

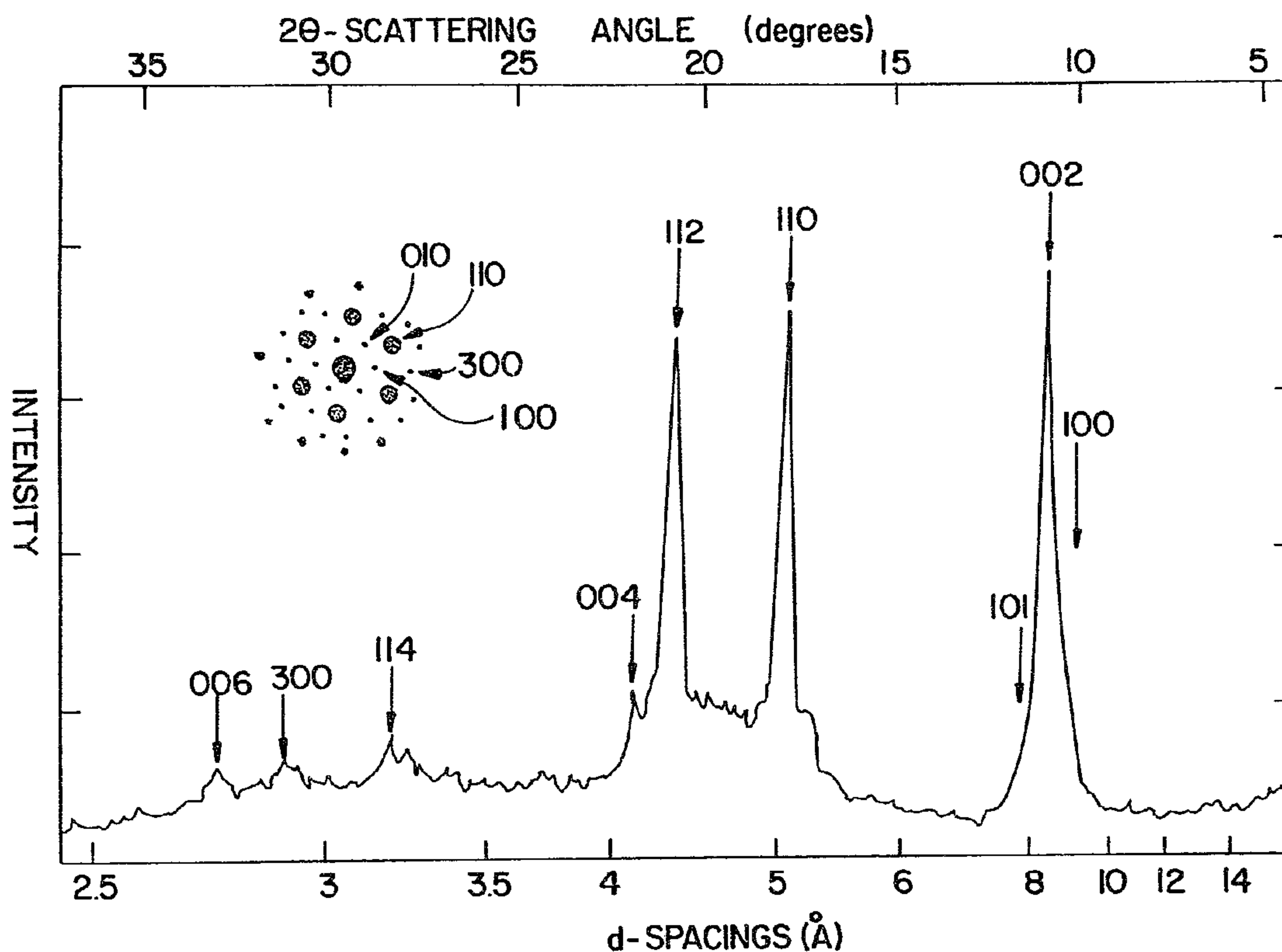


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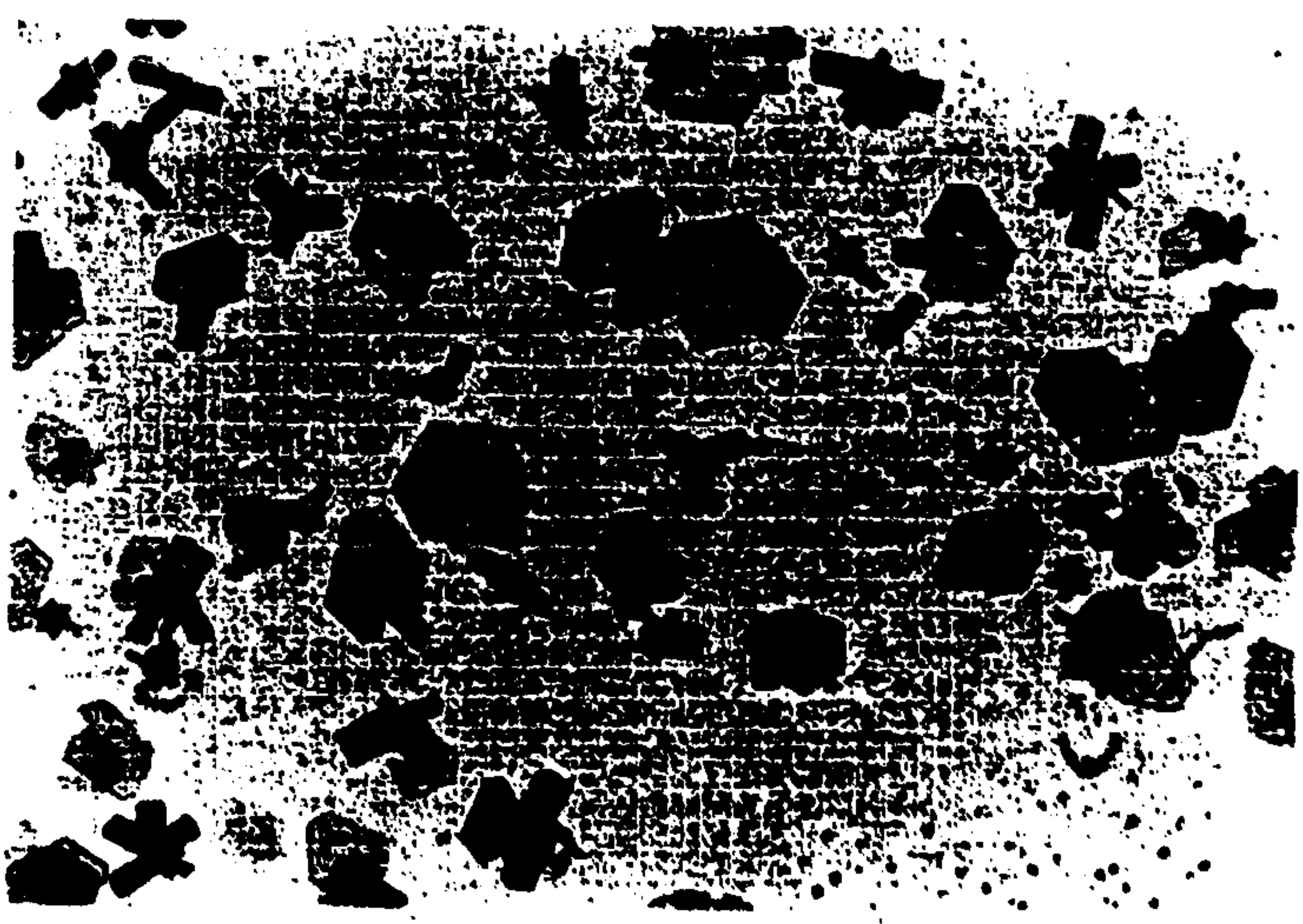


(57) Abrégé/Abstract:

C₆₀ and C₇₀ carbon atom compounds are prepared by evaporating graphite in an inert quenching gas. The vapor of carbon is collected and is selectively extracted with an organic non-polar solvent.



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(54) Title: NEW FORM OF CARBON  (57) Abstract C ₆₀ and C ₇₀ carbon atom compounds are prepared by evaporating graphite in an inert quenching gas. The vapor of carbon is collected and is selectively extracted with an organic non-polar solvent.		

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NEW FORM OF CARBON

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This invention relates to new forms of carbon as well as methods for the production and recovery thereof from carbon sources.

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In 1985, Kroto et al. postulated the existence of a highly stable molecule composed of 60 carbon atoms based solely on mass spectroscopic analysis of vaporized graphite (H.W. Kroto, et al., Nature, Vol. 318, 162, 14 November 1985). More specifically, all that was observed was a peak in the mass spectra of said carbon vapor. However, Kroto et al. did not isolate any of said compound.

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A model for this compound was proposed in which 60 carbon atoms are placed at the vertices of a truncated icosahedron forming a perfect "soccerball" structure. Subsequent thereto, many publications have strengthened the evidence for the existence of this molecule. The 60 carbon atom compound (hereinafter C_{60}) was presumably produced in situ for the spectroscopic determination reported in these publications. Yet, to date, no one has been successful in verifying the existence of this molecule since no one has been successful in isolating the molecule in measurable amounts. Thus, no processes for producing recoverable amounts of this new compound have been described to the present time.

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In the aforesaid publication by Kroto, et al., the authors proposed many uses for the new substance, C_{60} if it could be produced in quantity such as C_{60} transition metal compounds, e.g., $C_{60}Fe$; or halogenated species like $C_{60}F_{60}$ which might be a super lubricant; molecules including oxygen and lanthanum in the C_{60} interior; C_{60} would provide a topologically novel

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aromatic nucleus for new branches of organic and inorganic chemistry; and C_{60} being especially stable and symmetrical provides possible catalyst and/or intermediate in modelling prebiotic chemistry.

Another form of carbon containing 70 carbon atoms (C_{70}) has also been postulated (Kroto, Chemistry in Britain, 40-45 (1990), Kroto, Science, 1139-1145 (1988)). Like the C_{60} to date, no one has been successful in verifying the existence of the C_{70} . Heretofore, no one has been successful in obtaining the molecule in any appreciable amounts.

