The Supporting Information

Asymmetric Total Synthesis of Halicholactone

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General. Melting points are uncorrected. IR spectra were obtained using a Horiba FT-210 spectrometer. ¹H NMR spectra were obtained using JEOL JNM-GX-500 (500 MHz) and JEOL JNM-LA-500 (500 MHz) spectrometer. ¹³C NMR spectra were obtained using JEOL JNM-EX-270 (67.8 MHz), JEOL JNM-AL-300 (75.5 MHz) and JNM-LA-500 (125.7 MHz) spectrometer. Optical rotations were measured with a JASCO DIP-360 polarimeter. Mass spectra (MS) were measured with a Shimadzu GCMS-QP-1000 spectrometer. High resolution mass spectra (HRMS) were measured with JEOL JMS-D300 and JEOL JMS-600 spectrometer. Column chromatography was carried out using Merck Kieselgel 60. Toluene and THF were distilled from sodium benzophenone ketyl radical under argon. Dichloromethane was freshly distilled from calcium hydride. Dry ether and acetonitrile were obtained from Kanto Chemicals.

(2R,3S,4S,7R,8R,4E,6E)-Tricarbonyliron[(η⁴-4-7)-1,8-bis(*tert*-butyldimethylsilyloxy)trideca-4,6-dienyl-2,3-diol] (14b). To a stirred solution of **8** (251 mg, 0.503 mmol) in pyridine (2.0 ml) was added TBSOTf (0.12 ml, 0.503 mmol) at -15 °C under nitrogen atmosphere. After 30 min, brine was added to the reaction mixture, and the resulting mixture was extracted with AcOEt. The extract was washed with brine, dried over MgSO₄, and then concentrated *in vacuo*, and pyridine was removed azeotropically with toluene. The residue was purified by column chromatography (SiO₂, hexane:AcOEt=10:1) to give **14b** (174 mg, 56%) and **8**

(92 mg, 37%). **14b**: yellow crystals; mp 65–66 °C (hexane/AcOEt); R_f 0.44 (hexane/AcOEt = 4/1); $[\alpha]_D^{23} +26.3$ ($c = 1.00$, CHCl_3); ^1H NMR (CDCl_3) δ 0.07 (s, 3H), 0.10 (s, 3H), 0.30 (s, 6H), 0.89 (t, 3H, $J = 5.5$ Hz), 0.91 (s, 18H), 1.23–1.46 (m, 8H), 1.56–1.60 (m, 2H), 2.62 (d, 1H, $J = 6.1$ Hz), 3.02 (d, 1H, $J = 4.9$ Hz), 3.43 (ddd, 1H, $J = 5.5, 5.5, 9.2$ Hz), 3.51 (ddd, 1H, $J = 1.8, 4.9, 7.3$ Hz), 3.64 (dddd, 1H, $J = 1.8, 3.7, 5.5, 6.1$ Hz), 3.72 (d, 1H, $J = 5.5, 10.4$ Hz), 3.77 (dd, 1H, $J = 4.3, 10.4$ Hz), 5.19 (dd, 1H, $J = 4.9, 9.1$ Hz), 5.33 (dd, 1H, $J = 4.9, 9.1$ Hz); ^{13}C NMR (CDCl_3 , 75.5 MHz) δ -5.2, -4.2, -3.7, -3.6, 14.0, 18.11, 18.19, 22.6, 24.5, 25.8 (3C), 25.9 (3C), 31.7, 39.2, 61.8, 66.0, 67.1, 73.7, 74.2, 74.8, 83.4, 84.6, 211.3; IR (KBr) 3426, 2050, 1982 cm^{-1} ; MS (EI) m/z (%) 397 ($\text{M}^+ - 3\text{CO} - \text{TBS}$, 2), 75 (100); *Anal.* Calcd for $\text{C}_{28}\text{H}_{52}\text{FeO}_7\text{Si}_2$: C, 54.88; H, 8.55. Found: C, 54.59; H, 8.43.

(1S,2S,5R,6R,2E,4E)-Tricarbonyliron{[(η^4 -2-5)-1-[(1R)-1-chloroacetoxy-2-*tert*-butyldimetylsilyloxyethyl]-6-*tert*-butyldimetylsilyloxyundeca-2,4-dienyl]} Chloroacetate (16b)
 To a stirred solution of **14b** (400 mg, 0.653 mmol) in dry CH_2Cl_2 (6.0 ml) was added DMAP (398 mg, 3.26 mmol) and chloroacetic anhydride (500 mg, 2.93 mmol) at 0 °C under nitrogen atmosphere. The reaction mixture was stirred for 30 min at 0 °C and at room temperature for 2.5 h. After brine was added to the reaction mixture, and the resulting mixture was extracted with AcOEt. The extract was washed with brine, dried over MgSO_4 , and then concentrated *in vacuo*. The residue was purified by column chromatography (SiO_2 , hexane/AcOEt = 10/1) to give **16b** (402 mg, 81%) as yellow crystals: mp 103–104 °C (pentane); R_f 0.57 (hexane/AcOEt = 10/1 $\times$ 2); $[\alpha]_D^{28} +5.0$ ($c = 1.01$, CHCl_3); ^1H NMR (CDCl_3) δ 0.01 (s, 3H), 0.03 (s, 3H), 0.07 (s, 3H), 0.09 (s, 3H), 0.84 (s, 9H), 0.88 (s, 12H), 1.02 (dd, 1H, $J = 9.1, 9.1$ Hz), 1.24–1.42 (m, 7H), 1.54–1.58 (m, 2H), 3.44 (ddd, 1H, $J = 4.8, 5.5, 8.5$ Hz), 3.52 (t, 1H, $J = 9.2$ Hz), 3.80 (dd, 1H, $J = 5.5, 9.7$ Hz), 4.08 (s, 4H), 4.94 (ddd, 1H, $J = 2.4, 4.8, 5.5$ Hz), 5.09 (dd, 1H, $J = 2.4, 9.1$ Hz), 5.18 (dd, 1H, $J = 4.9, 8.5$ Hz), 5.52 (dd, 1H, $J = 4.9, 8.5$ Hz); ^{13}C NMR (CDCl_3 , 67.8 MHz) δ -5.8, -5.7, -4.2, -3.7, 14.0, 18.0, 18.1, 22.6, 24.2, 25.6 (3C), 25.9 (3C), 31.7, 39.0, 40.7, 40.9, 53.9, 59.3, 67.7, 74.5, 74.9, 77.4, 84.0, 85.4, 166.5, 166.6, 211.0; IR (KBr) 2036, 1971, 1764 cm^{-1} ; MS (EI) m/z (%) 456 ($\text{M}^+ - 3\text{CO} - \text{OTBS} - \text{ClCH}_2\text{CO}_2$, 5), 308 (58), 75 (100); *Anal.* Calcd for $\text{C}_{32}\text{H}_{54}\text{Cl}_2\text{FeO}_9\text{Si}_2$: C, 50.17; H, 7.11. Found: C, 50.18; H, 7.05.

