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PREPARATION OF BUCKMINSTERFULLERENE, C_{60}

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ABSTRACT

In view of the great interest generated by the recent discovery of closed-cage molecules of carbon (fullerenes), we describe a procedure for preparing nearly pure buckminsterfullerene, C_{60}, which has considerable potential as a material.

MATERIALS INDEX: Carbon, fullerene, buckminsterfullerene.

Introduction

The initial identification of a stable, fascinating molecule consisting of sixty carbon atoms, C_{60}, owing its stability to the geodesic properties inherent in a truncated icosahedral hollow cage, was made by Kroto et al [1] in a cluster beam produced by the laser-ablation of graphite. By employing different experimental conditions, it was soon demonstrated that there is a whole range of related closed-cage molecules of carbon called fullerenes, of which C_{60}, buckminsterfullerene, is the dominant species. A technique for preparing macroscopic quantities of C_{60} and C_{70}, generally in admixture, recently described by Krätschmer et al [2] has generated intense activity in the investigation of the structure and properties of this new breed of carbon

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molecules. The technique involves the collection of the carbon species produced by vaporization in a contact arc between two graphite electrodes in an inert atmosphere. The quantities of C_{60} and the other carbon species obtained depend on the electrodes used, arcing conditions and other operating parameters [1-5]. All preparations of buckminsterfullerene, C_{60}, generally contain C_{70} in varying proportions as determined by NMR spectroscopy. Chemical purification of the product and the seperation of C_{60} from C_{70} have been achieved [6]. In order to investigate the various properties of C_{60}, we have designed a generator which

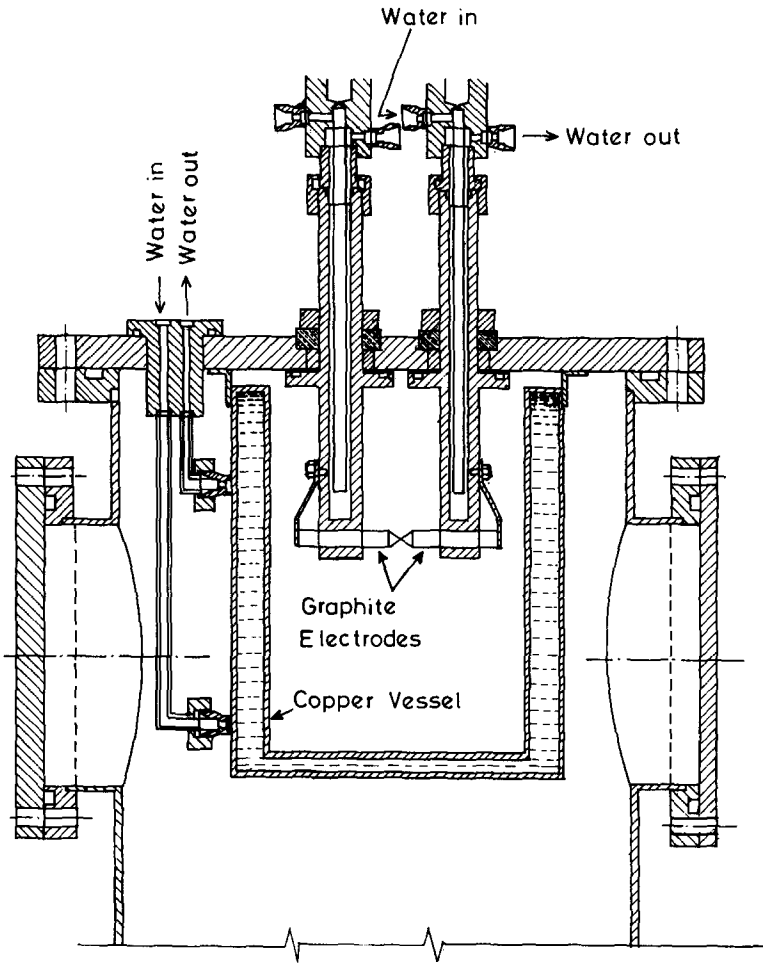

FIG. 1

Schematic diagram of the C_{60} generator

under certain operating conditions has yielded solely C_{60}. This finding has prompted us to describe the C_{60} generator fabricated by us along with the preparative conditions and characterization of the product, to enable other workers to readily generate this novel material.

EXPERIMENTAL

A schematic diagram of the C_{60} generator fabricated by us is shown in Fig. 1. Two water-cooled copper electrodes and a water-cooled double-walled copper vessel form the essential parts of the generator. The graphite electrodes and the copper vessel are housed in a 30" long stainless steel vacuum vessel of 8" diameter mounted on a 4" diffusion pump through a liquid nitrogen trap. Graphite rods of 5 mm diameter (specpure, Johnson Matthey Chemical Ltd., England) were used in the evaporation.

In order to facilitate the evaporation process, both the graphite electrodes were sharpened to a conical shape and held at loose contact with each other with the help of two tungsten springs. The system was pumped to $\sim 10^{-6}$ torr and helium let into the copper vessel through a liquid nitrogen trap. The pressure of helium was maintained around 180 torr with slow pumping and by controlling the leak rate, with the help of a variable leak valve. A current in the 100-180 amp range (5-8 volts AC, 60Hz) was passed through the electrodes to initiate the arc. A gray plume was observed under these conditions. After three hours of evaporation, about 500 mg of soot could be collected from the copper vessel and the electrode surfaces. Still larger quantities of soot could be obtained after prolonged evaporation. The soot was extracted with touluene and filtered. The resulting red solution is evaporated to yield a solid which was washed with diethyl ether to remove the hydrocarbons [2]. The solid left behind was characterised by mass spectrometry and ^{13}C NMR spectroscopy.

