Supporting Information for

The Preparation and Characterization of the Fullerene Diols 1,2-$C_{60}(OH)_2$, 1,2-$C_{70}(OH)_2$, and 5,6-$C_{70}(OH)_2$

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1. Experimental Procedures

General. Fullerenes C_{60} and C_{70} were obtained from MER Corp. A solution of RuO_4 in CCl_4 was prepared as follows: A separatory funnel in a well ventilated area was used for generation of RuO_4• RuO_2 hydrate (338 mg, 2 mmol, assuming dihydrate) was suspended in a mixture of water (30 mL) and CCl_4 (30 mL). Sodium periodate (2.14 g, 10 mmol) was added at once and the mixture was shaken vigorously until solid disappeared. The yellow CCl_4 layer was separated and the water layer was extracted with 20 mL of CCl_4. The combined CCl_4 layers were dried over anhydrous $MgSO_4$ for 20 min. then filtered through a layer of anhydrous sodium sulfate into an addition funnel.

1,2-$C_{60}(OH)_2$ (1). C_{60} (720mg, 1 mmol) was dissolved in 1,2,4-trichlorobenzene (200 mL). To this solution was added dropwise (30 min.) a freshly-prepared solution of RuO_4 (2 mmol) in carbon tetrachloride (50 mL). The resulting black suspension was stirred for 3 h, then suction filtered through a 0.2 μ nylon filter membrane. The filtrate contained

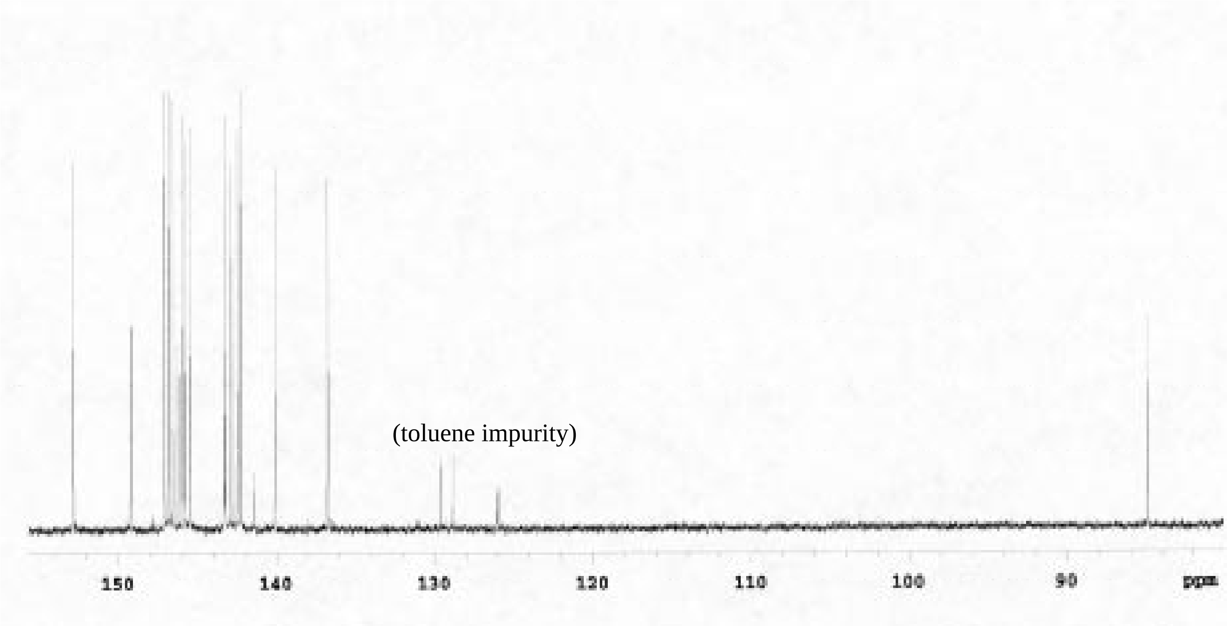
162 mg (0.225 mmol) of unreacted C_{60}. The filter cake was resuspended in toluene and centrifuged. This was repeated three times. The resulting pellet was dried in air to give 1.05 g of a black powder that was insoluble in common solvents. The solid was suspended in water/dioxane (1:25, 280 mL) containing 2 mL conc. aq. HCl and sonicated for 30 min. at 40°C. The clear solution was stirred while 300 mL of toluene was added over 5 min. The aqueous layer was separated and the organic solution was dried over anhydrous sodium sulfate for 10 hrs, filtered and evaporated to dryness. The resulting brown solid (574 mg) was redissolved in toluene/dioxane (80:20) mixture and purified using column chromatography on Florisil (40 g) eluting with toluene/dioxane (80:20 to 50:50) solvent system. Fractions containing $C_{60}(OH)_2$ were combined and concentrated. Crude 1,2-$C_{60}(OH)_2$ (137 mg) was further purified by HPLC on a semipreparative PBB Cosmosil column (toluene/THF = 90:10) to give 64 mg (8.5%, 11% based on reacted C_{60}). ^{13}C NMR (CS_2/dioxane-d_8) δ (relative intensity) 84.94, 136.85, 140.18, 142.34, 142.55, 142.93, 143.30, 143.38, 145.55, 145.90, 145.99, 146.30, 146.64, 146.93, 147.17, 149.30, 152.90; IR (KBr) ν 3285 cm^{-1}; FAB MS (negative ion, *m*-nitrobenzyl alcohol) *m*/*z* (%) 754 (M$^-$,40), 736 (67), 720 (100).

