Efficient Access to Orthoquinols and Their [4+2] Cyclodimers via SIBX-Mediated Hydroxylative Phenol Dearomatization

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General Experimental Methods. All reactions were run under N₂ using stabilized *o*-iodoxybenzoic acid (SIBX), the IBX content of which being 49% by weight of SIBX. Tetrahydrofuran (THF) was purified by distillation from sodium/benzophenone under N₂ prior to use. Dichloromethane (CH₂Cl₂), ethyl acetate (EtOAc), toluene, acetone and dimethyl sulfoxide (DMSO) were used as received. Evaporations were conducted under reduced pressure at temperatures less than 45 °C unless otherwise noted. Column chromatography was carried out under positive pressure using 40-63 µm silica gel and the indicated solvents. Further drying of the residues was accomplished under high vacuum, except for volatile products. Melting points are uncorrected. NMR spectra of samples in the indicated solvent were run at 250, 300 or 400 MHz. Carbon multiplicities were determined by DEPT-135 or *J*-MOD experiments. Electron impact mass spectra were obtained at 70 eV. Low and high resolution electron impact, chemical ionisation and liquid secondary ion mass spectrometry data (EIMS, CIMS, LSIMS, HRMS) were obtained either from the mass spectrometry laboratory at the Institut Européen de Chimie et Biologie, Pessac, France, from the Centre d'Etude Structurale et d'Analyse des Molécules Organiques (CESAMO) de l'Université de Bordeaux, Talence, France, or from the Centre Régional de Mesures Physiques de l'Ouest (CRMPO), Rennes, France.

3,10-Dihydroxy-3,5,8,10-tetramethyltricyclo[6.2.2.0^{2,7}]dodeca-5,11-diene-4,9-dione (9a). The SIBX-mediated HPD of 2,6-dimethylphenol **7a** (1.22 g, 10 mmol) was run according to the general procedure A. Purification by column chromatography, eluting with hexanes/acetone (3:1), gave dimer **9a** (1.32 g, 96%) as a white solid. Crystallization from a mixture of CH₂Cl₂ and hexanes afforded colourless prisms, which were used for X-ray analysis. The resulting crystal structure was identical to that previously reported for **9a**.^{10b} mp 172 °C (lit.^{10a} mp 194–196 °C); IR (NaCl, neat) 3416, 1713, 1670 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz) δ 1.24 (s, 3H), 1.32 (s, 3H), 1.43 (s, 3H), 1.85 (s, 3H), 2.33 (s, 1H), 2.80-2.95 (m, 1H), 3.25 (dd, *J* = 1.7, 8.3 Hz, 1H), 3.38 (d, *J* = 6.8 Hz, 1H), 4.00 (s, 1H), 5.51 (d, *J* = 8.1 Hz, 1H), 6.20-6.35 (m, 2H); ¹³C NMR (CDCl₃, 100 MHz) δ 214.8, 203.0, 139.3, 135.7, 135.3, 133.2, 73.6, 72.9, 53.7, 44.2, 43.6, 42.7, 31.7, 26.2, 16.3, 15.6; EIMS *m/z* (rel. intensity) 276 (M⁺, 5), 242 (3), 216 (3), 138 (42), 122 (15), 121 (33), 43 (100).

3,10-Dihydroxy-3,5,7,8,10,11-hexamethyl-tricyclo[6.2.2.0^{2,7}]dodeca-5,11-diene-4,9-dione (9b). The SIBX-mediated HPD of 2,4,6-trimethylphenol **7b** (1.36 g, 10 mmol) was run according to the general procedure A. Purification by column chromatography, eluting with hexanes/acetone (3:1), gave dimer **9b** (1.42 g, 94%) as a white solid. Crystallization from a mixture of CH₂Cl₂ and hexanes afforded colourless prisms, which were used for X-ray analysis. The resulting crystal data were deposited at the Cambridge Crystallographic Data Centre under the number CCDC 640072. **9b**: mp 159–162 °C (lit.^{10a} mp 182–183 °C); IR (NaCl, neat) 3482, 2979, 2876, 1686 cm⁻¹; ¹H NMR

(CDCl₃, 400 MHz) δ 1.16 (s, 3H), 1.24 (s, 6H), 1.36 (s, 3H), 1.70 (d, J = 1.7 Hz, 3H), 1.83 (d, J = 1.5 Hz, 3H), 2.23 (bs, 1H), 2.80 (d, J = 2.1 Hz, 1H), 3.14 (t, J = 2.1 Hz, 1H), 3.92 (s, 1H), 5.04 (s, 1H), 6.03 (s, 1H); ¹³C NMR (CDCl₃, 75.5 MHz) δ 214.0, 202.3, 145.1, 144.9, 132.8, 127.3, 73.6, 71.9, 57.6, 48.6, 48.5, 45.1, 32.3, 25.0, 23.0, 21.0, 16.1, 12.3; EIMS m/z (rel. intensity) 304 (M^+ , 1), 152 (18), 136 (32), 110 (55), 28 (71).

6-Hydroxy-2,5,6-trimethylcyclohexa-2,4-dienone (8c) and 3,10-Dihydroxy-3,5,6,8,10,12-hexamethyl-tricyclo[6.2.2.0^{2,7}]dodeca-5,11-diene-4,9,-dione (9c). The SIBX-mediated HPD of 2,3,6-trimethylphenol **7c** (1.36 g, 10 mmol) was run according to the general procedure A apart from the fact that a saturated aqueous solution of Na₂CO₃ was used instead of an aqueous 1 M solution of NaOH during the work-up. Purification by column chromatography, eluting with hexanes/acetone from (10:1) to (3:1), afforded orthoquinol **8c**^{13a} (480 mg, 32%) as a bright yellow oil and the dimer **9c** (471 mg, 31%) as a colourless solid. **8c**: R_f = 0.6 [hexanes/acetone (3:1)]; IR (NaCl) 3415, 2922, 1716, 1115 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz) δ 1.34 (s, 3H), 1.89 (s, 3H), 1.98 (s, 3H), 5.79 (d, J = 6.0 Hz, 1H), 6.71 (d, J = 6.0 Hz, 1H); ¹³C NMR (CDCl₃, 75 MHz) δ 207.7, 152.9, 139.5, 129.1, 117.0, 78.8, 29.3, 17.6, 14.8; CIMS (NH₃) m/z (rel. intensity) 170 (MNH₄⁺, 100); HRMS (EI) calcd for C₉H₁₂O₂ 152.0837, found 152.0842. Crystallization of **9c** from a mixture of CH₂Cl₂ and hexanes afforded colourless prisms, which were used for X-ray analysis. The resulting crystal data were deposited at the Cambridge Crystallographic Data Centre under the number CCDC 640073. **9c**: mp 180 °C (lit.^{13a} mp 191–192 °C); R_f = 0.1 [hexanes/acetone (3:1)]; IR (NaCl, neat) 3404, 2913, 1640, 1623, 1664, 1377, 1121 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz) δ 1.17 (s, 3H), 1.22 (s, 6H), 1.43 (s, 3H), 1.81 (s, 3H), 1.98 (s, 3H), 2.88 (d, J = 7.8 Hz, 1H), 2.98 (d, J = 7.8 Hz, 1H), 3.32 (d, J = 6.1 Hz, 1H), 5.96 (d, J = 6.1 Hz, 1H); ¹³C NMR (CDCl₃, 75 MHz) δ 213.7, 202.8, 148.3, 136.9, 132.3, 131.0, 73.7, 71.9, 56.4, 48.9, 44.1, 41.2, 32.0, 26.2, 23.6, 18.7, 15.1, 13.5; EIMS m/z (rel. intensity) 304 (M^+ , 9), 240 (16), 152 (42), 135 (85), 123 (73), 109 (71), 95 (100). HRMS (EI) calcd for C₁₈H₂₄O₄ 304.1675, found 304.1671.

6-Hydroxy-5-methoxy-2,6-dimethylcyclohexa-2,4-dienone (8d) and 3,10-Dihydroxy-6-methoxy-3,5,8,10-tetramethyl-tricyclo[6.2.2.0^{2,7}]dodeca-5-ene-4,9,12-trione (9d). The SIBX-mediated HPD of 3-methoxy-2,6-dimethylphenol **7d** (152 mg, 1 mmol) was run according to the general procedure A apart from the fact that a saturated aqueous solution of Na₂CO₃ was used instead of an aqueous 1 M solution of NaOH during the work-up. Purification by column chromatography, eluting with CH₂Cl₂/MeOH from (100:1) to (50:1), afforded orthoquinol **8d** (70 mg, 42%) as a bright yellow oil and dimer **9d** (72 mg, 43%) as a colourless solid. **8d**: R_f = 0.3 [CH₂Cl₂/MeOH (100:1)]; IR (NaCl) 3370, 2930, 1730, 1140 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz) δ

1.46 (s, 3H), 1.88 (d, $J = 1.3$ Hz, 6H), 3.43 (s, 1H), 3.76 (s, 3H), 5.13 (d, $J = 7.2$ Hz, 1H), 6.89 (qd, $J = 1.3, 7.2$ Hz, 1H); ^{13}C NMR (CDCl_3 , 75 MHz) δ 203.2, 167.6, 141.4, 123.9, 91.3, 76.8, 56.1, 29.4, 14.6; CIMS (NH_3) m/z (rel. intensity) 186 (MNH_4^+ , 100); HRMS (EI) calcd for $\text{C}_9\text{H}_{12}\text{O}_3$ 168.0786, found 168.0790. **9d**: mp 153–154 °C; $R_f = 0.1$ [hexanes/acetone (3:1)]; IR (NaCl, neat) 3469, 2952, 1740, 1718, 1664, 1322, 1215, 1042 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz) δ 1.19 (s, 3H), 1.37 (s, 6H), 1.86 (s, 3H), 2.12 (d, $J = 21.7$ Hz, 1H), 2.47 (d, $J = 21.7$ Hz, 1H), 2.87 (s, 1H), 3.10 (s, 1H), 3.20 (s, 1H), 3.77 (s, 3H), 4.11 (br s, 1H); ^{13}C NMR (CDCl_3 , 75 MHz) δ 212.3, 204.8, 202.0, 168.6, 117.4, 76.5, 72.3, 66.4, 60.0, 45.5, 38.0, 37.7, 31.2, 22.9, 12.5, 10.3; EIMS m/z (rel. intensity) 322 (M^+ , 1), 277(92), 233 (60), 155 (83), 140 (100); HRMS (EI) calcd for $\text{C}_{15}\text{H}_{19}\text{O}_5$ [$\text{M}^+ - \text{CHO}$] 279.1232, found 279.1235.