(2R,3S,4S,7R,8R,4E,6E)-Tricarbonyliron[(η^4 -4-7)-1-*tert*-butyldimethylsilyloxy-8-phenylthiotrideca-4,6-dienyl-2,3-diol] (15) Method A: To a stirred solution of **14b** (30.8 mg, 0.0503 mmol) in dry CH_2Cl_2 (0.5 ml) was added TMSSPh (10.5 μl , 0.0553 mmol) and $\text{Sc}(\text{OTf})_3$ (5.0 mg, 0.0101 mmol) at -78°C . The reaction mixture was stirred at -78°C to room temperature for 5 h under nitrogen atmosphere. After a saturated NaHCO_3 solution was added to the reaction mixture, and the resulting mixture was extracted with AcOEt . The extract was washed with brine, dried over MgSO_4 , and then concentrated *in vacuo*. The residue was purified by column chromatography (SiO_2 , hexane/ AcOEt = 5/1) to give **15** (18.3 mg, 62%) as a yellow oil. Method B: To a stirred solution of **14b** (22.0 mg, 0.0359 mmol) in dry CH_2Cl_2 (0.5 ml) was added $\text{CH}(\text{SPh})_3$ (24.5 mg, 0.0728 mmol) and *p*- $\text{TsOH}\cdot\text{H}_2\text{O}$ (1.0 mg, 5.28 μmol) at room temperature. After 30 min, water was added to the reaction mixture, and the resulting mixture was extracted with AcOEt . The extract was washed with brine, dried over MgSO_4 , and then concentrated *in vacuo*. The residue was purified by column chromatography (SiO_2 , hexane/ AcOEt = 10/1 to 5/1 to 2/1) to give **15** (5.6 mg, 27%). Method C: To a stirred solution of **14b** (32.4 mg, 0.0529 mmol) in dry CH_2Cl_2 (0.6 ml) was added $\text{CH}(\text{SPh})_3$ (27.0 mg, 0.0793 mmol) and Dowex 50W (34 mg) at room temperature. After 3 h, the solvent was removed *in vacuo*. The residue was purified by column chromatography (SiO_2 , hexane/ AcOEt = 6/1) to give **15** (18.6 mg, 63%); R_f 0.29 (hexane/ AcOEt = 4/1). $[\alpha]_D^{25} +67.9$ (c = 0.90, CHCl_3); ^1H NMR (CDCl_3) δ : 0.088 (s, 3H), 0.094 (s, 3H), 0.90 (s, 9H), 0.92 (t, 3H, J = 6.7 Hz), 1.24–1.37 (m, 7H), 1.55–1.63 (m, 2H), 1.77–1.84 (m, 1H), 2.56 (d, 1H, J = 6.1 Hz), 2.81 (dt, 1H, J = 8.5, 3.1 Hz), 3.00 (d, 1H, J = 4.9 Hz), 3.46 (ddd, 1H, J = 1.8, 4.9, 7.3 Hz), 3.62 (dddd, 1H, J = 1.8, 3.7, 5.5, 6.1 Hz), 3.71 (dd, 1H, J = 5.5, 10.6 Hz), 3.76 (dd, 1H, J = 3.7, 10.6 Hz), 4.52 (dd, 1H, J = 4.9, 8.5 Hz), 5.15 (dd, 1H, J = 4.9, 8.5 Hz), 7.33–7.34 (m, 3H), 7.45 (m, 2H); ^{13}C NMR (CDCl_3 , 75.5 MHz) δ –5.6, –5.5, 14.1, 18.2, 22.5, 25.8 (3C), 26.1, 31.5, 36.6, 55.4, 61.5, 66.2, 67.6, 73.6, 74.4, 82.9, 83.4, 128.2, 129.0 (2C), 133.7, 135.2 (2C), 203.2; IR (KBr) 3425, 2042, 1975 cm^{-1} ; MS (FAB) m/z 597 ($\text{M}+\text{Li}$) $^+$; HRMS (FAB) Calcd for $\text{C}_{28}\text{H}_{42}\text{FeLiO}_6\text{SSi}$ ($\text{M}+\text{Li}$) $^+$: 597.7981. Found: 597.7998.

The cyclopropanation of 22. To a stirred solution of **22** (41.6 mg, 0.104 mmol) in dry CH_2Cl_2 (1.1 ml) was added dropwise diethyl zinc (1.0 M in hexane, 0.63 ml, 0.626 mmol) and diiodomethane (83.7 μl , 1.04 mmol) at 0°C under argon atmosphere. After 4 h, a saturated NH_4Cl

solution was added to the mixture, and the resulting mixture was extracted with AcOEt. The extract was washed with brine, dried over MgSO_4 , and then concentrated *in vacuo*. The residue was purified by PTLC (hexane/AcOEt = 5/1 $\times$ 3) to give mono-cyclopropane **B** (4.4 mg, 10%) and **23** (4.5 mg, 10%). **B**: a colorless oil; R_f 0.39 (hexane/AcOEt = 5/1 $\times$ 3); ^1H NMR (CDCl_3) δ 0.009 (s, 3H), 0.0013 (s, 3H), 0.52 (m, 2H), 0.87–0.90 (m, 13H), 0.98 (m, 1H), 1.25–1.51 (m, 9H), 1.39 (s, 3H), 1.43 (s, 3H), 3.28 (m, 1H), 3.58 (m, 2H), 4.07 (dd, 1H, J = 6.1, 8.5 Hz), 4.22 (dd, 1H, J = 3.0, 11.6 Hz), 4.51 (ddd, 1H, J = 6.1, 6.1, 7.3 Hz), 5.69 (dd, 1H, J = 7.3, 15.3 Hz), 5.90 (dd, 1H, J = 5.5, 15.3 Hz); IR (KBr) 3406 cm^{-1} ; MS (FAB) m/z 435 ($\text{M}+\text{Na}^+$); HRMS (FAB) Calcd for $\text{C}_{23}\text{H}_{44}\text{NaO}_4\text{Si}$ ($\text{M}+\text{Na}^+$): 435.2907. Found: 435.2903; **23**: a colorless oil; R_f 0.38 (hexane/AcOEt = 5/1 $\times$ 3); ^1H NMR (CDCl_3) δ 0.02 (s, 3H), 0.03 (s, 3H), 0.44–0.55 (m, 4H), 0.88–0.90 (m, 14H), 1.05 (m, 2H), 1.19–1.53 (m, 9H), 1.33 (s, 3H), 1.43 (s, 3H), 2.57 (dd, 1H, J = 7.9, 7.9 Hz), 3.29 (m, 1H), 3.55 (m, 1H), 3.68 (m, 1H), 4.05 (m, 1H); IR (KBr) 3421 cm^{-1} ; MS (FAB) m/z 449 ($\text{M}+\text{Na}^+$); HRMS (FAB) Calcd for $\text{C}_{24}\text{H}_{46}\text{NaO}_4\text{Si}$ ($\text{M}+\text{Na}^+$): 449.3067. Found: 449.3051.

(1S)-1-((1R,2R)-2-[(1R,4R,2E)-4-*tert*-Butyldimethylsilyloxy-1-(2-trimethylsilyl)ethoxymethoxynon-2-enyl]cyclopropyl)but-3-enyl (S)- α -Methoxy- α -(trifluoromethyl)phenylacetate. To a stirred solution of **28b** (3.5 mg, 6.82 μmol) in dry CH_2Cl_2 (0.5 ml) was added (S)-methoxy- α -(trifluoromethyl)phenylacetic acid (3.7 mg, 0.0157 mmol), DMAP (1.9 mg, 1.57 μmol) and DCC (4.6 mg, 0.0225 mmol) at room temperature. After 16 h, the solvent was removed *in vacuo*. The residue was purified by PTLC (hexane/AcOEt = 7/1) to give (S)-MTPA ester (3.1 mg, 61%) as a colorless oil: R_f 0.45 (hexane/AcOEt = 7/1); ^1H NMR (CDCl_3) δ -0.01 (s, 3H), 0.02 (s, 9H), 0.03 (s, 3H), 0.52 (ddd, 1H, J = 4.3, 4.9, 8.5 Hz), 0.69 (ddd, 1H, J = 4.9, 4.9, 8.5 Hz), 0.86–0.90 (m, 5H), 0.89 (s, 9H), 1.18 (m, 1H), 1.24–1.30 (m, 7H), 1.42 (m, 2H), 2.42 (m, 2H), 3.52 (ddd, 1H, J = 6.1, 9.7, 14.0 Hz), 3.58 (s, 3H), 3.67 (ddd, 1H, J = 6.1, 9.7, 14.0 Hz), 3.94 (m, 1H), 4.06 (q, 1H, J = 6.1 Hz), 4.55 (d, 1H, J = 6.7 Hz), 4.60 (d, 1H, J = 6.7 Hz), 4.63 (m, 1H), 4.99 (m, 2H), 5.34 (dd, 1H, J = 7.3, 15.3 Hz), 5.60 (dd, 1H, J = 5.5, 15.3 Hz), 5.63 (m, 1H), 7.39 (m, 3H), 7.56 (m, 2H); IR (KBr) $1747, 1171\text{ cm}^{-1}$; MS (FAB) m/z 751 ($\text{M}+\text{Na}^+$); HRMS (FAB) Calcd for $\text{C}_{38}\text{H}_{63}\text{FNaO}_6\text{Si}_2$ ($\text{M}+\text{Na}^+$): 751.4013. Found: 751.3999.

