Supporting Information

Stereocontrolled Total Synthesis of a Polyfunctional Carotenoid, Peridinin

Noriyuki Furuichi, Hirokazu Hara, Takashi Osaki, Masayuki Nakano, Hajime Mori,
and Shigeo Katsumura*

*School of Science and Technology, Kwansei Gakuin University,
Gakuen 2-1, Sanda, Hyogo 669-1337, Japan*

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I. General Procedure

Unless otherwise specified, the reactions were carried out in oven-dried glassware under an argon atmosphere. All commercially available reagents were used without further purification. All solvents were used after distillation. Tetrahydrofuran (THF), diethyl ether, benzene, toluene, and dimethoxyethane (DME) were refluxed over and distilled from sodium-benzophenone ketyl. Dichloromethane was refluxed over and distilled from P₂O₅. Dimethylformamide (DMF), dimethyl sulfoxide (DMSO), and hexamethylphosphoric triamide (HMPA) were distilled from CaH₂ under reduced pressure. Methanol was refluxed over and distilled from Mg(OMe)₂. Triethylamine, diisopropylamine, and diisopropylethylamine were refluxed over and distilled from KOH. Preparative separation was performed by column chromatography on silica gel or alumina (~150 mesh, 58 Å). ¹H and ¹³C NMR spectra were recorded at 400 and 100 MHz, respectively, and chemical shifts were represented as δ -values relative to the internal standard TMS. IR spectra were recorded with an FT-IR spectrometer. Melting points were uncorrected.

II. Experimental

(4R)-tert-Butyldimethylsiloxy-2,2,6-trimethylcyclohexene -1-methanol (6). To a solution of **5**² (23.0 g, 57.1 mmol) in DMF (170 mL) was added tetrakis(triphenylphosphine)palladium (1.95 g, 1.69 mmol), triethylamine (24.3 mL, 171 mmol) and methanol (70 mL). After being stirred for 3 h at 70 °C under CO atmosphere, the reaction mixture was poured into a saturated aqueous NH₄Cl solution, and then extracted with ethyl acetate. The organic layers were combined, washed with brine, dried over MgSO₄, filtered and concentrated *in vacuo*. Purification by silica gel column chromatography (from 5% to 10% ethyl acetate in hexane) afforded the corresponding methyl ester (17.4 g, 97%) as a colorless oil: [α]_D²⁴ –50.45 (c 0.79, CHCl₃); IR (NaCl, cm^{–1}) 2975, 2930, 2359, 1724, 1462, 1433, 1383, 1252, 1227, 1086, 1040; ¹H NMR (400 MHz, CDCl₃) δ 3.99 (m, 1H), 3.74 (s, 3H), 2.22 (ddd, 1H, J = 17.3, 5.6, 1.5 Hz), 2.02 (ddd, 1H, J = 17.3, 9.3, 1.0 Hz), 1.67 (s, 3H), 1.63 (ddd, 1H, J = 12.4, 3.7, 1.7 Hz), 1.47 (dd, 1H, J = 12.0, 12.0 Hz), 1.20 (s, 3H), 1.04 (s, 3H), 0.89 (s, 9H), 0.053 (s, 3H), 0.047 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 170.9, 134.5, 132.3, 65.2, 51.1, 47.7, 41.5, 35.8, 29.2, 28.5, 25.8, 21.1, 18.1, –4.65, –4.66; EI⁺ HRMS Found m/z 312.2122, Calcd. for C₁₇H₃₂O₃Si M⁺ 312.2119.

To a suspension of lithium aluminum hydride (4.23 g, 111.4 mmol) in THF (200 mL) was added dropwise a solution of the methyl ester (17.4 g, 55.7 mmol) obtained above in THF (50 mL) at 0 °C. The reaction mixture was stirred at 45 °C for 20 h, and water-saturated ether and then water were successively added. The precipitate was removed by filtration through a pad of Celite, and the filtrate was concentrated *in vacuo*. Purification of the obtained oil by silica gel column chromatography (from 10% to 30% ethyl acetate in hexane) afforded allyl alcohol **6**¹⁶ (9.18 g, 87%) as a white solid.

(1R,2R,4S)-4-tert-Butyldimethylsiloxy-1,2-epoxy-2,6,6-trimethylcyclohexane -1-methanol (7). To a mixture of (–)-diethyl-D-tartrate (1.80 mL, 10.5 mmol, dehydrated by azeotropic distillation) and powdered molecular sieves 4 Å (5.0 g) in dichloromethane (120 mL) was added tetraisopropyl orthotitanate (2.10 mL, 7.03 mmol, freshly distilled under reduced pressure) at –20 °C. After the mixture was stirred for 10 min at the same temperature, tert-butylhydroperoxide (1.5 M in toluene, 46.5 mL, 69.8 mmol, dehydrated by azeotropic distillation) was added dropwise, and the reaction mixture was stirred for 10 min. To this mixture was added a solution of **7** (10.0 g, 35.2 mmol) in dichloromethane (50 mL) at the same temperature, and the mixture was stirred for 30 min at –20 °C. The resulting mixture was poured into an aqueous solution of FeSO₄-citric acid at 0 °C. After being stirred for a further 20 min at room temperature, the mixture was extracted with chloroform. The organic layers were combined, dried over MgSO₄, filtered and concentrated *in vacuo*. Purification by silica gel column chromatography (from 10% to 30% ethyl acetate in hexane) afforded a mixture of a-epoxy alcohol **7** and its diastereomer **8** (10.4 g, 99%) as a colorless oil, whose ratio was determined to be 96 to 4 based on ¹H NMR analysis. The obtained diastereomeric mixture was used in the next reaction without further purification: IR (NaCl, cm^{–1}) 3449, 2957, 2859, 1639, 1469, 1387, 1253, 1088; ¹H NMR (400 MHz, CDCl₃) δ 3.83 (d, 1H, J = 12.0 Hz), 3.77 (m, 1H), 3.71 (d, 1H, J = 12.0 Hz), 2.22 (ddd, 1H, J = 14.4, 4.9, 2.0 Hz), 1.63 (dd, 1H, J = 14.2, 8.5 Hz), 1.44 (ddd, 1H, J = 12.9, 3.2, 2.0 Hz), 1.40 (s, 3H), 1.23 (dd, 1H, J = 10.5, 10.3 Hz), 1.14 (s, 3H), 1.06 (s, 3H), 0.87 (s, 9H), 0.05 (s, 3H), 0.04 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 68.2, 65.8, 64.3, 59.9, 48.0, 42.3, 34.1, 27.0, 25.8, 24.8, 19.7, 18.1, –4.65, –4.70; FAB⁺ HRMS Found m/z 301.2191, Calcd. for C₁₆H₃₂O₃Si [M+H]⁺ 300.2199.

(1S,2R,4S)-4-tert-Butyldimethylsiloxy-1,2-epoxy-2,6,6-trimethylcyclohexane -1-carbaldehyde (4). To a solution of oxalyl chloride (3.60 mL, 41.6 mmol) in dichloromethane (70 mL) was added

dropwise DMSO (3.50 mL, 49.9 mmol) at -78°C , and the mixture was stirred for 10 min at the same temperature. To this mixture was added a solution of **7** (5.00 g, 16.6 mmol) in dichloromethane (10 mL), and the resulting mixture was stirred for 40 min at -78°C , and then triethylamine (11.5 mL, 83.2 mmol) was added. After the mixture was stirred for 20 min at room temperature, water was added, and the mixture was extracted with diethyl ether. The organic layers were combined, dried over MgSO_4 , filtered and concentrated *in vacuo*. Purification by short silica gel column chromatography (5% ethyl acetate in hexane containing 3% triethylamine) afforded aldehyde **4** (4.92 g, 100%) as a colorless oil. The analytical sample was further purified by silica gel column chromatography (3% triethylamine in hexane) to separate the (5*S*,6*R*)-stereoisomer (carotenoid numbering): $[\alpha]_{\text{D}}^{22} -76.84$ (c 0.84, CHCl_3); IR (NaCl, cm^{-1}) 2957, 2932, 2858, 1728, 1470, 1385, 1254, 1088; ^1H NMR (400 MHz, CDCl_3) δ 9.80 (s, 1H), 3.85 (m, 1H), 2.24 (ddd, 1H, $J = 14.6, 5.4, 1.2$ Hz), 1.71 (dd, 1H, $J = 14.9, 7.6$ Hz), 1.49 (ddd, 1H, $J = 13.2, 3.4, 1.2$ Hz), 1.38 (s, 3H), 1.28 (dd, 1H, $J = 13.2, 9.0$ Hz), 1.28 (s, 3H), 1.07 (s, 3H), 0.88 (s, 9H), 0.05 (s, 3H), 0.04 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 200.4, 72.3, 66.1, 64.2, 46.3, 40.8, 33.6, 28.0, 26.2, 25.8, 20.6, 18.0, -4.79, -4.84; EI^+ HRMS Found m/z 298.1973, Calcd. for $\text{C}_{16}\text{H}_{30}\text{O}_3\text{Si}$ M^+ 298.1963.

(1*S*,3*R*,4*R*)-3,4-Epoxy-4-ethynyl-3,5,5-trimethylcyclohexanol (11). To a suspension of chloromethyltriphenylphosphonium chloride (6.05 g, 17.4 mmol) in THF (45 mL) was added dropwise $n\text{BuLi}$ (1.6 M in hexane, 10.0 mL, 16.1 mmol) at -78°C . After the mixture was stirred for 30 min at -78°C , a solution of **4** (4.00 g, 13.4 mmol) in THF (20 mL) was added. After being stirred for 3 h at -30°C , the reaction mixture was filtered through a pad of Celite. Water was added to the filtrate, and the resulting mixture was extracted with diethyl ether. The organic layers were combined, washed with brine, dried over MgSO_4 , filtered and concentrated *in vacuo* to afford the crude vinyl chloride **9** as a mixture of E and Z isomers, which was used in the next reaction without further purification.

To a solution of the crude vinyl chloride **9** thus obtained in DMSO (40 mL) was added potassium *tert*-butoxide (3.31 g, 29.5 mmol) at room temperature. After being stirred for 30 min at the same temperature, the reaction mixture was poured into a saturated aqueous NH_4Cl solution, and then extracted with diethyl ether. The organic layers were combined, washed with brine, dried over MgSO_4 , filtered and concentrated *in vacuo*. Purification by silica gel column chromatography (from 20% to 50% ethyl acetate in hexane) and crystallization from diethyl ether-hexane afforded acetylene **11**¹⁴ (1.27 g, 53% for 2 steps) as a white solid.

(2*E*)-3-Iodobut-2-en-1-ol (13). To a solution of iodine (7.0 g, 27.5 mmol), Na_2CO_3 (2.9 g, 27.5 mmol) in dichloromethane (80 mL) was added dropwise a solution of **12** (4.96 g, 13.7 mmol) in dichloromethane (20 mL) at 0°C . After stirred for 1 h at 0°C , the mixture was poured into a saturated aqueous $\text{Na}_2\text{S}_2\text{O}_3$ solution, and then extracted with diethyl ether. The organic layers were combined, washed with brine, dried over MgSO_4 , filtered and concentrated *in vacuo*. Purification by silica gel column chromatography (from 5% to 20% ethyl acetate in hexane) afforded iodide **13** (2.44 g, 90%): IR (NaCl, cm^{-1}) 3321, 2919, 2874, 1638, 1426, 1377, 1221, 1100, 1061, 1005; ^1H NMR (400 MHz, CDCl_3) δ 6.39 (tq, 1H, $J = 7.1, 1.5$ Hz), 4.07 (d, 2H, $J = 6.8$ Hz), 2.46 (t, 3H, $J = 0.70$ Hz); ^{13}C NMR (100 MHz, CDCl_3) δ 139.6, 98.3, 59.7, 28.0; EI^+ HRMS Found m/z 197.9505, Calcd. for $\text{C}_4\text{H}_7\text{OI}$ M^+ 197.9501.