3,10-Dihydroxy-3,6,10,12-tetramethyltricyclo[6.2.2.0^{2,7}]dodeca-5,11-diene-4,9-dione (9e). The SIBX-mediated HPD of 2,5-dimethylphenol (**7e**) (1.22 g, 10 mmol) was run according to the general procedure B. Purification by column chromatography, eluting with hexanes/acetone (30:1), gave dimer **9e** as a brown solid (730 mg), which was further purified by crystallization from a mixture of CH_2Cl_2 and hexanes to furnish pure **9e** (676 mg, 49%) as colourless prisms. These prisms were used for X-ray analysis, and the resulting crystal data were deposited at the Cambridge Crystallographic Data Centre under the number CCDC 640074. **9e**: mp 189 °C (lit.^{10c} mp 200–201 °C); $R_f = 0.10$ [hexanes/acetone, (3:1)]; IR (NaCl, neat) 3435, 1729, 1671, 1379, 1159 (cm^{-1}); ^1H NMR (CDCl_3 , 300 MHz) δ 1.25 (s, 3H), 1.30 (s, 3H), 1.61 (d, $J = 1.3$ Hz, 3H), 2.00 (d, $J = 1.0$ Hz, 3H), 2.28 (bs, 1H), 3.10–3.20 (m, 3H), 3.32 (dd, $J = 1.3, 6.8$ Hz, 1H), 4.00 (bs, 1H), 5.87 (d, $J = 6.8$ Hz, 1H), 6.02 (s, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 212.7, 201.4, 156.3, 136.5, 128.3, 124.9, 73.1, 72.9, 56.9, 44.7, 44.2, 41.1, 32.0, 25.9, 22.4, 21.5; EIMS m/z (rel intensity) 276 (M^+ , 69), 216 (13), 215 (52), 188 (35), 187 (100), 159 (65), 122 (43).

6,12-Di-tert-butyl-3,10-dihydroxy-3,10-dimethyltricyclo[6.2.2.0^{2,7}]dodeca-5,11-diene-4,9-dione (9f). The SIBX-mediated HPD of 5-tert-butyl-2-methylphenol **7f** (70 mg, 0.43 mmol) was run according to the general procedure B. Purification by column chromatography, eluting with hexanes/EtOAc from (4:1) to (1:1), furnished dimer **9f** (30 mg, 39%) as a colourless solid.^{9b}

3,10-Dihydroxy-6,12-di-isopropyl-3,10-dimethyl-tricyclo[6.2.2.0^{2,7}]dodeca-5,11-diene-4,9-dione (9g) and 2,3-dihydroxy-4-iso-propyl-1-methylbenzene (10g). Carcavrol (**7g**) (1.5 g, 10 mmol) was submitted to the SIBX-mediated HPD reaction according to the general procedure B. Purification by column chromatography, eluting with hexanes/ Et_2O (1:1), afforded 2,3-dihydroxy-4-iso-propyl-1-methylbenzene (**10g**)²⁵ as a brown oil (398 mg, 24%) and dimer **9g** as a yellow solid (930 mg). This solid was crystallized from a mixture of CH_2Cl_2 and hexanes to give pure **9g** as white

needles (795 mg, 48%). **10g**: $R_f = 0.4$ [hexanes/Et₂O, (1:1)]; IR (NaCl) 3453, 2963, 1467 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz) δ 1.26 (d, $J = 6.8$ Hz, 6H), 2.24 (s, 3H), 3.17 (sept., $J = 6.8$ Hz, 1H), 5.31 (bs, 2H), 6.69 (d, $J = 7.9$ Hz, 1H), 6.73 (d, $J = 8.1$ Hz, 1H); ¹³C NMR (CDCl₃, 75 MHz) δ 141.5, 141.0, 132.5, 121.9, 121.3, 117.4, 27.1, 22.6, 15.3; EIMS m/z (rel intensity) 166 (M^+ , 92), 151 (100), 133 (56), 105 (60). Colourless prisms were also obtained from slow evaporation of a solution of **9g** in CHCl₃. These prisms were used for X-ray analysis, and the resulting crystal structure was identical to that previously reported for this natural product (CCDC/CSD VATBOH).^{16a} **9g**: mp 136 °C (lit.^{16c} 138–139 °C); $R_f = 0.15$ [hexanes/Et₂O, (1:1)]; IR (NaCl, neat) 3451, 2967, 1724, 1676, 1157 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz) δ 0.84 (d, $J = 6.8$ Hz, 3H), 0.88 (d, $J = 6.6$ Hz, 3H), 1.11 (d, $J = 7.0$ Hz, 3H), 1.12 (d, $J = 6.6$ Hz, 3H), 1.22 (s, 3H), 1.24 (s, 3H), 1.83 (sept., $J = 6.8$ Hz, 1H), 2.48 (sept., $J = 6.8$ Hz, 1H), 3.12 (dd, $J = 2.5, 8.7$ Hz, 1H), 3.13–3.17 (m, 1H), 3.23 (dd, $J = 1.9, 8.7$ Hz, 1H), 3.35 (dq, $J = 1.0, 2.4, 6.8$ Hz, 1H), 5.84 (d, $J = 6.8$ Hz, 1H), 5.96 (s, 1H); ¹³C NMR (CDCl₃, 100 MHz) δ 212.3, 201.9, 166.5, 145.6, 126.1, 119.9, 73.4, 72.8, 55.8, 44.6, 41.9, 40.8, 33.2, 32.8, 32.2, 25.8, 22.9, 20.7, 20.0, 19.2; EIMS m/z (rel intensity) 332 (M^+ , 31), 315 (16), 289 (18), 271 (40), 243 (48), 166 (42), 149 (61), 125 (100).

3,10-Dihydroxy-3,10-di-isopropyl-6,12-dimethyl-tricyclo[6.2.2.0^{2,7}]dodeca-5,11-diene-4,9-dione (9h). Thymol (**7h**) (1.5 g, 10 mmol) was submitted to the SIBX-mediated HPD reaction according to the general procedure B. Purification by column chromatography, eluting with hexanes/Et₂O (1:1), furnished **10g**²⁵ as a brown oil (229 mg, 14%) and dimer **9h** as a yellow solid (970 mg). This solid was crystallized from a mixture of CH₂Cl₂ and hexanes to give pure **9h** as white needles (830 mg, 50%). Colourless prisms were also obtained from slow evaporation of a solution of **9h** in CHCl₃. These prisms were used for X-ray analysis. The resulting crystal data were deposited at the Cambridge Crystallographic Data Centre under the number CCDC 640075. **9h**: mp 159.5 °C (lit.¹⁷ mp 168–170 °C); $R_f = 0.27$ [hexanes/Et₂O, (1:1)]; IR (NaCl, neat) 3481, 2972, 2927, 1719, 1681, 1448, 1361 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 0.56 (d, $J = 6.6$ Hz, 3H), 0.81 (d, $J = 6.8$ Hz, 3H), 0.84 (d, $J = 7.1$ Hz, 3H), 0.95 (d, $J = 6.6$ Hz, 3H), 1.50–1.65 (m, 1H), 1.60 (s, 3H), 1.76 (sept., $J = 6.8$ Hz, 1H), 1.95 (s, 3H), 2.36 (bs, 1H), 3.08 (d, $J = 7.3$ Hz, 1H), 3.16 (s, 1H), 3.26 (d, $J = 8.3$ Hz, 1H), 3.29 (d, $J = 6.6$ Hz, 1H), 3.79 (s, 1H), 5.82 (d, $J = 6.1$ Hz, 1H), 5.97 (s, 1H); ¹³C NMR (CDCl₃, 100 MHz) δ 214.8, 201.8, 155.9, 135.7, 126.6, 125.3, 78.1, 77.8, 57.2, 47.1, 41.8, 37.2, 37.2, 32.5, 22.1, 21.3, 16.7, 16.6, 16.3, 16.0; ESIMS (MeOH) m/z (rel intensity) 333 (MH^+ , 100).

2-Hydroxy-2-methylnaphthalen-1(2H)-one (12). *Method A*. To a solution of 2-methylnaphthol (**11a**) (100 mg, 0.63 mmol) in THF (1.5 mL) was added SIBX (0.394 g, 0.69 mmol) as a solid in one portion. The resulting suspension was stirred at room temperature for 14h, after which time the

reaction mixture was poured into ice-cold water (5 mL), neutralized with aqueous 1 M NaOH, and extracted with CH₂Cl₂ (2 × 5 mL). The combined organic phases were then washed with water (10 mL), dried over Na₂SO₄, filtered and evaporated to furnish orthoquinol **12**²⁶ (110 mg, 100%) as a red oil. *Method B.* Treatment of the trimethylsilylated 2-methylnaphthol **11b** (50 mg, 0.2 mmol) in DMSO (1 mL) with SIBX (255 mg, 0.5 mmol, i.e., 2.5 equiv of IBX) was run at room temperature for 2 h, after which time the reaction mixture was diluted with EtOAc (20 mL), washed with saturated aqueous NaHCO₃ (3 × 5 mL), and brine (5 mL), dried over Na₂SO₄, filtered and evaporated to furnish orthoquinol **12** (35 mg, 100%) as a red oil. IR (NaCl, neat) 3460, 2881, 1686, 1599, 1291 cm⁻¹; ¹H NMR (CDCl₃, 250 MHz) δ 1.43 (s, 3H), 6.30 (d, *J* = 9.8 Hz, 1H), 6.44 (d, *J* = 9.8 Hz, 1H), 7.17 (d, *J* = 7.6 Hz, 1H), 7.31 (dt, *J* = 1.2, 7.6 Hz, 1H), 7.55 (dt, *J* = 1.2, 7.6 Hz, 1H), 7.95 (d, *J* = 7.9 Hz, 1H); ¹³C NMR (CDCl₃, 62.9 MHz) δ 204.1, 137.6, 136.9, 135.0, 128.0, 127.9, 127.3, 127.0, 124.6, 75.2, 28.2; EIMS *m/z* (rel. intensity) 174 (M⁺, 1) 131 (7), 43 (29), 28 (100).

