**Direct Lactone Formation Using Hypervalent Iodine(III) Reagents with KBr via
Selective C-H Abstraction Protocol**

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General Information

¹H NMR and ¹³C NMR spectra were recorded on a JEOL JMN-300 or EX-270 spectrometer in CDCl₃ with tetramethylsilane as an internal standard. Data are reported as follows: chemical shift in ppm (δ), integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, br = broad singlet, m = multiplet), and coupling constant (Hz). Infrared spectra (IR) were obtained on a Hitachi 270-50 spectrometer; absorptions are reported in reciprocal centimeters with the following relative intensities: s (strong), m (medium), or w (weak). Mass spectra were obtained on a Shimadzu GCMS-QP 5000 instrument with ionization voltages of 70 eV. High resolution mass spectra and Elemental analyses were performed by the Elemental Analysis Section of Osaka University. Column chromatography was performed with SiO₂ (Merck Silica Gel 60 (230-400 mesh)).

Materials

p-Anisidine diacetate **1** was obtained as a white powder from commercially available *p*-iodoanisole by the known method.¹ Carboxylic acid **2g**² and **2g**³ were prepared according to the literature procedure. Other starting materials were commercially

available and used as received.

General Procedure for Direct Lactone Forming Reaction from Carboxylic Acids Using Aryliodine(III) diacetates 1 with KBr (Table 1)

In a flame-dried two-necked round bottomed flask, under nitrogen, to a stirred suspension of 4-phenyl butyric acid **2a** (82 mg, 0.5 mmol) and KBr (60 mg, 0.5 mmol) in dry CH₂Cl₂ (5 mL), the iodine(III) reagent **1** (211 mg, 0.6 mmol) was added and vigorously stirred for 8 hours at 30 °C. After checking the reaction completion by TLC, saturated NaHCO₃ aq. was added to the mixture, and then stirred for an additional 5 minutes. The organic layer was separated, washed with saturated NaHCO₃ aq., dilute sodium thiosulfate aq. and dried over anhydrous Na₂SO₄. After removal of the solvents, the residue was subjected to silica-gel column chromatography (eluent: *n*-hexane/AcOEt) to give the γ -phenyl γ -butyrolactone **3a** (65 mg, 80%) as a white powder. Unreacted **2a** (9.8 mg, 12%) was recovered from the combined aqueous phase by extraction of dichloromethane after acidifying the solution.

Dihydro-5-phenyl-2(3H)-furanone (3a):⁴ white powder; m.p. 35-36 °C, *R_f* = 0.21 (hexane/EtOAc = 3/1); ¹H NMR (300 MHz, CDCl₃): δ 2.11-2.29 (1H, m), 2.56-2.74 (3H, m), 5.51 (1H, dd, *J* = 7.8, 6.3 Hz), 7.25-7.68 (5H, m); ¹³C NMR (75 MHz, CDCl₃): δ 28.91, 30.92, 81.18, 125.23, 128.40, 128.73, 139.33, 176.87 ; IR (KBr, cm⁻¹): 3034 m, 2930 m, 1767 s, 1606 w, 1497 m, 1455 m, 1417 m, 1329 m, 1286 m, 1215 s, 1176 s, 1141 s, 1080 m, 1021 s, 985 m, 940 s, 913 m, 890 m, 826 m, 757 s, 700 s, 678 m, 530 m; MS, *m/z*: 162 (M⁺).

Table . Direct Aryl Lactone Forming Reaction from Aliphatic Carboxylic Acids **2**

carboxylic acid (2)	aryl lactone (3)

R = H (2a)	(3a)
OMe (2b)	(3b)
NO ₂ (2c)	(3c)
F (2d)	(3d)
Br (2e)	(3e)
Ph (2f)	(3f)

(2g)	(3g)

(2h)	(3h)

(2i)	(3i)

(2j)	(3j)

