Supporting information

Selective Synthesis of Eight-membered Cyclic urea by the [6+2]
Cycloaddition Reaction of 2-Vinylazetidines and Electron-
Deficient Isocyanates.

Shunsuke Koya, Kenichi Yamanoi, Ryu Yamasaki, Hyuma Masu, Isao Azumaya, and
Shinichi Saito

*Department of Chemistry, Faculty of Science, Tokyo University of Science, Kagurazaka,
Shinjuku, Tokyo, Japan, 162-8601*

Experimental details for the synthesis of **1a-f**, **3aa-3fa**, **4aa-4ea**, **5aa**, **6ae**, **6fa**. S2-S14

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¹H and ¹³C spectra of **1a-f**, **3aa-3fa**, **4aa-4ea**, **5aa**, **6ae**, **6fa**. S16-S71

General Experimental. Reagents were obtained from commercial supplies and used without further purification unless otherwise noted. ^1H and ^{13}C NMR spectra were recorded on a Bruker Biospin DPX 300 (300 MHz), a JEOL JNM-LA500 FT NMR SYSTEM (500 MHz), or a Bruker AV600 (600 MHz). ^1H chemical shifts were referenced to the non-deuterated solvent signals in CDCl_3 (δ 7.24), $\text{DMSO}-d_6$ (δ 2.49). ^{13}C chemical shifts were referenced to the solvent signals in CDCl_3 (δ 77.00), $\text{DMSO}-d_6$ (δ 39.70). Multiplicity is indicated by s (singlet), d (doublet), t (triplet), q (quartet), quint (quintet), or m (multiplet). Coupling constants, J , are reported in Hz. IR spectra were recorded on a JASCO FT/IR-410 (FT/IR). High-resolution mass spectra were measured by a JEOL JMS-SX102A. Elemental analyses were measured by a YANAKO CHN-recorder MT-6. Thin layer chromatography (TLC) was performed on a Merck silica gel 60F-254 plate. Column chromatography was performed using Kanto Chemical silica gel 60N (spherical, neutral, 63-210 μm).

N-benzyl-2-formylazetidine,¹⁻³ *N*-benzyl-2-acetylazetidine,⁴ *N*-benzyl-2-methoxy-carbonyl-2-methylazetidine,⁵ isocyanate **2b**,⁶ allene **5a**,⁷ **5b**⁸⁻⁹ were prepared as reported in the literature. Isocyanates **2a**, **2c** and **2d** were commercially available.

***N*-Benzyl-2-vinylazetidine (1a).** Crude *N*-benzyl-2-formylazetidine³ (1.75 g, 10 mmol) in THF (10 mL) was added to a solution of methylenetriphenylphosphorane, prepared by the treatment of methyltriphenylphosphonium bromide (7.2 g, 20 mmol) in THF (72 mL) with *n*-BuLi (1.66 M hexane solution, 11.1 mL, 18.4 mmol) at 0 °C for 1.5 h under an argon atmosphere. The mixture was then stirred at room temperature for 20 h. The mixture was poured into water and the product was extracted with ether. The organic layer was washed with brine, dried over anhydrous Na_2SO_4 , and concentrated. The residue was purified by silica gel chromatography (hexane-AcOEt/5:1) to give **1a** (0.85 g, 4.9 mmol, 49% yield). Pale yellow oil; ^1H NMR (300 MHz, CDCl_3) δ 7.34-7.21 (m, 5H), 5.94-5.83 (ddd, J = 17.1, 10.2, 6.8, 1H), 5.17-5.10 (ddd, J = 17.1, 1.7, 1.1, 1H), 5.03-4.98 (ddd, J = 10.2, 1.7, 0.8, 1H), 3.77-3.73 (d, J = 12.6, 1H), 3.63-3.55 (dd, J = 7.9, 7.5, 1H), 3.45-3.41 (d, J = 12.8, 1H), 3.30-3.24 (td, J = 7.3, 1.1, 1H), 2.88-2.80 (dt, J = 9.6, 7.5, 1H), 2.16-1.98 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) 140.1, 138.2, 128.9, 128.2, 126.9, 115.0, 68.1, 62.1, 51.5, 24.9; IR (neat) 2958, 2821, 1495, 1454, 1361, 1182, 1029, 990.3, 922.8, 733.8, 698.1 cm^{-1} ; HR-MS (EI) calcd for $\text{C}_{12}\text{H}_{15}\text{N}$: 173.1204. Found: 173.1203.

***N*-Benzyl-2-(1-(4-methoxyphenyl)vinyl)azetidine (1d).**

***N*-Benzyl-2-(4-methoxybenzoyl)azetidine.** Crude *N*-benzyl-2-formylazetidine³ (0.53 g, 3.0 mmol) was dissolved in ether (3.5 mL)-THF (4.0 mL), and the solution was added to 4-methoxyphenylmagnesium bromide (6.0 mmol) in ether (2.0 mL) at 0 °C under an argon atmosphere. The reaction mixture was stirred at 0 °C for 2.5 h. The reaction was quenched with saturated NH₄Cl aq., and the product was extracted with ether. The organic solution was washed with brine, and dried over anhydrous Na₂SO₄. The solvent was removed under reduced pressure. This material was used for the next reaction without further purification. DMSO (0.47 mL, 6.6 mmol) was added to a CH₂Cl₂ solution (9.0 mL) of oxalyl chloride (0.28 mL, 3.3 mmol) at -78 °C under an argon atmosphere and stirred for 15 min. Then the crude alcohol in CH₂Cl₂ (3.0 mL) was added slowly, and the stirring was continued for an additional 30 min. Triethylamine (2.1 mL, 15 mmol) was added, and the reaction mixture was stirred for 5 min at -78 °C and allowed to warm to room temperature. The mixture was poured into water and the product was extracted with CH₂Cl₂. The organic layer was washed with brine, dried over anhydrous Na₂SO₄, and concentrated. The residue was purified by silica gel column chromatography (hexane-AcOEt/10:1) to give *N*-benzyl-2-(4-methoxybenzoyl)azetidine. Yield: 70% from *N*-benzyl-2-formylazetidine. Yellow solid; mp 75.0-76.5 °C; ¹H NMR (500 MHz CDCl₃) δ 7.86-7.84 (d, *J* = 8.9, 2H), 7.28-7.16 (m, 5H), 6.88-6.86 (d, *J* = 8.9, 2H), 4.41-4.38 (t, *J* = 8.7, 1H), 3.88-3.85 (d, *J* = 12.8, 1H), 3.81 (s, 3H), 3.53-3.51 (d, *J* = 12.8, 1H), 3.32-3.29 (m, 1H), 3.01-2.96 (q, *J* = 7.8, 1H), 2.37-2.32 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) 196.8, 163.4, 137.2, 130.4, 129.2, 128.2, 128.1, 127.1, 113.8, 68.4, 62.3, 55.4, 50.5, 24.1; IR (KBr) 2843, 2360, 1680, 1600, 1509, 1237, 1174, 1018, 846.6, 741.5, 701.0, 410.8 cm⁻¹; HR-MS (EI) calcd for C₁₈H₁₉NO₂: 281.1416. Found: 281.1418.

***N*-Benzyl-2-(1-(4-methoxyphenyl)vinyl)azetidine (1d).** *N*-Benzyl-2-(4-(methoxybenzoyl)-azetidine (0.84 g, 3.0 mmol) in THF (3.0 mL) was added to methylene

-triphenylphosphorane, prepared in THF (22 mL) by the treatment of methyl -triphenylphosphonium bromide (2.1 g, 6.0 mmol) with *n*-BuLi (1.66M hexane solution, 3.5 mL, 5.5 mmol) at 0 °C for 1.5 h. The mixture was then stirred at room temperature for 20 h. The mixture was poured into water and the product was extracted with ether. The organic layer was washed with brine, dried over anhydrous Na₂SO₄, and concentrated. The residue was purified by silica gel column chromatography (hexane-AcOEt/5:1) to give **1d** (0.44 g, 1.58 mmol, 53% yield from *N*-benzyl-2-formylazetidine). Yellow solid; mp 45.0-46.3 °C; ¹H NMR (300 MHz, CDCl₃) δ 7.34-7.20 (m, 7H), 6.87-6.82 (d, *J* = 8.9, 1H), 5.51-5.50 (t, *J* = 1.6, 1H), 5.332-5.329 (t, *J* = 1.0, 1H), 4.03-3.98 (t, *J* = 8.5, 1H), 3.93-3.89 (d, *J* = 13.0, 1H), 3.38 (s, 3H), 3.38-3.33 (d, *J* = 13.0, 1H), 3.30-3.25 (t, *J* = 6.9, 1H), 2.90-2.82 (dt, *J* = 9.2, 7.2, 1H), 2.32-2.23 (m, 1H), 2.03-1.94 (quint, *J* = 9.1, 1H); ¹³C NMR (125 MHz, CDCl₃) 159.1, 148.1, 138.5, 132.0, 128.7, 128.2, 127.2, 126.9, 120.2, 113.7, 110.0, 67.9, 62.2, 55.3, 51.0, 27.0; IR (KBr) 3002, 2963, 2828, 2785, 1604, 1512, 1458, 1297, 1243, 1168, 1028, 907.3, 837.0, 756.9, 707.8, 508.2 cm⁻¹; Anal. Calcd for C₁₉H₂₁NO: C, 81.7; H, 7.58; N, 5.01. Found: C, 81.6; H, 7.54; N, 5.03.