Methyl (2*E*,4*E*)-5-iodohexa-2,4-dienate (14). A mixture of **13** (5.00 g, 25.3 mmol) and manganese dioxide (25.0 g) in diethyl ether (150 mL) was stirred for 2 h at room temperature. The precipitate was filtered through a pad of Celite, and the filtrate was concentrated *in vacuo* to afford the crude aldehyde, which was used in the next reaction without further purification.

To a solution of trimethyl phosphonoacetate (5.98 g, 32.8 mmol) in THF (120 mL) was added

sodium hydride (730 mg, 30.4 mmol) at 0 °C, and the mixture was stirred for 10 min. To this mixture was added a solution of the crude aldehyde in THF (30 mL) at 0 °C. After being stirred for 5 min at room temperature, the reaction mixture was poured into water, and then extracted with ethyl acetate. The organic layers were combined, dried over MgSO₄, filtered and concentrated *in vacuo*. Purification by silica gel column chromatography (5% ethyl acetate in hexane) afforded methyl ester **14** (4.70 g, 74% for 2 steps): IR (NaCl, cm⁻¹) 2992, 2949, 2917, 2841, 1717, 1622, 1433, 1379, 1345, 1310, 1267, 1234; ¹H NMR (400 MHz, CDCl₃) δ 7.38 (dd, 1H, J = 15.1, 11.5 Hz), 6.93 (dd, 1H, J = 11.5, 0.73 Hz), 5.85 (d, 1H, J = 15.4 Hz), 3.76 (s, 3H), 2.66 (d, 3H, J = 1.2 Hz); ¹³C NMR (100 MHz, CDCl₃) δ 167.2, 138.5, 138.0, 120.8, 108.1, 51.7, 28.9; EI⁺ HRMS Found m/z 251.9630, Calcd. for C₇H₉O₂I M⁺ 251.9647.

(2E,4E)-7-[(1'R,2'R,4'S)-2',4'-Dihydroxy-2',6',6'-trimethylcyclohexylidene]-5-methylhepta-2,4,6-trien-1-ol (16). To a solution of **15** (2.80 g, 9.44 mmol) in dichloromethane (95 mL) was added dropwise diisobutylaluminum hydride (1.0 M in dichloromethane, 94.5 mL, 94.5 mmol) at 0 °C. After the reaction mixture was stirred for 10 min at the same temperature, water and 2 N aqueous HCl solution were added, and then the resulting mixture was extracted with ethyl acetate. The organic layers were combined, washed with brine, dried over MgSO₄, filtered and concentrated *in vacuo*. Purification by silica gel column chromatography (from 50% to 70% ethyl acetate in hexane) afforded allenic triol **16** (2.10 g, 80%) as a pale yellow solid: [α]_D²² -37.19 (c 0.46, MeOH); IR (KBr, cm⁻¹) 3356, 2965, 2928, 2870, 2209, 1935, 1725, 1663, 1603, 1456, 1375, 1258, 1157, 1071; ¹H NMR (400 MHz, CDCl₃) δ 6.56 (dd, 1H, J = 15.1, 11.2 Hz), 6.02 (d, 1H, J = 11.2 Hz), 5.98 (s, 1H), 5.85 (dt, 1H, J = 15.1, 5.9 Hz), 4.32 (m, 1H), 4.23 (d, 2H, J = 5.9 Hz), 2.26 (ddd, 1H, J = 12.9, 3.9, 2.2 Hz), 1.95 (ddd, 1H, J = 12.2, 3.6, 2.2 Hz), 1.76 (s, 3H), 1.50-1.20 (m, 2H), 1.34 (s, 3H), 1.33 (s, 3H), 1.06 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 202.0, 132.5, 131.7, 127.8, 126.5, 117.7, 102.6, 72.9, 64.3, 63.6, 49.4, 48.9, 35.7, 32.1, 31.3, 29.3, 13.8; EI⁺ HRMS Found m/z 278.1817, Calcd. for C₁₇H₂₆O₃ M⁺ 278.1882.

(2E,4E)-5-[(1'S,2'R,4'S)-4'-tert-Butyldimethylsiloxy-1',2'-epoxy-2',6',6'-trimethylcyclohex-1'-yl]-3-methoxycarbonylpenta-2,4-dienal (20). To a solution of α -hydroxybutenolide **19** (1.69 g, 4.28 mmol) in DMSO (35 mL) was added diisopropylethylamine (2.24 mL, 12.9 mmol) at room temperature. After the reaction mixture was stirred for 3 h at the same temperature, ethyl iodide (2.06 mL, 25.7 mmol) was added. After being stirred for an additional 1 h, the reaction mixture was poured into water, and then extracted with ethyl acetate. The organic layers were combined, washed with brine, dried over MgSO₄, filtered and concentrated *in vacuo*. Purification by silica gel column chromatography (from 3% to 10% ethyl acetate in hexane) afforded (Z)- α -alkoxycarbonyldienal **20** (1.80 g, 100%) as a pale yellow oil: [α]_D²² -76.55 (c 1.37, CHCl₃); IR (NaCl, cm⁻¹) 2959, 2932, 2859, 1732, 1682, 1626, 1589, 1471, 1375, 1346, 1256, 1208, 1179, 1096, 1017; ¹H NMR (400 MHz, CDCl₃) δ 9.83 (d, 1H, J = 7.6 Hz), 6.50 (d, 1H, J = 15.6 Hz), 6.35 (d, 1H, J = 15.6 Hz), 6.08 (d, 1H, J = 7.6 Hz), 4.40 (m, 2H), 3.85 (m, 1H), 2.25 (ddd, 1H, J = 14.6, 5.1, 1.5 Hz), 1.66 (dd, 1H, J = 14.6, 8.1 Hz), 1.51 (ddd, 1H, J = 13.2, 3.4, 1.7 Hz), 1.38 (t, 3H, J = 7.1 Hz), 1.25 (dd, 1H, J = 13.2, 9.8 Hz), 1.18 (s, 3H), 1.13 (s, 3H), 0.96 (s, 3H), 0.87 (s, 9H), 0.042 (s, 3H), 0.037 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 190.9, 165.8, 148.3, 138.4, 129.7, 128.9, 70.2, 67.6, 64.5, 62.1, 46.7, 41.2, 35.1, 29.1, 25.8, 25.1, 20.0, 18.1, 14.2, -4.8; EI⁺ HRMS Found m/z 422.2497, Calcd. for C₂₃H₃₈O₅Si M⁺ 422.2487.

(3Z,5E)-1,1-Dibromo-6-[(1'S,2'R,4'S)-4'-tert-butyldimethylsiloxy-1',2'-epoxy-2',6',6'-trimethylcyclohex-1'-yl]-4-ethoxycarbonylhexa-1,3,5-triene (21). To a solution of carbon tetrachloride (2.84 g, 8.57 mmol) in dichloromethane (12 mL) was added dropwise a solution of triphenylphosphine

(4.49 g, 17.1 mmol) in dichloromethane (24 mL) at $-20\text{ }^{\circ}\text{C}$. After the reaction mixture was stirred for 20 min, a solution of **20** (1.80 g, 4.28 mmol) and triethylamine (1.22 mL, 8.79 mmol) in dichloromethane (12 mL) was added at $-60\text{ }^{\circ}\text{C}$, and then the mixture was stirred for 1 h at the same temperature. After hexane was added, the precipitate was removed by filtration through a pad of Celite, and then the solvents were removed *in vacuo*. Purification by silica gel column chromatography (from 2% to 5% ethyl acetate in hexane) afforded the corresponding dibromide **21** (2.30 g, 93%) as a yellow oil: $[\alpha]_{\text{D}}^{22} -36.08$ (c 0.74, CHCl_3); IR (NaCl, cm^{-1}) 2957, 2930, 2857, 1721, 1630, 1586, 1543, 1472, 1379, 1252, 1221, 1179, 1154, 1084, 1026; ^1H NMR (400 MHz, CDCl_3) δ 7.53 (d, 1H, $J = 11.2$ Hz), 6.40 (d, 1H, $J = 11.2$ Hz), 6.29 (d, 1H, $J = 15.9$ Hz), 6.23 (d, 1H, $J = 15.9$ Hz), 4.32 (m, 2H), 3.84 (m, 1H), 2.24 (ddd, 1H, $J = 14.4, 5.1, 1.5$ Hz), 1.64 (dd, 1H, $J = 14.4, 8.3$ Hz), 1.50 (ddd, 1H, $J = 12.9, 3.4, 1.7$ Hz), 1.35 (t, 3H, $J = 7.1$ Hz), 1.24 (dd, 1H, $J = 12.9, 10.0$ Hz), 1.18 (s, 3H), 1.12 (s, 3H), 0.96 (s, 3H), 0.87 (s, 9H), 0.05 (s, 3H), 0.04 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.5, 133.4, 131.9, 131.3, 131.0, 130.1, 97.9, 70.3, 67.2, 64.6, 61.2, 47.0, 41.3, 35.1, 29.3, 25.9, 25.0, 20.0, 18.1, 14.3, -4.7, -4.8; EI⁺ HRMS Found m/z 576.0895, Calcd. for $\text{C}_{24}\text{H}_{38}\text{O}_4\text{Br}_2\text{Si M}^+$ 576.0904.

(3Z,5E)-6-[(1'S,2'R,4'S)-4'-tert-Butyldimethylsiloxy-1',2'-epoxy-2',6',6'-trimethylcyclohex-1'-yl]-4-ethoxycarbonylhexa-3,5-dien-1-yne (22). To a solution of dibromide **21** (2.0 g, 3.46 mmol) in THF (35 mL) was dropwise sodium hexamethyldisilazide (1.0 M in THF, 5.2 mL, 5.20 mmol) at $-100\text{ }^{\circ}\text{C}$. After the reaction mixture was stirred for 10 min at $-100\text{ }^{\circ}\text{C}$, methylmagnesium bromide (1.0 M in THF, 10.4 mL, 10.4 mmol) was added dropwise at the same temperature. After the mixture was stirred for 10 min at $-100\text{ }^{\circ}\text{C}$, methanol (14 mL) and water (14 mL) were added at $-100\text{ }^{\circ}\text{C}$. After being stirred for 10 min at room temperature, and the resulting mixture was extracted with diethyl ether. The organic layers were combined, washed with brine, dried over MgSO_4 , filtered and concentrated *in vacuo* to afford the corresponding crude acetylene **22**, which was used in the next reaction without further purification.

(2E,6Z,8E)-9-[(1'S,2'R,4'S)-4'-tert-Butyldimethylsiloxy-1',2'-epoxy-2',6',6'-trimethylcyclohex-1'-yl]-7-ethoxycarbonyl-3-methylnona-2,6,8-trien-4-yn-1-ol (23). To a solution of **13** (820 mg, 4.15 mmol), tetrakis(triphenylphosphine)palladium (360 mg, 0.31 mmol) and triethylamine (1.92 mL, 13.8 mmol) in THF (20 mL) was added a solution of the crude acetylene **22** in THF (10 mL) and CuI (118 mg, 0.62 mmol). After being stirred for 18 h at room temperature, the reaction mixture was poured into a saturated aqueous NH_4Cl solution, and then the resulting mixture was extracted with diethyl ether. The organic layers were combined, dried over MgSO_4 , filtered and concentrated *in vacuo*. Purification by silica gel column chromatography (from 2% to 30% ethyl acetate in hexane) afforded the coupling product **23** (654 mg, 40% for 2 steps) as a yellow oil: $[\alpha]_{\text{D}}^{22} -83.00$ (c 0.77, CHCl_3); IR (NaCl, cm^{-1}) 3651, 3449, 2959, 2930, 2859, 2182, 1732, 1628, 1472, 1379, 1252, 1179, 1154, 1084; ^1H NMR (400 MHz, CDCl_3) δ 6.23 (s, 2H), 6.01 (brtq, 1H), 5.95 (s, 1H), 4.32 (m, 2H), 4.24 (d, 2H, $J = 6.6$ Hz), 3.82 (m, 1H), 2.22 (ddd, 1H, $J = 14.6, 5.1, 1.2$ Hz), 1.83 (s, 3H), 1.62 (dd, 1H, $J = 14.6, 8.5$ Hz), 1.48 (ddd, 1H, $J = 13.2, 3.2, 1.5$ Hz), 1.34 (t, 3H, $J = 7.1$ Hz), 1.25 (dd, 1H, $J = 13.2, 10.0$ Hz), 1.15 (s, 3H), 1.10 (s, 3H), 0.93 (s, 3H), 0.86 (s, 9H), 0.030 (s, 3H), 0.026 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.2, 140.8, 136.9, 131.0, 129.1, 120.6, 114.1, 101.4, 84.9, 70.2, 67.3, 64.6, 61.0, 59.2, 47.0, 41.3, 35.1, 29.3, 25.8, 24.9, 19.9, 18.1, 17.3, 14.3, -4.77, -4.80; EI⁺ HRMS Found m/z 488.2959, Calcd. for $\text{C}_{28}\text{H}_{44}\text{O}_5\text{Si M}^+$ 488.2956.