Dihydro-5-(4-methoxyphenyl)-2(3H)-furanone (3b):⁵ white powder; m.p. 53-54 °C, R_f = 0.31 (hexane/EtOAc = 2/1); ¹H NMR (300 MHz, CDCl₃): δ 2.13-2.24 (1H, m), 2.57-2.70 (3H, m), 3.81 (3H, s), 5.43-5.49 (1H, m), 6.91 (2H, d, J = 8.7 Hz), 7.26 (2H, d, J = 8.7 Hz); ¹³C NMR (75 MHz, CDCl₃): δ 29.20, 30.87, 55.29, 81.32, 114.05,

126.92, 131.08, 159.69, 176.95; IR (KBr, cm^{-1}): 3001 m, 2914 m, 2837 m, 1769 s, 1614 s, 1587 m, 1514 s, 1418 m, 1250 s, 1217 s, 1142 s, 1111 m, 1032 s, 981 m, 937 m, 893 m, 808 m, 772 w, 669 m, 637 w, 530 m; MS, m/z : 192 (M^+).

Dihydro-5-(4-nitrophenyl)-2(3H)-furanone (3c):⁶ white powder; m.p. 226-228 °C, R_f = 0.33 (hexane/EtOAc = 1/1); ^1H NMR (300 MHz, CDCl_3): δ 2.09-2.25 (1H, m), 2.63-2.85 (3H, m), 5.58-5.65 (1H, m), 7.53 (2H, d, J = 8.7 Hz), 8.27 (2H, d, J = 8.7 Hz); ^{13}C NMR (75 MHz, CDCl_3): δ 28.60, 30.87, 79.60, 124.07, 125.91, 146.56, 147.80, 176.00; IR (KBr, cm^{-1}): 3080 w, 2928 w, 2855 w, 1788 s, 1607 m, 1520 s, 1456 w, 1416 m, 1350 s, 1292 m, 1263 m, 1215 m, 1175 s, 1142 s, 1109 m, 1032 s, 995 m, 941 m, 854 s, 818 m, 750 m, 700 m; MS, m/z : 207 (M^+).

5-(4-Fluorophenyl)-dihydro-2(3H)-furanone (3d):⁷ colorless oil; R_f = 0.14 (hexane/EtOAc = 3/1); ^1H NMR (300 MHz, CDCl_3): δ 2.12-2.27 (1H, m), 2.59-2.74 (3H, m), 5.50 (1H, dd, J = 8.3, 6.0 Hz), 7.04-7.15 (2H, m), 7.28-7.36 (2H, m); ^{13}C NMR (75 MHz, CDCl_3): δ 29.01, 31.06, 80.65, 115.60, 115.88, 127.13, 127.24, 176.63; IR (KBr, cm^{-1}): 3080 w, 2926 w, 1778 s, 1609 m, 1514 s, 1458 m, 1421 m, 1329 m, 1227 s, 1177 s, 1142 s, 1099 w, 1022 m, 986 m, 941 m, 891 m, 837 m, 808 m, 783 w, 743 w, 530 w; MS, m/z : 180 (M^+).

5-(4-Bromophenyl)-dihydro-2(3H)-furanone (3e):⁸ white powder; m.p. 81-82 °C, R_f = 0.35 (hexane/EtOAc = 2/1); ^1H NMR (300 MHz, CDCl_3): δ 2.06-2.23 (1H, m), 2.60-2.74 (3H, m), 5.47 (1H, dd, J = 8.1, 6.0 Hz), 7.22 (2H, d, J = 8.4 Hz), 7.52 (2H, d, J = 8.4 Hz); ^{13}C NMR (75 MHz, CDCl_3): δ 28.82, 30.89, 80.41, 122.35, 126.92, 131.88, 138.37, 176.53; IR (KBr, cm^{-1}): 3058 w, 2949 m, 1769 s, 1593 m, 1493 s, 1456 m, 1416 m, 1327 m, 1279 m, 1213 s, 1142 s, 1011 s, 988 m, 937 s, 889 m, 806 s, 716 m, 669 m, 629 w, 606 w, 529 m; MS, m/z : 240 (M^+).