Compounds **1b-c**, **1e** were prepared similarly. *N*-Benzyl-2-(4-(trifluoromethyl)-benzoyl)azetidine turned out to be very unstable, and the attempted purification of the compound was unsuccessful.

***N*-Benzyl-2-(1-methyl)vinylazetidine (1b).**

Yield: 33% from *N*-benzyl-2-formylazetidine. Pale yellow oil; ¹H NMR (300 MHz, CDCl₃) δ 7.25-7.16 (m, 5H), 4.94-4.93 (d, *J* = 1.0, 1H), 4.71 (s, 1H), 3.78-3.74 (d, *J* = 12.8, 1H), 3.51-3.46 (t, *J* = 8.1, 1H), 3.31-3.27 (d, *J* = 13.0, 1H), 3.20-3.14 (t, *J* = 7.2, 1H), 2.76-2.68 (dt, *J* = 9.2, 7.1, 1H), 2.08-1.87 (m, 1H), 1.67 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) 141.2, 138.5, 128.8, 128.2, 126.8, 109.7, 70.3, 62.4, 50.8, 24.3, 18.3; IR (neat) 3064, 3028, 2961, 2822, 1647, 1495, 1454, 1362, 1236, 1177, 1029, 895.8, 803.2, 735.7, 697.1, 464.8 cm⁻¹; HR-MS (EI) calcd for C₁₃H₁₇N: 187.1361. Found: 187.1361.

***N*-Benzyl-2-benzoylazetidine.**

Yield: 50% from *N*-benzyl-2-formylazetidine. Pale yellow solid; ^1H NMR (500 MHz, $\text{DMSO}-d_6$) δ 7.90-7.89 (d, $J = 7.6$, 2H), 7.62-7.59 (t, $J = 7.0$, 1H), 7.51-7.48 (t, $J = 7.7$, 2H), 7.29-7.17 (m, 5H), 4.62-4.59 (t, $J = 8.6$, 1H), 3.83-3.80 (d, $J = 13.1$, 1H), 3.50-3.48 (d, $J = 13.1$, 1H), 3.15-3.12 (m, 1H), 2.94-2.89 (dt, $J = 8.6$, 6.7, 1H), 2.45-2.39 (qd, $J = 8.3$, 2.2, 1H), 2.19-2.12 (quint, $J = 8.6$, 1H); ^{13}C NMR (125 MHz, $\text{DMSO}-d_6$) 198.0, 137.9, 134.8, 133.1, 128.7, 128.4, 128.0, 127.9, 126.8, 68.0, 61.6, 50.3, 23.0.

***N*-Benzyl-2-(1-phenyl)vinylazetidine (1c).**

Yield: 36% from *N*-benzyl-2-formylazetidine. Pale yellow solid; mp 49.5-50.5 °C; ^1H NMR (300 MHz, CDCl_3) δ 7.32-7.12 (m, 10H), 5.51-5.50 (t, $J = 1.6$, 1H), 5.32 (s, 1H), 3.98-3.92 (t, $J = 8.1$, 1H), 3.84-3.80 (d, $J = 12.9$, 1H), 3.30-3.26 (d, $J = 12.8$, 1H), 3.22-3.18 (t, $J = 6.6$, 1H), 2.83-2.75 (dt, $J = 9.2$, 7.5, 1H), 2.24-2.15 (td, $J = 7.3$, 1.1, 1H), 1.96-1.84 (quint, $J = 9.1$, 1H); ^{13}C NMR (125 MHz, CDCl_3) 148.8, 139.6, 138.5, 128.7, 128.27, 128.25, 127.4, 126.9, 126.1, 111.6, 67.8, 62.8, 51.1; IR (KBr) 3082, 2972, 2928, 2824, 1492, 1451, 1370, 1281, 1223, 1184, 1071, 991.2, 918.9, 758.9, 701.0 cm^{-1} ; HR-MS (EI) calcd for $\text{C}_{18}\text{H}_{19}\text{N}$: 249.1517. Found: 249.1515.

***N*-Benzyl-2-(1-(4-trifluoromethyl)phenyl)vinylazetidine (1e).**

Yield: 35% from *N*-benzyl-2-formylazetidine. Pale yellow solid; mp 50.5-52.0 °C; ^1H NMR (300 MHz, CDCl_3) δ 7.57-7.54 (d, $J = 8.3$, 2H), 7.49-7.46 (d, $J = 8.2$, 2H), 7.32-7.20 (m, 5H), 5.66 (s, 1H), 5.44 (s, 1H), 4.04-3.98 (t, $J = 7.8$, 1H), 3.90-3.86 (d, $J = 13.0$, 1H), 3.41-3.37 (d, $J = 12.8$, 1H), 3.31-3.27 (t, $J = 6.8$, 1H), 2.93-2.85 (ddd, $J = 9.4$, 7.3, 7.3, 1H), 2.30-2.22

(m, 1H), 2.03-1.94 (quint, $J = 9.0$, 1H); ^{13}C NMR (125 MHz, CDCl_3) 147.9, 143.2, 138.2, 128.7, 128.3, 127.0, 126.5, 125.20, 125.17, 125.1, 113.9, 67.7, 62.2, 51.0, 26.6, 27.0; IR (KBr) 1615, 1456, 1326, 1169, 1126, 1065, 915.1, 844.7, 736.7, 698.1, 459.0 cm^{-1} ; Anal. Calcd for $\text{C}_{19}\text{H}_{18}\text{F}_3\text{N}$: C, 71.9; H, 5.72; N, 4.41. Found: C, 71.7; H, 6.01; N, 4.26.

Preparation of vinyl azetidine (1f).

N-Benzyl-2-methoxycarbonyl-2-methylazetidine.

To a solution of *N*-benzyl-2-methoxycarbonyl-2-methylazetidinone^{5c} (0.35 g, 1.5 mmol) in dry THF (1.5 mL) was added, under Ar atmosphere, tris-(triphenylphosphine)rhodium(I) carbonyl hydride (14 mg, 1 mol%) and diphenylsilane (0.69 mL, 3.8 mmol). After 15 h of reaction at room temperature, the solvent was evaporated to give a residue that was dissolved in ether and washed twice with 1N HCl aq.. The aqueous layer was treated with 2N NaOH to pH 10 and extracted with EtOAc. The organic layer was washed with brine, dried over Na_2SO_4 , and concentrated. The residue was purified by short column chromatography (hexane-AcOEt/5:1) to give *N*-benzyl-2-methoxycarbonyl-2-methylazetidine (0.23 g, 1.1 mmol, 71%). Pale yellow oil; ^1H NMR (500 MHz, CDCl_3) δ 7.23-7.14 (m, 5H), 3.71-3.68 (d, $J = 12.8$, 1H), 3.67 (s, 3H), 3.52-3.50 (d, $J = 12.8$, 1H), 3.21-3.17 (ddd, $J = 8.3, 6.5, 4.5$, 1H), 3.06-3.01 (dt, $J = 8.3, 6.9$, 1H), 2.59-2.50 (ddd, $J = 10.8, 8.3, 7.1$, 1H), 1.89-1.84 (ddd, $J = 10.7, 8.3, 4.6$, 1H), 1.43 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) 175.1, 138.2, 128.7, 128.2, 126.9, 67.7, 55.7, 51.6, 49.1, 28.6, 18.8; IR (neat) 2965, 2835, 1733, 1495, 1453, 1371, 1308, 1192, 1149, 1121, 1085, 989.3, 737.6, 699.1, 462.8 cm^{-1} ; HR-MS (EI) calcd for $\text{C}_{13}\text{H}_{17}\text{NO}_2$: 219.1256. Found: 219.1261.

N-Benzyl-2-methyl-2-formylazetidine.

