

Undergraduate Lab Module in Fullerene Chemistry

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Rice University has now offered its laboratory module course in fullerene chemistry for four consecutive years. The course, which is open to upper-level undergraduate chemistry majors, runs for seven weeks and carries one semester-hour of credit. Working in pairs, students begin with Soxhlet extraction of commercial fullerene soot, using toluene as the solvent. Extracts are concentrated using rotary evaporators and then separated into C₆₀, C₇₀, and higher fractions by multiple HPLC injections onto a Cosmosil Buckyprep 4.6 x 250 mm column. Students evaporate their separated fractions and weigh the products. They then measure UV-vis absorption spectra for C₆₀ and C₇₀, finding absorption peaks and molar absorptivities. Samples are also submitted for ¹³C NMR and MALDI mass spectrometric analysis. Students build models of C₆₀ and C₇₀ to help them interpret the NMR spectra.

In the second half of the module, students use their fullerene samples in electrochemistry and photophysics experiments. A computer-controlled BAS potentiostat allows cyclic voltammetry and differential pulse polarography measurements. Multiple reduction potentials are determined for both C₆₀ and C₇₀. Students also measure triplet state decay kinetics for dilute solutions of C₆₀ and C₇₀. Samples are first degassed on a vacuum line to remove dissolved oxygen. They are then placed in a custom-built apparatus that irradiates them with pulses from a 532 nm laser. The resulting triplet state population is monitored as a function of time by the absorption of a diode laser beam, as recorded on a digitizing oscilloscope. Data analysis provides kinetic decay parameters for several combinations of sample concentration and excitation energy.

Although this module requires a substantial investment in instrumentation, most of the equipment can also be used in other projects. Course capacity is limited to eight students because of facilities and time constraints. Despite its relatively demanding content, this laboratory module has become a popular and oversubscribed course among Rice chemistry students.