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Supplementary Information

Dichloroiodoisocyanuric Acid: A New Reagent for Regioselective Coiodination of Alkenes and Iodination of Activated Arenes#

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Dichoroiodoisocyanuric acid (DCICA)

Crushed iodine (26.65 g, 105 mmol) and trichloroisocyanuric acid (23.25 g, 100 mmol) were added to a 100 cm^3 sealed tube and heated in a sand bath at 170 °C. After 18 h, the ICl produced was distilled off under reduced pressure and the sealed tube was heated again at 220 °C during 24 h. Evaporation of ICl under reduced pressure and heating gave a solid that was crushed and washed with CH_2Cl_2 to produce 30.13 g of dichloroiodoisocyanuric acid (DCICA). This reagent must be stored in the dark as some decomposition occurs in the presence of light.

Beige solid (93% yield); mp > 300 °C. Anal. Calc. for $C_3N_3O_3Cl_2I$: C, 11.11, N, 12.96. Found: C, 10.74, N, 12.48. ^{13}C MAS-NMR: δ 150.1 ppm (Figure S1). IR ν_{max}/cm^{-1}: 1837, 1716, 1697, 1662, 1457, 1362, 762, 534 (Figure S2).

General procedure for coiodination of alkenes with DCICA in oxygenated nucleophilic solvents

To a stirred solution of the alkene (2 mmol) in an appropriate solvent (10 cm^3 of acetone/2 cm^3 of H_2O for iodohydrins, or 10 cm^3 of alcohols for β-iodoethers, or 5 cm^3 acetic acid/5 cm^3 Ac_2O for β-iodoacetates), DCICA (2 mmol) was added at room temperature and in the absence of light. After 1 min, CH_2Cl_2 (10 cm^3) was added, cyanuric acid was filtered off and the resulting solution was treated with 10% aq. $NaHSO_3$ (50 cm^3). The aqueous phase was washed with CH_2Cl_2 (2 × 10 cm^3), the organic extract was dried (anhydrous Na_2SO_4) and filtered. The solvent was evaporated on a rotatory evaporator to give the product.

1-Methoxy-1-phenyl-2-iodoethane[1]

Yellowish liquid (99% yield). ^1H NMR ($CDCl_3$): δ 3.32 (s, 3H), 3.35 (d, 1H, *J* 7.3 Hz), 3.36 (d, 1H, *J* 5.6 Hz), 4.31 (dd, 1H, *J* 7.3 and 5.6 Hz), 7.29-7.43 (m, 5H) ppm (Figure S3). ^{13}C NMR ($CDCl_3$): δ 10.5, 57.3, 83.6, 126.6, 128.5, 128.7, 139.8 ppm (Figure S4). IR ν_{max}/cm^{-1}: 3083, 3062, 3027, 2985, 2931, 2900, 2877, 2823, 1602, 1493, 1454, 1410, 1354, 1225, 1174, 1108, 1088, 955, 766, 700, 580 (Figure S5). MS: *m/z* 262 (M^+), 135, 121 (100%), 104, 91, 77, 51 (Figure S6).

1-Ethoxy-1-phenyl-2-iodoethane[2]

Yellowish liquid (98% yield).^1H NMR ($CDCl_3$): δ 1.22 (t, 3H, *J* 7.0 Hz), 3.33 (d, 2H, *J* 6.5 Hz) 3.45 (q, 2H, *J* 7.0 Hz), 4.40(t, 1H, *J* 6.5 Hz), 7.34 (s, 5H) ppm (Figure S7). ^{13}C NMR ($CDCl_3$): δ 11.1, 15.4, 65.3, 82.0, 126.7, 128.5, 128.8, 140.7 ppm (Figure S8). IR ν_{max}/cm^{-1}: 3084, 3061, 3028, 2974, 2927, 2870, 1601, 1493, 1453, 1343, 1172, 1117, 1094, 763, 701, 582 (Figure S9). MS: *m/z* 276 (M^+), 149, 135 (100%), 121, 107, 104, 91, 79, 77, 65, 51, 43 (Figure S10).

2-Iodo-1-phenyl-1-isopropoxyethane[2]

Yellowish liquid (98% yield). ^1H NMR ($CDCl_3$): δ 1.12 (d, 3H, *J* 6.1 Hz), 1.25 (d, 3H, *J* 5.8 Hz), 3.32 (d, 2H, *J* 6.2 Hz), 3.58 (m, 1H), 4.53 (t, 1H, *J* 6.2 Hz), 7.35 (s, 5H) ppm (Figure S11). ^{13}C NMR ($CDCl_3$): δ 11.7, 21.4, 23.2, 70.5, 79.7, 126.6, 128.2, 128.6, 141.5 ppm (Figure S12). IR ν_{max}/cm^{-1}: 3084, 3062, 3028, 2970, 2929, 2833, 1601, 1493, 1454, 1410, 1377, 1335, 1119, 1090, 1070, 1047, 1007, 762, 700, 586 (Figure S13). MS: *m/z* 290 (M^+), 231, 149, 107 (100%), 105, 104, 79, 77, 43 (Figure S14).

Trans-1-iodo-2-methoxycyclohexane[3]

Colorless liquid (75% yield). ^1H NMR ($CDCl_3$): δ 1.24-2.44 (m, 8H), 3.25 (td, 1H, *J* 9.0 Hz, *J* 3.8 Hz), 3.42 (s, 3H), 4.08 (td, 1H, *J* 9.0 Hz, *J* 4.1 Hz) ppm (Figure S15). ^{13}C NMR ($CDCl_3$): δ 23.7, 27.3, 30.5, 35.5, 38.0, 57.0, 84.1 ppm (Figure S16). IR ν_{max}/cm^{-1}: 2978, 2935, 2858, 2823, 1446, 1367, 1192, 1163, 1113, 1086, 928, 661, 624, 444 (Figure S17). MS: *m/z* 240 (M^+), 113, 81 (100%), 71, 45, 41 (Figure S18).

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#Dedicated to Prof. José Barluenga for his many achievements in synthetic organic chemistry.

Trans-1-iodo-2-isopropoxycyiclohexane[4]

Colorless liquid (76% yield). ^{1}H NMR ($CDCl_3$): δ 1.15 (d, 3H, *J* 6.0 Hz), 1.22 (d, 3H, *J* 6.0 Hz), 1.30-2.44 (m, 8H), 3.37 (td, 1H, *J* 8.9 Hz, *J* 4.4 Hz), 3.78 (sep, 1H, *J* 6.1 Hz), 4.02 (qd, 1H, *J* 10.9 Hz, *J* 8.9 Hz, *J* 4.1 Hz) ppm (Figure S19). ^{13}C NMR ($CDCl_3$): δ 22.7, 23.2, 23.9, 27.3, 33.0, 37.0, 38.2, 71.2, 80.6 ppm (Figure S20). IR ν_{max}/cm^{-1}: 2970, 2935, 2858, 1724, 1448, 1377, 1367, 1334, 1165, 1117, 1086, 1018, 918, 661, 623 (Figure S21). MS: *m/z* 268 (M^+), 209, 141, 99, 81 (100%), 57, 43 (Figure S22).

1-Iodo-2-methoxyoctane[5] and 2-iodo-1-methoxyoctane (5:1 by HRGC-MS)

Colorless liquid (80% yield). ^{1}H NMR ($CDCl_3$): δ 0.89 (brs, 3H + 3H), 1.30 (sbr, 8H + 8H), 1.56 (sbr, 2Ha), 1.77 (m, 2Hb), 3.03 (quint, 1H, *J* 5.0 Hz), 3.27 (brs, 2H), 3.38 (s, 3H), 3.62(m, 2Hb), 4.17 (quint, 1H, *J* 6.4 Hz) ppm (Figure S23). ^{13}C NMR ($CDCl_3$): δ 9.9, 14.1, 22.6, 25.2, 28.6, 29.3, 31.7, 31.8, 34.1, 34.4, 36.5, 57.1, 58.7, 78.4, 79.9 ppm (Figure S24). IR ν_{max}/cm^{-1}: 2954, 2927, 2870, 2856, 1458, 1377, 1329, 1180, 1153, 1095, 738, 725, 623 (Figure S25).

1-Iodo-2-methoxyoctane (major)

MS: *m/z* 270 (M^+), 185, 143, 129 (100%), 97, 69, 58, 55, 45, 41 (Figure S26).

2-Iodo-1-methoxyoctane (minor)

MS: *m/z* 185 (M^+-Hex), 143, 111, 69 (100%), 58, 45 (Figure S27).

1-Phenyl-2-iodoethanol[2]

Yellowish liquid (87% yield). ^{1}H NMR ($CDCl_3$): δ 2.46 (brs, 1H, OH), 3.35-3.54 (m, 2H), 4.83 (dd, 1H, *J* 8.5 Hz, *J* 4.0 Hz), 7.37 (s, 5H) ppm (Figure S28). ^{13}C NMR ($CDCl_3$): δ 15.3, 74.1, 125.8, 128.4, 128.7, 141.2 ppm (Figure S29). IR ν_{max}/cm^{-1}: 3373, 3086, 3061, 3028, 2954, 2897, 1603, 1495, 1452, 1412, 1329, 1294, 1174, 1055, 764, 698, 569 (Figure S30). MS: *m/z* 248 (M^+), 121, 107 (100%), 103, 91, 79, 77, 65, 51, 43 (Figure S31).

2-Phenyl-1-iodo-2-propanol[6]

Yellowish liquid (99% yield). ^{1}H NMR ($CDCl_3$): δ 1.75 (s, 3H), 2.44 (brs, 1H, OH), 3.65 (s, 2H), 7.50-7.28 (m, 5H) ppm (Figure S32). ^{13}C NMR ($CDCl_3$): δ 24.3, 29.0, 72.7, 124.8, 127.5, 128.5, 144.3 ppm (Figure S33). IR ν_{max}/cm^{-1}: 3373, 3086, 3061, 3028, 2954, 2897, 1603, 1495, 1452, 1412, 1329, 1294, 1174, 1055, 764, 698, 569 (Figure S34). MS: *m/z* 262 (M^+), 135, 121, 105, 91, 77, 65, 51, 43 (100%) (Figure S35).

Trans-2-iodocyclohexanol[1]

Colorless liquid (83% yield). ^{1}H NMR ($CDCl_3$): δ 1.21-2.13 and 2.43-2.50 (m, 8H), 2.29 (s, 1H, OH), 3.65 (td, 1H, *J* 9.8 Hz, *J* 4.4 Hz), 4.04 (qt, 1H, *J* 12.3 Hz, *J* 9.8 Hz, *J* 4.4 Hz) ppm (Figure S36). ^{13}C NMR ($CDCl_3$): δ 24.5, 28.0, 33.8, 38.6, 43.3, 76.0 ppm (Figure S37). IR ν_{max}/cm^{-1}: 3302, 2933, 2856, 1444, 1159, 1066, 949, 654, 555 (Figure S38).MS: *m/z* 226 (M^+), 99, 81 (100%), 79, 69, 57, 55, 43, 41 (Figure S39).

Trans-2-iodo-1-methylcyclohexanol[7]

Red liquid (42% yield). ^{1}H NMR ($CDCl_3$): δ 1.20-1.33 and 1.53-2.09 and 2.32-2.42 (m, 8H), 1.39 (s, 1H), 2.17 (s, 1H, OH), 4.34 (dd, 1H, *J* 12.1 Hz, *J* 4.3 Hz) ppm (Figure S40). ^{13}C NMR ($CDCl_3$): δ 23.4, 26.0, 28.2, 37.6, 37.6, 50.1, 72.5 ppm (Figure S41). IR ν_{max}/cm^{-1}: 3412, 2978, 2935, 2860, 1444, 1375, 1188, 1120, 1099, 962, 924, 725, 660, 546 (Figure S42). MS: *m/z* 225 (M^+- Me), 113, 95, 71, 69, 67, 55, 45, 43 (100%), 41 (Figure S43).

1-Iodo-2-octanol[6] and 2-iodo-1-octanol (5:1 by HRGC-MS)

Red liquid (85% yield). ^{1}H NMR ($CDCl_3$): δ 0.78 (s, 3H), 0.82 (brs, 3H), 1.22 (brs, 8H, 1.40-1.60 (m, 2H), 1.63-1.83 (m, 2H), 1.97 (brs, 1H, OH), 3.16 (dd, 1H, *J* 10.2 Hz, *J* 6.8 Hz), 3.32 (dd, 1H, *J* 10.2 Hz, *J* 3.4 Hz), 3.39-3.50 (m, 1Ha), 3.62 (dd, 1H, *J* 12.3 Hz, *J* 5.1 Hz), 3.69 (dd, 1H, *J* 12.3 Hz, *J* 6.1 Hz), 4.09-4.22 (m, 1H) ppm (Figure S44). ^{13}C NMR ($CDCl_3$): δ 14.1, 16.9, 22.6, 25.7, 28.6, 29.2, 31.7, 31.8, 36.3, 36.7, 42.2, 53.5, 71.1 ppm (Figure S45). IR ν_{max}/cm^{-1}: 3373, 2954, 2927, 2856, 1460, 1414, 1377, 1180, 1124, 1020, 725, 623 (Figure S46).

1-Iodo-2-octanol (major)

MS: *m/z* 256 (M^+), 186, 171, 142, 129, 115, 97, 85, 69 (100%), 55, 44, 43, 41 (Figure S47).

2-Iodo-1-octanol (minor)

MS: *m/z* 171 (M^+- Hex), 129, 111, 83, 69 (100%), 55, 44, 41 (Figure S48).

2-Iodo-1-phenyethyl acetate[8]

Yellowish liquid (89% yield). ^{1}H NMR ($CDCl_3$): δ 2.12 (s, 3H), 3.45 (d, 1H, *J* 7.3 Hz), 3.46 (d, 1H, *J* 6.0 Hz), 5.87 (dd, 1H, *J* 7.3 and 6.0 Hz), 7.34 (m, 5H) ppm (Figure S49). ^{13}C NMR ($CDCl_3$): δ 8.0, 21.2, 75.3, 126.6, 128.8, 128.9, 138.6, 169.9 ppm (Figure S50). IR ν_{max}/cm^{-1}: 3483, 3088, 3062, 3032, 2962, 1747, 1495, 1454, 1372, 1232, 1207, 1178, 1059, 1018, 763, 700, 602, 566 (Figure S51). MS: *m/z* 247, 163, 121, 107, 103, 77, 43 (100%) (Figure S52).

2-Iodo-1-butoxy-1-methoxybutane[9]

Yellowish liquid (99% yield). ^{1}H NMR ($CDCl_3$): δ 0.93 (t, 3H), 1.35-1.45 (m, 2H), 1.55-1.59 (m, 2H), 3.17-3.25 (m, 2H), 3.37 (s, 3H), 3.44-3.69 (m, 2H), 4.54 (m, 1H) ppm (Figure S53). ^{13}C NMR ($CDCl_3$): δ 4.9, 13.9, 19.4, 31.8, 53.5, 66.8, 102.7 ppm (Figure S54). MS: *m/z* 227 (M^+ – OMe), 185, 171, 117, 61 (100%), 58, 57, 41 (Figure S55).

Trans-3-iodo-2-methoxy tetrahydropyran[10]

Red liquid (99% yield). ^{1}H NMR ($CDCl_3$): δ 1.49-1.85 (m, 2H), 1.94-2.12 (m, 1H), 2.30-2.45 (m, 1H), 3.46 (s, 3H), 3.54-3.66 (m, 1H), 3.94-4,12 (m, 2H), 4.54 (d, 1H, *J* 6 Hz) ppm (Figure S56). ^{13}C NMR ($CDCl_3$): δ 26.0, 29.3, 33.2, 56.2, 63.9, 104.0 ppm (Figure S57). MS: *m/z* 242 (M^+), 211, 197, 182, 154, 127, 115 (100%), 83, 61, 55, 45, 39 (Figure S58).

General procedure for iodination of activated arenes with DCICA

To a stirred solution of the arene (2 mmol) in acetonitrile (10 cm^3), was added DCICA (2 mmol) at room temperature and in the absence of light. The reaction was monitored by HRGC-MS and after the specified time showed in Table 3, CH_2Cl_2 (10 cm^3) was added, cyanuric acid was filtered off and the resulting solution was treated with 10% aq. $NaHSO_3$ (60 cm^3). The aqueous phase was extracted with CH_2Cl_2 (2 × 10 cm^3), the combined organic extract was washed with H_2O (60 cm^3), dried (anhydrous Na_2SO_4) and filtered. The solvent was evaporated on a rotatory evaporator to give the product.

4-Iodoanisole[11]

White solid (97% yield); mp 48-50 °C (Lit.[11] 48-50 °C). ^{1}H NMR ($CDCl_3$): δ 3.79 (s, 3H), 6.69 (d, 2H, *J* 8.8 Hz), 7.57 (d, 2H, *J* 8.8 Hz) ppm (Figure S59). ^{13}C NMR ($CDCl_3$): δ 55.4, 82.8, 116.5, 138.3, 159.5 ppm (Figure S60). MS: *m/z* 235 (M^++ 1), 234 (M^+, 100%), 219, 191, 92, 77, 64, 63, 50 (Figure S61).

1-Iodo-2-methoxynaphthalene[12]

Beige solid (99% yield); mp 84-86 °C (Lit.[12] 87 °C). ^{1}H NMR ($CDCl_3$): δ 4.03 (s, 3H), 7.21 (d, 1H, *J* 9.0 Hz), 7.40 (t, 1H, *J* 8.0 Hz), 7.56 (t, 1H, *J* 8.0 Hz), 7.76 (d, 1H, *J* 8.0 Hz), 7.83 (d, 1H, *J* 9.0 Hz), 8.17 (d, 1H, *J* 8.0 Hz) ppm (Figure S62). ^{13}C NMR ($CDCl_3$): δ 57.3, 87.8, 113.0, 124.4, 128.2, 128.3, 130.0, 130.4, 131.3, 135.7, 156.7 ppm (Figure S63). MS: *m/z* 285 (M^++ 1), 284 (M^+, 100%), 269, 241, 142, 127, 114, 88, 63 (Figure S64).

4-Iodoacetanilide[11]

White solid (96% yield); mp 183-185 °C (Lit.[11] 182-184 °C). ^{1}H NMR (DMSO-d_6): δ 2.03, (s, 3H), 7.41 (d, 2H, *J* 8.9 Hz), 7.61 (d, 2H, *J* 8.9 Hz) ppm (Figure S65). ^{13}C NMR (DMSO-d_6): δ 24.0, 86.3, 121.1, 137.3, 139.1, 168.5 ppm (Figure S66). MS: *m/z* 262 (M^++ 1), 261 (M^+), 219 (100%), 92, 65, 43 (Figure S67).

4-Iodotoluene[13] and 2-iodotoluene (3:2 by HRGC-MS)

Colorless liquid (91% yield). ^{1}H NMR ($CDCl_3$): δ 2.28 (s, 3H), 2.42 (s, 3H), δ 6.81-6.88 (m, 1H), 6.91 (d, 2H, *J* 8.0 Hz), 7.15-7.24 (m, 2H), 7.55 (d, 2H, *J* 8.0 Hz) 7.80 (d, 1H, *J* 8.0 Hz) ppm (Figure S68). ^{13}C NMR ($CDCl_3$): δ 21.1, 28.2, 90.3, 101.3, 127.5, 128.2, 129.8, 131.3, 137.3, 137.5 139.0, 141.4 ppm (Figure S69).

4-Iodotoluene (major)

MS: *m/z* 218 (M^+), 127, 91 (100%), 65, 51 (Figure S70).

2-Iodotoluene (minor)

MS: *m/z* 218 (M^+), 127, 91 (100%), 65, 51 (Figure S71).

1,4-Diiodo-2,3,5,6-tetramethylbenzene[14]

White solid (90% yield); mp 133-135 °C (Lit.[14] 135-137 °C). ^{1}H NMR ($CDCl_3$): δ 2.63 (s, 12H) ppm (Figure S72). ^{13}C NMR ($CDCl_3$): δ 29.9, 112.3, 138.0 ppm (Figure S73). MS: *m/z* 387 (M^++ 1), 386 (M^+, 100%), 259, 132, 117, 115, 91, 77, 65, 51 (Figure S74).

References

1. Barluenga, J.; Rodriguez, M. A.; Campos, P. J.; *J. Chem.Soc., Perkin Trans. 1* **1990**, 2807.
2. Villegas, R. A. S.; Santo Jr., J. L. E.; de Mattos, M. C. S.; de Aguiar, M. R. M. P.; Guarino, A. W. S.; *Catal. Commun*. **2007**, *8*, 97.
3. Cambie, R. C.; Hayward, R. C.; Roberts, J. L.; Rutledge, P. S.; *J. Chem Soc., Perkin Trans. 1* **1974**, 1858.
4. Villegas, R. A. S.; Santo Jr., J. L. E., Sanseverino, A. M.; de Mattos, M. C. S.; de Aguiar, M. R. M. P.; Guarino, A. W. S.; *Synth. Commun.* **2005**, *35*, 1627.
5. Davidson, R. I.; Kropp, P. J.; *J. Org. Chem.* **1982**, *47*, 1904.
6. Villegas, R. A. S.; de Aguiar, M. R. M. P.; de Mattos, M. C. S.; Guarino, A. W. S.; Barbosa, L. M.; Assumpção, L. C. F. N.; *J. Braz. Chem. Soc.* **2004**, *15*, 150.
7. Sanseverino, A. M.; de Mattos, M. C. S.; *Synthesis* **1998**, 1584.
8. de Mattos, M. C. S.; Sanseverino, A. M.; *J. Chem. Res.* **2004**, 638.
9. Ribeiro, R. S.; Esteves, P. M.; de Mattos, M. C. S.; *Tetrahedron Lett.* **2007**, *48*, 8747.

10. Jereb, M.; Zupan, M.; Stavber, S.; *Green Chem.* **2005**, *7*, 100.
11. Vaghei, R. G.; *Tetrahedron Lett.* **2003**, *44*, 7529.
12. Iskra, J.; Stavber, S.; Zupan, M.; *Synthesis* **2004**, 1869.
13. Ribeiro, R. S.; Esteves, P. M.; de Mattos, M. C. S.; *J. Braz. Chem. Soc.* **2008**, *19*, 1239.
14. Yusubov, M. S.; Funk, T. V.; Chi, K.-W.; Cha, E. H.; Kim, G. M.; Kirschning, A.; Zhdankin, V. V.; *J. Org. Chem.* **2008**, *73*, 295.

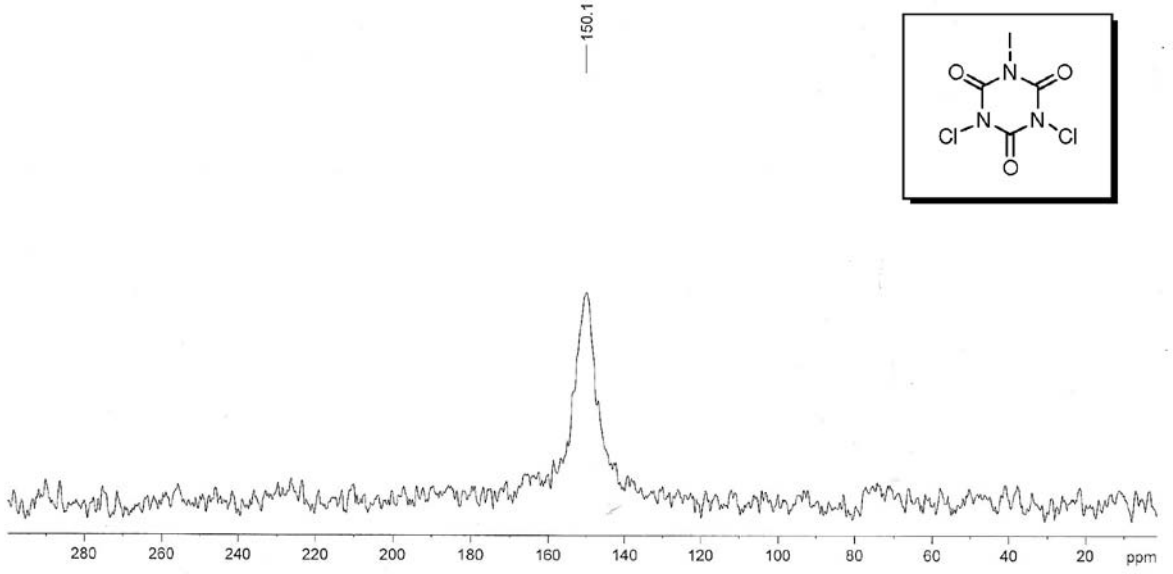

Figure S1. ^{13}C MA-NMR spectrum of dichloroiodoisocyanuric acid (DCICA) (300 MHz).