(1S)-1-((1R,2R)-2-[(1R,4R,2E)-4-*tert*-Butyldimethylsilyloxy-1-(2-trimethylsilyl)ethoxymethoxynon-2-enyl]cyclopropyl)but-3-enyl (R)- α -Methoxy- α -

(trifluoromethyl)phenylacetate. The same treatment of **28b** (3.6 mg, 7.02 μmol) with (*R*)-methoxy- α -(trifluoromethyl)phenylacetic acid (3.8 mg, 0.0161 mmol) as described for (*S*)-MTPA ester to give (*R*)-MTPA ester (3.0 mg, 60%) as a colorless oil; R_f 0.45 (hexane/AcOEt = 7/1); ^1H NMR (CDCl_3) δ -0.001 (s, 3H), 0.003 (s, 9H), 0.03 (s, 3H), 0.47 (ddd, 1H, J = 5.4, 5.4, 9.2 Hz), 0.66 (ddd, 1H, J = 4.9, 4.9, 8.5 Hz), 0.89 (s, 14H), 1.08 (m, 1H), 1.11–1.32 (m, 7H), 1.34–1.36 (m, 2H), 2.51 (m, 2H), 3.43 (m, 1H), 3.54 (s, 3H), 3.62 (ddd, 1H, J = 6.1, 9.7, 14.7 Hz), 3.75 (dd, 1H, J = 4.9, 7.9 Hz), 4.03 (m, 1H), 4.52 (d, 1H, J = 6.7 Hz), 4.55 (d, 1H, J = 6.7 Hz), 4.70 (m, 1H), 5.10 (m, 2H), 5.22 (dd, 1H, J = 7.9, 15.3 Hz), 5.57 (dd, 1H, J = 6.1, 15.3 Hz), 5.79 (m, 1H), 7.39 (m, 3H), 7.55 (m, 2H); IR (KBr) 1747, 1171 cm^{-1} ; MS (FAB) m/z 751 ($\text{M}+\text{Na}^+$); HRMS (FAB) Calcd for $\text{C}_{38}\text{H}_{63}\text{FNaO}_6\text{Si}_2$ ($\text{M}+\text{Na}^+$): 751.4013. Found: 751.3983.

(1S)-1-((1R,2R)-2-[(1R,4R,2E)-4-*tert*-Butyldimethylsilyloxy-1-(2-trimethylsilyl)ethoxymethoxynon-2-enyl]cyclopropyl}but-3-enyl Hex-5-enoate (29) To a stirred solution of **28b** (42.8 mg, 0.835 mmol) in dry CH_2Cl_2 (0.8 ml) was added 5-hexenoic acid (10.9 μl , 0.0918 mmol), DMAP (12.2 mg, 0.100 mmol) and DCC (20.7 mg, 0.100 mmol) at room temperature. After 17 h, the solvent was removed *in vacuo*. The residue was purified by PTLC (hexane/AcOEt = 7/1) to give **29** (29.1 mg, 57%) and **28b** (3.1 mg, 7%). **29**: a colorless oil; R_f 0.52 (hexane/AcOEt = 7/1); $[\alpha]_D^{26}$ -63.4 (c = 1.19, CHCl_3); ^1H NMR (CDCl_3) δ 0.019 (s, 9H), 0.03 (s, 3H), 0.04 (s, 3H), 0.55 (ddd, 1H, J = 5.5, 5.5, 8.5 Hz), 0.64 (ddd, 1H, J = 4.9, 4.9, 8.6 Hz), 0.86–0.97 (m, 6H), 0.89 (s, 9H), 1.06–1.11 (m, 1H), 1.27–1.40 (m, 6H), 1.44 (m, 2H), 1.71 (m, 2H), 2.08 (m, 2H), 2.28 (t, 2H, J = 7.3 Hz), 2.37 (m, 2H), 3.49 (ddd, 1H, J = 6.1, 10.4, 14.6 Hz), 3.57 (t, 1H, J = 7.3 Hz), 3.71 (ddd, 1H, J = 6.1, 10.4, 14.6 Hz), 4.10 (q, 1H, J = 5.5 Hz), 4.47 (m, 1H), 4.62 (s, 2H), 4.97–5.09 (m, 4H), 5.43 (dd, 1H, J = 7.3, 15.3 Hz), 5.62 (dd, 1H, J = 5.5, 15.3 Hz), 5.77 (m, 2H); ^{13}C NMR (CDCl_3 , 75.5 MHz) δ -4.8, -4.3, -1.4 (3C), 8.8, 14.0, 18.1, 18.2, 19.2, 21.0, 22.6, 24.2, 24.9, 25.9 (3C), 31.8, 33.1, 33.8, 38.4, 39.0, 64.9, 72.6, 75.5, 77.6, 91.3, 115.3, 117.5, 127.4, 133.7, 137.3, 137.7, 173.0; IR (KBr) 2954, 1736, 1643 cm^{-1} ; MS (FAB) m/z 615 ($\text{M}+\text{Li}^+$); HRMS (FAB) Calcd for $\text{C}_{34}\text{H}_{64}\text{LiO}_5\text{Si}_2$ ($\text{M}+\text{Li}^+$): 615.4452. Found: 615.4424.

15-*tert*-Butyldimethylsilyloxy-12-(2-trimethylsilyl)ethoxymethoxyhalicholactone (30) The ester **29** (31.0 mg, 0.0509 mmol) and freshly distilled $\text{Ti}(\text{O}i\text{-Pr})_4$ (4.5 μl , 0.0153 mmol) were dissolved in dry CH_2Cl_2 (8.0 ml), and the mixture was refluxed for 1 h under argon atmosphere. The