10 A process has now been developed for the production of recoverable amounts of C_{60} and C_{70} . The present invention relates to a method of producing C_{60} and C_{70} compounds which comprises evaporating graphite in an atmosphere of an inert quenching gas at effective pressures in an evacuated reactor, collecting the quenched carbon product produced therefrom and contacting the quenched carbon product with an extracting non-polar organic solvent under effective conditions to separate the C_{60} and C_{70} compounds therefrom. The present new process is accomplished by evaporating carbon rods in an atmosphere of an inert quenching gas maintained at reduced pressure in a reactor therefor. This process produces a sooty carbon product which is graphitic
20 carbon including a few percent of C_{60} and low levels of C_{70} which are recoverable from the product. However, an increase in the fraction of C_{70} molecules can be produced if the pressure is raised to greater than atmospheric pressures.

The recovery process is preferably accomplished by selective extraction of C_{60} and C_{70} with non-polar organic solvents from the sooty graphitic carbon. Such products can be characterized as being amorphous or crystalline particulate matter comprised of C_{60} or C_{70} .

In accordance with an embodiment of the present invention there is provided a method of producing C_{60} and C_{70} compounds which comprises evaporating graphite in an atmosphere of an inert quenching gas at pressures effective to quench the vaporized carbon in a reactor that had previously been
30 evacuated, collecting the quenched carbon product produced therefrom and

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contacting the quenched carbon product with an extracting non-polar organic, solvent under effective conditions to separate the C_{60} and C_{70} compounds therefrom.

In accordance with another embodiment of the present invention there is provided a carbon product, the mass spectrum of which shows a strong peak at mass 720 amu, the infrared bonds to which have four intense lines at 1424, 1183, 577 and 528 cm^{-1} , absorption peaks in the UV at 264 and 339 nm, soluble in non-polar organic solvents and sublimates at a temperature of from about 300° to 400°C, present in amounts sufficient to be discerned as a solid.

Another embodiment of the present invention provides a formed or molded product comprising C_{60} and C_{70} .

Yet another embodiment of the present invention provides a free flowing particulate comprised of C_{60} and C_{70} .

A substantially pure C_{60} and C_{70} is provided in another embodiment of the present invention.

Still another embodiment of the present invention provides an isolated carbon product, the mass spectrum of which shows a molecular ion at 840 amu, a broad peak in the ultraviolet at 216 nm, and soluble in non-polar organic solvents.

Other embodiments of the present invention provide isolated C_{60} or isolated C_{70} as well as the vapor of C_{60} and C_{70} .

In accordance with yet another embodiment of the present invention there is provided a method of extracting C_{60} and C_{70} from a carbon source containing same which comprises contacting the carbon source with a non-polar organic solvent.

A still further embodiment of the present invention provides a process for preparing C_{60} comprising: (a) vaporizing a carbon source in the presence of an inert quenching gas under conditions effective to form a sooty carbon product comprising C_{60} molecules, the C_{60} molecules being present in the sooty carbon product in amounts capable of extracting and recovering predominantly therefrom the C_{60} as a visible solid form; and (b) recovering C_{60} from the sooty carbon product.

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An embodiment of the present invention provides a process for preparing C_{60} comprising: (a) vaporizing a carbon source in the presence of an inert quenching gas under conditions effective to form a sooty carbon product comprising C_{60} molecules, said C_{60} molecules being present in said sooty carbon product in amounts sufficient to be capable of providing a visibly colored solution when extracted with sufficient amounts of benzene; and (b) recovering C_{60} from said sooty carbon product.

10 Another embodiment of the present invention provides a process for preparing C_{60} comprising: (a) vaporizing a carbon source in the presence of an inert quenching gas under conditions effective to form a sooty carbon product comprising C_{60} molecules, the C_{60} molecules being present in said sooty carbon product in macroscopic amounts; and (b) recovering macroscopic amounts of the C_{60} from said sooty carbon product.

In accordance with one embodiment of the present invention there is provided a process for preparing fullerenes comprising: (a) vaporizing a carbon source in the presence of an inert quenching gas under conditions effective to form a sooty carbon product comprising fullerene molecules, the fullerene molecules being present in the sooty carbon product in macroscopic amounts; and (b) recovering macroscopic amounts of fullerene from the sooty carbon product.

20 In accordance with another embodiment of the present invention there is provided a process for preparing a carbon allotrope comprising caged molecules consisting solely of carbon atoms which are soluble in non-polar organic solvents, the process comprising: (a) vaporizing a carbon source in the presence of an inert gas to produce a carbon vapor; (b) quenching the vapor of carbon in the inert gas under conditions effective to nucleate and condense the carbon allotrope, the allotrope being present in the sooty carbon product in amounts sufficient to be capable of extracting and recovering therefrom the allotrope in solid form; and (c) recovering the carbon allotrope from the sooty carbon product.

30 In accordance with a further embodiment of the present invention there is

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provided a method of extracting C_{60} from a carbon source containing same which comprises contacting the carbon source with a non-polar organic solvent.

In accordance with a still further embodiment of the present invention there is provided a carbon product comprising C_{60} , C_{70} or a mixture of C_{60} and C_{70} .

Various other embodiments of the present invention provide for solid C_{60} , solid C_{70} , crystalline C_{60} , crystalline C_{70} , substantially pure solid C_{60} , substantially pure solid C_{70} , substantially pure crystalline C_{60} , substantially pure crystalline C_{70} .

10 Also provided by embodiments of the present invention are C_{60} in amounts sufficient to isolate as a visible solid, C_{70} in amounts sufficient to isolate as a visible solid, macroscopic amounts of C_{60} , macroscopic amounts of C_{70} , substantially pure C_{60} in amounts sufficient to isolate as a visible solid, substantially pure C_{70} in amounts sufficient to isolate as a visible solid.

A still further embodiment of the present invention provides a solution of a carbon allotrope selected from the group consisting of C_{60} and C_{70} being dissolved in an organic non-polar solvent, the allotrope being present therein in amounts sufficient to be capable of recovering it as a solid when the solvent is evaporated.

20 In other preferred embodiments, there are provided a fullerene present in an amount sufficient to isolate as a solid and a macroscopic amount of fullerene.

In accordance with a further embodiment of the present invention there is provided a solid that consists essentially of a fullerene, the fullerene being isolated from a sooty carbon product formed from vaporizing a carbon source in the presence of an inert quenching gas, wherein the fullerene is recovered therefrom in macroscopic amounts.

30 In accordance with a still further embodiment of the present invention there is provided a cage carbon molecule consisting solely of carbon atoms that is isolated from a sooty carbon product formed from the vaporization of a carbon source in the presence of an inert quenching gas, the cage carbon molecule being recovered therefrom in amounts sufficient to be isolated as a visible solid.

In another embodiment of the present invention there is provided a cage

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carbon molecule product consisting solely of carbon atoms which is prepared by (a) vaporizing a carbon source in the presence of an inert quenching gas under conditions effective to produce a sooty carbon containing the caged carbon molecule product the inert gas providing a non-reactive atmosphere; (b) collecting the sooty carbon product; (c) separating the product from the sooty carbon product, the separated product being free of any sooty carbon product and consisting solely of carbon atoms, the product being present in amounts sufficient for the product to be perceived as a visible solid in solid form.

10 In accordance with a further embodiment of the present invention, there is provided fullerenes in an amount at least as large as that amount which is sufficient to see under an optical microscope.

 In accordance with a further embodiment of the present invention, there is provided a process for preparing cage molecules consisting solely of carbon atoms comprising vaporizing elemental carbon in the presence of an inert quenching gas under conditions effective to form a sooty carbon product containing the cage molecules of carbon, the cage molecules being present in the sooty carbon product in sufficient amounts to be capable of extracting and recovering therefrom the cage molecules as a solid; and extracting the cage
20 molecule of carbon from the sooty carbon product.

 In accordance with a further embodiment of the present invention, there is provided a process for preparing cage molecules consisting solely of carbon atoms comprising vaporizing elemental carbon in the presence of an inert quenching gas under conditions effective to provide a sooty carbon product comprising the cage molecules of carbon, the cage molecules being present in the sooty carbon product in amounts sufficient to be capable of extracting and recovering therefrom the cage molecules in solid form; depositing the sooty carbon product on a collecting surface; removing the sooty product from the collecting surface; and extracting a product which comprises cage molecules
30 consisting solely of carbon atoms from the sooty carbon product.

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In accordance with a further embodiment of the present invention, there is provided macroscopic amounts of an amorphous or crystalline particulate matter comprised of C_{60} or C_{70} .

In accordance with a further embodiment of the present invention, there is provided a carbon product comprising a macroscopic amount of a mixture of C_{60} or C_{70} .

In accordance with a further embodiment of the present invention, there is provided isolated C_{60} or isolated C_{70} in amounts sufficient to be visible.

10 In accordance with a further embodiment of the present invention, there is provided a process for preparing C_{60} comprising vaporizing a carbon source in the presence of an inert quenching gas under conditions effective to form a sooty carbon product comprising C_{60} molecules, said C_{60} molecules being present in said sooty carbon product in amounts capable of extracting and recovering predominantly therefrom said C_{60} as a visible solid form; and recovering C_{60} from said sooty carbon product.

20 In accordance with a further embodiment of the present invention, there is provided a process for preparing C_{60} in macroscopic amounts comprising vaporizing a carbon source in the presence of an inert quenching gas under conditions effective to produce a carbon product comprising C_{60} molecules, said C_{60} molecules being present in amounts capable of extracting and recovering therefrom said C_{60} in macroscopic amounts; depositing the sooty carbon product on a collecting surface; removing the sooty carbon product from the collecting surface; and recovering a product comprising C_{60} in macroscopic amounts from said sooty carbon product.

30 In accordance with a further embodiment of the present invention, there is provided a (a) vaporizing a carbon source in the presence of an inert quenching gas under conditions effective for form a sooty carbon product comprising C_{60} molecules, said C_{60} molecules being present in the sooty carbon product in amounts sufficient to be capable of providing a visibly colored solution when extracted with sufficient amounts of benzene; and (b) recovering C_{60} from the sooty carbon product.

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In accordance with a further embodiment of the present invention, there is provided a process for preparing a carbon allotrope which is C₆₀ or C₇₀ or a mixture thereof comprising molecules consisting solely of carbon atoms, which are soluble in non-polar organic solvents, the process comprising: (a) vaporizing a carbon source in the presence of an inert gas to produce a carbon vapor; (b) quenching the vapor of carbon in the inert gas under conditions effective to nucleate and condense the carbon allotrope, the allotrope being present in the sooty carbon product in amounts sufficient to be capable of extracting and recovering therefrom the allotrope in solid form; and (c) recovering the carbon
10 allotrope of C₆₀ or C₇₀ or a mixture thereof from the sooty carbon product.

