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Copper Chloride as a Selective Precipitation Agent for Purifying Endohedral Metallofullerenes

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Abstract

Goal: Develop a new separation strategy for isolating new molecules containing metals, nitrogen, or oxygen trapped inside carbon cages. These metallofullerenes possess an electron-rich surface that reacts with electron-deficient Lewis acids. We report an improved separation approach which replaces the currently used aluminum chloride method with copper (II) chloride. Our results demonstrate the successful and selective separation of a diverse array of metallofullerene systems.

Experimental

- **Plasma Arc Synthesis of Metallofullerenes:** 1 inch cored and packed graphite rods of 100% Sc_2O_3 or Er_2O_3, gas flow of 0.8-6 torr air/min, and 40-60 minutes vaporization time.
- **HPLC Analysis:** 4.6 x 250 mm PYE column, 0.75 mL/min toluene mobile phase, 360 nm UV detection, 50-500 µL injection volume.
- **Reaction Conditions:** Metallofullerene extract is placed in a round bottom flask and dissolved in CS_2. While stirring, Lewis acid (i.e., $CuCl_2$ or $AlCl_3$) is added to initiate selective complexation and precipitation. Upon filtration, the solid material is placed in a beaker for decomplexation with ice, water, and subsequent dissolution into the CS_2 organic layer. HPLC traces and MALDI mass spectral data are provided below.

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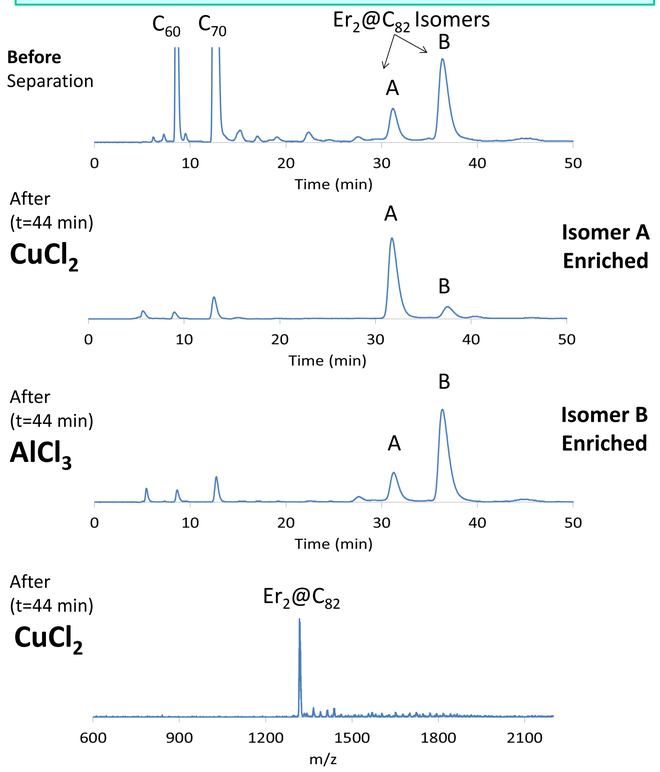
Conclusions

- **Wide Range of Use:** $CuCl_2$ success for metallic oxide, metallic nitride, and classical metallofullerenes.
- **New Separation Method:** Discovery that $CuCl_2$ is better and replaces the currently practiced method of using $AlCl_3$ to purify endohedral metallofullerenes

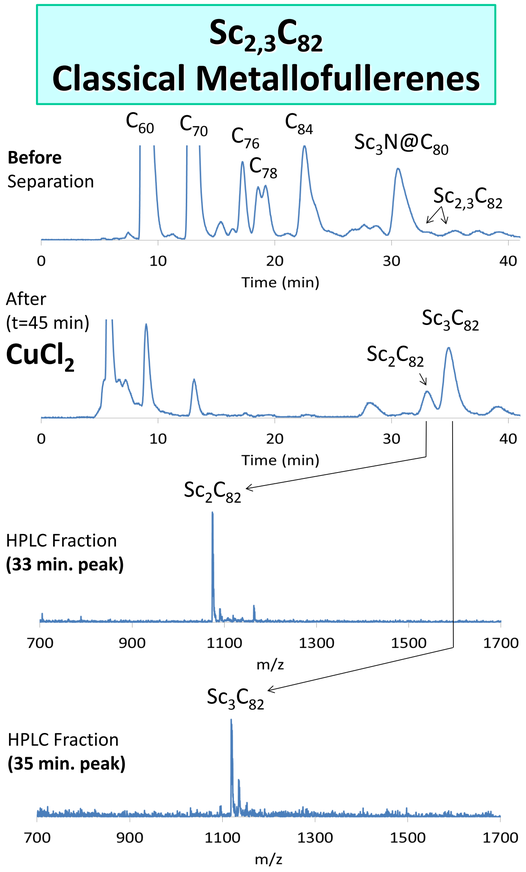
Results (Erbium-based Molecules)

$Er_2@C_{82}$ Classical Metallofullerenes

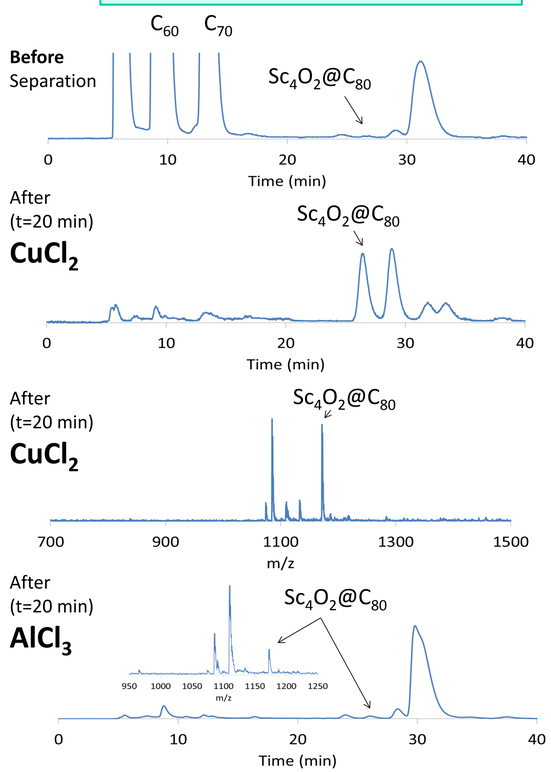


Results (Scandium-based Molecules)

$Sc_{2,3}C_{82}$ Classical Metallofullerenes



$Sc_4O_2@C_{80}$ Metallic Oxide Fullerenes



$Sc_3N@C_{78}$ Metallic Nitride Fullerenes