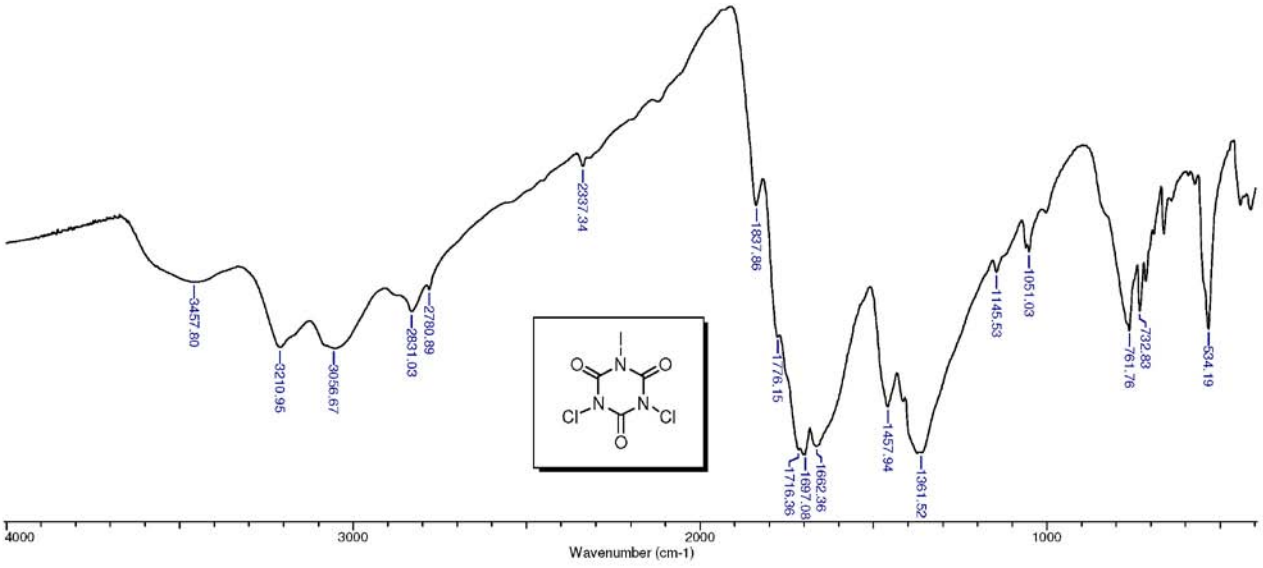

Figure S2. IR spectrum of dichloroiodoisocyanuric acid (DCICA) (KBr).

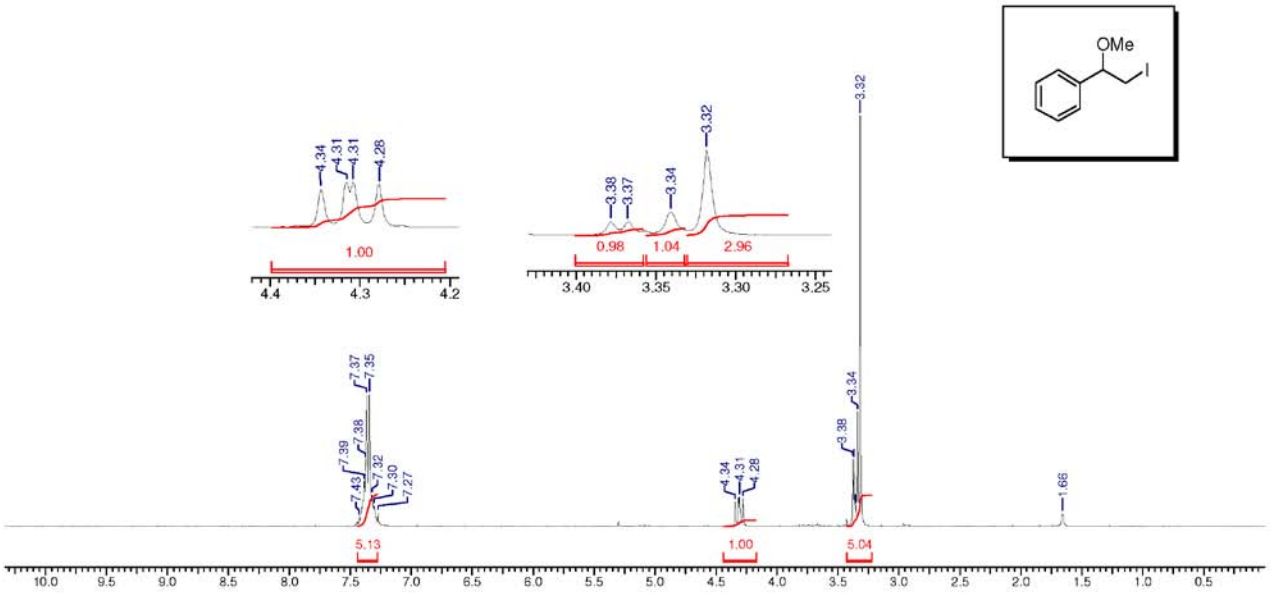

Figure S3. [1]H NMR spectrum of 1-methoxy-1-phenyl-2-iodoethane ($CDCl_3$, TMS, 200 MHz).

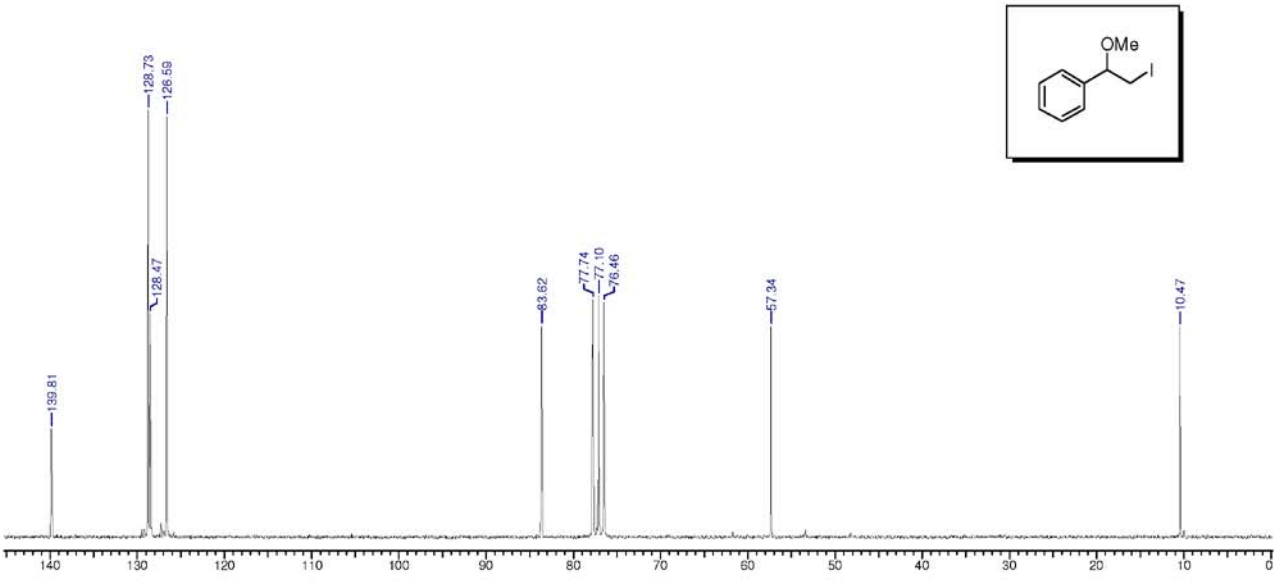

Figure S4. [13]C NMR spectrum of 1-methoxy-1-phenyl-2-iodoethane ($CDCl_3$, TMS, 50 MHz).

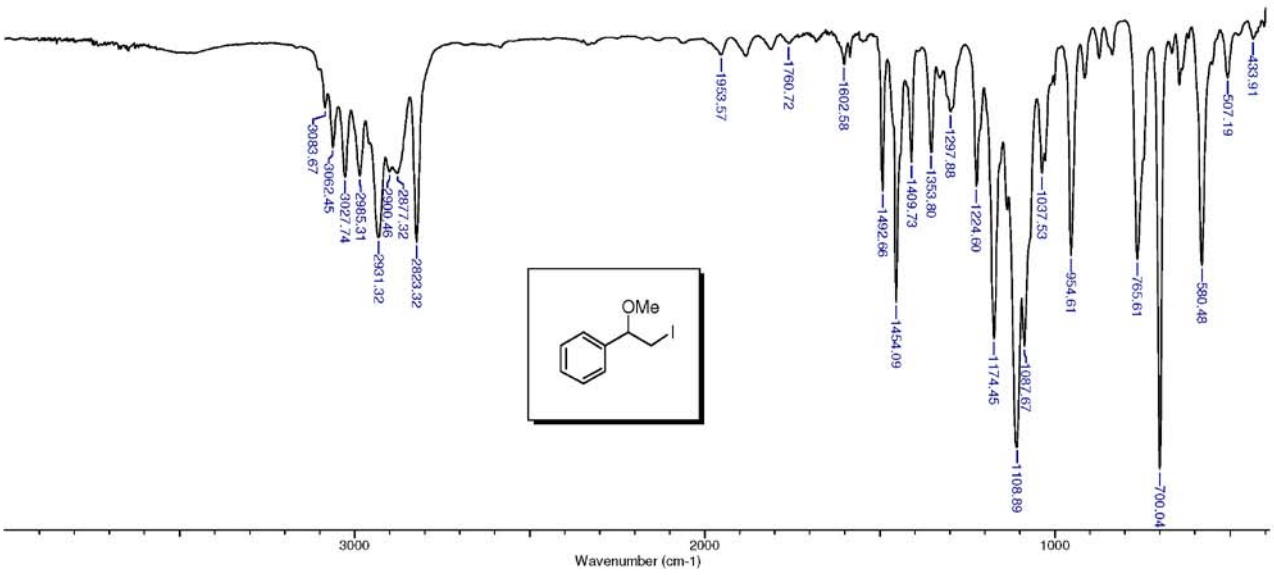

Figure S5. IR spectrum of 1-methoxy-1-phenyl-2-iodoethane (film).

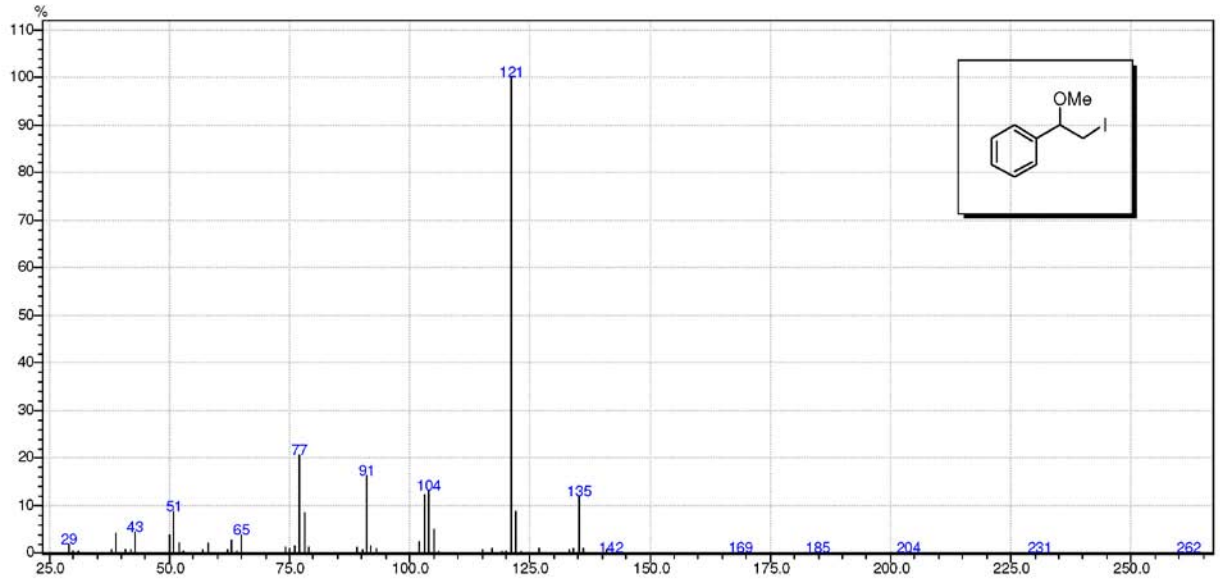

Figure S6. Mass spectrum of 1-methoxy-1-phenyl-2-iodoethane (70 eV).

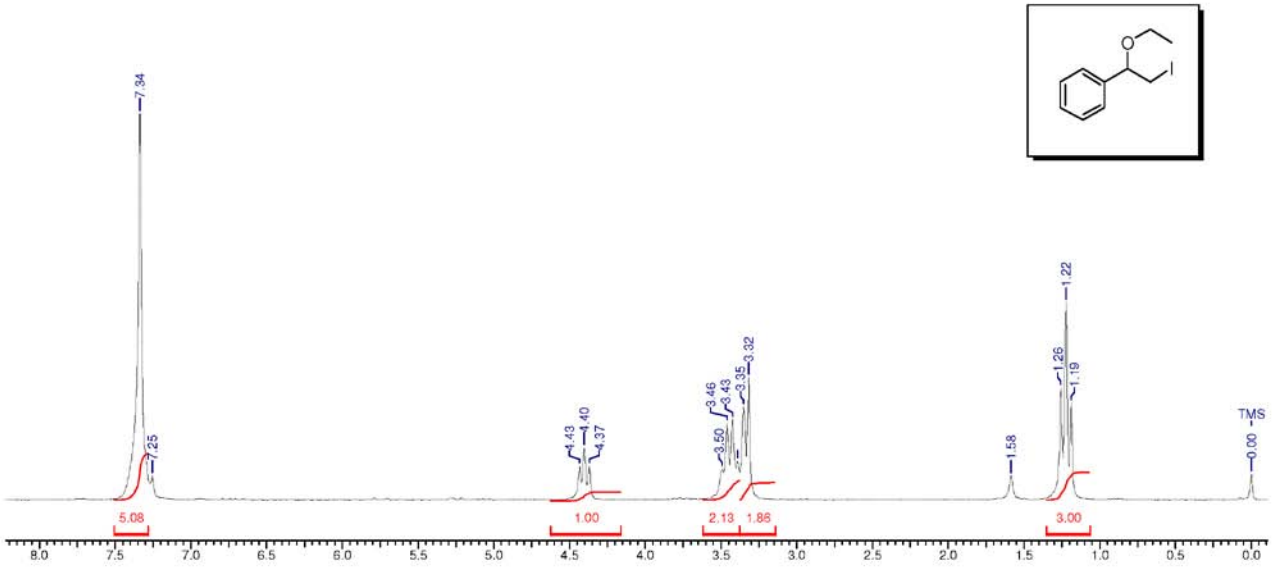

Figure S7. [1]H NMR spectrum of 1-ethoxy-1-phenyl-2-iodoethane ($CDCl_3$, TMS, 200 MHz).

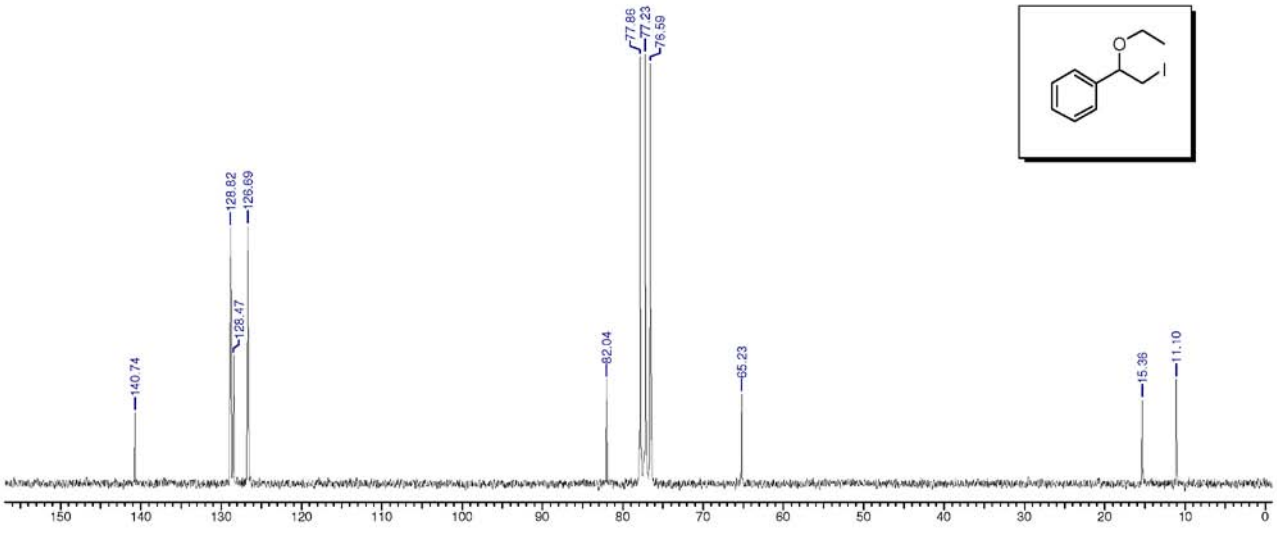

Figure S8. [13]C NMR spectrum of 1-ethoxy-1-phenyl-2-iodoethane ($CDCl_3$, TMS, 50 MHz).

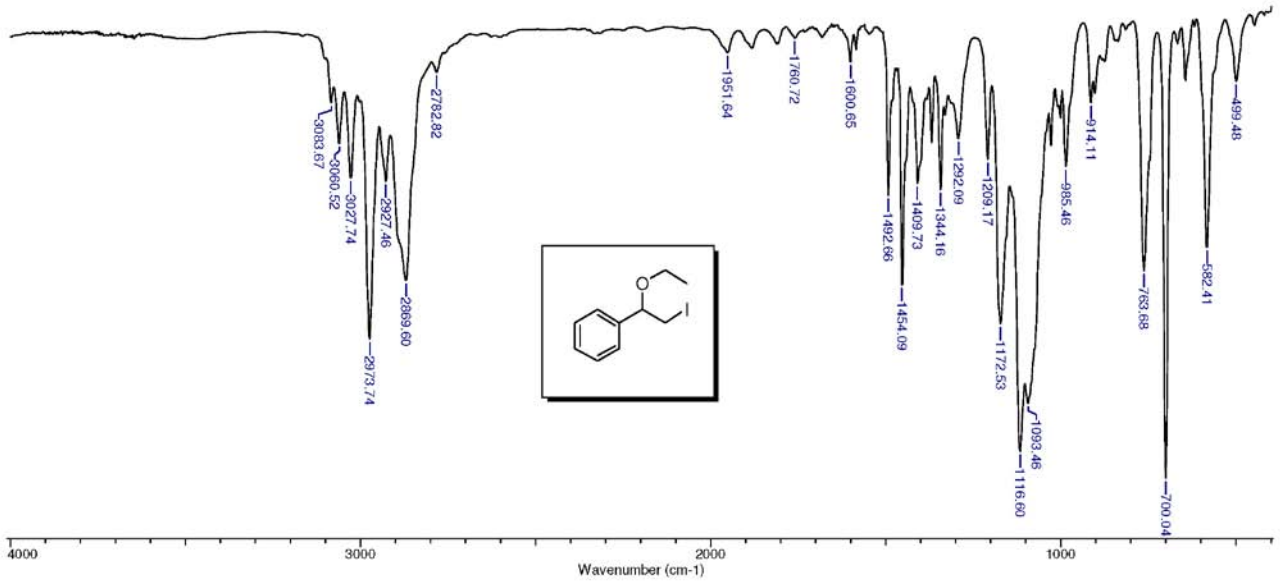

Figure S9. IR spectrum of 1-ethoxy-1-phenyl-2-iodoethane (film).

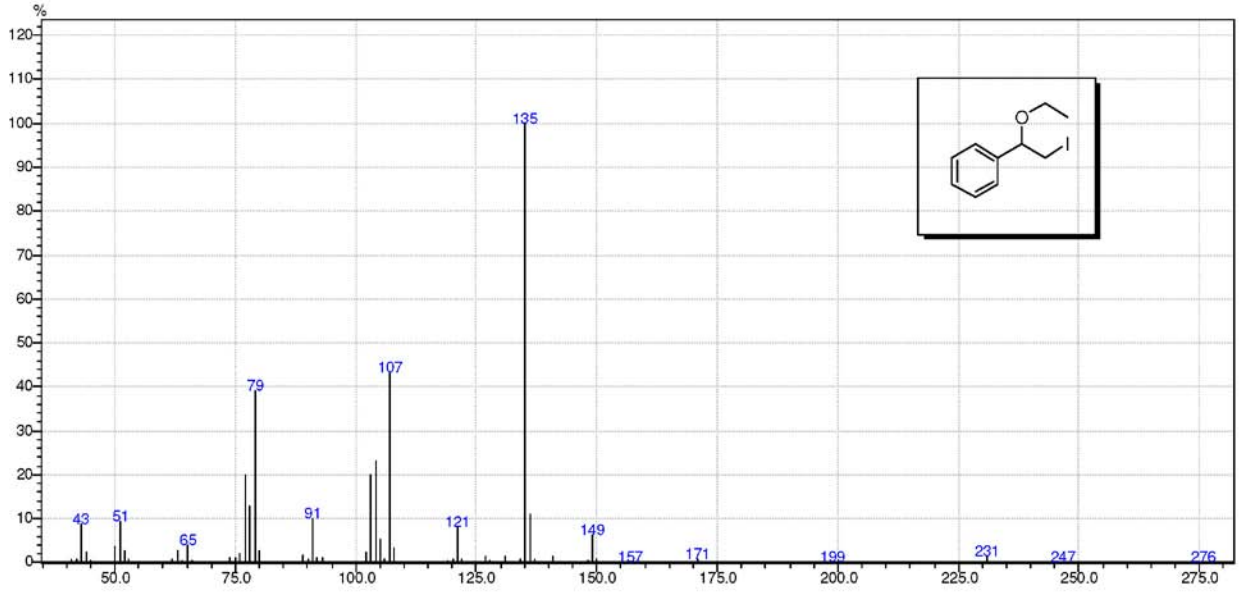

Figure S10. Mass spectrum of 1-ethoxy-1-phenyl-2-iodoethane (70 eV).

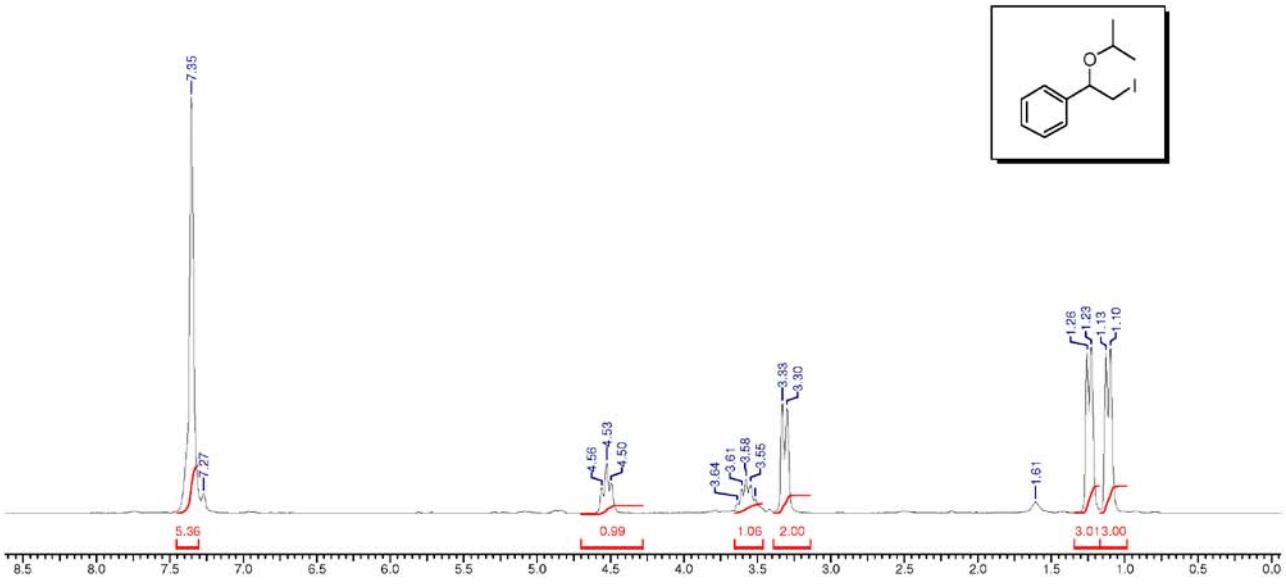

Figure S11. ^{1}H NMR spectrum of 2-iodo-1-phenyl-1-isopropoxyethane ($CDCl_3$, TMS, 200 MHz).