In accordance with a further embodiment of the present invention, there is provided a carbon product comprising C₆₀ in macroscopic amounts, C₇₀ in macroscopic amounts as a mixture of C₆₀ and C₇₀ in macroscopic amounts.

In accordance with another aspect of the present invention, there is provided solid C₆₀ and solid C₇₀.

In accordance with a further embodiment of the present invention, there is provided crystalline C₆₀ and crystalline C₇₀.

In accordance with another aspect of the present invention, there is provided substantially pure solid C₆₀ and solid C₇₀.

20 In accordance with a further embodiment of the present invention, there is provided substantially pure crystalline C₆₀.

In accordance with a further embodiment of the present invention, there is provided substantially pure crystalline C₇₀.

In accordance with a further embodiment of the present invention, there is provided C₆₀ in amounts sufficient to isolate as a visible solid. Further, there is provided C₇₀ in amounts sufficient to isolate as a visible solid.

In accordance with a further aspect of the present invention, there is provided macroscopic amounts of C₆₀.

30 In accordance with a further aspect of the present invention, there is provided macroscopic amounts of C₇₀.

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In accordance with a further embodiment of the present invention, there is provided substantially pure C_{60} in amounts sufficient to isolate as a visible solid.

In accordance with a further embodiment of the present invention, there is provided substantially pure C_{70} in amounts sufficient to isolate as a visible solid.

In accordance with a further aspect of the present invention, there is provided a solution of a carbon allotrope selected from the group consisting of C_{60} and C_{70} being dissolved in an organic non-polar solvent, the allotrope being present therein in amounts sufficient to be capable of recovering it as a solid when the solvent is evaporated.

10 In accordance with another embodiment of the present invention, there is provided C_{60} in an amount at least as large as that amount which is sufficient to see under an optical microscope.

In accordance with a further embodiment of the present invention, there is provided C_{70} in an amount at least as large as that amount which is sufficient to see under an optical microscope.

In accordance with a further aspect of the present invention, there is provided a product containing C_{70} and substantially pure solid C_{60} and a product containing substantially pure solid C_{60} and substantially pure solid C_{70} .

20 In accordance with a further embodiment of the present invention, there is provided a product containing C_{60} and substantially pure solid C_{70} .

In accordance with a further embodiment of the present invention, there is provided macroscopic amounts of substantially pure C_{60} and macroscopic amounts of substantially pure C_{70} .

30 In accordance with another embodiment of the present invention, there is provided a carbon product prepared by (a) vaporizing a carbon source in the presence of an inert quenching gas under conditions effective to produce a sooty carbon product comprising C_{60} , the C_{60} being present in the sooty carbon product in sufficient amounts to allow the C_{60} to be separated and isolated from the sooty carbon product as a visible solid in solid form; (b) collecting the sooty, carbon product produced therefrom; (c) separating the C_{60} from the sooty carbon product, the product being comprised of C_{60} present in amounts sufficient for the

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C₆₀ therein to be perceived as a visible solid in solid form, the C₆₀ being free of sooty carbon product.

In accordance with another aspect of the present invention, there is provided a solid carbon product comprising C₆₀ prepared by the process comprising: (a) vaporizing a carbon source in the presence of an inert quenching gas under conditions effective to provide a sooty carbon product comprising C₆₀ molecules; (b) depositing the sooty carbon product on a collecting substrate; (c) removing the sooty carbon product from the collecting substrate; (d) separating C₆₀ from the sooty carbon product, the C₆₀ being
10 produced in amounts sufficient to be isolated in solid form, the C₆₀ being free from the sooty carbon product.

In accordance with a further aspect of the present invention, there is provided a product comprised of C₆₀ prepared by the process comprising: (a) vaporizing a carbon source in the presence of an inert quenching gas under conditions effective to produce a sooty carbon product comprising C₆₀ molecules; (b) depositing the sooty carbon product into a collecting substrate; (c) removing the sooty carbon product from the collecting substrate; (d) contacting the sooty carbon product with a non-polar organic solvent effective to dissolve the C₆₀, the C₆₀ being present in the non-polar organic solvent amounts
20 sufficient to provide a visibly colored solution and separating the solution containing the dissolved C₆₀ from the sooty carbon product.

In accordance with a further aspect of the present invention, there is provided a formed or molded product comprising substantially pure solid C₆₀ or substantially pure C₇₀.

In accordance with a further embodiment of the present invention, there is provided a free flowing particulate comprising substantially pure solid C₆₀ or substantially pure solid C₇₀.

In accordance with another aspect of the present invention, there is provided a process for preparing C₆₀ comprising: (a) vaporizing elemental
30 carbon in the presence of an inert quenching gas under conditions effective to form a sooty carbon product comprising C₆₀ molecules, the C₆₀ molecules being

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present in the sooty carbon product in amounts capable of extracting therefrom the C_{60} in macroscopic amounts and in solid form; and (b) extracting C_{60} in macroscopic amounts.

In accordance with another embodiment of the present invention there is provided a process for preparing C_{60} comprising: (a) vaporizing elemental carbon in the presence of an inert quenching gas under conditions effective to form a sooty carbon product comprising C_{60} molecules, the C_{60} molecules being present in the sooty carbon product in amounts sufficient to be capable of providing a visibly colored solution when extracted with sufficient amounts of benzene; and (b) extracting C_{60} from the sooty carbon product in amounts sufficient to provide a visibly colored solution when extracted with benzene in amounts sufficient to dissolve the C_{60} present in the sooty carbon product.

In accordance with another preferred aspect of the present invention there is provided a process for preparing C_{60} comprising: (a) vaporizing elemental carbon in the presence of an inert quenching gas under conditions effective to provide a sooty carbon product comprising C_{60} molecules, the C_{60} molecules being present in the sooty carbon product in amounts sufficient to be capable of providing a visibly colored solution when extracted with benzene; (b) depositing the sooty carbon product on a collecting surface; (c) removing the sooty carbon product from the collecting surface; and (d) extracting product which is predominantly C_{60} from the sooty carbon product, the C_{60} being present in sufficient quantities to provide a visibly colored solution when extracted with benzene present in amounts sufficient to dissolve the C_{60} present in the sooty carbon product.

In accordance with another preferred aspect of the present invention there is provided a process for preparing C_{60} comprising: (a) vaporizing elemental carbon in the presence of an inert quenching gas under conditions effective to form a sooty carbon product comprising C_{60} molecules, the C_{60} molecules being present in the sooty carbon product in amounts capable of extracting therefrom the C_{60} in solid form; and (b) extracting in solid form C_{60} from the sooty carbon product.

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In accordance with another embodiment of the present invention there is provided a process for preparing C_{60} comprising: (a) vaporizing elemental carbon in the presence of an inert quenching gas at a pressure sufficient to generate a sooty carbon product comprising C_{60} , the C_{60} being present in the sooty carbon product in sufficient amounts to produce and collect therefrom crystalline C_{60} ; (b) separating said C_{60} from the sooty carbon product under conditions effective to recover crystalline C_{60} .

10 In accordance with a further embodiment of the present invention there is provided a process for preparing C_{60} comprising: (a) vaporizing elemental carbon to form carbon vapor in an atmosphere of an inert gas; (b) quenching the carbon vapor in the inert gas under conditions sufficient to effectively condense and nucleate the vapor to form a sooty carbon product comprising C_{60} molecules in sufficient quantities to extract therefrom an amount sufficient to collect the C_{60} as a crystalline product; (c) collecting the sooty carbon product; (d) separating the C_{60} from the sooty carbon product and recovering therefrom the C_{60} in crystalline form.

20 In accordance with a further aspect of the present invention there is provided a process for producing C_{60} comprising: (a) vaporizing elemental carbon in an atmosphere of an inert gas at a pressure sufficient to generate a sooty carbon product comprising C_{60} ; the C_{60} being present in sufficient quantities to recover therefrom C_{60} in amounts to be discernible as a colored solid; (b) separating the C_{60} from the sooty carbon product under conditions effective to recover therefrom a colored crystalline C_{60} .

In accordance with another aspect of the present invention there is provided a process for preparing C_{60} comprising vaporizing elemental carbon selected from the group consisting of graphite, amorphous carbon and glassy carbon in an inert quenching gas at a pressure of at least 50 torr so as to generate a carbon soot comprising C_{60} and separating the C_{60} from the soot under conditions effective to recover substantially pure crystalline C_{60} therefrom.

30 In accordance with a further embodiment of the present invention there is provided a process for preparing C_{60} comprising: (a) vaporizing elemental

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carbon in the presence of an inert quenching gas under conditions effective to form a sooty carbon product comprising C_{60} molecules in macroscopic amounts; (b) depositing the sooty carbon product on a collecting surface remote from the situs of vaporization; (c) removing the sooty carbon product from the collecting surface; and (d) extracting a product comprising a macroscopic amount of C_{60} from the sooty carbon product.

10 In accordance with a further aspect of the present invention there is provided a process for preparing C_{60} comprising: (a) vaporizing elemental carbon in the presence of an inert quenching gas at a pressure ranging from less than 1 atmosphere up to a pressure of 10 atmospheres under conditions effective to form a sooty carbon product comprising C_{60} in quantities sufficient to isolate C_{60} as a solid when extracted from the sooty carbon product; (b) depositing the sooty carbon product on a collecting surface remote from the situs of vaporization; (c) removing the sooty carbon product from the collecting surface; and (d) extracting C_{60} from the sooty carbon product in quantities sufficient to isolate C_{60} as a solid when extracted from the sooty carbon product.

20 In accordance with a further aspect of the present invention there is provided a process for preparing C_{60} comprising: (a) vaporizing elemental carbon in the presence of an inert quenching gas under a pressure ranging from less than 1 atmosphere up to 10 atmospheres under conditions effective to form a sooty carbon product comprising C_{60} in quantities sufficient to isolate C_{60} as a discernible solid when extracted from the sooty carbon product; (b) extracting C_{60} from the sooty carbon product in quantities sufficient to isolate the C_{60} as a discernible solid.

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In the accompanying figures, Fig. 1 is a micrograph of typical crystals of the 98% C₆₀, 2% C₇₀ material showing thin platelets, rods and stars of hexagonal symmetry.

Fig. 2 is an x-ray diffraction of a microcrystalline powder of the 98% C₆₀, 2% C₇₀ solid material. Inset at upper left is a single crystal electron diffraction pattern indexed with Miller indices compatible with the x-ray pattern, taken on a thin platelet as in Figure 1 with the electron beam perpendicular to the flat face.