ruthenium carbene (**A**) (3.4 mg, 4.07 μmol) in dry CH_2Cl_2 (0.5 ml) was then added, and reflux was continued for 30 h. The mixture was cooled to room temperature and filtered through a short pad of SiO_2 . The solvent was removed *in vacuo*. The residue was purified by PTLC (hexane/AcOEt = 7/1) to give **30** (5.5 mg, 19%) and dimer (4.6 mg, 8%). **30**: a colorless oil; R_f 0.41 (hexane/AcOEt = 7/1); $[\alpha]_D^{26} -97.1$ ($c = 0.23$, CHCl_3); ^1H NMR (CDCl_3) δ 0.025 (s, 9H), 0.030 (s, 3H), 0.04 (s, 3H); 0.60 (ddd, 1H, $J = 4.9, 4.9, 8.5$ Hz), 0.68 (ddd, 1H, $J = 4.9, 4.9, 8.6$ Hz), 0.86–0.95 (m, 5H), 0.89 (s, 9H), 0.99 (m, 1H), 1.06–1.11 (m, 1H), 1.25–1.32 (m, 6H), 1.39–1.49 (m, 2H), 1.72–1.81 (m, 2H), 2.04–2.15 (m, 2H), 2.20–2.32 (m, 2H), 2.45 (m, 2H), 3.50 (ddd, 1H, $J = 6.7, 10.3, 14.0$ Hz), 3.58 (t, 1H, $J = 7.3$ Hz), 3.72 (ddd, 1H, $J = 6.1, 10.3, 14.0$ Hz), 4.10 (q, 1H, $J = 6.1$ Hz), 4.23 (m, 1H), 4.64 (s, 2H), 5.45–5.50 (m, 3H), 5.63 (dd, 1H, $J = 6.1, 15.3$ Hz); ^{13}C NMR (CDCl_3 , 75.5 MHz) δ -4.8, -4.2, -1.4 (3C), 8.6, 14.0, 18.1, 18.2, 19.2, 21.4, 22.6, 24.9, 25.2, 25.9 (3C), 26.5, 31.8, 33.6, 33.9, 38.4, 64.9, 72.7, 75.9, 77.7, 91.4, 124.8, 127.7, 134.6, 137.1, 174.1; IR (KBr) 2953, 1743 cm^{-1} ; MS (FAB) m/z 603 ($\text{M}+\text{Na}^+$); HRMS (FAB) Calcd for $\text{C}_{32}\text{H}_{60}\text{NaO}_5\text{Si}_2$ ($\text{M}+\text{Na}^+$): 603.3877. Found: 603.3871; dimer of **30**: a colorless oil; R_f 0.31 (hexane/AcOEt = 7/1); ^1H NMR (CDCl_3) δ 0.02 (s, 18H), 0.03 (s, 6H), 0.04 (s, 6H), 0.55 (m, 2H), 0.62 (m, 2H), 0.87–0.97 (m, 30H), 0.99 (m, 2H), 1.06–1.13 (m, 2H), 1.20–1.30 (m, 12H), 1.42–1.48 (m, 2H), 1.62 (m, 4H), 1.99 (m, 4H), 2.13–2.44 (m, 8H), 3.50 (m, 2H), 3.57 (t, 2H, $J = 7.3$ Hz), 3.71 (m, 2H), 4.10 (m, 2H), 4.50 (m, 2H), 4.62 (s, 4H), 5.29–5.47 (m, 6H), 5.91–5.64 (m, 2H); IR (KBr): 2954, 1735 cm^{-1} ; MS (FAB) m/z 1184 ($\text{M}+\text{Na}^+$); HRMS (FAB) Calcd for $\text{C}_{64}\text{H}_{120}\text{NaO}_{10}\text{Si}_4$ ($\text{M}+\text{Na}^+$): 1183.7854. Found: 1183.7865.

Table 1. Crystal data and structure refinement for 16b.

Identification code	16b
Empirical formula	C ₃₂ H ₅₄ Cl ₂ Fe O ₉ Si ₂
Formula weight	765.68
Temperature	293(2) K
Wavelength	0.71069 Å
Crystal system	Monoclinic
Space group	P2
Unit cell dimensions	a = 7.542(2) Å alpha = 90.000(14) deg. b = 15.619(4) Å beta = 93.720(13) deg. c = 17.529(3) Å gamma = 90.001(12) deg.
Volume	2060.5(7) Å ³
Z	2
Density (calculated)	1.234 Mg/m ³
Absorption coefficient	0.598 mm ⁻¹
F(000)	812
Crystal size	0.7 x 0.5 x 0.3 mm
Theta range for data collection	2.33 to 25.00 deg.
Index ranges	-9 ≤ h ≤ 9, -20 ≤ k ≤ 20, -22 ≤ l ≤ 22
Reflections collected	8134
Independent reflections	7241 [R(int) = 0.1275]
Absorption correction	PSI
Max. and min. transmission	0.998 and 0.521
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	6609 / 1 / 469
Goodness-of-fit on F ²	1.034
Final R indices [I > 2σ(I)]	R1 = 0.1167, wR2 = 0.2830
R indices (all data)	R1 = 0.2368, wR2 = 0.5906
Absolute structure parameter	-0.03(6)
Largest diff. peak and hole	0.879 and -1.121 e.Å ⁻³

Table 2. Atomic coordinates (x 10⁻⁴) and equivalent isotropic displacement parameters (Å² x 10⁻³) for 1. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
Fe(1)	5839(2)	-5068(1)	-30(1)	45(1)
Cl(1)	-41(6)	-8179(3)	272(3)	87(2)
Cl(2)	-398(9)	-1979(4)	1745(4)	122(2)
Si(1)	3758(5)	-4905(3)	3679(2)	66(1)
Si(2)	3264(6)	-3345(3)	-2685(3)	77(1)
O(1)	3822(12)	-5286(7)	2800(5)	68(3)

O(2)	1473(10)	-5977(5)	1050(5)	41(2)
O(3)	2522(13)	-7285(6)	1292(7)	66(3)
O(4)	1482(10)	-4238(6)	1407(6)	50(2)
O(5)	2945(16)	-2987(8)	1608(9)	91(4)
O(6)	3661(16)	-4299(8)	-2363(7)	77(3)
O(7)	9219(11)	-5197(8)	-758(7)	75(3)
O(8)	4437(17)	-6795(7)	-290(7)	84(3)
O(9)	7716(11)	-5163(10)	1478(7)	81(3)
C(1)	2386(17)	-5638(9)	2379(8)	52(3)
C(2)	2872(15)	-5564(8)	1541(8)	42(3)
C(3)	3141(15)	-4698(8)	1282(8)	45(3)
C(4)	3492(14)	-4601(8)	405(8)	44(3)
C(5)	4577(19)	-3963(8)	188(10)	56(4)
C(6)	5046(19)	-3963(9)	-611(9)	58(4)
C(7)	4407(17)	-4624(9)	-1042(9)	55(3)
C(8)	5010(22)	-4694(12)	-1850(9)	68(5)
C(9)	5167(24)	-5597(12)	-2128(9)	68(5)
C(10)	5966(67)	-5658(21)	-2953(14)	161(17)
C(11)	6007(82)	-6508(27)	-3224(17)	201(23)
C(12)	6754(100)	-6510(31)	-4016(19)	317(44)
C(13)	8609(86)	-6533(32)	-3993(23)	252(31)
C(14)	2173(24)	-4053(16)	3658(15)	103(7)
C(15)	3091(29)	-5718(17)	4325(11)	100(7)
C(16)	6085(21)	-4527(12)	3913(9)	69(4)
C(17)	7315(24)	-5282(25)	3898(16)	142(13)
C(18)	6627(20)	-3875(17)	3341(14)	96(7)
C(19)	6327(38)	-4104(22)	4713(13)	123(9)
C(20)	1504(15)	-6819(9)	973(9)	52(3)
C(21)	40(17)	-7071(10)	404(10)	62(4)
C(22)	1566(25)	-3411(11)	1593(10)	76(5)
C(23)	-265(25)	-3105(12)	1716(12)	85(6)
C(24)	2864(26)	-2538(13)	-1940(15)	93(6)
C(25)	5182(36)	-2976(22)	-3183(16)	120(8)
C(26)	1267(30)	-3494(14)	-3323(13)	97(6)
C(27)	-203(39)	-3873(26)	-2876(32)	183(17)
C(28)	700(33)	-2593(23)	-3702(24)	145(14)
C(29)	1716(41)	-4076(25)	-4020(16)	145(12)
C(30)	7866(16)	-5179(12)	-507(8)	64(4)
C(31)	5005(19)	-6166(9)	-188(10)	57(4)
C(32)	6970(13)	-5119(12)	882(9)	56(3)

Table 3. Selected bond lengths [Å] and angles [deg] for 1.