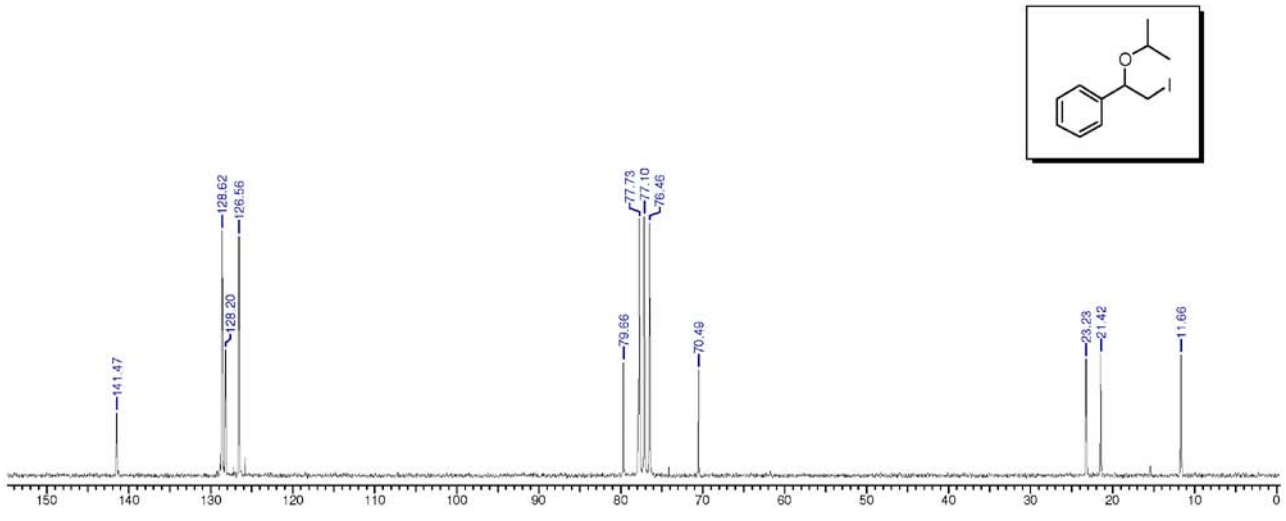

Figure S12. ^{13}C NMR spectrum of 2-iodo-1-phenyl-1-isopropoxyethane ($CDCl_3$, TMS, 50 MHz).

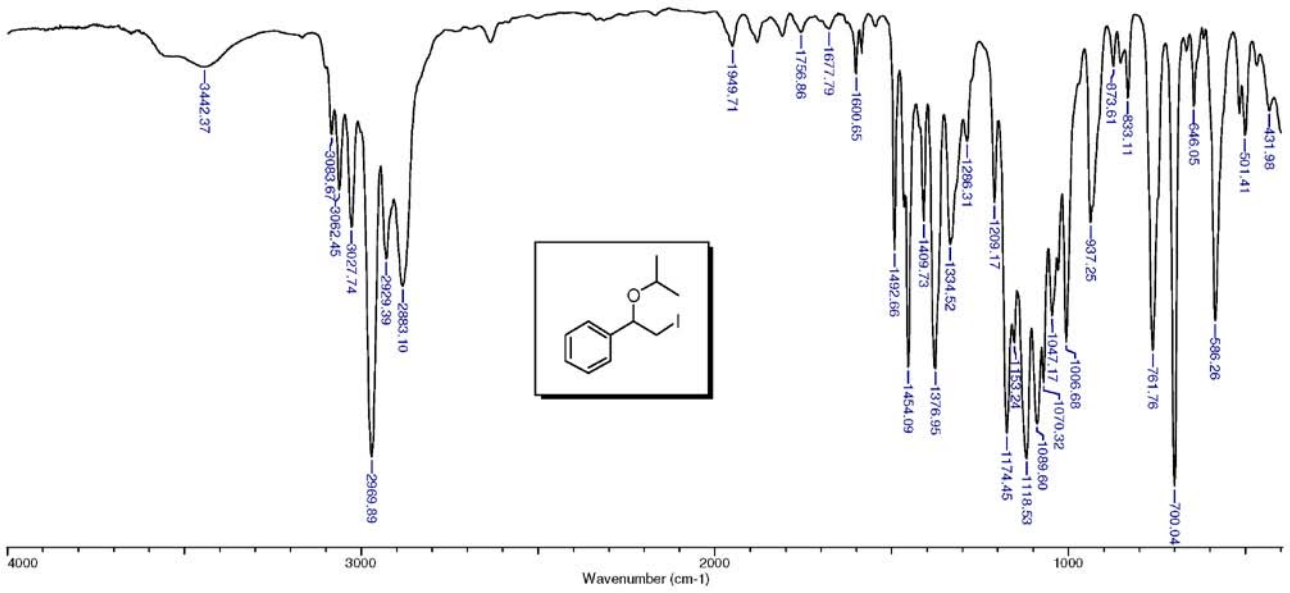

Figure S13. IR spectrum of 2-iodo-1-phenyl-1-isopropoxyethane (film).

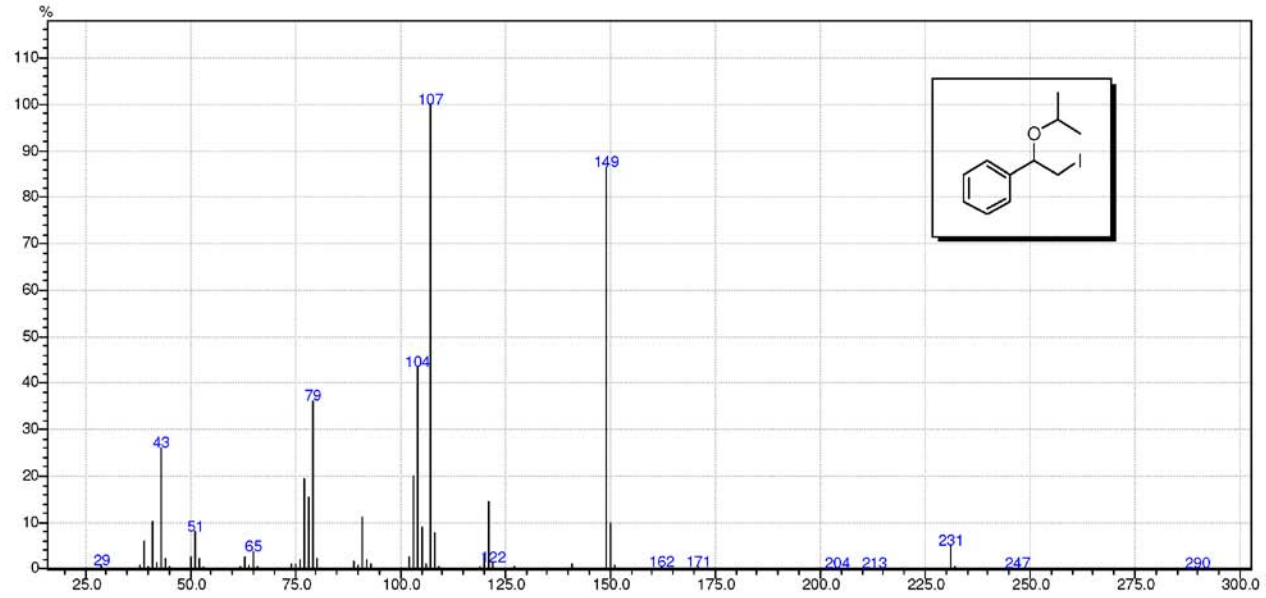

Figure S14. Mass spectrum of 2-iodo-1-phenyl-1-isopropoxyethane (70 eV).

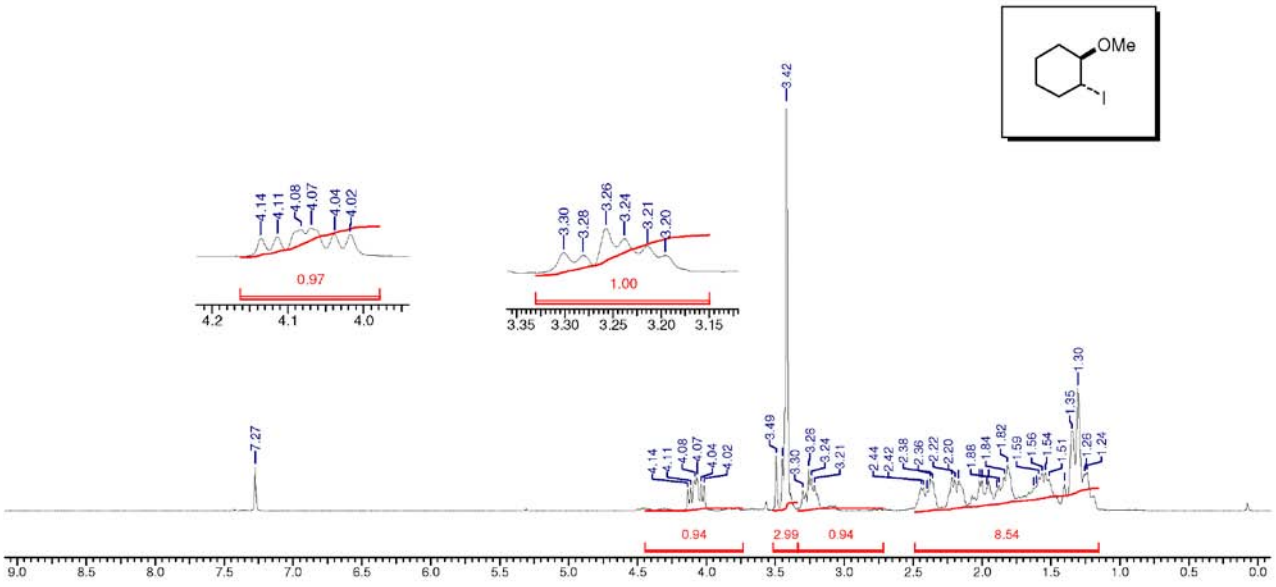

Figure S15. ^{1}H NMR spectrum of *trans*-1-iodo-2-methoxycyclohexane ($CDCl_3$, TMS, 200 MHz).

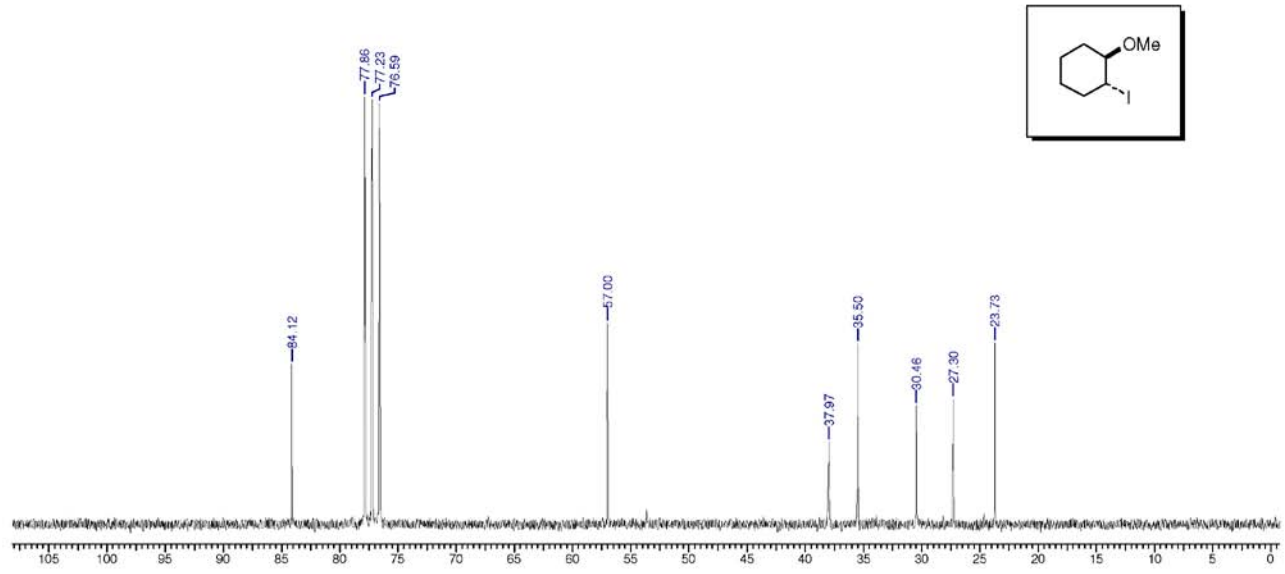

Figure S16. ^{13}C NMR spectrum of *trans*-1-iodo-2-methoxycyclohexane ($CDCl_3$, TMS, 50 MHz).

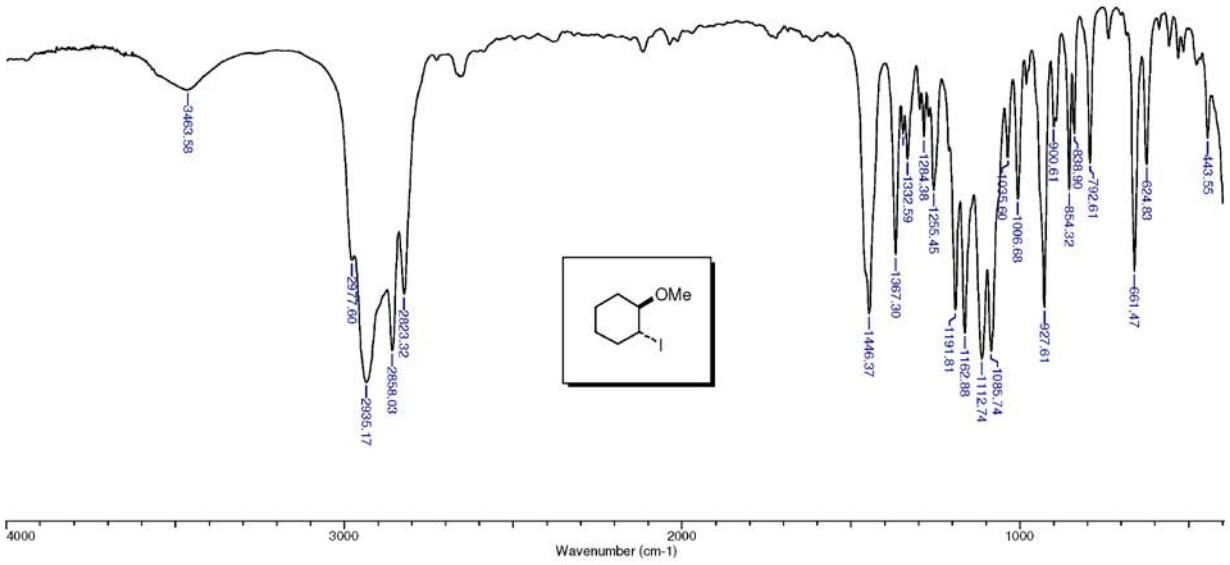

Figure S17. IR spectrum of *trans*-1-iodo-2-methoxycyclohexane (film).

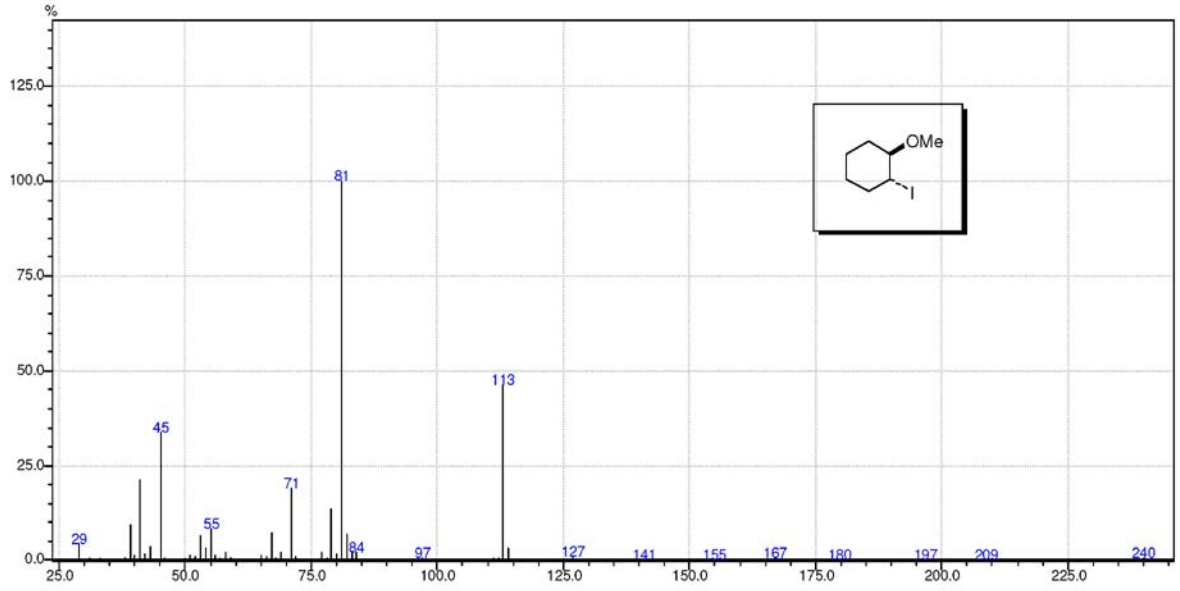

Figure S18. Mass spectrum of *trans*-1-iodo-2-methoxycyclohexane (70 eV).

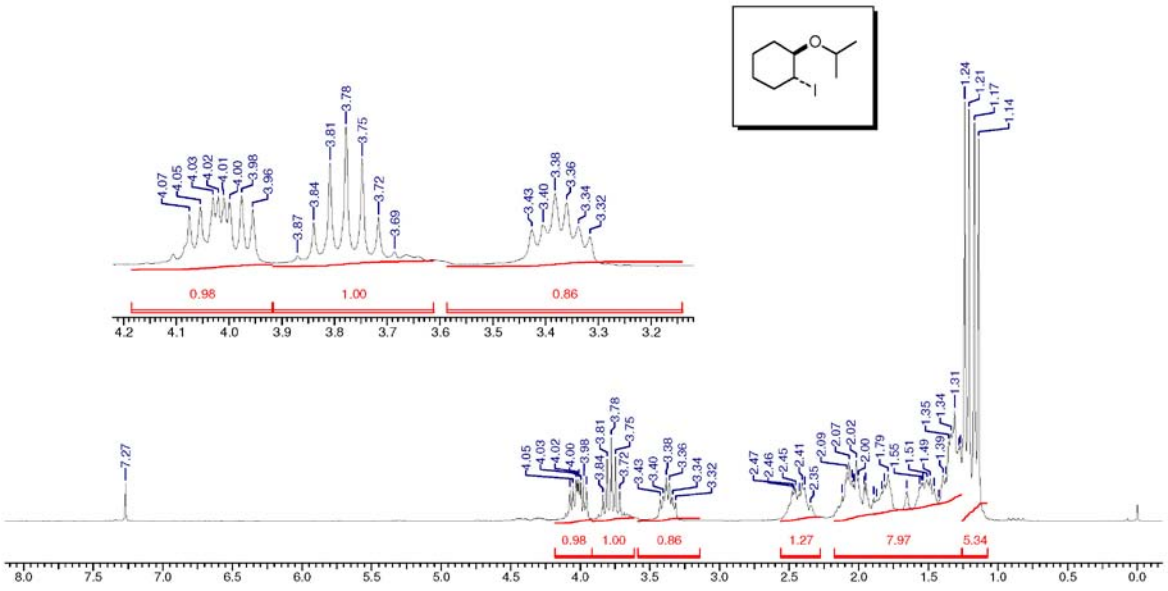

Figure S19. ^{1}H NMR spectrum of *trans*-1-iodo-2-isopropoxycyclohexane ($CDCl_3$, TMS, 200 MHz).

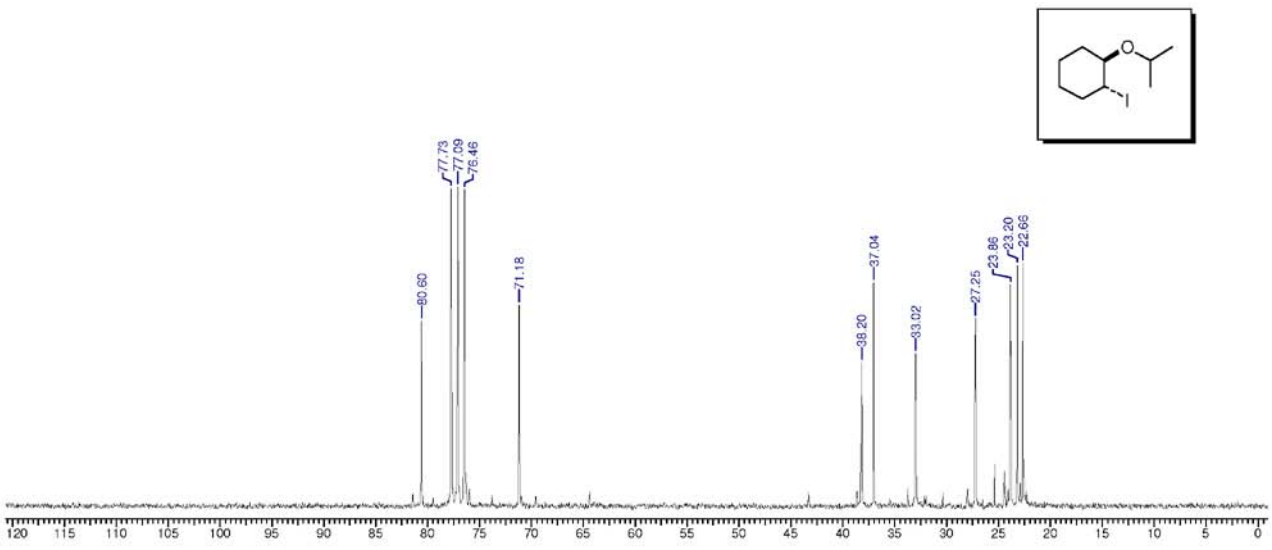

Figure S20. ^{13}C NMR spectrum of *trans*-1-iodo-2-isopropoxycyclohexane ($CDCl_3$, TMS, 50 MHz).

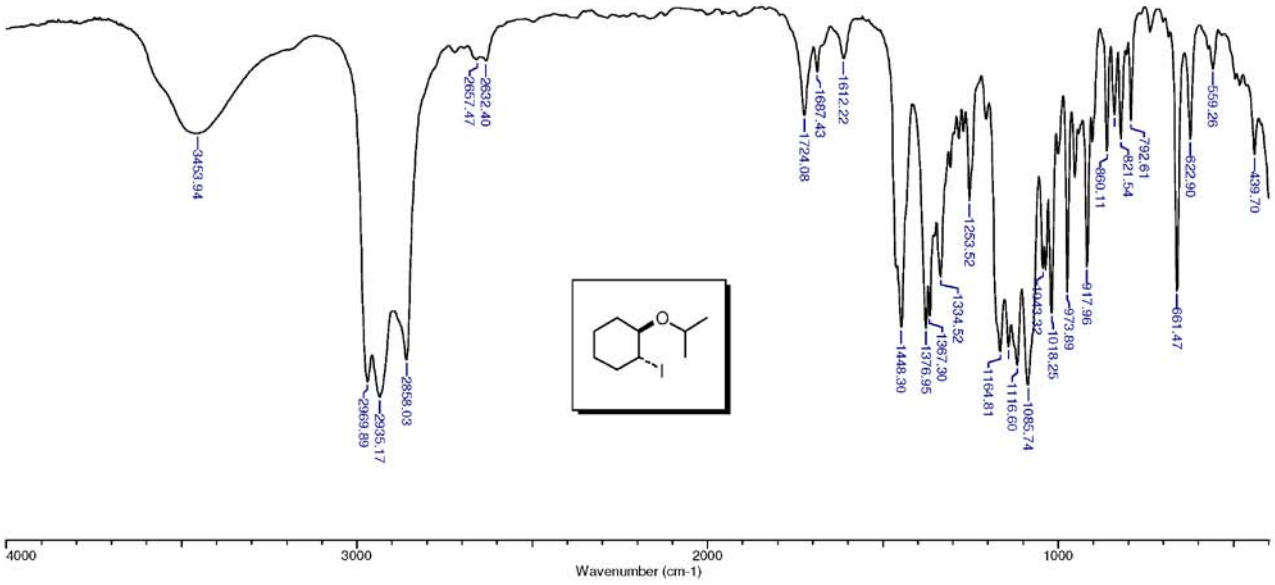

Figure S21. IR spectrum of *trans*-1-iodo-2-isopropoxycyclohexane (film).