Fig. 3 is an infrared absorption spectrum of an approximately 2 micrometer thick coating of the 98% C₆₀, 2% C₇₀ material on a silicon substrate, referenced to a clean silicon substrate. Absorption is given as optical density= $\log_{10} (1/T)$, where T is transmission. Apparent negative absorptions are due to the coating acting in part as a non-reflecting layer.

Fig. 4 is a visible-ultraviolet absorption spectrum of an approximately 0.1 micrometer thick coating of the 98% C₆₀, 2% C₇₀ material on quartz. Shown at the bottom are positions and relative oscillator strengths for allowed transitions calculated for the C₆₀ molecule by Larsson, et al.

The first step in the production of C₆₀ and C₇₀ molecules is vaporizing carbon from any source containing carbon in its various forms, e.g. graphite, amorphous and glassy carbon. It is preferred that this vaporization takes place in an evacuated reactor (e.g., a bell jar). The carbon is vaporized by heating in the presence of an inert quenching gas. The carbon vapor is nucleated in the presence of the inert quenching gas to form smoke particles.

1 In the production of C_{60} and C_{70} , any procedure
for vaporizing carbon can be used, although the preferred
method relies on the use of a high intensity electrical
current with graphite rods as electrodes. These rods are
5 constructed to permit vaporization of carbon at the tip
of the rod to produce a high density vapor of carbon.
For best results, the end of one of the rods is reduced
in diameter so that the vaporization occurs at the
reduced tip. The rods can be prepared using any of the
10 various forms of carbon, such as graphite, amorphous and
glassy carbon.

The inert quenching gas can be any of the usual
inert gases such as the noble gas. Argon and helium are
preferred, the latter being most preferred. Other inert
15 gases commonly employed to provide a non-reactive
atmosphere can also be used as quenching gas.

The amount of C_{60} and C_{70} produced from this
carbon source is dependent upon the pressure of the
quenching gas. At lower pressures relatively pure C_{60}
20 molecules can be produced in high yield with minor
concentrations of C_{70} . For the production of
predominantly C_{60} molecules, the pressure at which the
quenching gas is maintained should be subatmospheric and
preferably about 50-400 torr. Especially preferred is a
25 pressure of approximately 100 torr. The use of any lower
pressure may result in reduced yield of C_{60} .

However, as the pressure is raised, the ratio
of $C_{70}:C_{60}$ is also increased.

30 If the pressure is increased to at least two
atmospheres, the greatest percentage of C_{70} product is
formed. Theoretically, the pressure can be raised to any
level just below the point where the reactor would

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- 1 explode from the increased pressure. However, at the
higher pressures, the yield of the overall product (C_{60}
and C_{70}) is reduced even though the ratio of $C_{70}:C_{60}$ is
also increased. Therefore, as a practical consideration,
5 the pressure of the quenching gas should not be greater
than 10 atmospheres. The preferred pressure for
maximizing the amount of C_{70} produced is 2-3 atmospheres.

10 The produced quenched vapor of carbon, i.e.,
the smoked particles coats the internal surface of the
reactor and of collecting substrates as black soot.
These collecting surfaces are inert to the vaporized
carbon. They can be transparent and/or coated with an
inert metal. Examples include glass, or gold coated
15 glass surfaces and the like. These collecting surfaces
are located in the reactor in the path of the carbon
smoke. The black coating can be removed by any suitable
means, e.g., by scraping the solids from the coated
surfaces. The C_{60} and C_{70} molecules can be removed from
20 this collected quenched product by contacting said
quenched product with an extracting solvent. In other
words, the black soot is placed in a container containing
the extracting solvent, or the extracting solvent is
poured onto the black soot placed in a container. In
25 either case, the C_{60} and C_{70} molecules become dissolved in
the solvent, while the remainder of the black soot
remains insoluble. The insoluble material is separated
from the solution containing the C_{60} and C_{70} molecules,
e.g., by decanting, or by filtration, and the like.

30 Suitable solvents include non-polar organic
solvents, such as the alkanes containing 5-10 carbon
atoms (e.g. pentanes, hexanes, heptanes, octanes),
benzene and alkyl-benzenes (e.g. toluene, xylene), carbon
disulfide, carbon tetrachloride, naphtha, 1,1,1-

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1 trichloroethane, and the like. Simple solubility
determinations using classical laboratory methods will
5 permit selection of other suitable solvents. The
preferred solvents are carbon disulfide, benzene, carbon
tetrachloride and toluene. Especially preferred are
benzene, carbon tetrachloride and carbon disulfide.

The product obtained contains a mixture of C_{60}
and C_{70} . As described hereinabove, the amounts of C_{60}
10 and C_{70} present is dependent upon the pressure used. If
subatmospheric pressures are used, such as 50-400 torr,
the product is predominately pure C_{60} with a minor amount
of C_{70} present. Thus, when the collected product is
dispersed in the extracting solvent, the product obtained
15 is a mixture of C_{60} and C_{70} . For example, when the
pressure is 100 torr, the product formed is about 98% C_{60}
and about 2% C_{70} . This product can be separated from the
organic solvent solution by standard methods as by
evaporation of the solvent or by dilution of the solvent
20 solution with a non-solvent for C_{60} . The product can be
crystallized by careful evaporation of the organic
solvent or by sublimation procedures.

In a preferred embodiment of producing C_{60} and
 C_{70} , pure graphite rods are vaporized by passing high
25 electrical current (either dc or ac) through narrowed
tips of graphite rods. Electron beam, laser and RF
heating can be used in lieu of electrical heating. This
is done in a reactor (such as a bell jar) that has been
evacuated, purged and filled with inert gas at or
30 preferably below atmospheric pressure, e.g., pressures
ranging from about 50 to about 400 torr. and even higher.
The graphite rods are typically 1/4 inch in diameter with

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1 about 1 cm length of one rod reduced in diameter to about
5 mm. The electrical heating vaporizes the constricted
tip of the graphite rod producing a high density vapor of
carbon, which quickly condenses into a smoke consisting
5 of very fine particles (of the order of 0.1 microns) of
graphitic carbon with an admixture of a few percent of
the desired C_{60} molecule. At this point in the process
there is a heavy black coating on collecting substrates
and/or on the walls of the chamber which can be easily
10 scraped off for the recovery step.

For recovery, the sooty product is treated with
benzene to provide a brownish-red solution. After
separation of the undissolved graphitic carbon, the
benzene solution is evaporated to obtain microcrystalline
15 product. Alternatively, the product can be sublimed from
the sooty carbon at 300° to 400°C. and the sublimation
product obtained by condensation on a conventional
substrate.

When the pressure of inert quenching gas is 100
20 torr, the product formed is 98% C_{60} and 2% C_{70} . This
product, as obtained from the solvent extract of the
sooty graphitic carbon, is a dark brown to black
crystalline material. When obtained by sublimation in
vacuum or inert atmosphere, the product is obtained as a
25 brown to gray coating depending on thickness.

On analysis by mass spectroscopy, the spectrum
clearly shows a strong peak at mass 720 amu (i.e., the
mass of C_{60}) and a clean peak at 840 amu (i.e., the mass
of C_{70}). Significant differences in the spectra occur
30 only in the abundances in the mass domain lower than 300
amu. Most of these differences seem to originate from
the different ionization techniques in the mass
spectrometer and from the different kinds of sample
desorption. So far, the cleanest mass spectra have been
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obtained when the material was evaporated and ionized in the vapor phase by electrons. In such spectra the mass range above 40 amu is dominated by the C_{60} mass along with its expected isotope lines. The only other large mass found in any abundance corresponds to C_{70} , with a ratio of C_{70} to C_{60} of about .02.

10 Studies by optical microscopy of the C_{60} material which is left after evaporating the benzene solution show a variety of what appear to be crystals -- mainly rods, platelets, and star-like flakes. Figure 1 shows a microphotograph of such a crystal assemblage. All crystals tend to exhibit six-fold symmetry. In transmitted light they appear red to brown in color; in reflected light the larger crystals have a metallic appearance, whereas the platelets show interference colors consistent with an index of refraction of about 2.

20 The platelets can be rather thin and thus are ideally suited for electron diffraction studies in an electron microscope. (See the insert in Figure 2).

30 In order to determine if the C_{60} molecules form a regular lattice, electron and x-ray diffraction studies on the individual crystals and on the powder were carried out. A typical X-ray diffraction pattern of the purified C_{60} powder is shown in Figure 2. To aid in comparing the electron diffraction results with the X-ray results the electron diffraction pattern is inserted into the corner of Figure 2. From the hexagonal array of diffraction spots indexed as shown in the Figure, a d-spacing of 8.7 Å was deduced corresponding to the (100) reciprocal lattice vector of a hexagonal lattice. The most obvious correspondence between the two types of diffraction is between the 5.01 Å peak of the X-ray pattern and the (100) spot of the electron diffraction pattern, which gives a spacing of about 5.0 Å. Assuming that the C_{60}

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molecules are behaving approximately as spheres stacked in a hexagonal close packed lattice with a c/a ratio of 1.633, d-spacings can be calculated. The results are shown in Table I.

Table I: X-Ray Diffraction Results and Assignments
For a Hexagonal Lattice Using

$$a = 10.02 \text{ \AA}, c = 16.36 \text{ \AA}$$

$$\frac{1}{d^2} = \frac{4}{3} \left(\frac{h^2 + hk + k^2}{a^2} \right) + \frac{l^2}{c^2}$$

Measured 2θ (degrees)	Measured d-spacing (Å)	Calculated d-spacing (Å)	Assignment (hkl)
10.2 shoulder	8.7	8.68	(100)
10.81	8.18	8.18	(002)
		7.67	(101)
17.69	5.01	5.01	(110)
20.73	4.28	4.27	(112)
21.63	4.11	4.09	(004)
28.1	3.18	3.17	(114)
30.8	2.90	2.89	(300)
32.7	2.74	2.73	(006)

The values derived from this interpretation are $a = 10.02 \text{ \AA}$ and $c = 16.36 \text{ \AA}$. The nearest neighbor distance is thus $10.02 \text{ \AA}$. For such a crystal structure the density is calculated to be 1.678 g/cm^3 , which is consistent with a value of 1.65 ± 0.05 determined by suspending crystal samples in aqueous GaCl_3 solutions of

1 known densities. Although the agreement shown in Table 1
is good, the absence of the characteristically strong
(101) diffraction in hcp and the broad continuum in
certain regions suggest a less than perfect crystalline
5 order. Furthermore, X-ray diffraction patterns obtained
on carefully grown crystals up to 500 micrometers in size
with well developed faces yielded no clear spot pattern
(in contrast to the electron diffraction pattern on
micron-size crystals). It thus appears that these larger
10 crystals do not exhibit long range periodic order in all
directions.

