What are Pure Carbon Fullerenes (C60)?

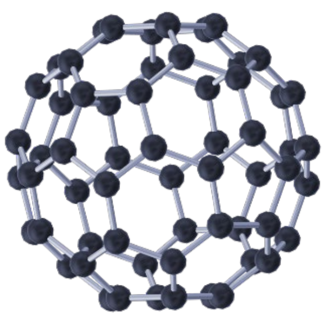
By Robert Wong and Chris Burres Co-Founders of SES Research, Inc.

The use of Carbon Fullerenes - Carbon 60 (C60), Carbon 70 (C70) and Carbon 84 (C84) - in various industrial and scientific applications has become increasingly common due to research and development over the past 30 years.

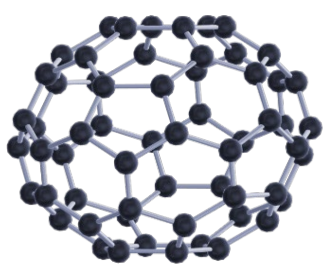
Various grades / purities can be used in most of the product's application to achieve the desired result. Those responsible for selecting the appropriate grade /purity of all ingredients will have to make the right choice to balance product performance vs the final cost of the finished product. This white paper should help in making the right decision.

Introduction

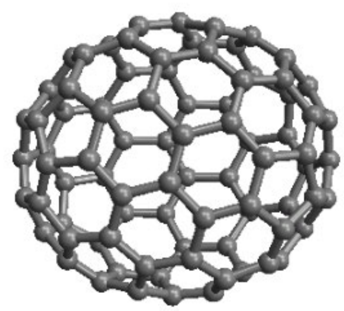
Carbon Fullerenes were discovered in 1985 at Rice University in Houston, Texas. This newly discovered pure form of carbon amazed scientists worldwide with its hollow spherical closed cage structure. In fact all 10 of the most cited scientific papers in 1992 where on the topic of Fullerenes (https://www.the-scientist.com/the-most-cited-papers-in-chemistry-1991-93-by-subject-percent-of-total-58840). The most abundant of the carbon molecules are C60, C70, and C84. All of which are spherical closed caged molecules with the most abundant being C60. All these forms of Carbon Fullerenes are easily dissolved in non-polar solvents.



C60



C70



C84

Production method

Carbon Fullerenes can be produced using a number of processes in the laboratory. On the industrial scale, two primary processes are employed: Combustion Synthesis and Carbon Arc method. Both methods produce a fine **Soot** powder produced contains the Fullerenes, called Fullerenic Soot. This Fullerenic Soot, which is very similar to carbon black in density and appearance, yields between 3-20 percent by weight of Fullerenes. The remaning 80-97 percent is amorphous carbon. The predominant Fullerene produced is, as the 3-20 by weight material, is C60, which is the most stable form of the Fullerenes.

The extraction of fullerenes from the amorphous carbon is done through chemical liquid solvent extraction. A property of Carbon Fullerenes is that they can be fully dissolved in organic solvents, unlike other forms of carbon, including amorphous carbon, that do not dissolve in solvents. In this process, the fullerenes, which are dissolved in an organic solvent, are separated from the amorphous carbon by filtering. The amorphous carbon, which does not dissolve in the organic solvent will remain on top of the filter while the Fullerenes dissolved in the organic solvent will go through the filter as a solution. This is similar to if you wanted to separate sugar and sand. You would first add water which would dissolve the sugar. You would then filter the mixture and the sand (amourphos carbon) would remain on top of the filter and the sugar would go through the filter. The final step is to boil off the solvent leaving the isolated Fullerenes called Fullerenic Extract.

Fullerene Purity

At this point the concentrated material, called Fullerenic Extract, is comprised of about 80% C60, 19% C60 and 1% higher Fullerenes (C76, C84, C96). This Fullereneic Extract needs further processing in order to isolate pure C60 from the other higher Fullerenes. This is accomplished by using a standard chemistry process called chromatography. The Fullerenic Extract is again dissolved in a polar solvent and the solution flows through a separation column of material. Each of the Fullerenes will flow through the column at different rates. By selecting the right fraction of solution as it exits the column you can collect highly concentrated C60. More about chromatography can be found here https://byjus.com/chemistry/differential-extraction-chromatography/

Pure fractions of C60 and C70 may not always be achieved during the first separation stage and the fraction separated which has higher concentrations of C60 or C70 will be repeated through the same process until desired purity of C60 is obtained. Purity is expressed by the concentration of C60 relative to C70, such as 99.5%, 99.75% or 99.99%. Assessment of the purity is performed using industry standard High Performance Liquid Chromatography (HPLC), which measures the purity of C60 vs C70. Once the desired purity is reached, the C60 or C70 in the liquid solvent is evaporated to leave a dried powder. However, some solvent may remain trapped in the dried powder.

