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Exploring Carbon's Allotropy: A Pupil-Led Synthesis of Fullerenes from Graphite

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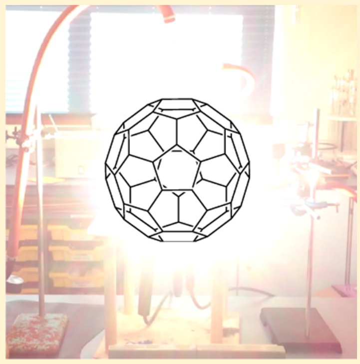
S *Supporting Information*

ABSTRACT: A successful pupil-led effort to build a fullerene generator and to use it to synthesize C_{60} and C_{70} is reported, demonstrating that such an activity is feasible in schools. Suggestions are made as to how fullerene synthesis and purification could be used in conventional high school chemistry lessons, particularly in the context of teaching the concept of allotropy, but also to illustrate some of the contrasting properties of simple versus giant covalent structures.



KEYWORDS: *High School, Introductory Chemistry, Laboratory Instruction, Problem Solving, Enrichment, Student-Centered Learning, Synthesis*

In this communication we would like to report the success of a team of 17−18 year old school pupils who were set the challenge of constructing a fullerene generator and using it to produce buckminsterfullerene (C_{60}) at relatively low cost, relying wherever possible upon existing resources available within their school. Previously in this journal, Potter[1] and Craig[2] have each reported means of generating fullerenes aimed at educators of undergraduates, based on the method of Krätschmer et al.[3] We would like to augment those reports by describing how Potter's method can be adapted to safely and affordably synthesize fullerenes in a reasonably well resourced school. The benefits to the pupils involved in our "C_{60} Project" were immense, and so we hope that this communication will stimulate other teachers to set up similar projects. The inherent beauty of C_{60}'s structure means that it cannot fail to invoke aesthetic appreciation[4] and so it readily captures the imagination of chemistry students.[5] Furthermore, its association with the 1996 Nobel Prize in Chemistry[6] and its connection to the subsequent boom in fullerene chemistry[7] means that teachers will find little difficulty in inspiring pupils to get involved in laboratory work not only to synthesize fullerenes but also to build apparatus for this purpose.[8]

EXPERIMENTAL DETAILS

Equipment

We needed to spend around £220 on essential resources and equipment which were not already available within our school (e.g., an arc welder, £176). In addition to the items in Box 1, we also made use of an in-line pressure regulator and a Soxhlet extractor, but neither of these items is essential (see Supporting Information for details).

Box 1: A summary of the key equipment and resources needed for fullerene generation, purification and characterization in a high school setting.

Non-consumable equipment	Consumable resources
Vacuum pump	Graphite components
Arc welder	Helium
Bell jar and thick acrylic base	Vacuum grease
Aluminium rod and copper cable	Silica thin layer chromatography plates
Thick walled tubing, connectors and clips	Toluene
In-line pressure gauge	Hexane

Method

Our pupils followed a simplified version of Potter's method,[1] with additional guidance provided by The Creative Science Centre.[9] Carbon was vaporized by using an arc welder to create an electrical arc between two high purity graphite electrodes contained within a helium atmosphere. A glass bell jar was used to house the electrodes, with the outlet at the top of the bell jar

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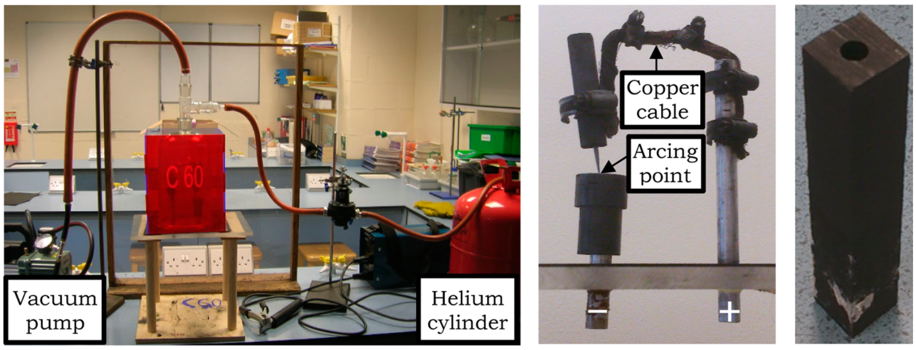

Figure 1. Left: The top of the bell jar divided to a high vacuum pump (left) and to a helium cylinder (right). The bell jar itself is concealed in this photo by an acrylic surround, employed in case of an implosion. Middle: Close-up photograph of the acrylic base (viewed "edge-on") penetrated by the two aluminum rods (labeled according to their polarity when linked up to the arc welder). Jubilee clips have been used for the connections, and for preventing the copper cable from fraying. Right: Close-up photograph of the graphite electrode holder, which in the middle photograph is shown containing the sharp-tipped positive electrode.

dividing to a helium cylinder and to a high vacuum pump (Figure 1, left). The bell jar sat on a 1 cm thick acrylic base (an intervening rubber sheet helped to achieve an airtight seal between the base and the bell jar), through which passed two aluminum rods (Figure 1, middle). Below the acrylic base, these rods served as attachment points for the leads from the arc welder. Above the acrylic base, one of the aluminum rods (the rod connected to the negative terminal of the arc welder) had a graphite block fixed to it. By way of a bent copper cable stemming from the second aluminum rod, a graphite electrode holder was suspended vertically above this fixed graphite block (Figure 1, middle and right). The purpose of the graphite electrode holder was to keep the positive electrode in position horizontally while at the same time allowing it to slide through the holder in the vertical direction. Since only the positive electrode vaporizes when using direct current, this arrangement allowed the electrode to drop down gradually as its tip vaporized away. The tip of the positive electrode was sharpened to a fine point in order to help start the arcing process (this fine tip is visible at the arcing point as labeled in Figure 1, middle).