$C_{70}(OH)_2$ (2 and 3). C_{70} was reacted with RuO_4 according to the procedure described above for $C_{60}(OH)_2$. Starting from 420 mg (0.5 mmol), 143 mg of unreacted C_{70} was recovered after initial reaction with RuO_4. Also, Florisil was replaced by silica gel in the process of preliminary purification. Two isomers of $C_{70}(OH)_2$ were isolated using the work-up procedure described above: 45 mg (10.5%, 16% based on reacted C_{70}) of (1,2) isomer together with 14 mg (3.2%, 4.8% based on reacted C_{70}) of (5,6) isomer.

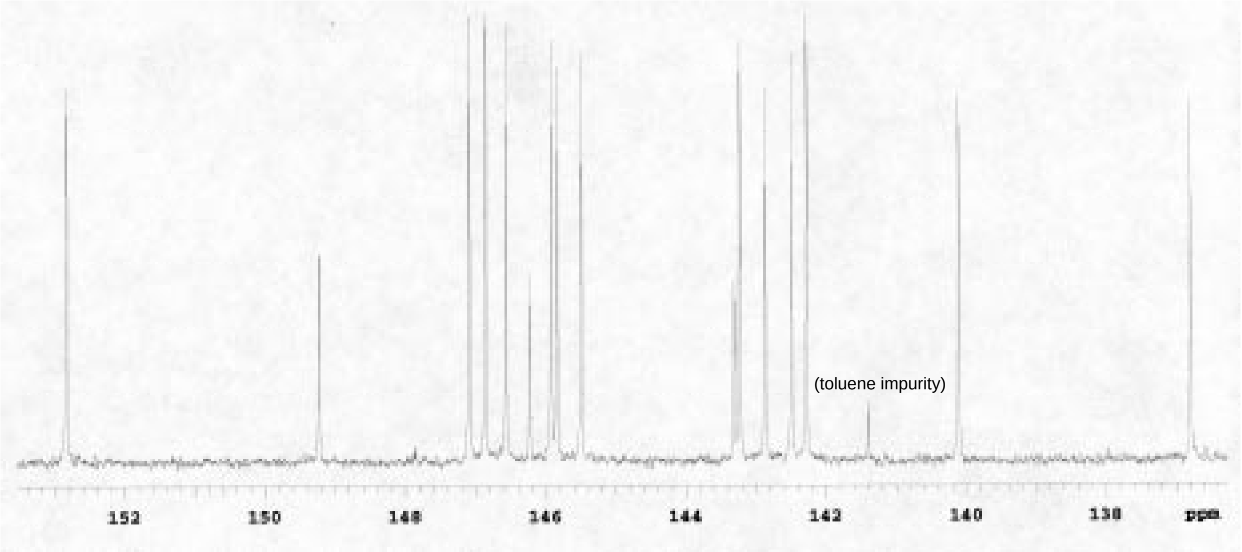
1,2-$C_{70}(OH)_2$ (2): ^{13}C NMR (CS_2/dioxane-d_8) δ (relative intensity) 80.36, 81.60, 132.07, 132.09, 132.75, 133.38, 134.15, 138.00, 141.09, 141,54, 144.01, 144.16, 144.26, 144.49, 146.30, 146.68, 147.28, 147.29, 147.96, 147.98, 148.06, 148.41, 149.13, 149.74, 149.90, 150.06, 150.09, 150.47, 150.83, 151.31, 151.79, 151.82, 152.03, 152.11, 154.98, 157.37, 157.38. IR (KBr) ν 3280 cm^{-1}; FAB MS (negative ion, *m*-nitrobenzyl alcohol) *m*/*z* (%) 874 (M^-,55), 856 (100), 840 (100).