A likely explanation for the unusual
diffraction lies in the disordered stacking arrangement
of the molecules in planes normal to the c-axis. It is
15 well known that the position taken by spheres in the
third layer of stacking determines which of the
close-packed structures occurs, the stacking arrangement
in fcc being ABCABC while that in hcp is ABABAB. If the
stacking sequence varies, the X-ray lines due to certain
20 planes will be broadened by the disorder while other
lines will remain sharp. Such disordered crystalline
behavior was observed long ago in the close packed
structure of cobalt, where X-ray diffraction lines such
as (101), (102) and (202) were found to be substantially
25 broadened due to the stacking disorder. Reflections from
planes such as (002) remain sharp since these planes have
identical spacings in both fcc and hcp structures. A
general expression for which peaks are broadened by this
kind of disorder have been given in terms of Miller
30 indices (h,k,l) as $h - k = 3t \pm 1$, $l \neq 0$, where t is an
integer. None of these broadened reflections are
apparent in the X-ray pattern of Figure 2. This may
explain the weakness of the characteristically strong

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1 (101) peak. Whether or not this stacking disorder is
related to the presence of the possibly elongated C_{70}
molecules is yet to be determined.

5 In small crystals at least, the C_{60} molecules
appear to be assembling themselves into a somewhat
ordered array as though they are effectively spherical,
which is entirely consistent with the soccer ball
hypothesis for their structure. The additional diameter
over the calculated $7.1 \text{ \AA}$ value for the carbon cage
10 itself must represent the effective van der Waals
diameter set by the repulsion of the pi electron clouds
extending outward from each carbon atom. Scanning
tunnelling spectroscopy of the C_{60} molecules clearly
shows the spherical nature of the C_{60} molecules.

15 Some scanning tunnelling microscope images of a
carbon sample prepared in accordance with the procedure
described hereinabove at pressures of helium at 100 torr
show a spherical molecule of twice the diameter of the
 C_{60} molecules. This is evidence of the existence of a
20 caged molecule containing 240 carbon atoms or a C_{240}
molecule.

Samples were prepared for spectroscopy by
subliming pure material onto transparent substrates for
transmission measurements. Depending on the pressure of
25 helium in the sublimation chamber, the nature of the
coatings can range from uniform films (at high vacuum) to
coatings of C_{60} smoke (i.e., sub-micron microcrystalline
particles of solid C_{60}) with the particle size depending
to some extent on the pressure.

30 Figure 3 shows the transmission spectrum of an
approximately 2 micrometer thick C_{60} coating on a silicon
substrate. The infrared bands show the four most intense
lines at 1429, 1183, 577, and 528 cm^{-1} , with no
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1 underlying continuum remaining from the soot. In early
tries at purifying C_{60} material, the infrared spectrum
showed a strong band in the vicinity of 3.0 micrometers,
which is characteristic of a CH stretching mode. After
5 much effort, this contaminant was successfully removed by
washing the soot with ether and using distilled benzene
in the extraction. The spectrum in Figure 3 was obtained
when the material cleaned in such a manner was sublimed
under vacuum onto the substrate. The spectrum shows very
10 little indication of CH impurities.

The presence of only four strong bands is what
is expected for the free, truncated icosahedral molecule
with its unusually high symmetry. Also present are a
number of other weak infrared lines which may be due to
15 other causes, among which may be absorption by the C_{70}
molecule or symmetry breaking produced, for example, by
isotopes other than C^{12} in the C_{60} molecule or by mutual
interaction of the C_{60} molecules in the solid.
Noteworthy, are weaker features at about 2330 and 2190
20 cm^{-1} which are located in the near vicinity of the free
 CO_2 and CO stretching modes. This may imply some
attachment of CO_2 or CO to a small fraction of the total
number of C_{60} molecules. Another noteworthy effect can
be observed in the feature at $675\ cm^{-1}$, which is weak in
25 the thin film samples but almost as strong as the four
main features in the crystals. This vibrational mode may
be of solid state rather than molecular origin.

Figure 4 shows an absorption spectrum taken on
a uniform film coated onto a quartz glass substrate. The
30 ultraviolet features are no longer obscured by the
graphitic carbon background as in our previous spectra.
Broad peaks at 216, 264 and 339 nm dominate the spectra.
Weaker structures show up in the visible, including a

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1 plateau with ends at about 460 and 500 nm and a very weak
peak near 625 nm. At the bottom of Figure 4 are shown
positions and relative oscillator strengths taken from
Larsson, et al. (Chem. Phys. Lett. 137, 501-504)
5 calculated for the C_{60} molecule. This reference also
shows a variety of forbidden bands with the lowest energy
ones in the vicinity of 500 nm. There seems to be a
rough correspondence between the present measurements on
thin films and the allowed transitions predicted for the
10 molecule. There was no band at 386 nm in our films of
 C_{60} , as disclosed by Heath, et al. (J. Chem. Phys. 87,
4236-4238 (1987)) using a laser depletion spectroscopy
method and attributed to the C_{60} molecule. Quite similar
spectra to that in Figure 4 have been recorded for
15 microcrystalline coatings deposited at helium pressures
of 100 torr, for example. The peaks occur at the
slightly shifted positions of 219, 268, and 345 nm.

The C_{70} molecule is larger than the C_{60}
molecule. The C_{70} molecule shows a molecular ion peak at
20 840 amu. Furthermore, a noticeable peak in the
ultraviolet spectrum of the C_{70} molecule taken on a
uniform film coated onto a quartz glass substrate is
exhibited at about 216 nm. This is a broad peak.
Surprisingly, it appears that the C_{70} molecule is more
25 stable than C_{60} .

Thus, using the procedures described
hereinabove, at quenching pressures of less than 1
atmospheric pressure and especially at pressures of
50-400 torr, a product is produced which is predominantly
30 C_{60} and contains minor amounts of C_{70} . The C_{60} product
can be used or can be further purified.

Further purification and separation of C_{60} and
 C_{70} can be made by many conventional techniques known to
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1 one skilled in the art, e.g., fractional crystallization,
column chromatography, capillary electrophoresis, HPLC,
preparative thin-layer chromatography, and the like.

5 Because the molecular figuration of C_{60} and C_{70}
are different, the attractive intermolecular forces are
different which allows for the two molecules to be
readily separated.

10 Furthermore, the solubility of C_{60} and C_{70} in
pure solvents and mixed solvents are also different from
each other, which also makes the two compounds separable
by using conventional techniques known to one skilled in
the art, such as crystallization, extraction, and the
like.

15 For example, pure C_{60} and pure C_{70} molecules
can be isolated as follows. The black sooty mixture of
 C_{60} and C_{70} which is produced according to the procedure
described hereinabove is placed in the extracting
solvent, such as benzene. The insoluble residue is
20 removed and the resulting benzene solution containing C_{60}
and C_{70} molecules is concentrated. The C_{60} and C_{70}
solution is added to a packed column with an adsorbent,
such as alumina. The column is eluted with an eluent
such as benzene or a mixture of benzene and toluene.
Various fractions of set volume e.g., 10 mL, are
25 collected. The eluent i.e., the solvent is removed from
each fraction such as by evaporation to dryness. The
fractions with product will contain microcrystals, the
identity of which can be confirmed by spectroscopy, e.g.,
mass spectroscopy.

30 Thus, the process of the present invention can
produce a product which is predominantly C_{60} , which, if
desired, can be further purified by the purification and
separation techniques described hereinabove.

1 Furthermore, the present invention contemplates
two different variations of the procedure described
hereinabove to make C_{70} molecules. First, if
subatmospheric pressures of quenching gases are used in
5 the initial step, small amounts of C_{70} are produced,
which can be separated from the C_{60} molecules using the
purification techniques described hereinabove. However,
if the pressure of the quenching gas is raised to at
least 2 atmospheres, after separation and purification, a
10 greater percentage of substantially pure C_{70} would be
produced from each vaporization of carbon.

The present new products, C_{60} , C_{70} , or mixtures
thereof have the similar utilities as graphite. However,
they are particularly valuable for forming products of a
15 higher order of stability than those formed from
graphitic carbon, and can be processed into formed or
molded products such as C_{60} fibers, C_{70} fibers, or
mixtures thereof using standard processing techniques.
In this regard, free-flowing, particulate C_{60} and C_{70} are
20 of special value particularly for use in producing molded
products, especially those extended in at least one
direction. C_{60} and C_{70} are also useful for producing a
low temperature C_{60} vapor (300°-400°C.) and C_{70} vapor of
the respective molecules to produce low temperature
25 atomic and molecular beams of carbon which is not now
possible using graphite as the carbon source. Further,
the synthesis of compounds such as $C_{60}H_{60}$ and $C_{60}F_{60}$ can
be accomplished by introducing hydrogen and fluorine,
respectively, into a reactor containing C_{60} vapor.
30 Furthermore, the C_{60} product and the C_{70} product may be
used as an industrial paint pigment or as a lubricant.
Moreover, since the C_{60} and C_{70} molecule are hollow, they
could be used for binding and/or storing molecules e.g.,
toxic material.

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EXAMPLE 1

5 C_{60} -containing carbon dust was produced in a conventional bell-jar carbon evaporator which was first evacuated to 10^{-4} torr by either an oil diffusion pump or a turbo pump, both equipped with liquid nitrogen traps, and then filled with an inert quenching gas. Helium and argon were used at pressures ranging up to 400 torr. Then graphite rods (as previously described herein) were
10 evaporated using a current of about 100 amps (either AC or DC).

The smoke which formed in the vicinity of the evaporating carbon rods was collected on substrates which were placed within 5 cm to 10 cm of the evaporating
15 carbon rods.

The evaporator was opened after a cool down period of 10-30 min. and the carbon dust samples removed by scraping substrate surfaces and the internal surfaces of the bell-jar. After washing with ether, the collected
20 dust samples were then extracted with benzene to produce a wine-red to brown solution. On evaporation of the solution, C_{60} was obtained as a microcrystalline residue.