To a stirred solution of *N*-benzyl-2-methoxycarbonyl-2-methylazetidine (3.89 g, 17.8 mmol) in dry THF (40 mL) at -78°C under Ar was added a 1 M solution of diisobutylaluminium hydride in hexane (28.7 mL, 28.4 mmol) dropwise via a cannula over 1 h. The reaction mixture was stirred for 20 h at -70°C . The mixture was quenched

by the careful addition of several small portions of $\text{Na}_2\text{SO}_4 \cdot (\text{H}_2\text{O})_{10}$. The mixture was stirred at room temperature, diluted with EtOAc, filtered through Celite and concentrated in vacuo to give *N*-benzyl-2-methyl-2-formylazetidine. This material was used for the next reaction without further purification. Pale yellow oil; ^1H NMR (500 MHz, CDCl_3) δ 9.58 (s, 1H), 7.26-7.15 (m, 5H), 3.64-3.62 (d, J = 13.1, 1H), 3.57-3.54 (d, J = 13.1, 1H), 3.37-3.34 (ddd, J = 8.2, 6.9, 3.4, 1H), 3.24-3.21 (td, J = 8.1, 6.9, 1H), 2.39-2.34 (dt, J = 11.0, 8.3, 1H), 1.74-1.69 (ddd, J = 11.3, 8.3, 3.4, 1H), 1.29 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) 203.4, 138.0, 128.5, 128.3, 127.1, 70.6, 55.4, 50.5, 25.7, 13.5.

***N*-Benzyl-2-methyl-2-vinylazetidine (1f).** Crude aldehyde (0.176 g, 0.93 mmol) in THF (1.0 mL) was added to methylenetriphenylphosphorane, prepared in THF (7.2 mL) by the treatment of methyltriphenylphosphonium bromide (0.71 g, 1.86 mmol) with *n*-BuLi (1.66 M hexane solution, 1.15 mL, 1.83 mmol) at 0 °C for 1.5 h. The mixture was then stirred at room temperature for 20 h. The mixture was poured into water and the product was extracted with ether. The organic layer was washed with brine, and dried over anhydrous Na_2SO_4 , and concentrated. The residue was purified by silica gel column chromatography (hexane-AcOEt/5:1) to give **1f** (59 mg, 0.32 mmol, 34%). Pale yellow oil; ^1H NMR (300 MHz, CDCl_3) δ 7.27-7.12 (m, 5H), 6.03-5.94 (dd, J = 17.4, 10.5, 1H), 5.12-5.09 (dd, J = 17.4, 1.5, 1H), 5.02-4.98 (dd, J = 10.6, 1.7, 1H), 3.54-3.50 (d, J = 13.2, 1H), 3.44-3.40 (d, J = 13.0, 1H), 3.15-3.02 (m, 1H), 3.45-3.41 (d, J = 12.8, 1H), 3.30-3.24 (td, J = 7.3, 1.1, 1H), 2.88-2.80 (dt, J = 9.6, 7.5, 1H), 2.11-2.02 (ddd, J = 10.2, 8.0, 7.1, 1H), 1.89-1.81 (dd, J = 9.6, 7.5, 1H), 1.27 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) 143.6, 139.2, 128.7, 128.3, 128.1, 126.6, 112.4, 66.3, 55.4, 49.3, 31.1, 20.5; IR (neat) 3442, 2961, 2821, 1636, 1494, 1454, 1369, 1191, 1028, 918.9, 726.7, 696.2 cm^{-1} ; HR-MS (EI) calcd for $\text{C}_{13}\text{H}_{17}\text{N}$: 187.1361. Found: 187.1364.

Cycloaddition of 2-vinylazetidine (1a) and tosyl isocyanate (2a). A Representative Procedure. To a solution of 2-vinylazetidine **1a** (87 mg, 0.5 mmol) in CH_2Cl_2 (10 mL) was added freshly distilled tosyl isocyanate **2a** (114 μl , 0.75 mmol). The mixture was stirred at room temperature for 1 h. The reaction mixture was concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hexane-AcOEt) to give **3aa** and **4aa**.

Compound 3aa

Colorless solid; mp 148.0-150.0 °C; ^1H NMR (300 MHz, CDCl_3) δ 7.95-7.92 (d, $J = 8.4$, 2H), 7.34-7.24 (m, 10H), 5.69-5.28 (m, 2H), 4.48 (s, 2H), 4.02-4.00 (d, $J = 6.3$, 2H), 3.61-3.57 (t, $J = 6.6$, 2H), 2.50-2.44 (q, $J = 6.6$, 2H), 2.42 (s, 3H); ^{13}C HMR (150 MHz, CDCl_3) 156.3, 144.3, 136.3, 135.8, 132.3, 129.6, 128.9, 128.8, 128.2, 127.9, 124.3, 51.3, 47.0, 46.2, 26.8, 21.8; IR (KBr) 2930, 1687, 1596, 1473, 1424, 1344, 1216, 1156, 1089, 855, 820, 735, 703, 683, 689, 579, 543 cm^{-1} ; Anal. Calcd for $\text{C}_{20}\text{H}_{22}\text{N}_2\text{O}_3\text{S}$: C, 64.84; H, 5.99; N, 7.56. Found: C, 64.56; H, 6.01; N, 7.74.

Compound 4aa

Colorless solid; mp 148.5-150.5 °C; ^1H NMR (300 MHz, CDCl_3) δ 7.80-7.78 (d, $J = 8.3$, 2H), 7.27-7.17 (m, 8H), 5.72-5.54 (m, 2H), 5.01-4.99 (dd, $J = 5.2, 0.9$, 2H), 4.56 (s, 2H), 3.47-3.43 (t, $J = 6.4$, 2H), 2.42-2.36 (m, 5H), 2.42 (s, 3H); ^{13}C HMR (125 MHz, CDCl_3) 159.8, 141.9, 141.0, 135.5, 131.1, 129.0, 128.7, 128.3, 128.0, 126.6, 124.8, 72.6, 52.9, 46.1, 28.4, 21.5; IR (KBr) 1569, 1496, 1474, 1449, 1365, 1281, 1246, 1171, 1148, 1090, 962, 912, 843, 817, 764, 699, 670, 580, 553 cm^{-1} ; Anal. Calcd for $\text{C}_{20}\text{H}_{22}\text{N}_2\text{O}_3\text{S}$: C, 64.84; H, 5.99; N, 7.56. Found: C, 64.57; H, 6.16; N, 7.45.

Compound 3ab

Colorless solid; mp 120.0-121.5 °C; ^1H NMR (500 MHz, CDCl_3) δ 7.59-6.92 (m, 10H), 5.89-5.81 (quint, $J = 5.6$ Hz, 1H), 5.77-5.69 (quint, $J = 5.6$, 1H), 4.49-4.47 (d, $J = 5.7$, 2H), 4.36 (s, 2H), 3.32-3.28 (t, $J = 6.6$, 1H), 2.50-2.44 (q, $J = 6.3$, 1H); ^{13}C NMR (125 MHz, CDCl_3) 169.3, 158.0, 136.0, 135.8, 130.9, 130.8, 128.5, 128.3, 128.2, 127.7, 127.2, 126.3; IR (KBr) 1670, 1475, 1425, 1662, 1246, 1200, 798.4, 720.3, 701.0, 643.1, 617.1 cm^{-1} ; HR-MS (EI) calcd for $\text{C}_{20}\text{H}_{20}\text{N}_2\text{O}_2$: 320.1512. Found 320.1523.

Compound 3ba

Colorless solid; mp 107.9-109.0 °C; ^1H NMR (500 MHz, CDCl_3) δ 7.98-7.96 (d, J = 8.3, 2H), 7.33-7.24 (m, 7H), 5.26-5.23 (dd, J = 6.9, 6.0, 1H), 4.44 (s, 2H), 3.92 (s, 2H), 3.51-3.48 (t, J = 7.1, 2H), 2.46-2.42 (m, 5H), 1.521-1.519 (d, J = 1.2, 3H); ^{13}C NMR (125 MHz, CDCl_3) 155.8, 144.0, 136.3, 135.7, 132.5, 129.3, 128.7, 128.5, 128.2, 127.6, 125.6, 51.7, 51.0, 46.2, 25.6, 23.1, 21.6; IR (neat) 1668, 1424, 1349, 1164, 821.5, 733.8, 666.3, 587.2, 546.7 cm^{-1} ; Anal. Calcd for $\text{C}_{21}\text{H}_{24}\text{N}_2\text{O}_3\text{S}$: C, 65.60; H, 6.29; N, 7.29. Found: C, 65.47; H, 6.51; N, 7.08.

Compound 4ba

Colorless solid; mp 124.8-125.4 °C; ^1H NMR (500 MHz, CDCl_3) δ 7.26-7.16 (m, 7H), 5.43-5.40 (td, J = 6.7, 1.5, 1H), 4.95 (s, 2H), 4.53 (s, 2H), 3.36-3.33 (t, J = 6.7, 2H), 2.39-2.36 (m, 5H), 1.78-1.77 (d, J = 1.3, 3H); ^{13}C NMR (125 MHz, CDCl_3) 159.8, 141.8, 141.1, 135.6, 133.8, 129.0, 128.6, 128.3, 128.0, 126.5, 126.0, 75.8, 53.9, 47.2, 27.0, 22.5, 21.4; IR (neat) 1561, 1477, 1440, 1364, 1281, 1252, 1171, 1145, 1114, 1085, 954.6, 906.4, 882.3, 850.5, 789.7, 747.3, 702.9, 656.6, 581.4, 548.7 cm^{-1} ; Anal. Calcd for $\text{C}_{21}\text{H}_{24}\text{N}_2\text{O}_3\text{S}$: C, 65.4; H, 6.29; N, 7.29. Found: C, 65.34; H, 6.42; N, 7.24.