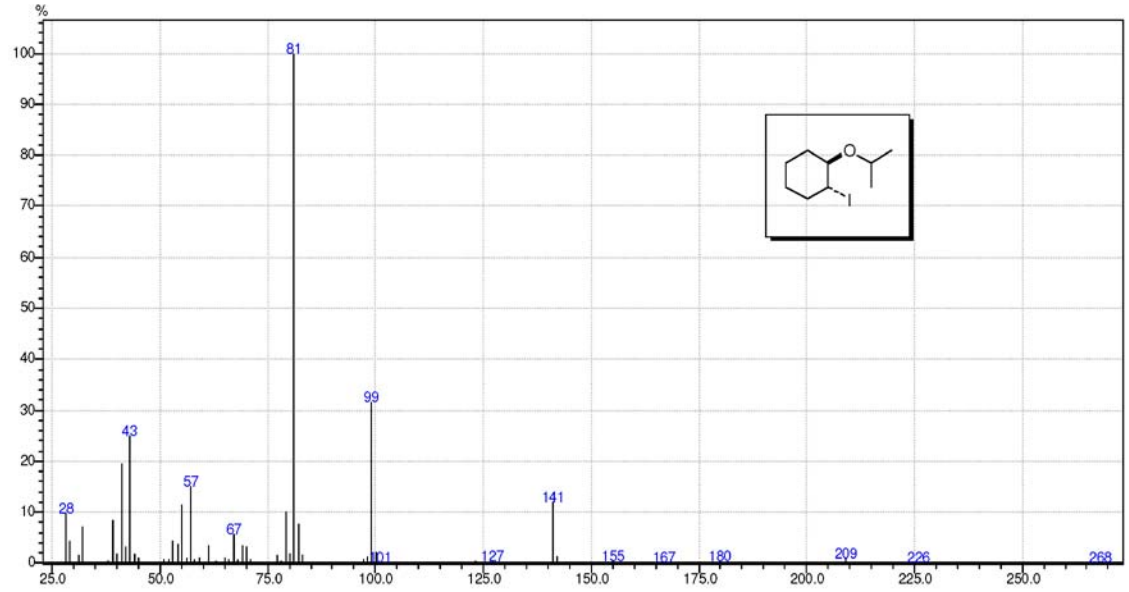

Figure S22. Mass spectrum of *trans*-1-iodo-2-isopropoxycyclohexane (70 eV).

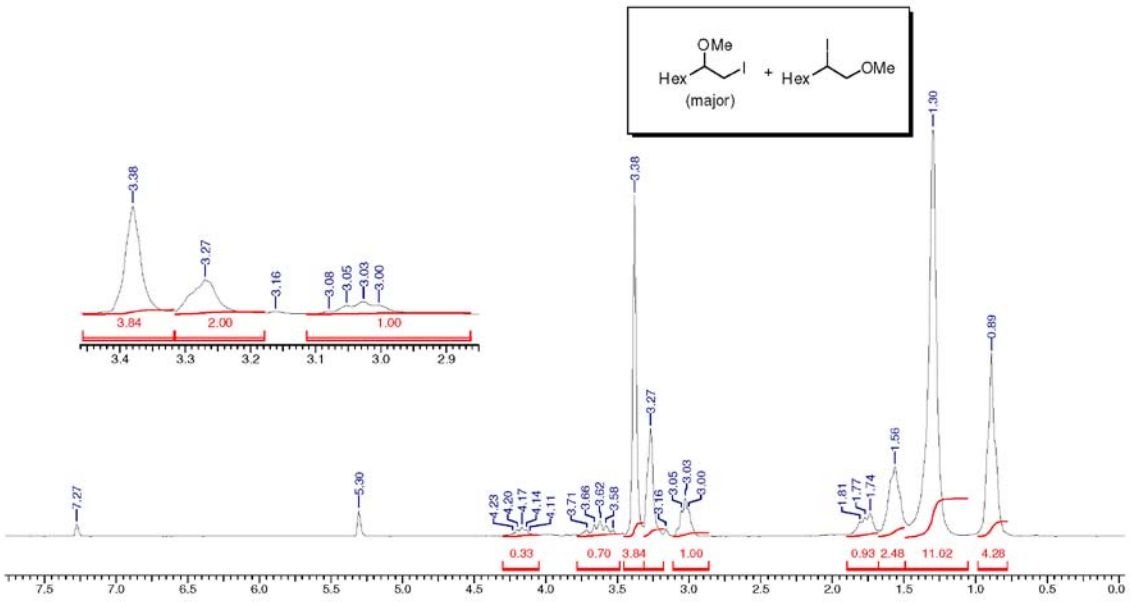

Figure S23. ^{1}H NMR spectrum of 1-iodo-2-methoxyoctane and 2-iodo-1-methoxyoctane ($CDCl_3$, TMS, 200 MHz).

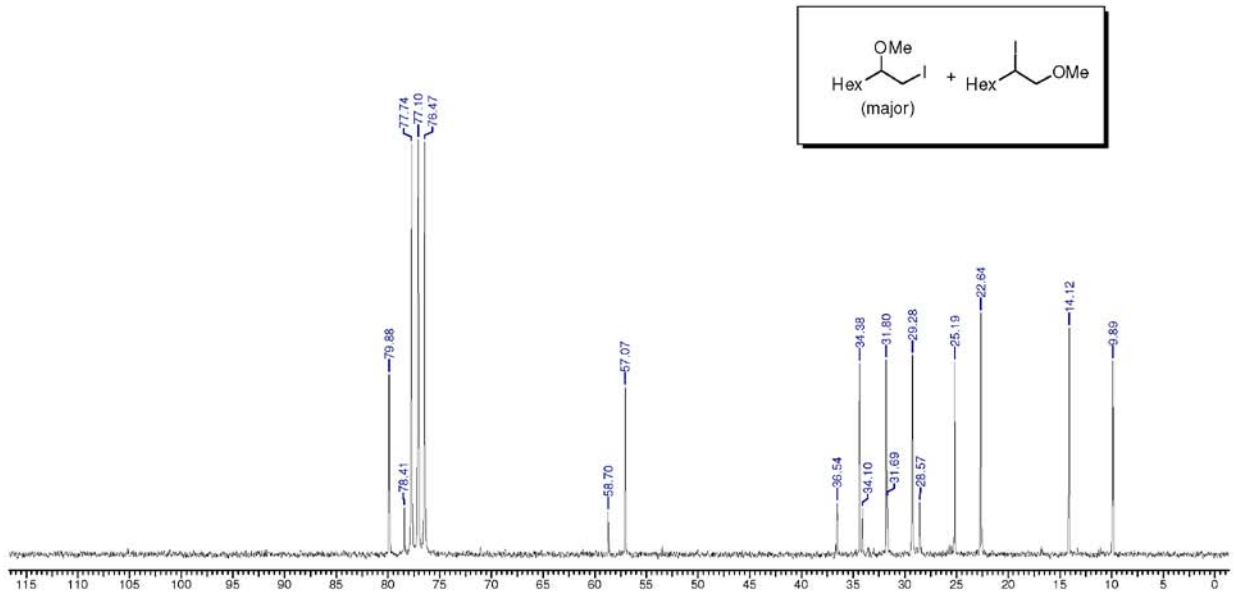

Figure S24. ^{13}C NMR spectrum of 1-iodo-2-methoxyoctane and 2-iodo-1-methoxyoctane ($CDCl_3$, TMS, 50 MHz).

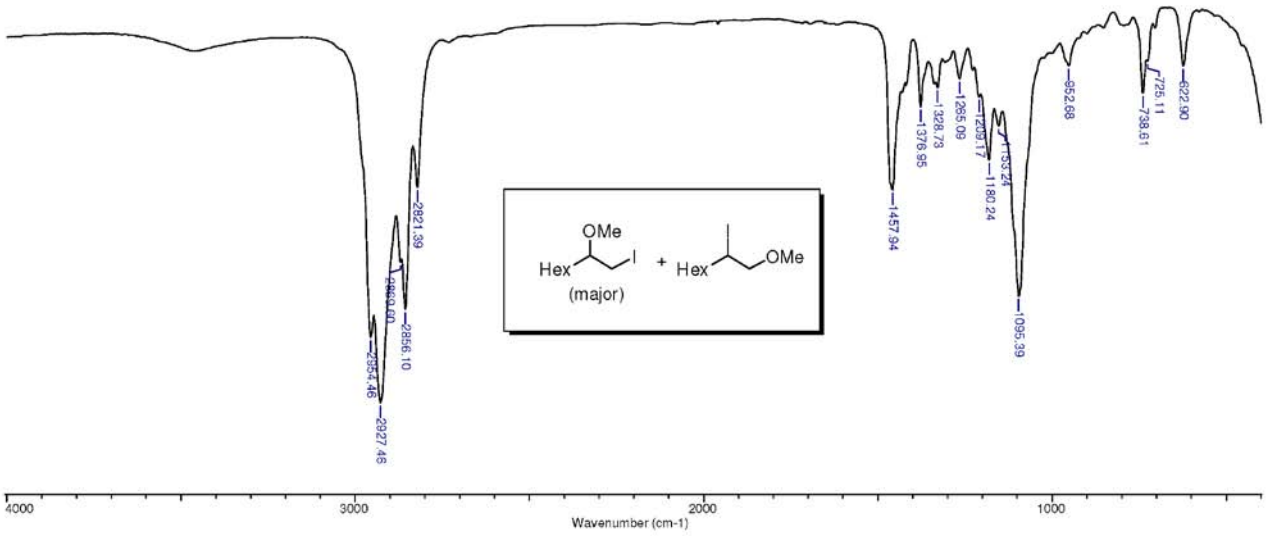

Figure S25. IR spectrum of 1-iodo-2-methoxyoctane and 2-iodo-1-methoxyoctane (film).

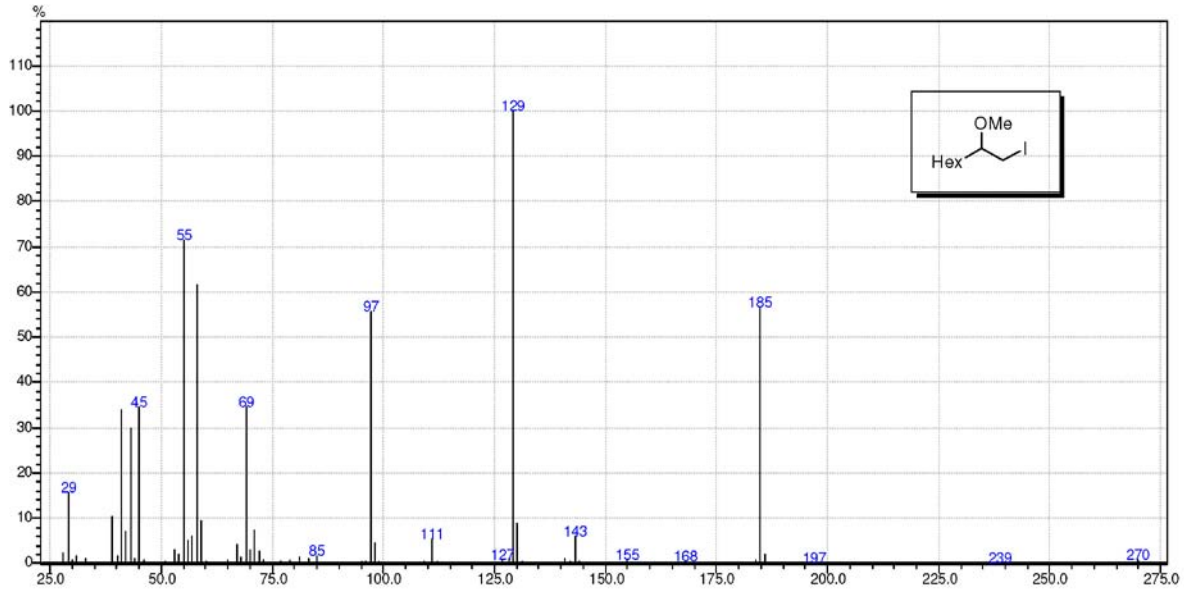

Figure S26. Mass spectrum of 1-iodo-2-methoxyoctane (70 eV).

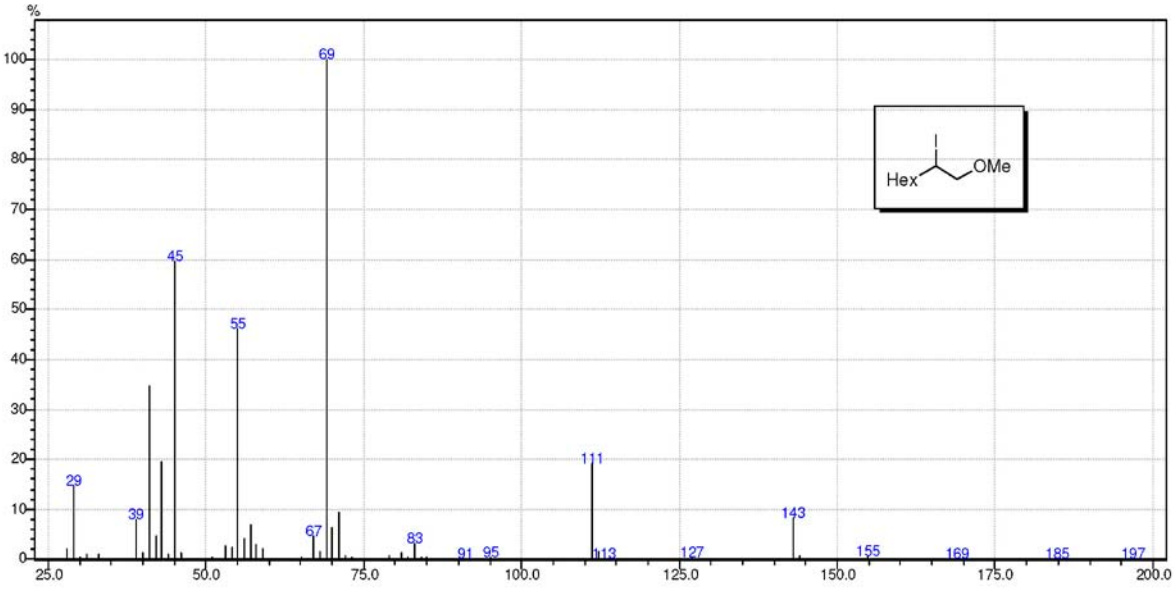

Figure S27. Mass spectrum of 2-iodo-1-methoxyoctane (70 eV).

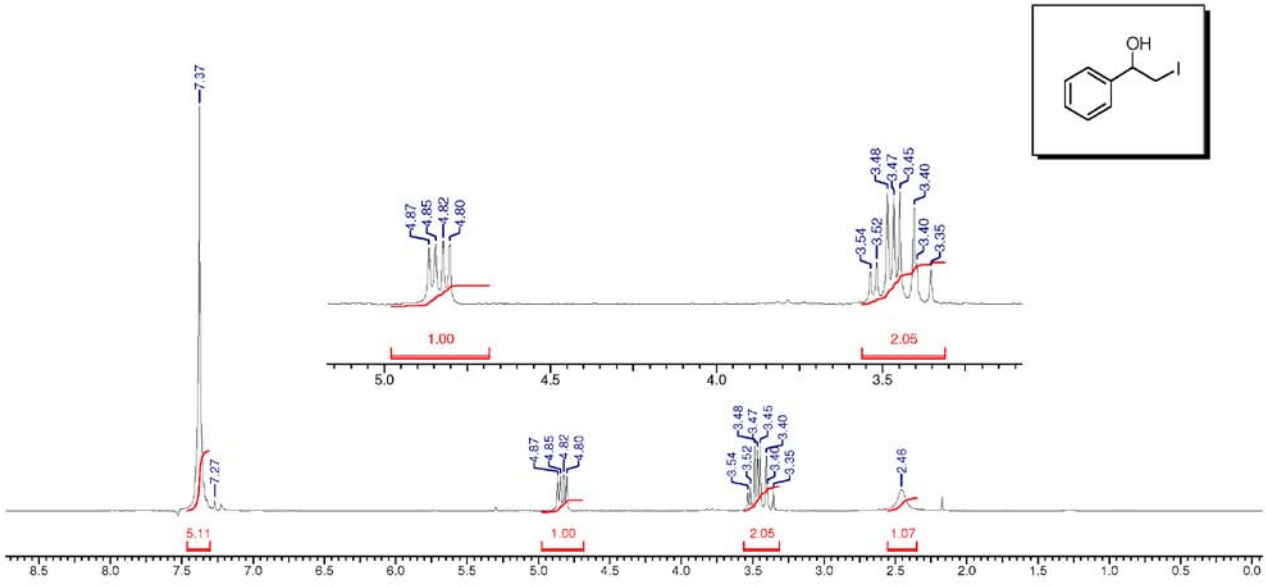

Figure S28. ^{1}H NMR spectrum of 1-phenyl-2-iodoethanol ($CDCl_3$, TMS, 200 MHz)**.**

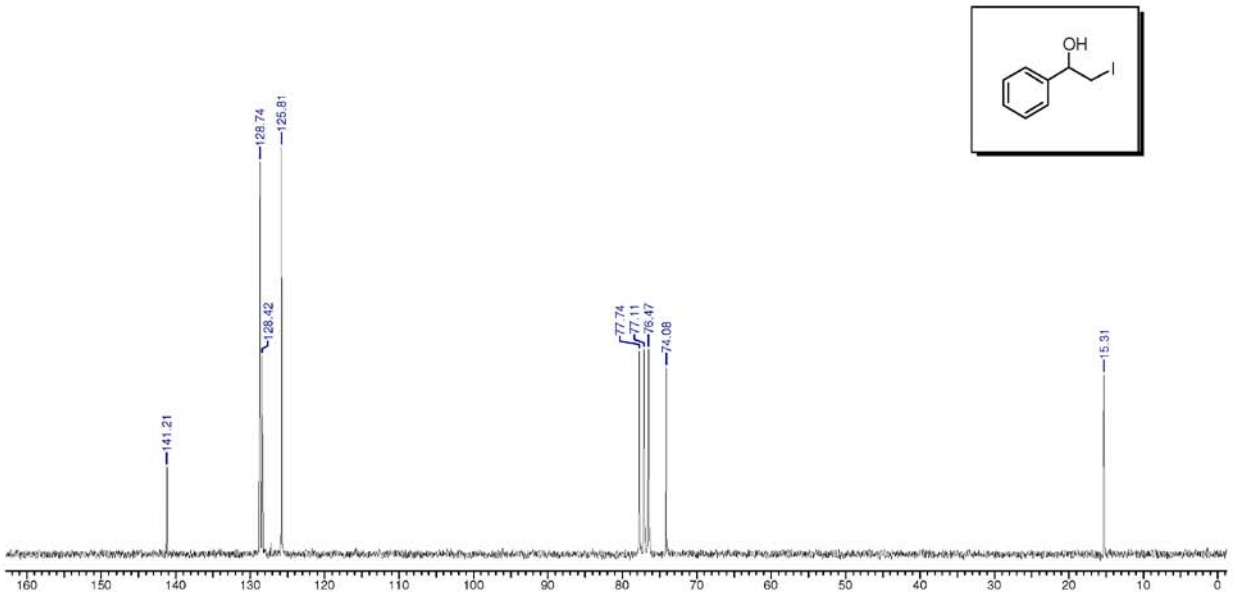

Figure S29. ^{1}H NMR spectrum of 1-phenyl-2-iodoethanol ($CDCl_3$, TMS, 50 MHz).

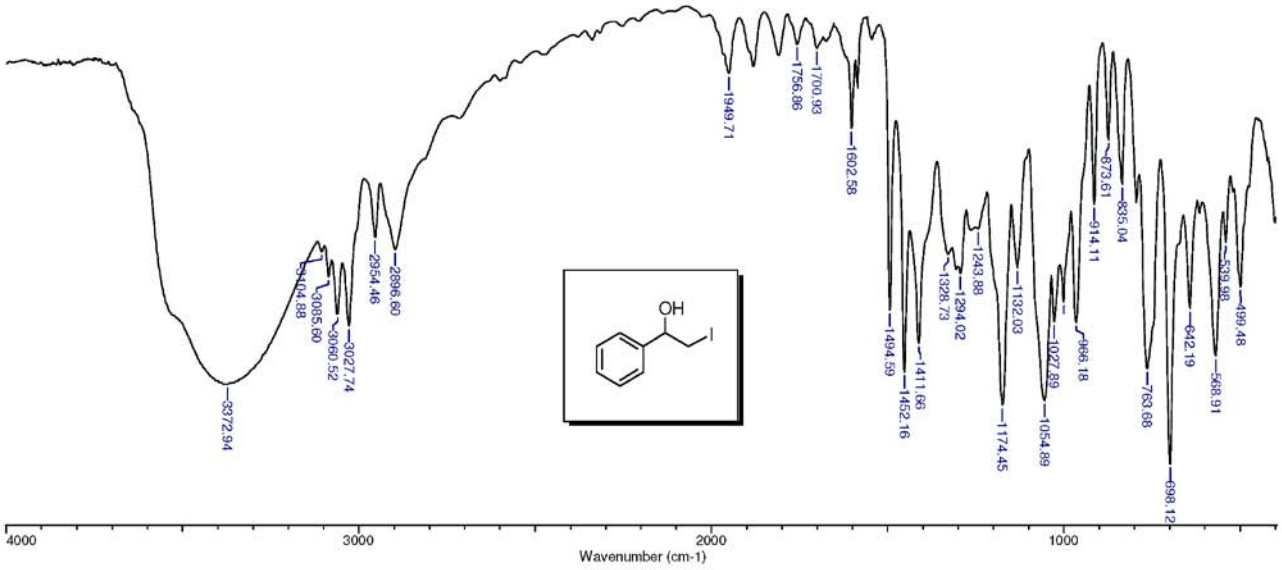

Figure S30. IR spectrum of 1-phenyl-2-iodoethanol (film).

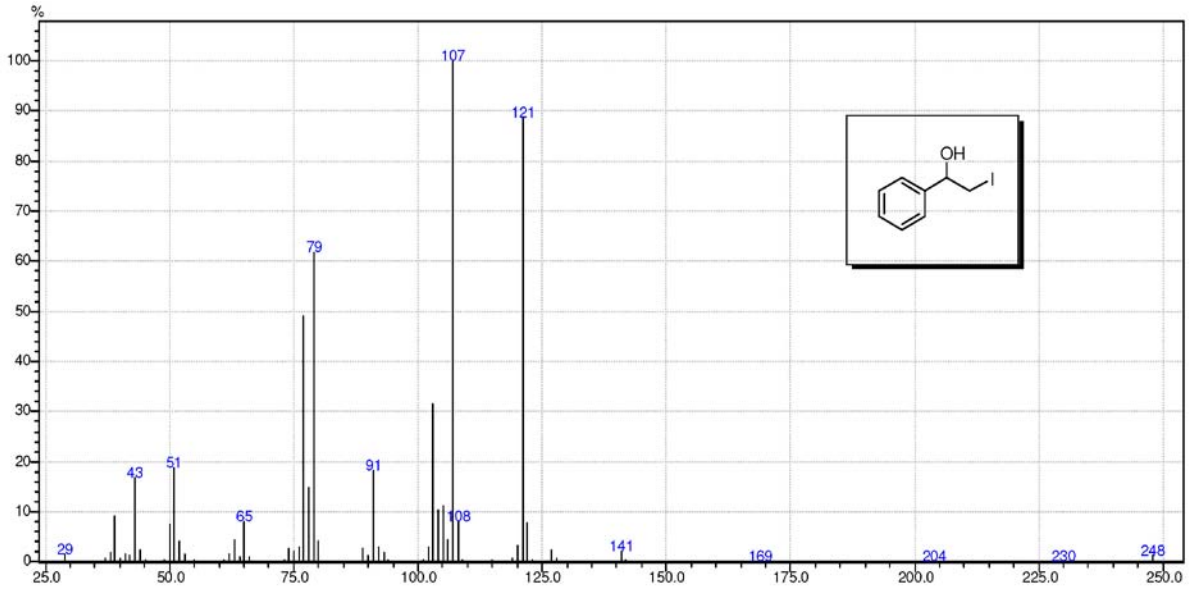

Figure S31. Mass spectrum of 1-phenyl-2-iodoethanol (70 eV).

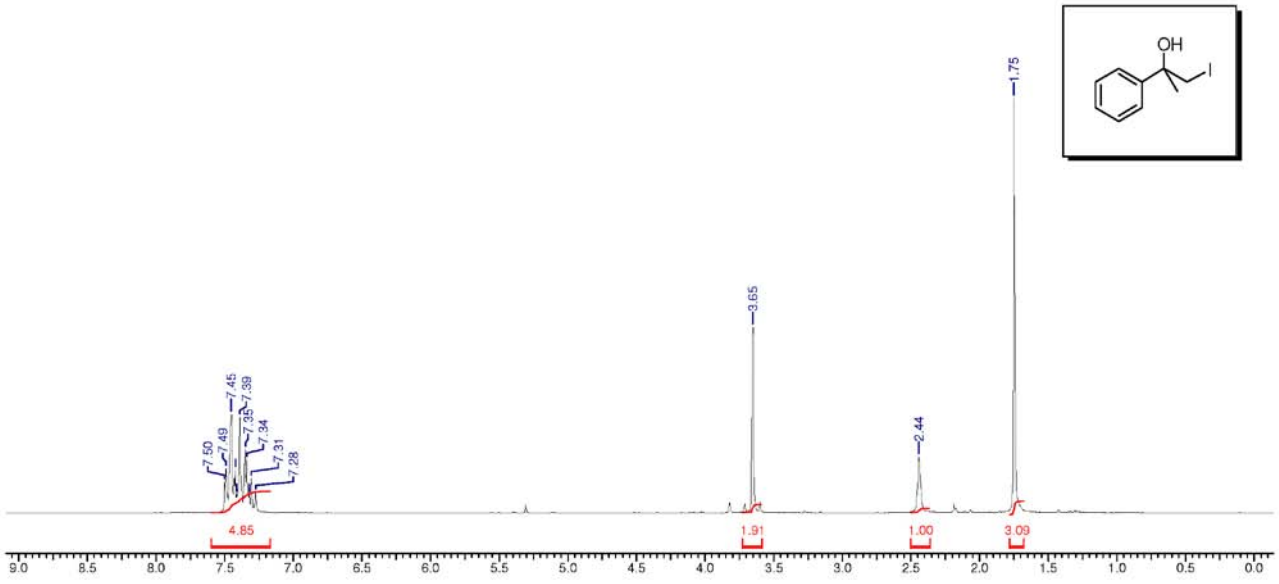

Figure S32. ^{1}H NMR spectrum of 2-phenyl-1-iodo-2-propanol ($CDCl_3$, TMS, 200 MHz).