1,2-Naphthoquinone (see Scheme 1). To a solution of 1-naphthol (100 mg, 0.70 mmol) in THF (15 mL) was added SIBX (435 mg, 0.76 mmol) as a solid in one portion. The resulting suspension was stirred at room temperature in the dark for 14h, after which time water (30 mL) was added. This mixture was then extracted with CH₂Cl₂ (2 × 15 mL), and the combined organic phases were washed with saturated aqueous NaHCO₃ (6 × 15 mL) and water (20 mL), dried over Na₂SO₄, filtrated and evaporated to give 1,2-naphthoquinone (108 mg, 99%) as a red solid.^{8a,27}

Scheme 1. Plausible Mechanism of the SIBX-Mediated Oxidation of 1-Naphthol into 1,2-Naphthoquinone

Scheme 2. Preparation of Functionalized Naphthols **11d-e**

The epoxide **11d** was generated in three steps from 1-naphthol (Scheme 2). After *O*-allylation of 1-naphthol with 2-methylallyl chloride and Claisen rearrangement in refluxing DMF,²⁸ the resulting 2-(2-methylallyl)-naphthalen-1-ol (**16a**) was treated with *m*-CPBA in CH₂Cl₂ to furnish **11d** in 62% yield. Similarly, the *O*-allylation of 1-naphthol with allyl chloride, followed by a Claisen rearrangement, gave 2-allylnaphthalen-1-ol (**16b**) in 90% yield (Scheme 2).²⁸ This allylated naphthol was first protected as a triethylsilyloxy derivative and then converted to the epoxide **17** by treatment with *m*-CPBA in CH₂Cl₂. Opening of this epoxide with the Nagata's reagent in toluene gave a mixture of triethylsilylated naphtholic and free naphtholic β-hydroxynitriles **18a** and **18b** in 43% and 32%, respectively. Hence, the tertiary alcohol of the β-hydroxynitrile **18a** was protected as a *tert*-butyldimethylsilyl ether, and the triethylsilyloxy group was selectively cleaved using acetic acid to furnish the naphthol **11e** in 43% over the two steps (Scheme 2).

2-[(2-methyloxiran-2-yl)methyl]naphthalen-1-ol (11d). To a solution of 2-(2-methylallyl)-naphthol (**16b**)²⁸ (200 mg, 1.0 mmol) in CH₂Cl₂ (10 mL) at 0 °C was slowly added *m*-CPBA (373 mg, 1.5 mmol). This reaction mixture was stirred at 0 °C for 6 h, after which time it was carefully

quenched with saturated aqueous Na_2SO_3 (10 mL) and extracted with CH_2Cl_2 (3×20 mL). The combined organic phases were washed with saturated aqueous NaHCO_3 (4×30 mL), brine (20 mL), dried over Na_2SO_4 , and evaporated to give a residue, which was purified by column chromatography, eluting with cyclohexane/ Et_2O (3:1), to afford **11d** (133 mg, 62%) as a pale yellow oil: IR (NaCl, neat) 3308, 1297 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 1.38 (s, 3H), 2.87 (d, $J = 3.6$ Hz, 1H), 2.88 (d, $J = 15.6$ Hz, 1H), 2.96 (d, $J = 3.9$ Hz, 1H), 3.28 (d, $J = 14.9$ Hz, 1H), 7.18 (d, $J = 8.3$ Hz, 1H), 7.40 (d, $J = 8.1$ Hz, 1H), 7.49-7.52 (m, 2H), 7.80 (d, $J = 7.2$ Hz, 1H), 8.28 (s, 1H), 8.39 (d, $J = 9.0$ Hz, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 151.4, 134.1, 129.4, 127.1, 125.9, 125.6, 125.0, 122.4, 119.5, 116.0, 60.0, 55.6, 39.5, 20.0; EIMS m/z (rel. intensity) 214 (88), 156 (100); Anal. calcd for $\text{C}_{14}\text{H}_{14}\text{O}_2$: C, 78.48; H, 6.59. Found: C, 78.20; H, 6.50.

2-(Oxiran-2-yl)methyl-1-triethylsilyloxynaphthalene (17). Triethylamine (6.3 mL, 45.3 mmol) was added dropwise to a stirred ice-cold solution of 2-allylnaphthol (**16a**)²⁸ (6.4 g, 34.8 mmol) in CH_2Cl_2 (200 mL). This mixture was then stirred for 5 min, after which time Et_3SiCl (6.4 mL, 38.3 mmol) was slowly added at 0 °C. After stirring for 12 h at this temperature, the reaction mixture was diluted with CH_2Cl_2 (80 mL), washed with aqueous 1 M H_3PO_4 (2×20 mL), brine (2×20 mL), dried over Na_2SO_4 , and evaporated to give 2-allyl-1-triethylsilyloxynaphthalene (10.2 g, 98%) as a reddish oil: IR (NaCl, neat) 2956, 2917, 2877 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 0.92-0.97 (q, $J = 7.8$ Hz, 6H), 1.05-1.11 (q, $J = 8.1$ Hz, 9H), 3.67 (d, $J = 6.4$ Hz, 2H), 5.21-5.25 (m, 2H), 6.07-6.21 (m, 1H), 7.38 (d, $J = 8.6$ Hz, 1H), 7.48-7.57 (m, 3H), 7.85 (d, $J = 8.1$ Hz, 1H), 8.18 (d, $J = 8.1$ Hz, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 149.3, 137.4, 134.2, 128.7, 128.6, 128.1, 125.7, 125.5, 124.9, 122.9, 121.9, 116.3, 34.9, 7.3, 6.2; EIMS m/z (rel. intensity) 298 (M^+ , 100), 217 (93); HMRS (EI) calcd for $\text{C}_{19}\text{H}_{26}\text{OSi}$ 298.1753, found 298.1741.

This silylated allylnaphthol (5.0 g, 16.8 mmol) was dissolved into CH_2Cl_2 (150 mL). To the resulting ice-cooled solution was added slowly *m*-CPBA (6.2 g, 25.1 mmol). This reaction mixture was stirred at 0 °C for 3 days, after which time it was carefully quenched with a saturated aqueous Na_2SO_3 (20 mL) and separated. The aqueous phase was extracted with CH_2Cl_2 (3×50 mL), and the combined organic phases were washed with saturated aqueous NaHCO_3 (4×40 mL) and brine (30 mL), dried over Na_2SO_4 , and evaporated to give a residue, which was purified by column chromatography, eluting with cyclohexane/ Et_2O (10:1), to furnish **17** (1.1 g, 20%) as a yellow oil: IR (NaCl, neat) 2957, 2913, 2877 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 0.82-0.86 (m, 6 H), 0.96-1.00 (m, 9H), 2.61 (dd, $J = 4.9, 2.5$ Hz, 1H), 2.80 (dd, $J = 4.6, 4.2$ Hz, 1H), 2.95 (dd, $J = 14.4, 5.4$ Hz, 1H), 3.16 (dd, $J = 14.4, 5.4$ Hz, 1H), 3.24 (m, 1H), 7.37-7.49 (m, 4H), 7.79 (d, $J = 7.4$ Hz, 1H), 8.07 (d, $J = 7.6$ Hz, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 149.4, 133.9, 128.2, 128.1, 127.7, 125.5, 125.2,

122.5, 122.1, 121.6, 51.9, 47.0, 33.4, 6.8, 5.7; EIMS m/z (rel. intensity) 314 (M^+ , 85), 285 (36), 257 (89), 227 (92); HMRS (EI) calcd for $C_{19}H_{26}O_2Si$ 314.1702, found 314.1711.

2-[(3-cyano-2-hydroxy)propyl]-1-triethylsilyloxynaphthalene (18a) and 2-[(3-cyano-2-hydroxy)propyl]naphthalen-1-ol (18b). Et_2AlCN (1.0 M in toluene, 8.6 mL, 8.6 mmol) was added dropwise to a solution of **17** (675 mg, 2.1 mmol) in toluene (20 mL) at $-40\text{ }^{\circ}C$. This reaction mixture was stirred at this temperature for 40 min, after which time it was quenched with aqueous 1 M HCl solution (1 mL). The resulting mixture was stirred at rt for 40 min and separated. The aqueous phase was extracted with EtOAc ($2 \times 15\text{ mL}$), and the combined organic phases were washed with aqueous 1 M HCl ($2 \times 10\text{ mL}$) and brine (20 mL), dried over Na_2SO_4 , and evaporated to give a residue, which was purified by column chromatography, eluting with cyclohexane/ Et_2O (2:1), to furnish **18a** (311 mg, 43 %) and **18b** (156 mg, 32%) as pale yellow oils. **18a**: IR (NaCl, neat) 2926, 2252 cm^{-1} ; 1H NMR ($CDCl_3$, 400 MHz) δ 0.89-0.96 (m, 6H), 1.03-1.07 (m, 9H), 2.52-2.63 (m, 2H), 2.83 (s, 1H), 3.13-3.25 (m, 2H), 4.30-4.36 (m, 1H), 7.35 (d, 1H), 7.52-7.58 (m, 3H), 7.86-7.89 (m, 1H), 8.10-8.13 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 150.0, 134.2, 128.6, 128.0, 127.8, 126.0, 125.5, 122.4, 122.3, 121.5, 117.6, 68.3, 38.2, 25.2, 6.7, 5.7; EIMS m/z (rel. intensity) 341 (M^+ , 77), 312 (66), 271 (96); HMRS (EI) calcd for $C_{20}H_{27}NO_2Si$ 341.1811, found 341.1800. **18b**: IR (NaCl, neat) 3372, 2952, 2257 cm^{-1} ; 1H NMR ($CDCl_3$, 400 MHz) δ 2.49 (d, $J = 6.1\text{ Hz}$, 2H), 2.96-3.08 (m, 2H), 4.13 (s, 1H), 4.32-4.37 (m, 1H), 7.10 (d, $J = 8.3\text{ Hz}$, 1H), 7.37 (d, $J = 8.1\text{ Hz}$, 1H), 7.45-7.47 (m, 2H), 7.75-7.78 (m, 1H), 8.23-8.25 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 151.0, 134.1, 129.2, 127.4, 126.2, 125.6, 125.4, 122.1, 120.1, 117.6, 116.2, 69.5, 38.2, 25.2; EIMS m/z (rel. intensity) 227 (M^+ , 100), 210 (87) HMRS (EI) calcd for $C_{13}H_{13}NO_2$ 227.0946, found 227.0955.

2-[(2-tert-butyldimethylsilyloxy-3-cyano)propyl]naphthalen-1-ol (11e). TBDMSOTf (143 μL , 0.62 mmol) was added to a mixture of **18a** (74 mg, 0.2 mmol) and 2,6-lutidine (73 μL , 0.62 mmol) in CH_2Cl_2 (4 mL) at $0\text{ }^{\circ}C$. After stirring for 3 h at this temperature, saturated aqueous $NaHCO_3$ (5 mL) was added to the reaction mixture, which was then separated. The aqueous phase was extracted with Et_2O (20 mL), and the combined organic phases were washed with brine (20 mL), dried over Na_2SO_4 , and evaporated to furnish the desired silylated derivative (98 mg), which was used in the next step without purification: IR (NaCl, neat) 2956, 2930, 2880, 2957, 2349 cm^{-1} ; 1H NMR ($CDCl_3$, 400 MHz) δ -0.14 (s, 3H), 0.08 (s, 3H), 0.82-0.89 (m, 18H), 0.93-0.99 (m, 6H), 2.35-2.48 (m, 2H), 3.00-3.11 (m, 2H), 4.21-4.26 (m, 1H), 7.26 (d, $J = 8.3\text{ Hz}$, 1H), 7.44-7.47 (m, 3H), 7.78 (d, $J = 7.1\text{ Hz}$, 1H), 8.04 (d, $J = 8.6\text{ Hz}$, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 149.8, 134.0, 129.0, 128.1, 127.7, 125.7, 125.2, 122.5, 122.0, 121.6, 118.0, 68.9, 38.7, 25.7, 25.6, 17.9, 6.7, 5.7, -3.0, -4.9; HMRS (EI) calcd for $C_{26}H_{41}NO_2Si_2$ 455.2676, found 455.2654.