Example: 1000 gram of 99.5% C60 with contain a minimum 995 grams of C60 and up to 5 grams of C70

VOC Removal (Volatile Organic Compounds)

Trace amounts of non-polar Solvent or VOC will remain in the dry powder. This is acceptable for some users of C60 or C70, since they will be using the material in industrial applications or redissolving the pure C60 in non-polar solvent to perform a reaction to produce derivative C60 compounds, such as [60] PCBM *([6,6]-Phenyl C61 butyric acid methyl ester)* which is useful in solar cell technology. *(https://www.sesres.com/pcbm/)*

If the material is going to be consumed by people or pets, a solvent free material is required and the C60 will undergo additional processing to remove all traces of the solvents. This final processing includes Vacuum drying, thermal drying, Wiped Film Evaporators (WFE), and a final material wash. In any of the above-mentioned processes, care must be taken to ensure that the C60 is not destroyed by oxidation. The removal of solvents is confirmed by doing a VOC test. This highly refined solvent free version of C60 for people or pets is called ESS60 (*https://www.sesres.com/fullerene/).*

Theory of Sublimation

There has been increasing mention of sublimated C60, or sublimed C60 in the literature from companies that sale industrial C60 for humans and pets. The process of sublimation is a standard process in modern chemistry and has been successfully used in industrial manufacturing, such as in the production of sunglasses, solar panels, and semiconductors.

The process of making a sublimed C60 always starts with a pure C60**,** and not from raw Fullerenic soot which was extract with solvents. Essentially, the method involves placing C60 sample in an apparatus, usually a quartz tube, where it is heated to the vaporization point of C60 around 1478 F°. It is important to do this in a 100% oxygen free environment or the C60 will oxidize and be rendered useless. The C60 vapor is then allowed to condense on a separate cooled section of the apparatus. It is well established that the sublimation process is wasteful, as much of the material escapes down the tube and out of the necessary vacuum pump. The wasteful and technical requirements for this process can nearly double or triple the cost of a sublimed product as compared to a material properly processed to remove all the solvents. The reality of the sublimation process is that it is exceptionally low-scale and costly.

As an additional note the health and wellness industry regularly uses solvents in the manufacture of supplements. The process of removing solvents is neither a complicated nor risky effort.

A Review of the Sublimated C60 Industry

At SES Research Inc., we felt it important to assess the viability of sublimated products on the market. If in fact sublimated products produced a higher quality product and if we could make that product cost effective it would be the best decision for our customers. At SES Resarch Inc. we use the laboratory Eurofins Scientific (https://www.eurofins.com/) as a third-party testing service to understand the remaining solvents, if any, in as produced C60, ESS60 and sublimed C60 purchased from all the North American suppliers. The test to assess this is GC/MS VOA Testing Method 8260D. Below are the results of recent tests for SES Research ESS60, SES Research standard Industrial grade C60, and other sublimed C60 purchased from various suppliers.