Symmetry transformations used to generate equivalent atoms:

Table 4. Bond lengths [Å] and angles [deg] for 1.

Fe(1)-C(32)	1.76(2)
Fe(1)-C(30)	1.798(14)
Fe(1)-C(31)	1.842(14)
Fe(1)-C(5)	2.019(13)
Fe(1)-C(6)	2.072(14)
Fe(1)-C(4)	2.103(12)
Fe(1)-C(7)	2.13(2)
Cl(1)-C(21)	1.75(2)
Cl(2)-C(23)	1.76(2)
Si(1)-O(1)	1.655(9)
Si(1)-C(14)	1.79(2)
Si(1)-C(15)	1.79(2)
Si(1)-C(16)	1.87(2)
Si(2)-O(6)	1.614(13)
Si(2)-C(26)	1.83(2)
Si(2)-C(25)	1.83(3)
Si(2)-C(24)	1.85(2)
O(1)-C(1)	1.38(2)
O(2)-C(20)	1.32(2)
O(2)-C(2)	1.47(2)
O(3)-C(20)	1.17(2)
O(4)-C(22)	1.33(2)
O(4)-C(3)	1.47(2)
O(5)-C(22)	1.23(2)
O(6)-C(8)	1.45(2)
O(7)-C(30)	1.14(2)
O(8)-C(31)	1.08(2)
O(9)-C(32)	1.16(2)
C(1)-C(2)	1.54(2)
C(2)-C(3)	1.45(2)
C(3)-C(4)	1.58(2)
C(4)-C(5)	1.36(2)
C(5)-C(6)	1.47(2)
C(6)-C(7)	1.35(2)

C(7)-C(8)	1.52(2)
C(8)-C(9)	1.50(2)
C(9)-C(10)	1.60(3)
C(10)-C(11)	1.41(5)
C(11)-C(12)	1.53(4)
C(12)-C(13)	1.40(9)
C(16)-C(17)	1.50(4)
C(16)-C(18)	1.50(3)
C(16)-C(19)	1.55(3)
C(20)-C(21)	1.49(2)
C(22)-C(23)	1.49(2)
C(26)-C(27)	1.52(4)
C(26)-C(29)	1.58(4)
C(26)-C(28)	1.60(3)
C(32)-Fe(1)-C(30)	92.4(5)
C(32)-Fe(1)-C(31)	103.6(8)
C(30)-Fe(1)-C(31)	97.6(8)
C(32)-Fe(1)-C(5)	94.2(7)
C(30)-Fe(1)-C(5)	126.6(7)
C(31)-Fe(1)-C(5)	131.6(6)
C(32)-Fe(1)-C(6)	126.2(8)
C(30)-Fe(1)-C(6)	94.5(7)
C(31)-Fe(1)-C(6)	128.1(6)
C(5)-Fe(1)-C(6)	42.0(6)
C(32)-Fe(1)-C(4)	93.2(5)
C(30)-Fe(1)-C(4)	164.4(7)
C(31)-Fe(1)-C(4)	95.2(6)
C(5)-Fe(1)-C(4)	38.5(5)
C(6)-Fe(1)-C(4)	70.5(5)
C(32)-Fe(1)-C(7)	163.2(7)
C(30)-Fe(1)-C(7)	92.6(6)
C(31)-Fe(1)-C(7)	91.6(6)
C(5)-Fe(1)-C(7)	70.1(6)
C(6)-Fe(1)-C(7)	37.4(6)
C(4)-Fe(1)-C(7)	78.2(5)
O(1)-Si(1)-C(14)	108.0(10)
O(1)-Si(1)-C(15)	111.1(8)
C(14)-Si(1)-C(15)	108.9(11)
O(1)-Si(1)-C(16)	103.5(6)
C(14)-Si(1)-C(16)	112.7(10)
C(15)-Si(1)-C(16)	112.5(9)
O(6)-Si(2)-C(26)	102.9(8)
O(6)-Si(2)-C(25)	108.8(12)
C(26)-Si(2)-C(25)	113.1(12)

O(6)-Si(2)-C(24)	114.6(10)
C(26)-Si(2)-C(24)	110.6(9)
C(25)-Si(2)-C(24)	107.0(13)
C(1)-O(1)-Si(1)	124.7(9)
C(20)-O(2)-C(2)	118.7(9)
C(22)-O(4)-C(3)	118.8(11)
C(8)-O(6)-Si(2)	135.7(11)
O(1)-C(1)-C(2)	104.6(11)
C(3)-C(2)-O(2)	109.8(10)
C(3)-C(2)-C(1)	114.6(12)
O(2)-C(2)-C(1)	108.6(10)
C(2)-C(3)-O(4)	105.8(10)
C(2)-C(3)-C(4)	115.5(10)
O(4)-C(3)-C(4)	107.1(10)
C(5)-C(4)-C(3)	118.9(12)
C(5)-C(4)-Fe(1)	67.4(7)
C(3)-C(4)-Fe(1)	121.1(8)
C(4)-C(5)-C(6)	117.0(14)
C(4)-C(5)-Fe(1)	74.1(8)
C(6)-C(5)-Fe(1)	70.9(8)
C(7)-C(6)-C(5)	115.8(13)
C(7)-C(6)-Fe(1)	73.7(9)
C(5)-C(6)-Fe(1)	67.1(7)
C(6)-C(7)-C(8)	117.3(13)
C(6)-C(7)-Fe(1)	68.9(9)
C(8)-C(7)-Fe(1)	125.9(10)
O(6)-C(8)-C(9)	105.4(14)
O(6)-C(8)-C(7)	107.9(13)
C(9)-C(8)-C(7)	113.9(13)
C(8)-C(9)-C(10)	113(2)
C(11)-C(10)-C(9)	112(3)
C(10)-C(11)-C(12)	109(4)
C(13)-C(12)-C(11)	114(5)
C(17)-C(16)-C(18)	109(2)
C(17)-C(16)-C(19)	108(2)
C(18)-C(16)-C(19)	107(2)
C(17)-C(16)-Si(1)	109(2)
C(18)-C(16)-Si(1)	111.0(11)
C(19)-C(16)-Si(1)	112.7(14)
O(3)-C(20)-O(2)	125.7(12)
O(3)-C(20)-C(21)	126.0(14)
O(2)-C(20)-C(21)	108.3(12)
C(20)-C(21)-Cl(1)	111.6(11)
O(5)-C(22)-O(4)	123.5(14)

O(5)-C(22)-C(23)	128(2)
O(4)-C(22)-C(23)	108(2)
C(22)-C(23)-Cl(2)	112.3(14)
C(27)-C(26)-C(29)	112(2)
C(27)-C(26)-C(28)	112(2)
C(29)-C(26)-C(28)	105(3)
C(27)-C(26)-Si(2)	109(2)
C(29)-C(26)-Si(2)	110(2)
C(28)-C(26)-Si(2)	109(2)
O(7)-C(30)-Fe(1)	173.4(13)
O(8)-C(31)-Fe(1)	176.5(14)
O(9)-C(32)-Fe(1)	179(2)

Symmetry transformations used to generate equivalent atoms:

Table 5. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for 1.