(2E,6Z,8E)-9-[(1'S,2'R,4'S)-4'-tert-Butyldimethylsiloxy-1',2'-epoxy-2',6',6'-trimethylcyclohex-1'-yl]-1-hydroxy-3-methylnona-2,6,8-trien-4-yne-7-carboxylic acid (24). To a mixture of 2 N aqueous KOH solution and ethanol (1 : 4 mixture, 8.0 mL) was added a solution of **23** (203 mg, 0.42 mmol) in

ethanol (2 mL) at 70 °C. After starting material was disappeared by TLC analysis (ca. 40 min later), the reaction mixture was cooled to 0 °C, and diluted with diethyl ether. The mixture was acidified by addition of a 3 N aqueous HCl solution under vigorous stirring, and the resulting mixture was extracted with diethyl ether. The organic layers were combined, washed with brine, dried over MgSO_4 , filtered and concentrated *in vacuo*. Purification by silica gel column chromatography (from 20% to 50% ethyl acetate in hexane) afforded carboxylic acid **24** (97 mg, 51%): $[\alpha]_D^{22}$ -52.10 (c 0.14, CHCl_3); IR (KBr, cm^{-1}) 3426, 2957, 2930, 2859, 2182, 1763, 1709, 1626, 1472, 1383, 1364, 1256, 1182, 1163, 1082, 1032, 1007; ^1H NMR (400 MHz, CDCl_3) δ 6.41 (d, 1H, $J = 15.6$ Hz), 6.29 (d, 1H, $J = 15.6$ Hz), 6.13 (s, 1H), 6.07 (brtq, 1H), 4.26 (d, 2H, $J = 6.6$ Hz), 3.84 (m, 1H), 2.25 (ddd, 1H, $J = 14.6, 4.6, 1.2$ Hz), 1.86 (s, 3H), 1.65 (dd, 1H, $J = 14.6, 8.3$ Hz), 1.50 (ddd, 1H, $J = 13.2, 3.2, 1.5$ Hz), 1.25 (dd, 1H, $J = 13.4, 9.5$ Hz), 1.18 (s, 3H), 1.13 (s, 3H), 0.96 (s, 3H), 0.88 (s, 9H), 0.05 (s, 3H), 0.04 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.6, 139.0, 137.5, 131.3, 128.7, 120.7, 116.4, 103.5, 85.2, 70.3, 67.4, 64.6, 59.2, 47.0, 41.3, 35.2, 29.3, 25.9, 25.0, 20.0, 18.1, 17.2, -4.7, -4.8; EI⁺ HRMS Found m/z 460.2653, Calcd. for $\text{C}_{26}\text{H}_{40}\text{O}_5\text{Si}$ M⁺ 460.2643.

(5Z)-[(2'E)-4'-Hydroxy-2'-methyl-1'-butenyldiene]-3-[(1'E)-2''-(1'''S,2'''R,4'''S)-4'''-tert-butyltrimethylsiloxy-1'''',2'''-epoxy-2'''',6'''',6'''-trimethylcyclohex-1'''-yl]-2(5H)-furanone (25). To a mixture of bis(triphenylphosphine)palladium dichloride (2 mg, 0.003 mmol), CuI (1 mg, 0.005 mmol), diphenylphosphonoethane (dppe, 1 mg, 0.003 mmol) and triethylamine (0.02 mL, 0.15 mmol) in THF (2 mL) was added a solution of **24** (47 mg, 0.102 mmol) in THF (1 mL) at room temperature. After being stirred for 30 sec at the same temperature, the reaction mixture was filtered through a pad of Celite, and then the filtrate was concentrated *in vacuo*. Purification by silica gel column chromatography (from 10% to 20% ethyl acetate in hexane) afforded butenolide **25** (31 mg, 66%) along with trace amounts of dimer **26**.

Data for **25**: $[\alpha]_D^{22}$ -61.27 (c 0.049, CHCl_3); IR (KBr, cm^{-1}) 3632, 3422, 3081, 2957, 2930, 2857, 1753, 1640, 1618, 1462, 1383, 1364, 1283, 1256, 1186, 1152, 1076; ^1H NMR (400 MHz, CDCl_3) δ 7.20 (d, 1H, $J = 15.6$ Hz), 7.03 (s, 1H), 6.35 (d, 1H, $J = 15.6$ Hz), 5.97 (brt, 1H), 5.63 (s, 1H), 4.35 (d, 2H, $J = 6.6$ Hz), 3.85 (m, 1H), 2.25 (ddd, 1H, $J = 14.6, 4.9, 1.2$ Hz), 2.10 (s, 3H), 1.67 (dd, 1H, $J = 14.4, 8.3$ Hz), 1.51 (ddd, 1H, $J = 13.2, 3.2, 1.2$ Hz), 1.28 (dd, 1H, $J = 13.4, 10.2$ Hz), 1.18 (s, 3H), 1.17 (s, 3H), 0.94 (s, 3H), 0.88 (s, 9H), 0.05 (s, 3H), 0.04 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 168.7, 146.7, 136.8, 134.6, 134.0, 126.0, 121.3, 117.5, 70.6, 67.5, 64.6, 59.5, 47.0, 41.3, 35.1, 29.4, 25.9, 25.0, 20.0, 18.1, 15.5, -4.7, -4.8; EI⁺ HRMS Found m/z 460.2656, Calcd. for $\text{C}_{26}\text{H}_{40}\text{O}_5\text{Si}$ M⁺ 460.2643.

Data for **26**: ^1H NMR (400 MHz, CDCl_3) δ 7.26 (d, 1H, $J = 15.4$ Hz), 6.90 (s, 1H), 6.32 (d, 1H, $J = 15.4$ Hz), 5.84 (brt, 1H), 4.32 (d, 2H, $J = 6.6$ Hz), 3.82 (m, 1H), 2.25 (ddd, 1H, $J = 14.6, 4.9, 1.2$ Hz), 2.05 (s, 3H), 1.67 (dd, 1H, $J = 14.4, 8.3$ Hz), 1.51 (ddd, 1H, $J = 13.2, 3.2, 1.2$ Hz), 1.28 (m, 1H), 1.18 (s, 3H), 1.17 (s, 3H), 0.94 (s, 3H), 0.88 (s, 9H), 0.05 (s, 3H), 0.04 (s, 3H); EI⁺ HRMS Found m/z 918.5175, Calcd. for $\text{C}_{26}\text{H}_{40}\text{O}_5\text{Si}$ M⁺ 918.5134.

III. Model Study for First-Generation Synthesis of the Ylidenebutenolide Segment

Epoxide (ii). To a solution of **i** (2.83 g, 10.90 mmol) in dichloromethane (50 mL) was added a solution of *m*CPBA (2.80 g, 16.20 mmol) in dichloromethane (50 mL) at 0 °C. After being stirred for 5 h at 0 °C, the reaction mixture was poured into water, and then extracted with diethyl ether. The organic layers were combined, washed with a saturated aqueous NaHCO_3 solution and brine, dried over MgSO_4 , filtered and concentrated *in vacuo* to afford the crude epoxide **ii** (2.10 g, 70%) as a yellow oil: IR (neat, cm^{-1}) 2957, 2872, 1734, 1680, 1626, 1591, 1439, 1364, 1251, 1163, 1128, 974; ^1H NMR (400 MHz, CDCl_3) δ 9.81 (d, 1H, $J = 7.3$ Hz), 6.49 (d, 1H, $J = 15.6$ Hz), 6.38 (d, 1H, $J = 15.6$ Hz), 6.12 (d, 1H, $J = 7.3$ Hz), 3.94 (s, 3H), 1.95-1.88 (m, 1H), 1.79-1.73 (m, 1H), 1.46-1.39 (m, 4H), 1.16 (s, 3H), 1.11 (s, 3H), 0.94 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 190.9, 166.3, 147.9, 138.4, 129.7, 128.8, 71.1, 66.2, 52.6, 35.6, 33.7, 29.9, 25.9, 25.8, 20.8, 16.9; EI^+ HRMS Found m/z 278.1519, Calcd. for $\text{C}_{16}\text{H}_{22}\text{O}_4$ M^+ 278.1518.

Dibromide (iii): IR (KBr, cm^{-1}) 2965, 2940, 2899, 1713, 1439, 1381, 1238, 1196, 1167, 984, 897, 874, 828, 793; ^1H NMR (400 MHz, CDCl_3) δ 7.53 (d, 1H, $J = 11.2$ Hz), 6.44 (d, 1H, $J = 11.2$ Hz), 6.27 (s, 2H), 3.86 (s, 3H), 1.94-1.86 (m, 1H), 1.77-1.72 (m, 1H), 1.48-1.40 (m, 4H), 1.17 (s, 3H), 1.09 (s, 3H), 0.94 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 167.0, 133.4, 133.0, 131.3, 131.2, 131.1, 130.0, 98.1, 71.2, 65.8, 52.0, 35.7, 33.7, 30.0, 26.0, 25.9, 20.9, 17.0; EI^+ HRMS Found m/z 431.9933, Calcd. for $\text{C}_{17}\text{H}_{22}\text{O}_3\text{Br}_2$ M^+ 431.9936.

Acetylene (iv): IR (NaCl, cm^{-1}) 3273, 2948, 2098, 1735, 1442, 1380, 1233; ^1H NMR (400 MHz, CDCl_3) δ 6.24 (s, 2H), 5.83 (d, 1H, $J = 2.7$ Hz), 3.87 (s, 3H), 3.41 (d, 1H, $J = 2.7$ Hz), 1.91-1.87 (m, 1H), 1.78-1.71 (m, 1H), 1.45-1.39 (m, 4H), 1.15 (s, 3H), 1.08 (s, 3H), 0.93 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 143.3, 132.1, 128.6, 112.6, 86.6, 80.2, 71.0, 65.9, 52.0, 35.7, 30.0, 25.8, 25.8, 20.6, 17.0; EI^+ HRMS Found m/z 274.1565, Calcd. for $\text{C}_{17}\text{H}_{22}\text{O}_3$ M^+ 274.1569.

Coupling product (v): IR (neat, cm^{-1}) 3444, 2932, 2170, 1728, 1438, 1380, 1224, 1158, 976; ^1H NMR (400 MHz, CDCl_3) δ 6.25 (d, $J = 15.9$ Hz, 2H), 6.04 (t, $J = 6.8$ Hz, 1H), 5.99 (s, 1H), 4.26 (d, $J = 6.4$ Hz, 2H), 3.86 (s, 3H), 1.94-1.86 (m, 1H), 1.86 (s, 3H), 1.76-1.70 (m, 1H), 1.48-1.40 (m, 4H), 1.15 (s,

3H), 1.08 (s, 3H), 0.93 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.7, 140.6, 136.9, 131.08, 129.0, 120.7, 114.2, 101.5, 85.1, 77.2, 71.1, 65.9, 59.2, 51.8, 35.7, 33.7, 30.0, 25.8, 20.9, 17.4, 17.0; EI^+ HRMS Found m/z 344.1985, Calcd. for $\text{C}_{21}\text{H}_{28}\text{O}_4 \text{M}^+$ 344.1988.

