Direct isolation of metallofullerenes by chemical reduction

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Endohedral metallofullerenes have attracted much interest because of their unique geometrical and electronic structures. Production, isolation, and characterization of a variety of metallofullerenes have been performed during recent years since the first macroscopic production of $La@C_{82}$ and the first isolation of metallofullerenes $Sc_2@C_{84}$ and $La@C_{82}$ by high performance liquid chromatography (HPLC). Since then, HPLC isolation is always a unique method to purify metallofullerenes. However, HPLC is limited to those soluble metallofullerenes and is a tedious, time-consuming procedure and this makes it difficult to obtain macroscopic quantities of pure metallofullerenes and restrict their experimental characterization of the solid-state properties and further practical applications.

In our recent report, $Gd@C_{82}$ and $Gd_2@C_{80}$ anion mixtures were reduced selectively and extracted directly from the raw soot in a mixed solvent of THF/toluene, but $Gd@C_{82}$ and $Gd_2@C_{80}$ were not isolated, respectively. For the polarity of solvents exhibits significant contribution to the reduction potentials of fullerenes, the different THF/toluene ratios are used to adjust the reduction potentials of fullerenes. Here we report that pure $Gd_2@C_{80}$ anions can be obtained directly from the raw soot by precisely controlling reduction potentials. Subsequently, pure $Gd@C_{82}$ anions can also be obtained the reduced soot by further reduction. $Gd@C_{82}$ and $Gd_2@C_{80}$ anions were characterized by negative-ion MALDI-TOF mass spectrometry and UV-vis-NIR absorption spectra. This study may pave a pathway to isolate metallofullerenes in large quantities.