This disilylated compound (200 mg from two runs, 0.44 mmol) was then dissolved in THF/AcOH/H₂O/EtOH (1:6:3:2, 22 mL). The resulting mixture was stirred at rt for 4 days, after which time it was extracted with Et₂O (20 mL), washed with saturated aqueous NaHCO₃ (20 mL), dried over Na₂SO₄ and evaporated to give a residue, which was purified by column chromatography, eluting with cyclohexane/Et₂O (3:1), to furnish **11e** (64 mg, 43%) as a pale yellow oil: IR (NaCl, neat) 3289, 2955, 2929, 2857, 2252 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 0.07 (s, 3H), 0.18 (s, 3H), 0.98 (s, 9H), 1.44 (s, 1H), 2.46 (s, 2H), 3.13-3.24 (m, 2H), 4.45 (s, 1H), 7.18 (d, *J* = 8.0 Hz, 1H), 7.37-7.40 (m, 1H), 7.47 (d, 4.8 Hz, 1H), 7.77-7.78 (m, 1H), 7.94 (d, *J* = 2.7 Hz, 1H), 8.27 (d, *J* = 4.8 Hz, 1H); ¹³C NMR (CDCl₃, 100 MHz) δ 151.1, 134.2, 129.4, 127.3, 126.1, 125.4, 125.2, 122.2, 120.0, 116.9, 115.2, 70.4, 39.1, 25.7, 24.7, 18.0, -4.9; HMRS (EI) calcd for C₂₀H₂₇NO₂Si 341.1811, found 341.1812.

Additional references

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- (26) Krohn, K.; Brüggmann, K.; Doering, D.; Jones, P. G. *Chem. Ber.* **1992**, *125*, 2439-2442.
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CCDC 640072

9b

CCDC 640073

9c

CCDC 640074

9e

CCDC 640075

9h

Table 3. Influence of an acidic medium on the IBX-mediated HPD of 2,6-dimethylphenol (**7a**)

entry ^a	acid	reaction time	IBA-bearing dimer	free dimer (9a)
1	SIBX	12 h		✓
2	IBX	2 days	✓	
3	IBX + benzoic acid (3 equiv)	2 days	✓	
4	IBX + isophthalic acid (3 equiv)	2 days	✓	
5	IBX + benzoic (22%) and isophthalic (29%) acids (i.e., SIBX formulation)	2 days	✓	
6	IBX + TFA (3 equiv)	1 h		✓
7	IBX, then TFA (1 equiv) ^b	2 days		✓
8	IBX + TFA (0.1 equiv)	12 h		✓
9	IBX + CSA (0.1 equiv)	12 h		✓
10	IBX + HCl conc. (0.1 equiv)	12 h		✓

^a All reactions were run on 2,6-dimethylphenol (**7a**) (100 mg, 0.82 mmol) in THF (2 mL) at rt using IBX (1.0 equiv), except the reference reaction (entry 1) for which SIBX (1.1 equiv) was used. ^b TFA was added after running the reaction for 2 days; the reaction mixture was then stirred at rt 1 h. Below are examples of ¹H NMR spectra of crude reaction mixtures before (top, IBA-bearing dimer) and after (bottom, free dimer) treatment with TFA:

JGD48-1 - DPX400 - 091005 -

JGD48-1 - 300MHz - 260406 -

Peak List

File Name : c:\docume~1\julien\bureau\jge048~1\1\pdata\1\1R.1R
 Peak Results saved in File : C:\WIN1D\TMP\EDIT.ASC
 Nucleus : off
 SF : 300.130006 MHz
 OFFSET : 16.4635 ppm
 SW_p : 6188.12 Hz
 SI : 32768

9a

Peak Picking Parameter

Peak constant PC = 1.00
 Noise = 105885
 Sens. level = 423542

Peak Picking region

Start (ppm/Hz)	End (ppm/Hz)	MI (%)	MAXI (%)
5.86/ 1758.8	5.32/ 1597.7	6.09	23.96
4.43/ 1330.5	3.86/ 1159.6	27.17	50.86
3.48/ 1045.7	3.15/ 945.4	4.28	24.56
2.39/ 717.7	2.14/ 642.9	16.12	25.56
1.99/ 595.8	1.80/ 538.8	60.10	67.73
1.57/ 472.0	1.16/ 348.3	97.04	102.86
1.67/ 501.4	1.57/ 472.0	19.54	26.97
8.38/ 2515.7	6.61/ 1985.0	48.42	83.45

Peak Picking results

Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	14627	2178.94	7.2600	167867168	54.3
2	17387	1657.73	5.5234	27754644	9.0
3	17430	1649.61	5.4963	26035728	8.4
4	19800	1202.04	4.0051	90750840	29.3
5	20770	1018.86	3.3947	26100242	8.4
6	20806	1012.06	3.3721	26257764	8.5
7	20978	979.58	3.2639	23403428	7.6
8	20987	977.88	3.2582	23146184	7.5
9	21022	971.27	3.2362	27259416	8.8
10	21031	969.57	3.2305	26779684	8.7
11	22463	699.14	2.3295	51884976	16.8
12	23224	555.43	1.8506	187980608	60.8
13	23622	480.27	1.6002	63490100	20.5
14	24031	403.03	1.3429	305954624	98.9
15	24072	395.29	1.3171	309223520	100.0
16	24196	371.87	1.2390	300153600	97.1

JGD47-1 - DPX400 - 251005 -

JGD47-1 - 300MHz - 260406 -

Peak List

File Name : c:\docume~1\julien\mesdoc~1\b\jgd47-
 1\1\pdata\1\1R.1R
 Peak Results saved in File : C:\WIN1D\TMP\EDIT.ASC
 Nucleus : off
 SF : 300.130006 MHz
 OFFSET : 16.4635 ppm
 SW_p : 6188.12 Hz
 SI : 32768

9b

Peak Picking Parameter

Peak constant PC = 1.00
 Noise = 77493
 Sens. level = 309974
 Peak Picking region

Start (ppm/Hz)	End (ppm/Hz)	MI (%)	MAXI (%)
1.84/ 552.6	1.80/ 539.8	45.64	53.12
1.72/ 516.7	1.66/ 497.3	45.13	55.33
1.63/ 489.3	1.61/ 483.3	31.86	33.56
1.38/ 415.5	1.12/ 334.7	80.00	91.40
1.33/ 400.0	1.21/ 361.7	98.42	119.85
2.25/ 675.9	2.20/ 659.5	9.28	24.93
3.45/ 1034.9	2.76/ 828.3	4.66	18.73
6.16/ 1849.6	5.87/ 1760.5	10.27	15.60
7.63/ 2291.1	7.09/ 2129.1	26.46	31.97

Peak Picking results

Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	14627	2178.94	7.2600	92131568	26.7
2	16587	1808.81	6.0267	36251212	10.5
3	18161	1511.56	5.0364	29690160	8.6
4	19933	1176.93	3.9214	89203896	25.9
5	21160	945.21	3.1493	22372652	6.5
6	21171	943.13	3.1424	40809868	11.8
7	21182	941.06	3.1355	22326620	6.5
8	21713	840.78	2.8014	39722180	11.5
9	21724	838.70	2.7945	37706848	10.9
10	22625	668.55	2.2275	35303276	10.2
11	23252	550.14	1.8330	157818704	45.8
12	23260	548.63	1.8280	159965696	46.4
13	23453	512.19	1.7065	160686480	46.6
14	23462	510.49	1.7009	157186112	45.6
15	24002	408.51	1.3611	287281632	83.3
16	24196	371.87	1.2390	344749408	100.0
17	24318	348.83	1.1623	291640384	84.6

JGE33-1 - 071106 - DPX300 -

JGE33-1 - DPX300 - 071106 -

Peak List

File Name : d:\rmn\nov06\jge33-1\1\pdata\1\1R.1R
 Peak Results saved in File : C:\WIN1D\TMP\EDIT.ASC
 Nucleus : off
 SF : 300.130006 MHz
 OFFSET : 16.4654 ppm
 SW_p : 6188.12 Hz
 SI : 32768

8c

Peak Picking Parameter

Peak constant PC = 1.00
 Noise = 90929
 Sens. level = 363715

Peak Picking region

Start (ppm/Hz)	End (ppm/Hz)	MI (%)	MAXI (%)
10.20/ 3060.5	5.03/ 1510.0	3.41	26.30
3.77/ 1131.0	-0.13/ -40.4	65.08	115.02

Peak Picking results

Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	14630	2178.94	7.2600	10420506	3.8
2	15485	2017.48	6.7220	23411674	8.5
3	15517	2011.44	6.7019	27573094	10.0
4	16947	1741.39	5.8021	23651942	8.6
5	16979	1735.34	5.7820	26332572	9.6
6	23027	593.20	1.9765	187695456	68.1
7	23161	567.90	1.8922	188485152	68.4
8	24029	403.98	1.3460	275485024	100.0

JGE33-3 - 300MHz -

JGE33-3 - 300MHz - 081206 -

Peak List

File Name : d:\rmn\nmrdpx~1\jge33-3\2\pdata\1\1R.1R
Peak Results saved in File : C:\WIN1D\TMP\EDIT.ASC
Nucleus : off
SF : 300.130006 MHz
OFFSET : 16.4638 ppm
SW_p : 6188.12 Hz
SI : 32768

Peak Picking Parameter

Peak constant PC = 1.00
Noise = 235226
Sens. level = 940903
Peak Picking region

Start (ppm/Hz)	End (ppm/Hz)	MI (%)	MAXI (%)
6.01/ 1804.9	5.78/ 1734.9	6.69	8.46
3.49/ 1046.1	2.77/ 831.1	4.92	9.48
7.46/ 2239.7	6.87/ 2062.3	13.01	19.19
2.27/ 681.9	0.95/ 284.3	45.32	110.59

Peak Picking results

Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	14628	2178.85	7.2597	75113744	13.8
2	16681	1791.15	5.9679	38063856	7.0
3	16713	1785.10	5.9478	39184880	7.2
4	20864	1001.20	3.3359	40378696	7.4
5	20897	994.97	3.3151	41076120	7.5
6	21403	899.41	2.9967	29295308	5.4
7	21443	891.86	2.9716	40775472	7.5
8	21572	867.50	2.8904	41520704	7.6
9	21615	859.38	2.8634	29613826	5.4
10	23014	595.18	1.9831	249466272	45.7
11	23295	542.12	1.8063	280418176	51.4
12	23888	430.13	1.4331	297664288	54.6
13	24224	366.68	1.2217	545501312	100.0
14	24308	350.81	1.1689	267318064	49.0