Carboxylic Acid (vi). To a mixture of a 1 N aqueous $\text{Ba}(\text{OH})_2$ solution (4.0 mL) and methanol (5.0 mL) was added a solution of the coupling product **v** (100 mg, 0.29 mmol) in methanol (1.0 mL) at room temperature. After being stirred for 4 h at room temperature, the reaction mixture was acidified by addition of a 3 N aqueous HCl solution under vigorous stirring, and then extracted with diethyl ether. The organic layers were combined, washed with brine, dried over MgSO_4 , filtered and concentrated *in vacuo*. Purification by silica gel column chromatography (from 20% to 50% ethyl acetate in hexane) afforded carboxylic acid **vi** (70 mg, 73%): IR (neat, cm^{-1}) 3408, 2932, 2200, 1718, 1458, 1382, 1230, 1174, 978; ^1H NMR (400 MHz, CDCl_3) δ 6.39 (d, 1H, $J = 15.6$ Hz), 6.30 (d, 1H, $J = 15.6$ Hz), 6.12 (s, 1H), 6.06 (t, 1H, $J = 6.8$ Hz), 4.26 (d, 2H, $J = 6.6$ Hz), 1.94-1.88 (m, 1H), 1.86 (s, 3H), 1.78-1.72 (m, 1H), 1.48-1.40 (m, 4H), 1.17 (s, 3H), 1.10 (s, 3H), 0.94 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 170.0, 139.3, 137.3, 131.1, 128.7, 120.7, 116.0, 103.2, 85.3, 71.3, 66.0, 59.1, 35.7, 33.7, 30.0, 25.9, 25.8, 20.9, 17.2, 17.0; EI^+ HRMS Found m/z 300.1828, Calcd. for $\text{C}_{20}\text{H}_{26}\text{O}_4 \text{M}^+$ 300.1830.

Ylidenebutenolide (vii = 30). To a mixture of bis(triphenylphosphine)palladium dichloride (15 mg, 0.02 mmol), CuI (4 mg, 0.02 mmol), and triethylamine (0.06 mL, 0.42 mmol) in THF (2 mL) was added a solution of **vi** (70 mg, 0.21 mmol) in THF (0.5 mL) at room temperature. After being stirred for 1 h at the same temperature, the reaction mixture was filtered through a pad of Celite, and then the filtrate was concentrated *in vacuo*. Purification by silica gel column chromatography (from 10% to 20% ethyl acetate in hexane) afforded butenolide **vii** (52 mg, 74%): IR (NaCl, cm^{-1}) 3428, 2948, 1774, 1742, 1620, 1462, 1234, 1050; ^1H NMR (400 MHz, CDCl_3) δ 7.18 (d, 1H, $J = 15.6$ Hz), 7.03 (s, 1H), 6.37 (d, 1H, $J = 15.6$ Hz), 5.95 (t, 1H, $J = 6.6$ Hz), 5.64 (s, 1H), 4.35 (d, 1H, $J = 6.4$ Hz), 2.10 (s, 3H), 1.91 (m, 1H), 1.75 (m, 1H), 1.42-1.46 (m, 4H), 1.16 (s, 6H), 0.93 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 168.7, 146.7, 136.6, 134.0, 126.1, 121.3, 117.4, 71.5, 66.0, 59.5, 35.6, 33.7, 30.0, 25.9, 25.8, 20.9, 17.0, 15.5; EI^+ HRMS Found m/z 300.1834, Calcd. for $\text{C}_{20}\text{H}_{26}\text{O}_4 \text{M}^+$ 300.1830.

IV. Model Study for Second-Generation Synthesis of the Ylidenebutenolide Segment

Allyl ester (ii): IR (NaCl, cm^{-1}) 2961, 2874, 1732, 1680, 1626, 1589, 1456, 1377, 1342, 1258, 1206, 1169, 1100, 1063, 1046; ^1H NMR (400 MHz, CDCl_3) δ 9.82 (d, 1H, $J = 7.6$ Hz), 6.49 (d, 1H, $J = 15.6$ Hz), 6.37 (d, 1H, $J = 15.6$ Hz), 6.10 (d, 1H, $J = 7.6$ Hz), 5.98 (ddt, 1H, $J = 17.0, 10.4, 5.9$ Hz), 5.42 (dd, 1H, $J = 17.0, 1.2$ Hz), 5.33 (dd, 1H, $J = 10.4, 1.2$ Hz), 4.82 (m, 2H), 1.90 (m, 1H), 1.73 (m, 1H), 1.00-1.60 (m, 4H), 1.16 (s, 3H), 1.09 (s, 3H), 0.93 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 190.9, 165.6, 148.1, 138.5, 131.0, 129.6, 128.8, 120.2, 71.1, 66.5, 66.3, 35.6, 33.7, 29.9, 25.9, 20.8, 16.9; EI^+ HRMS Found m/z 304.1675, Calcd. for $\text{C}_{19}\text{H}_{24}\text{O}_4 \text{M}^+$ 304.1668.

Dibromide (iii): IR (NaCl, cm^{-1}) 3084, 2959, 2872, 1718, 1649, 1628, 1586, 1543, 1453, 1383, 1364, 1254, 1221, 1157, 1117; ^1H NMR (400 MHz, CDCl_3) δ 7.55 (dd, 1H, $J = 11.2, 0.49$ Hz), 6.43 (dd, 1H, $J = 11.2, 0.49$ Hz), 6.28 (s, 2H), 5.99 (ddt, 1H, $J = 17.0, 10.4, 5.9$ Hz), 5.40 (dd, 1H, $J = 17.0, 1.2$ Hz), 5.32 (dd, 1H, $J = 10.4, 1.2$ Hz), 4.76 (m, 2H), 1.90 (m, 1H), 1.75 (m, 1H), 1.00-1.60 (m, 4H), 1.18 (s, 3H), 1.09 (s, 3H), 0.97 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.3, 133.4, 133.2, 131.5, 131.1, 130.0, 119.3, 98.0, 71.3, 65.8, 65.7, 35.7, 33.7, 30.2, 25.9, 25.8, 20.9, 17.0; EI^+ HRMS Found m/z 458.0092, Calcd. for $\text{C}_{19}\text{H}_{24}^{79}\text{Br}_3\text{O}_3 \text{M}^+$ 458.0085.

Acetylene (iv = 29a). To a solution of **iii** (1.70 g, 3.72 mmol) in THF (30 mL) was added tetra-*n*-butylammonium fluoride (4.00 g, 15.2 mmol) at room temperature. After being stirred for 20 h at room temperature, the reaction mixture was poured into a saturated aqueous NH_4Cl solution, and then extracted with diethyl ether. The organic layers were combined, washed with brine, dried over MgSO_4 , filtered and concentrated *in vacuo*. Purification by short silica gel column chromatography (from 10% to 30% ethyl acetate in hexane) afforded terminal acetylene **iv** (825 mg, 75%) as an orange oil, which was immediately used in next reaction: IR (NaCl, cm^{-1}) 3437, 3293, 3088, 2961, 1736, 1649, 1458, 1379, 1240, 1161, 1046; ^1H NMR (400 MHz, CDCl_3) δ 6.27 (d, 1H, $J = 15.9$ Hz), 6.23 (d, 1H, $J = 15.9$ Hz), 6.00 (ddt, 1H, $J = 17.1, 10.5, 5.9$ Hz), 5.82 (d, 1H, $J = 2.7$ Hz), 5.41 (dd, 1H, $J = 17.3, 1.5$ Hz), 5.28 (dd, 1H, $J = 10.5, 1.5$ Hz), 4.78 (m, 2H), 3.39 (d, 1H, $J = 2.7$ Hz), 1.90 (m, 1H), 1.77 (m, 1H), 1.00-1.50 (m, 4H), 1.18 (s, 3H), 1.10 (s, 3H), 0.96 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 165.8, 143.2, 132.1, 131.6, 128.7, 118.9, 112.6, 86.8, 80.2, 71.0, 65.9, 67.8, 35.7, 33.7, 30.0, 25.8, 20.8, 17.0; EI^+ HRMS Found m/z 300.1731, Calcd. for $\text{C}_{19}\text{H}_{24}\text{O}_3 \text{M}^+$ 300.1725.

Ylidenebutenolide (v = 30). To a solution of **iv** (70 mg, 0.23 mmol) and **13** (46 mg, 0.23 mmol) in

triethylamine (2.5 mL) was added formic acid (0.22 mL, 0.46 mmol), tetrakis(triphenylphosphine) palladium (27 mg, 0.02 mmol), and cuprous iodide (4.5 mg, 0.02 mmol). After being stirred for 20 h at room temperature, the reaction mixture was poured into a saturated aqueous NH_4Cl solution, and then extracted with diethyl ether. The organic layers were combined, washed with brine, dried over MgSO_4 , filtered and concentrated *in vacuo*. Purification by short silica gel column chromatography (from 10% to 20% ethyl acetate in hexane) afforded butenolide **v** (30 mg, 43%).

**V. ^1H NMR Analysis (400 MHz, CDCl_3) for the Thermodynamic Isomerization
of 15-*cis* Peridinin to *all-trans*-congener**

- a) Just after the purification by silica gel column chromatography
- b) After 1 days in CDCl_3 solution at 25 °C in the dark
- c) After 2 days in CDCl_3 solution at 25 °C in the dark
- d) After 3 days in CDCl_3 solution at 25 °C in the dark
- e) *all-trans*-peridinin (1) purified by HPLC

VI. CD Spectra of the Synthesized Peridinin

CD spectra in ether-isopentane-ethanol (5:5:2) solution of the synthesized peridinin

DFILE
SF FILE
COMNT
EXMOD
IRMOD
POINT
FREQU
SCANS
DUMMY
ACQTM
PD
RGAIN
PW1

DFILE
SF FILE
COMNT
EXMOD
IRMOD
POINT
FREQU
SCANS
DUMMY
ACQTM
PD
RGAIN
PW1

```
OBNUC : 1H
OBFRQ : 399.65 MHZ
OBSET : 134500.00 HZ
```

```

IRNUC      : 1H
IRFRQ      : 399.65 MHz
IRSET      : 134500.00 Hz
IRATN      : 511
IRBPW      : 50.0 usec
IRBP1      : 27
IRBP2      : 6
IRRNS      : 0

```

```
ADBIT : 16
CTEMP : 22.8 c
CSPED : 10 Hz
SLVNT : CDCL3
```

RESOL :	0.24	Hz
BF	0.01	Hz
T1	0.00	%
T2	0.00	%
T3	90.00	%
T4	100.00	%
REFVL	7.26	ppm
XE	2038.90	Hz
XS	1012.86	Hz
operator

7-SEP-1999 05:38:26.70

DFILE :
 SFIL :
 COMNT :
 EXMOD : SINGL
 IRMOD : BCM
 POINT : 32768
 FREQ : 27100.27 Hz
 SCANS : 163
 DUMMY : 4
 ACQTM : 1.2091 sec
 PD : 0.8000 sec
 RGAIN : 23
 PW1 : 4.75 usec