5-[1,1'-Biphenyl]-4-yl-dihydro-2(3H)-furanone (3f):⁹ white powder; m.p. 105-106 °C, R_f = 0.54 (hexane/EtOAc = 1/1); ¹H NMR (300 MHz, CDCl₃): δ 2.15-2.33 (1H, m), 2.63-2.76 (3H, m), 5.56 (1H, t, J = 7.5 Hz), 7.25-7.45 (5H, m), 7.57- 7.68 (4H, m); ¹³C NMR (75 MHz, CDCl₃): δ 28.93, 30.91, 81.01, 125.74, 127.05, 127.43, 127.52, 128.80, 138.26, 140.36, 141.39, 176.82; IR (KBr, cm⁻¹): 3041 w, 2926 w, 1767 s, 1566 w, 1487 m, 1414 m, 1327 m, 1294 m, 1219 m, 1182 s, 1148 s, 1119 w, 1018 m, 980 m, 941 s, 895 m, 843 m, 812 m, 725 m, 690 m, 642 w; MS, m/z : 238 (M⁺).

Dihydro-5,5-diphenyl-2(3H)-furanone (3g):¹⁰ white powder; m.p. 88-89 °C, R_f = 0.71 (hexane/EtOAc = 1/1); ¹H NMR (300 MHz, CDCl₃): δ 2.54-2.65 (2H, m), 2.87-2.99 (2H, m), 7.23-7.50 (10H, m); ¹³C NMR (75 MHz, CDCl₃): δ 29.00, 35.62, 89.67, 125.34, 127.85, 128.56, 142.97, 176.06; IR (KBr, cm⁻¹): 3060 m, 3028 m, 3003 w, 2929 w, 1770 s, 1598 m, 1583 w, 1492 s, 1449 s, 1417 m, 1286 m, 1215 s, 1163 s, 1090 m, 1043 s, 981 s, 921 s, 903 s, 883 m, 846 w, 811 m, 780 w, 751 s, 738 s, 699 s, 663 m, 612 m, 561 w; MS, m/z : 238 (M⁺).

In the reaction of carboxylic acid **2h**, *trans*-lactone product **3h** produced as a major product together with a regioisomeric *cis*-lactone under the reaction conditions. After the typical work-up, a mixture of crude products **3h** and the *cis*-regioisomer was obtained by evaporation, and then it was subjected to ¹H NMR to determine a ratio of **3h** and its *cis*-regioisomer.¹¹ Integrations of the benzyl proton of **3h** (δ 4.9 ppm, 1H) and the *cis*-regioisomer (δ 5.5, 1H) indicated that these isomers presented in a ratio of ca. 100: 5 (Chart 1). Purification by column chromatography (SiO₂/ hexane-AcOEt) gave **3h** containing 4% of the *cis*-regioisomer in 50% yield.

***trans*-Dihydro-4-methyl-5-phenyl-2(3H)-furanone (3h, containing 4% of**

cis-regioisomer):¹¹ white powder; R_f = 0.29 (hexane/EtOAc = 3/1); ^1H NMR (300 MHz, CDCl_3): δ 1.20 (3H, d, J = 6.4 Hz), 2.28-2.55 (2H, m), 2.80 (1H, dd, J = 16.8, 7.5 Hz), 4.94 (1H, d, J = 8.4 Hz), 7.22–7.56 (5H, m); ^{13}C NMR (75 MHz, CDCl_3): δ 16.45, 37.21, 39.83, 88.14, 125.89, 128.68 (x 2), 137.88, 176.13; IR (KBr, cm^{-1}): 3034 m, 2963 m, 2930 m, 2876 m, 1788 s, 1497 m, 1456 m, 1418 m, 1383 m, 1279 s, 1211 s, 1142 s, 1076 m, 1005 s, 947 m, 912 m, 891 w, 862 m, 802 m, 756 s, 700 s, 633 m, 598 w, 550 w; MS, m/z : 176 (M^+).

