Isolation of Individual Fullerenes C_{60} and C_{70} by Extraction and Column Adsorption Chromatography on Graphite

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Individual fullerenes (C_{60} and C_{70}) can be rapidly and effectively isolated from initial sooty material by extraction from the solid phase with organic solvents and column absorption chromatography on graphite.

The separation of individual fullerenes from soot is still rather difficult. In this communication we describe an effective column chromatography method for the isolation of fullerenes C_{60} and C_{70}, extracted from the initial soot obtained in the Institute of Solid State Physics, Russian Academy of Sciences by vaporization of pure graphite (*cf.*, *e.g.*, ref. 1).

The soluble soot fraction was extracted in a Soxhlet apparatus by hexane during 20–25 h of continuous boiling. Hexane extracts from soot yielded a C_{60}/C_{70} mixture in the proportion 8:1–9:1, corresponding to published work.[2]

There are reports on separation techniques for fullerenes in which they are isolated individually by sublimation,[3] column chromatography on alumina[4] and HPLC on silica.[5]

In common with other workers, we tested alumina of activity 1 as a sorbent. This useful sorbent does ensure separation, but the chromatographic zones are diffuse and their volumes are large. As a result the isolation process is lengthy, with low efficiency.

This appears to us quite natural since the main adsorption mechanism in this case is the surface electrostatic induction produced by the electrostatic field of oxygen and aluminum ions.[6]

For similar reasons, silica gel is of little use since adsorption often proceeds by means of hydrogen bond formation between adsorbate and surface hydroxyl groups;[7] this possibility does not exist for fullerenes.

Satisfactory results may be achieved using sorbents on which adsorption is determined primarily by the molecular mass of the sample, and proceeds by the dispersion forces mechanism.

In this communication we used pure graphite for the separation of individual fullerenes. This material has a low porosity and a high affinity for the components to be separated. The separation of fullerenes was performed as follows. The hexane extract from a Soxhlet apparatus was evaporated to dryness, a C_{60}/C_{70} mixture (50 mg) was dissolved in 20 ml toluene, and the solution was fed into a 3×30 cm glass column filled with sorbent, prepared from graphite (>99%, MPG 6A, Russia). The eluent for C_{60} was hexane–toluene (50:50) and the eluent for C_{70} was toluene–xylene (70:50). Under these conditions the volume of the C_{60}-containing magenta fraction was up to 200 ml, and the volume of the C_{70}-containing yellow–orange fraction was up to 500 ml.

However, the eluted volumes obtained on alumina under our optimal conditions were, for an analogous sample, 2.5–3 times higher than those on graphite. The method described above is preferable to the one described earlier,[8] where the utilization of graphite as a sorbent allows the isolation of only C_{60}. Using our method it is possible to isolate both C_{60} and C_{70} continuously; our column has not lost its separating ability even after three months of continuous work.

After distillation of the solvents 40–45 mg of C_{60} and *ca.* 5 mg of C_{70} were obtained. The purity of the individual fullerenes was tested spectrophotometrically in the UV and visible regions by HPLC, IR-Fourier and mass spectrometric methods (*cf.* ref. 9). Purity: $99.7 \pm 0.1\%$ per mass for C_{60} and $98.0 \pm 1.0\%$ per mass for C_{70}. Mass spectra of the pure fullerenes are shown in Fig. 1.

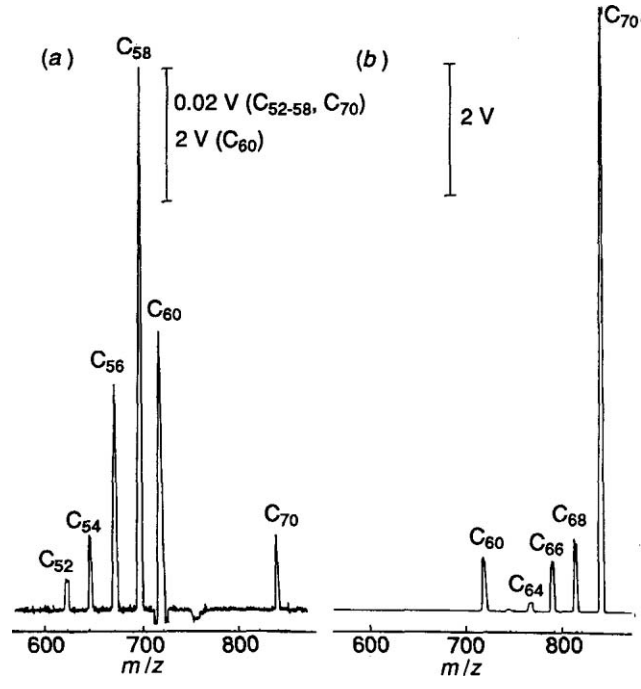

Fig. 1 Mass spectra of pure C_{60} (*a*) and C_{70} (*b*); ionization with electron impact (70 eV)

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Received: Moscow, 14th April 1992
Cambridge, 28th May 1992; Com. 2/02070J