JGE17-1 - 300MHz - 130406 -

JGE17-1 - 130406 - 300MHz -

Peak List

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File Name           : d:\rmn\avril2~1\jge017-1\2\pdata\1\1R.1R
Peak Results saved in File : C:\WIN1D\TMP\EDIT.ASC
Nucleus             : off
SF                  : 300.130006 MHz
OFFSET             : 16.4642 ppm
SW_p               : 6188.12 Hz
SI                 : 32768

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Peak Picking Parameter

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-----
Peak constant PC =      1.00
Noise            =      27811
Sens. level      =     111245
Peak Picking region
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Start (ppm/Hz)	End (ppm/Hz)	MI (%)	MAXI (%)
7.37/ 2212.0	6.81/ 2044.3	4.66	18.52
7.00/ 2099.4	6.66/ 1997.7	1.84	8.11
5.39/ 1617.1	5.12/ 1538.0	10.27	13.28
5.29/ 1587.1	5.02/ 1505.7	10.24	13.42
5.19/ 1557.8	0.67/ 200.4	16.93	126.84

Peak Picking results

Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	14628	2178.94	7.2600	48860608	15.6
2	15193	2072.25	6.9045	6612144	2.1
3	15199	2071.11	6.9007	17127500	5.5
4	15206	2069.79	6.8963	18126372	5.8
5	15213	2068.47	6.8919	8223451	2.6
6	15231	2065.07	6.8806	7313546	2.3
7	15237	2063.94	6.8768	17744136	5.7
8	15244	2062.61	6.8724	18199820	5.8
9	15251	2061.29	6.8680	7941595	2.5
10	17991	1543.85	5.1439	34983940	11.2
11	18029	1536.68	5.1200	34261948	10.9
12	20184	1129.71	3.7641	313468576	100.0
13	20711	1030.19	3.4325	58054208	18.5
14	23167	566.38	1.8871	145618496	46.5
15	23174	565.06	1.8827	156699872	50.0
16	23851	437.21	1.4567	306735872	97.9

JGE17B-2 - DPX300 - 241106 -

JGE17B-2 - 300MHz - 241106 -

Peak List

```

File Name           : d:\rmn\nov06\jge17b~2\1\pdata\1\1R.1R
Peak Results saved in File : C:\WIN1D\TMP\EDIT.ASC
Nucleus             : off
SF                  : 300.130092 MHz
OFFSET              : 16.1772 ppm
SW_p                : 6188.12 Hz
SI                  : 32768

```

Peak Picking Parameter

```

Peak constant PC =      1.00
Noise            =     151439
Sens. level      =     605754

```

Peak Picking region

Start (ppm/Hz)	End (ppm/Hz)	MI (%)	MAXI (%)
4.81/ 1444.1	0.77/ 231.4	53.25	109.10
3.38/ 1014.3	3.03/ 910.3	8.08	13.79
3.01/ 903.1	2.78/ 834.0	10.87	13.97
4.29/ 1286.8	4.07/ 1220.5	6.07	9.35
2.61/ 783.4	2.41/ 722.7	6.86	12.39
2.31/ 692.5	1.99/ 598.7	7.53	21.01
8.07/ 2421.2	6.97/ 2090.8	23.80	46.64

Peak Picking results

Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	14172	2178.94	7.2600	76209064	25.7
2	19180	1233.20	4.1089	19068232	6.4
3	19718	1131.60	3.7704	234802512	79.1
4	20618	961.64	3.2041	24944178	8.4
5	20774	932.18	3.1059	30768208	10.4
6	21152	860.80	2.8681	33345568	11.2
7	21733	751.08	2.5025	21251498	7.2
8	21840	730.87	2.4352	28756494	9.7
9	22278	648.16	2.1596	57132168	19.2
10	22401	624.93	2.0822	23918726	8.1
11	22756	557.89	1.8588	238028016	80.2
12	23527	412.29	1.3737	296919328	100.0
13	23822	356.58	1.1881	165477232	55.7

JGD52-2 - DPX400 - 050306 -

JGD52-2 - 300MHz - 240406 -

Peak List

```

File Name           : d:\rmn\avril2~1\jgd52-2\2\pdata\1\1R.1R
Peak Results saved in File : C:\WIN1D\TMP\EDIT.ASC
Nucleus             : off
SF                  : 300.130006 MHz
OFFSET              : 16.4642 ppm
SW_p                : 6188.12 Hz
SI                  : 32768

```

Peak Picking Parameter

```

Peak constant PC =      1.00
Noise            =     115425
Sens. level      =     461701

```

Peak Picking region

Start (ppm/Hz)	End (ppm/Hz)	MI (%)	MAXI (%)
3.40/ 1021.7	3.29/ 987.5	8.91	12.16
7.82/ 2348.3	5.56/ 1667.9	6.60	64.41
2.56/ 768.6	2.06/ 618.7	15.63	24.83
2.32/ 695.6	1.45/ 434.2	66.15	76.04
1.72/ 515.0	0.55/ 165.3	92.02	103.83
4.80/ 1441.3	3.79/ 1137.6	28.48	61.12
4.70/ 1410.5	3.78/ 1133.9	27.09	60.77

Peak Picking results

Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	14628	2178.94	7.2600	154825552	53.8
2	16603	1805.97	6.0173	52506044	18.2
3	16822	1764.62	5.8795	24401028	8.5
4	16858	1757.82	5.8569	23834774	8.3
5	19815	1199.40	3.9963	80981936	28.1
6	20868	1000.54	3.3337	30579236	10.6
7	20875	999.22	3.3293	30421046	10.6
8	20904	993.74	3.3110	29702136	10.3
9	20911	992.42	3.3066	28719682	10.0
10	22548	683.28	2.2766	47104736	16.4
11	22979	601.89	2.0054	198407904	68.9
12	22984	600.94	2.0023	196714720	68.3
13	23596	485.37	1.6172	195402176	67.9
14	23603	484.05	1.6128	192196544	66.8
15	24093	391.51	1.3045	278892128	96.9
16	24182	374.71	1.2485	287810528	100.0

AOA44 - jv171f.105 - 250MHz -

AOA44/CDCI3/popo

Peak List

 File Name : d:\nmr\aoa\aoa44\17010318\001001.1R
 Peak Results saved in File : c:\WIN1D\TMP\EDIT.ASC
 Nucleus : off
 SF : 300.130003 MHz
 OFFSET : 19.7730 ppm
 SW_p : 6172.84 Hz
 SI : 16384
 Peak Picking Parameter

 Peak constant PC = 1.00
 Noise = 10716
 Sens. level = 42862
 Peak Picking region

Start(ppm/Hz)	End(ppm/Hz)	MI(%)	MAXI(%)
2.11/ 634.2	0.68/ 205.5	84.14	126.68
2.66/ 797.7	2.42/ 725.4	0.66	11.36
4.19/ 1258.5	3.02/ 907.4	9.08	67.54
3.06/ 919.0	2.98/ 894.9	2.37	3.70
3.48/ 1043.8	3.41/ 1024.5	6.77	9.63
3.41/ 1023.4	3.39/ 1016.6	2.98	3.84
3.39/ 1018.5	3.32/ 997.8	3.99	5.26
4.70/ 1411.5	3.92/ 1177.5	6.15	17.71
1.23/ 367.8	1.21/ 363.3	33.13	42.76

Peak Picking results

Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	10915	1822.14	6.0712	36992452	11.2
2	10948	1809.71	6.0297	11953892	3.6
3	10967	1802.55	6.0059	11130506	3.4
4	12479	1232.89	4.1078	28641400	8.7
5	13022	1028.31	3.4262	23170818	7.0
6	13039	1021.90	3.4049	10918567	3.3
7	13050	1017.76	3.3911	10541285	3.2
8	13064	1012.48	3.3735	13641368	4.1
9	13088	1003.44	3.3433	14369223	4.4
10	13336	910.00	3.0320	10556803	3.2
11	13344	906.99	3.0220	9993312	3.0
12	13360	900.96	3.0019	8991985	2.7
13	13368	897.95	2.9919	8278457	2.5
14	13746	755.53	2.5173	4196589	1.3
15	14763	372.37	1.2407	329254848	100.0
16	14781	365.58	1.2181	115452784	35.1
17	15060	260.47	0.8679	286738240	87.1

JGC266-4 - DPX400 - 050805 -

JGE13A-2-recrist - 300MHz - 220406 -

Peak List

File Name : d:\rmn\jge013~1\2\pdata\1\1R.1R
 Peak Results saved in File : C:\WIN1D\TMP\EDIT.ASC
 Nucleus : off
 SF : 300.130006 MHz
 OFFSET : 16.4635 ppm
 SW_p : 6188.12 Hz
 SI : 32768

Peak Picking Parameter

Peak constant PC = 1.00
 Noise = 99902
 Sens. level = 399608

Peak Picking region

Start (ppm/Hz)	End (ppm/Hz)	MI (%)	MAXI (%)
2.59/ 776.0	2.35/ 706.7	0.61	7.54
1.90/ 570.9	1.87/ 560.2	0.16	2.21
1.78/ 533.3	1.75/ 526.5	0.22	1.08
1.79/ 537.3	1.77/ 532.6	1.81	2.82
1.82/ 545.6	1.81/ 541.8	4.07	4.48
1.84/ 551.5	1.83/ 547.9	5.31	5.88
1.86/ 557.1	1.85/ 555.2	3.48	5.04
1.77/ 530.9	1.76/ 526.9	0.17	0.98
3.11/ 934.6	3.09/ 927.3	5.94	10.41

Peak Picking results

Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	14627	2178.94	7.2600	118798960	37.1
2	16695	1788.41	5.9588	69934056	21.8
3	16862	1756.87	5.8537	25119614	7.8
4	16898	1750.07	5.8311	26192540	8.2
5	20816	1010.17	3.3658	18879232	5.9
6	20821	1009.23	3.3626	25221260	7.9
7	20828	1007.91	3.3582	22982432	7.2
8	20834	1006.77	3.3545	26291602	8.2
9	20852	1003.38	3.3431	20041456	6.3
10	20857	1002.43	3.3400	25194152	7.9
11	20865	1000.92	3.3350	23107572	7.2
12	20870	999.98	3.3318	26338258	8.2
13	21002	975.05	3.2488	18611726	5.8
14	21012	973.16	3.2425	22176124	6.9
15	21048	966.36	3.2198	30863660	9.6
16	21058	964.47	3.2135	37362020	11.7
17	21181	941.24	3.1361	41119756	12.8
18	21194	938.79	3.1279	36483872	11.4
19	21227	932.56	3.1072	22358040	7.0
20	21240	930.10	3.0990	22000848	6.9
21	22119	764.11	2.5459	2412906	0.8
22	22154	757.50	2.5239	6916284	2.2
23	22190	750.70	2.5012	15946376	5.0
24	22226	743.90	2.4786	21626308	6.8

