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MATERIALS AND METHODS

Isotopically labeled (10-15% ^{13}C) fullerenes were purchased from MER Corporation, Tucson, Az. Enriched C_{60} was isolated via the method of Tour (*J. Am. Chem. Soc.* **1992**, *114*, 7917). The remaining chemicals were reagent grade (Aldrich) and were used without further purification. The ^{13}C NMR spectrum was acquired with a Bruker AM-400 spectrometer. A Beckman DU-7 spectrophotometer was used to obtain UV-vis spectra. All laser-desorption mass spectra were acquired with a PerSeptive Biosystems Voyager linear laser-desorption time-of flight mass spectrometer equipped with a nitrogen laser (337 nm) and operated in positive ion mode. The accelerating voltage was held at 28 kV. A one-point calibration of the mass spectrometer was performed using C_{60} .

EXPERIMENTAL PROCEDURES

Preparation of $(\text{C}_{60})_2\text{C}_2$. Following the known procedure (Shevlin, P. B. *J. Am. Chem. Soc.* **1972**, *94*, 1379), diazotetrazole was formed via slow addition of concentrated HCl (0.4 mL) to a solution of 5-aminotetrazole monohydrate (0.082 g, 0.79 mmol, 11 equiv) in THF (1.5 mL) followed by addition of isoamylnitrite (0.1 mL, 0.74 mmol, 11 equiv) at 5 °C. This solution was poured into diethyl ether (2 mL) and water (2 mL). The layers were quickly separated and the organic layer immediately added to a solution of C_{60} (0.050g, 0.069 mmol, 1 equiv) in benzene or toluene (40 mL) and heated to reflux. Upon cooling, the mixture was filtered through a 1 inch pad of SiO_2 with toluene as eluant. Fourteen HPLC peaks were observed (Cosmosil Buckyprep column, 4.6 x 250 mm, 10% hexane/toluene mobile phase, flow rate 1 mL/min, monitored at $\lambda=325$ nm): 15.0 (C_{60}), 17.9, 18.8, 20.5, 21.4, 24.9, 28.0, 29.6, 33.3, 34.9, 39.1, 41.4, 46.4, 50.9, 56.9 and 63.0 (1) min. $(\text{C}_{60})_2\text{C}_2$ (**1**) was isolated employing 100 % toluene as the mobile phase (20.2 min retention time) and obtained in 3 % yield (3.0 mg, 0.0021 mmol). C_{119} , **4**, **5** and **6** were observed by LD-TOF mass spectrometry in fractions isolated from a cluster of peaks centered at 11.0 min. The carbon-13 NMR data for **1** is shown in Table 1:

Table I. ^{13}C NMR data for **1** (*o*-dichlorobenzene- d_4).

Compound 1					
δ (ppm)	#C	δ (ppm)	#C	δ (ppm)	#C
145.31	8	143.95	8	142.81	8
145.12 ^a	16	143.51	8	141.86	8
144.97	8	143.30	8	138.51	8
144.49	8	143.20	8	60.10	4
144.30	4	143.00	4		
144.15	6	142.93	8		

(a) Represents close or overlapping resonances