The crystals were sublimed by heating in vacuo or in a quenching inert gas to 400°C. and collected on a
25 substrate. The sublimed product was brown to gray in color.

In powder form, the present new carbon allotrope is brownish-red.

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EXAMPLE 2

The procedure of Example 1 is repeated except, in the original step, the graphite rods are evaporated at 2 or more atmospheres of helium pressure in the chamber.

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The product obtained from this procedure contains a greater percentage of C₇₀ than does the product in Example 1.

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EXAMPLE 3

Pure C₆₀ and pure C₇₀ are obtained as follows:

5 The C₆₀ and C₇₀ mixtures prepared in either Examples 1 or 2 are dissolved in benzene and added to an alumina column. Using benzene as the eluent, the fractions are collected and each elute fraction is evaporated to dryness. The presence of C₆₀ or C₇₀ in the fraction can be determined by taking mass spectroscopy thereof.

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1 The above embodiments and examples are given to
illustrate the scope and spirit of the instant invention.
These embodiments and examples are within the
contemplation of the present invention. Therefore, the
5 present invention should be limited only by the appended
claims.

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WHAT IS CLAIMED IS:

1. A method of producing C₆₀ and C₇₀ compounds which comprises evaporating graphite in an atmosphere of an inert quenching gas at pressures effective to quench the vaporized carbon in a reactor that had previously been evacuated, collecting the quenched carbon product produced therefrom and contacting the quenched carbon product with an extracting non-polar organic solvent under effective conditions to separate the C₆₀ and C₇₀ compounds therefore.
2. The method according to Claim 1 wherein the quenched carbon is collected on a collecting substrate.
3. The method according to Claim 1 or 2 wherein the C₆₀ and C₇₀ compounds are recovered from the organic solvent.
4. The method according to Claim 3 wherein the separating step comprises evaporating the solvent.
5. The method according to Claim 1 wherein the solvent is benzene or carbon tetrachloride.
6. The method according to any one of Claims 1 to 5 wherein the evaporation of graphite is effected by passing high electrical current through graphite rods.
7. The method according to any one of Claims 1 to 6 wherein the inert gas is helium or argon.
8. The method according to any one of Claims 1 to 7 wherein the graphite is evaporated at pressures ranging from about 50 to about 400 torr.
9. The method according to any one of Claims 1 to 7 wherein the pressure ranges from about 2 to about 3 atmospheres.

10. The method according to any one of Claims 1 to 9 further comprising separating the C₆₀ compound from the C₇₀ compound.

11. Macroscopic amounts of an amorphous or crystalline particulate matter comprised of C₆₀ or C₇₀.

12. A carbon product comprising a macroscopic amount of a mixture of C₆₀ or C₇₀.

13. A substantially pure carbon product, the mass spectrum of which shows a strong peak at mass 720 amu, the infrared bonds to which have four intense lines at 1424, 1183, 577 and 528 cm⁻¹, absorption peaks in the UV at 264 and 339 nm, soluble in non-polar organic solvents and sublimates at a temperature of from about 300 to 400°C, present in amounts sufficient to be discerned as a solid.

14. The C₆₀ or C₇₀ produced in macroscopic amounts in accordance with any one of Claims 1 to 10.

15. A formed or molded product comprising macroscopic amounts of C₆₀ or C₇₀.

16. The product according to Claim 15 which is extended in at least one direction.

17. A free flowing particulate comprised of macroscopic amounts of C₆₀ or C₇₀.

18. Macroscopic amounts of substantially pure C₆₀ or C₇₀.

19. An isolated substantially pure carbon product in macroscopic amounts, the mass spectrum of which shows a molecular ion at 840 amu, a broad peak in the ultraviolet at 216 nm, and soluble in non-polar organic solvents.

20. Isolated C₆₀ or isolated C₇₀ in amounts sufficient to be visible.
21. The vapor of C₆₀ or C₇₀, said C₆₀ and/or C₇₀ being present in amounts sufficient to isolate said C₆₀ or C₇₀ as a solid when the vapor is condensed.
22. A method of extracting C₆₀ and C₇₀ from a carbon source containing same which comprises contacting the carbon source with a non-polar organic solvent.
23. A method according to Claim 22 wherein the C₆₀ and C₇₀ are recovered from the organic solvent.
24. A method according to Claim 22 or 23 wherein the solvent is benzene, carbon tetrachloride or carbon disulfide.
25. A method according to Claim 23 wherein a carbon source containing C₆₀ and C₇₀ is contacted with benzene and recovering C₆₀ and C₇₀ from the benzene solution thus formed.
26. A process for preparing C₆₀ comprising:
 - (a) vaporizing a carbon source in the presence of an inert quenching gas under conditions effective to form a sooty carbon product comprising C₆₀ molecules, said C₆₀ molecules being present in said sooty carbon product in amounts capable of extracting and recovering predominantly therefrom said C₆₀ as a visible solid form; and
 - (b) recovering C₆₀ from said sooty carbon product.
27. A process for preparing C₆₀ in macroscopic amounts comprising:
 - (a) vaporizing a carbon source in the presence of an inert quenching gas under conditions effective to produce a carbon product comprising C₆₀ molecules, said C₆₀ molecules being present in amounts capable of extracting and recovering therefrom said C₆₀ in macroscopic amounts;

- (b) depositing the sooty carbon product on a collecting surface;
- (c) removing the sooty carbon product from the collecting surface; and
- (d) recovering a product comprising C_{60} in macroscopic amounts from said sooty carbon product.

28. A process for preparing C_{60} comprising:

- (a) vaporizing a carbon source in the presence of an inert quenching gas under conditions effective for form a sooty carbon product comprising C_{60} molecules, said C_{60} molecules being present in said sooty carbon product in amounts sufficient to be capable of providing a visibly colored solution when extracted with sufficient amounts of benzene; and
- (b) recovering C_{60} from said sooty carbon product.

29. A process for preparing C_{60} comprising:

- (a) vaporizing a carbon source in the presence of an inert quenching gas under conditions effective to form a sooty carbon product comprising C_{60} molecules, said C_{60} molecules being present in said sooty carbon product in macroscopic amounts; and
- (b) recovering macroscopic amounts of said C_{60} from said sooty carbon product.

30. A process for preparing a carbon allotrope which is C_{60} or C_{70} or a mixture thereof comprising molecules consisting solely of carbon atoms, which are soluble in non-polar organic solvents, said process comprising:

- (a) vaporizing a carbon source in the presence of an inert gas to produce a carbon vapor;
- (b) quenching said vapor of carbon in said inert gas under conditions effective to nucleate and condense said carbon allotrope, said allotrope being present in said sooty carbon product in amounts sufficient to be capable of extracting and recovering therefrom said allotrope in solid form; and
- (c) recovering said carbon allotrope of C_{60} or C_{70} or a mixture thereof from said sooty carbon product.

31. The process according to any one of Claims 26 to 29 wherein the recovering step comprises contacting the sooty carbon product with a non-polar organic solvent effective to dissolve said C_{60} , said solvent being present in amounts sufficient to dissolve the C_{60} in said sooty carbon product.

32. The process according to Claim 31 wherein the recovering step comprises subliming the C_{60} from the sooty carbon product and condensing the sublimed C_{60} .

33. The process according to Claim 30 wherein the carbon allotrope is recovered in solid form.

34. The process according to Claim 30 wherein the carbon allotrope is recovered in solution.

35. The process according to Claim 34 wherein the carbon allotrope is recovered in a solution of a non-polar organic solvent.

36. The process according to any one of Claims 26 to 29 wherein said C_{60} is recovered in solid form.

37. The process according to any one of Claims 26 to 29 wherein said C_{60} is recovered in solution.

38. The process according to any one of Claims 31 to 35 wherein the non-polar organic solvent is benzene, toluene, carbon tetrachloride, 1,1,1-trichloroethane, xylenes or alkanes having 5-10 carbon atoms.

39. The process according to Claim 36, wherein said C_{60} in solid form is a crystalline solid of C_{60} .

40. The process of Claim 39, wherein said crystalline solid of C_{60} is substantially pure crystalline C_{60} .

41. The process of Claim 36, wherein C_{60} in solid form is substantially pure solid C_{60} .

42. The process according to any one of Claims 26 to 29 wherein C_{70} is additionally present in the sooty carbon product.

43. The process according to any one of Claims 26 to 29 wherein C_{70} is additionally present in the sooty carbon product and is additionally recovered therefrom.

44. The process according to Claim 42 wherein the C_{70} is separated from the C_{60} .

45. The process according to Claim 44 wherein the C_{70} separated from said C_{60} is substantially pure solid C_{70} .

46. The process according to Claim 45, wherein substantially pure solid C_{70} is substantially pure crystalline C_{70} .

47. The process of Claim 27, wherein the depositing step comprises collecting the sooty carbon product on a collecting surface distanced 5-10 cm from said vaporization.

48. The process according to any one of Claims 26 to 30 wherein the carbon source is vaporized in an evacuated reactor.

49. The process according to any one of Claims 26 to 30 wherein the carbon source subject to vaporization is graphite or amorphous or glass carbon.

50. The process according to any one of Claims 26 to 30 wherein the carbon source subject to vaporization is graphite.

51. The process according to any one of Claims 26 to 30 wherein the carbon source is vaporized by passing an electrical current of sufficient intensity through said carbon source to produce the sooty carbon product.

52. The process according to any one of Claims 26 to 30 wherein the inert gas is a noble gas.

53. The process according to Claim 52 wherein the noble gas is helium or argon.

54. The process according to Claim 38 wherein the solvent is benzene.

55. The process according to any one of Claims 26 to 30 wherein the recovering step comprises evaporating the solvent.

56. The process according to Claim 36 further comprising purifying the solid C₆₀.

57. The process according to any one of Claims 26 to 56 wherein the carbon source is vaporized in the presence of an inert quenching gas at a pressure of at least about 50 torr.

58. A method of extracting C₆₀ from a carbon source containing same which comprises contacting the carbon source with a non-polar organic solvent.

59. The method according to Claim 58 wherein the non-polar organic solvent is benzene, carbon tetrachloride, toluene, carbon disulfide, xylene, or alkane having 5-10 carbon atoms.