Tetrahydro-6-phenyl-2H-pyran-2-one (3i):¹² white powder; m.p. 76-78 °C, R_f = 0.21 (hexane/EtOAc = 3/1); ^1H NMR (300 MHz, CDCl_3): δ 1.82-2.04 (3H, m), 2.13-2.21 (1H, m), 2.50-2.76 (2H, m), 5.35 (1H, dd, J = 10.5, 3.3 Hz), 7.25-7.42 (5H, m); ^{13}C NMR (75 MHz, CDCl_3): δ 18.50, 29.42, 30.42, 81.56, 125.63, 128.17, 128.51, 139.67, 171.30; IR (KBr, cm^{-1}): 3063 m, 3033 m, 2955 m, 2881 m, 1738 s, 1605 w, 1495 m, 1455 s, 1444 m, 1330 s, 1238 s, 1187 s, 1158 s, 1043 s, 1002 m, 965 s, 934 s, 875 m, 822 w, 755 s, 700 s, 653 m, 625 m, 571 w, 533 m; MS, m/z : 176 (M^+).

7-Phenyl-2-oxepanone (3j):¹³ white powder; m.p. 66-67 °C, R_f = 0.58 (hexane/EtOAc = 1/1); ^1H NMR (300 MHz, CDCl_3): δ 1.54-1.87 (2H, m), 1.96-2.33 (4H, m), 2.70-2.84 (2H, m), 5.26-5.37 (1H, m), 7.24-7.49 (5H, m); ^{13}C NMR (75 MHz, CDCl_3): δ 22.83, 28.62, 34.95, 37.46, 82.12, 125.84, 128.09, 128.56, 140.75, 174.92 ; IR (KBr, cm^{-1}): 3029 w, 2936 m, 2862 w, 1732 s, 1497 w, 1447 m, 1329 m, 1254 s, 1229 s, 1165 s, 1140 s, 1090 m, 1045 m, 1011 s, 897 w, 858 m, 752 m, 700 s, 584 m; MS, m/z : 190 (M^+).

Chart 1.

General Procedure for Direct Lactone Forming Reaction from Benzoic Acids Using PIDA with KBr

In a flame-dried two-necked round bottomed flask, under nitrogen, to a stirred suspension of 2-ethyl benzoic acid (75 mg, 0.5 mmol) and KBr (60 mg, 0.5 mmol) in dry CH₂Cl₂ (5 mL), thePIDA (193 mg, 0.6 mmol) was added. The resulting mixture was stirred under N₂ (balloon) at 40 °C. for 8 h. TLC check of the reaction mixture confirmed full conversion of the starting benzoic acid. After cooling, saturated NaHCO₃ aq. was added to the mixture, and then stirred for an additional 5 minutes. Product **4b** (60 mg, 81%) was isolated as colorless oil from the organic layer by evaporation of the solvents, and successive silica-gel column chromatography (eluents: *n*-hexane/AcOEt).

In the reaction of 2-alkyl benzoic acid leading to phthalides **4**, more reactive phenyliodine(III) diacetate (PIDA) usually gave better results than *p*-anisiodine diacetate **1**. When using the reagent **1**, large amount of starting materials were remained after 24 hours even at higher temperature (50 °C).

3-Methylphthalide (4b):¹⁴ colorless oil; R_f = 0.30 (hexane/EtOAc = 4/1); ¹H NMR (300 MHz, CDCl₃): δ 1.65 (3H, d, J = 6.6 Hz), 5.58 (1H, q, J = 6.6 Hz), 7.46 (1H, d, J = 7.8 Hz), 7.53 (1H, t, J = 7.5 Hz), 7.66-7.73 (1H, m), 7.89 (1H, d, J = 7.8 Hz); ¹³C NMR (75 MHz, CDCl₃): δ 20.30, 77.66, 121.50, 125.56, 125.67, 128.97, 133.99, 151.11, 170.38 ; IR (KBr, cm⁻¹): 3063 w, 2982 m, 2932 m, 1759 s, 1614 m, 1597 m, 1468 m,

1454 m, 1377 m, 1346 s, 1286 s, 1215 s, 1196 m, 1155 m, 1105 m, 1080 m, 1043 s, 1013 s, 959 m, 924 w, 887 w, 802 m, 764 s, 739 w, 696 s, 602 m, 573 m, 530 m; MS, m/z : 148 (M^+).