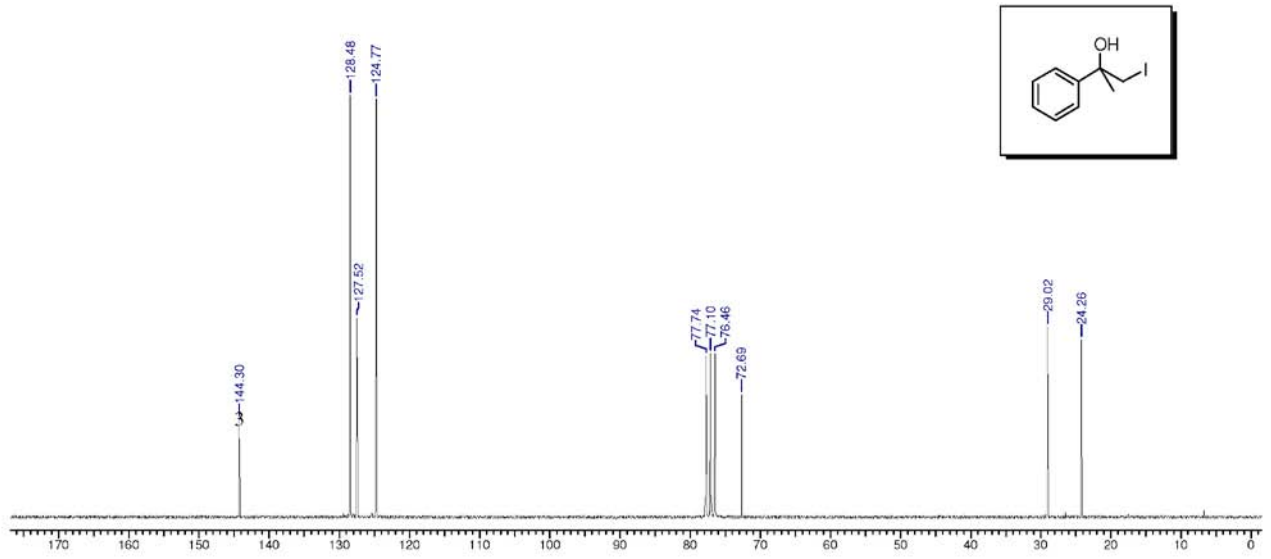

Figure S33. ^{13}C NMR spectrum of 2-phenyl-1-iodo-2-propanol ($CDCl_3$, TMS, 50 MHz).

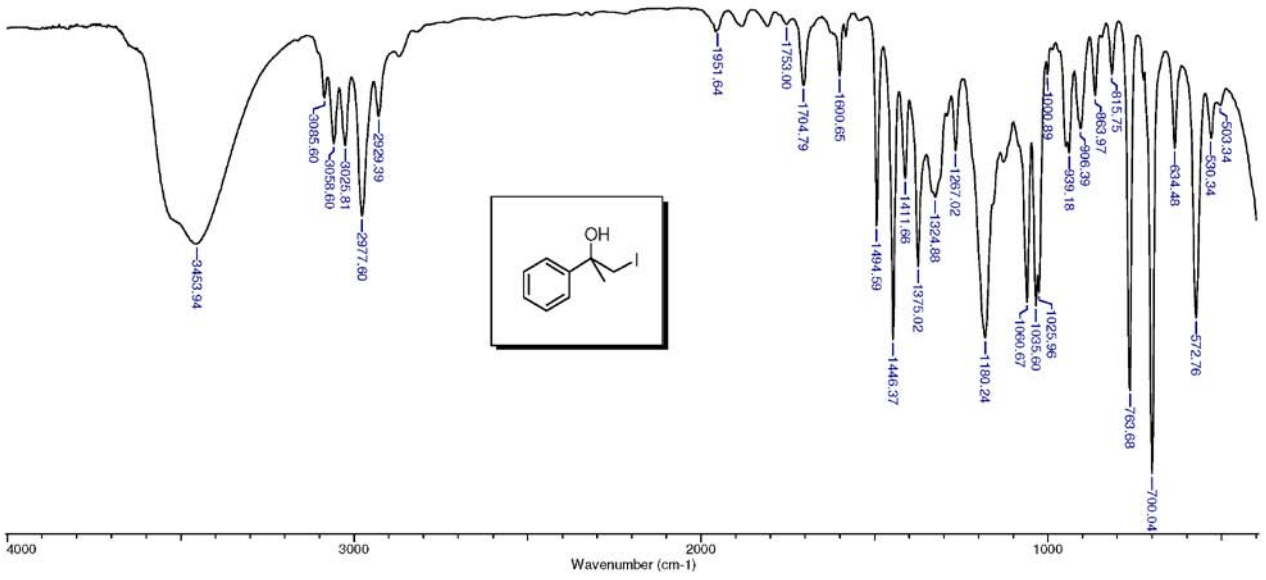

Figure S34. IR spectrum of 2-phenyl-1-iodo-2-propanol (film).

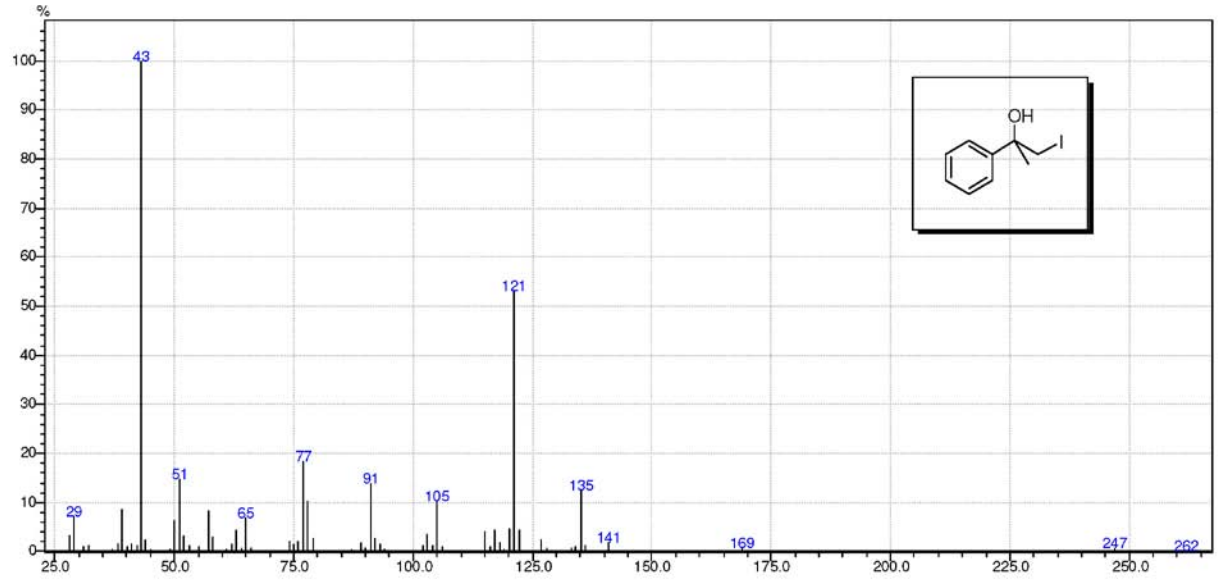

Figure S35. Mass spectrum of 2-phenyl-1-iodo-2-propanol (70 eV).

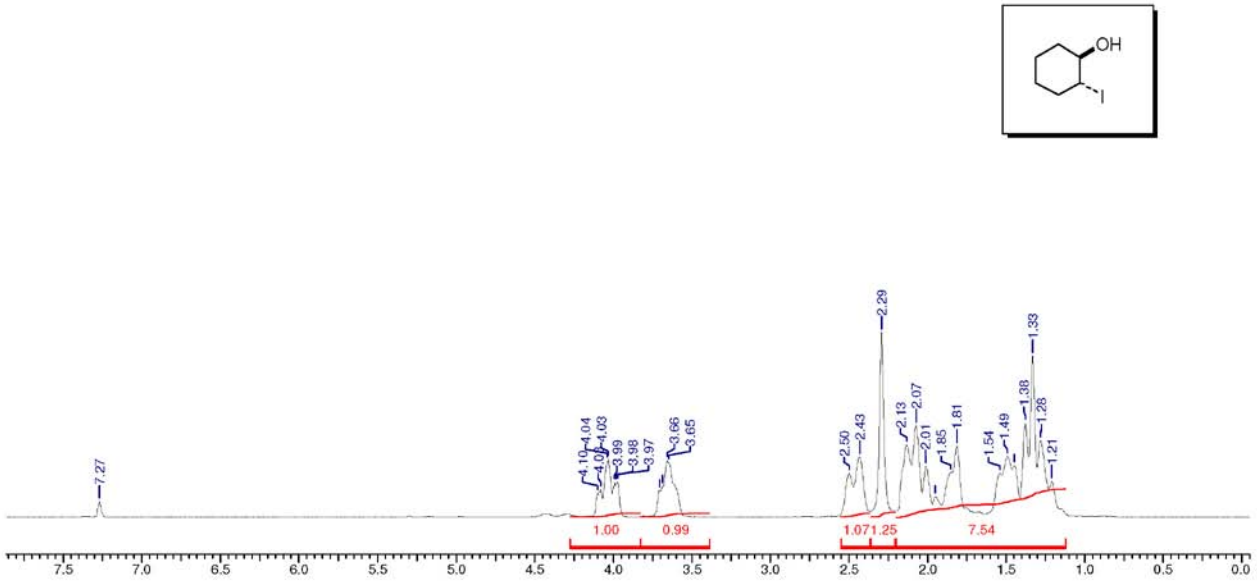

Figure S36. ^{1}H NMR spectrum of *trans*-2-iodocyclohexanol ($CDCl_3$, TMS, 200 MHz).

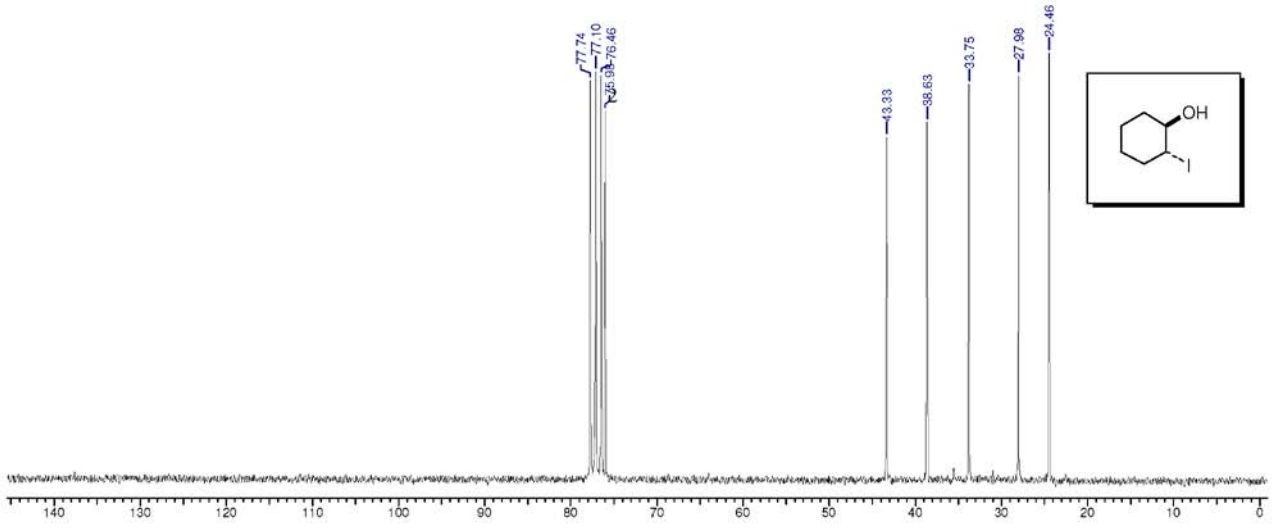

Figure S37. ^{13}C NMR spectrum of *trans*-2-iodocyclohexanol ($CDCl_3$, TMS, 50 MHz).

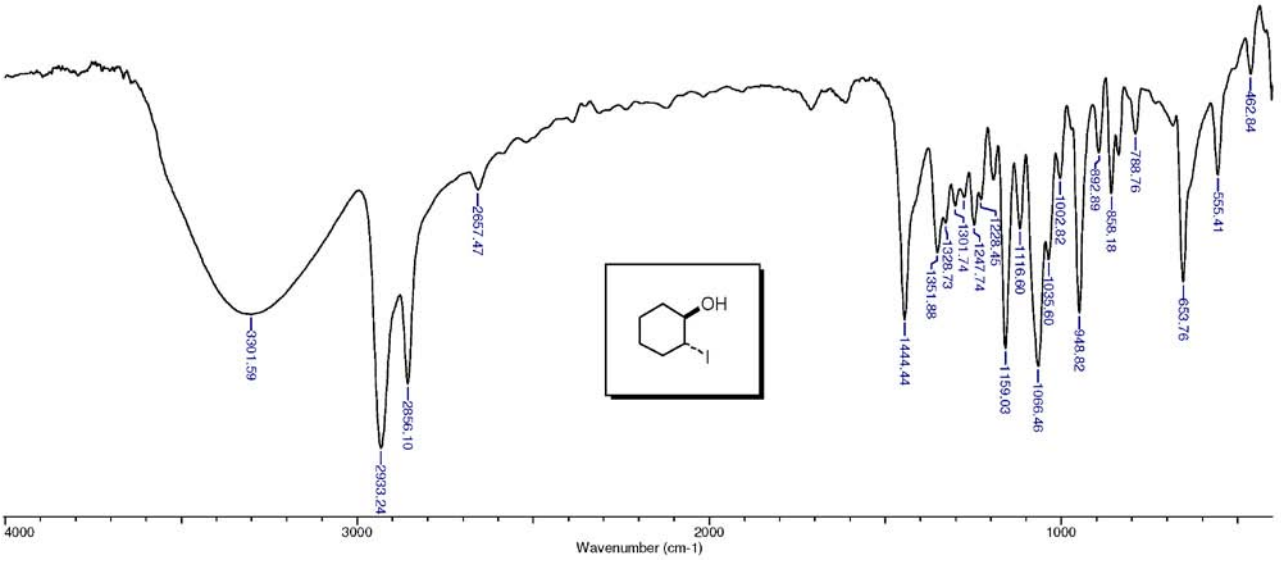

Figure S38. IR spectrum of *trans*-2-iodocyclohexanol (film).

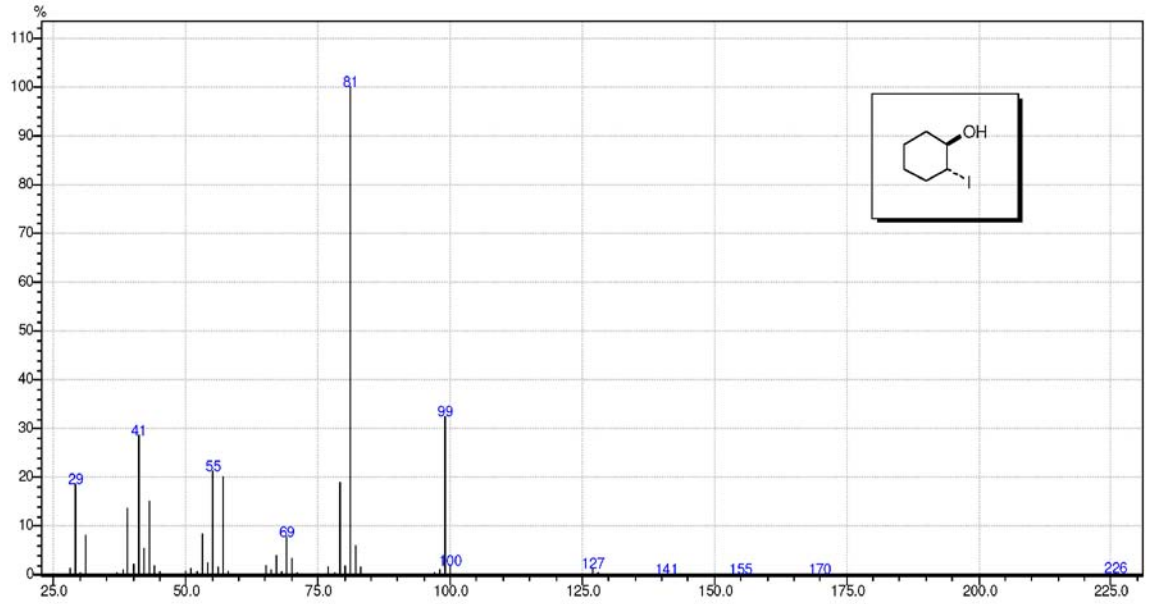

Figure S39. Mass spectrum of *trans*-2-iodocyclohexanol (70 eV).

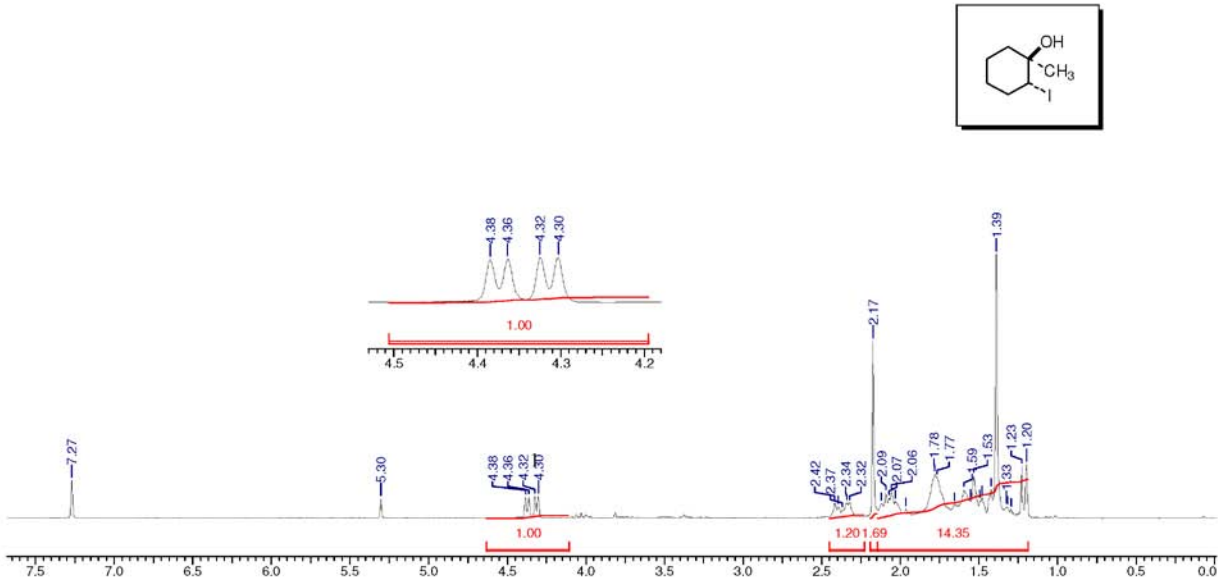

Figure S40. ^{1}H NMR spectrum of *trans*-2-iodo-1-methylcyclohexanol ($CDCl_3$, TMS, 200 MHz).

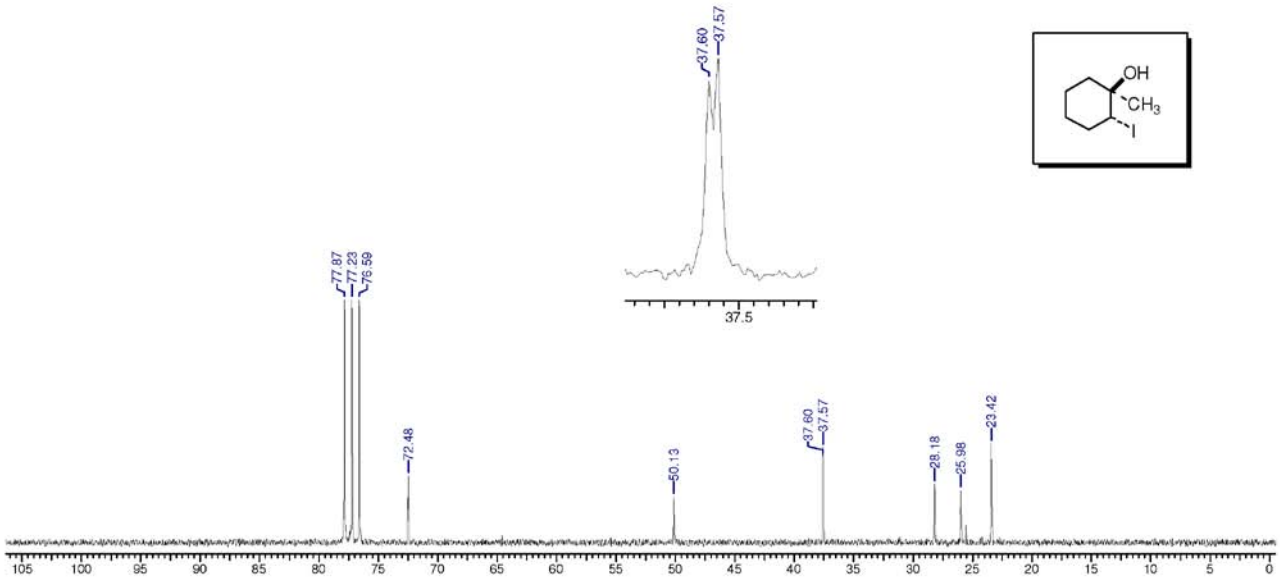

Figure S41. ^{13}C NMR spectrum of *trans*-2-iodo-1-methylcyclohexanol ($CDCl_3$, TMS, 50 MHz).

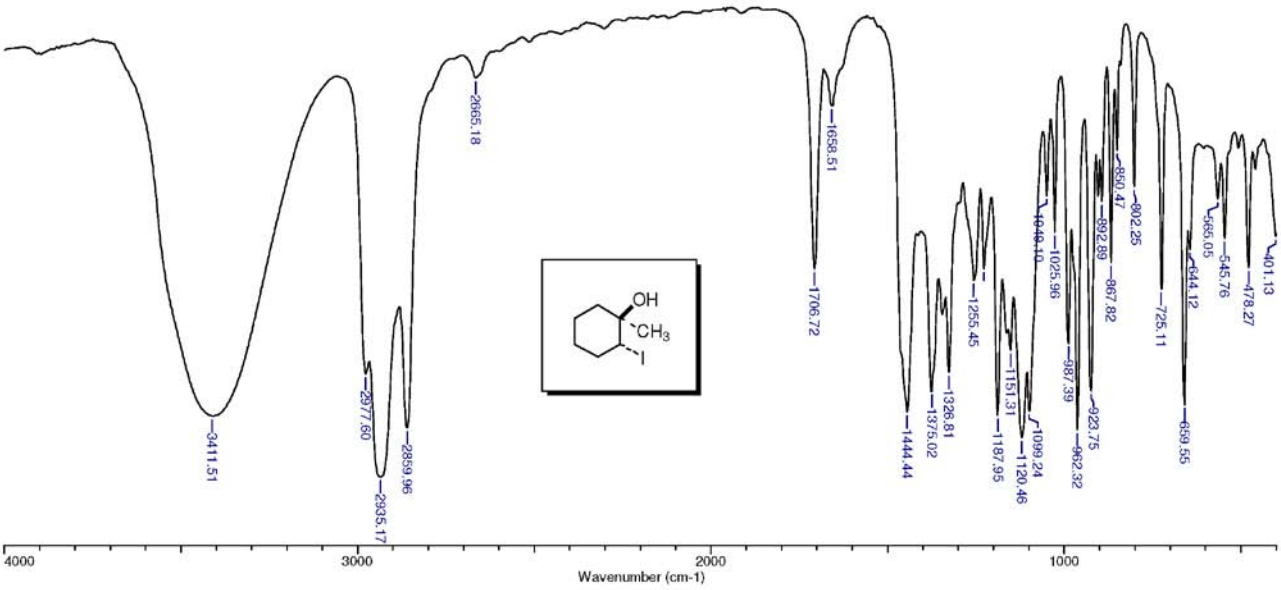

Figure S42. IR spectrum of *trans*-2-iodo-1-methylcyclohexanol (film).