60. The method according to Claim 58 wherein the C₆₀ is removed from the non-polar solvent.

61. The method according to Claim 58 wherein C₇₀ is additionally present and is separated from the C₆₀.

62. The C₆₀ produced in accordance with Claim 26 or 27.
63. A carbon product comprising C₆₀ in macroscopic amounts, C₇₀ in macroscopic amounts as a mixture of C₆₀ and C₇₀ in macroscopic amounts.
64. Solid C₆₀.
65. Solid C₇₀.
66. Crystalline C₆₀.
67. Crystalline C₇₀.
68. Substantially pure solid C₆₀.
69. Substantially pure solid C₇₀.
70. Substantially pure crystalline C₆₀.
71. Substantially pure crystalline C₇₀.
72. C₆₀ in amounts sufficient to isolate as a visible solid.
73. C₇₀ in amounts sufficient to isolate as a visible solid.
74. Macroscopic amounts of C₆₀.
75. Macroscopic amounts of C₇₀.
76. Substantially pure C₆₀ in amounts sufficient to isolate as a visible solid.
77. Substantially pure C₇₀ in amounts sufficient to isolate as a visible solid.

78. A solution of a carbon allotrope selected from the group consisting of C_{60} and C_{70} being dissolved in an organic non-polar solvent, said allotrope being present therein in amounts sufficient to be capable of recovering it as a solid when the solvent is evaporated.

79. The solution according to Claim 78, wherein the non-polar organic solvent is toluene, benzene, xylene, carbon disulfide, carbon tetrachloride or alkane having 5 – 10 carbon atoms.

80. The product of any one of Claims 66, 68, 70, 72, 74 and 76, dissolved in a non-polar organic solvent.

81. The product of any one of Claims 65, 67, 69, 71, 73, 75 and 77, dissolved in a non-polar organic solvent.

82. The solid product of Claim 80 or 81 wherein the non-polar organic solvent is benzene, toluene, xylene, alkane having 5-10 carbon atoms, carbon tetrachloride or 1,1,1-trichloroethane.

83. The method according to any one of Claims 1 to 10 wherein the pressure ranges from subatmospheric up to and including 10 atmospheres.

84. The process according to Claim 83 wherein the pressure ranges from about 50-400 torr.

85. C_{60} in an amount at least as large as that amount which is sufficient to see under an optical microscope.

86. C_{70} in an amount at least as large as that amount which is sufficient to see under an optical microscope.

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87. A product containing C_{70} and substantially pure solid C_{60} .
88. A product containing substantially pure solid C_{60} and substantially pure solid C_{70} .
89. A product containing C_{60} and substantially pure solid C_{70} .
90. Macroscopic amounts of substantially pure C_{60} .
91. Macroscopic amounts of substantially pure C_{70} .
92. A carbon product prepared by
 - (a) vaporizing a carbon source in the presence of an inert quenching gas under conditions effective to produce a sooty carbon product comprising C_{60} , said C_{60} being present in said sooty carbon product in sufficient amounts to allow said C_{60} to be separated and isolated from said sooty carbon product as a visible solid in solid form;
 - (b) collecting said sooty, carbon product produced therefrom;and
 - (c) separating said C_{60} from said sooty carbon product, said product being comprised of C_{60} present in amounts sufficient for the C_{60} therein to be perceived as a visible solid in solid form, said C_{60} being free of sooty carbon product.
93. A solid carbon product comprising C_{60} prepared by the process comprising:
 - (a) vaporizing a carbon source in the presence of an inert quenching gas under conditions effective to provide a sooty carbon product comprising C_{60} molecules;

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(b) depositing the sooty carbon product on a collecting substrate;

(c) removing the sooty carbon product from the collecting substrate; and

(d) separating C₆₀ from the sooty carbon product, said C₆₀ being produced in amounts sufficient to be isolated in solid form, said C₆₀ being free from the sooty carbon product.

94. The solid carbon product of Claim 93 in which step (d) comprises contacting the sooty carbon product with a non-polar organic solvent effective to dissolve the C₆₀ molecules therein, said solvent being present in an amount effective to dissolve the C₆₀ molecules in the sooty carbon product, separating the solvent containing the C₆₀ from the sooty carbon product recovering the non-polar organic solvent containing the dissolved C₆₀, and removing the solvent.

95. The carbon product of Claim 92 in which step (c) comprises contacting the sooty carbon product with a non-polar organic solvent effective to dissolve the C₆₀ molecules therein, said solvent being present in an amount effective to dissolve the C₆₀ molecules in the sooty carbon product, separating the solvent containing the C₆₀ from the sooty carbon product recovering the non-polar organic solvent containing the dissolved C₆₀, and removing the solvent.

96. The carbon product of Claim 93 in which the solvent is removed by evaporation.

97. The carbon product of Claim 92 in which the solvent is removed by evaporation.

98. The carbon product according to Claim 92 in which separating comprises subliming the carbon product comprising C₆₀ from the sooty carbon product.

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99. The carbon product according to Claim 93 in which separating comprises subliming the carbon product comprising C₆₀ from the sooty carbon product.

100. The solid carbon product according to Claim 92 wherein the carbon source is vaporized by passing an electrical current of sufficient intensity to produce the sooty carbon product.

101. The solid carbon product according to Claim 93 wherein the carbon source is vaporized by passing an electrical current of sufficient intensity to produce the sooty carbon product.

102. The solid carbon product according to Claim 92 wherein the inert quenching gas of step (a) is a noble gas.

103. The solid carbon product according to Claim 93 wherein the inert quenching gas of step (a) is a noble gas.

104. The solid carbon product of Claim 92 wherein the carbon source is vaporized in the presence of said inert quenching gas at a pressure ranging from subatmospheric to about 10 atmospheres.

105. The solid carbon product of Claim 93 wherein the carbon source is vaporized in the presence of said inert quenching gas at a pressure ranging from subatmospheric to about 10 atmospheres.

106. The solid carbon product of Claim 105 wherein the pressure of the inert quenching gas ranges from 50-400 torr.

107. The solid carbon product of Claim 106 wherein the pressure of the inert quenching gas ranges from 50-400 torr.

108. The solid carbon product of Claim 107 wherein the pressure of the inert quenching gas is about 100 torr.

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109. The solid carbon product of Claim 104 wherein the pressure of the inert quenching gas is about 100 torr.

110. A product comprised of C_{60} prepared by the process comprising:

- (a) vaporizing a carbon source in the presence of an inert quenching gas under conditions effective to produce a sooty carbon product comprising C_{60} molecules;
- (b) depositing the sooty carbon product into a collecting substrate;
- (c) removing the sooty carbon product from the collecting substrate; and
- (d) contacting the sooty carbon product with a non-polar organic solvent effective to dissolve the C_{60} , said C_{60} being present in the non-polar organic solvent amounts sufficient to provide a visibly colored solution and separating the solution containing the dissolved C_{60} from the sooty carbon product.

111. The product of claim 110 wherein the solvent containing the dissolved C_{60} is separated from the C_{60} molecules, said product being free of any sooty carbon product.

112. The carbon product of any one of Claims 92 to 111 which additionally contains C_{70} .

113. A formed or molded product comprising substantially pure solid C_{60} or substantially pure C_{70} .

114. The product according to Claim 110 which is extended in at least one direction.

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115. A free following particulate comprising substantially pure solid C₆₀ or substantially pure solid C₇₀.

116. A process for preparing C₆₀ comprising:

(a) vaporizing elemental carbon in the presence of an inert quenching gas under conditions effective to form a sooty carbon product comprising C₆₀ molecules, said C₆₀ molecules being present in said sooty carbon product in amounts capable of extracting therefrom said C₆₀ in macroscopic amounts and in solid form; and

(b) extracting C₆₀ in macroscopic amounts.

117. The process according to Claim 116 wherein said extracted C₆₀ is in solid form.

118. The process according to Claim 116 wherein said extracted C₆₀ is in solution.

119. The process according to Claim 118 wherein said extracted C₆₀ is in solution of a non-polar organic solvent.

120. The process according to Claim 119 wherein the non-polar organic solvent is benzene, toluene, carbon tetrachloride, 1,1,1-trichloroethane, xylene or alkanes having 5-10 carbon atoms.

121. The process according to Claim 116 wherein said extracted C₆₀ is in solid form.

122. The process according to Claim 116 wherein said extracted C₆₀ is in solution.

123. The process according to Claim 122 wherein said extracted C₆₀ is in solution of a non-polar organic solvent.

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124. The process according to Claim 123 wherein said non-polar organic solvent is benzene, toluene, carbon tetrachloride, 1,1,1-trichloroethane, xylene or alkanes having 5-10 carbon atoms.

125. The process according to Claim 116 or 117 wherein the C_{60} in solid form is a crystalline solid.

126. The process of Claim 125 wherein the C_{60} in solid form is substantially pure solid C_{60} .

127. The process of Claim 125 wherein the C_{60} in solid form is substantially pure crystalline C_{60} .

128. The process of Claim 116 wherein C_{70} is additionally present in the sooty carbon product and is additionally extracted therefrom.

129. The process of Claim 116 wherein C_{70} is additionally present in sooty carbon product and is separated from the C_{60} .

130. The process of Claim 129 wherein the C_{70} separated from said C_{60} is substantially pure solid C_{70} .

131. The process of Claim 130 wherein the substantially pure solid C_{70} is substantially pure crystalline C_{70} .

132. The process according to Claim 116 wherein extracting comprises contacting the sooty carbon product with a non-polar organic solvent effective to dissolve the C_{60} , said solvent being present in amounts sufficient to dissolve the C_{60} present in said sooty carbon product.

133. The process according to Claim 132 wherein extracting further comprises separating from said solvent a solid C_{60} .

134. The process of Claim 116 wherein extracting comprises subliming the C_{60} from the sooty carbon product and condensing the sublimed C_{60} .

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135. The process of Claim 116 wherein depositing step comprises collecting the sooty carbon product on a collecting surface distanced 5-10 cm from said vaporization.

136. The process of Claim 116 wherein the elemental carbon is placed into an evacuated reactor prior to the vaporization thereof.

137. The process of Claim 116 wherein the elemental carbon is vaporized in a bell jar carbon evaporator.

138. The process of Claim 116 wherein the elemental carbon subject to vaporization is graphite, or amorphous or glassy carbon.

139. The process of Claim 138 wherein the elemental carbon subject to vaporization is graphite.

140. The process of Claim 116 wherein the elemental carbon subject to vaporization is graphite rods.

141. The process of Claim 116 wherein the elemental carbon is vaporized by passing an electrical current of sufficient intensity through said carbon source to produce the sooty carbon product.

