Fullerenes without Symmetry: Crystallographic Characterization of C_1(30)-C_{90} and C_1(32)-C_{90}†

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Supporting Information

Table of contents

Experimental and computation section 2

Figure SI-1 HPLC profiles 3

Figure SI-2 HPLC profiles 4

Figure SI-3 LDI-TOF mass spectra of C_{90}(II) and C_{90}(III) 4

Figure SI-4 Numbering Schemes for C_1(32)-C_{90} 5

Figure SI-5 Numbering Schemes for C_1(30)-C_{90} 5

Synthesis of C_{90} isomers. A 8 × 150 mm graphite rod filled with Sm_2O_3 and graphite powder (Sm:C atomic ratio 1:40) was vaporized as the anode in DC arc discharge under optimized conditions.1 The raw soot was sonicated in odicholorobenzene for eight hours and then filtered with aid of vacuum. After removing the solvent with a rotary evaporator, chlorobenzene was added to re-dissolve the dry extract. The resulting solution was subjected to a five-stage HPLC isolation process without recycling.

The first stage was carried out using a Buckyprep-M column with chlorobenzene mobile phase. Figure SI-1 shows the initial chromatogram. The eluent in the range from 9.1 to 11.8 minutes, which is labeled as F in Figure SI-1a, was collected. Fraction F was then reinjected into a 5PBB column with chlorobenzene eluent, and fraction F1 and F2 were collected (Figure SI-1b). The C_{90}(I) isomer was obtained from F1 after further chromatographic separation as described previously.

F2 was injected into Buckyprep-M column with toluene as eluent to remove the dominant endohedral fullerenes, F21 was collected in figure SI-2a. The same HPLC condition purified F21 was then passed through Buckyprep column toluene flow, with F211and F212 collected in SI-2b. C_{90}(II) and C_{90}(III) were obtained from purified F211 and F212 in SI-2c and SI-2d, respectively.

Mass spectra (negatively charged mode) of isolated pure samples of C_{90}(II) and C_{90}(III) are shown figure SI-3 LDI-TOF.

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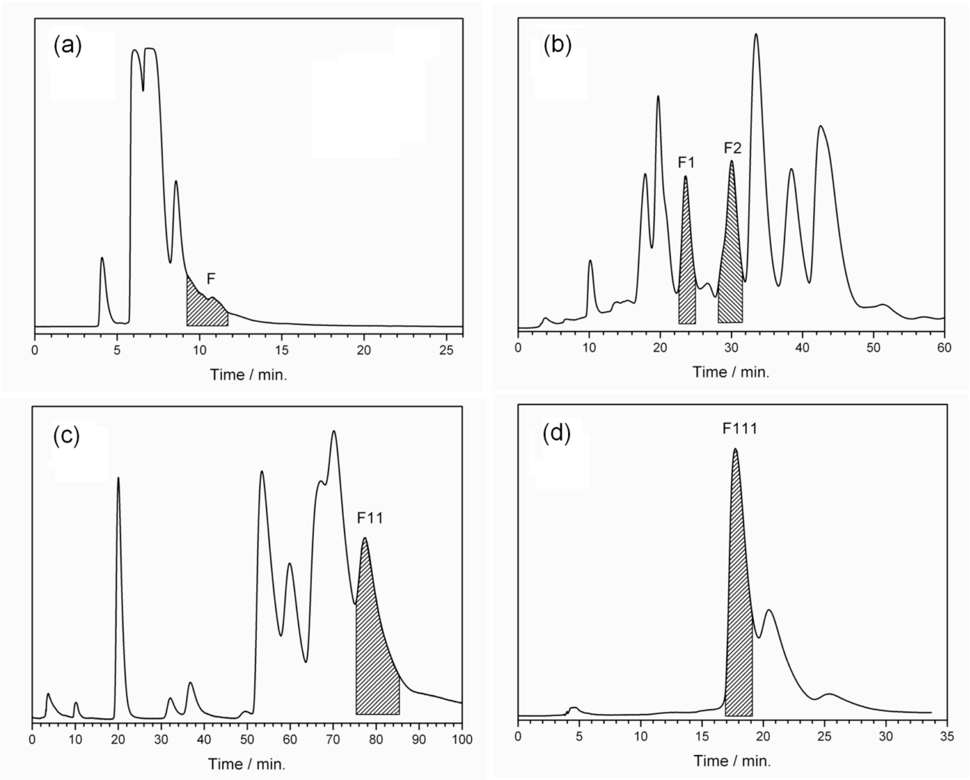