We have recently incorporated another set of electrodes to the system shown in Fig. 1 in a manner that enables us to move them from outside. This helps to sustain the arc for longer periods. With this design, larger quantities of C_{60} could be produced in a shorter period (50 mg in less than an hour).

RESULTS AND DISCUSSION

Since the arcing conditions are clearly crucial in determining the nature of carbon clusters in the soot, we decided to use relatively small currents to explore whether we could avoid many of the species other than C_{60}. To our surprise, the soot obtained with a current of 150 amps or less, after removal of the hydrocarbon impurities [2], yielded only C_{60} as evidenced from the mass spectrum (Fig. 2) and the ^{13}C NMR spectrum (Fig. 3). The ^{13}C NMR spectrum of C_{70} has signals at 130.9, 145.2, 147.3, 148.6 and 150.5 ppm with intensity ratios of 10/20/10/20/10. It is indeed remarkable that we only see C_{60} in

both the mass spectrum and the NMR spectrum. Furthermore, the ratio of C_{60}^{2+} to C_{60}^{+} in the mass spectrum is around 1.2 as expected. The procedure detailed above has enabled us to

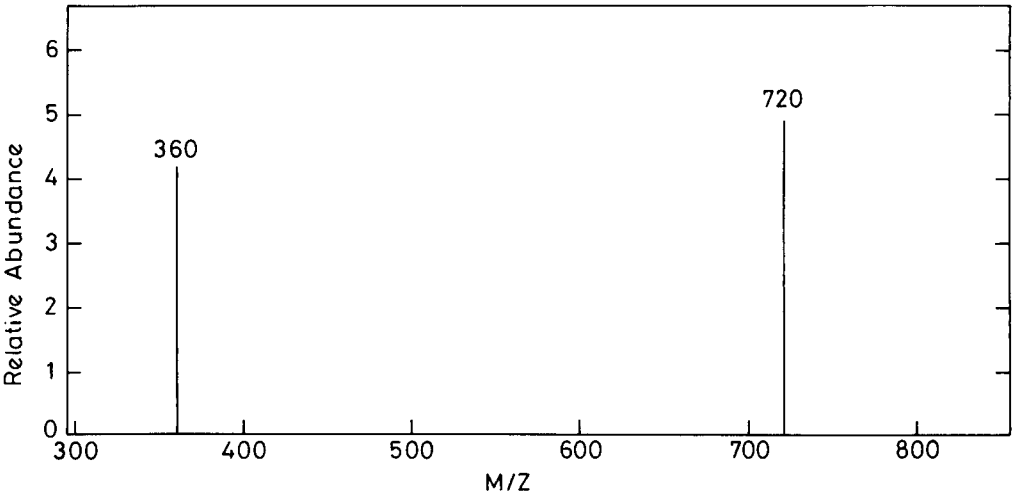

FIG. 2

Mass spectrum of the fullerene obtained after removing the hydrocarbons from the soot

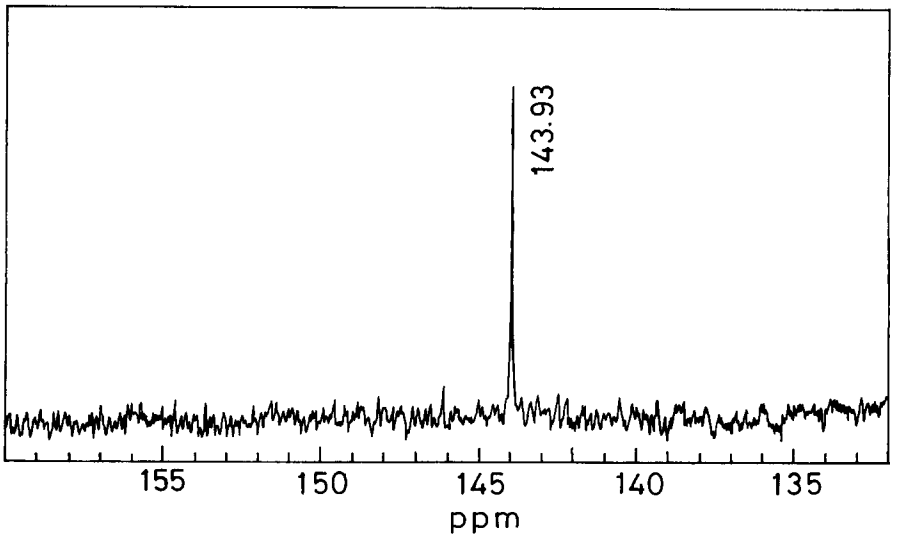

FIG. 3

^{13}C NMR spectrum of the fullerene obtained after removing the hydrocarbons from the soot

conveniently obtain nearly pure C_{60} in one step, without the need for chromatographic seperation from C_{70} etc. We have prepared several batches of C_{60} in this manner with yields of $\sim$ 10% with respect to the soot. The preferential formation of C_{60} in the present study suggests that the fragments containing the fused six-membered rings that initially come out of the graphite surface predominently contain the precursor species corresponding to C_{60}.

CONCLUSION

The present study demonstrates that it is indeed possible to obtain C_{60} essentially in pure form by arc evaporation of graphite.

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