

Article

## Extraction of Fullerene C<sub>60</sub> from the Surface of the Herbal Pipe after Combustion of Coal and Wood

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**Abstract:** From the soot of the chimney by completely burning wood together with brown coal, a small amount of graphite was extracted with fullerene C<sub>60</sub>. It was found that during the burning of coal and wood a large amount of heat is generated accompanying the evaporation of carbon carrying free electrons transforming the trigonal configuration with the further formation of mono-, di- and trimeric carbon radicals condensing to the formation of C<sub>60</sub>.

**Keywords:** fullerene, radical, carbon, crystal, spectrum, chromatography, graphite

### 1. Introduction

Fullerenes in a quantitative ratio in nature was found in the carboniferous mineral Shungite in the Zazhoginsky deposit of Karelia of the Russian Federation, the content of which was 0.01 mass%. However, preference is given to the electric arc method for obtaining and isolating the solid crystalline C<sub>60</sub> fullerene [1, 2]. In addition different ways of obtaining fullerenes are proposed, for example: from liquid and gaseous hydrocarbons, alcohols and other organic materials [2, 3, 4]. There are data on the production of carbon clusters by heating vapors of condensed polycyclic compounds containing five and six-membered rings [5].

A group of scientists from the United States believe that when the graphite evaporates with a laser, individual clusters merge into larger ones and go over into the shape of a single closed loop, reaching the number of carbon atoms in the ring to 40 which can form a sphere [6]. In the papers devoted to the theoretical analysis of the mechanism of fullerene growth, it is assumed that the process can go through the addition of particles from two [7, 8] or from three carbon atoms [9]. In work [10], it is believed that the formation of fullerenes occurs by "sticking together" excited clusters with the subsequent breakdown into two fragments of different masses. There is also evidence that small amounts of C<sub>60</sub> have been found in wood charcoal [11].

## 2. Materials and Methods

### 2.1. Extraction of Fullerenes C<sub>60</sub> from Soot of the Chimney

100g of soot black stovepipe in a three-liter conical flask and poured 0.5 liters of freshly distilled toluene. The contents are stirred with a mechanical stirrer at 50-60°C for 10 hours. The reaction mixture is then filtered in a Bünsen flask equipped with a Buchner funnel. The filtrate is evaporated to a minimum volume, the residue is concentrated in vacuo. 4.66 g of a dark mass are obtained which is treated with dry ether and decanted several times with methyl alcohol. The remaining black mass is transferred to a pear-shaped cone, 7 ml of acetone is added and the mixture is refluxed for an hour. The precipitate is filtered and weighed. 18.3 mg% of a fullerene C<sub>60</sub> black color are obtained which, when heated to 450°C, in a block for the melting point, a change in the melting point did not occur, upon further heating, decomposition began.