The anisotropic displacement factor exponent takes the form:

$$-2\pi^2 [h^2 a^{*2} U_{11} + \dots + 2hk a^* b^* U_{12}]$$

	U11	U22	U33	U23	U13	U12
Fe(1)	40(1)	38(1)	57(1)	2(1)	4(1)	1(1)
Cl(1)	111(3)	49(2)	99(4)	-16(2)	-18(3)	-21(2)
Cl(2)	164(5)	74(3)	130(5)	-19(3)	18(4)	65(4)
Si(1)	66(2)	93(4)	38(2)	-7(2)	-1(2)	-7(2)
Si(2)	86(3)	70(3)	74(3)	6(2)	-3(3)	16(2)
O(1)	82(5)	101(10)	19(4)	-15(5)	-9(4)	-23(6)
O(2)	42(4)	24(4)	57(6)	0(4)	-3(4)	-3(3)
O(3)	67(6)	39(5)	88(8)	2(5)	-22(6)	1(5)
O(4)	45(4)	50(6)	56(6)	0(4)	2(4)	8(4)
O(5)	75(7)	68(8)	130(13)	-44(8)	7(7)	-7(6)
O(6)	103(8)	65(7)	62(7)	4(6)	-1(6)	14(6)
O(7)	53(5)	75(8)	101(8)	-8(7)	29(5)	7(6)
O(8)	115(9)	48(7)	90(9)	-4(6)	12(7)	-26(6)
O(9)	54(4)	108(10)	78(8)	12(8)	-20(5)	-4(7)
C(1)	60(8)	53(9)	41(8)	-12(6)	3(6)	-4(6)
C(2)	41(6)	42(8)	43(8)	8(6)	-3(6)	-4(5)
C(3)	37(6)	44(7)	53(8)	-15(6)	-1(5)	-6(5)
C(4)	44(6)	38(7)	49(8)	-3(6)	-8(5)	-6(5)
C(5)	70(8)	10(5)	88(12)	8(6)	10(8)	1(5)
C(6)	69(8)	33(8)	72(11)	10(7)	12(8)	0(6)
C(7)	53(7)	43(8)	69(10)	6(7)	3(7)	-11(6)
C(8)	83(10)	97(13)	24(7)	6(8)	-1(7)	-11(9)

C(9)	99(12)	68(11)	39(9)	-3(8)	23(8)	11(9)
C(10)	299(52)	117(22)	66(16)	-7(14)	6(19)	105(29)
C(11)	361(61)	172(33)	76(19)	11(19)	70(25)	165(39)
C(12)	618(105)	247(48)	101(25)	44(26)	145(43)	328(65)
C(13)	456(82)	193(46)	115(31)	0(26)	82(41)	184(52)
C(14)	78(12)	108(18)	124(20)	-1(16)	27(12)	29(11)
C(15)	118(16)	123(19)	60(12)	-16(12)	10(11)	-23(14)
C(16)	70(9)	90(12)	46(9)	-18(8)	-19(7)	-9(8)
C(17)	59(10)	254(42)	109(19)	41(23)	-12(10)	46(18)
C(18)	58(9)	139(19)	91(15)	22(14)	4(9)	-5(11)
C(19)	137(21)	166(27)	67(15)	-20(17)	2(12)	-44(22)
C(20)	37(6)	58(10)	61(10)	4(7)	0(6)	-11(6)
C(21)	61(8)	45(8)	79(12)	-15(8)	-2(7)	-10(7)
C(22)	109(13)	52(10)	66(11)	-21(8)	-6(9)	49(10)
C(23)	96(12)	65(11)	101(15)	-14(10)	53(12)	24(9)
C(24)	92(12)	60(11)	124(18)	-20(12)	-8(11)	31(10)
C(25)	125(17)	125(22)	111(21)	6(17)	13(16)	-29(17)
C(26)	127(16)	85(14)	77(14)	12(11)	-12(12)	29(13)
C(27)	86(15)	139(27)	329(52)	-47(33)	49(24)	28(18)
C(28)	109(18)	129(24)	189(34)	65(28)	-56(19)	5(16)
C(29)	144(21)	192(36)	95(20)	-16(22)	-32(18)	-25(24)
C(30)	59(7)	72(11)	58(9)	-22(8)	-11(6)	24(8)
C(31)	63(8)	29(8)	79(12)	-3(7)	-3(8)	-7(6)
C(32)	38(5)	51(8)	80(10)	-9(9)	3(6)	1(7)

Table 6. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 1.

	x	y	z	U(eq)
H(1A)	2231	-6236	2517	56(39)
H(1B)	1304	-5330	2459	36(30)
H(2)	3973	-5885	1487	67(44)
H(3)	4131	-4430	1594	53(38)
H(4)	2947	-4963	55	2000(3957)
H(5)	5006	-3544	520	234(157)
H(6)	5744	-3533	-799	39(31)
H(7)	3625	-5022	-862	57(34)
H(8)	6135	-4402	-1881	128(79)
H(9A)	5921	-5911	-1758	78(53)
H(9B)	4006	-5856	-2161	35(33)
H(10A)	7241	-5451	-2913	154(151)

H(10B)	5357	-5257	-3315	57(36)
H(11A)	6488	-6921	-2874	185(122)
H(11B)	4657	-6708	-3327	298(307)
H(12A)	6540	-5920	-4255	194(113)
H(12B)	6415	-6910	-4353	43(33)
H(13A)	9083	-6780	-4451	111(64)
H(13B)	9223	-5945	-3954	50(38)
H(13C)	9150	-6843	-3557	75(48)
H(14A)	2148	-3805	4162	147(99)
H(14B)	2490	-3621	3305	81(59)
H(14C)	1010	-4272	3508	89(54)
H(15A)	2729	-5465	4782	23(25)
H(15B)	2189	-6064	4088	41(33)
H(15C)	4136	-6084	4460	106(68)
H(17A)	6664	-5788	4143	55(43)
H(17B)	7512	-5458	3405	225(173)
H(17C)	8358	-5204	4214	197(115)
H(18A)	5699	-3460	3265	50(34)
H(18B)	7690	-3578	3561	32(27)
H(18C)	6887	-4141	2884	303(170)
H(19A)	6309	-4545	5100	72(50)
H(19B)	7499	-3835	4761	60(38)
H(19C)	5442	-3698	4773	135(107)
H(21A)	-1083	-6886	599	44(32)
H(21B)	184	-6793	-71	1008(481)
H(23A)	-1074	-3314	1299	184(134)
H(23B)	-651	-3340	2186	57(36)
H(24A)	1881	-2198	-2097	0(18)
H(24B)	2692	-2825	-1469	328(280)
H(24C)	3926	-2181	-1870	176(119)
H(25A)	4715	-2594	-3598	34(30)
H(25B)	5920	-2641	-2836	28(25)
H(25C)	5764	-3436	-3382	239(197)
H(27A)	372	-4210	-2442	315(290)
H(27B)	-812	-3399	-2619	19(23)
H(27C)	-982	-4199	-3160	402(592)
H(28A)	1584	-2372	-4006	24(28)
H(28B)	-422	-2626	-3981	43(33)
H(28C)	556	-2165	-3279	166(157)
H(29A)	2669	-3795	-4309	56(42)
H(29B)	2306	-4613	-3825	140(101)
H(29C)	771	-4208	-4353	276(217)

1H Line

15.18

10.67

4.58

3.89

Date : Fri Jan 16 15:50:16 1998

Filename : LoadingID.nmdata

Comment : 1H Line

SlcHistory : non

EXMODE :