Compound 3ca

Colorless solid; mp 176.0-177.0 °C; ^1H NMR (500 MHz, CDCl_3) δ 7.97-7.94 (d, J = 8.3, 2H), 7.30-6.91 (m, 12H), 5.70-5.65 (t, J = 7.1, 1H), 4.48 (s, 2H), 4.38 (s, 2H), 3.68-3.63 (t, J = 7.1, 2H), 2.71-2.64 (q, J = 7.1, 2H), 2.44 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) 155.4, 144.0, 141.0, 136.7, 136.2, 135.6, 129.4, 129.3, 128.7, 128.5, 128.3, 128.2, 127.7, 127.3, 125.9, 51.7, 50.1, 45.9, 26.0, 21.6; IR (KBr) 1683, 1418, 1347, 1207, 1168, 1092, 739.6, 661.5, 584.3 cm^{-1} ; Anal. Calcd for $\text{C}_{26}\text{H}_{26}\text{N}_2\text{O}_3\text{S}$: C, 69.93; H, 5.87; N, 6.27. Found: C, 69.75; H, 6.00; N, 6.23.

Compound 3da

Colorless solid; mp 135.0-136.5 °C; ^1H NMR (300 MHz, CDCl_3) δ 7.97-7.94 (dd, J = 6.8, 1.7, 2H), 7.30-7.24 (m, 7H), 6.87-6.84 (dd, J = 6.7, 2.2, 2H), 6.65-6.62 (dd, J = 6.6, 2.1, 2H), 5.63-5.57 (t, J = 7.0, 1H), 4.47 (s, 2H), 4.34 (s, 2H), 3.76 (s, 3H), 3.65-3.61 (t, J = 7.1, 2H), 2.68-2.61 (q, J = 7.0, 2H), 2.44 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) 159.0, 155.3, 143.9, 136.2, 136.1, 135.6, 133.5, 129.3, 128.7, 128.5, 128.3, 127.8, 127.6, 127.1, 113.6, 55.2, 51.7, 50.1, 45.9, 25.9, 21.6; IR (KBr) 1668, 1609, 1514, 1420, 1350, 1251, 1167, 819.6, 730.9, 696.2, 656.6, 584.3 cm^{-1} ; Anal. Calcd for $\text{C}_{27}\text{H}_{28}\text{N}_2\text{O}_4\text{S}$: C, 68.04; H, 5.92; N, 5.88. Found: C, 67.78; H, 6.04; N, 5.84.

Compound 4da

Colorless solid; mp 133.7-135.2 °C; ^1H NMR (600 MHz, CDCl_3) δ 7.80-7.79 (d, J = 7.9, 2H), 7.40-7.38 (d, J = 8.6, 2H), 7.27-7.17 (m, 7H), 6.88-6.87 (d, J = 8.6, 2H), 5.82-5.80 (t, J = 7.9, 1H), 5.46 (s, 2H), 4.55 (s, 2H), 3.81 (s, 3H), 3.40-3.38 (t, J = 6.6, 2H), 2.62-2.58 (q, J = 6.9, 2H), 2.37 (s, 3H); ^{13}C NMR (150 MHz, CDCl_3) 155.2, 154.7, 137.2, 136.3, 134.0, 130.7, 127.1, 124.3, 123.9, 123.5, 123.2, 123.1, 122.3, 121.7, 109.3, 70.4, 50.6, 50.1, 43.7, 22.9, 16.7; IR (KBr) 3853, 3734, 3648, 2924, 1735, 1559, 1508, 1457, 1247, 1151, 812.9, 658.6, 453.2, 424.3 cm^{-1} ; HR-MS (EI) calcd for $\text{C}_{27}\text{H}_{28}\text{N}_2\text{O}_4\text{S}$: 476.1770. found: 476.1768.

Compound 3ea

Colorless solid; mp 157.0-158.5 °C; ^1H NMR (300 MHz, CDCl_3) δ 7.96-7.94 (d, J = 8.3, 2H), 7.36-7.24 (m, 9H), 7.00-6.98 (d, J = 8.1, 2H), 5.74-5.69 (t, J = 7.0, 1H), 4.48 (s, 2H), 4.36 (s, 2H), 3.70-3.65 (t, J = 7.0, 2H), 2.72-2.65 (q, J = 7.0, 2H), 2.44 (s, 3H); ^{13}C NMR (75

MHz, CDCl₃) 155.3, 144.4, 144.3, 136.1, 135.8, 135.4, 131.6, 129.4, 128.8, 128.6, 128.4, 127.9, 126.3, 125.3, 125.2, 125.2; IR (KBr) 1665, 1436, 1330, 1170, 1124, 1069, 813.8, 734.8, 662.4, 582.4 cm⁻¹; HR-MS (EI) calcd for C₂₇H₂₅F₃N₂O₃S: 514.1538. found: 514.1541.

Compound 4ea

Colorless solid; mp 142.0-144.0 °C; ¹H NMR (500 MHz, CDCl₃) δ 7.80-7.78 (d, *J* = 8.3, 2H), 7.61-7.60 (d, *J* = 8.6, 2H), 7.57-7.55 (d, *J* = 8.2, 2H), 7.27-7.03 (m, 7H), 5.98-5.95 (t, *J* = 7.6, 1H), 5.49 (s, 2H), 4.55 (s, 2H), 3.46-3.43 (t, *J* = 6.6, 2H), 2.67-2.63 (q, *J* = 7.0, 2H), 2.37 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) 159.6, 142.1, 140.9, 138.3, 135.4, 131.1, 129.1, 128.7, 128.3, 128.1, 126.9, 126.4, 125.63, 125.61, 120.5, 120.43, 120.40, 74.5, 54.8, 48.0, 29.7, 27.8, 21.4; IR (KBr) 1665, 1437, 1330, 1170, 1124, 1069, 813.8, 734.8, 662.4, 582.4 cm⁻¹; HR-MS (EI) calcd for C₂₇H₂₅F₃N₂O₃S: 514.1538. found: 514.1540.

Compound 3fa

Colorless solid; mp 125.0-126.5 °C; ¹H NMR (500 MHz, CDCl₃) δ 7.93-7.92 (d, *J* = 8.2, 2H), 7.29-7.21 (m, 7H), 5.34-5.31 (td, *J* = 7.2, 0.8, 1H), 4.41 (s, 2H), 3.97-3.96 (d, *J* = 7.1, 2H), 3.52-3.49 (t, *J* = 6.7, 2H), 2.44-2.42 (t, *J* = 6.8, 2H), 2.38 (s, 3H), 1.48 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) 155.9, 143.9, 142.4, 136.4, 136.1, 129.3, 128.7, 128.5, 128.2, 127.6, 117.5, 51.4, 46.4, 45.3, 31.4, 25.4, 21.6; IR (KBr) 1676, 1469, 1423, 1347, 1224, 1164, 1107, 1089, 1047, 912.2, 817.7, 735.7, 707.8, 662.4, 578.5, 548.7 cm⁻¹; Anal. Calcd for C₂₁H₂₄N₂O₃S: C, 65.60; H, 6.29; N, 7.29. Found: C, 65.89; H, 6.49; N, 7.08.

Compound 6fa

Colorless solid; mp 160.0-161.5 °C; ^1H NMR (500 MHz, CDCl_3) δ 7.84-7.82 (d, J = 8.3, 2H), 7.31-7.19 (m, 7H), 5.56-5.50 (dd, J = 17.4, 11.0, 1H), 5.10-5.06 (d, J = 17.4, 1H), 5.05-5.03 (d, J = 11.0, 1H), 3.08-3.03 (m, 2H), 2.37 (s, 3H), 1.86-1.83 (td, J = 6.4, 2.3, 2H), 1.29 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) 154.9, 141.6, 141.1, 137.5, 135.2, 128.7, 128.6, 128.2, 128.0, 127.4, 116.1, 82.0, 53.7, 41.3, 30.8, 26.5, 21.4; IR (KBr) 2982, 1576, 1488, 1457, 1327, 1273, 1174, 1151, 1084, 950.7, 842.7, 718.4, 673.0, 608.4, 585.3, 551.5 cm^{-1} ; Anal. Calcd for $\text{C}_{21}\text{H}_{24}\text{N}_2\text{O}_3\text{S}$: C, 65.60; H, 6.29; N, 7.29. Found: C, 65.35; H, 6.44; N, 7.24.