### 2.2. Instruments

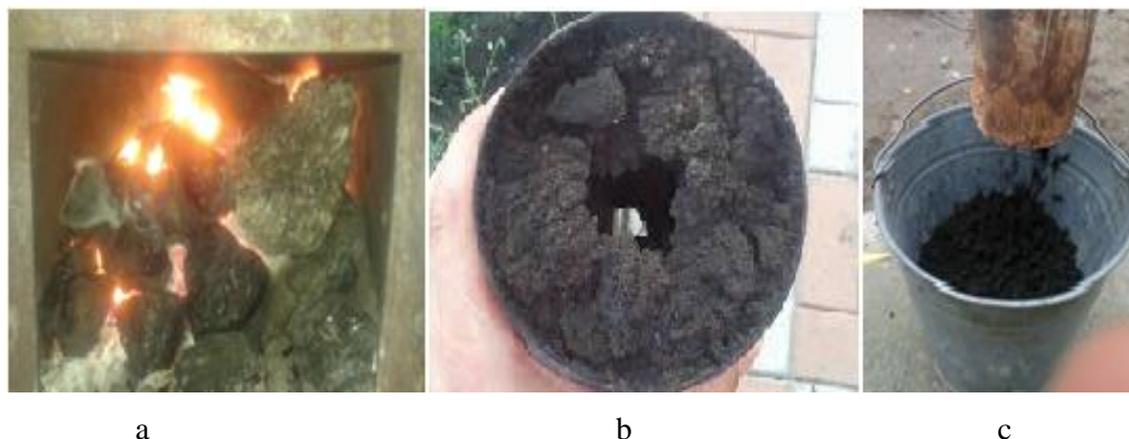
IR instruments: Shimadzu FTIR

Mass spectrometry: Bruker Ultra Flex 11

HPLC: ALTEX 340 equipped with a computer AXXLOM 710 HPLC, a spectrophotometer 757, a KIPP ZONEN recorder, an integrator of Shimadzu CRIB chromatinas. The column of the silicone is 5μ, 250x4mm (internal diameter), the space velocity is 4.99 ml / min, the pressure at the entry to the column is 120 atm, the detection by absorption at 290 nm of the eluent is toluene, the tape speed is 0.5 cm / min.

## 3. Results and Discussion

We tried to look for the formation of fullerenes in soot deposited on the inner walls of the iron pipe of the chimney of the cast iron stove inside which were burned brown coal instead of with wood (Fig. 1a, b, c).



**Figure 1:** a. Combustion of coal in the stove, b. Soot on the pipe walls, c. Finished Soot

Wood contributes to a better combustion of coal, due to the oxygen of the air accumulated during the drying of wood, taking the place of evaporated moisture outdoors. At the same time, the temperature inside the stove can rise to 1200-1300°C and part of the hydrocarbon products including graphite are converted to carbon vapor, converting carbon into a sublimated state and leaving the stove in the form of an excited sp-hybridized carbon three-dimensional radical, di- and trimeric, carbon radicals forming excited clusters and by “sticking” onto the hemispherical surface of graphite soot (crystallites), condensing, form a fullerene molecule C<sub>60</sub>. The process of formation of C<sub>60</sub> can be depicted according to the scheme 1.

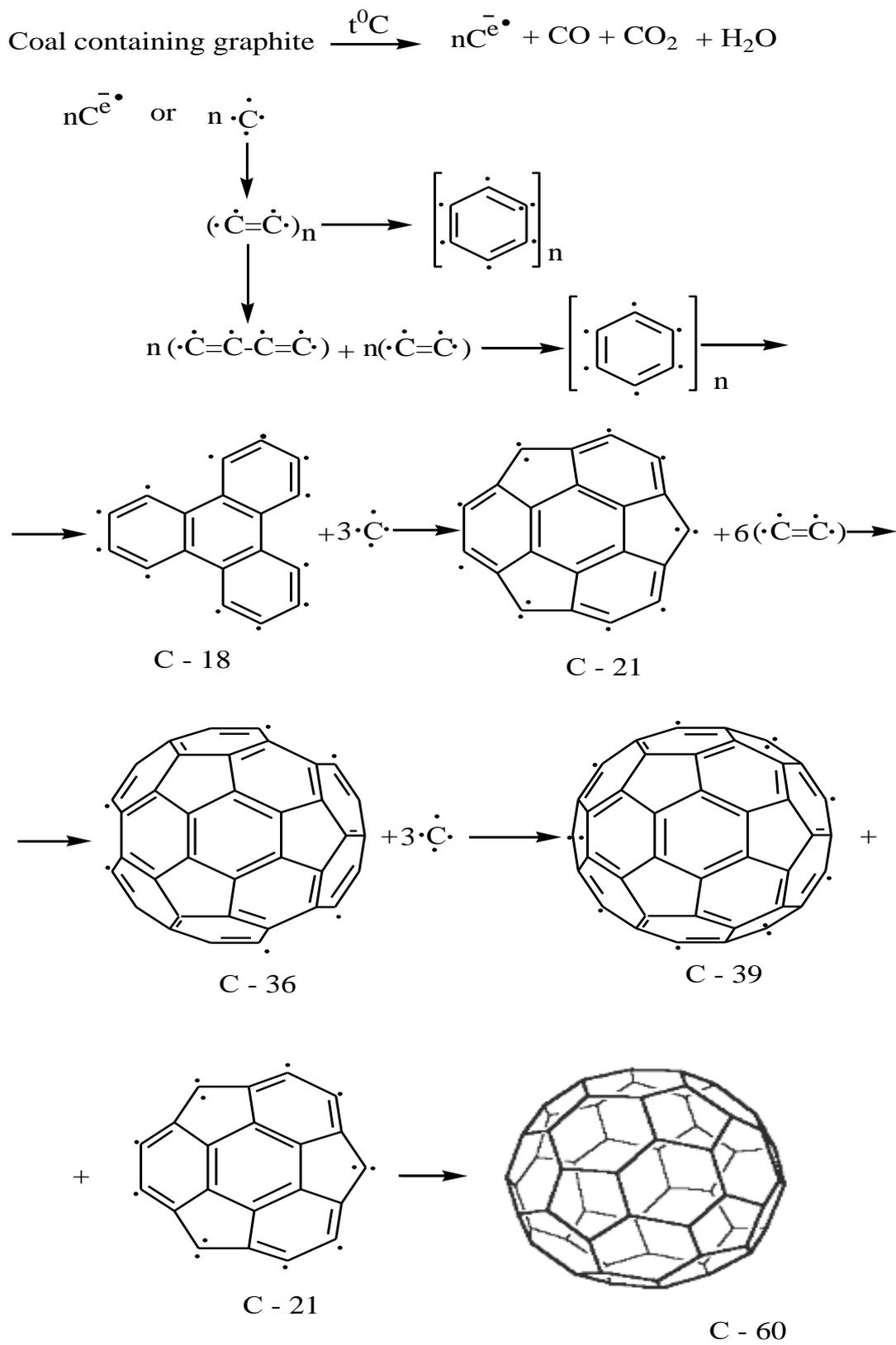
At the same time, a certain amount of fullerene-formed C<sub>60</sub> is combined with other organic products of soot, precipitating on the inner part of the metal pipe creating a black formation (Fig. 1b).

