

Purification of C60 Fullerene using Non-aqueous Reverse Phase with RediSep Rf Gold® Columns

Abstract

The fullerene family, especially C60, has appealing photo, electrochemical, and physical properties which can be exploited in both medical and material science applications. Fullerenes are formed in soot produced in an inert atmosphere. This process results in a mixture of fullerenes which typically range in size from C20 through C70 and larger. Purification of fullerenes is hampered by solubility in most solvents. The use of non-aqueous reverse phase and adroit choice of solvents allow the isolation of C60 fullerene from soot with RediSep Rf Gold® C18 columns.

Experimental

Method development

Initial method development was performed with RediSep® TLC plates. Authentic samples of C60 and C70 were also run with the soot mixture. Various combinations of hexane/ toluene, and hexane/2-propanol, hexane/dichloromethane, and hexane/carbon disulfide were run with RediSep® Rf silica TLC plates (PN 69-2203-400), neutral RediSep® Rf alumina TLC plates (PN 69-2203-569), RediSep Rf Gold® amine TLC plates (PN 69-2203-573), and RediSep Rf Gold® diol TLC plates (PN 69-2203-574). RediSep® Rf C18 TLC plates (PN 69-2203-586) were run as non-aqueous reverse phase using 2-propanol/ethyl acetate, 2-propanol/dichloromethane, 2-propanol/toluene and toluene/carbon disulfide. Plates were visualized with 254nm UV light.

The RediSep Rf Gold diol plate developed with 90:10 hexanes: carbon disulfide exhibited retention and resolution with minimal tailing, but the solubility of fullerenes in this solvent system was low so that only a small sample load could be purified with any resolution. RediSep Rf C18 run with 2-propanol and toluene also showed retention with some resolution. The 2-propanol/toluene solvent system allowed a larger sample load to be run due to improved solubility of fullerenes in these solvents making it the preferred solvent system.

Purification

A soot sample (25 mg) was dissolved in 2.5 mL carbon disulfide and loaded onto a 5 g RediSep® Rf C18 solid load cartridge (PN 69-3873-237) A RediSep Gold® C18 column (30 g, PN 69-2203-335) was run with a 2-propanol/toluene gradient according to *Table 1*. Purification was run on a CombiFlash® Rf 200 with a UV-vis detector (PN 62-5230-008) at 310 nm.

Table 1: Gradient table used to purify C60 fullerene. Solvent A is 2-propanol; solvent B is toluene

Segment Duration (column volumes)	% B
Start	25
20	25
15	100
15	100
0	0
10	0

The Rf solvent system was determined via TLC using RediSep Rf TLC plates. Fraction purity was determined by TLC with RediSep diol TLC plates (10% carbon disulfide in hexanes). TLC was compared to authentic samples of C60 and C70 fullerenes.

Results and discussion

Method development

RediSep Rf silica TLC plates were initially tested because they work well for many situations; however, they tend to show poor retention with very non-polar samples such as fullerenes. RediSep Rf alumina TLC plates often work well with non-polar compounds, but failed to resolve the fullerenes with the solvents tested.

RediSep Rf Gold diol TLC plates and RediSep Rf amine TLC plates are less polar than RediSep Rf alumina and RediSep Rf silica TLC plates and also exhibit different selectivity. These media run as normal phase when developed with organic solvents^{1,2}. While the amine plate failed to resolve the fullerenes, the RediSep Rf diol TLC plate did resolve C60 and C70 with a solution of 90:10 hexanes: carbon disulfide. Unfortunately, this solvent system has limited fullerene solubility which limits the amount of sample that could be loaded on a column. However, the diol plate allowed evaluation of fraction purity.

RediSep Rf C18 TLC plates run in 60:40 2-propanol: toluene also showed separation of the fullerenes, with

1. Silver, J.E.; Bellinghausen, P.; Fowler, N.; Pipes, R. Diol columns – pretend they're normal phase, presented at the American Chemical Society meeting Fall 2009
2. Silver, J.E.; Bellinghausen, P.; Fowler, N.; Pipes, R. Method Development Strategies for Amine Bonded Phase Columns for Medium Pressure Liquid Chromatography, presented at the American Chemical Society meeting Fall 2009

slightly reduced resolution compared to the diol plates. Fullerenes are more soluble in the RediSep Rf Gold C18 solvent system compared to the RediSep Rf diol TLC solvent system, so the fullerenes were purified on RediSep Rf Gold C18. A single run on a RediSep Rf Gold C18 column with a focusing gradient from 0 to 30% toluene in 2-propanol showed that 30% toluene would provide better resolution compared to 40% toluene.

The solvents chosen for the RediSep Rf TLC runs are not traditional flash solvents. The solvents were chosen on the basis of known fullerene solubility and availability. Although non-aqueous reverse phase is not a commonly employed technique, it is useful when purifying non-polar compounds.^{3, 4}

Purification

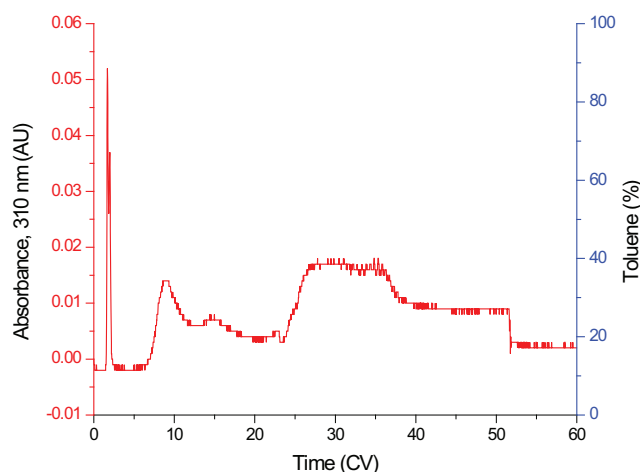


Figure 1: Purification of C₆₀ using an Rf-200 flash system and RediSep Gold C18 column

The peak from 3-11 CV was combined and dried to give 9.6 mg of pure C₆₀ (38%). RediSep Rf diol TLC showed C₆₀ tailing into the gradient. The peak observed at ~15 CV contains C₇₀. Peaks after the gradient begins are a mixture of other compounds in the soot, some C₇₀ and C₆₀, and a baseline change due to absorbance from the solvent.

- Snyder, L.R.; Kirkland, J.J.; Glajch, J.L.; *Practical HPLC Method Development*, 2nd Ed, John Wiley & Sons, 1997, p.264
- Silver, J.E.; Bellinghausen, P.; Fowler, N.; Pipes, R. Facile Isolation of Carotenoid Antioxidants from *Solanum lycopersicum* using Flash Chromatography, presented at the American Chemical Society meeting Fall 2009



Figure 2: Fractions on right exhibited the characteristic purple color of purified C₆₀ fullerene

Conclusion

The CombiFlash Rf-200 and RediSep Rf Gold C18 columns allow for the flash purification of C₆₀ fullerenes. RediSep TLC plates allow a large variety of solvent systems to be tested quickly with minimal sample usage. The major impediment to resolving fullerenes is solubility in solvent systems that provide good interaction of the fullerenes with the columns.

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