Compound 3ae

Colorless solid; mp 156.5-157.5 °C; ^1H NMR (300 MHz, CDCl_3) δ 7.29-7.21 (m, 5H), 5.78-5.76 (m, 1H), 5.69-5.64 (dt, J = 10.6, 4.8, 1H), 4.75 (s, 1H), 4.48 (s, 2H), 3.74-3.72 (d, J = 8.1, 2H), 3.55-3.51 (t, J = 6.7, 2H), 2.40-2.35 (dt, J = 6.6, 5.1, 2H); ^{13}C NMR (125 MHz, CDCl_3) 161.7, 138.2, 131.5, 128.4, 128.1, 127.2, 125.1, 50.8, 44.7, 41.7, 27.9; IR (KBr) 3851, 3647, 3204, 1635, 1506, 1421, 1290, 961.3, 781.0, 703.9, 419.0 cm^{-1} ; Calcd for $\text{C}_{13}\text{H}_{16}\text{N}_2\text{O}$: 216.1263. Found: 216.1263.

Compound 4ae

Colorless solid; mp 80.0-82.0 °C; ^1H NMR (300 MHz, CDCl_3) δ 7.34-7.30 (m, 5H), 5.61-5.51 (m, 2H), 4.88-4.87 (dd, J = 3.0, 1.5, 2H), 4.74 (s, 2H), 3.59-3.55 (t, J = 6.3, 2H), 2.49-2.43 (q, J = 6.0, 2H); ^{13}C NMR (150 MHz, CDCl_3) 168.0, 164.8, 135.4, 128.8, 128.6, 128.3, 128.0, 126.0, 71.8, 53.7, 46.6, 29.6; IR (KBr) 3373, 1682, 1576, 1472, 1434, 1347, 1230, 1111, 977.7, 826.4, 701.0, 665.3, 419.4 cm^{-1} ; HR-MS (EI) calcd for $\text{C}_{15}\text{H}_{15}\text{Cl}_3\text{N}_2\text{O}_2$: 360.0199. Found: 360.0203.

Compound 6ae

Colorless oil; ^1H NMR (300 MHz, CDCl_3) δ 7.36-7.29 (m, 5H), 5.90-5.80 (ddd, $J = 17.1$, 10.6, 4.4, 1H), 5.624-5.616 (dq, $J = 17.2$, 0.8, 1H), 5.36-5.31 (dq, $J = 10.7$, 0.8, 1H), 4.99-4.93 (m, 1H), 4.89-4.84 (d, $J = 14.5$, 1H), 4.72-4.67 (d, $J = 14.3$, 1H), 3.34-3.30 (td, $J = 5.5$, 1.4, 1H), 2.24-2.14 (m, 1H), 2.01-1.89 (sextet, $J = 7.1$, 1H); ^{13}C NMR (150 MHz, CDCl_3) 162.6, 155.0, 130.3, 128.3, 124.2, 123.8, 123.6, 114.1, 72.6, 49.8, 37.6, 21.1; IR (neat) 1682, 1557, 1456, 830.2, 663.2, 419.4 cm^{-1} ; HR-MS (EI) calcd for $\text{C}_{15}\text{H}_{15}\text{Cl}_3\text{N}_2\text{O}_2$: 360.0199. Found: 360.0207.

Cycloaddition of 2-vinylazetidine (1a) and tosyl isocyanate (2a) in DMF.

To a solution of 2-vinylazetidine **1a** (87 mg, 0.5 mmol) in DMF (10 mL) was added freshly distilled tosyl isocyanate **2a** (114 μl , 0.75 mmol). The mixture was stirred at room temperature for 1 h. The mixture was poured into water and the product was extracted with ether (10 ml $\times$ 3). The organic layer was washed with brine, and dried over anhydrous Na_2SO_4 , and concentrated. The residue was purified by silica gel column chromatography (hexane-AcOEt) to give **5aa**.

Compound 5aa

Viscous liquid; ^1H NMR (300 MHz, CDCl_3) δ 7.83-7.80 (d, $J = 8.2$, 2H), 7.33-7.18 (m, 7H), 5.73-5.62 (ddd, $J = 17.1$, 10.7, 4.9, 1H), 5.32-5.22 (m, 2H), 5.26-5.22 (d, $J = 10.5$, 1H), 4.79-4.69 (m, 2H), 4.61-4.56 (d, $J = 14.9$, 1H), 3.20-3.16 (t, $J = 6.2$, 2H), 2.37 (s, 3H), 2.11-2.01 (m, 2H), 1.90-1.78 (sextet, $J = 7.3$, 1H); ^{13}C NMR (125 MHz, CDCl_3) 155.1, 141.6, 141.0, 135.1, 133.1, 128.8, 128.7, 128.3, 128.1, 127.1, 118.7, 77.7, 53.8, 42.3, 26.1, 21.4; IR (neat) 2925, 1733, 1575, 1456, 1298, 1139, 1086, 989.3, 838.9, 668.2, 588.2, 553.5 cm^{-1} ; HR-MS (EI) calcd for $\text{C}_{20}\text{H}_{22}\text{N}_2\text{O}_3\text{S}$: 370.1351. Found: 370.1349.

Cycloaddition of 2-vinylazetidine (1a) and electron-deficient allene (7). A representative procedure.

To a solution of 2-vinylazetidine **1a** (173 mg, 1.0 mmol) in THF (1 mL) was added freshly distilled acetyl allene **7a** (246 mg, 3.0 mmol) for 6 h. The mixture was stirred at room temperature for 15 h. The reaction mixture was concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hexane-AcOEt) to give **8aa**.

Compound 8aa

Colorless solid; mp 78.0-80.5 °C; ^1H NMR (300 MHz, CDCl_3) δ 7.36-7.15 (m, 5H), 5.73-5.65 (quint, $J = 5.9$, 1H), 5.49-5.41 (quint, $J = 5.9$, 1H), 5.07 (s, 1H), 4.42 (s, 2H), 3.67-3.63 (t, $J = 6.4$, 2H), 3.51-3.47 (t, $J = 6.9$, 2H), 2.61-2.54 (q, $J = 7.0$, 2H), 2.45-2.41 (q, $J = 6.2$, 2H); ^{13}C NMR (125 MHz, CDCl_3) 194.2, 166.1, 136.3, 130.7, 128.7, 126.6, 125.0, 94.7, 54.7, 48.9, 31.9, 28.6, 27.4, 26.4; IR (KBr) 1623, 1541, 1473, 1352, 1173, 952.7, 721.3 cm^{-1} ; Anal. Calcd for $\text{C}_{17}\text{H}_{21}\text{NO}$: C, 79.96; H, 8.29; N, 5.49. Found: C, 79.79; H, 8.27; N, 5.46.

Compound 8ab

Pale yellow solid; mp 100.0-101.5 °C; ^1H NMR (300 MHz, CDCl_3) δ 7.59-7.57 (d, $J = 7.7$, 2H), 7.36-7.21 (m, 8H), 5.75-5.70 (m, 1H), 5.49-5.44 (quint, $J = 5.8$, 1H), 4.50 (s, 2H), 3.75-3.72 (t, $J = 6.4$, 2H), 3.61-3.58 (t, $J = 7.1$, 2H), 2.70-2.67 (dt, $J = 7.0$, 5.8, 2H), 2.46-2.45 (q, $J = 6.1$, 2H); ^{13}C NMR (125 MHz, CDCl_3) 187.5, 167.6, 142.7, 136.1, 130.6, 130.1, 128.8, 127.8, 127.3, 127.2, 126.5, 124.9, 92.7, 55.3, 49.6, 28.8, 27.5, 27.0; IR (KBr) 1615, 1532, 1475, 1345, 1203, 1171, 986.4, 878.4, 769.5, 711.6 cm^{-1} ; Anal. Calcd for $\text{C}_{22}\text{H}_{23}\text{NO}$: C, 83.24; H, 7.30; N, 4.41. Found: C, 83.30; H, 7.21; N, 4.28.