Part of fullerene C<sub>60</sub> under the pressure of smoke-forming gases, CO, CO<sub>2</sub> and a pair of water through the pipe, is discharged into the open atmosphere.

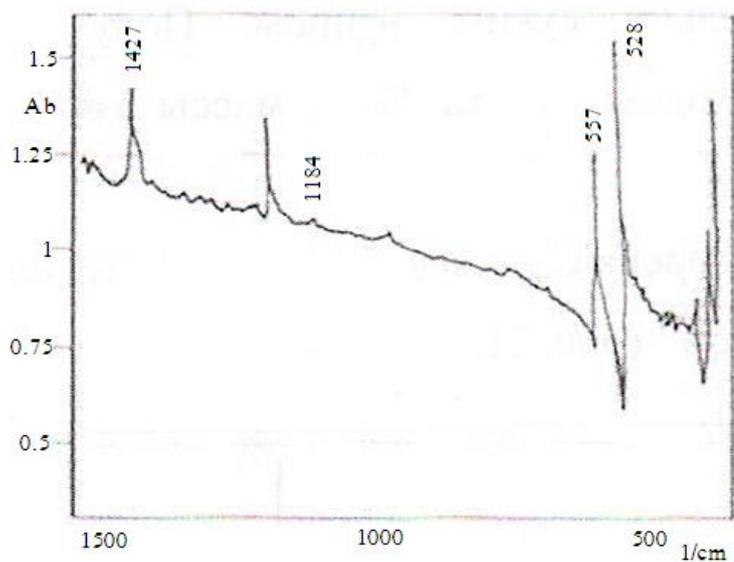
The soot formed from the tube is gently scraped and extracted with toluene. After evaporation of the toluene, the remaining dense mass was 3% of the extracted amount of carbon black, which was treated with ether, methanol and hexane to a minimum amount of the extract, which was transferred to a pear-shaped cone and refluxed in acetone for 2 hours. During this time, gradually more than half of the extract passes into acetone and at the bottom of the cone remains a black mass, which is treated additionally with dry ether. A black crystalline fullerene C<sub>60</sub> is obtained in a yield of 18.3 mg%.

In the IR-spectrum of the obtained C<sub>60</sub> fullerene there are distinguishable spectra at energies of 1586, 1427, 1184, 577 and 528 cm<sup>-1</sup> (Fig. 2).