5,6-$C_{70}(OH)_2$ (3): ^{13}C NMR (CS_2/dioxane-d_8) δ (relative intensity) 78.03, 126.93, 129.33, 130.97, 133.01, 133.20, 141.44, 143.44, 143.55, 144.53, 144.69, 145.04, 145.52, 145.63, 146.01, 146.09, 146.60, 146.80, 146.86, 147.27, 147.57, 147.64, 147.75, 148.66, 149.46, 149.61, 149.64, 149.90, 150.10, 150.62, 150.79, 151.30, 151.31, 152.28, 152.48, 155.61, 166.67. IR (KBr) ν 3285 cm^{-1}; FAB MS (negative ion, *m*-nitrobenzyl alcohol) *m*/*z* (%) 891 (11), 874 (M^+,32), 856 (80), 840 (100).

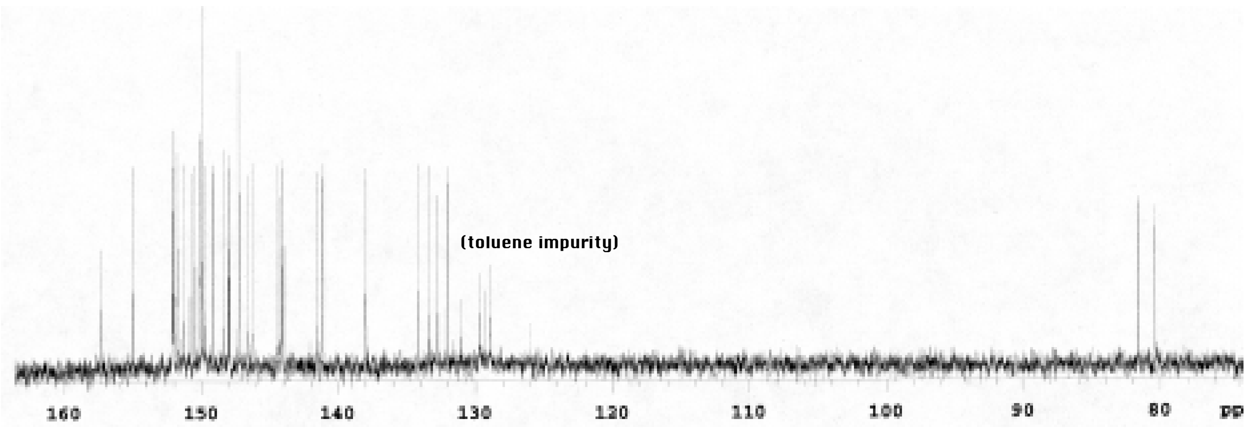