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¹³C NMR spectrum (CDCl₃) of compound 10. The x-axis represents the chemical shift in ppm, ranging from 0 to 175. The spectrum shows a large solvent peak at 77.2550 ppm. Other labeled peaks include:

Chemical Shift (ppm)
148.8094
139.5639
138.4698
128.7225
128.2701
128.2537
127.4393
126.8882
126.0739
111.6297
77.2550
77.0000
76.7450
67.8038
62.1857
51.0154
26.9802

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 RGAIN 18

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sk6-58-2

DFILE C:\Documents and Settings\admin
COMMT sk6-58-2
DATIM Thu Jul 30 20:05:00 2009
ORNUC 13C
bcm
EXMOD 125.65 MHz
OBFRQ 0.00 KHz
OBSET 127958.0 Hz
OBFIN 32768
POINT 33898.3 Hz
SCANS 814
ACQTM 0.967 sec
PD 2.033 sec
PW1 5.1 us
1H
TENUC 1H
CTEMP 26.8 c
CDCL3
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 30

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical data\sk4-88-4_data.als

C:\Documents and Settings\admin

1H

- EXMOD 300.01 MHz
- OBSFQ 1.85 KHz
- OBSFT 2.7 Hz
- POINT 32768
- FREQ 6172.8 Hz
- SCANS 8
- ACQTM 0.000 sec
- PD 0.000 sec
- PW1 10.0 us
- TENUC 0.0 c
- CTEMP 7.24 ppm
- SLVNT EXREF 1.20 Hz
- BF 0
- RGAIN 0

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical data\sk4-88-4\data.C.als
sk4-88-4

DFILE C:\Documents and Settings\admin
COMNT sk4-88-4
DATIM Mon Mar 23 17:18:28 2009
OBNUC 13C
EXMOD bcm
OBFRQ 125.65 MHz
OBSET 0.00 KHz
OBFIN 127958.0 Hz
POINT 32768
FREQ 33898.3 Hz
SCANS 899
ACQTM 0.967 sec
PD 2.033 sec
PM1 5.1 us
IRNUC 1H
CTEMP 465.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 31

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical data\sk5-22-2_data.als

C:\Documents and Settings\admin

1H
300.01 MHz
1.85 KHz
2.7 Hz
32768
6172.8 Hz
8
0.000 sec
0.000 sec
10.0 us
0.0 c
7.24 ppm
1.20 Hz
0

DEFILE
COMMT
DATIM
ORNUC
EXMOD
OBFRO
OBFRO
OBFRO
OBFRO
POINT
FREQ
SCANS
ACQTM
PD
PW1
TENUC
CTEMP
SLVNT
EXREF
BF
RGAIN

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical data\sk5-22-2\data, C, .als
sk5-22-2

DFILE C:\Documents and Settings\admin
COMMT sk5-22-2
DATIM Thu Jun 18 21:54:05 2009
OBNUC 13C
EXMOD bcm
EXMOD 125.65 MHz
OBFREQ 0.00 KHz
OBSFET 127958.0 Hz
OBSFIN 32768
POINT 33898.3 Hz
FREQ 907
SCANS 907
ACQTM 0.967 sec
PD 2.033 sec
PW1 5.1 us
1H
TENUC 1H
CTEMP 25.8 c
CDCL3
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 30

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical data\sk4-26-2\data,.als
sk4-26-2

DEFILE C:\Documents and Settings\adr
COMMT sk4-26-2
DATIM Sun Jul 19 15:14:16 2009
OBNUC 1H
EXMOD non
OBFREQ 500.00 MHz
OBSFET 0.00 KHz
OBSFIN 162160.0 Hz
POINT 32768
FREQU 10000.0 Hz
SCANS 8
ACQTM 3.277 sec
PD 3.723 sec
PW1 6.2 us
TENUC 1H
CTEMP 24.4 C
SLVNT CDCL3
EXREF 7.24 ppm
BF 1.20 Hz
RGAIN 14

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical data\sk4-26-2, C, data. als
sk4-26-2

DFILE C:\Documents and Settings\admin
 COMMT sk4-26-2
 DATIM Sun Jul 19 16:02:53 2009
 OBNUC 13C
 bcm
 EXMOD 125.65 MHz
 OBFRQ 0.00 KHz
 OBSFT 127958.0 Hz
 OFIN 32768
 POINT 33898.3 Hz
 FREQU 916
 SCANS 0.967 sec
 ACQTM 2.033 sec
 PD 5.1 us
 PW1 1H
 TENUC 26.4 c
 CTEMP CDCL3
 SLVNT 77.00 ppm
 EXREF 1.20 Hz
 BF 30
 RGAIN

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical data\sk5-45-2,data.als
sk5-45-2

DFILE C:\Documents and Settings\admin
 SK5-45-2
 COMMT sk5-45-2
 DATIM Fri Aug 21 15:06:07 2009
 OBNUC 1H
 EXMOD non
 OBFRQ 500.00 MHz
 OBSFET 0.00 KHz
 OBSFIN 162160.0 Hz
 POINT 32768
 FREQU 10000.0 Hz
 SCANS 8
 ACQTM 3.277 sec
 PD 3.723 sec
 PW1 6.2 us
 TENUC 1H
 CTEMP 24.3 C
 SLVNT CDCL3
 EXREF 7.24 ppm
 BF 1.20 Hz
 RGAIN 12

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical data\sk5-45-2clean.C\data.als
sk5-45-2

C:\Documents and Settings\admin
sk5-45-2
Fri Aug 21 15:41:46 2009
13C
bcm
EXMOD 125.65 MHz
OBPRQ 0.00 KHz
OBSET 127958.0 Hz
OBFIN 32768
POINT 33898.3 Hz
FREQU 672
SCANS 0.967 sec
ACQTM 2.033 sec
PD 5.1 us
PW1 1H
IRNUC 1H
CTEMP 26.7 c
CDCL3
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 30

C:\Documents and Settings\admin\My Documents\NMR\koya\NMR\chemical data\sk5-51-1_data.als

C:\Documents and Settings\adr

1H
 300.01 MHz
 1.85 KHz
 2.7 Hz
 32768
 6172.8 Hz
 8
 0.000 sec
 0.000 sec
 10.0 us
 0.0 c
 7.24 ppm
 1.20 Hz
 0

DEFILE
 COMMT
 DATIM
 OBNUC
 EXMOD
 OBFREQ
 OBSSET
 OBFIN
 POINT
 FREQU
 SCANS
 ACQTM
 PD
 PW1
 TENUC
 CTEMP
 SLVNT
 EXREF
 BF
 RGAIN

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical_data\sk5-51-1_data, C.als
sk5-51-1

DFILE C:\Documents and Settings\admin
COMNT sk5-51-1
DATIM Sat Apr 04 17:52:10 2009
OBNUC 13C
bcm
EXMOD 125.65 MHz
OBFREQ 0.00 KHz
OBSFET 127958.0 Hz
OBFIN 32768
POINT 33898.3 Hz
FREQU 381
SCANS 0.967 sec
ACQTM 2.033 sec
PD 5.1 us
PW1 1H
TENUC 465.0 c
CTEMP CDCL3
SLVNT 77.00 ppm
EXREF 1.20 Hz
BF 30
RGAIN

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical_data\sk5-1-2_data.als

C:\Documents and Settings\admin

1H
 300.01 MHz
 1.85 KHz
 2.7 Hz
 32768
 6172.8 Hz
 8
 0.000 sec
 0.000 sec
 10.0 us
 0.0 c
 7.24 ppm
 1.20 Hz
 0

DFILE
 COMMT
 DATIM
 ORNUC
 EXMOD
 OFRQ
 OBSF
 OFIN
 POINT
 FREQU
 SCANS
 ACQTM
 PD
 PW1
 TENUC
 CTEMP
 SLVNT
 EXREF
 BF
 RGAIN

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical_data\sk5-1-2.data.C.als
sk5-1-2

DFILE C:\Documents and Settings\admin
COMMT sk5-1-2
DATIM Mon Mar 23 15:40:13 2009
OBNUC 13C
bcm
EXMOD 125.65 MHz
OBFREQ 0.00 KHz
OBSFET 127958.0 Hz
OBFIN 32768
POINT 33898.3 Hz
FREQ 840
SCANS 840
ACQTM 0.967 sec
PD 2.033 sec
PW1 5.1 us
1H
TENUC 465.0 c
CTEMP CDCL3
SLVNT 77.00 ppm
EXREF 1.20 Hz
BF 30
RGAIN

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical data\sk5-1-3_data.als

C:\Documents and Settings\admin

1H
 300.01 MHz
 1.85 KHz
 2.7 Hz
 32768
 6172.8 Hz
 8
 0.000 sec
 0.000 sec
 10.0 us
 0.0 c
 7.24 ppm
 1.20 Hz
 0
 RGAIN

DFILE
 COMMT
 DATIM
 OBNUC
 EXMOD
 OBFRO
 OBSFT
 OBFIN
 POINT
 FREQU
 SCANS
 ACQTM
 PD
 PW1
 TENUC
 CTEMP
 SLVNT
 EXREF
 BF
 RGAIN

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical data\sk5-1-3.data.C.als
sk5-1-3

DFILE C:\Documents and Settings\adr
COMNT sk5-1-3
DATIM Thu Apr 09 19:09:09 2009
OENUC 13C
EXMOD bcm
OBFRQ 125.65 MHz
OBSET 0.00 KHz
OBFIN 127958.0 Hz
POINT 32768
FREQU 33898.3 Hz
SCANS 724
AQTM 0.967 sec
PD 2.033 sec
PW1 5.1 us
TENUC 1H
CTEMP 465.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 31