Phthalide (4a):¹⁵ white powder; m.p. 73-74 °C, R_f = 0.38 (hexane/EtOAc = 2/1); ^1H NMR (300 MHz, CDCl_3): δ 5.34 (2H, s), 7.49-7.66 (2H, m), 7.68-7.76 (1H, m), 7.93 (1H, d, J = 7.5 Hz); ^{13}C NMR (75 MHz, CDCl_3): δ 69.62, 122.06, 125.64, 125.68, 128.98, 133.97, 146.47, 171.08 ; IR (KBr, cm^{-1}): 3061 w, 2943 w, 2878 w, 1747 s, 1620 m, 1597 m, 1366 s, 1313 m, 1288 s, 1219 s, 1157 m, 1107 m, 1051 s, 1001 s, 974 s, 912 w, 858 w, 800 m, 739 s, 698 m, 681 m, 590 w, 565 w; MS, m/z : 134 (M^+).

Spiro[cyclohexane-1,1'(3'H)-isobenzofuran]-3'-one (4c):¹⁶ white powder; m.p. 77-78 °C, R_f = 0.39 (hexane/EtOAc = 4/1); ^1H NMR (300 MHz, CDCl_3): δ 1.60-2.10 (10H, m), 7.40 (1H, d, J = 7.5 Hz), 7.50 (1H, t, J = 7.2 Hz), 7.61-7.69 (1H, m), 7.87 (1H, d, J = 7.8 Hz); ^{13}C NMR (75 MHz, CDCl_3): δ 22.28, 24.69, 36.32, 86.96, 120.92, 125.46, 125.77, 128.90, 133.86, 154.88, 170.12; IR (KBr, cm^{-1}): 3042 w, 2936 s, 2861 m, 1760 s, 1614 m, 1599 m, 1494 m, 1464 m, 1449 m, 1368 m, 1353 m, 1328 m, 1282 s, 1270 s, 1239 m, 1203 m, 1089 s, 1017 m, 946 s, 925 s, 861 m, 191 w, 760 s, 727 m, 693 s, 662 w, 601 m, 542 m; MS, m/z : 202 (M^+).

From 3-phenethyl benzoic acid, two isomers, phthalide **4d** (minor product) and isocoumarin **4d'** (major product), were formed in total 81% yield (Eq. 1). These isomers were separable by usual column chromatography techniques (SiO_2 (neutral)/ n -hexane-AcOEt). The isolated yield of **4d** and **4d'** is 18 % and 63 %, respectively. Thus, the ratio of **4d/4d'** is calculated to 22:78.

3-Phenylmethyl-1(3H)-isobenzofuranone (4d, minor product):¹⁷ white powder; m.p. 60-61 °C, R_f = 0.36 (hexane/EtOAc = 3/1); ^1H NMR (270 MHz, CDCl_3): δ 3.16 (1H, dd, J = 14.1, 6.2 Hz), 3.29 (1H, dd, J = 14.1, 6.8 Hz), 5.69 (1H, t, J = 6.5 Hz), 7.12-7.81 (8H, m), 7.84 (1H, d, J = 7.6 Hz); ^{13}C NMR (75 MHz, CDCl_3): δ 40.83, 81.19, 122.27, 125.68, 126.21, 127.14, 128.53, 129.19, 129.69, 133.69, 134.96, 149.08, 170.25; IR (KBr, cm^{-1}): 3030 w, 2922 w, 1765 s, 1601 w, 1495 m, 1466 m, 1454 m, 1350 m, 1286 m, 1267 m, 1213 m, 1155 w, 1065 s, 1015 m, 989 m, 912 w, 756 m, 737 m, 696 s, 627 w, 546 m; MS, m/z : 224 (M^+).