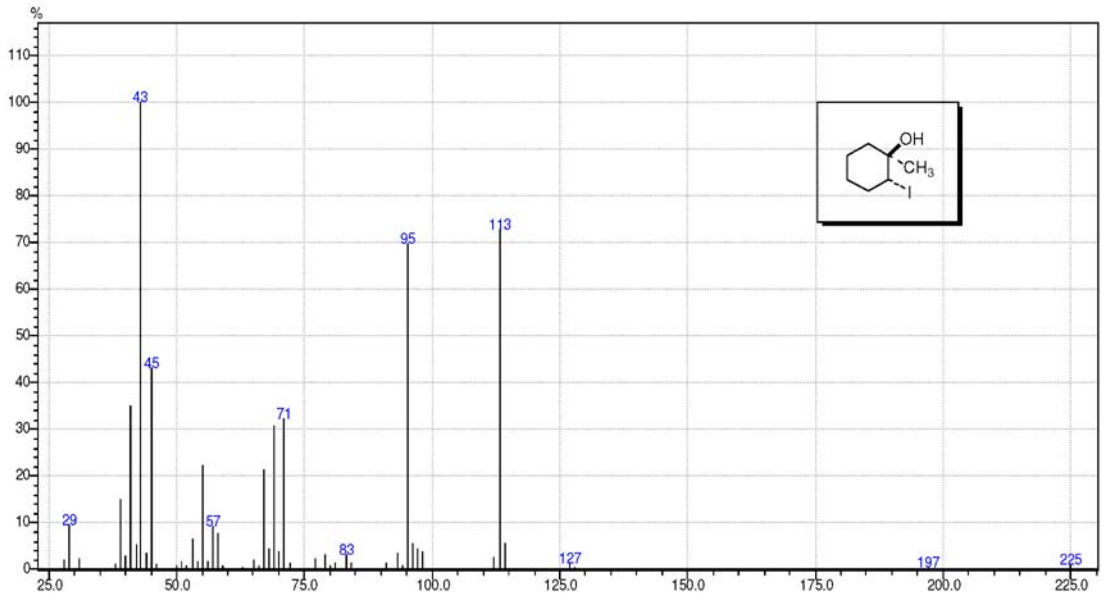

Figure S43. Mass spectrum of *trans*-2-iodo-1-methylcyclohexanol (70 eV).

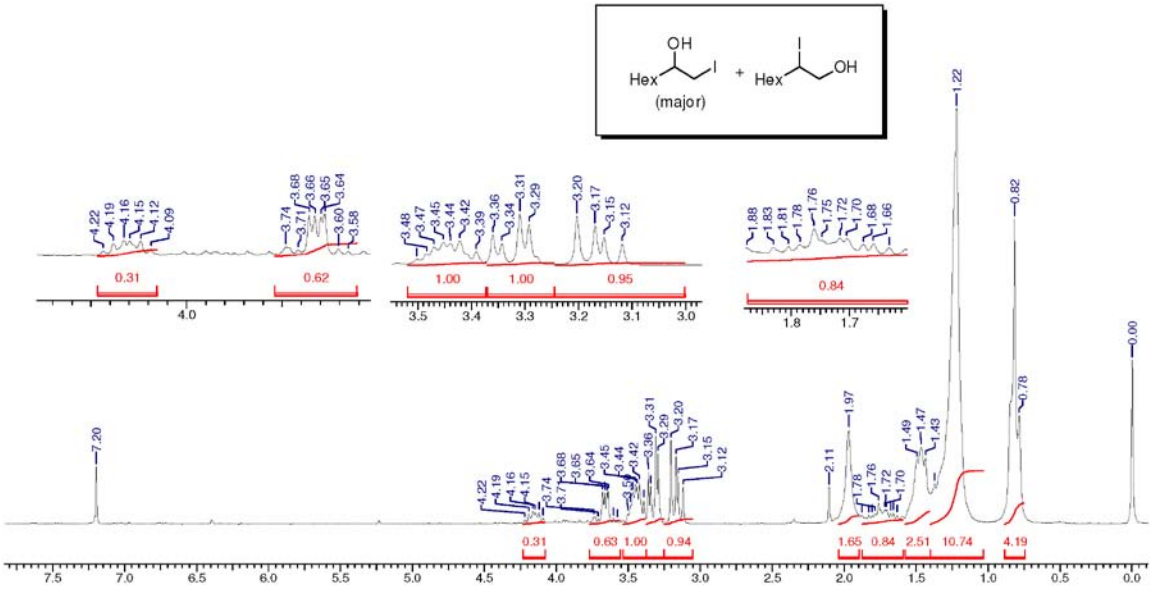

Figure S44. ^{1}H NMR spectrum of 1-iodo-2-octanol and 2-iodo-1-octanol ($CDCl_3$, TMS, 200 MHz).

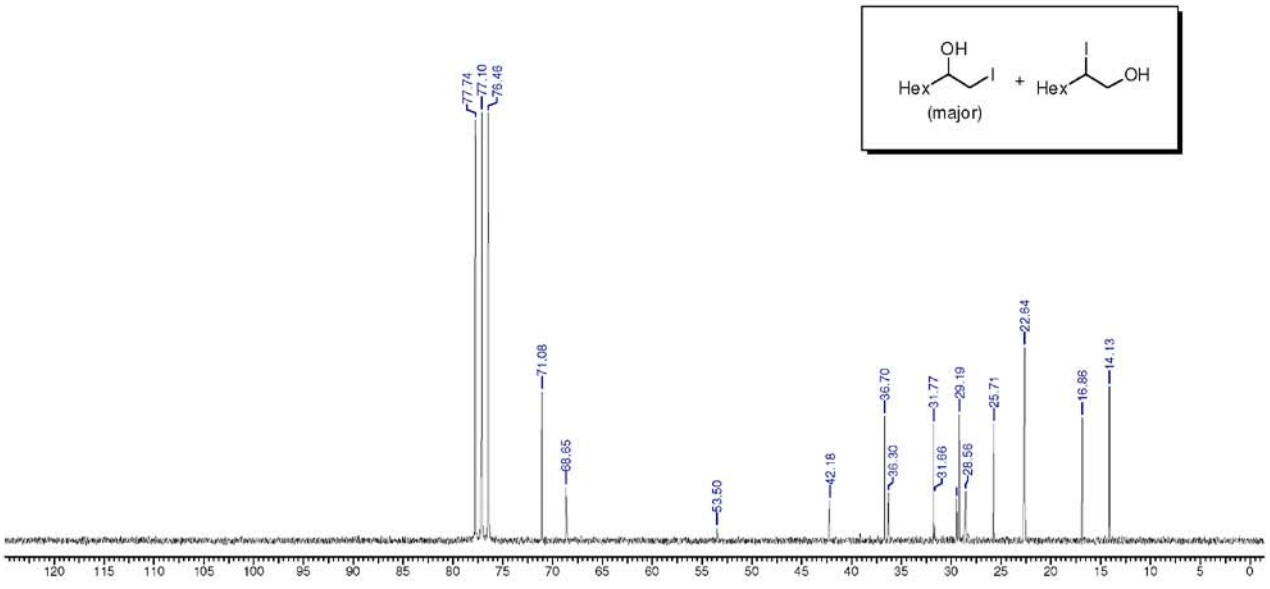

Figure S45. ^{13}C NMR spectrum of 1-iodo-2-octanol and 2-iodo-1-octanol ($CDCl_3$, TMS, 50 MHz).

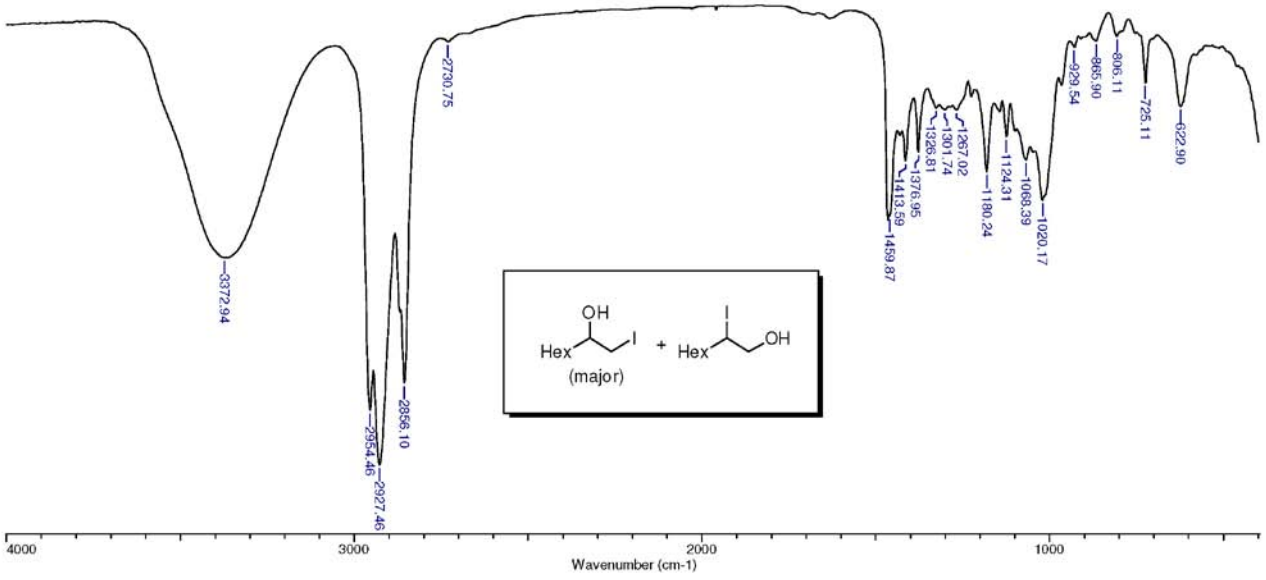

Figure S46. IR spectrum of 1-iodo-2-octanol and 2-iodo-1-octanol (film).

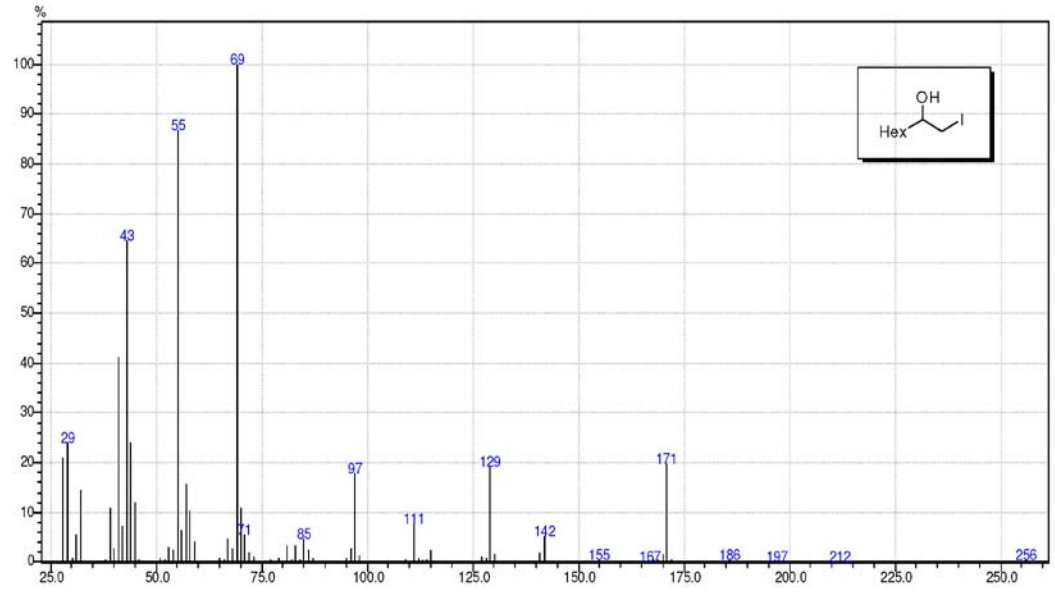

Figure S47. Mass spectrum of 1-iodo-2-octanol (70 eV).

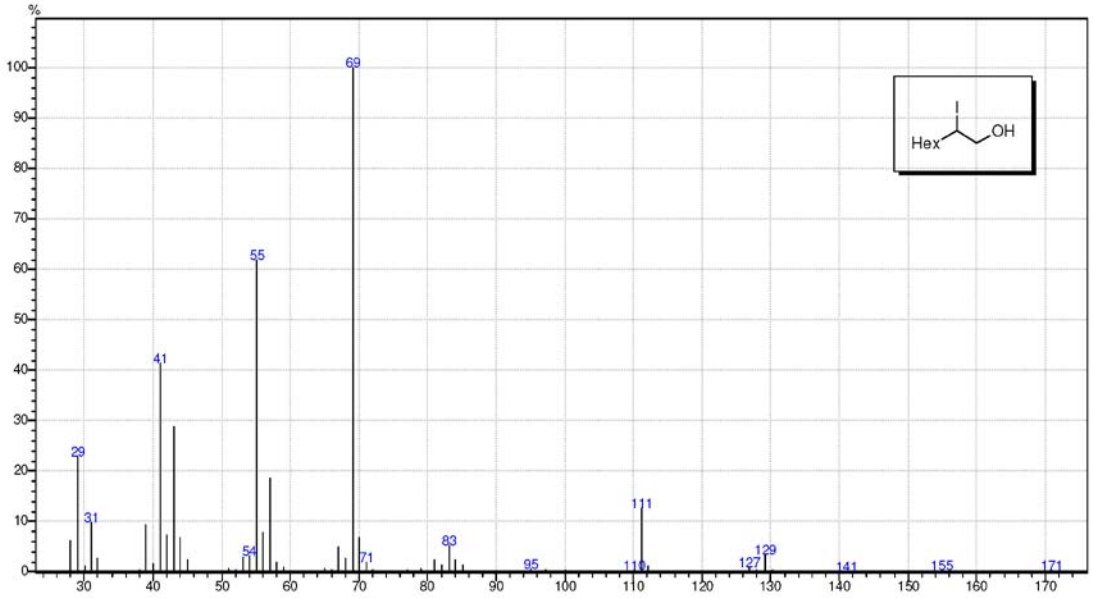

Figure S48. Mass spectrum of 2-iodo-1-octanol (70 eV).

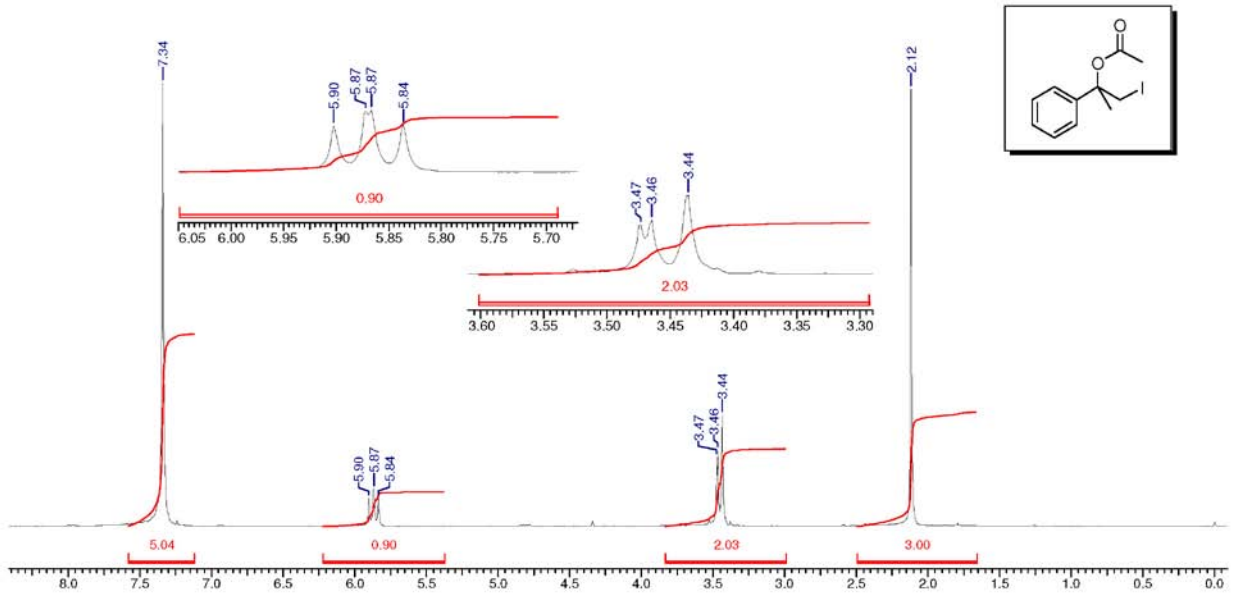

Figure S49. ^{1}H NMR spectrum of 2-iodo-1-phenylethyl acetate ($CDCl_3$, TMS, 200 MHz).

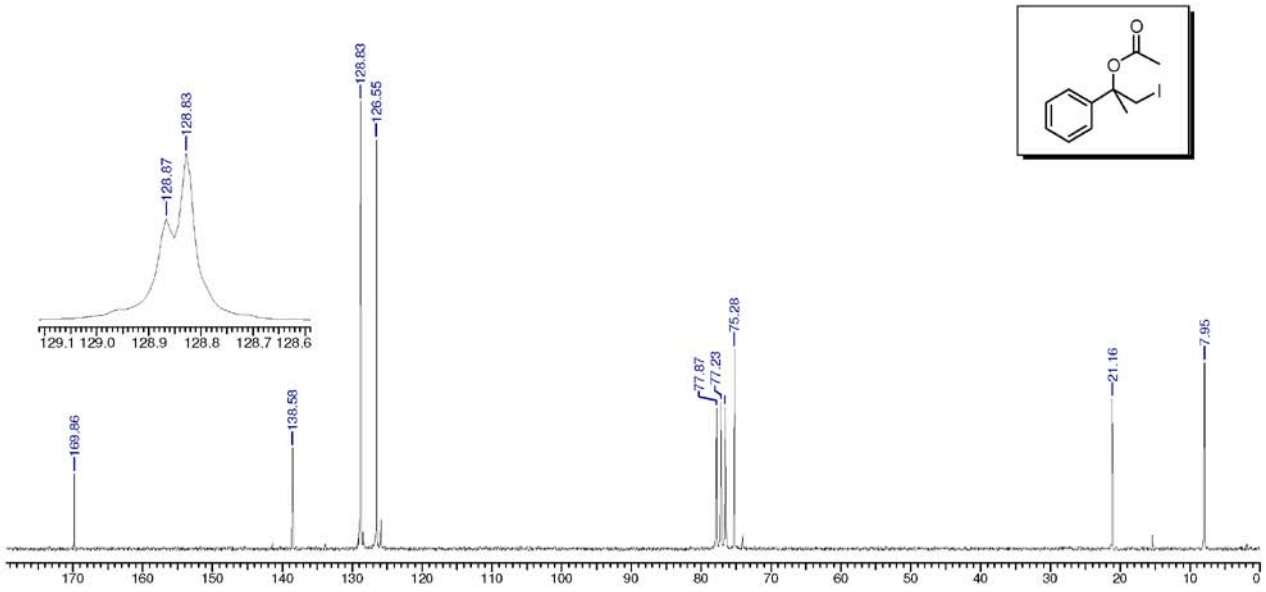

Figure S50. ^{13}C NMR spectrum of 2-iodo-1-phenylethyl acetate ($CDCl_3$, TMS, 50 MHz).

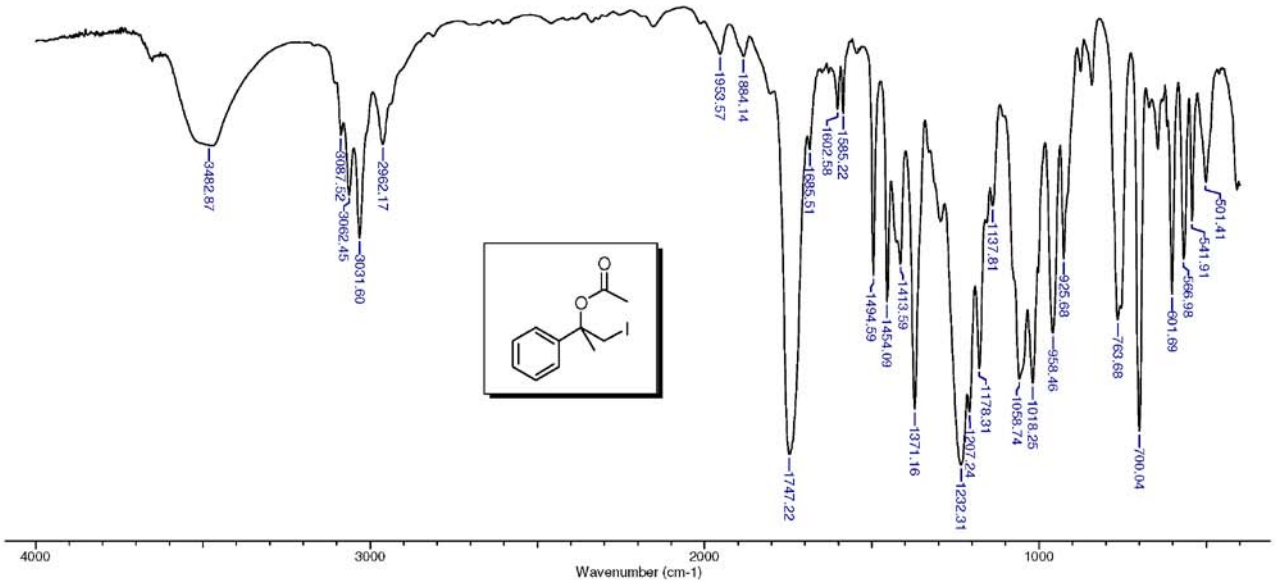

Figure S51. IR spectrum of 2-iodo-1-phenylethyl acetate (film).

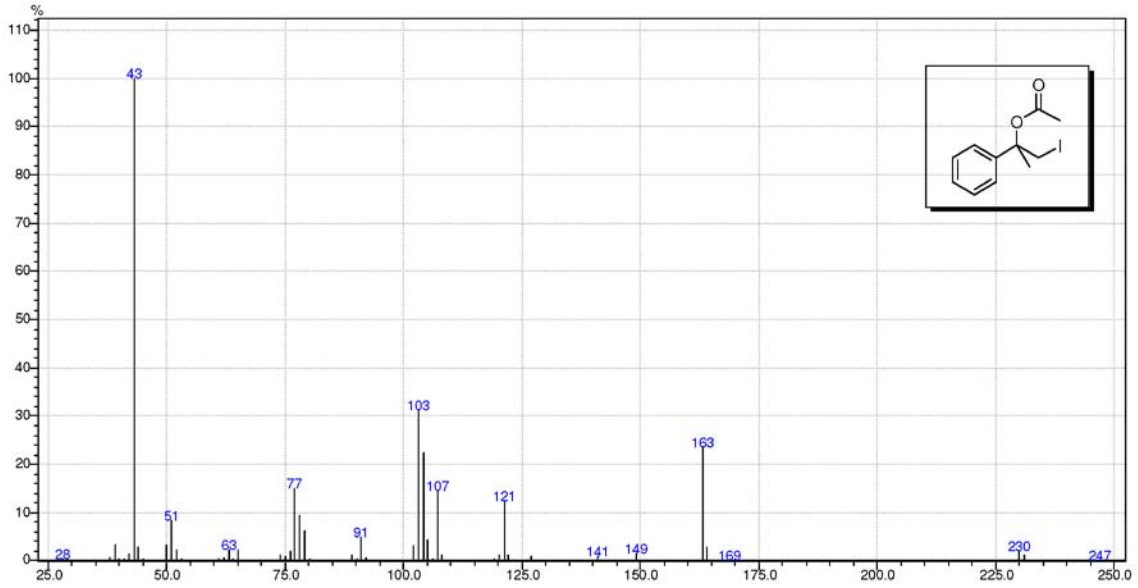

Figure S52. Mass spectrum of 2-iodo-1-phenylethyl acetate (70 eV).