Figure SI-1 Chromatograms of the first to fourth stage separations, (a) the first stage using Buckprep-M column with chlorobenzene elute, (b) the second stage using 5PBB column with chlorobenzene elute, (c) the third stage using 5PBB column with toluene and (d) the fourth stage using Buckyprep-M with toluene elute. The HPLC conditions are: (a) and (d) flow rate 4.0 mL/min, (b) and (c) 4.5 mL/min. All used detecting wavelength of 450 nm.

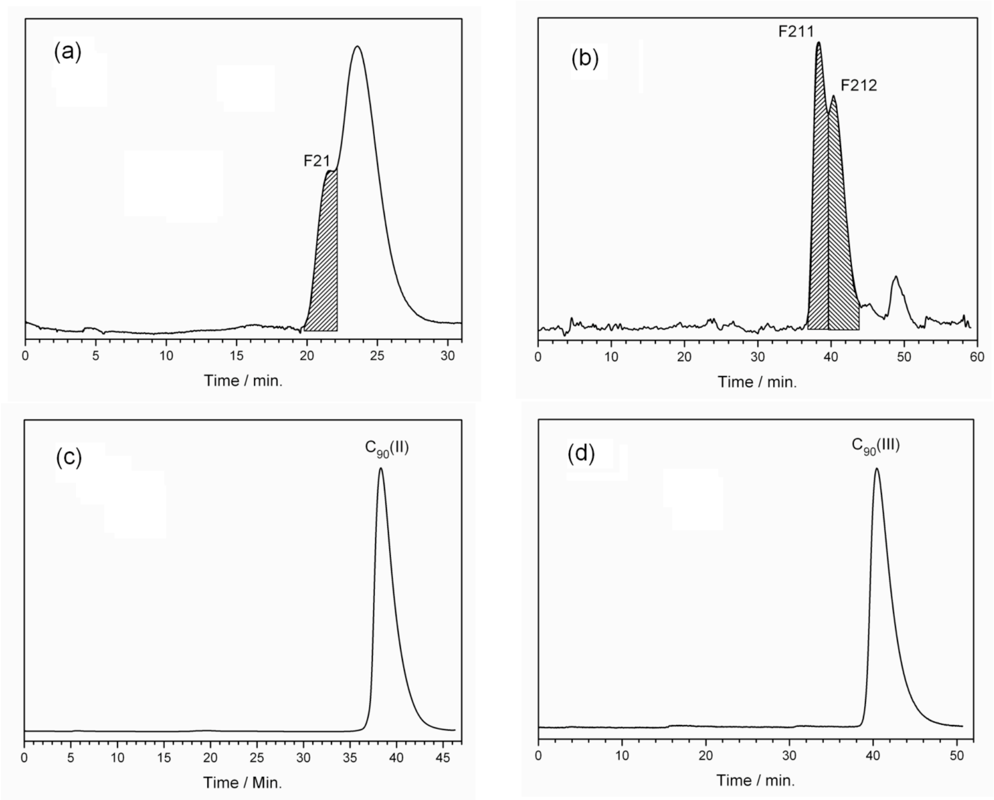

Figure SI-2 Chromatograms of the third to fourth stage separations for C_{90}(II) and C_{90}(III) isomers, (a) the third stage using Buckyprep-M column with toluene elute, (b) the fourth stage using Buckyprep column with toluene elute, (c) and (d) the fifth stage using Buckyprep column with toluene for the final separations of C_{90}(II) and C_{90}(III), respectively. The HPLC conditions are: (a) flow rate 4.0 mL/min; (b), (c) and (d) flow rate 4.5 mL/min. All used detecting wavelength of 450 nm.