25	22261	737.29	2.4566	16956178	5.3
26	22297	730.49	2.4339	7522006	2.3
27	22333	723.69	2.4113	1976065	0.6
28	23149	569.60	1.8978	683631	0.2
29	23185	562.80	1.8752	4528644	1.4
30	23221	556.00	1.8525	12173032	3.8
31	23257	549.20	1.8299	17320846	5.4
32	23293	542.40	1.8072	13733128	4.3
33	23329	535.60	1.7846	5845228	1.8
34	23365	528.80	1.7619	1077140	0.3
35	24197	371.68	1.2384	284078656	88.7
36	24218	367.72	1.2252	320151872	100.0
37	24359	341.09	1.1365	127368280	39.8
38	24381	336.94	1.1226	149856192	46.8
39	24394	334.48	1.1145	135602400	42.4
40	24418	329.95	1.0994	134062128	41.9
41	24743	268.57	0.8949	129483536	40.4
42	24778	261.96	0.8728	125000136	39.0
43	24819	254.22	0.8470	139360176	43.5
44	24855	247.42	0.8244	130770768	40.8

JGE15-1-1 - 260406 - 300MHz -

JGE15-1-1 - 260406 - 300MHz -

Peak List

File Name : d:\rmn\avril2~1\jge015~1\1\pdata\1\1R.1R
 Peak Results saved in File : C:\WIN1D\TMP\EDIT.ASC
 Nucleus : off
 SF : 300.130006 MHz
 OFFSET : 16.4642 ppm
 SW_p : 6188.12 Hz
 SI : 32768

Peak Picking Parameter

Peak constant PC = 1.00
 Noise = 60060
 Sens. level = 240242

Peak Picking region

Start (ppm/Hz)	End (ppm/Hz)	MI (%)	MAXI (%)
8.84/ 2653.5	6.38/ 1916.3	2.53	31.67
5.70/ 1709.5	4.92/ 1475.3	1.32	5.83
3.35/ 1006.0	3.07/ 921.2	0.28	7.86
3.33/ 998.8	3.00/ 901.4	-0.07	7.56
3.25/ 976.4	3.23/ 969.2	0.39	1.95
3.11/ 934.4	3.10/ 930.1	0.22	0.90
3.11/ 932.0	3.10/ 929.7	0.14	1.25
2.39/ 716.7	2.09/ 627.9	52.57	70.27
1.39/ 416.6	1.14/ 341.3	94.70	107.78

Peak Picking results

Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	14628	2178.94	7.2600	18604374	6.3
2	15445	2024.66	6.7459	13122386	4.4
3	15488	2016.54	6.7189	65008652	22.0
4	15507	2012.95	6.7069	60433952	20.5
5	15549	2005.02	6.6805	11988676	4.1
6	17730	1593.14	5.3082	4671816	1.6
7	21020	971.84	3.2381	1480282	0.5
8	21055	965.23	3.2160	5847721	2.0
9	21091	958.43	3.1934	13519367	4.6
10	21128	951.44	3.1701	17922724	6.1
11	21164	944.64	3.1474	13720642	4.6
12	21200	937.85	3.1248	5821022	2.0
13	21235	931.24	3.1028	1234400	0.4
14	22598	673.84	2.2452	172747296	58.5
15	24134	383.77	1.2787	295080352	100.0
16	24170	376.97	1.2560	283679264	96.1

JGC266-2 - DPX400 - 200306 -

JGC266-2 - DPX400 - 200306 -

Peak List

File Name : d:\rmn\mars20~1\jgc266-2\1\pdata\1\1R.1R
 Peak Results saved in File : C:\WIN1D\TMP\EDIT.ASC
 Nucleus : off
 SF : 400.130018 MHz
 OFFSET : 17.9688 ppm
 SW_p : 8012.82 Hz
 SI : 32768

Peak Picking Parameter

Peak constant PC = 1.00
 Noise = 466561
 Sens. level = 1866243
 Peak Picking region

Start (ppm/Hz)		End (ppm/Hz)		MI (%)	MAXI (%)
1.82/	726.4	1.81/	725.4	-0.04	1.40
1.72/	686.8	1.71/	685.6	-0.37	2.31
1.72/	687.5	1.71/	685.1	0.41	2.43
1.72/	687.5	1.71/	683.4	-0.94	2.43
1.72/	686.3	1.71/	683.4	0.09	2.59
1.80/	719.8	1.73/	691.0	2.27	12.71
2.16/	864.6	1.49/	595.3	87.71	102.30
1.05/	421.0	0.41/	163.3	42.73	81.53
8.71/	3486.9	5.55/	2219.8	8.45	41.24

Peak Picking results

Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	17523	2904.94	7.2600	41002096	11.2
2	19626	2390.69	5.9748	100410904	27.4
3	19867	2331.76	5.8275	46729700	12.8
4	19892	2325.65	5.8122	48710092	13.3
5	23204	1515.76	3.7882	106926824	29.2
6	24006	1319.64	3.2980	55805432	15.2
7	24034	1312.80	3.2809	59639316	16.3
8	24060	1306.44	3.2650	55540808	15.2
9	24094	1298.12	3.2443	62196256	17.0
10	24232	1264.38	3.1599	88022480	24.0
11	24348	1236.01	3.0890	49771492	13.6
12	24379	1228.43	3.0701	42685428	11.7
13	25544	943.55	2.3581	62504548	17.1
14	26207	781.43	1.9529	366314944	100.0
15	26434	725.92	1.8142	1978485	0.5
16	26461	719.32	1.7977	12396355	3.4
17	26488	712.72	1.7812	30681066	8.4
18	26516	705.87	1.7641	44556748	12.2
19	26544	699.02	1.7470	34509840	9.4
20	26571	692.42	1.7305	15366506	4.2
21	26597	686.06	1.7146	2954734	0.8
22	26781	641.07	1.6021	360734976	98.5
23	27827	385.29	0.9629	210710624	57.5
24	27854	378.69	0.9464	206983168	56.5
25	28009	340.78	0.8517	220006896	60.1
26	28038	333.69	0.8340	237691472	64.9
27	28058	328.80	0.8217	238469120	65.1
28	28086	321.95	0.8046	215654816	58.9
29	28465	229.28	0.5730	199410304	54.4
30	28492	222.67	0.5565	201934272	55.1

JGE006B-2 - DPX300 - 120406 -

JGE006-2 - DPX300 - 120406 -

Peak List

File Name : d:\rmn\nmrdpx~1\jge006~1\1\pdata\1\1R.1R
 Peak Results saved in File : C:\WIN1D\TMP\EDIT.ASC
 Nucleus : off
 SF : 300.130006 MHz
 OFFSET : 16.4642 ppm
 SW_p : 6188.12 Hz
 SI : 32768

Peak Picking Parameter

Peak constant PC = 1.00
 Noise = 40369
 Sens. level = 161478
 Peak Picking region

Start (ppm/Hz)	End (ppm/Hz)	MI (%)	MAXI (%)
7.07/ 2121.5	7.05/ 2115.7	16.26	16.84
6.95/ 2084.7	6.93/ 2078.9	13.36	15.25
6.92/ 2076.4	6.91/ 2072.8	6.45	8.10
7.01/ 2104.2	6.97/ 2090.6	12.14	15.10
6.97/ 2090.8	6.95/ 2086.6	8.60	11.80
7.39/ 2219.2	7.33/ 2201.0	10.11	15.22
7.27/ 2180.8	7.24/ 2172.9	10.11	12.21
7.49/ 2246.7	7.41/ 2223.5	12.50	33.52
6.38/ 1916.3	4.71/ 1413.2	89.37	138.27

Peak Picking results

Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	14073	2283.75	7.6092	9964120	3.1
2	14080	2282.43	7.6048	16708741	5.3
3	14087	2281.11	7.6004	10444851	3.3
4	14109	2276.96	7.5866	14750619	4.6
5	14119	2275.07	7.5803	44556608	14.0
6	14129	2273.18	7.5740	15652865	4.9
7	14152	2268.84	7.5595	21680074	6.8
8	14159	2267.51	7.5551	32609024	10.3
9	14165	2266.38	7.5513	17830458	5.6
10	14302	2240.51	7.4651	63843336	20.1
11	14343	2232.77	7.4393	95135528	29.9
12	14382	2225.40	7.4148	42945060	13.5
13	14424	2217.47	7.3884	33686344	10.6
14	14432	2215.96	7.3833	38959688	12.3
15	14464	2209.91	7.3632	35966076	11.3
16	14472	2208.40	7.3582	42121040	13.3
17	14628	2178.94	7.2600	33188480	10.4
18	15047	2099.82	6.9964	44042412	13.9
19	15090	2091.70	6.9693	39780824	12.5
20	15099	2090.00	6.9636	34351620	10.8
21	15105	2088.86	6.9599	28202584	8.9
22	15139	2082.44	6.9385	45215088	14.2
23	15145	2081.31	6.9347	43142936	13.6
24	15178	2075.08	6.9139	21139724	6.7
25	15184	2073.95	6.9102	20697004	6.5
26	17606	1616.56	5.3862	317711424	100.0

JGE07B-2-R - 300MHz - 230406 -

JGE007B-2-R - 300MHz -

Peak List

```

File Name           : d:\rmn\mai200~1\jge007~1\1\pdata\1\1R.1R
Peak Results saved in File : C:\WIN1D\TMP\EDIT.ASC
Nucleus             : off
SF                  : 300.130006 MHz
OFFSET              : 16.4635 ppm
SW_p                : 6188.12 Hz
SI                  : 32768

```

Peak Picking Parameter

```

Peak constant PC =      1.00
Noise             =     206160
Sens. level       =     824639

```

Peak Picking region

Start (ppm/Hz)	End (ppm/Hz)	MI (%)	MAXI (%)
9.12/ 2736.0	6.61/ 1985.0	93.38	140.61
8.32/ 2497.2	7.84/ 2353.6	35.12	45.71
6.84/ 2051.7	5.82/ 1745.5	7.50	28.06
3.82/ 1146.1	3.63/ 1088.4	14.15	25.36
3.81/ 1143.9	3.61/ 1084.0	9.79	25.77
4.33/ 1298.2	4.08/ 1223.2	16.43	30.14
4.67/ 1403.0	4.43/ 1330.3	16.22	32.21