3,4-Dihydro-3-phenyl-1H-2-benzopyran-1-one (4d', major product):¹⁸ white powder; m.p. 90-91 °C, R_f = 0.41 (hexane/EtOAc = 3/1); ^1H NMR (300 MHz, CDCl_3): δ 3.14 (1H, dd, J = 16.5, 3.3 Hz), 3.36 (1H, dd, J = 16.5, 12.0 Hz), 5.57 (1H, dd, J = 12.0, 3.3 Hz), 7.00-7.60 (3H, m), 8.16 (1H, d, J = 7.8 Hz); ^{13}C NMR (75 MHz, CDCl_3): δ 35.62, 79.97, 125.11, 126.11, 127.34, 127.88, 128.65, 128.68, 130.43, 133.91, 138.52, 138.92, 165.34; IR (KBr, cm^{-1}): 3034 w, 2924 w, 2853 w, 1728 s, 1605 m, 1487 w, 1421 w, 1346 m, 1271 s, 1227 m, 1119 s, 1084 s, 1067 m, 1030 m, 1001 m, 912 m, 800 w, 743 s, 698 s, 640 w; MS, m/z : 224 (M^+).

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060137-1

DFILE
COMNT
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OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
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SCANS
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NON
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130.00 KHz
1150.00 Hz
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6006.01 Hz
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0.00 ppm
0.12 Hz
15

3b

(13C) 5-(4-Methoxyphenyl)dihydrofuran-2-one

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 BF 0.12 Hz
 RGAIN 22

3d

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 BF 0.12 Hz
 RGAIN 24

3d

1H-4-Br-lactone

DFILE E:\(1H)-3e 5-(4-Bromo-phen
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 IRNUC 1H
 CTEMP 20.0 C
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 17

¹³C-4-Br-Lactone

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
B1
RGAIN

E:\(13C)-3e 5-(4-Bromo-phe
13C-4-Br-Lactone
Wed May 09 12:42:50 2007
13C

BCM
75.45 MHz
124.00 KHz
1840.00 Hz
32768
20356.23 Hz
450
1.6097 sec
1.3900 sec
4.50 usec
1H
20.3 c
CDCL3
77.00 ppm
0.12 Hz
24

DFILE E:\(1H)-3f 5-(4-Biphenyl)d
 COMNT 5-(4-Fluorophenyl)dihydrof
 DATIM Tue May 15 21:10:01 2007

OBNUC 1H
 EXMOD NON
 OFFRQ 300.40 MHz
 OBSET 130.00 KHz
 OFFIN 1150.00 Hz
 POINT 32768
 FREQU 6006.01 Hz
 SCANS 17
 ACQTM 5.4559 sec
 PD 1.0000 sec
 PW1 5.80 usec
 IRNUC 1H
 CTEMP 22.3 C
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 17

5-(4-Biphenyl)dihydrofuran-2-one

DFILE E:\(13C)-3f 5-(4-Biphenyl)
 COMNT 5-(4-Biphenyl)dihydrofuran
 DATIM Thu Dec 07 15:26:09 2006

OBNUC 13C
 EXMOD BCM
 OBFRQ 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.00 Hz
 POINT 32768
 FREQU 20356.23 Hz
 SCANS 600
 ACQTM 1.6097 sec
 PD 1.3900 sec
 PW1 4.50 usec
 IRNUC 1H
 CTEMP 23.3 C
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 26

DFILE E:\(1H)-3g.als
 COMNT
 DATIM Thu Dec 21 20:25:31 2006
 OBNUC 1H
 EXMOD NON
 OBFRQ 300.40 MHz
 OBSET 130.00 KHz
 OBFIN 1150.00 Hz
 POINT 32768
 FREQU 6006.01 Hz
 SCANS 16
 ACQTM 5.4559 sec
 PD 1.5440 sec
 PW1 5.50 usec
 IRNUC 1H
 CTEMP 21.4 C
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 14