Summary

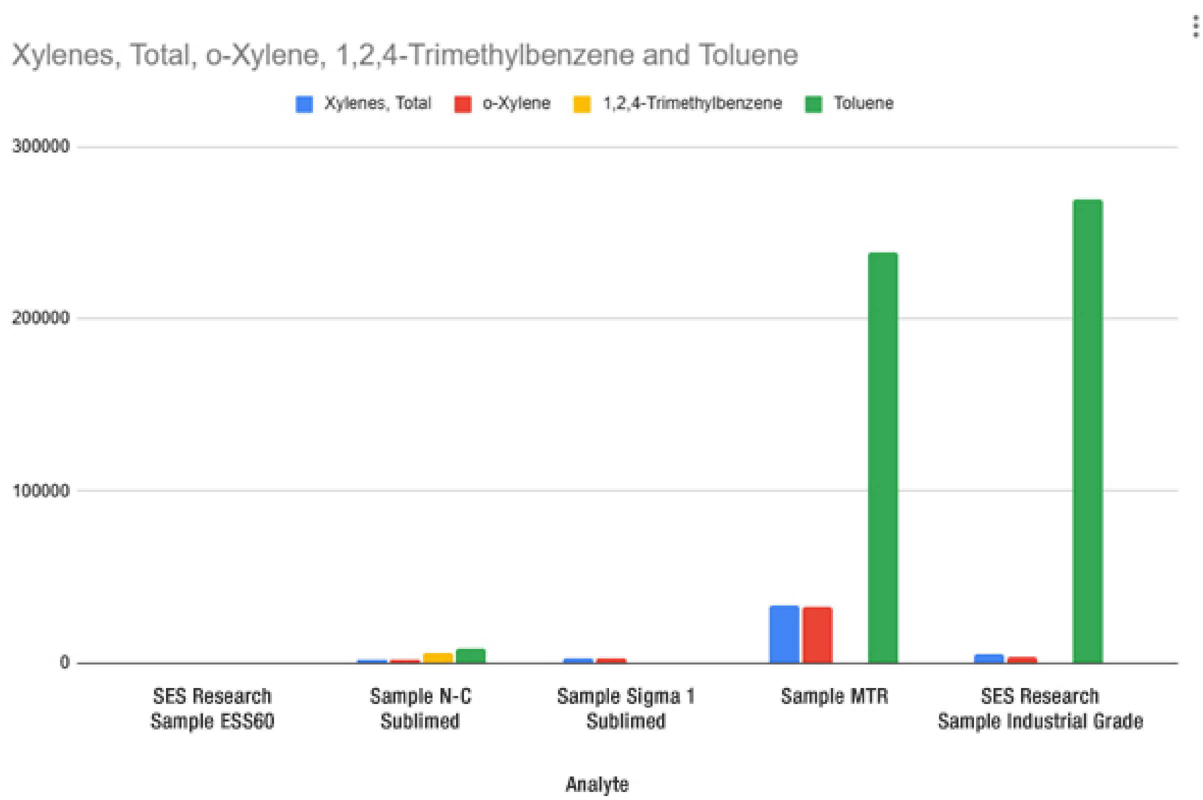


Fig 1

Detection Summary

Client: SES Research

Project/Site: Carbon 60 Dry Powder VOCs

Job ID: 860-XXX-XX

Client Sample ID: Sample ESS60 - Baked — Lab Sample ID: 860-51810-1

No Detections.

Client Sample ID: Sample N-C Sublimed — Lab Sample ID: 860-51810-2

Analyte	Result	Qualifier	RL	MDL	Unit	Dil Fac	D	Method	Prep Type
Xylenes, Total	1820	J	2780	1110	ug/Kg	50		8260D	Total/NA
o-Xylene	1820		1390	497	ug/Kg	50		8260D	Total/NA
1,2,4-Trimethylbenzene	5650	J	6940	2180	ug/Kg	50		8260D	Total/NA
Toluene	8740		6940	2170	ug/Kg	50		8260D	Total/NA

Client Sample ID: Sample Sigma 1 Sublimed — Lab Sample ID: 860-51810-3

Analyte	Result	Qualifier	RL	MDL	Unit	Dil Fac	D	Method	Prep Type
Xylenes, Total	2650		2500	1000	ug/Kg	50		8260D	Total/NA
o-Xylene	2650		1250	448	ug/Kg	50		8260D	Total/NA

Client Sample ID: Sample MTR — Lab Sample ID: 860-48533-1

Analyte	Result	Qualifier	RL	MDL	Unit	Dil Fac	D	Method	Prep Type
Xylenes, Total	33200		510	230	ug/Kg	50		8260D	Total/NA
m,p-Xylenes	852		510	204	ug/Kg	50		8260D	Total/NA
o-Xylene	32300		255	230	ug/Kg	50		8260D	Total/NA
Ethylbenzene	296		255	170	ug/Kg	50		8260D	Total/NA
Naphthalene	631	J	2550	510	ug/Kg	50		8260D	Total/NA
Toluene - DL	239000		51000	19500	ug/Kg	2000		8260D	Total/NA

Below is **SES Research, Industrial grade**

Client Sample ID: Sample 2 Non-Oven — Lab Sample ID: 860-48533-3

Analyte	Result	Qualifier	RL	MDL	Unit	Dil Fac	D	Method	Prep Type
Xylenes, Total	5350		490	221	ug/Kg	50		8260D	Total/NA
m,p-Xylenes	1510		490	196	ug/Kg	50		8260D	Total/NA
o-Xylene	3840		245	221	ug/Kg	50		8260D	Total/NA
Ethylbenzene	677		245	163	ug/Kg	50		8260D	Total/NA
Naphthalene	647	J	2450	490	ug/Kg	50		8260D	Total/NA
Toluene - DL	270000		49000	18700	ug/Kg	2000		8260D	Total/NA