Peak Picking results

Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	13354	2419.35	8.0610	136513184	37.5
2	13393	2411.98	8.0365	150018096	41.2
3	13552	2381.95	7.9364	137095568	37.7
4	13591	2374.59	7.9119	150595264	41.4
5	14627	2178.94	7.2600	364004160	100.0
6	15671	1981.79	6.6031	34103332	9.4
7	15690	1978.20	6.5911	36090004	9.9
8	15724	1971.78	6.5698	42200328	11.6
9	15743	1968.19	6.5578	40524284	11.1
10	15910	1936.65	6.4527	36298164	10.0
11	15948	1929.48	6.4288	65546816	18.0
12	15986	1922.30	6.4049	42864020	11.8
13	16278	1867.16	6.2212	73585480	20.2
14	16330	1857.34	6.1884	64611316	17.8
15	16585	1809.18	6.0280	35948484	9.9
16	16621	1802.38	6.0053	55562332	15.3
17	16656	1795.77	5.9833	34015588	9.3
18	19045	1344.62	4.4801	66509288	18.3
19	19108	1332.72	4.4405	94347032	25.9
20	19416	1274.56	4.2467	90293968	24.8
21	19479	1262.66	4.2070	68090912	18.7
22	20291	1109.32	3.6961	58567752	16.1
23	20325	1102.90	3.6747	58082472	16.0

AOC97 - 250MHz -

AOC97 - 250MHz -

Peak List

```

File Name           : f:\nmr\2004\12\aoe97\1haoa~4\105001.1R
Peak Results saved in File : C:\WIN1D\TMP\EDIT.ASC
Nucleus             : 1H
SF                  : 250.132854 MHz
OFFSET              : 15.7772 ppm
SW_p                : 5000.00 Hz
SI                  : 16384

```

12

Peak Picking Parameter

```

Peak constant PC =      0.50
Noise             =      1
Sens. level       =      2

```

Peak Picking region

Start (ppm/Hz)	End (ppm/Hz)	MI (%)	MAXI (%)
7.36/ 1841.6	7.28/ 1821.8	9.94	11.00
7.38/ 1846.8	7.32/ 1831.8	6.48	7.99
7.29/ 1823.6	7.24/ 1812.0	4.61	5.77
7.27/ 1817.8	7.24/ 1811.1	3.18	3.70
7.21/ 1803.8	7.14/ 1784.8	8.09	10.65
7.68/ 1921.6	7.48/ 1871.5	2.86	11.61
8.02/ 2005.2	7.87/ 1967.3	7.69	10.43
6.83/ 1707.3	5.94/ 1486.4	8.36	24.58
7.38/ 1845.3	7.27/ 1817.8	3.42	10.48

Peak Picking results

Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	6406	1991.44	7.9615	833	7.9
2	6432	1983.51	7.9298	933	8.9
3	6720	1895.62	7.5784	311	3.0
4	6724	1894.39	7.5736	460	4.4
5	6744	1888.29	7.5492	762	7.2
6	6749	1886.77	7.5431	1059	10.1
7	6769	1880.66	7.5187	547	5.2
8	6774	1879.14	7.5126	674	6.4
9	6908	1838.24	7.3491	500	4.7
10	6912	1837.02	7.3442	706	6.7
11	6933	1830.61	7.3186	792	7.5
12	6937	1829.39	7.3137	1066	10.1
13	6958	1822.98	7.2881	406	3.9
14	6962	1821.76	7.2832	497	4.7
15	6981	1815.96	7.2600	340	3.2
16	7043	1797.04	7.1844	1005	9.5
17	7068	1789.41	7.1539	866	8.2
18	7634	1616.68	6.4633	903	8.6
19	7666	1606.92	6.4243	1442	13.7
20	7753	1580.37	6.3181	2147	20.4
21	7785	1570.60	6.2791	1249	11.9
22	11759	357.83	1.4306	10532	100.0

aoc123 - 300MHz - 240105 -

SQ2/CDCl3/300MHz

Peak List

File Name : c:\armn\sq2ok\3\pdata\1\1R.1R
 Peak Results saved in File : C:\TEMP\EDIT.ASC
 Nucleus : off
 SF : 300.130006 MHz
 OFFSET : 16.4642 ppm
 SW_p : 6188.12 Hz
 SI : 32768

Peak Picking Parameter

Peak constant PC = 1.00
 Noise = 407395
 Sens. level = 1629581

Peak Picking region

Start (ppm/Hz)	End (ppm/Hz)	MI (%)	MAXI (%)
4.23/ 1269.3	3.24/ 971.3	61.13	127.04
6.23/ 1870.0	5.92/ 1776.1	6.71	25.76
6.86/ 2057.7	6.64/ 1992.0	8.53	27.88
8.19/ 2458.8	7.35/ 2205.4	3.08	29.09

Peak Picking results

Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	13457	2400.08	7.9968	32340990	7.6
2	13497	2392.53	7.9716	36739124	8.6
3	14015	2294.71	7.6457	18806772	4.4
4	14048	2288.48	7.6249	41206592	9.7
5	14054	2287.34	7.6212	42302108	9.9
6	14087	2281.11	7.6004	27609236	6.5
7	14094	2279.79	7.5960	27321884	6.4
8	14365	2228.61	7.4255	29093712	6.8
9	14406	2220.87	7.3997	47473564	11.1
10	14444	2213.69	7.3758	23633632	5.5
11	15434	2026.73	6.7529	48508960	11.4
12	15486	2016.91	6.7201	54421760	12.8
13	16405	1843.36	6.1419	55750972	13.1
14	16457	1833.54	6.1092	50983800	12.0
15	20300	1107.81	3.6911	426480288	100.0

Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	10821	3360.74	8.3991	12709	8.0
2	10830	3358.54	8.3936	12047	7.5
3	10858	3351.69	8.3765	13693	8.6
4	11013	3313.79	8.2818	54516	34.1
5	11759	3131.37	7.8259	14383	9.0
6	11787	3124.52	7.8088	14782	9.3
7	11796	3122.32	7.8033	14420	9.0
8	12256	3009.84	7.5221	17963	11.3
9	12263	3008.12	7.5179	20485	12.8
10	12268	3006.90	7.5148	21801	13.7
11	12282	3003.48	7.5063	31587	19.8
12	12295	3000.30	7.4983	21658	13.6
13	12307	2997.36	7.4910	14383	9.0
14	12430	2967.29	7.4158	20734	13.0
15	12464	2958.97	7.3950	24577	15.4
16	12806	2875.34	7.1860	28019	17.5
17	12840	2867.03	7.1652	23470	14.7
18	19159	1321.83	3.3035	26513	16.6
19	19220	1306.92	3.2662	31827	19.9
20	19716	1185.63	2.9631	26658	16.7
21	19732	1181.72	2.9533	30025	18.8
22	19812	1162.15	2.9044	27643	17.3
23	19861	1150.17	2.8745	42613	26.7
24	19876	1146.50	2.8653	45467	28.5
25	22313	550.58	1.3760	159662	100.0

Peak Picking results

Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	11580	3173.93	7.9322	6102	9.9
2	11609	3166.84	7.9145	8558	13.9
3	11630	3161.70	7.9017	7054	11.4
4	12151	3034.30	7.5833	6006	9.7
5	12182	3026.72	7.5643	12895	20.9
6	12212	3019.39	7.5460	8135	13.2
7	12511	2946.27	7.3633	8336	13.5
8	12542	2938.69	7.3443	14425	23.4
9	12573	2931.11	7.3254	7640	12.4
10	12680	2904.94	7.2600	7996	13.0
11	12781	2880.25	7.1983	8466	13.7
12	12794	2877.07	7.1903	9976	16.2
13	12811	2872.91	7.1799	9273	15.0
14	12824	2869.73	7.1720	8982	14.6
15	13902	2606.13	6.5132	6722	10.9
16	13919	2601.97	6.5028	8024	13.0
17	13942	2596.34	6.4888	10365	16.8
18	13959	2592.19	6.4784	10513	17.0
19	14117	2553.55	6.3818	9548	15.5
20	14157	2543.77	6.3574	7008	11.4
21	14195	2534.48	6.3341	10214	16.6
22	14235	2524.70	6.3097	7580	12.3
23	18613	1454.14	3.6342	13124	21.3

24	18639	1447.78	3.6183	12768	20.7
25	20184	1069.98	2.6741	8554	13.9
26	20203	1065.33	2.6625	10562	17.1
27	20390	1019.60	2.5482	10186	16.5
28	20409	1014.96	2.5366	9194	14.9
29	20464	1001.51	2.5030	37163	60.2
30	20986	873.86	2.1839	8428	13.7
31	21045	859.44	2.1479	10895	17.7
32	21229	814.44	2.0354	6802	11.0
33	21287	800.26	2.0000	10744	17.4
34	21461	757.71	1.8937	12085	19.6
35	21519	743.53	1.8582	7634	12.4
36	21659	709.29	1.7727	11303	18.3
37	21717	695.11	1.7372	9463	15.3
38	22262	561.84	1.4041	61549	99.8
39	22285	556.22	1.3901	61697	100.0

Peak Picking results

Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	11339	3233.59	8.0814	3486	7.8
2	11371	3225.77	8.0618	3747	8.4
3	11797	3121.60	7.8015	3375	7.6
4	11826	3114.51	7.7837	3868	8.7
5	12307	2996.89	7.4898	5299	11.9
6	12340	2988.82	7.4696	8063	18.0
7	12359	2984.17	7.4580	6404	14.3
8	12366	2982.46	7.4537	5880	13.2
9	12390	2976.59	7.4391	3373	7.5
10	12395	2975.37	7.4360	3363	7.5
11	12464	2958.50	7.3938	7112	15.9
12	12498	2950.18	7.3731	4434	9.9
13	12683	2904.94	7.2600	2453	5.5
14	19258	1297.15	3.2418	2634	5.9
15	19337	1277.83	3.1935	2807	6.3
16	19358	1272.69	3.1807	1627	3.6
17	19395	1263.65	3.1581	3571	8.0
18	19417	1258.27	3.1446	2776	6.2
19	19677	1194.69	2.9857	3605	8.1
20	19699	1189.31	2.9723	3276	7.3
21	19735	1180.51	2.9503	2595	5.8
22	19758	1174.88	2.9362	2312	5.2
23	19957	1126.22	2.8146	3033	6.8
24	19974	1122.06	2.8042	4945	11.1
25	19993	1117.42	2.7926	3140	7.0
26	20269	1049.93	2.6240	3898	8.7
27	20279	1047.48	2.6178	4249	9.5
28	20289	1045.03	2.6117	4011	9.0
29	20300	1042.34	2.6050	3454	7.7
30	22912	403.63	1.0087	14465	32.4
31	22946	395.31	0.9880	44703	100.0
32	22976	387.98	0.9696	28079	62.8
33	23073	364.26	0.9103	3619	8.1
34	23105	356.43	0.8908	8969	20.1
35	23133	349.59	0.8737	12209	27.3
36	23160	342.98	0.8572	23623	52.8
37	23194	334.67	0.8364	18526	41.4
38	23224	327.33	0.8181	4620	10.3
39	23230	325.87	0.8144	4337	9.7