OBNUC : 13C
 OBFRQ : 100.40 MHz
 OBSET : 135500.00 Hz

IRNUC : 1H
 IRFRQ : 399.65 MHz
 IRSET : 134500.00 Hz
 IRATN : 511
 IRRPW : 50.0 usec
 IRBP1 : 27
 IRBP2 : 6
 IRRNS : 0

ADBIT : 16
 CTEMP : 23.6 c
 CSPED : 11 Hz
 SLVNT : CDCL3

RESOL : 0.83 Hz
 BF : 2.00 Hz
 T1 : 0.00 %
 T2 : 0.00 %
 T3 : 90.00 %
 T4 : 100.00 %
 REFVL : 77.00 ppm
 XE : 21109.23 Hz
 XS : 558.25 Hz
 operator

7-SEP-1999 13:13:32.81

DFILE :
SFILE :
COMNT :
EXMOD : SINGL
IRMOD : NON
POINT : 32768
FREQ : 7993.61 Hz
SCANS : 8
DUMMY : 4
ACQTM : 4.0993 sec
PD : 2.9007 sec
RGAIN : 15
PW1 : 5.35 usec

OBNUC : 1H
OBFRQ : 399.65 MHz
OBSET : 134500.00 Hz

IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRATN : 511
IRRPW : 50.0 usec
IRBP1 : 27
IRBP2 : 6
IRNS : 0

ADBIT : 16
CTEMP : 29.8 C
CSPED : 10 Hz
SLVNT : CDCL3

RESOL : 0.24 Hz
BF : 0.01 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
REFVL : 7.26 ppm
XE : 2038.90 Hz
XC : 1013.11 Hz
operator

```

DFILE : :
SFILE : :

COMMT : :
EXMOD : :
IRMOD : :
POINT : :
FREQU : :
SCANS : :
DUMMY : :
ACQTM : :
PD : :
RGAIN : :

PW1 : :

```

```

OBNUC : 13C
OBFRQ : 100.40 MHz
OBSET : 135500.00 HZ

IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 HZ
IRATN : 511
IRAPW : 50.0 usec
IRBP1 : 27
IRBP2 : 6
IRANS : 0

ADBIT : 16
CTEMP : 24.0 C
CSPED : 12 HZ
SLVNT : CDCL3

```

RESOL :	0.83 Hz
BF	2.00 Hz
T1	0.00 %
T2	0.00 %
T3	90.00 %
T4	100.00 %
REFVL	77.00 ppm
XE	21109.23 Hz
XS	559.08 Hz
operator

```

DFILE :
SFILE : HARA-EPOXYALCOHOL

```

COMNT : KATSUMURA-LAB.

EXMOD : SINGL

IRMOD : NON

POINT: 32768

FREQU : 7993.61 HZ

SCANS : 8

DUMMY : 4

ACQTM : 4.0993 sec

PD : 2.9007 sec

RGAIN :	12
---------	----

PW1 : 5.35 usec

OBNUC : 1H

OBFRQ : 399.65 MHZ

OBSET : 134500.00 HZ

IRNUC : 1H

IRFRQ : 399.65 MHZ

IRSET : 134500.00 HZ

IRATN : 511

```
IRPPW : 50.0 usec
```

IRBP1 : 27

IRBP2 : 6

TRANS : 0

ADBIT : 16

CTEMP : 23.1 C

CSPED : 10 HZ

SLVNT : CDCL3

RESOL : 0.24 Hz

HF	0.01 Hz	0.1 Hz	1 Hz
HF	0.01 Hz	0.1 Hz	1 Hz

	T1	T2	T3	T4	T5	T6	T7	T8	T9	T10	T11	T12	T13	T14	T15	T16	T17	T18	T19	T20	T21	T22	T23	T24	T25	T26	T27	T28	T29	T30	T31	T32	T33	T34	T35	T36	T37	T38	T39	T40	T41	T42	T43	T44	T45	T46	T47	T48	T49	T50	T51	T52	T53	T54	T55	T56	T57	T58	T59	T60	T61	T62	T63	T64	T65	T66	T67	T68	T69	T70	T71	T72	T73	T74	T75	T76	T77	T78	T79	T80	T81	T82	T83	T84	T85	T86	T87	T88	T89	T90	T91	T92	T93	T94	T95	T96	T97	T98	T99	T100	T101	T102	T103	T104	T105	T106	T107	T108	T109	T110	T111	T112	T113	T114	T115	T116	T117	T118	T119	T120	T121	T122	T123	T124	T125	T126	T127	T128	T129	T130	T131	T132	T133	T134	T135	T136	T137	T138	T139	T140	T141	T142	T143	T144	T145	T146	T147	T148	T149	T150	T151	T152	T153	T154	T155	T156	T157	T158	T159	T160	T161	T162	T163	T164	T165	T166	T167	T168	T169	T170	T171	T172	T173	T174	T175	T176	T177	T178	T179	T180	T181	T182	T183	T184	T185	T186	T187	T188	T189	T190	T191	T192	T193	T194	T195	T196	T197	T198	T199	T200	T201	T202	T203	T204	T205	T206	T207	T208	T209	T210	T211	T212	T213	T214	T215	T216	T217	T218	T219	T220	T221	T222	T223	T224	T225	T226	T227	T228	T229	T230	T231	T232	T233	T234	T235	T236	T237	T238	T239	T240	T241	T242	T243	T244	T245	T246	T247	T248	T249	T250	T251	T252	T253	T254	T255	T256	T257	T258	T259	T260	T261	T262	T263	T264	T265	T266	T267	T268	T269	T270	T271	T272	T273	T274	T275	T276	T277	T278	T279	T280	T281	T282	T283	T284	T285	T286	T287	T288	T289	T290	T291	T292	T293	T294	T295	T296	T297	T298	T299	T300	T301	T302	T303	T304	T305	T306	T307	T308	T309	T310	T311	T312	T313	T314	T315	T316	T317	T318	T319	T320	T321	T322	T323	T324	T325	T326	T327	T328	T329	T330	T331	T332	T333	T334	T335	T336	T337	T338	T339	T340	T341	T342	T343	T344	T345	T346	T347	T348	T349	T350	T351	T352	T353	T354	T355	T356	T357	T358	T359	T360	T361	T362	T363	T364	T365	T366	T367	T368	T369	T370	T371	T372	T373	T374	T375	T376	T377	T378	T379	T380	T381	T382	T383	T384	T385	T386	T387	T388	T389	T390	T391	T392	T393	T394	T395	T396	T397	T398	T399	T400	T401	T402	T403	T404	T405	T406	T407	T408	T409	T410	T411	T412	T413	T414	T415	T416	T417	T418	T419	T420	T421	T422	T423	T424	T425	T426	T427	T428	T429	T430	T431	T432	T433	T434	T435	T436	T437	T438	T439	T440	T441	T442	T443	T444	T445	T446	T447	T448	T449	T450	T451	T452	T453	T454	T455	T456	T457	T458	T459	T460	T461	T462	T463	T464	T465	T466	T467	T468	T469	T470	T471	T472	T473	T474	T475	T476	T477	T478	T479	T480	T481	T482	T483	T484	T485	T486	T487	T488	T489	T490	T491	T492	T493	T494	T495	T496	T497	T498	T499	T500	T501	T502	T503	T504	T505	T506	T507	T508	T509	T510	T511	T512	T513	T514	T515	T516	T517	T518	T519	T520	T521	T522	T523	T52
--	----	----	----	----	----	----	----	----	----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	-----

0.00%

T3	100.00 %
T4	0.00 %

T2	100.00 %
T4	100.00 %

REFVFL : 0.00 ppm

XE : 1718.84 HZ

X S : 1177.77 Hz

operator

anti-7

30-NOV-1998 15:06:23.13

DFILE :
 SFIL : HARA-EPOXYALCOHOL
 COMNT : KATSUMURA-LAB.
 EXMOD : SINGL
 IRMOD : BCM
 POINT : 32768
 FREQU : 27100.27 Hz
 SCANS : 512
 DUMMY : 4
 ACQTM : 1.2091 sec
 PD : 0.8000 sec
 RGAIN : 23
 PW1 : 4.75 usec

OBNUC : ¹³C
 OBFRQ : 100.40 MHz
 OBSET : 135500.00 Hz

IRNUC : ¹H
 IRFRQ : 399.65 MHz
 IRSET : 134500.00 Hz
 IRATN : 511
 IRRPW : 50.0 usec
 IRBP1 : 27
 IRBP2 : 6
 IRBNS : 0

ADBIT : 16
 CTEMP : 23.3 C
 CSPED : 11 Hz
 SLVNT : CDCL3

RESOL : 0.83 Hz
 BF : 1.00 Hz
 T1 : 0.00 %
 T2 : 0.00 %
 T3 : 100.00 %
 T4 : 100.00 %
 REFVL : 77.00 ppm
 XE : 10051.78 Hz
 XS : 6085.32 Hz
 operator

anti-7

```

DFILE :
SSFILE : HARA-EPOXYALDET
COMNT : KATSUMURA-LAB.
EXMOD : SINGL
IRMOD : NON
POINT : 32768
FREQU : 7993.61 Hz
SCANS : 8
DUMMY : 4
ACQTM : 4.0993 sec
PPD : 2.9007 sec
RGAIN : 14
PW1 : 5.35 usec

```

```

OBNUC : 1H
OBFRQ : 399.65 MHz
OBSET : 134500.00 Hz

IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRATN : 511
IRAPW : 50.0 usec
IRBP1 : 27
IRBP2 : 6
IRBNS : 0

AQB1T : 16
AQTEMP : 24.2 C
AQSPED : 11 Hz
AQSLVNT : CDCL3

RESOL : 0.24 Hz
BF : 0.01 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
REFVL : 0.00 ppm
XE : 4077.55 Hz
XS : -2.32 Hz
operator

```

21-AUG-1999 18:46:01.24

DFILE :
SFIL : HARA-EPOXYALDEHYDE-13C

COMNT : KATSUMURA-LAB.

EXMOD : SINGL

IRMOD : BCM

POINT : 32768

FREQU : 27100.27 Hz

SCANS : 512

DUMMY : 4

ACQTM : 1.2091 sec

PD : 0.8000 sec

RGAIN : 23

PW1 : 4.75 usec

OBNUC : 13C

OBFRQ : 100.40 MHz

OBSET : 135500.00 Hz

IRNUC : 1H

IRFRQ : 399.65 MHz

IRSET : 134500.00 Hz

IRATN : 511

IRRPW : 50.0 usec

IRBP1 : 27

IRBP2 : 6

IRRNS : 0

ADBIT : 16

CTEMP : 23.9 c

CSPED : 12 Hz

SLVNT : CDCL3

RESOL : 0.83 Hz

BF : 1.00 Hz

T1 : 0.00 %

T2 : 0.00 %

T3 : 90.00 %

T4 : 100.00 %

REFVL : 77.00 ppm

XE : 2214.91 Hz

XS : 53.76 Hz

operator

A2

29-OCT-2000 07:34:08.27

DFILE :
SFILE :
COMNT : A2-Solid
EXMOD : SINGL
IRMOD : NON
POINT : 32768
FREQU : 7993.61 Hz
SCANS : 8
DUMMY : 4
ACQTM : 4.0993 sec
PD : 2.9007 sec
RGAIN : 16
PW1 : 10.50 usec
OBNUC : 1H
OBFRQ : 399.65 MHz
OBSET : 134500.00 Hz
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRATN : 511
IRRPW : 50.0 usec
IRBP1 : 27
IRBP2 : 6
IRRNS : 0
ADBIT : 16
CTEMP : 24.3 C
CSPED : 11 Hz
SLVNT : CDCL3
RESOL : 0.24 Hz
BF : 0.01 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
REFVL : 0.00 ppm
XE : 1638.83 Hz
XS : 1215.82 Hz
operator