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SAMPO 16384 points

FREQ 10000.0 Hz

FILTR 5000 Hz

DELA 40.0 usec

DEAD 58.7 usec

INTVL 100.0 usec

TIMES 16 times

DUMMY 1 times

PD 5.3616 sec

ACQTM 1638.3999 msec

PREDL 0.01000 msec

ININT 1000.0000 msec

RESOL 0.51 Hz

PW1 2.63 usec

1H 500.00 MHz

OBNUC 162167.63 Hz

OBFRQ 17

OBSET

RGAIN

SCANS 16 times

SLVNT CDCL3

SPINNT 14 Hz

TEMP 23.7 C

15-JAN-98 10:33:51

DFILE Q13C

OBNUC 13C

EXMOD BCM

OFR 67.80 MHz

OBSET 135.00 kHz

OBFIN 5200.0 Hz

POINT 32768

FREQ 20000.0 Hz

SCANS 165

ACQTM 0.819 sec

PD 2.181 sec

PW1 3.8 us

IRNUC 1H

CTEMP 21.3 c

SLVNT CDCL3

EXREF 77.00 ppm

BF 1.50 Hz

RGAIN 27

OPERATOR :

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 DATIM Fri May 01 13:21:59 1998
 OBNUC 13C
 EXMOD BCM
 OBPRQ 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.0 Hz
 POINT 32768
 FREQU 20408.1 Hz
 SCANS 128
 ACQTM 1.606 sec
 PD 1.394 sec
 PW1 4.0 us
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 CTMP 24.8 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 24

1H Line

Date : Wed Jul 14 20:38:02 1999

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Comment : 1H Line

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EXMODE : non

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 DUMMY 1 times
 PD 5.3616 sec
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 INIWT 1000.0000 msec
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 OBPRQ 500.00 MHz
 OBSET 162167.63 Hz
 RGAIN 21

SCANS : 16 times

SLVNT : CDCL3

SPINNING : 13 Hz

TEMP : 22.8 C

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18.32

12.09

3.51

1.04

0.72

1.11

2.19

3.54

1.27

2.25

1.00

1.00

11.31

200MHz

0

2

4

6

DFILE C:\WINNMR95\COMMON_DEFAULT.
COMNT diol
DATIM Fri Aug 20 14:25:23 1999
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EXMOD BCM
OBFREQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
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ACQTM 1.606 sec
PD 1.394 sec
PWL 4.0 us
IRNUC 1H
CTEMP 22.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 24

1H Line

Date : Wed Sep 1 10:29:34 1999

FileName : LoadingID.rmdata

Comment : 1H Line

SlcHistory : non

EXMODE

POINT 16384 points

SAMPD 16384 points

FREQ 10000.0 Hz

FILTR 5000 Hz

DELAY 40.0 usec

DEADT 58.7 usec

INTVL 100.0 usec

TIMES 32 times

DUMM1 1 times

PO 5.3616 sec

ACQTM 1638.3999 msec

PREDL 0.01000 msec

IN1MT 1000.0000 msec

RESOL 0.61 Hz

PH1 2.63 usec

OBNUC 1H

OBFRQ 500.00 MHz

OBSET 162167.63 Hz

PGAIN 24

SCANS 32 times

SLVNT CDCL3

SPINNING 13 Hz

TEMP 24.3 C

DFILE C:\WINNMR95\COMMON_DEFAULT.ALS
 COMNT allyl(S,lesspolar)
 DATIM Tue Aug 31 15:14:20 1999
 OBNUC 13C
 EXMOD BCM
 OBFRQ 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.0 Hz
 POINT 32768
 FREQU 20408.1 Hz
 SCANS 166
 ACQTM 1.606 sec
 PD 1.394 sec
 PW1 4.0 us
 IRNUC 1H
 CTEMP 21.6 C
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 24.

1H Line

Date : Wed Sep 1 10: 41: 30 1999
 FileName : .LoadingFID.nmdata
 Comment : 1H Line
 SliceHistory : non
 EXMODE :
 POINT : 16384 points
 SAMPD : 16384 points
 FREQU : 10000.0 Hz
 FILTR : 5000 Hz
 DELAY : 40.0 usec
 DEADT : 58.7 usec
 INVL : 100.0 usec
 TIMES : 32 times
 DUMMY : 1 times
 PO : 5.3616 sec
 ACQTM : 1638.3999 msec
 PREDL : 0.01000 msec
 INIWT : 1000.0000 msec
 RESOL : 0.61 Hz
 PM1 : 2.63 usec
 OBNUC : ¹H
 OBPRQ : 500.00 MHz
 OBSET : 162167.63 Hz
 RGAIN : 24
 SCANS : 32 times
 SLVNT : CDCL3
 SPINNING : 12 Hz
 TEMP : 24.0 C

DFILE C:\WINNMR95\COMMON\DEFAULT.ALS
 COMNT allyl(R,polar)
 DATIM Tue Aug 31 15:44:45 1999
 OBNUC 13C
 EXMOD BCM
 OBFRQ 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.0 Hz
 POINT 32768
 FREQU 20408.1 Hz
 SCANS 513
 ACQTM 1.606 sec
 PD 1.394 sec
 PW1 4.0 us
 IRNUC 1H
 CTEMP 21.4 C
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 24

1H Line

Date : Fri Nov 5 15:56:16 1999
 FileName : LoadingFID.mdata
 Comment : 1H Line
 SliceHistory : non
 EXMODE : non

POINT 16384 points
 SAMPO 16384 points
 FREQ 10000.0 Hz
 FILTR 5000 Hz
 DELAY 40.0 usec
 DEATH 58.7 usec
 INTR 100.0 usec
 TIMES 16 times
 DUMMY 1 times
 PD 5.3616 sec
 ACQTM 1638.3999 msec
 PREOL 0.01000 msec
 ININT 1000.0000 msec
 RESOL 0.61 Hz
 PH1 2.63 usec
 OBNJC 500.00 MHz
 OBFRO 162167.63 Hz
 OBSET 22
 RGAIN 16 times

SCANS 16 times
 SLVNT CDCL3
 SPINNING 10 Hz
 TEMP 27.2 C

```

C:\WINMR95\COMMON_DEFAULT.ALS
-----
FILE      COUNT
DATE      EE
TIME      THU Nov 04 13:30:47 1999
NAME      13C
PROC      EXMOD BCM
F1        75.45 MHz
F2        124.00 KHz
F3        1840.0 Hz
F4        32768
F5        20408.1 Hz
F6        513
F7        1.606 sec
F8        1.394 sec
F9        PD
F10       4.0 us
F11       1H
F12       IRNUC
F13       CTMP
F14       24.7 C
F15       CDCL3
F16       SLVNT
F17       77.00 ppm
F18       EXREF
F19       1.20 Hz
F20       BF
F21       24
F22       RGAIN

```

1H Line

Date : Wed Nov 10 13:18:57 1999

FileName : se1201.nmdata

Comment : 1H Line

SliceHistory :

EXMODE : non

POINT : 16384 points

SAMPD : 16384 points

FREQD : 100000.0 Hz

FILTR : 5000 Hz

DELAY : 40.0 usec

DEADT : 58.7 usec

INVL : 100.0 usec

TIMES : 32 times

DUMMY : 1 times

PD : 5.3616 sec

ACQTM : 1638.3999 msec

PREDL : 0.01000 msec

INITI : 1000.0000 msec

RESOL : 0.61 Hz

PWI : 2.63 usec

OBNUC : 1H

OBFRQ : 500.00 MHz

OBSET : 162167.63 Hz

RGAIN : 24

SCANS : 32 times

SLVNT : CDCL3

SPINNT : 15 Hz

TEMP : 30.3 C

DFILE C:\WINMR95\COMMON_DEFAULT.ALS
 CONNT diol(Ee)
 DATIM Mon Nov 08 10:24:32 1999
 OBNUC 13C
 EXMOD BCM
 OBFREQ 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.0 Hz
 POINT 32768
 FREQU 20408.1 Hz
 SCANS 291
 ACQTM 1.606 sec
 PD 1.394 sec
 PW1 4.0 us
 IRNUC 1H
 CTEMP 24.7 C
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 24