Peak Picking results

Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	11389	3252.66	8.1290	17109	8.8
2	11391	3252.17	8.1278	17262	8.9
3	11395	3251.20	8.1253	15411	7.9
4	11398	3250.46	8.1235	14987	7.7
5	11422	3244.59	8.1088	13538	7.0
6	11425	3243.86	8.1070	14842	7.6
7	11428	3243.13	8.1052	15321	7.9
8	11783	3156.32	7.8882	17682	9.1
9	11813	3148.98	7.8699	17902	9.2
10	11820	3147.27	7.8656	13641	7.0
11	12279	3035.03	7.5851	32586	16.7
12	12301	3029.65	7.5717	29076	14.9
13	12314	3026.47	7.5637	41311	21.2
14	12329	3022.80	7.5546	31686	16.3
15	12338	3020.60	7.5491	14880	7.6
16	12349	3017.91	7.5423	12801	6.6
17	12356	3016.20	7.5381	10476	5.4
18	12650	2944.31	7.3584	30504	15.7
19	12684	2935.99	7.3376	39736	20.4
20	17548	1746.59	4.3651	2723	1.4
21	17570	1741.21	4.3516	9268	4.8
22	17594	1735.34	4.3369	13218	6.8
23	17620	1728.98	4.3211	9576	4.9
24	17643	1723.36	4.3070	3138	1.6
25	19380	1298.61	3.2455	5252	2.7
26	19409	1291.52	3.2277	4829	2.5
27	19436	1284.91	3.2112	20441	10.5
28	19465	1277.82	3.1935	22418	11.5
29	19483	1273.42	3.1825	23806	12.2
30	19505	1268.04	3.1691	23237	11.9
31	19539	1259.73	3.1483	8064	4.1
32	19561	1254.35	3.1349	6910	3.6
33	20068	1130.37	2.8250	6012	3.1
34	20386	1052.61	2.6307	3498	1.8
35	20407	1047.47	2.6178	3513	1.8
36	20454	1035.98	2.5891	26206	13.5
37	20476	1030.60	2.5757	49837	25.6
38	20503	1024.00	2.5592	29358	15.1
39	20546	1013.48	2.5329	5973	3.1
40	20571	1007.37	2.5176	5809	3.0
41	22946	426.61	1.0662	42036	21.6
42	22978	418.78	1.0466	184355	94.8
43	22980	418.29	1.0454	194547	100.0
44	23009	411.20	1.0277	142451	73.2
45	23128	382.10	0.9549	39709	20.4
46	23134	380.64	0.9513	41678	21.4

47	23160	374.28	0.9354	109850	56.5
48	23194	365.96	0.9146	93039	47.8
49	23223	358.87	0.8969	36967	19.0
50	23231	356.92	0.8920	36679	18.9

Peak Nr. Data Point Frequency PPM Intensity %Int.

1	11063	3301.33	8.2506	28111	29.6
2	11077	3297.91	8.2421	28764	30.3
3	11102	3291.79	8.2268	29876	31.4
4	11832	3113.29	7.7807	23153	24.4
5	11857	3107.17	7.7654	25507	26.8
6	11870	3103.99	7.7575	26038	27.4
7	12335	2990.29	7.4733	57238	60.2
8	12346	2987.60	7.4666	41468	43.6
9	12351	2986.37	7.4635	42473	44.7
10	12359	2984.42	7.4586	43460	45.7
11	12374	2980.75	7.4495	53314	56.1
12	12483	2954.09	7.3828	39019	41.0
13	12517	2945.78	7.3621	45668	48.0
14	12683	2905.19	7.2606	30821	32.4
15	12922	2846.75	7.1146	52318	55.0
16	12956	2838.43	7.0938	45432	47.8
17	17424	1745.86	4.3632	5784	6.1
18	17450	1739.51	4.3473	17832	18.8
19	17461	1736.82	4.3406	19304	20.3
20	17476	1733.15	4.3315	20750	21.8
21	17487	1730.46	4.3247	18909	19.9
22	17514	1723.86	4.3082	7570	8.0
23	17799	1654.16	4.1341	3146	3.3
24	19516	1234.30	3.0848	11461	12.1
25	19528	1231.37	3.0774	13000	13.7
26	19576	1219.63	3.0481	38170	40.2
27	19588	1216.70	3.0408	37006	38.9
28	19626	1207.40	3.0175	34066	35.8
29	19655	1200.31	2.9998	33199	34.9
30	19687	1192.49	2.9802	12204	12.8
31	19715	1185.64	2.9631	12543	13.2
32	20479	998.82	2.4962	89198	93.8
33	20505	992.46	2.4803	95066	100.0

Peak Picking results

Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	10995	3312.58	8.2788	12303	5.1
2	11015	3307.69	8.2665	11751	4.8
3	11548	3177.35	7.9408	24449	10.0
4	11557	3175.15	7.9353	23476	9.6
5	11811	3113.04	7.7801	12034	4.9
6	11830	3108.39	7.7685	13352	5.5
7	12312	2990.53	7.4739	25914	10.6
8	12331	2985.88	7.4623	25058	10.3
9	12446	2957.76	7.3920	10816	4.4
10	12470	2951.89	7.3773	13446	5.5
11	12662	2904.94	7.2600	5537	2.3
12	12674	2902.01	7.2527	5384	2.2
13	12763	2880.25	7.1983	13195	5.4
14	12774	2877.56	7.1916	13566	5.6
15	12796	2872.18	7.1781	11596	4.8
16	17253	1782.30	4.4543	11111	4.6
17	17264	1779.61	4.4476	11103	4.6
18	19236	1297.39	3.2424	6572	2.7
19	19298	1282.23	3.2045	19085	7.8
20	19365	1265.85	3.1636	12721	5.2
21	19404	1256.31	3.1398	4101	1.7
22	19427	1250.69	3.1257	4522	1.9
23	20513	985.12	2.4620	31769	13.0
24	20519	983.66	2.4583	32861	13.5
25	22191	574.80	1.4365	27287	11.2
26	22953	388.47	0.9709	243452	100.0
27	24257	69.60	0.1739	75573	31.0
28	24446	23.38	0.0584	76037	31.2

Peak Nr. Data Point Frequency PPM Intensity %Int.

1	11576	3175.64	7.9365	4085	1.7
2	11610	3167.33	7.9157	10380	4.3
3	11643	3159.26	7.8956	7137	2.9
4	12106	3046.04	7.6126	5325	2.2
5	12111	3044.82	7.6096	4403	1.8
6	12136	3038.70	7.5943	10816	4.5
7	12142	3037.24	7.5906	7984	3.3
8	12167	3031.12	7.5753	6099	2.5
9	12173	3029.66	7.5717	4201	1.7
10	12473	2956.30	7.3883	4791	2.0
11	12484	2953.61	7.3816	6581	2.7
12	12487	2952.87	7.3798	6569	2.7
13	12504	2948.72	7.3694	7782	3.2
14	12514	2946.27	7.3633	9969	4.1
15	12531	2942.11	7.3529	3604	1.5
16	12535	2941.13	7.3504	3567	1.5
17	12545	2938.69	7.3443	4363	1.8
18	12683	2904.94	7.2600	11752	4.9
19	12750	2888.56	7.2191	6230	2.6
20	12765	2884.89	7.2099	9746	4.0
21	12779	2881.47	7.2013	5788	2.4
22	12795	2877.56	7.1916	7836	3.2
23	13893	2609.06	6.5205	4894	2.0
24	13934	2599.03	6.4955	6961	2.9
25	13953	2594.39	6.4839	9028	3.7
26	13993	2584.61	6.4594	11022	4.6
27	14217	2529.83	6.3225	7151	3.0
28	14257	2520.05	6.2981	6352	2.6
29	14278	2514.92	6.2852	12760	5.3
30	14318	2505.13	6.2608	9869	4.1
31	17563	1711.63	4.2777	962	0.4
32	17584	1706.49	4.2648	2463	1.0
33	17604	1701.60	4.2526	2930	1.2
34	17632	1694.76	4.2355	2096	0.9
35	17795	1654.90	4.1359	2093	0.9
36	17825	1647.56	4.1176	5567	2.3
37	17843	1643.16	4.1066	4856	2.0
38	17851	1641.20	4.1017	5482	2.3
39	17881	1633.87	4.0833	1696	0.7
40	18495	1483.72	3.7081	2862	1.2
41	18567	1466.12	3.6641	1643	0.7
42	18606	1456.58	3.6403	2031	0.8
43	18797	1409.88	3.5235	4095	1.7
44	18857	1395.20	3.4869	2432	1.0
45	18877	1390.31	3.4747	1864	0.8
46	18884	1388.60	3.4704	2034	0.8
47	18911	1382.00	3.4539	1722	0.7
48	18946	1373.44	3.4325	2187	0.9

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49	18974	1366.59	3.4154	1082	0.4
50	20001	1115.46	2.7877	2987	1.2
51	20019	1111.06	2.7767	3239	1.3
52	20068	1099.08	2.7468	8047	3.3
53	20085	1094.92	2.7364	7654	3.2
54	20134	1082.94	2.7065	8720	3.6
55	20152	1078.54	2.6955	7973	3.3
56	20278	1047.72	2.6185	9190	3.8
57	20302	1041.86	2.6038	8873	3.7
58	20340	1032.56	2.5806	6576	2.7
59	20345	1031.34	2.5775	6350	2.6
60	20362	1027.18	2.5671	6452	2.7
61	20370	1025.23	2.5622	5891	2.4
62	20408	1015.94	2.5390	3640	1.5
63	20430	1010.56	2.5256	3566	1.5
64	20998	871.66	2.1784	5779	2.4
65	21029	864.08	2.1595	5677	2.3
66	21058	856.99	2.1418	7456	3.1
67	21090	849.16	2.1222	7519	3.1
68	21110	844.27	2.1100	3439	1.4
69	21131	839.14	2.0972	3271	1.4
70	21169	829.85	2.0739	4339	1.8
71	21189	824.96	2.0617	4167	1.7
72	21234	813.95	2.0342	6177	2.6
73	21300	797.81	1.9939	1593	0.7
74	21442	763.09	1.9071	8024	3.3
75	21455	759.91	1.8992	7395	3.1
76	21502	748.42	1.8704	6170	2.5
77	21516	744.99	1.8619	5608	2.3
78	21589	727.14	1.8173	4094	1.7
79	21618	720.05	1.7995	4063	1.7
80	21647	712.96	1.7818	3405	1.4
81	21677	705.62	1.7635	3391	1.4
82	23158	343.47	0.8584	140333	58.0
83	23216	329.29	0.8230	242069	100.0
84	24394	41.23	0.1030	38594	15.9
85	24458	25.58	0.0639	50226	20.7
86	24475	21.42	0.0535	89023	36.8
87	24661	-24.06	-0.0601	70120	29.0