11

A2

29-OCT-2000 08:10:33.32

DFILE :
SFIL :COMNT : A2-Solid
EXMOD : SINGL
IRMOD : BCM
POINT : 32768
FREQU : 27100.27 Hz
SCANS : 1369
DUMMY : 4
ACQTM : 0.6046 sec
PD : 0.8000 sec
RGAIN : 23
PW1 : 9.50 usecOBNUC : 13C
OBFRQ : 100.40 MHz
OBSET : 135500.00 HzIRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRATN : 511
IRRPW : 50.0 usec
IRBP1 : 27
IRBP2 : 6
IRBNS : 0ADBIT : 16
CTEMP : 24.6 c
CSPED : 12 Hz
SLVNT : CDCL3RESOL : 0.83 Hz
BF : 2.00 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
REFVL : 77.00 ppm
XE : 11056.63 Hz
XS : 5585.38 Hz
operator21.401
25.391
29.694
34.005
39.739
45.720
63.771
66.527
74.129
76.687
77.000
77.321
80.521ppm
0
10
20
30
40
50
60
70
80
90

11

25-NOV-1999 14:01:14.77

DFILE :
 SFIL :
 COM1 :
 EXMOD : SINGL
 IRMOD : NON
 POINT : 32768
 FREQU : 7993.61 Hz
 SCANS : 8
 DUMMY : 4
 ACQTM : 4.0993 sec
 PD : 2.9007 sec
 RGAIN : 16
 PW1 : 5.35 usec

OBNUC : 1H
 OBFRQ : 399.65 MHz
 OBSET : 134500.00 Hz
 IRNUC : 1H
 IRFRQ : 399.65 MHz
 IRSET : 134500.00 Hz
 IRATN : 511
 IRRPW : 50.0 usec
 IRBP1 : 27
 IRBP2 : 6
 IRRNS : 0

ADBIT : 16
 CTEMP : 22.7 C
 CSPED : 10 Hz
 SLVNT : CDCL3

RESOL : 0.24 Hz
 BF : 0.01 Hz
 T1 : 0.00 %
 T2 : 0.00 %
 T3 : 90.00 %
 T4 : 100.00 %
 REFVL : 0.00 ppm
 XE : 3997.53 Hz
 XS : 37.20 Hz
 operator

4-AUG-1999 10:42:06.47

DFILE :
SFIL :
COMNT :
EXMOD : SINGL
IRMOD : BCM
POINT : 32768
FREQ : 27100.27 Hz
SCANS : 172
DUMMY : 4
ACQTM : 1.2091 sec
PD : 0.8000 sec
RGAIN : 23
PW1 : 4.75 usec

OBNUC : 13C
OBFRQ : 100.40 MHz
OBSET : 135500.00 Hz

IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRATN : 511
IRRPW : 50.0 usec
IRBP1 : 27
IRBP2 : 6
IRBNS : 0

ADBIT : 16
CTEMP : 23.6 c
CSPED : 12 Hz
SLVNT : CDCL3

RESOL : 0.83 Hz
BF : 2.00 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
REFVL : 77.00 ppm
XE : 21109.23 Hz
XS : 555.77 Hz
operator

A3

§ 25

operator

A3

526

8-FEB-2000 19:06:25.92

DFILE :
SFILF :COMNT : A3
EXMOD : SINGL
IRMOD : BCM
POINT : 32768
FREQU : 27100.27 Hz
SCANS : 926
DUMMY : 4
ACQTM : 0.6046 sec
PD : 0.8000 sec
RGAIN : 23
PW1 : 9.50 usecOBNUC : 13C
OBFRQ : 100.40 MHz
OBSET : 135500.00 HzIRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRATN : 511
IRRPW : 50.0 usec
IRBP1 : 27
IRBP2 : 6
IRRNS : 0ADBIT : 16
CTEMP : 24.7 C
CSPED : 12 Hz
SLVNT : CDCL3RESOL : 0.83 Hz
BF : 2.00 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
REFVL : 77.00 ppm
XE : 20104.39 Hz
XS : 1057.36 Hz
operator

operator

9-FEB-2000 17:55:13.27

DFILE :
SFILE : A4COMNT : A4
EXMOD : SINGL
IRMOD : BCM
POINT : 32768
FREQ : 27100.27 Hz
SCANS : 1264
DUMMY : 4
ACQTM : 0.6046 sec
PD : 0.8000 sec
RGAIN : 23

PW1 : 9.50 usec

OBNUC : 13C
OBFRQ : 100.40 MHz
OBSET : 135500.00 HzIRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRATN : 511
IRRPW : 50.0 usec
IRBP1 : 27
IRBP2 : 6
IRBNS : 0ADBIT : 16
CTEMP : 24.8 C
CSPED : 13 Hz
SLVNT : CDCL3RESOL : 0.83 Hz
BF : 2.00 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
REFVL : 77.00 ppm
XE : 23119.75 Hz
XS : -447.01 Hz
operator

13.750
29.307
31.323
32.105
35.700
48.888
49.398
63.590
64.248
72.903
76.679
77.000
77.313
102.619
117.658
126.445
127.844
131.702
132.500
202.011

ppm

16-FEB-2000 14:50:43.22

3821.36
3813.31

2975.26
2963.66
2948.77
2902.42
2495.94
2487.23
2452.59
2444.54
2437.46
2429.41
2423.80
2418.43
2415.21
2410.82
2407.90
2122.28
2126.43
2129.60
2133.75
2137.90
2140.82

932.05
910.61
909.14
902.07
902.42
897.92
897.43
892.09
887.42
882.54
798.05
792.05
784.00
779.38
770.09
601.04
589.09
576.64
561.52
559.06
556.64
544.69
537.61
530.54
491.75
465.55
464.68
478.58
477.60
440.52
430.77
427.96
417.58

DFILE :
SFILF : osaki a6CCMNT : KATSUMURA-LAB.
EXMOD : SINGL
IRMOD : NON

POINT : 32768

FREQ : 7993.61 Hz

SCANS : 8

DUMMY : 4

ACQTM : 4.0993 sec

PD : 2.9007 sec

RGAIN : 11

PW1 : 10.70 usec

OBNUC : 1H
OBFRQ : 399.65 MHz
OBSET : 134500.00 HzIRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRATN : 511
IRRPW : 50.0 usec
IRBP1 : 27
IRBP2 : 6
IRBNS : 0ADBIT : 16
CTEMP : 24.4 C
CSPED : 13 Hz
SLVNT : CDCL3RESOL : 0.24 Hz
BF : 0.01 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 70.00 %
T4 : 100.00 %
REFVL : 7.26 ppm
XE : 3997.53 Hz
XS : 34.76 Hz

16-FEB-2000 15:37:59.05

DFILE :
SFILE : osaki a6COMNT : KATSUMURA-LAB
EXMOD : SINGL
IRMOD : BCMPOINT : 32768
FREQ : 27100.27 Hz
SCANS : 1563
DUMMY : 4
ACQTM : 0.6046 sec
PD : 0.8000 sec
RGAIN : 23

PW1 : 9.50 usec

OBNUC : 13C
OBFRQ : 100.40 MHz
OBSET : 135500.00 HzIRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRATN : 511
IRRPW : 50.0 usec
IRBP1 : 27
IRBP2 : 6
IRRNS : 0ADBIT : 16
CTEMP : 24.9 C
CSPED : 13 Hz
SLVNT : CDCL3RESOL : 0.83 Hz
BF : 2.00 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 70.00 %
T4 : 100.00 %
REFVL : 77.00 ppm
XE : 23119.75 Hz
XS : -455.28 Hz
GENERATION

13-NOV-2000 20:27:11.48

DFILE :
SFIL : FURU-A7

COMNT : A7
 EXMOD : SINGL
 IRMOD : NON
 POINT : 32768
 FREQU : 7993.61 Hz
 SCANS : 8
 DUMMY : 4
 ACQTM : 4.0993 sec
 PD : 2.9007 sec
 RGAIN : 16
 PW1 : 10.60 usec

OBNUC : 1H
 OBFRQ : 399.65 MHz
 OBSET : 134500.00 Hz

IRNUC : 1H
 IRFRQ : 399.65 MHz
 IRSET : 134500.00 Hz
 IRATN : 511
 IRRPW : 50.0 usec
 IRBP1 : 27
 IRBP2 : 6
 IRRNS : 0

ADBIT : 16
 CTEMP : 24.3 C
 CSPED : 11 Hz
 SLVNT : CDCL3

RESOL : 0.24 Hz
 BF : 0.01 Hz
 T1 : 0.00 %
 T2 : 0.00 %
 T3 : 90.00 %
 T4 : 100.00 %
 REFVL : 0.00 ppm
 XE : 3997.53 Hz
 XS : 36.71 Hz
 Generator

A7

13-NOV-2000 20:22:29.48

DFILE :
SFIL : FURU-A7

COMNT : A7
 EXMOD : SINGL
 IRMOD : BCM
 POINT : 32768
 FREQU : 27100.27 Hz
 SCANS : 22589
 DUMMY : 4
 ACQTM : 0.6046 sec
 PD : 0.8000 sec
 RGAIN : 23
 PW1 : 9.50 usec

OBNUC : 13C
 OBFRQ : 100.40 MHz
 OBSET : 135500.00 Hz

IRNUC : 1H
 IRFRQ : 399.65 MHz
 IRSET : 134500.00 Hz
 IRATN : 511
 IRPW : 50.0 usec
 IRBP1 : 27
 IRBP2 : 6
 IRFNS : 0

ADBIT : 16
 CTEMP : 24.9 c
 CSPED : 11 Hz
 SLVNT : CDCL3

RESOL : 0.83 Hz
 BF : 2.00 Hz
 T1 : 0.00 %
 T2 : 0.00 %
 T3 : 90.00 %
 T4 : 100.00 %
 REFVL : 77.00 ppm
 XE : 23119.75 Hz
 XS : -445.36 Hz
 operator

13.758
 21.385
 29.151
 31.000
 31.224
 32.031
 32.446
 35.642
 45.202
 45.400
 63.598
 67.991
 72.582
 76.687
 77.000
 77.321
 102.784
 117.436
 126.610
 127.786
 131.793
 132.352
 170.427
 201.962

ppm
 0
 50
 100
 150
 200

Y2

23

22-JAN-2001 21:52:00.17

DFILE : Y2
 SFIL : Y2
 COMNT : Y2
 EXMOD : SINGL
 IRMOD : NON
 POINT : 32768
 FREQU : 7993.61 Hz
 SCANS : 8
 DUMMY : 4
 ACQTM : 4.0993 sec
 PD : 2.9007 sec
 RGAIN : 16
 PW1 : 5.30 usec

OBNUC : 1H
 OBFRQ : 399.65 MHz
 OBSET : 134500.00 Hz

IRNUC : 1H
 IRFRQ : 399.65 MHz
 IRSET : 134500.00 Hz
 IRATN : 511
 IRRPW : 50.0 usec
 IRBP1 : 27
 IRBP2 : 6
 IRRNS : 0

ADBIT : 16
 CTEMP : 24.5 C
 CSPED : 11 Hz
 SLVNT : CDCL3

RESOL : 0.24 Hz
 BF : 0.01 Hz
 T1 : 0.00 %
 T2 : 0.00 %
 T3 : 90.00 %
 T4 : 100.00 %
 REFVL : 0.00 ppm
 XE : 3997.53 Hz
 XS : 36.96 Hz

30-OCT-1999 12:59:47.50

DFILE :
SFIL :

COMNT :
EXMOD : SINGL
IRMOD : BCM

POINT : 32768
FREQ : 27100.27 Hz
SCANS : 171

DUMMY : 4
ACQTM : 1.2091 sec
PD : 0.8000 sec
RGAIN : 23

PW1 : 4.75 usec

OBNUC : ¹³C
OBFRQ : 100.40 MHz
OBSET : 135500.00 Hz

IRNUC : ¹H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRATN : 511
IRAPW : 50.0 usec
IRBP1 : 27
IRBP2 : 6
IRBNS : 0

ADBIT : 16
CTEMP : 23.5 C
CSPED : 12 Hz
SLVNT : CDCL3

RESOL : 0.83 Hz
BF : 2.00 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
REFVL : 77.00 ppm
XE : 21109.23 Hz
XS : 556.59 Hz
Operator

02-AUG-1999 00:44:45.73

DEFILE : ET

```
COMINT : COM3
EYMOD : SINCL
```

IRMOD : VOR-

POINT : 32768

REF: 7993.61 HZ

SCANS : SNYJS 8

DUMMY : 4

ACQTM : 1.0993 sec.