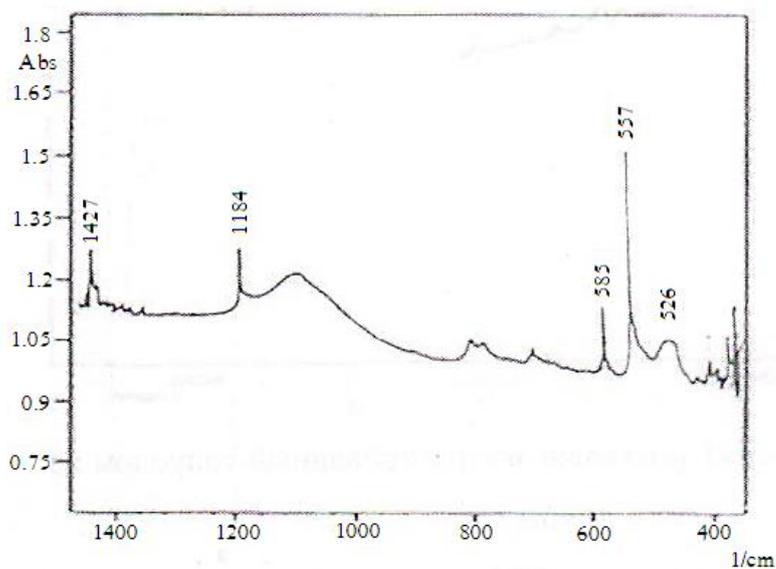
The same infrared spectra exist in the IR- absorption of the ordinary fullerene (standard) obtained in the electric arc synthesis of Fig. 3.



Scheme 1

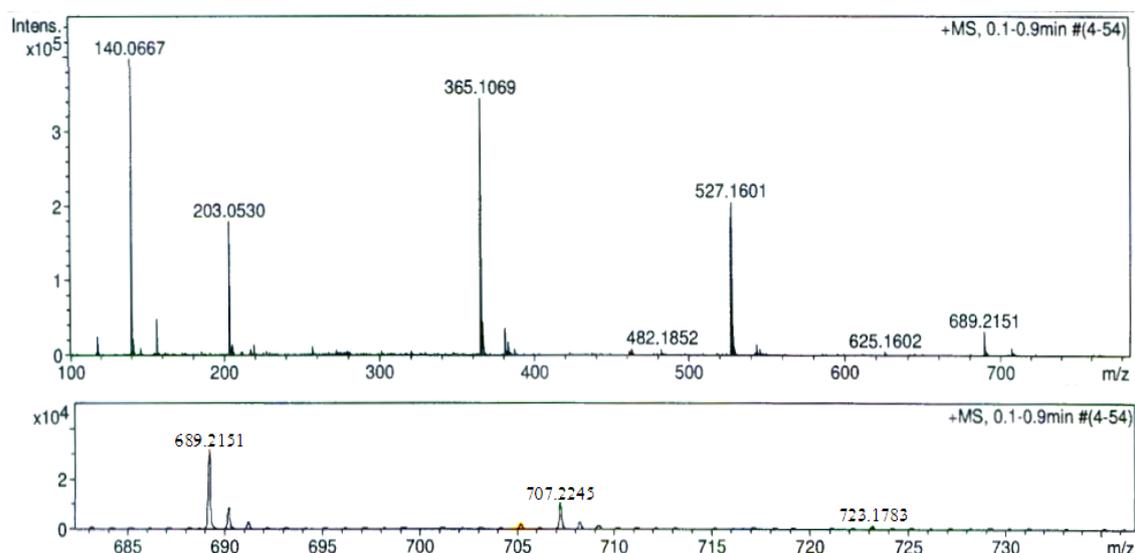


**Figure 2.** IR - spectrum of fullerene C<sub>60</sub> from soot of furnace pipe extracted with toluene



**Figure 3.** IR- spectrum of the reference standard

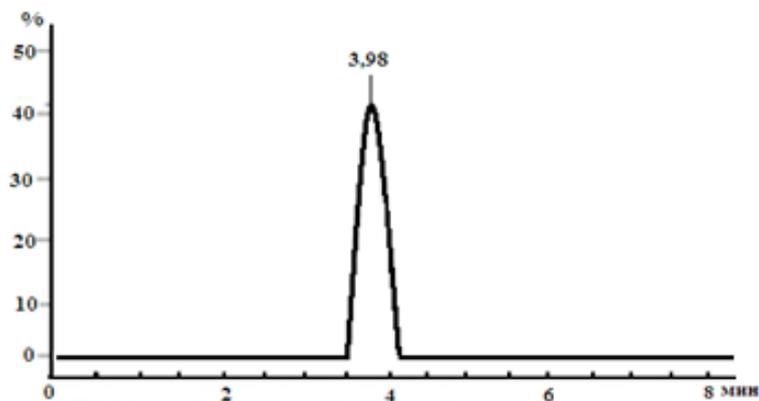
In the mass spectrum of the C<sub>60</sub> fullerene obtained (Fig. 4), the process of fragmentation and formation of a series of ions is observed, fragments with odd and even masses of 723, 707, 689, 625, 527, 482, 365, 203, 140. The spectrum with a mass of 723 corresponds to protonized C<sub>60</sub> 3H<sup>+</sup>.