DFILE E:\(13C)-3g.als
 COMNT
 DATIM Fri Dec 22 20:28:32 2006
 OBNUC 13C
 EXMOD BCM
 OBFRQ 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.00 Hz
 POINT 32768
 FREQU 20356.23 Hz
 SCANS 520
 ACQTM 1.6097 sec
 PD 1.3900 sec
 PW1 4.50 usec
 IRNUC 1H
 CTEMP 22.5 C
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 26

C:\Documents and Settings\
 DFILE COMNT
 DATIM Mon Jun 04 08:56:45 2007
 OBNUC 1H
 EXMOD NON
 OBFREQ 300.40 MHz
 OBSET 130.00 KHz
 OBFIN 1150.00 Hz
 POINT 32768
 FREQU 6006.01 Hz
 SCANS 32
 ACQTM 5.4559 sec
 PD 1.0000 sec
 PW1 5.80 usec
 IRNUC 1H
 CTEMP 20.2 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 19

3h

C:\Documents and Settings\

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFREQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

Thu May 31 12:57:28 2007
13C
ECM
75.45 MHz
124.00 KHz
1840.00 Hz
32768
20356.23 Hz
1359
1.6097 sec
1.3900 sec
4.50 usec
1H
19.9 c
CDCL3
77.00 ppm
0.12 Hz
24

3h

060111-2

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFREQ
OBFSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

C:\Documents and Settings\
060111-2
Sat Dec 02 14:19:31 2006
1H
NON
300.40 MHz
130.00 KHz
1150.00 Hz
32768
6006.01 Hz
16
5.4559 sec
1.5440 sec
5.50 usec
1H
22.3 c
CDCL3
0.00 ppm
0.12 Hz
15

060111-2

DFILE 060111-2
 COMNT 060111-2
 DATIM Sat Dec 02 14:59:18 2006
 OBNUC 13C
 EXMOD BCM
 OBFRQ 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.00 Hz
 POINT 32788
 FREQU 20356.23 Hz
 SCANS 600
 ACQTM 1.6097 sec
 PD 1.3900 sec
 PW1 4.50 usec
 IRNUC 1H
 CTEMP 23.1 c
 SLVNT CDCl3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 26

C:\Documents and Settings\

COMNT
DATIM Tue May 29 20:01:41 2007
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.00 Hz
POINT 32768
FREQU 6006.01 Hz
SCANS 64
ACQTM 5.4559 sec
PD 1.0000 sec
PW1 5.80 usec
IRNUC 1H
CTEMP 19.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 22

C:\Documents and Settings\
 DFILE COMNT
 DATIM Thu May 31 02:44:33 2007
 OBNUC 13C
 EXMOD BCM
 OBFRQ 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.00 Hz
 POINT 32768
 FREQU 20356.23 Hz
 SCANS 1181
 ACQTM 1.6097 sec
 PD 1.3900 sec
 PW1 4.50 usec
 IRNUC 1H
 CTEMP 20.5 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 25

060147-1-1

DFILE C:\Documents and Settings\
 COMNT 060147-1-1
 DATIM Tue Apr 24 13:20:03 2007
 OBNUC 1H
 EXMOD NON
 OBFRQ 300.40 MHz
 OBSET 130.00 KHz
 OBFIN 1150.00 Hz
 POINT 32768
 FREQU 6006.01 Hz
 SCANS 16
 ACQTM 5.4559 sec
 PD 1.0000 sec
 PW1 5.80 usec
 IRNUC 1H
 CTEMP 17.4 c
 SLVNT CDCl3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 16

DFILE
 COMNT
 DATIM Thu May 31 01:40:48 2007
 OBNUC 13C
 EXMOD BCM
 OBFRQ 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.00 Hz
 POINT 32768
 FREQU 20356.23 Hz
 SCANS 1066
 ACQTM 1.6097 sec
 PD 1.3900 sec
 PW1 4.50 usec
 IRNUC 1H
 CTEMP 20.5 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 24

