  -0.048 pp
F2                  -24.23 Hz
PPMCM                0.39720 pp
HZCM               198.65346 Hz
```



8

```
Current Data Parameters
NAME                  j121106
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameter
Date_                20061211
Time                    14.45
INSTRUM                 spect
PROBHD   5 mm PHBBO BB-
PULPROG                    zg
TD                      32768
SOLVENT                 CDCl3
NS                          8
DS                          0
SWH                17985.611 Hz
FIDRES              0.548877 Hz
AQ                  0.9110282 se
RG                      161.3
DW                   27.800 us
DE                    7.00 us
TE                   300.8 K
D1                1.00000000 se

======== CHANNEL f1 ======
NUC1                       1H
P1                    5.00 us
PL1                  -4.00 dB
SFO1           500.1319002 MH

F2 - Processing parameters
SI                      32768
SF             500.1300233 MH
WDW                        EM
SSB                         0
LB                   0.20 Hz
GB                          0
PC                    4.00

1D NMR plot parameters
CX                   20.00 cm
CY                   50.00 cm
F1P                   7.853 pp
F1                 3927.48 Hz
F2P                  -0.390 pp
F2                 -195.12 Hz
PPMCM                0.41215 pp
HZCM               206.12964 Hz
```


OSiMe3
10
Current Data Parameters
NAME j121106
EXPNO 2
PROCNO 1
F2 - Acquisition Parameter
Date_ 20061211
Time 14.45
INSTRUM spect
PROBHD 5 mm PHBBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 17985.611 Hz
FIDRES 0.548877 Hz
AQ 0.9110282 se
RG 161.3
DW 27.800 us
DE 7.00 us
TE 300.8 K
D1 1.00000000 se
======== CHANNEL f1 ======
NUC1 1H
P1 5.00 us
PL1 -4.00 dB
SFO1 500.1319002 MH
F2 - Processing parameters
SI 32768
SF 500.1300233 MH
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 20.00 cm
CY 13.00 cm
F1P 7.853 pp
F1 3927.48 Hz
F2P -0.390 pp
F2 -195.12 Hz
PPMCM 0.41215 pp
HZCM 206.12964 Hz


9b
Table 2, Entry 2
Current Data Parameters
NAME j121906
EXPNO 1
PROCNO 1
F2 - Acquisition Parameter
Date_ 20061219
Time 10.23
INSTRUM spect
PROBHD 5 mm PHBBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 17985.611 Hz
FIDRES 0.548877 Hz
AQ 0.9110282 se
RG 161.3
DW 27.800 us
DE 7.00 us
TE 301.0 K
D1 1.00000000 se
======== CHANNEL f1 ======
NUC1 1H
P1 5.00 us
PL1 -4.00 dB
SFO1 500.1319002 MH
F2 - Processing parameters
SI 32768
SF 500.1300233 MH
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 20.00 cm
CY 8.00 cm
F1P 7.767 pp
F1 3884.76 Hz
F2P -0.048 pp
F2 -24.23 Hz
PPMCM 0.39080 pp
HZCM 195.44934 Hz