Prior to switching on the arc welder, the air within the system was replaced with helium to 1/7 atmospheric pressure. The fullerenes were then generated by applying a current through the electrodes of 105 A (the current was initially run for a few seconds at half this value in order to heat the electrodes: this helped to "kick start" the arcing process once the maximum current was applied). Owing to intense resistive heating, the current could be applied for only around 30 s before threatening to damage the apparatus (Figure 2, left). After allowing the apparatus to cool, the bell jar was lifted off in a fume hood and the crude soot scraped out (Figure 2, middle). A Soxhlet extraction, with toluene as the solvent, produced from the crude material an orange-red solution; this was then evaporated to dryness (Figure 2, right). Thin layer chromatography (TLC) against reference samples was sufficient to strongly suggest that C_{60} and C_{70} had been made, but unambiguous proof was provided by mass spectrometry (see Supporting Information for more details on the characterization, and also on how the apparatus was assembled).

Figure 2. Left: The arcing process underway. Middle: Following arcing, the bell jar is coated with a crude, sooty product. Right: Close-up photograph of the Soxhlet extractor. The orange-red coloration is due to C_{70}, which was also formed by the arcing process, and which masks the magenta color of C_{60} in toluene.

■ HAZARDS

The synthesis phase requires the use of a high current and of a glass bell jar being heated under reduced pressure. Arcing produces an extremely bright light, which should only be viewed through a welder's visor. The purification phase requires the handling of a finely powdered crude product and the use of flammable and harmful solvents. As such, we would advise any teachers intending to lead such a project to ensure that a rigorous and comprehensive risk assessment is carried out. In particular, we would recommend the implementation of a fail-safe "switch on" and "switch off" procedure with regard to the use of the arc welder so as to eliminate the possibility of electrocution.

■ IMPACT

This project had an enormous impact on the students involved owing to the wide range of skills that were required for a successful outcome: Design and engineering skills when constructing the various apparatus components, tenacity when attempting the actual synthesis (numerous subtle modifications were made to the apparatus before the final successful design described herein was achieved), and fundamental chemist's lab skills when carrying out the purification and characterization. Throughout the project excellent team working skills were required, as was the constant reference to safety protocols. The overall impact of this year-long project was perhaps best summed up by one of the pupils involved when he said

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J. Chem. Educ. XXXX, XXX, XXX−XXX

This experience has taught me that the most satisfying successes are those for which you have to work hard. The C_{60} Project pushed me beyond the limits of my practical and intellectual abilities in a way in which the A Level course did not. Furthermore, the prolonged collaboration with both staff and peers was an experience from which I learnt a huge amount, especially the ability to work in a team where each person's individual skill set is used to the greatest effect. The analytical and creative approach that we had to develop to tackle the problems we faced will be incredibly valuable to me in higher education and beyond.

CONCLUSION

It is both affordable and feasible for high school pupils to carry out fullerene synthesis projects. The benefits to the pupils involved are considerable and out of all proportion to the monetary expense demanded by such a project. We would urge teachers in other schools to instigate similar projects, and not to be put off by a feeling that such a project can only be conducted in a university setting. Moreover, while the initial design, build, and optimization of a fullerene generator is best conducted as an extracurricular project, once this work has been completed the apparatus could be used in the classroom as part of a sequence of lessons concerning the subtle concept of allotropy[10] and the structure−property relationships of covalent substances. For example, in the first lesson of a three lesson sequence, the arcing process could be demonstrated, dramatically illustrating the high energy conditions needed to vaporize a giant covalent structure.[11] The demonstration also underlines other teaching points such as the chemical inertness of helium and the conductivity of graphite. More significantly, the demonstration is probably unique in being the only way of showing in a school an interconversion between allotropes of carbon, thus complementing other well-established demonstrations of allotropic interconversions, such as those of sulfur[12] and phosphorus.[13] The Soxhlet extraction of fullerenes from the crude soot could be run as a demonstration during the second lesson, thus exemplifying the facts that C_{60} and C_{70} are not only soluble but also give rise to strongly colored solutions. In a third and final lesson, the pupils themselves could carry out TLC analysis of the crude Soxhlet extract against a reference sample. Not only would this provide a meaningful experience of the use of chromatography, but if a graphite pencil is used to draw a baseline on the TLC plate the practical would nicely contrast the solubility of simple covalent structures with the intractable nature of their giant counterparts.

ASSOCIATED CONTENT

Supporting Information

Details on the sourcing and use of the apparatus and resources, and on the characterization of the products. This material is available via the Internet at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest.

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(11) For reasons of safety pupils should not directly view the arcing process, unless through a welder's visor or equivalent. Nonetheless, this is an exciting, "exocharmic" demonstration owing to the reflection of bright light off the classroom walls and ceiling, accompanied by the distinct sound of the electrical arc.

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J. Chem. Educ. XXXX, XXX, XXX−XXX