060113

C:\Documents and Settings\
 060113
 COMNT 060113
 DATIM Sat Dec 02 17:24:22 2006
 OBNUC 1H
 EXMOD NON
 OBFREQ 300.40 MHz
 OBSET 130.00 KHz
 OBFIN 1150.00 Hz
 POINT 32768
 FREQU 6006.01 Hz
 SCANS 16
 ACQTM 5.4559 sec
 PD 1.5440 sec
 PW1 5.50 usec
 IRNUC 1H
 CTEMP 21.4 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 EF 0.12 Hz
 RGAIN 13

4b

C:\Documents and Settings\

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PWL
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

Sat Dec 02 18:01:28 2006
13C
BCM
75.45 MHz
124.00 KHz
1840.00 Hz
32768
20356.23 Hz
600
1.6097 sec
1.3900 sec
4.50 usec
1H
23.0 c
CDCL3
77.00 ppm
0.12 Hz
25

060171-2

DEFILE C:\Documents and Settings\
 COMNT 060171-2
 DATIM Thu May 24 19:11:43 2007
 ORNUC 1H
 EXMOD NON
 OBFRQ 300.40 MHz
 OBSET 130.00 KHz
 OBFIN 1150.00 Hz
 POINT 32768
 FREQU 6006.01 Hz
 SCANS 16
 ACQTM 5.4559 sec
 PD 1.5440 sec
 PWL 5.80 usec
 IRNUC 1H
 CTEMP 19.3 C
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 14

DFILE C:\Documents and Settings\
 COMNT Wed May 30 12:55:11 2007
 DATIM 13C
 OBNUC BCM
 EXMOD 75.45 MHz
 OBFRQ 124.00 KHz
 OBSET 1840.00 Hz
 OBFIN 32768
 POINT 20356.23 Hz
 FREQU 993
 SCANS 1.6097 sec
 ACQTM 1.3900 sec
 PD 4.50 usec
 PW1 1H
 IRNUC 20.3 c
 CTEMP CDCL3
 SLVNT 77.00 ppm
 EXREF 0.12 Hz
 BF 24
 RGAIN

C:\Documents and Settings\
 COMNT Thu May 31 19:49:30 2007
 DATIM 1H
 OBNUC 1H
 EXMOD NON
 OBFREQ 270.05 MHz
 OBSET 112.00 KHz
 OBFIN 5800.00 Hz
 POINT 32768
 FREQU 5402.40 Hz
 SCANS 64
 ACQTM 6.0655 sec
 PD 0.9350 sec
 PW1 5.50 usec
 IRNUC 1H
 CTEMP 19.4 C
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 22

DFILE E:\(13C)-4d.als
 COMNT
 DATIM Fri Jun 01 08:53:18 2007
 OBNUC 13C
 EXMOD BCM
 OBFRQ 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.00 Hz
 POINT 32768
 FREQU 20356.23 Hz
 SCANS 8929
 ACQTM 1.6097 sec
 PD 1.3900 sec
 FW1 4.50 usec
 IRNUC 1H
 CTEMP 19.7 C
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 24

4d

DFILE C:\Documents and Settings\
 COMNT Thu May 31 15:15:44 2007
 DATIM 1H
 ORNUC 1H
 EXMOD NON
 OBFRQ 300.40 MHz
 OBSET 130.00 KHz
 OBFIN 1150.00 Hz
 POINT 32768
 FREQU 6006.01 Hz
 SCANS 32
 ACQTM 5.4559 sec
 PD 1.5440 sec
 PW1 5.80 usec
 IRNUC 1H
 CTEMP 19.7 C
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 12

4d'

DFILE C:\Documents and Settings\
 COMNT Thu May 31 08:46:41 2007
 DATIM 13C
 OBNUC BCM
 EXMOD 75.45 MHz
 OBFRQ 124.00 KHz
 OBSET 1840.00 Hz
 OBFIN 32768
 POINT 20356.23 Hz
 FREQU 7195
 SCANS 1.6097 sec
 ACQTM 1.3900 sec
 PD 4.50 usec
 PWL 1H
 IRNUC 20.1 C
 CTEMP CDCL3
 SLVNT 77.00 ppm
 EXREF 0.12 Hz
 BF 24
 RGAIN

4d'