PD 2.9007 sec

BEATN 15

Case	Time (sec)
1	5.35
2	5.35
3	5.35
4	5.35
5	5.35
6	5.35
7	5.35
8	5.35
9	5.35
10	5.35
11	5.35
12	5.35
13	5.35
14	5.35
15	5.35
16	5.35
17	5.35
18	5.35
19	5.35
20	5.35
21	5.35
22	5.35
23	5.35
24	5.35
25	5.35
26	5.35
27	5.35
28	5.35
29	5.35
30	5.35
31	5.35
32	5.35
33	5.35
34	5.35
35	5.35
36	5.35
37	5.35
38	5.35
39	5.35
40	5.35
41	5.35
42	5.35
43	5.35
44	5.35
45	5.35
46	5.35
47	5.35
48	5.35
49	5.35
50	5.35

OBNUC : 1H

03FRO : 3

OBSSET : 134500.00 HZ

TRANUC : 1H

IRFRO

IRSET : 13450.50 Hz

BRAIN : 511

188PW : 50.0 usec

T83P1 27

TABLE 6

TRBNS

ADBIT :

CTEMP

USPED : 12 H7

SYNOPSIS: 1000

RESOL :

100

—

12

...

24

REFE

YU. A. IZRAEL, 42

5.7%

operator

22-AUG-1999 01:02:18.08

DFILE :
SFIL : ET C

COMNT :
EXMOD : SINGL
IRMOD : BCM
POINT : 32768
FREQ : 27100.27 Hz
SCANS : 256
DUMMY : 4
ACQTM : 1.2091 sec
PD : 0.8000 sec
RGAIN : 23

PW1 : 4.75 usec

OBNUC : 13C
OBFRQ : 100.40 MHz
OBSET : 135500.00 Hz

IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRATN : 511
IRRPW : 50.0 usec
IRBP1 : 27
IRBP2 : 6
IRNS : 0

ADBIT : 16
CTEMP : 23.6 c
CSPED : 13 Hz
SLVNT : CDCL3

RESOL : 0.83 Hz
BF : 2.00 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
REFVL : 77.00 ppm
XE : 21109.23 Hz
XS : 558.25 Hz
operator

20

25-AUG-1999 09:54:25.32

DFILE :
SFIL :
COMNT :
EXMOD :
IRMOD :
POINT :
FREQ :
SCANS :
DUMMY :
ACQTM :
PD :
RGAIN :
PW1 :
5.35 usec

OBNUC : 1H
OBFRQ : 399.65 MHz
OBSET : 134500.00 Hz
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRATN : 511
IRRPW : 50.0 usec
IRBP1 : 27
IRBP2 : 6
IRBNS : 0
ADBIT : 16
CTEMP : 23.2 C
CSPED : 11 Hz
SLVNT : CDCL3

RESOL : 0.24 Hz
BF : 0.01 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
REFVL : 0.00 ppm
XE : 3997.53 Hz
XS : 34.76 Hz
concentration

17-NOV-1999 08:59:09.96

DFILE :
SFIL : Y6-ET

COMNT :
EXMOD : SINGL
IRMOD : BCM
POINT : 32768
FREQ : 27100.27 Hz
SCANS : 256
DUMMY : 4
ACQTM : 1.2091 sec
PD : 0.8000 sec
RGAIN : 23
PW1 : 4.75 usec

OBNUC : 13C
OBFRQ : 100.40 MHz
OBSET : 135500.00 Hz

IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRATN : 511
IRRPW : 50.0 usec
IRBP1 : 27
IRBP2 : 6
IRRNS : 0

ADBIT : 16
CTEMP : 22.8 C
CSPED : 11 Hz
SLVNT : CDCL3

RESOL : 0.83 Hz
BF : 2.00 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
REFVL : 77.00 ppm
XE : 21109.23 Hz
XS : 557.42 Hz
operator

ADBIT	:	16
CTEMP	:	24.9 C
CSPED	:	12 Hz
SLVNT	:	CDCL3
RESOL	:	0.24 Hz
BF	:	0.25 Hz
T1	:	0.00 %
T2	:	0.00 %
T3	:	90.00 %
T4	:	100.00 %
REFVL	:	0.00 ppm
XE	:	2678.28 Hz
XS	:	694.39 Hz
operator

10-FEB-1999 20:43:16.91

DFILE : HARA-CARBOXYL-ACTIVE
 SFIL : HARA-CARBOXYL-ACTIVE
 COMNT : KATSUMURA-LAB.
 EXMOD : SINGL
 IRMOD : BCM
 POINT : 32768
 FREQU : 27100.27 Hz
 SCANS : 731
 DUMMY : 4
 ACQTM : 1.2091 sec.
 PD : 0.8000 sec.
 RGAIN : 23
 PW1 : 4.75 usec

OBNUC : ¹³C
 OBFRQ : 100.40 MHz
 OBSET : 135500.00 Hz
 IRNUC : ¹H
 IRFRQ : 399.65 MHz
 IRSET : 134500.00 Hz
 IRATN : 511
 IRRPW : 50.0 usec
 IRBP1 : 27
 IRBP2 : 6
 IRBNS : 0

ADBIT : 16
 CTEMP : 25.3 C
 CSPED : 12 Hz
 SLVNT : CDCL3

RESOL : 0.83 Hz
 BF : 1.00 Hz
 T1 : 0.00 %
 T2 : 0.00 %
 T3 : 90.00 %
 T4 : 100.00 %
 REFVL : 77.00 ppm
 XE : 20104.39 Hz
 XS : 1061.50 Hz
 operator

19-NOV-1999 10:00:17.73

DFILE :
SFIL : YB

COMNT :
EXMOD : SINGL
IRMOD : BCM
POINT : 32768
FREQ : 27100.27 Hz
SCANS : 256
DUMMY : 4
ACQTM : 1.2091 sec
PD : 0.8000 sec
RGAIN : 23

PW1 : 4.75 usec

OBNUC : 13C
OBFRQ : 100.40 MHz
OBSET : 135500.00 Hz

IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRATN : 511
IRRPW : 50.0 usec
IRBP1 : 27
IRBP2 : 6
IRBNS : 0

ADBIT : 16
CTEMP : 22.8 c
CSPED : 12 Hz
SLVNT : CDCL3

RESOL : 0.83 Hz
BF : 2.00 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
REFVL : 77.00 ppm
XE : 21109.23 Hz
XS : 558.25 Hz

77.321
77.000
76.679
70.558
67.473
64.610
59.509
46.971
41.303
35.099
29.384
26.860
24.980
20.003
18.118
15.494
7.728
4.761

168.650
146.675
136.745
134.631
133.997
126.025
121.287
117.469

ppm

25

22-JAN-2001 22:22:44.47

DFILE :
SFILE : Y3-C

COMNT : Y3
EXMOD : SINGL
IRMOD : BCM
POINT : 16384
FREQ : 27100.27 Hz
SCANS : 5000
DUMMY : 4
ACQTM : 0.6046 sec
PD : 2.3954 sec
RGAIN : 23

PW1 : 4.80 usec

OBNUC : 13C
OBFRQ : 100.40 MHz
OBSET : 135500.00 Hz

IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRATN : 511
IRRPW : 50.0 usec
IRBP1 : 27
IRBP2 : 6
IRRNS : 0

ADBIT : 16
CTEMP : 24.5 c
CSPED : 10 Hz
SLVNT : CDCL3

RESOL : 1.65 Hz
BF : 1.50 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
REFVL : 77.00 ppm
XE : 21109.23 Hz
XS : 559.08 Hz
operator

44

```

DFILE :
SFILE : Y4
COMNT : Y4
EXMOD : SINGL
IRMOD : NON
POINT : 32768
FREQ : 7993.61 Hz
SCANS : 4
DUMMY : 4
ACQTM : 4
PPD : 2.9007 sec
RGAIN : 12
PW1 : 10.60 usec

```

```

CBNUC : 1H
OBFRQ : 399.65 MHZ
OBSET : 134500.00 HZ

```

```

IRNUC      : 14      :
IRFRQ      : 359.65  MHz
IRSET      : 134500.00 Hz
IRATN      : 511     :
IRBPW      : 50.0    usec
IRBP1      : 27      :
IRBP2      : 5       :
IRNS       : 0       :

```

```
ADBIT : 1C
CTEMP : 24.5 c
CSPED : 14 Hz
SLVNT : CDCL3
```

RESULT:	0.24 Hz
8BF	0.01 Hz
T1	0.00 %
T2	0.00 %
T3	90.00 %
T4	100.00 %
REFVL	0.00 ppm
XE	3997.53 Hz
XS	39.15 Hz

operator

27-JAN-2001 02:01:37.94

DFILE :
SFIL : Y41BCM

COMNT :
EXMOD : SINGL
IRMOD : BCM
POINT : 16384
FREQ : 27100.27 Hz
SCANS : 10000
DUMMY : 4
ACQTM : 0.6046 sec
PD : 2.3954 sec
RGAIN : 23

PW1 : 4.80 usec

OBNUC : ¹³C
OBFRQ : 100.40 MHz
OBSET : 135500.00 Hz

IRNUC : ¹H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRATN : 511
IRRPW : 50.0 usec
IRBP1 : 27
IRBP2 : 6
IRNS : 0

ADBIT : 16
CTEMP : 24.2 C
CSPED : 13 Hz
SLVNT : CDCL3

RESOL : 1.65 Hz
BF : 1.50 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
REFVL : 77.00 ppm
XE : 21109.23 Hz
XS : 557.42 Hz
operator

14-MAR-2001 08:03:11.98

```

DFILE :
SFILF :
COMNT : Y5
EXMOD : SINGL
IRMOD : NON
POINT : 32768
FREQU : 7993.61 Hz
SCANS : 8
DUMMY : 4
ACQTM : 4.0993 sec
PD : 2.9007 sec
RGAIN : 14
PW1 : 5.30 usec

```

OBNUC :	1H	
OBFRQ :	399.65 MHz	
OBSET :	134500.00 Hz	
IRNUC :	1H	
IRFRQ :	399.65 MHz	
IRSET :	134500.00 Hz	
IRATN :	511	
IRRPW :	50.00 usec	
IRBP1 :	27	
IRBP2 :	6	
IRRNS :	0	

```
ADBIT : 16
CTEMP : 23.7 C
CSPED : 14 Hz
SLVNT : CDCL3
```

RESOL :	0.24 Hz
BF :	0.01 Hz
T1 :	0.00 %
T2 :	0.00 %
T3 :	90.00 %
T4 :	100.00 %
REFVL :	0.00 ppm
XE :	3997.53 Hz
XS :	43.06 Hz
operator	

operator

14-MAR-2001 21:30:26.28

DFILE :
SFILE : Y51BCM

COMNT : 13C SINGL BCM Y5
EXMOD : SINGL
IRMOD : BCM
POINT : 16384
FREQU : 27100.27 Hz
SCANS : 5000
DUMMY : 4
ACQTM : 0.6046 sec
PD : 2.3954 sec
RGAIN : 23
PW1 : 4.80 usec

OBNUC : 13C
OBFRQ : 100.40 MHz
OBSET : 135500.00 Hz
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRATN : 511
IRRPW : 50.0 usec
IRBP1 : 27
IRBP2 : 6
IRRNS : 0
ADBIT : 16
CTEMP : 23.4 C
CSPED : 14 Hz
SLVNT : CDCL3

RESOL : 1.65 Hz
BF : 1.50 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
REFVL : 77.00 ppm
XE : 21109.23 Hz
XS : 557.42 Hz
OI