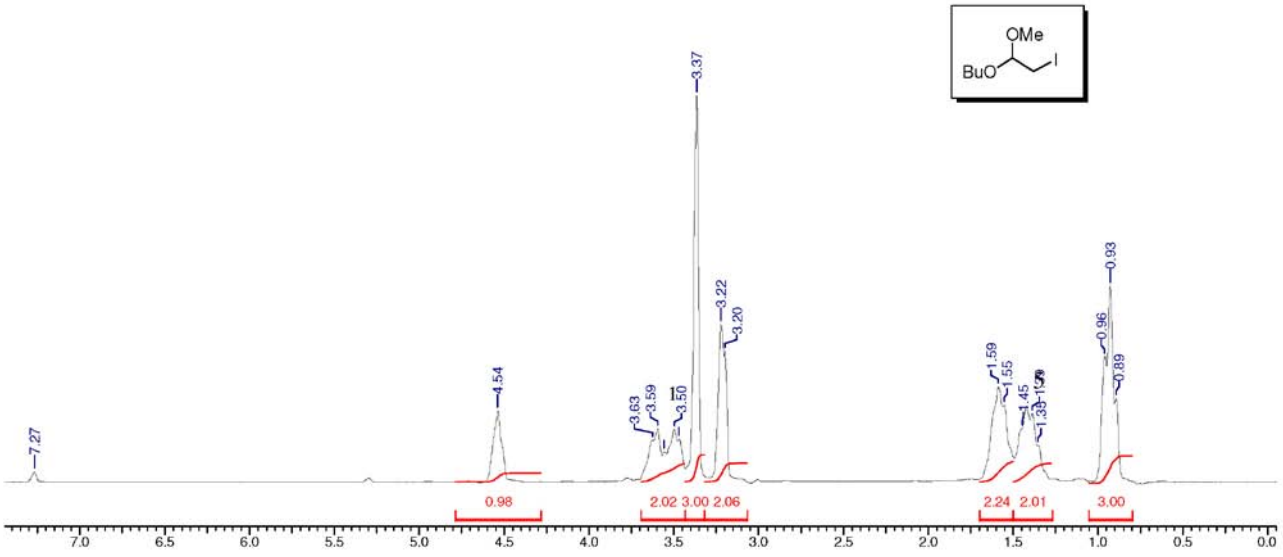

Figure S53. ^{1}H NMR spectrum of 2-iodo-1-butoxy-1-methoxybutane ($CDCl_3$, TMS, 200 MHz).

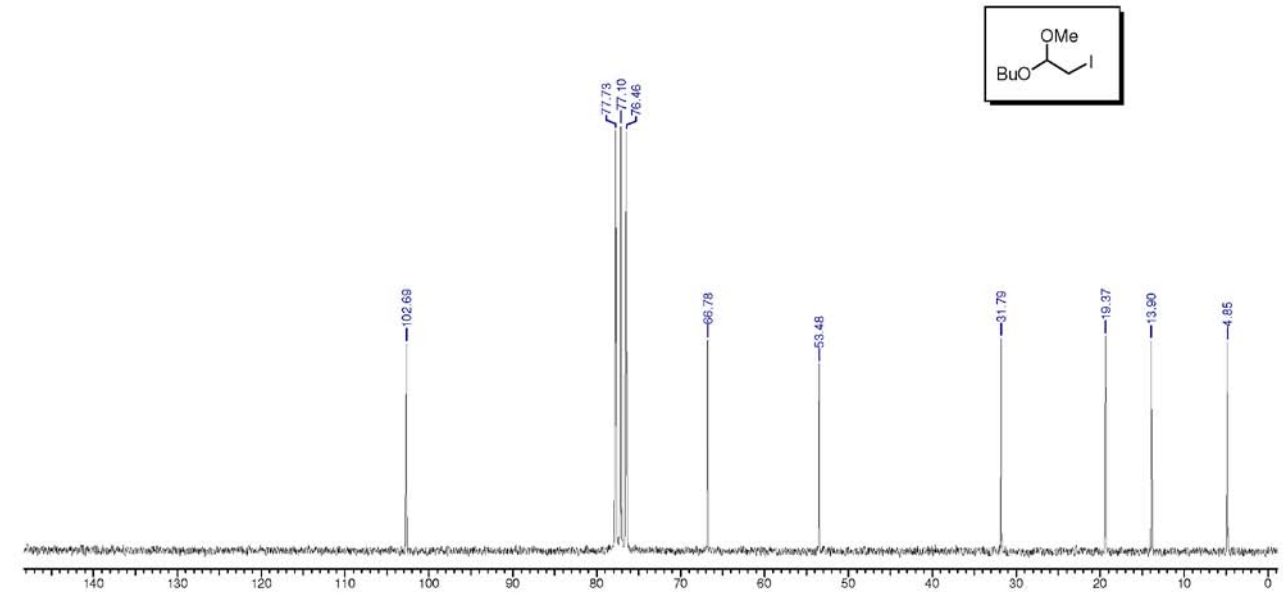

Figure S54. ^{13}C NMR spectrum of 2-iodo-1-butoxy-1-methoxybutane ($CDCl_3$, TMS, 50 MHz).

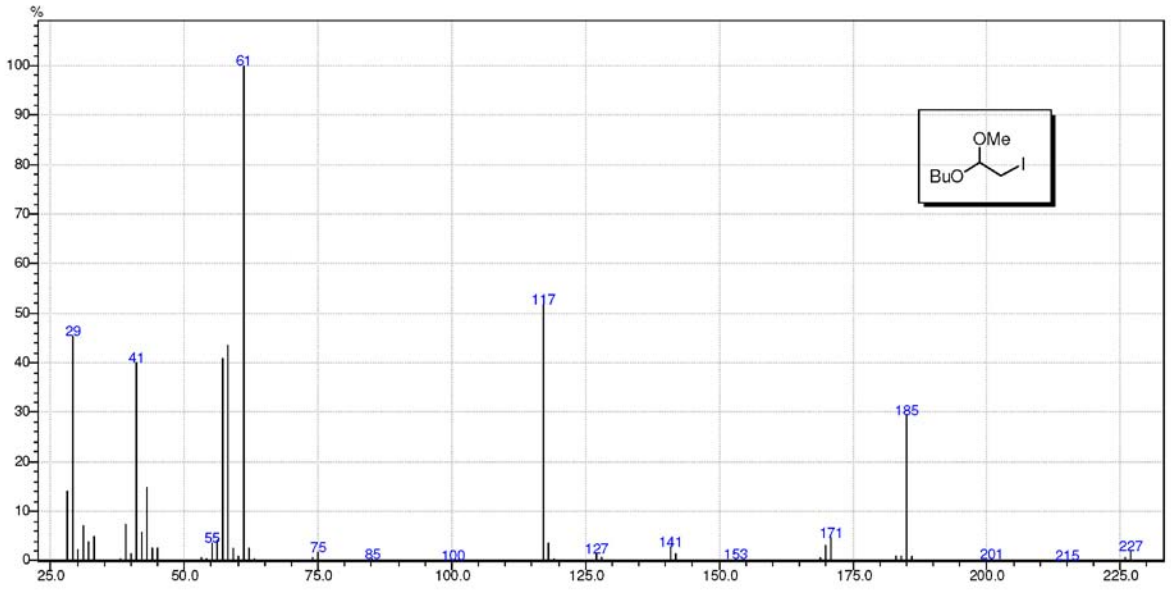

Figure S55. Mass spectrum of 2-iodo-1-butoxy-1-methoxybutane (70 eV).

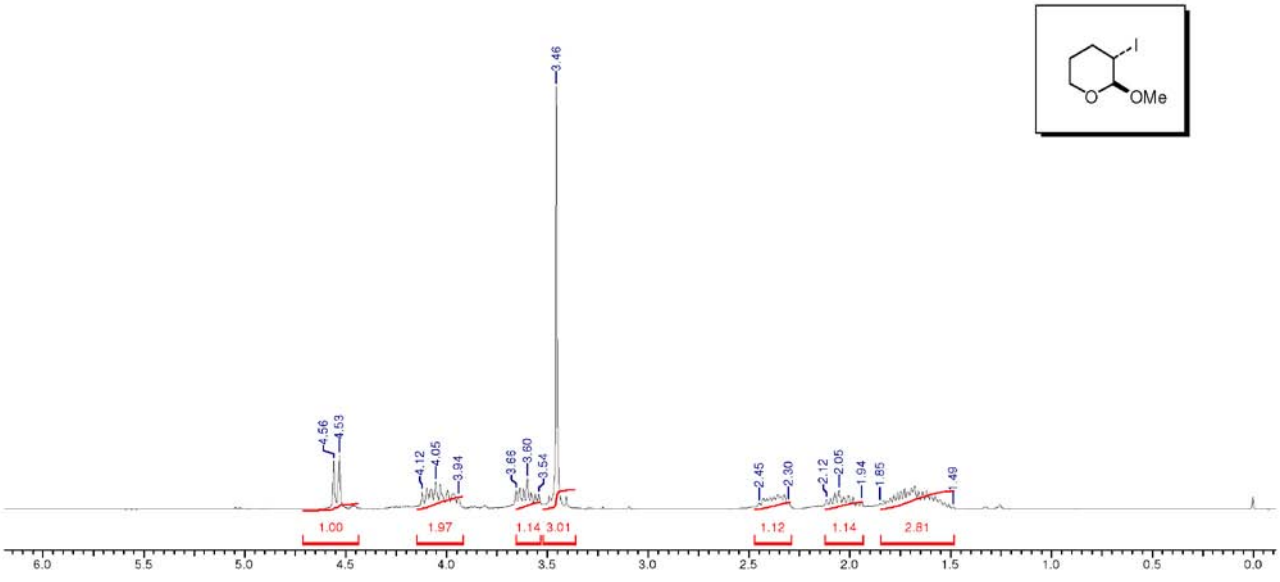

Figure S56. ^{1}H NMR spectrum of *trans*-3-iodo-2-methoxy tetrahydropyran ($CDCl_3$, TMS, 200 MHz).

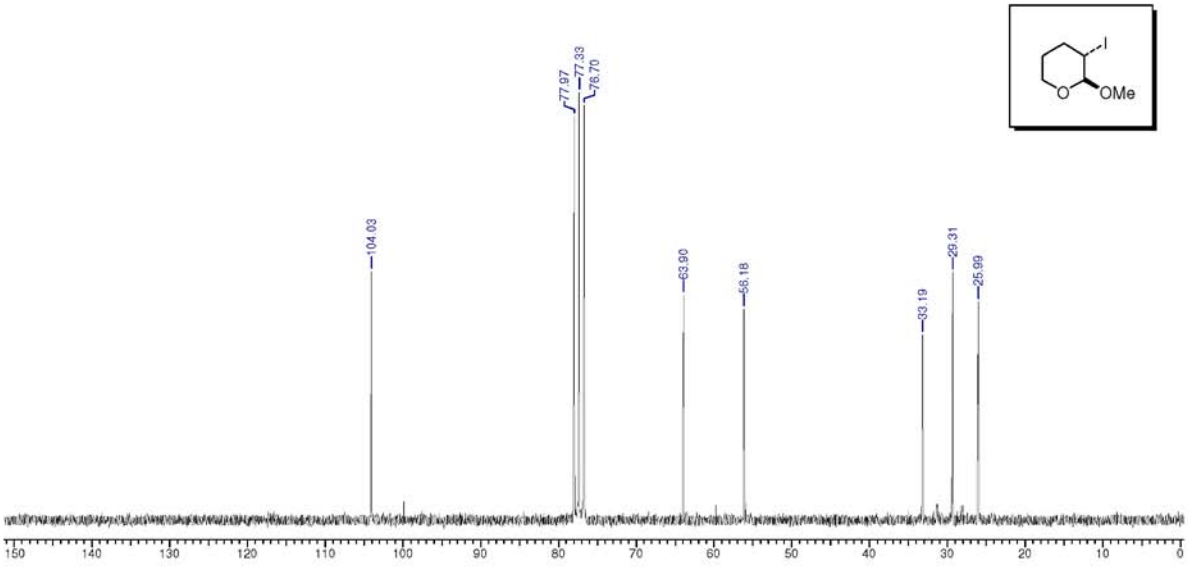

Figure S57. ^{13}C NMR spectrum of *trans*-3-iodo-2-methoxy tetrahydropyran ($CDCl_3$, TMS, 50 MHz).

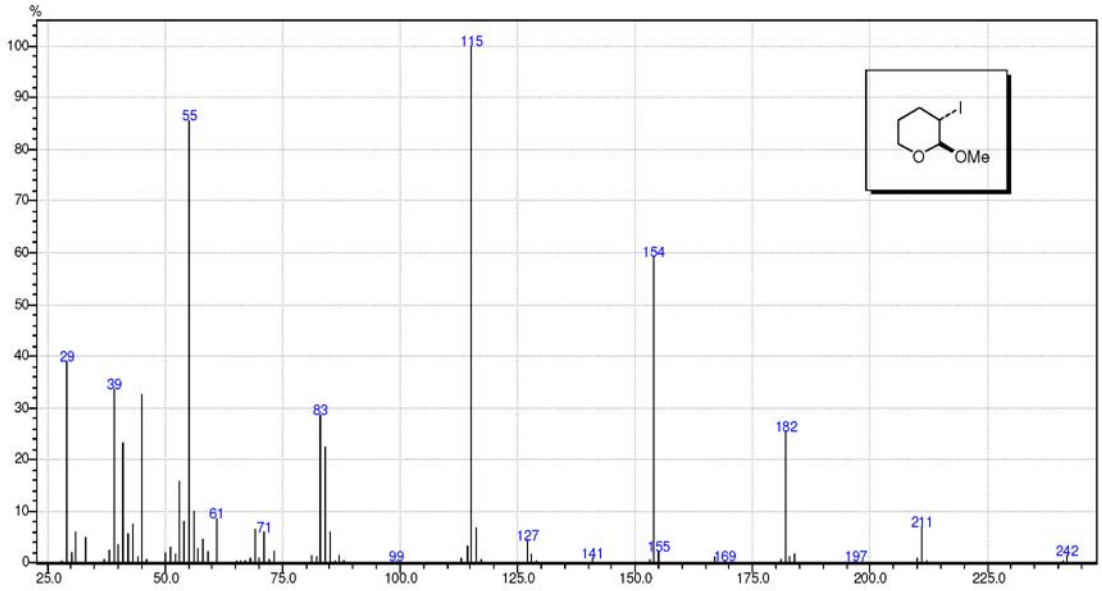

Figure S58. Mass spectrum of *trans*-3-iodo-2-methoxy tetrahydropyran (70 eV).

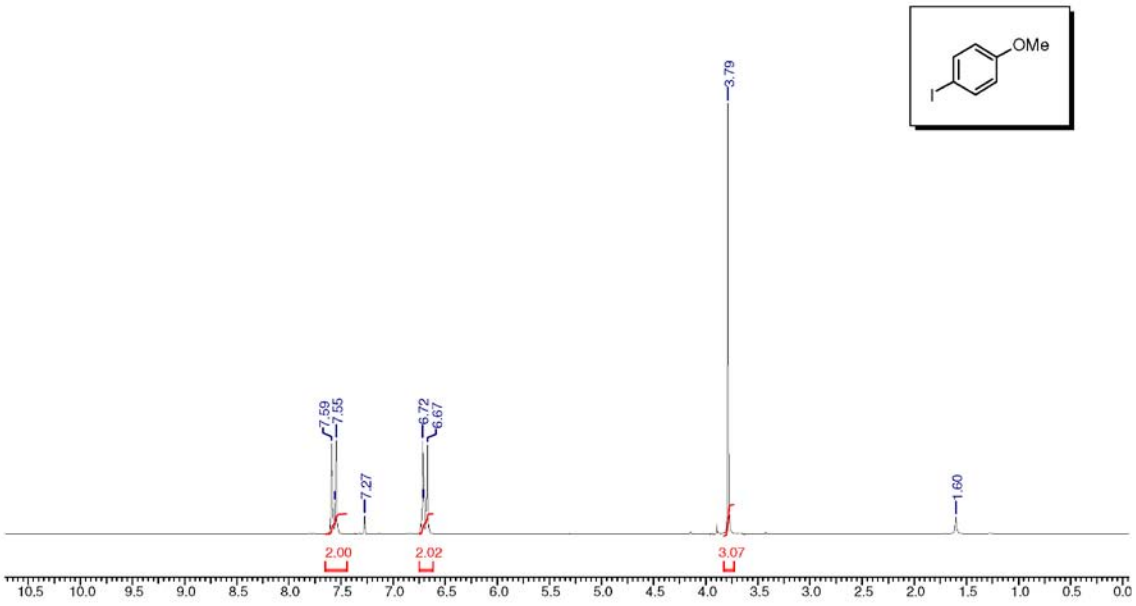

Figure S59. ^{1}H NMR spectrum of 4-iodoanisole ($CDCl_3$, TMS, 200 MHz).

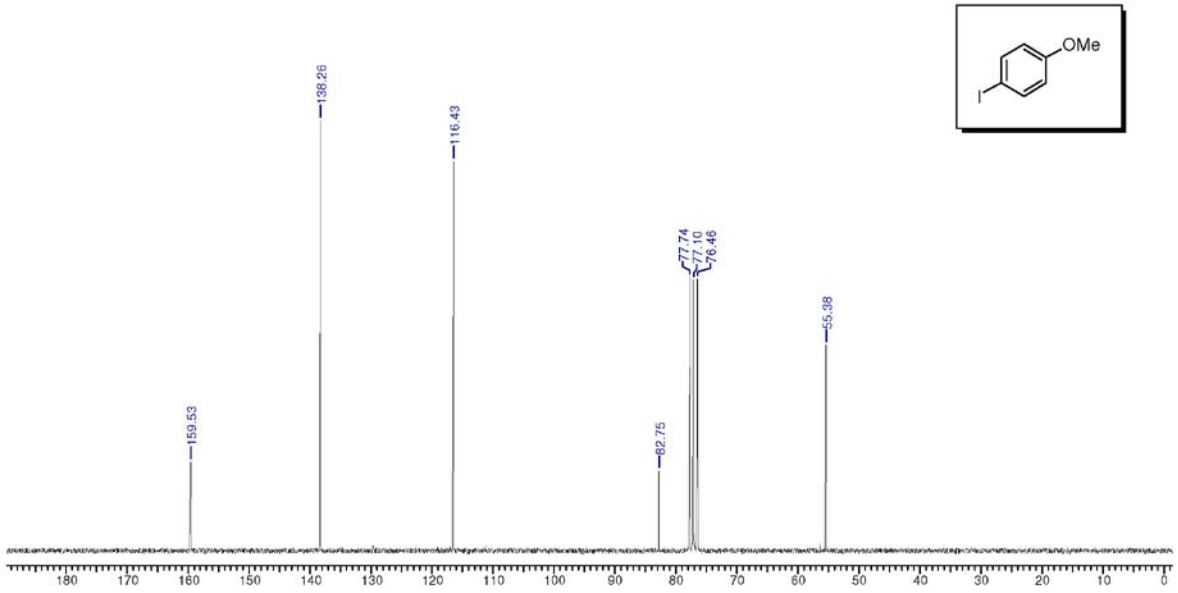

Figure S60. ^{13}C NMR spectrum of 4-iodoanisole ($CDCl_3$, TMS, 50 MHz).

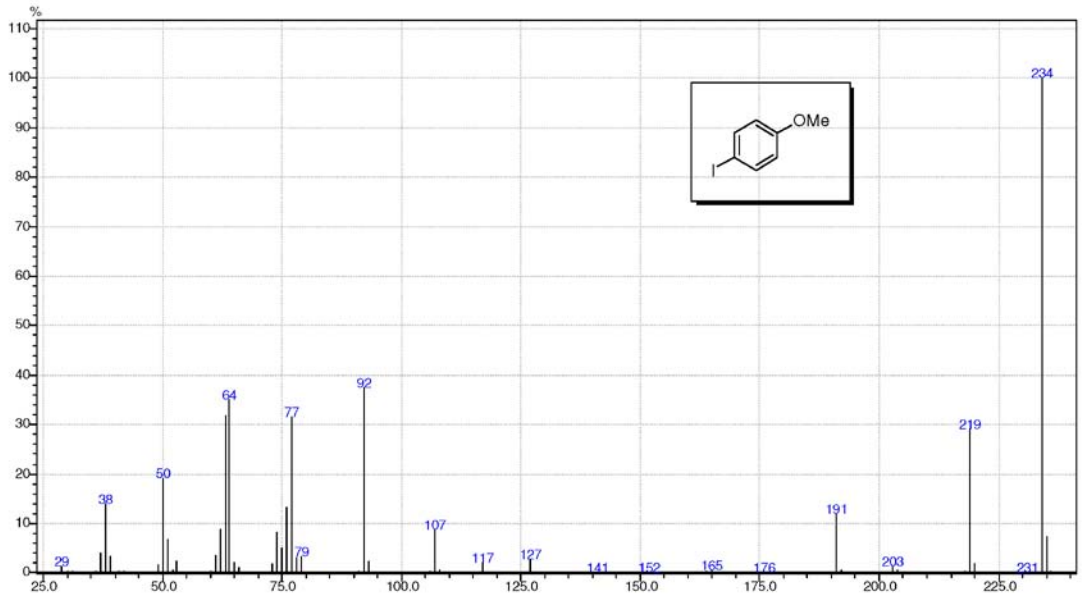

Figure S61. Mass spectrum of 4-iodoanisole (70 eV).

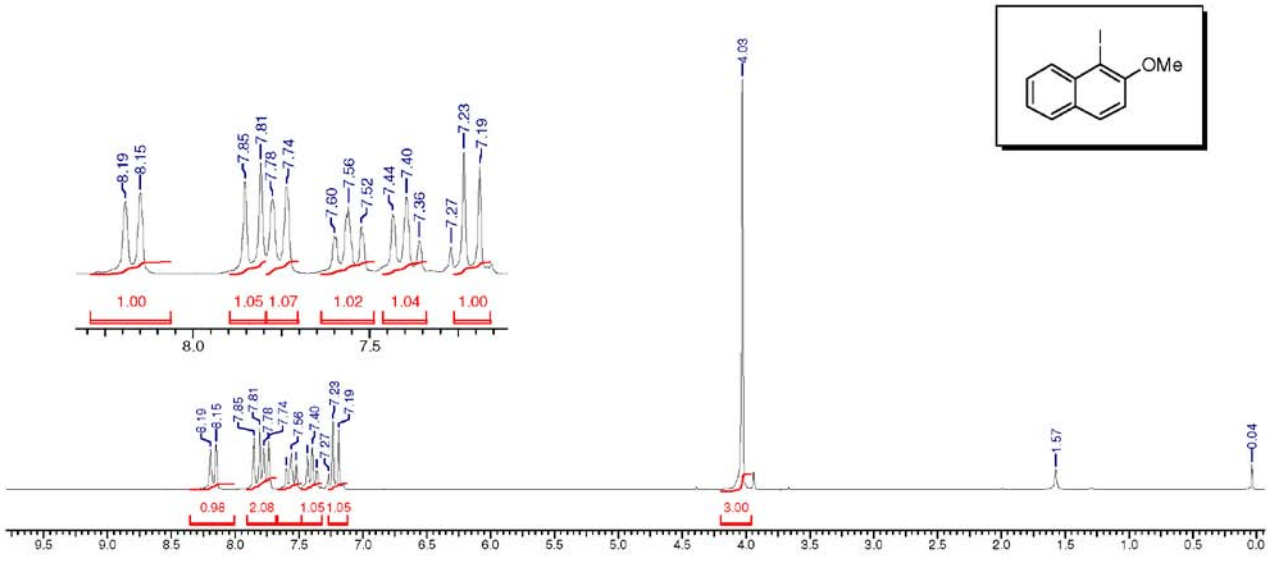

Figure S62. ^{1}H NMR spectrum of 1-iodo-2-methoxynaphthalene ($CDCl_3$, TMS, 200 MHz).

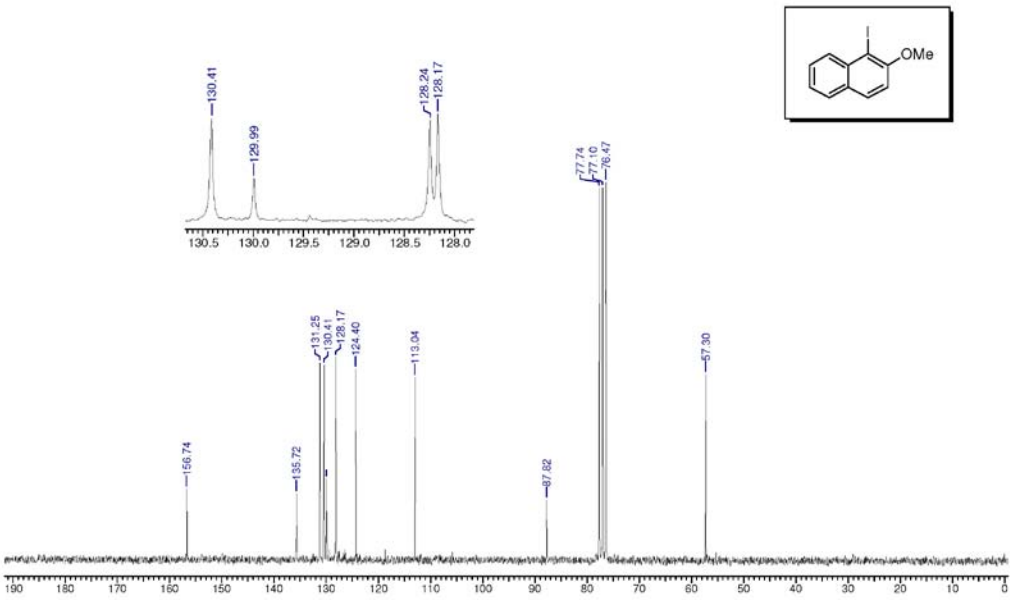

Figure S63. ^{13}C NMR spectrum of 1-iodo-2-methoxynaphthalene ($CDCl_3$, TMS, 50 MHz).