**Figure 4.** Mass-spectrum of the obtained fullerene C<sub>60</sub> from soot of the chimney

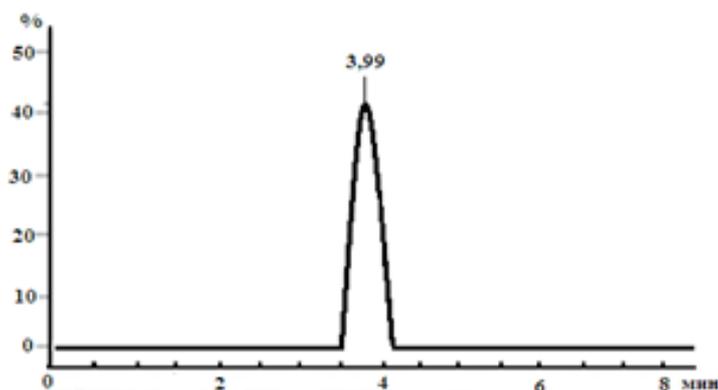
Identification of the obtained fullerene C<sub>60</sub> was carried out by the retention time of the matrix component by.

To clarify the identity, first enter the usual fullerene (standard) in toluene (Fig. 5) and elute with toluene. A single peak appears on the chromatogram, the retention time of which is 3.98 minutes.



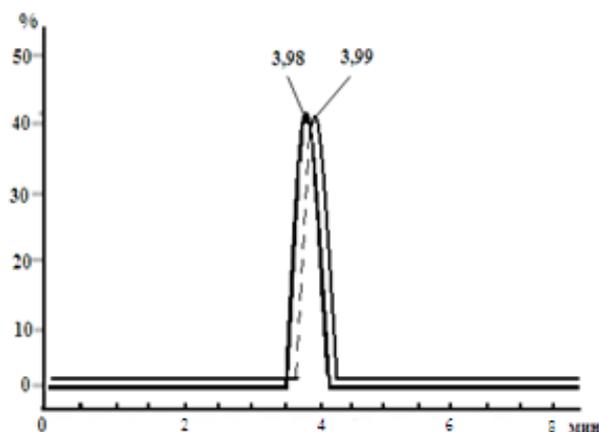
**Figure 5.** HPLC of an ordinary fullerene C<sub>60</sub> as a reference

Then, under the same condition, the extracted fullerene C<sub>60</sub> from the carbon black is passed through the column (Fig. 6), and one single peak will appear at 3.99 min.



**Figure 6.** HPLC of the isolated fullerene C<sub>60</sub> from soot of the chimney

A solution of the composite of these two C<sub>60</sub> fullerenes in toluene is then prepared and, if appropriate, is introduced into the column and eluted with toluene (Fig.7), two peaks are obtained with a retention time, which is almost the same 3.98 and 3.99 min. They were similar to previous analyzes alone.



**Figure 7.** Analytical HPLC of fullerene C<sub>60</sub> isolated from a carbon black extract (----) and a standard (fullerene C<sub>60</sub>) (-) on a column of 250x4 mm filled with a SNS 5 $\mu$ , a volumetric velocity of 4.99 ml / min, a tape speed of 0.5 cm / min, the pressure at the outlet of 120 atm., the detection by absorption at 290 nm, the mass of the sample to be separated is 0.5  $\mu$ g / l.

#### 4. Conclusion

From the soot of the chimney deposited on the inner walls of the chimney when burning brown coal together with wood in a cast-iron stove was extracted with toluene. The fullerene C<sub>60</sub> was obtained.

Instrumental analyses show fullerene C<sub>60</sub> obtained in this way has same properties as that of fullerene C<sub>60</sub> obtained by electric arc method.

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