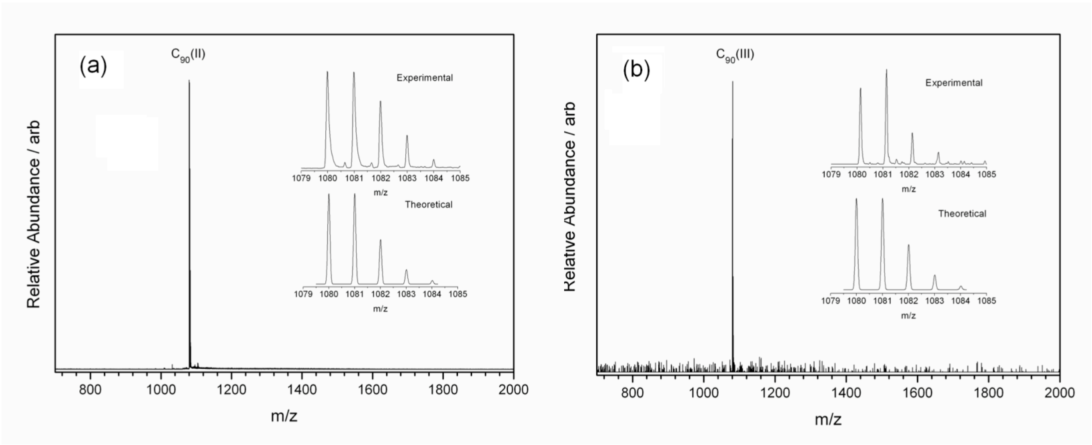

Figure SI-3 LDI-TOF mass spectra (negatively charged mode) of isolated pure samples of C_{90}(II) and C_{90}(III).

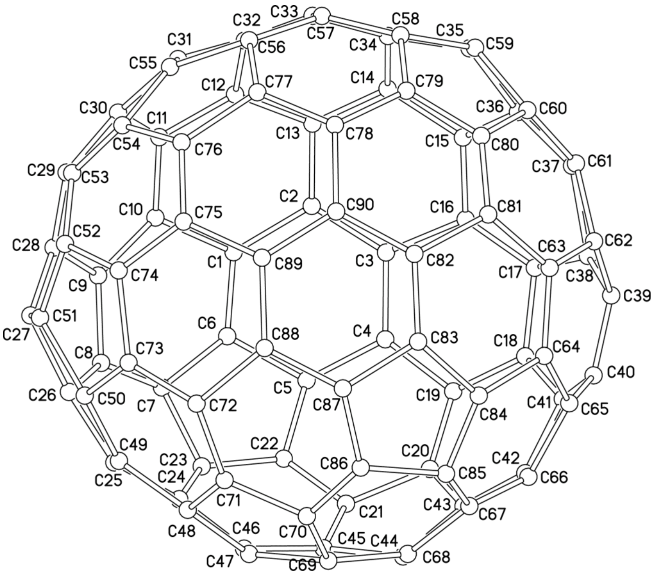

Figure SI-4 The numbering scheme used for C_1(32)-C_{90}.

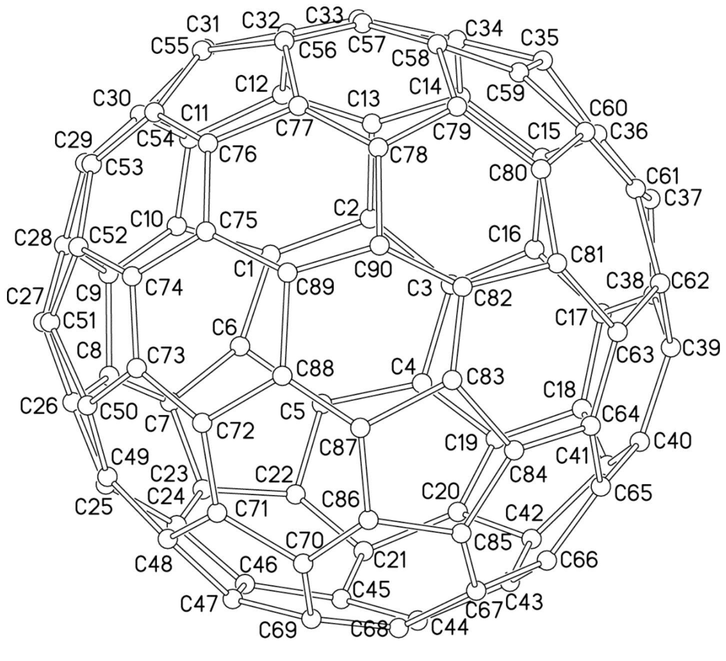

Figure SI-5 The numbering scheme used for C_1(30)-C_{90}.