2. ^{13}C NMR Spectra



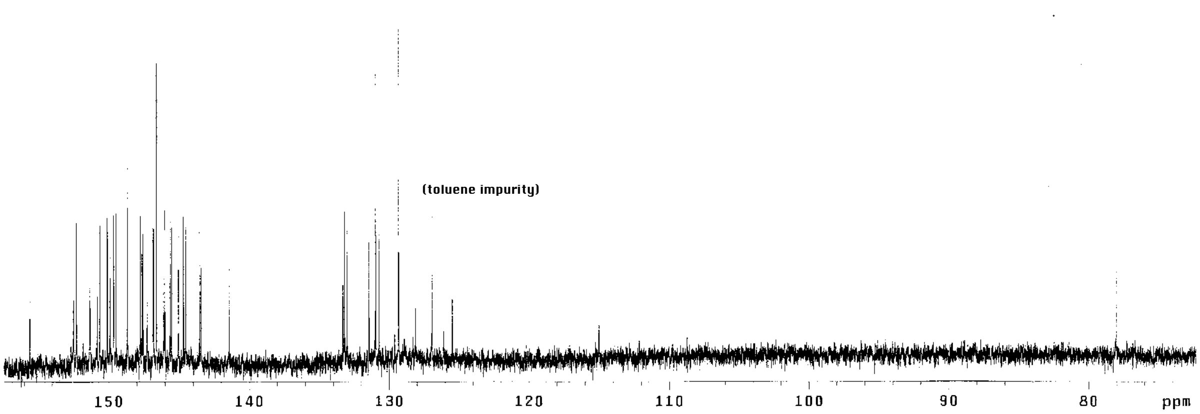
The ^{1}H-decoupled ^{13}C NMR spectrum of **1** in 50:50 CS_2/dioxane-d_8.



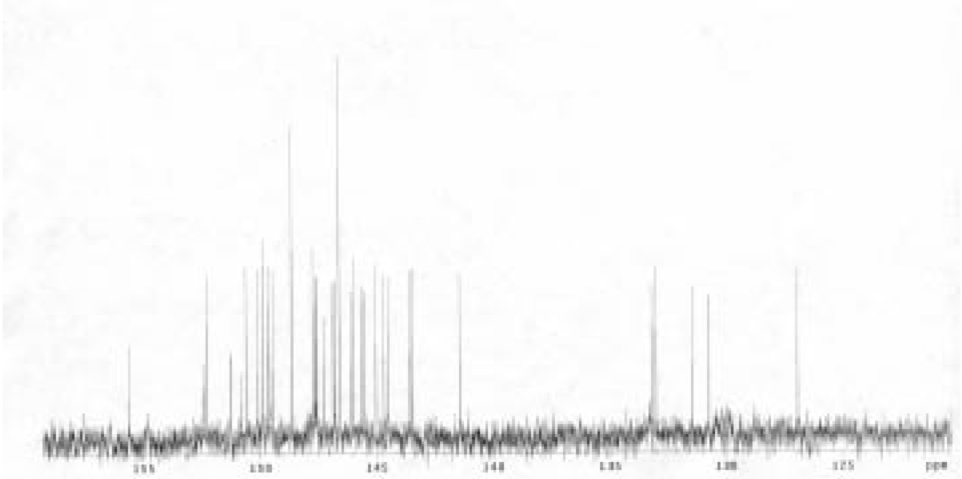
Expansion of the sp^{2} region of the ^{1}H-decoupled ^{13}C NMR spectrum of **1**



^{1}H-decoupled ^{13}C NMR spectrum of **2** in 50:50 CS_2/dioxane-d_8



^{1}H-decoupled ^{13}C NMR spectrum of **3** in 50:50 CS_2/dioxane-d_8.



sp^{2} Region of the ^{1}H-coupled spectrum of **3**. This experiment supresses the resonances from residual toluene, which have an associated nOe.