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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$_2$O$_3$ or Er$_2$O$_3$, 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 tolune 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 complexionation 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.

Results (Erbium-based Molecules)

**Er$_2$@C$_{82}$ Classical Metallofullerenes**

Before Separation

After (t=44 min) CuCl$_2$

After (t=44 min) AlCl$_3$

Results (Scandium-based Molecules)

**Sc$_{2,3}$C$_{42}$ Classical Metallofullerenes**

Before Separation

After (t=45 min) CuCl$_2$

After (t=20 min) AlCl$_3$

HPLC Fraction (33 min. peak)

**Sc$_{4}$O$_2$@C$_{80}$ Metallic Oxide Fullerenes**

Before Separation

After (t=20 min) CuCl$_2$

After (t=60 min) AlCl$_3$

HPLC Fraction (35 min. peak)

**Sc$_3$N@C$_{78}$ Metallic Nitride Fullerenes**

Before Separation

After (t=60 min) CuCl$_2$

After (t=60 min) AlCl$_3$

HPLC Fraction (55 min. peak)

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.

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HPLC Analysis:

- **Arc Synthesis of Metallofullerenes**: Prof. Steven Stevenson (Prof. Steven Stevenson)