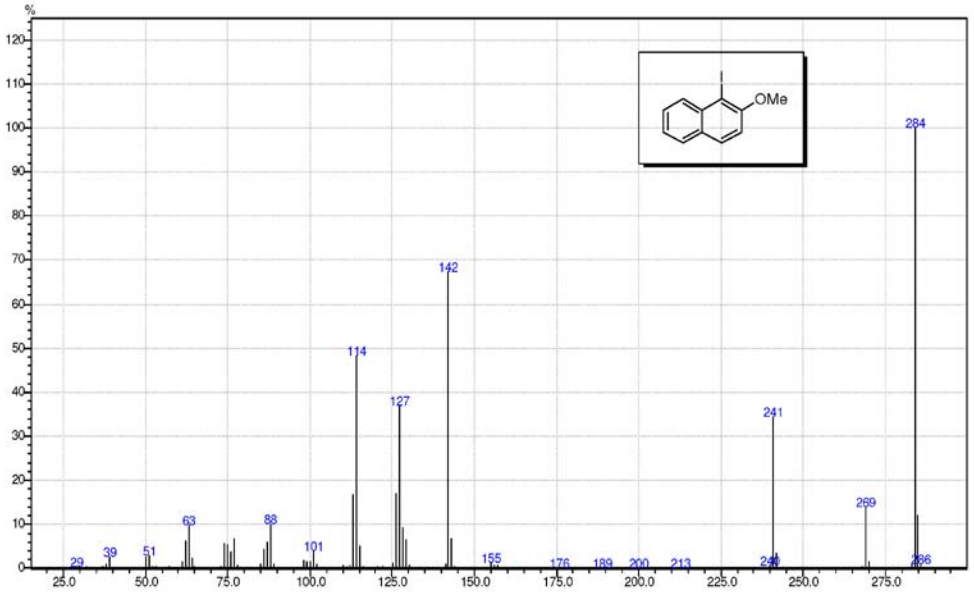

Figure S64. Mass spectrum of 1-iodo-2-methoxynaphthalene (70 eV).

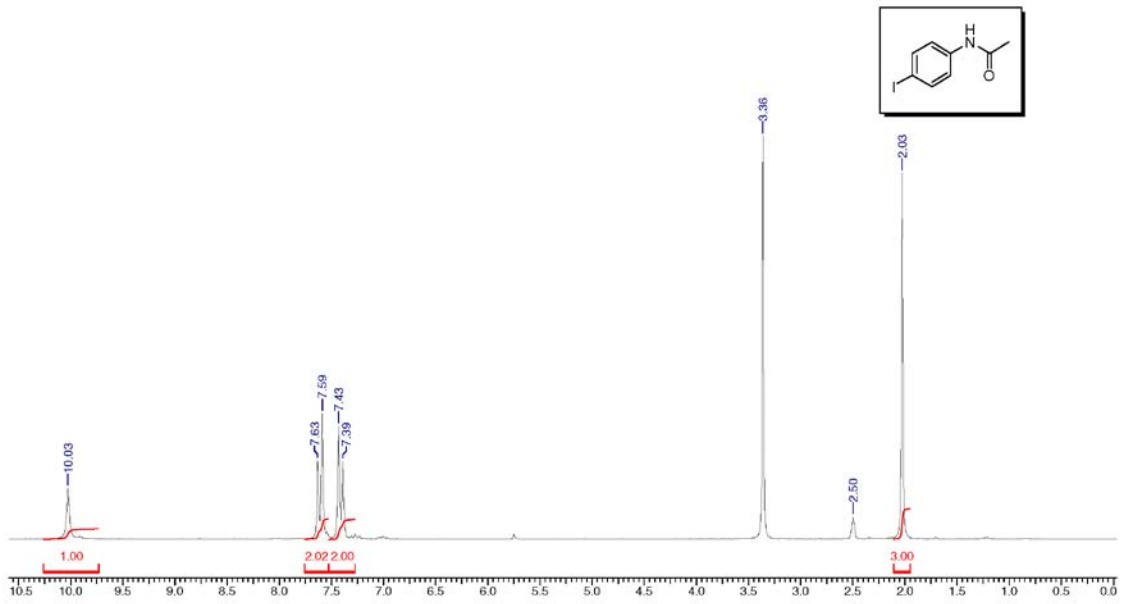

Figure S65. ^{1}H NMR spectrum of 4-iodoacetanilide (DMSO-d_6, TMS, 200 MHz).

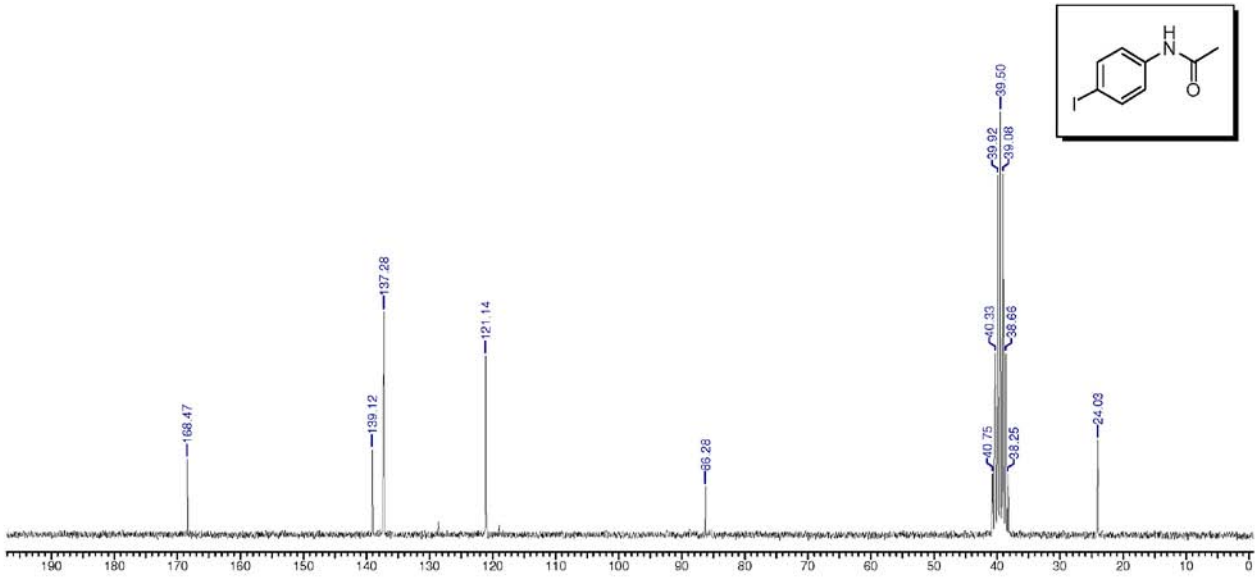

Figure S66. ^{13}C NMR spectrum of 4-iodoacetanilide (DMSO-d_6, TMS, 50 MHz).

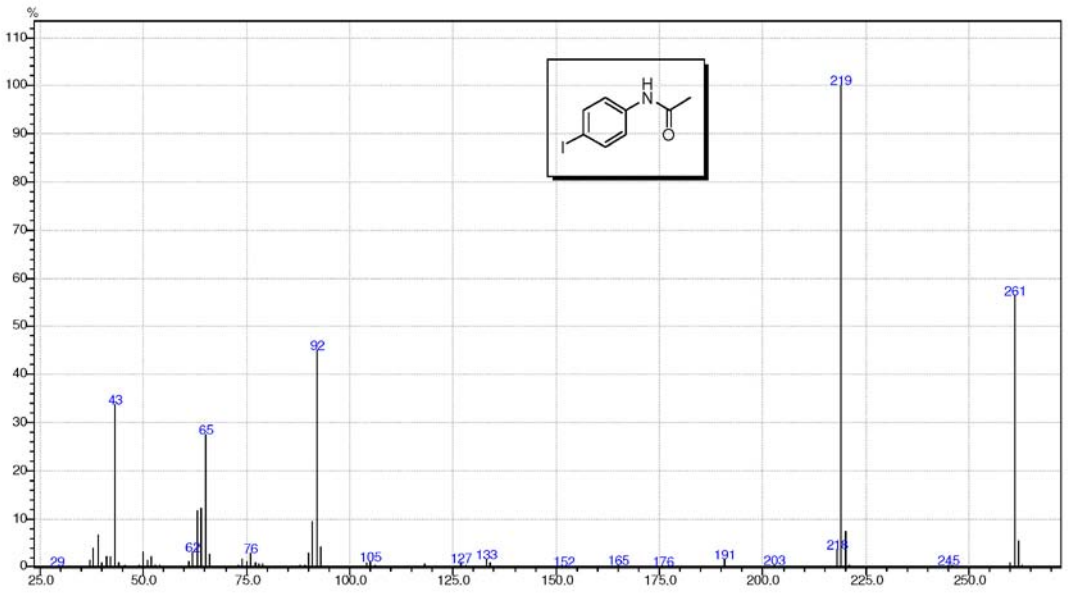

Figure S67. Mass spectrum 4-iodoacetanilide (70 eV).

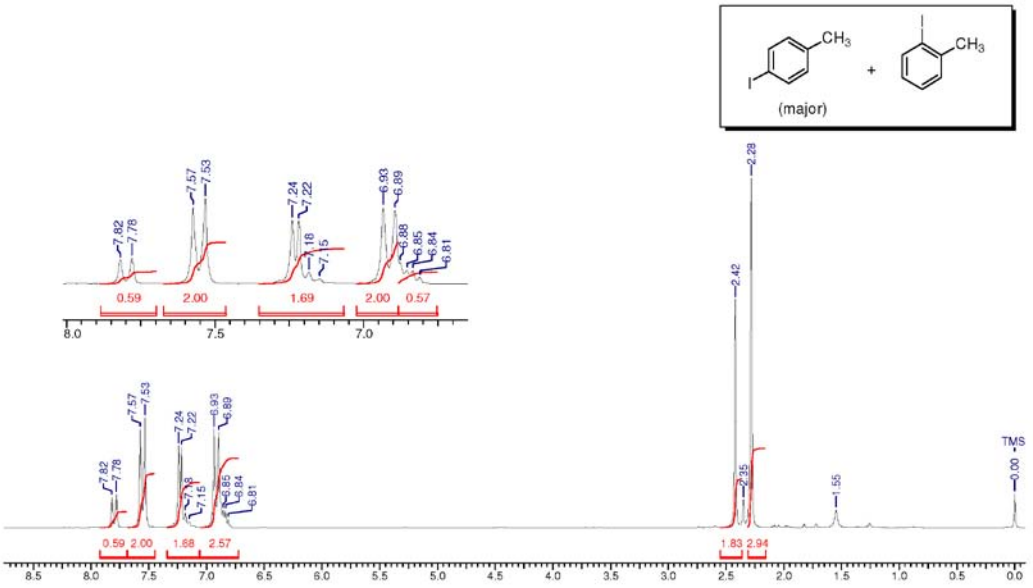

Figure S68. ^{1}H NMR spectrum of 4-iodo- and 2-iodotoluene ($CDCl_3$, TMS, 200 MHz).

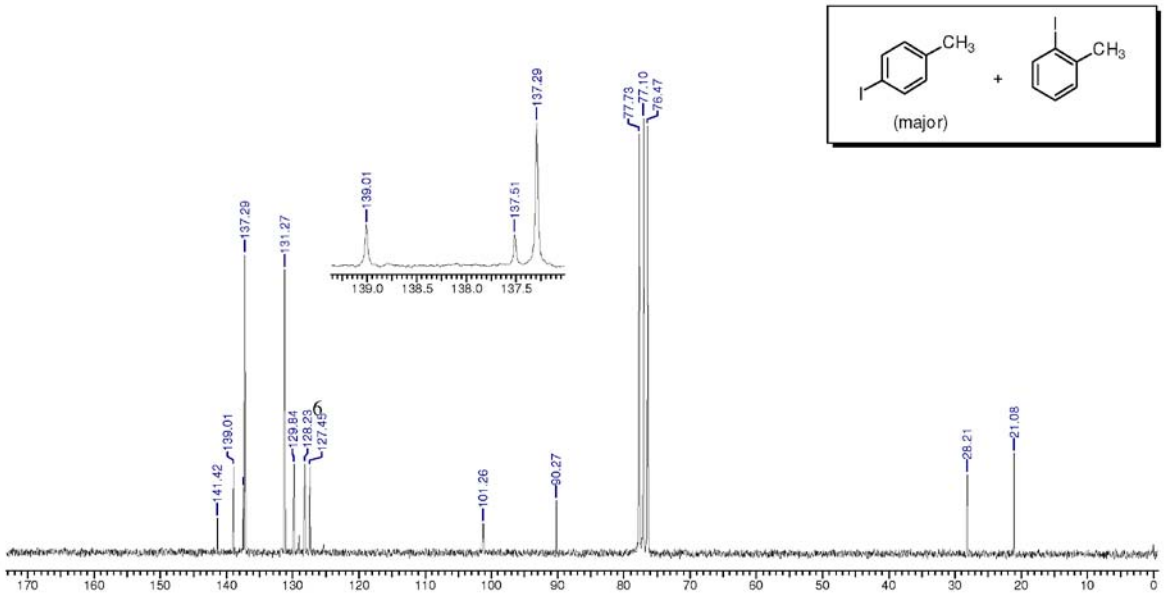

Figure S69. ^{13}C NMR spectrum of 4-iodo- and 2-iodotoluene ($CDCl_3$, TMS, 50 MHz).

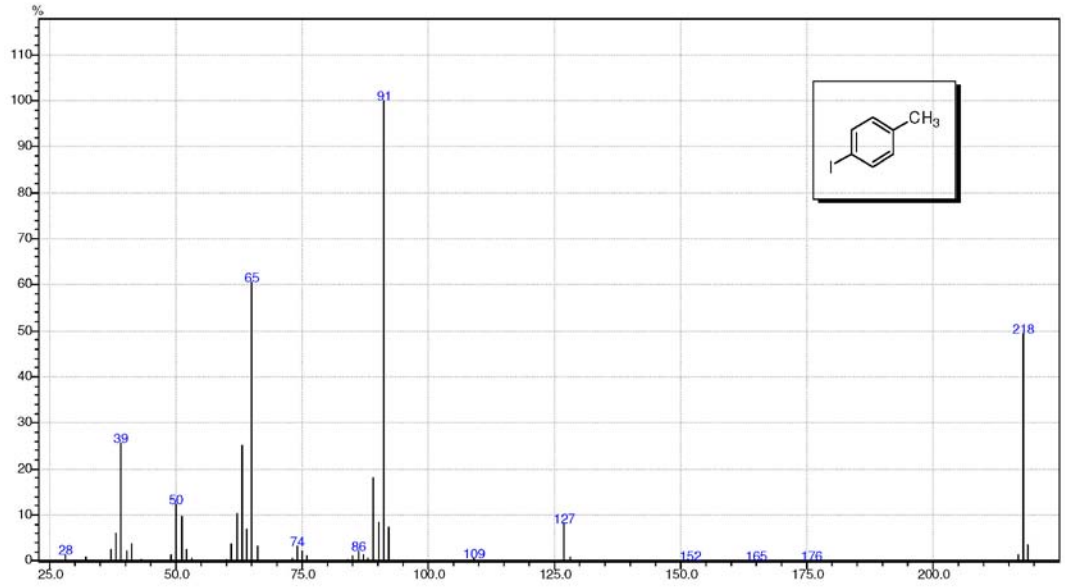

Figure S70. Mass spectrum of 4-iodotoluene (70 eV).

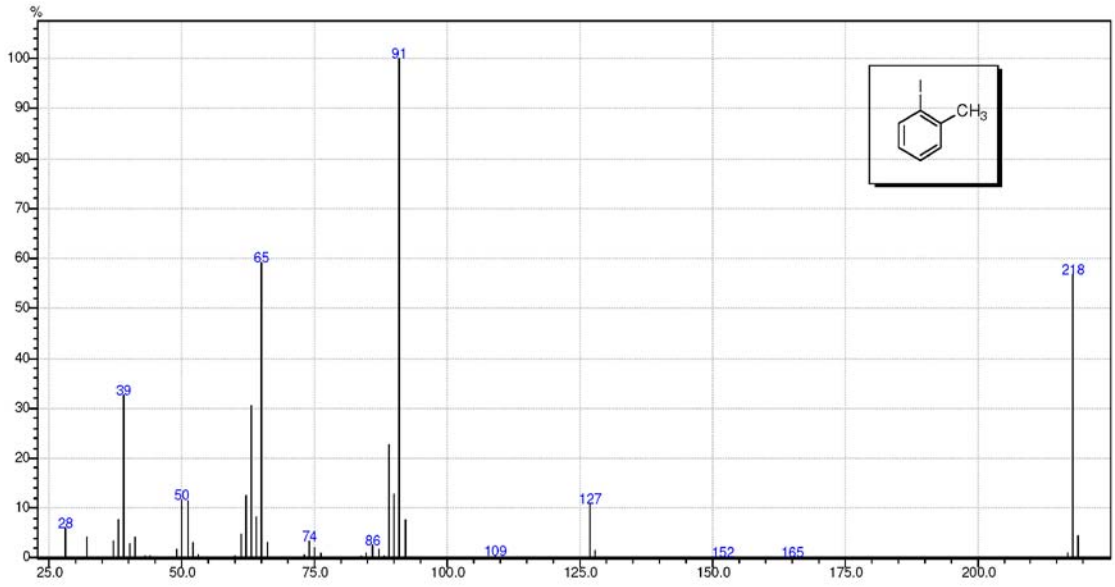

Figure S71. Mass spectrum of 2-iodotoluene (70 eV).

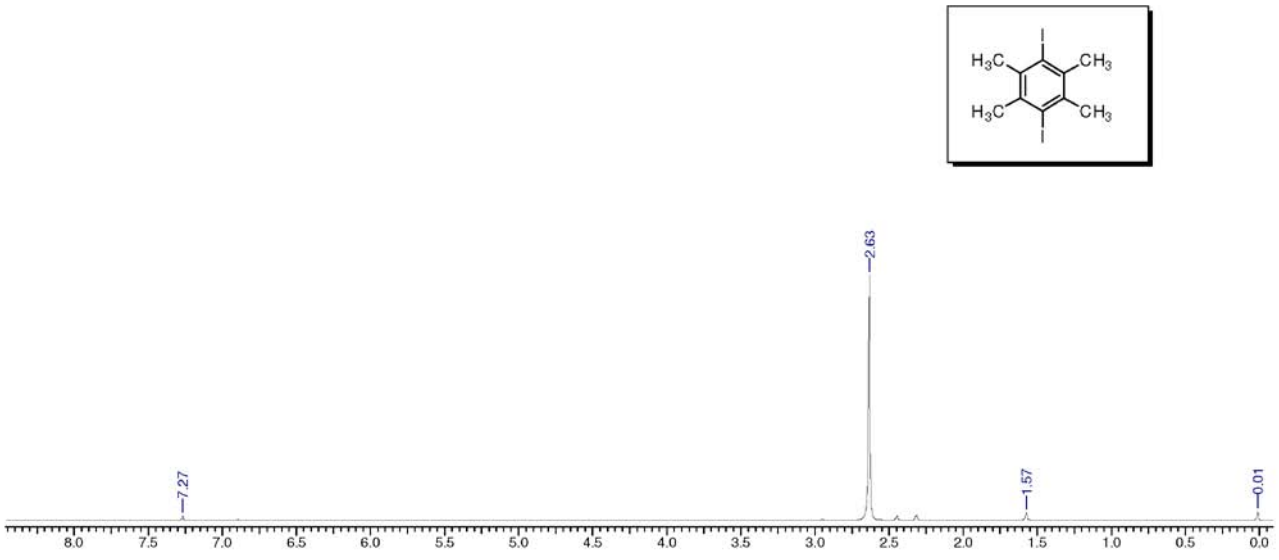

Figure S72. ^{1}H NMR spectrum of 1,4-diiodo-2,3,5,6-tetramethylbenzene ($CDCl_3$, TMS, 200 MHz).

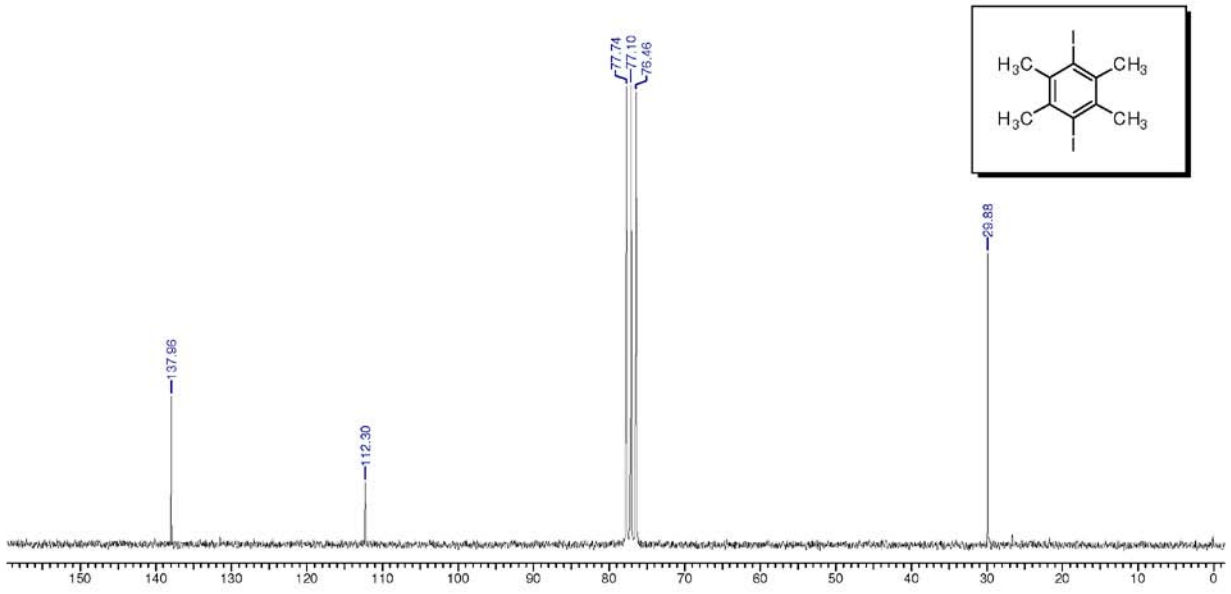

Figure S73. ^{13}C NMR spectrum of 1,4-diiodo-2,3,5,6-tetramethylbenzene ($CDCl_3$, TMS, 50 MHz).

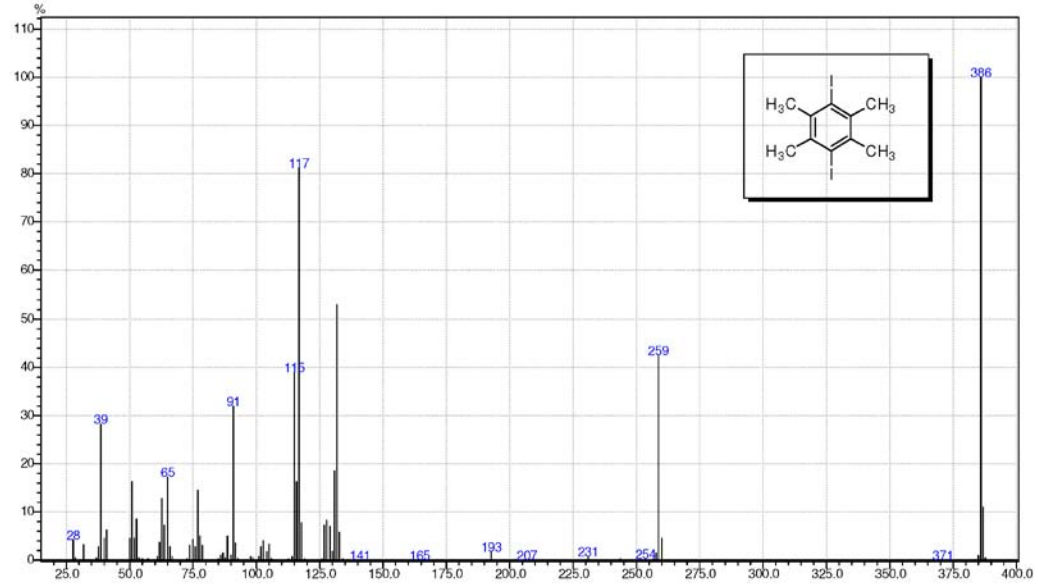

Figure S74. Mass spectrum of 1,4-diiodo-2,3,5,6-tetramethylbenzene (70 eV).