1H Line

Date : Wed Nov 10 14:00:56 1999

FileName : se1202.nmdata

Comment : 1H Line

SliceHistory

EXMODE : non

POINT : 16384 points

SAMPD : 16384 points

FREQU : 10000.0 Hz

FILTR : 5000 Hz

DELAY : 40.0 usec

DEAOT : 58.7 usec

INVL : 100.0 usec

TIMES : 32 times

DUMMY : 1 times

PD : 5.3616 sec

ACQTM : 1638.3999 msec

PREDL : 0.01000 msec

ININT : 1000.0000 msec

RESOL : 0.81 Hz

PWT : 2.63 usec

OBNUC : 1H

OBFRQ : 500.00 MHz

OBSET : 162167.63 Hz

RGAIN : 25

SCANS : 32 times

SLVNT : CDCL3

SPINNT : 13 Hz

TEMP : 28.7 C

C:\WINNER95\COMMON_DEFAULT.ALS
 COMNT Ac(EE)
 DATIM Tue Nov 09 22:11:11 1999
 OBNUC 13C
 EXMOD BCM
 OBFRQ 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.0 Hz
 POINT 32768
 FREQU 20408.1 Hz
 SCANS 201
 ACQTM 1.606 sec
 PD 1.394 sec
 PW1 4.0 us
 IRNUC 1H
 CTEMP 24.3 C
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 24

Date	File Name	Comment	Loading ID	nmdata
Time	File Name	Comment	1H Line	
Wed Oct 13 10:13:48 1999	EXMODE	SliceHistory	non	
	POINT			16384 points
	SAMPO			16384 points
	FREQU			10000.0 Hz
	FILTR			5000 Hz
	DELAY			40.0 usec
	DEAOT			58.7 usec
	INTVL			100.0 usec
	TIMES			16 times
	DUMMY			1 times
	PD			5.3616 sec
	ACQTM			1638.3959 msec
	PREDL			0.01000 msec
	INITI			1000.0000 msec
	RESOL			0.61 Hz
	PW1			2.63 usec
	OBNUC		1H	
	OBFRQ			500.00 MHz
	OBSET			162167.63 Hz
	RGAIN			22
	SCANS			16 times
	SOLVN		COCL3	
	SPINNING			
	TEMP			12 Hz
				22.6 C

DFILE C:\WINMR95\COMMON\DEFAULT.ALS
 COMNT OHdIAC
 DATIM Sat Nov 13 16:43:23 1999
 OBNUC 13C
 EXMOD BCM
 OBFREQ 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.0 Hz
 POINT 32768
 FREQU 20408.1 Hz
 SCANS 341
 ACQTM 1.606 sec
 PD 1.394 sec
 PW1 4.0 us
 IRNUC 1H
 CTEMP 25.5 C
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 24

8.784
 13.935
 20.512
 21.278
 22.457
 22.663
 24.682
 31.432
 34.111
 41.569
 73.646
 74.289
 76.571
 76.992
 77.420

117.755
 130.183
 131.123
 134.551

170.295
 170.800

PPM

1H Line

Date: Wed Nov 17 10:48:27 1999
 Filename: LoadingFD.nmdata
 Comment: 1H Line
 SliceHistory: non
 EXMODE: non
 POINT: 16384 points
 SAMPD: 16384 points
 FREQD: 10000.0 Hz
 FILTR: 5000 Hz
 DELAY: 40.0 usec
 DEAT: 58.7 usec
 INTR: 100.0 usec
 TIMES: 48 times
 DUMY: 1 times
 PD: 5.3616 sec
 ACQIM: 1638.3999 msec
 PREDL: 0.01000 msec
 ININT: 1000.0000 msec
 RESOL: 0.61 Hz
 PW1: 2.63 usec
 1H: 500.00 MHz
 162167.63 Hz
 21
 SCANS: 48 times
 SLVNT: C6D6
 SPINNT: 12 Hz
 TEMP: 27.7 C

C:\WINMR95\COMMON_DEFAULT.ALS
 triene(OAc)
 Tue Nov 16 17:53:28 1999
 13C
 EXMOD BCM
 OBFRQ 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.0 Hz
 POINT 32768
 FREQU 20408.1 Hz
 SCANS 253
 ACQTM 1.606 sec
 PD 1.394 sec
 PW1 4.0 us
 IRNUC 1H
 CTEMP 24.0 C
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 24

1H Line

Date : Wed Oct 27 14:01:36 1999
 FileName : seizonl.nmdata
 Comment : 1H Line
 SliceHistory : non

POINT 16384 points
 SAMP0 16384 points
 FREQ0 10000.0 Hz
 FILTR 5000 Hz
 DELAY 40.0 usec
 DEADT 58.7 usec
 INTVL 100.0 usec
 TIMES 128 times
 DUMM1 1 times
 PD 5.3616 sec
 ACOTM 1638.3999 msec
 PREDL 0.01000 msec
 INIHT 1000.0000 msec
 RESOL 0.61 Hz
 PWT 2.63 usec
 OBNUC 1H 500.00 MHz
 OBFRQ 162167.63 Hz
 OBSET 25
 RGAIN 128 times
 SCANS 128 times
 SLVNT C6D6
 SPINNING 12 Hz
 TEMP 24.5 C

C:\WINMR95\COMMON_DEFAULT.ALS
 CONNT RCM
 DATIM Fri Nov 19 21:22:18 1999
 OBNUC 13C
 EXMOD BCM
 OBERQ 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.0 Hz
 POINT 32768
 FREQU 20408.1 Hz
 SCANS 1401
 ACQTM 1.606 sec
 PD 1.394 sec
 PW1 4.0 us
 IRNUC 1H
 CTEMP 24.5 C
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 24

1H Line

Date : Wed Nov 24 14:55:02 1999
File Name : LoadingFID.nmdata
Comment : 1H Line
Slice History : non
EXMODE :
POINT : 16384 points
SAMPD : 16384 points
FREQU : 10000.0 Hz
FILTR : 5000 Hz
DELAY : 40.0 usec
DEADT : 58.7 usec
INTVL : 100.0 usec
TIMES : 128 times
DUMMY : 1 times
PD : 5.3616 sec
ACQTM : 1638.3999 msec
PREDL : 0.01000 msec
ININT : 1000.0000 msec
RESOL : 0.61 Hz
PWI : 2.63 usec
1H :
OBNUC :
OBFRQ : 500.00 MHz
OBSET : 162167.63 Hz
RGAIN : 25
SCANS : 128 times
SLVNT : C6D6
SPINNING : 11 Hz
TEMP : 27.7 C

1H Line

Date : Mon Dec 6 17:23:48 1999
 FileName : seizo7.nmdata
 Comment : 1H Line
 SliceHistory :
 EXMODE : bcm

POINT 16384 points
 SAMPO 16384 points
 FREQ 33898.3 Hz
 F1L1R 16950 Hz
 DELAY 11.8 usec
 DEAT 15.1 usec
 IN1VL 29.5 usec
 TIMES 2000 times
 DUMMY 1 times
 P0 2.5167 sec
 ACQTM 483.3280 msec
 PREDL 0.01000 msec
 IN1T 1000.0000 msec
 RESOL 2.07 Hz
 PW1 5.10 usec
 13C 125.65 MHz
 OBPRO 127958.00 Hz
 OBSET 28
 RGAIN
 LRNUC 500.00 MHz
 LRSET 162160.00 Hz
 LRPH 48.0 usec
 LRNS 0

SCANS 2000 times
 SLVNT CDCL3
 SPINNING 13 Hz
 TEMP 32.6 C

Figure 1. X-ray structure of **16b** drawn by Chem 3D