Fig 2

Definitions/Glossary

Client: SES Research
Project/Site: Carbon 60 Dry Powder VOCs

Job ID: 860-48533-1

Qualifiers

GC/MS VOA

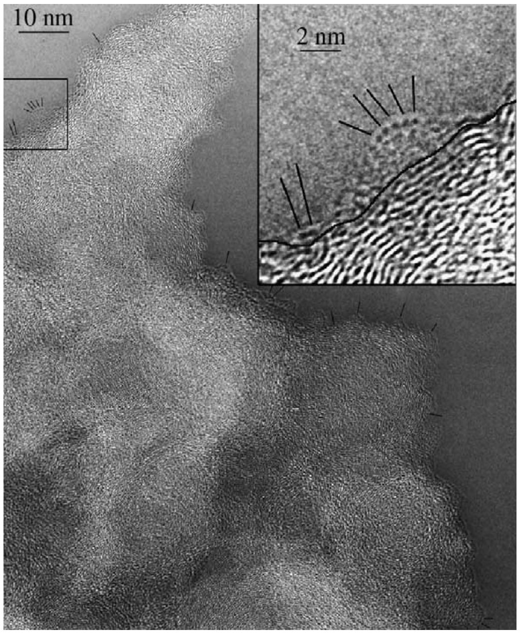
Qualifier	Qualifier Description
*+	LCS and/or LCSD is outside acceptance limits, high biased.
*1	LCS/LCSD RPD exceeds control limits.
J	Result is less than the RL but greater than or equal to the MDL and the concentration is an approximate value.
U	Indicates the analyte was analyzed for but not detected.

Glossary

Abbreviation	These commonly used abbreviations may or may not be present in this report.
¤	Listed under the "D" column to designate that the result is reported on a dry weight basis
%R	Percent Recovery
CFL	Contains Free Liquid
CFU	Colony Forming Unit
CNF	Contains No Free Liquid
DER	Duplicate Error Ratio (normalized absolute difference)
Dil Fac	Dilution Factor
DL	Detection Limit (DoD/DOE)
DL, RA, RE, IN	Indicates a Dilution, Re-analysis, Re-extraction, or additional Initial metals/anion analysis of the sample
DLC	Decision Level Concentration (Radiochemistry)
EDL	Estimated Detection Limit (Dioxin)
LOD	Limit of Detection (DoD/DOE)
LOQ	Limit of Quantitation (DoD/DOE)
MCL	EPA recommended "Maximum Contaminant Level"
MDA	Minimum Detectable Activity (Radiochemistry)
MDC	Minimum Detectable Concentration (Radiochemistry)
MDL	Method Detection Limit
ML	Minimum Level (Dioxin)
MPN	Most Probable Number
MQL	Method Quantitation Limit
NC	Not Calculated
ND	Not Detected at the reporting limit (or MDL or EDL if shown)
NEG	Negative / Absent
POS	Positive / Present
PQL	Practical Quantitation Limit
PRES	Presumptive
QC	Quality Control
RER	Relative Error Ratio (Radiochemistry)
RL	Reporting Limit or Requested Limit (Radiochemistry)
RPD	Relative Percent Difference, a measure of the relative difference between two points
TEF	Toxicity Equivalent Factor (Dioxin)
TEQ	Toxicity Equivalent Quotient (Dioxin)
TNTC	Too Numerous To Count

High Resolution Transmission Electron Microscopy (HRTEM) Image of C60

Image of Carbon 60 under High Resolution TEM with accuracy of ±0.05nm magnification. C60 on carbon black and Pure single C60 Molecules



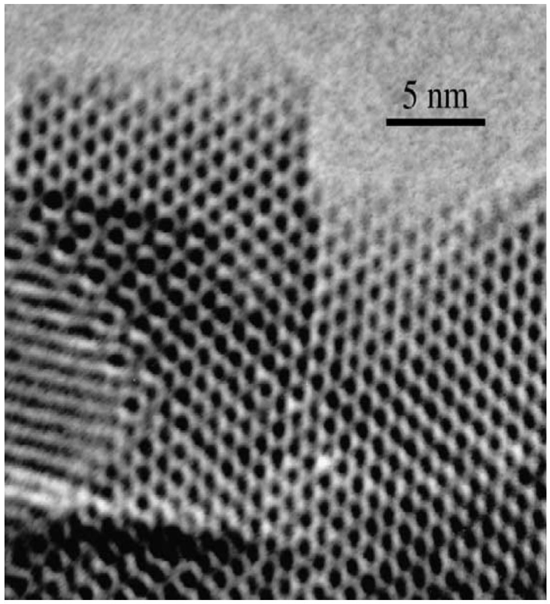


Image by Drs. Paula Jardim, Paulo Ferreira and K. Das Chowdhury at MIT

Carbon 42 (2004) 1907–1915

Closing Summary

Carbon fullerenes, particularly C60, are a remarkable class of molecules with a broad range of industrial and scientific applications. Since their discovery in 1985, advances in production, purification, and quality assurance have made these molecules integral to fields like energy, electronics, and health supplements. SES Research has demonstrated the importance of rigorous processes to ensure high-purity and solvent-free products, particularly for applications involving human and pet consumption, as seen in the development of ESS60.

While alternative methods like sublimation are promoted in some markets, care must be taken when to verify if it really sublimated since the process is costly, wasteful, and not scalable beyond the laboratory application and solvent still remains (see Fig 1 and Fig 2). SES Research's commitment to using advanced testing and refining protocols underscores its dedication to delivering superior products while balancing cost-effectiveness. Remember the age old adage , you pay for what you get and it sounds too good to be true it must not be true.