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical data\sk3-62-3.als

C:\Documents and Settings\adr

1H
 300.01 MHz
 1.85 KHz
 2.7 Hz
 32768
 6172.8 Hz
 8
 0.000 sec
 0.000 sec
 10.0 us
 0.0 c
 7.24 ppm
 1.20 Hz
 0
 RGAIN

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical_data\sk3-62-3_data, C.als
sk4-47-4

DFILE C:\Documents and Settings\admin
COMMT sk4-47-4
DATIM Thu May 21 09:29:42 2009
ORNUC 13C
bcm
EXMOD 125.65 MHz
OBFRQ 0.00 KHz
OBSET 127958.0 Hz
OBFIN 32768
POINT 33898.3 Hz
FREQU 13552
SCANS 0.967 sec
ACQTM 2.033 sec
PD 5.1 us
PWL 1H
TENUC 25.8 c
CTEMP CDCL3
SLVNT 77.00 ppm
EXREF 1.20 Hz
BF 30
RGAIN

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical data\sk5-8-2.data.als
sk5-8-2

DFILE C:\Documents and Settings\admin
COMMT sk5-8-2
DATIM Thu Apr 09 21:02:20 2009
OBNUC 1H
EXMOD non
OBFRQ 500.00 MHz
OBSET 0.00 KHz
OBFIN 162160.0 Hz
POINT 32768
FREQ 10000.0 Hz
SCANS 8
ACQTM 3.277 sec
PD 3.723 sec
PWL 6.2 us
TENUC 1H
CTEMP 465.0 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 1.20 Hz
RGAIN 18

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical data\sk5-8-2, data, C.als
sk5-8-2

DFILE C:\Documents and Settings\admin
COMMT sk5-8-2
DATIM Thu Apr 09 21:45:10 2009
OBNUC 13C
bcm
EXMOD 125.65 MHz
OBFREQ 0.00 KHz
OBSFET 127958.0 Hz
OBFIN 32768
POINT 33898.3 Hz
FREQU 824
SCANS 824
ACQTM 0.967 sec
PD 2.033 sec
PW1 5.1 us
1H
TENUC
CTEMP 465.0 c
CDCL3
SLVNT
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 31

C:\Documents and Settings\admin\My Documents\NMR\koya\chemical data\sk5-8-3_data.als
sk5-8-3

D:\Documents and Settings\admin
sk5-8-3
COMNT sk5-8-3
DATIM Thu May 07 22:17:21 2009
OBNUC 1H
EXMOD non
OBFRQ 500.00 MHz
OBSEF 0.00 KHz
OBFIN 162160.0 Hz
POINT 32768
FREQU 10000.0 Hz
SCANS 24
ACQTM 3.277 sec
PD 3.723 sec
PW1 6.2 us
TENUC 1H
CTEMP 465.0 C
SLVNT CDCL3
EXREF 7.24 ppm
BF 1.20 Hz
RGAIN 19

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical_data\sk5-8-3_data.C.als
sk5-8-3

DFILE C:\Documents and Settings\admin
COMNT sk5-8-3
DATIM Fri May 08 09:38:33 2009
OBNUC 13C
EXMOD bcm
OBFRQ 125.65 MHz
OBSET 0.00 KHz
OBFIN 127958.0 Hz
POINT 32768
FREQU 33898.3 Hz
SCANS 13582
ACQTM 0.967 sec
PD 2.033 sec
PW1 5.1 us
TENUC 1H
CTEMP 465.0 C
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 30

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical_data\sk5-9-2-data.als

C:\Documents and Settings\admin

1H
 EXMOD 300.01 MHz
 OBFRQ 1.95 KHz
 OBSET 2.7 Hz
 POINT 32768
 FREQU 6172.8 Hz
 SCANS 8
 ACQTM 0.000 sec
 PD 0.000 sec
 PW1 10.0 us
 TENUC 0.0 c
 CTMP 7.34 ppm
 EXREF 1.20 Hz
 BF 0
 RGAIN 0

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical_data\sk5-9-2_data.C.als
sk5-9-2.C

DFILE C:\Documents and Settings\admin
COMNT sk5-9-2.C
DATIM Sat May 09 09:36:58 2009
OBNUC 13C
EXMOD bcm
OBFRQ 125.65 MHz
OBSET 0.00 KHz
OBSF1 127958.0 Hz
POINT 32768
FREQU 33898.3 Hz
SCANS 13814
ACQTM 0.967 sec
PD 2.033 sec
PW1 5.1 us
TENUC 1H
CTEMP 465.0 C
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 30

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical data\sk5-10-2-data.als

C:\Documents and Settings\admin

1H
 EXMOD 300.01 MHz
 OBSFQ 1.85 KHz
 OBSFT 2.7 Hz
 POINT 32768
 FREQU 6172.8 Hz
 SCANS 8
 ACQTM 0.000 sec
 PD 0.000 sec
 PW1 10.0 us
 TENUC 0.0 c
 CTMP 7.34 ppm
 EXREF 1.20 Hz
 BF 0
 RGAIN 0

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical_data\sk5-10-2,data,C.als
sk5-10-2

DFILE C:\Documents and Settings\admin
COMNT sk5-10-2
DATIM Thu Apr 30 16:21:23 2009
OBNUC 13C
EXMOD bcm
OBFRQ 125.65 MHz
OBSET 0.00 KHz
OBFIN 127958.0 Hz
POINT 32768
FREQU 33898.3 Hz
SCANS 989
ACQTM 0.967 sec
PD 2.033 sec
PW1 5.1 us
1H
TENUC
CTEMP 465.0 C
CDCL3
SLVNT
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 30

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical_data\sk5-10-3_data.als

C:\Documents and Settings\admin

1H
 600.13 MHz
 3.70 KHz
 6.0 Hz
 32768
 12376.2 Hz
 8
 0.000 sec
 0.000 sec
 10.0 us
 0.0 c
 7.24 ppm
 1.20 Hz
 0
 RGAIN

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical_data\sk5-10-3_data,Ca.als

C:\Documents and Settings\admin

13C

EXMOD	150.91 MHz
OBFRQ	7.89 KHz
OBSET	8.8 Hz
OBFIN	32768
POINT	35971.2 Hz
FREQ	8
SCANS	0.000 sec
ACQTM	0.000 sec
PD	10.0 us
PW1	0.0 c
TEMP	215.51 ppm
SLVNT	1.20 Hz
EXREF	0
BF	
RGAIN

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical data\sk5-27-2\data.als

C:\Documents and Settings\admin

1H
 EXMOD 300.01 MHz
 OBSFQ 1.95 KHz
 OBSFT 2.7 Hz
 POINT 32768
 FREQU 6172.8 Hz
 SCANS 8
 ACQTM 0.000 sec
 PD 0.000 sec
 PW1 10.0 us
 TENUC 0.0 c
 CTMP 7.34 ppm
 EXREF 1.20 Hz
 BF 0
 RGAIN 0

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical_data\sk5-27-2_data.C.als

C:\Documents and Settings\admin

13C

DFILE	COMMT	DATIM	OBNUC	EXMOD	OBFRQ	OBSET	OBFIN	POINT	FREQU	SCANS	ACQTM	PD	PW1	TENUC	CTEMP	SLVNT	EXREF	BF	RGAIN
			13C		75.44 MHz	5.11 KHz	8.3 Hz	32768	17985.6 Hz	8	0.000 sec	0.000 sec	10.0 us		0.0 C		215.54 ppm	1.20 Hz	0

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical data\sk5-27-3, data.als
sk5-27-3

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical_data\sk5-27-3_data.C.als
sk5-27-3

DFILE C:\Documents and Settings\admin
 COMNT sk5-27-3
 DATIM Fri May 01 09:52:02 2009
 OBNUC 13C
 EXMOD bcm
 OBFRQ 125.65 MHz
 OBSET 0.00 KHz
 OBFIN 127958.0 Hz
 POINT 32768
 FREQU 33898.3 Hz
 SCANS 14011
 ACQTM 0.967 sec
 PD 2.033 sec
 PW1 5.1 us
 TENUC 1H
 CTEMP 465.0 C
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 30

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical data\sk5-7-2.data.als
sk5-7-2

DFILE C:\Documents and Settings\admin
COMNT sk5-7-2
DATIM Mon Apr 06 17:03:33 2009
OBNUC 1H
EXMOD non
EXFREQ 500.00 MHz
OBSEF 0.00 KHz
OBFIN 162160.0 Hz
POINT 32768
FREQU 10000.0 Hz
SCANS 8
ACQTM 3.277 sec
PD 3.723 sec
PW1 6.2 us
TENUC 1H
CTEMP 465.0 C
SLVNT CDCL3
EXREF 7.24 ppm
BF 1.20 Hz
RGAIN 18

