

### **3-D carbon by Marie-Pascale Manseau**

Physical sciences

Experiment

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#### **Project summary**

At the beginning of my fourth year of secondary school, I decided to take on the challenge of synthesizing fullerenes. To do so, I designed a device in conjunction with Cégep de la Pocatière. The experiment was conclusive and enabled me to learn a lot about the subject.

#### **Project report**

At the beginning of my fourth year of secondary school, I decided to learn about a subject that was completely foreign to me. I decided to synthesize fullerenes using a device that did not yet exist. This new family of carbons—especially its most illustrious member,  $C_{60}$ —quickly captured my attention with its unusual characteristics and properties. A little-known molecule with tremendous potential, an uncommon synthesis and a number of things to discover were among the ingredients that led me to plunge head first into this Science Fair project.

To begin with, let me introduce you to the large family of fullerenes ( $C_n$ ), the most recently discovered carbons. Let's discuss the most abundant and promising member of this illustrious family: buckminsterfullerene,  $C_{60}$ . As its chemical formula indicates, it is composed of 60 carbon atoms joined together by single or double bonds to form an icosahedron with 12 pentagonal and 20 hexagonal faces, a design that resembles a soccer ball. This particular molecule, which revolves around itself 100 million times per

second, crystallizes in a face-centred cubic arrangement. The other fullerenes are carbon cage structures made up of 12 pentagons and an even number of hexagons.

Discovered in 1985 by R.F. Curl, H.W. Kroto and R.E. Smalley, this atomic ball derives its name from the American architect R. Buckminster Fuller, whose widely renowned geodesic dome design is similar to the structure of fullerenes. In the case of C<sub>60</sub>, the highly symmetrical structure makes it incredibly resistant.

We therefore undertook the task of synthesizing these fascinating molecules. First, we looked for documentation on the subject at the library and discovered articles that briefly explained the protocol to follow in order to carry out this experiment. The principle was simple: we had to induce the evaporation of graphite using an electric arc of  $\pm 100$  A, in a low-pressure atmosphere of inert gas. Certain parameters had to be respected. The pressure had to approach 100 torrs (13.3 kPa), etc. Since we had neither the technical skills or the equipment needed to manufacture the device required for this synthesis, we contacted a technician at Cégep de la Pocatière. Through discussions with the technician, and by referring to articles and relying on our imagination, we were able to determine which components we needed and to develop a model of the device.

The following is a diagram of the device:

Steps involved in the synthesis:

1. Make the necessary adjustments (pressure, flow, amperage, refrigeration).
2. Activate the electric arc.
3. Rinse the device with toluene and set the liquid aside.
4. Use a Büchner funnel to filter with celite and collect the filtrate.
5. Evaporate the toluene.
6. Store the residue in a dark area under nitrogen.

We relied on several different articles to make the adjustments. Thanks to our cooled electrodes, the device was able to operate continuously, which greatly contributed to the system's effectiveness.

A more difficult step involved the transferring of the toluene from the device to the beaker. To facilitate this step, we used a glass rod (principle of capillary action).

We decided to use a Büchner filter. The suction created by the tap helped speed up the operation.

To preserve the fullerenes, we allowed the solvent to evaporate in a small tinted bottle in order to prevent too much light exposure. We then introduced some nitrogen to decrease the amount of oxygen present, since these two factors—UV and O<sub>2</sub>—degrade the product.

Of course, we encountered a variety of technical problems. One of the first was the alignment of the electrodes, which was not perfect. We had to constantly support them to keep them from spreading apart: this would cause the electrodes to degrade, without significant fullerene production. Then, during our initial filtration, we noticed that some soot was penetrating the filter. We used celite to remedy the problem. After experiencing some difficulty with the celite, we placed a filter beneath the dampened powder and another above it to prevent piercing the celite layer when pouring the solution to be filtered.

Several articles mentioned that toluene takes on a reddish hue when it contains fullerenes in solution. We were therefore tremendously satisfied to observe that our filtrate no longer had the transparency of pure toluene. We considered this obvious colouring as proof of the experiment's success. After evaporating the toluene, we obtained 40 mg of residue, or fullerenes. We decided to go even further and paid a visit to the prestigious Université Laval. There, a specialist wanted to produce IR and NMR spectra of our sample, as well as those of the pure C<sub>60</sub> that we had ordered. We encountered a problem on the first attempt: our samples contained traces of toluene, which masked the fullerenes. So we produced a second set of spectra. Those of our sample versus that of the pure C<sub>60</sub> were not identical. We attributed this to the fact that our sample contains several different types of fullerenes and, unlike the commercial sample, a few impurities. It is also possible that the toluene interfered.

From their earliest discovery, buckyballs (C<sub>60</sub>), as they are referred to by some, captured the interest of scientists throughout the world. They showed tremendous promise in a number of fields. From a miracle lubricant for electronic components to superconductors, this molecule, of the order of an angstrom, was destined to conquer the world. Scientists' enthusiasm waned, however, when they turned their attention to a derivative: the nanotube—a hollow tube made up of one or more layers of rolled-up carbon atoms whose extremities are half-C<sub>60</sub>.

Whether doped or not (a doped structure is one in which one or more elements have been introduced), nanotubes also have fascinating properties and a range of possible applications. Given their tremendous resistance, they could be used to produce solid, lightweight cables, to store hydrogen or to manufacture electronic components. As is the case with fullerenes, the main drawbacks involve difficulty producing large quantities and the transition from the laboratory to the manufacturing plant.

Although basic research and experiments involving fullerenes never really stopped, there has been renewed interest in terms of their concrete applications. This time, health-care researchers are examining these molecular balls and trying to develop certain medical applications. One company, C-Sisty Inc., announced the beginning of production based on fullerenes for the treatment of HIV.

We will probably be witnessing new discoveries derived from these fascinating molecules in the coming years, since hundreds of researchers are working feverishly to pursue such objectives.

The hundreds of hours invested in this project did bear fruit, from both an experimental and personal standpoint. Synthesizing fullerenes required learning a lot about these fascinating molecules, which was very enriching. One day, I hope to be able to work on this subject again and to share my passion for this promising nanometric world.