17-MAR-2001 01:04:43.34

DFILE :
SFILE : Y8

COM1 : KATSUMURA-LAB
EXMOD : SINGL
IRMOD : NON
POINT : 32768
FREQU : 7993.61 Hz
SCANS : 8
DUMMY : 4
ACQTM : 4.0993 sec
PD : 2.9007 sec
RGAIN : 18
PW1 : 10.60 usec

OBNUC : ¹H
OBFRQ : 399.65 MHz
OBSET : 134500.00 Hz

IRNUC : ¹H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRATN : 514
IRRPW : 50.0 usec
IRBP1 : 27
IRBP2 : 6
IRBNS : 0

ADBIT : 16
CTEMP : 23.8 C
CSPED : 14 Hz
SLVNT : CDCL3

RESOL : 0.24 Hz
BF : 0.01 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
REFVL : 0.00 ppm
XE : 3997.53 Hz
XS : 37.20 Hz

17-MAR-2001 01:38:31.23

DFILE :
SFIL : YB1BCM

COMNT :
EXMOD : SINGL
IRMOD : BCM
POINT : 16384
FREQ : 27100.27 Hz
SCANS : 5000
DUMMY : 4
ACQTM : 0.6046 sec
PD : 2.3954 sec
RGAIN : 23

PW1 : 4.80 usec

OBNUC : 13C
OBFRQ : 100.40 MHz
OBSET : 135500.00 Hz

IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRATN : 511
IRRPW : 50.0 usec
IRBP1 : 27
IRBP2 : 6
IRRNS : 0

ADBIT : 16
CTEMP : 24.3 C
CSPED : 13 Hz
SLVNT : CDCL3

RESOL : 1.65 Hz
BF : 1.50 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
REFVL : 77.00 ppm
XE : 21109.23 Hz
XS : 559.08 Hz
operator

28-JUN-2001 00:31:13.33

DFILE :
 SFIL :
 COMNT :
 EXMOD : SINGL
 IRMOD : NON
 POINT : 32768
 FREQU : 7993.61 Hz
 SCANS : 8
 DUMMY : 4
 ACQTM : 4.0993 sec
 PD : 2.9007 sec
 RGAIN : 18
 PW1 : 5.30 usec

OBNJC : ¹H
 OBFRQ : 399.65 MHz
 OBSET : 134500.00 Hz
 IRNJC : ¹H
 IRFRQ : 399.65 MHz
 IRSET : 134500.00 Hz
 IRATN : 511
 IRRPW : 50.0 usec
 IRBP1 : 27
 IRBP2 : 6
 IRRNS : 0

ADBIT : 16
 CTEMP : 22.9 C
 CSPED : 13 Hz
 SLVNT : CDCL3
 RESOL : 0.24 Hz
 BF : 0.01 Hz
 T1 : 0.00 %
 T2 : 0.00 %
 T3 : 90.00 %
 T4 : 100.00 %
 REFVL : 0.00 ppm
 XE : 3997.53 Hz
 XS : 37.20 Hz
 oper

29a

28-JUN-2001 00:55:01.48

DFILE :
SFIL :

COMNT :
EXMOD : SINGL
IRMOD : BCM
POINT : 32768
FREQ : 27100.27 Hz
SCANS : 765
DUMMY : 4
ACQTM : 0.6046 sec
PD : 0.8000 sec
RGAIN : 23
PW1 : 9.50 usec

OBNUC : 13C
OBFRQ : 100.40 MHz
OBSET : 135500.00 Hz

IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRATN : 511
IRRPW : 50.0 usec
IRBP1 : 27
IRBP2 : 6
IRANS : 0

ADBIT : 16
CTEMP : 24.0 C
CSPED : 13 Hz
SLVNT : CDCL3

RESOL : 0.83 Hz
BF : 2.00 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
REFVL : 77.00 ppm
XE : 21109.23 Hz
XS : 559.08 Hz
operator

29a

21-MAY-1998 16:35:30.29

DFILE :
 SFILE : IBS ether
 COMNT : KATSUMURA-LAB.
 EXMOD : SINGL
 IRMOD : NON
 POINT : 32768
 FREQU : 7993.61 Hz
 SCANS : 8
 DUMMY : 4
 ACQTM : 4.0993 sec
 PD : 2.9007 sec
 RGAIN : 19
 PW1 : 5.35 usec

OBNUC : 1H
 OBFRQ : 399.65 MHz
 OBSET : 134500.00 Hz
 IRNUC : 1H
 IRFRQ : 399.65 MHz
 IRSET : 134500.00 Hz
 IRATN : 511
 IRRPW : 50.0 usec
 IRBP1 : 27
 IRBP2 : 6
 IRRNS : 0
 ADBIT : 16
 CTEMP : 21.6 C
 CSPED : 11 Hz
 SLVNT : CDCL3

RESOL : 0.24 Hz
 BF : 0.01 Hz
 T1 : 0.00 %
 T2 : 0.00 %
 T3 : 90.00 %
 T4 : 100.00 %
 REFVL : 0.00 ppm
 XE : 3997.53 Hz
 XS : 36.47 Hz
 operator

21-MAY-1998 21.04.10.94

DFILE :
 SF FILE :
 COMNT : KATSUMURA-LAB.
 EXMOD : SINGL
 IRMOD : BCM
 POINT : 32768
 FREQU : 27100.27 Hz
 SCANS : 800
 DUMMY : 4
 ACQTM : 1.2091 sec
 PD : 0.8000 sec
 RGAIN : 23
 PW1 : 4.75 usec

OBNUC : 13C
 OBFRQ : 100.40 MHz
 OBSET : 135500.00 Hz

IRNUC : 1H
 IRFRQ : 399.65 MHz
 IRSET : 134500.00 Hz
 IRATN : 511
 IRRPW : 50.0 usec
 IRBP1 : 27
 IRBP2 : 6
 IRRNS : 0

ADBIT : 16
 CTEMP : 21.8 C
 CSPED : 12 Hz
 SLVNT : CDCL3

RESOL : 0.83 Hz
 BF : 1.00 Hz
 T1 : 0.00 %
 T2 : 0.00 %
 T3 : 90.00 %
 T4 : 100.00 %
 REFVL : 77.00 ppm
 XE : 20104.39 Hz
 XS : 1050.67 Hz
 operator

30

COMNT :	SINGL	
EXMOD :	NON	
IRMOD :		32768
POINT :		7993.61 Hz
FREQU :		8
SCANS :		4
DUMMY :		4.0993 sec
ACQTM :		2.9007 sec
PD :		15
RGAIN :		
PW1 :		10.60 usec

```
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRATN : 511
IRAPW : 50.0 usec
IRBP1 : 27
IRBP2 : 6
IRANS : 0
```

RESOL	:	0.24	Hz
BF	:	0.01	Hz
T1	:	0.00	%
T2	:	0.00	%
T3	:	90.00	%
T4	:	100.00	%
REFVL	:	0.00	pdm
XE	:	3997.53	Hz
XS	:	35.98	Hz

12-MAR-2001 21:58:16.73

DFILE :
SFIL : A81BCM

COMNT :
EXMOD : SINGL
IRMOD : BCM
POINT : 16384
FREQU : 27100.27 Hz
SCANS : 5000
DUMMY : 4
ACQTM : 0.6046 sec
PD : 2.3954 sec
RGAIN : 23

PW1 : 4.80 usec

OBNUC : 13C
OBFRQ : 100.40 MHz
OBSET : 135500.00 Hz

IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRATN : 511
IRRPW : 50.0 usec
IRBP1 : 27
IRBP2 : 6
IRRNS : 0

ADBIT : 16
CTEMP : 23.6 C
CSPED : 13 Hz
SLVNT : CDCL3

RESOL : 1.65 Hz
BF : 1.50 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
REFVL : 77.00 ppm
XE : 22903.90 Hz
XS : -841.09 Hz
NAME : nmrator

1H SINGL NON A9

14-MAR-2001 00:10:40.71

DFILE :
SF FILE : A90N0N

COMNT : 1H SINGL NON A9
EXMOD : SINGL
IRMOD : NON
POINT : 16384
FREQ : 793.51 Hz
SCANS : 16
DUMMY : 4
AQ : 2.0495 sec
PD : 4.3504 sec
RGAIN : 14
PW1 : 5.30 usec

ORNU2 : 1H
ORFRQ : 399.65 MHz
ORSET : 134500.00 Hz

IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRATN : 511
IRRPW : 50.0 usec
IRBP1 : 27
IRBP2 : 6
IRBNS : 0

ADBIT : 16
CTEMP : 24.4 C
CSPED : 14 Hz
SLVNT : CDCL3

RESOL : 0.49 Hz
BF : 0.24 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
REFVL : 0.00 ppm
XE : 3997.29 Hz
YS : 41.71 Hz

13C SINGL B.M A3

14-MAR-2001 06:04:19.57

DFILE : A91B.M
 EXMOD : SINGL
 INMOD : BCM
 POINT : 16384
 FREQ : 27100.17 HZ
 SCANS : 5000
 DUMMY : 4
 ACQTM : 0.5045 sec
 PD : 2.3954 sec
 RGAIN : 23

W1 : 4.80 use

BNUC : 13C
 ORFRQ : 100.40 MHz
 JRSET : 135500.00 HZ

INUC : 1H
 IRFRQ : 300.00 MHz
 IRSET : 134500.00 HZ
 IRATN : 514
 IRPW : 50.0 usec
 IRBP1 : 27
 IRBP2 : 5
 TRNS : 0

ADBIT : 16
 CTEMP : -4.1 C
 CPED : 12 HZ
 PLVNT : COL3

RESOL : 1.25 HZ
 BF : 1.50 HZ
 T1 : 0.00 %
 T2 : 0.00 %
 T3 : 0.00 %
 T4 : 100.00 %
 REFVL : 77.00 ppm
 XE : 23118.92 HZ
 XS : -440.02 HZ

17--MAR-2001 21: 31: 57.48

```

DFILE :
SFILE : Peridin-100d

```

```

COMNT : SINGL
EXMOD : NON
IRMOD :
POINT : 32768
FREQU : 7993.61 Hz
SCANS : 8217
DUMMY : 4
ACQTIM : 4.0993 sec
PD : 2.9007 sec
RGAIN : 23

```

PW1 : 10.60 usec

```
OBNUC : 1H
OBFRQ : 399.65 MHz
OBSET : 134500.00 Hz
```

```

IRNUC      : 1H
IRFRQ      : 399.65 MHz
IRSET      : 134500.00 HZ
IRATN      : 511
IRRPW      : 50.0 usec
IRBP1      : 27
IRBP2      : 6
IRRNS      : 0

```

```
ADBIT : 16
CTEMP : 23.8 c
CSPED : 12 Hz
SLVNT : CDCL3
```

RESOL	:	0.24	Hz
BF	:	0.01	Hz
T1	:	0.00	%
T2	:	0.00	%
T3	:	90.00	%
T4	:	100.00	%
REFVL	:	7.26	ppm
XE	:	3038.10	Hz

19-MAR-2004 20:35:06.34

DFILE :
SFILE :

COMNT :
EXMOD :
IRMOD :
POINT :
FREQ :
SCANS :
DUMMY :
ACQTM :
PD :
RGAIN :

SINGL
BOM
32768
27400.27 Hz
92724

4
0.6045 sec
0.3009 sec
23

PW1 : 9.50 usec

ORNU : 130
ORFRQ : 100.40 MHz
ORSET : 135500.00 Hz

IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRATN : 514
IRRPW : 50.0 usec
IRBP1 : 17
IRBP2 : 6
IRBNS : 0

ADBIT : 16
JTEMP : 24.7 C
JSPED : 12 Hz
SLVNT : CDCL3

RESOL : 0.83 Hz
BF : 2.00 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
REFVL : 77.00 ppm
XEL : 3119.75 Hz