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical_data\sk5-7-2_data.C.als
sk5-7-2

DFILE C:\Documents and Settings\admin
COMNT sk5-7-2
DATIM Mon Apr 06 17:39:03 2009
OBNUC 13C
EXMOD bcm
OBFRQ 125.65 MHz
OBSET 0.00 KHz
OBFIN 127958.0 Hz
POINT 32768
FREQU 33898.3 Hz
SCANS 666
ACQTM 0.967 sec
PD 2.033 sec
PW1 5.1 us
TENUC 1H
CTEMP 465.0 C
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 30

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical data\sk5-7-3.data.als
sk5-7-3

C:\Documents and Settings\admin\sk5-7-3
Thu Apr 23 20:55:48 2009
1H
non
EXMOD 500.00 MHz
OBFRQ 0.00 KHz
OBSET 162160.0 Hz
OBFIN 32768
POINT 10000.0 Hz
SCANS 8
ACQTM 3.277 sec
PD 3.723 sec
PW1 6.2 us
TENUC 1H
CTEMP 465.0 C
SLVNT CDCL3
EXREF 7.24 ppm
BF 1.20 Hz
RGAIN 20

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical_data\sk5-7-3_data.C.als
sk5-7-3

DFILE C:\Documents and Settings\admin
COMNT sk5-7-3
DATIM Thu Apr 23 21:45:22 2009
OBNUC 13C
EXMOD bcm
OBFRQ 125.65 MHz
OBSET 0.00 KHz
OBFIN 127958.0 Hz
POINT 32768
FREQU 33898.3 Hz
SCANS 928
ACQTM 0.967 sec
PD 2.033 sec
PW1 5.1 us
TENUC 1H
CTEMP 465.0 C
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 31

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical data\sk5-61-2.data.als

C:\Documents and Settings\adr

1H
 EXMOD 300.01 MHz
 OBSFQ 1.85 KHz
 OBSFT 2.7 Hz
 POINT 32768
 FREQU 6172.8 Hz
 SCANS 8
 ACQTM 0.000 sec
 PD 0.000 sec
 PW1 10.0 us
 TENUC 0.0 c
 CTEMP 7.24 ppm
 SLVNT 1.20 Hz
 EXREF 0
 RGAIN 0

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical data\sk5-61-2,data,C.als
sk5-61-2

DFILE C:\Documents and Settings\admin
COMMT sk5-61-2
DATIM Mon Jun 01 09:19:34 2009
OBNUC 13C
EXMOD bcm
OBFRQ 125.65 MHz
OBSET 0.00 KHz
OBFIN 127958.0 Hz
POINT 32768
FREQU 33898.3 Hz
SCANS 13824
ACQTM 0.967 sec
PD 2.033 sec
PW1 5.1 us
TENUC 1H
CTEMP 25.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 30

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical_data\sk5-61-3_data.als

C:\Documents and Settings\admin

Parameter	Value
DFILE	1H
COMNT	
DATIM	
OBNUC	
EXMOD	300.01 MHz
OBFRQ	1.95 KHz
OBSET	2.7 Hz
OBFIN	32768
POINT	6172.8 Hz
FREQU	8
SCANS	0.000 sec
ACQTM	0.000 sec
PD	10.0 us
PW1	0.0 c
TEHUC	
CTEMP	7.34 ppm
SLVNT	1.20 Hz
EXREF	
BF	0
RGAIN	

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical data\sk5-61-3\data, C.als
sk5-61-3

DFILE C:\Documents and Settings\adr
COMMT sk5-61-3
DATIM Fri May 29 15:22:20 2009
OBNUC 13C
EXMOD bcm
OBFREQ 125.65 MHz
OBFSET 0.00 KHz
OBFIN 127958.0 Hz
POINT 32768
FREQU 33898.3 Hz
SCANS 882
ACQTM 0.967 sec
PD 2.033 sec
PW1 5.1 us
TENUC 1H
CTEMP 25.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 30

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical data\sk5-61-4.als

C:\Documents and Settings\adr

1H

- 300.01 MHz
- 1.85 KHz
- 2.7 Hz
- 32768
- 6172.8 Hz
- 8
- 0.000 sec
- 0.000 sec
- 10.0 us
- 0.0 c
- 7.24 ppm
- 1.20 Hz
- 0

DFILE
COMMT
DATIM
ORNUC
EXMOD
OBFREQ
OBSSET
OBSFIN
POINT
FREQU
SCANS
ACQTM
PD
PWL
TENUIC
CTEMP
SLVNT
EXREF
BF
RGAIN

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical_data\sk5-61-4_data.C.als

C:\Documents and Settings\admin

DFILE
 COMNT
 DATIM
 OBNUC 13C
 EXMOD
 OBFRQ 150.91 MHz
 OBSET 7.89 KHz
 OBFIN 8.8 Hz
 POINT 32768
 FREQU 35971.2 Hz
 SCANS 8
 ACQTM 0.000 sec
 PD 0.000 sec
 PW1 10.0 us
 TENUC
 CTEMP 0.0 C
 SLVNT
 EXREF 215.51 ppm
 BF 1.20 Hz
 RGAIN 0

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical data\sk3-66-3.data.als

C:\Documents and Settings\adr

1H
300.01 MHz
1.85 KHz
2.7 Hz
32768
6172.8 Hz
8
0.000 sec
0.000 sec
10.0 us
0.0 c
7.24 ppm
1.20 Hz
0

DEFILE
COMMT
DATIM
ORNUC
EXMOD
OBFRO
OBSFT
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
TENUC
CTEMP
SLVNT
EXREF
BF
RGAIN

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical data\sk3-66-3\data, C.als
sk3-66-3

DFILE C:\Documents and Settings\admin
COMMT sk3-66-3
DATIM Sun May 10 17:55:49 2009
OBNUC 13C
bcm
EXMOD 125.65 MHz
OBFRQ 0.00 KHz
OBSET 127958.0 Hz
OBFIN 32768
POINT 33898.3 Hz
FREQU 1822
SCANS 0.967 sec
ACQTM 2.033 sec
PD 5.1 us
PW1 1H
TENUC 465.0 c
CTEMP CDCL3
SLVNT 77.00 ppm
EXREF 1.20 Hz
BF 30
RGAIN

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical data\sk3-49-2.data.als

C:\Documents and Settings\admin

1H
 EXMOD 300.01 MHz
 OBSFQ 1.95 KHz
 OBSFT 2.7 Hz
 POINT 32768
 FREQU 6172.8 Hz
 SCANS 8
 ACQTM 0.000 sec
 PD 0.000 sec
 PW1 10.0 us
 TENUC 0.0 c
 CTMP 7.34 ppm
 EXREF 1.20 Hz
 BF 0
 RGAIN 0

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical_data\sk3-49-2\data, C.als
sk3-49-2

DFILE C:\Documents and Settings\admin
 COMNT sk3-49-2
 DATIM Sun May 31 18:52:21 2009
 OBNUC 13C
 EXMOD bcm
 OBFRQ 125.65 MHz
 OBSEF 0.00 KHz
 OBFIN 127958.0 Hz
 POINT 32768
 FREQU 33898.3 Hz
 SCANS 2451
 ACQTM 0.967 sec
 PD 2.033 sec
 PW1 5.1 us
 TENUC 1H
 CTMP 26.0 c
 CDCL3
 SLVNT
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 30

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical data\sk3-10-2\data.als
sk3-10-2

DFILE C:\Documents and Settings\admin
 sk3-10-2
 COMNT Sun May 31 14:06:05 2009
 DATIM 1H
 OBNUC non
 EXMOD 500.00 MHz
 OBFRQ 0.00 KHz
 OBSET 162160.0 Hz
 OFIN 32768
 POINT 10000.0 Hz
 FREQU 8
 SCANS 3.277 sec
 ACQTM 3.723 sec
 PD 6.2 us
 PW1 1H
 TENUC 23.6 C
 CTMP CDCL3
 SLVNT 7.34 ppm
 EXREF 1.20 Hz
 BF 13
 RGAIN

C:\Documents and Settings\admin\My Documents\NMR\koya\HNMR\chemical data\sk3-10-2,data,C.als
sk3-10-2

DFILE sk3-10-2
 COMNT Sun May 31 14:54:55 2009
 DATIM 13C
 OBNUC bcm
 EXMOD 125.65 MHz
 OBFRQ 0.00 KHz
 OBSET 127958.0 Hz
 OBFIN 32768
 POINT 33898.3 Hz
 SCANS 922
 FREQU 0.967 sec
 ACQTM 2.033 sec
 PD 5.1 us
 PW1 1H
 TENUC 25.8 c
 CTMP CDCL3
 SLVNT 77.00 ppm
 EXREF 1.20 Hz
 BF 28
 RGAIN