142. The process of Claim 141 wherein the electrical current is about 100 amps.

143. The process of Claim 116 wherein the inert quenching gas is a noble gas.

144. The process of Claim 143 wherein the noble gas is helium or argon.

145. The process of Claim 143 wherein the collecting substrate is a glass surface.

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146. The process of Claim 132 wherein the non-polar organic solvent is carbon disulfide, benzene, carbon tetrachloride or toluene.

147. The process of Claim 146 wherein the solvent is benzene.

148. The process of Claim 133 wherein separating the solid C₆₀ from the solvent comprises evaporating the solvent.

149. The process of Claim 133 further comprising the step of purifying the solid C₆₀.

150. The process of Claim 149 wherein the purifying step is sublimation, crystallization, column chromatography, capillary electrophoresis, HPLC, preparative thin-layer chromatography or extraction.

151. The process of Claim 134 wherein the C₆₀ is sublimed from the sooty carbon product at 300-400°C.

152. The process of Claim 134 wherein the sublimation step comprising heating the C₆₀ is a vacuum or inert atmosphere at effective sublimation temperatures to extract C₆₀ from said sooty carbon product.

153. A process for preparing C₆₀ comprising:

(a) vaporizing elemental carbon in the presence of an inert quenching gas under conditions effective to form a sooty carbon product comprising C₆₀ molecules, said C₆₀ molecules being present in said sooty carbon product in amounts sufficient to be capable of providing a visibly colored solution when extracted with sufficient amounts of benzene; and

(b) extracting C₆₀ from said sooty carbon product in amounts sufficient to provide a visibly colored solution when extracted with benzene in amounts sufficient to dissolve the C₆₀ present in said sooty carbon product.

154. A process for preparing C₆₀ comprising:

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(a) vaporizing elemental carbon in the presence of an inert quenching gas under conditions effective to provide a sooty carbon product comprising C_{60} molecules, said C_{60} molecules being present in said sooty carbon product in amounts sufficient to be capable of providing a visibly colored solution when extracted with benzene;

(b) depositing the sooty carbon product on a collecting surface;

(c) removing the sooty carbon product from the collecting surface; and

(d) extracting product which is predominantly C_{60} from said sooty carbon product, said C_{60} being present in sufficient quantities to provide a visibly colored solution when extracted with benzene present in amounts sufficient to dissolve the C_{60} present in said sooty carbon product.

155. The process of Claim 116 wherein the depositing step comprises collecting the sooty carbon product on a collecting surface distanced 5-10 cm from said vaporization.

156. A process for preparing C_{60} comprising:

(a) vaporizing elemental carbon in the presence of an inert quenching gas under conditions effective to form a sooty carbon product comprising C_{60} molecules, said C_{60} molecules being present in said sooty carbon product in amounts capable of extracting therefrom said C_{60} in solid form; and

(b) extracting in solid form C_{60} from said sooty carbon product.

157. The process according to Claim 132 wherein extracting further comprises evaporating the solvent off, thereby forming a residue, and subliming the C_{60} from said residue.

158. A process for preparing C_{60} comprising:

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(a) vaporizing elemental carbon in the presence of an inert quenching gas at a pressure sufficient to generate a sooty carbon product comprising C_{60} , said C_{60} being present in said sooty carbon product in sufficient amounts to produce and collect therefrom crystalline C_{60} ; and

(b) separating said C_{60} from said sooty carbon product under conditions effective to recover crystalline C_{60} .

159. The process according to Claim 158 wherein the pressure is less than 1 atmosphere.

160. The process according to Claim 159 wherein the pressure is at least about 50 torr.

161. The process according to Claim 160 wherein the pressure ranges from about 50 to about 400 torr.

162. The process according to Claim 158 wherein C_{70} is additionally present in the sooty carbon product.

163. The process of Claim 158 wherein C_{70} is additionally present in the sooty carbon product and is separated from the sooty carbon product and is additionally present in the crystalline C_{60} .

164. The process according to Claim 163 wherein said C_{70} is separated from the crystalline C_{60} .

165. The process according to Claim 158 wherein said crystalline C_{60} is substantially pure crystalline C_{60} .

166. The process according to Claim 158 wherein the elemental carbon is graphite, amorphous carbon or glassy carbon.

167. A process for preparing C_{60} comprising:

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(a) vaporizing elemental carbon to form carbon vapor in an atmosphere of an inert gas;

(b) quenching said carbon vapor in said inert gas under conditions sufficient to effectively condense and nucleate said vapor to form a sooty carbon product comprising C_{60} molecules in sufficient quantities to extract therefrom an amount sufficient to collect said C_{60} as a crystalline product;

(c) collecting said sooty carbon product; and

(d) separating said C_{60} from said sooty carbon product and recovering therefrom said C_{60} in crystalline form.

168. The process according to Claim 167 wherein the inert gas is helium or argon.

169. The process according to Claim 167 wherein the carbon vapor is quenched for a sufficient distance from the situs of vaporization to form said sooty carbon product.

170. The process according to Claim 167 wherein said distance is about 5 to 10 cm from the situs of vaporization.

171. The process according to Claim 167 wherein the C_{60} recovered therefrom is substantially pure crystalline C_{60} .

172. The process according to Claim 167 wherein C_{70} is additionally present in the sooty carbon product and is additionally present in the carbon product and is additionally present in the crystalline product.

173. The process according to Claim 167 wherein the C_{60} is separated from C_{70} .

174. The process according to Claim 167 wherein the quenching gas is at a pressure less than 1 atmosphere.

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175. The process according to claim 174 wherein the pressure ranges from about 50 torr to about 400 torr.

176. A process for producing C₆₀ comprising:

(a) vaporizing elemental carbon in an atmosphere of an inert gas at a pressure sufficient to generate a sooty carbon product comprising C₆₀; said C₆₀ being present in sufficient quantities to recover therefrom C₆₀ in amounts to be discernible as a colored solid; and

(b) separating said C₆₀ from said sooty carbon product under conditions effective to recover therefrom a colored crystalline C₆₀.

177. The process according to Claim 176 wherein the pressure is less than 1 atmosphere pressure.

178. The process according to Claim 177 wherein the pressure is greater than about 50 torr.

179. The process according to Claim 178 wherein the pressure ranges from about 50 torr to about 400 torr.

180. The process according to Claim 176 wherein C₇₀ is additionally present in the sooty carbon product.

181. The process according to Claim 176 wherein C₇₀ is additionally present in the sooty carbon product and is additionally separated from the sooty carbon product and is present in the crystalline C₆₀.

182. The process according to Claim 181 wherein the C₇₀ is separated from the crystalline C₆₀.

183. The process according to Claim 176 wherein the crystalline C₆₀ is substantially pure crystalline C₆₀.

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184. A process for preparing C₆₀ comprising vaporizing elemental carbon selected from the group consisting of graphite, amorphous carbon and glassy carbon in an inert quenching gas at a pressure of at least 50 torr so as to generate a carbon soot comprising C₆₀ and separating said C₆₀ from said soot under conditions effective to recover substantially pure crystalline C₆₀ therefrom.

185. The process according to Claim 184 wherein the pressure ranges from about 50 torr to about 400 torr.

186. A process for preparing C₆₀ comprising:

- (a) vaporizing elemental carbon in the presence of an inert quenching gas under conditions effective to form a sooty carbon product comprising C₆₀ molecules in macroscopic amounts;
- (b) depositing the sooty carbon product on a collecting surface remote from said situs of vaporization;
- (c) removing the sooty carbon product from the collecting surface; and
- (d) extracting a product comprising a macroscopic amount of C₆₀ from said sooty carbon product.

187. A process for preparing C₆₀ comprising:

- (a) vaporizing elemental carbon in the presence of an inert quenching gas at a pressure ranging from less than 1 atmosphere up to a pressure of 10 atmospheres under conditions effective to form a sooty carbon product comprising C₆₀ in quantities sufficient to isolate C₆₀ as a solid when extracted from the sooty carbon product;
- (b) depositing the sooty carbon product on a collecting surface remote from said situs of vaporization;

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(c) removing the sooty carbon product from the collecting surface; and

(d) extracting C_{60} from said sooty carbon product in quantities sufficient to isolate C_{60} as a solid when extracted from the sooty carbon product.

188. A process for preparing C_{60} comprising:

(a) vaporizing elemental carbon in the presence of an inert quenching gas under a pressure ranging from less than 1 atmosphere up to 10 atmospheres under conditions effective to form a sooty carbon product comprising C_{60} in quantities sufficient to isolate C_{60} as a discernible solid when extracted from the sooty carbon product; and

(b) extracting C_{60} from said sooty carbon product in quantities sufficient to isolate said C_{60} as a discernible solid.

189. The process according to Claim 187 or 188 wherein said elemental carbon is placed into an evacuated reactor prior to the vaporization thereof.

190. The process according to Claim 187 wherein depositing comprises collecting the sooty carbon product on a collecting surface distanced 5-10 cm from said vaporization situs.

191. The process according to Claim 187 or 188 wherein the elemental carbon is vaporized in a bell jar carbon evaporator.

192. The process according to Claim 187 or 188 wherein extracting comprises contacting the sooty carbon product with a non-polar organic solvent effective to dissolve the C_{60} , said solvent being present in amounts sufficient to dissolve the C_{60} present in said sooty carbon product.

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193. The process according to Claim 192 wherein said non-polar organic solvent is benzene, toluene, carbontetrachloride, 1,1,1-trichloroethane, xylene or an alkane having 5-10 carbon atoms.

194. The process according to Claim 187 or 188 wherein C_{70} is additionally present in the extracted C_{60} .

195. The process of Claim 194 which additionally comprises separating the C_{70} from C_{60} .

196. The process according to Claim 187 or 188 which additionally comprises purifying the extracted C_{60} product.

197. The process according to Claim 187 or 188 wherein the product is substantially pure C_{60} .

198. The process according to Claim 187 or 188 wherein extracting comprises subliming the C_{60} from the sooty carbon product and condensing the sublimed C_{60} .

199. The process according to Claim 187 or 188 wherein the pressure of the vaporization is conducted at a pressure ranging from 50 to 400 torr.

200. The process according to Claim 187 or 188 wherein the extracted product is in solution.

201. The process according to Claim 187 or 188 wherein said extracted C_{60} product is in solution of a non-polar organic solvent.

202. The process according to Claim 200 wherein the non-polar organic solvent is benzene, toluene, carbon tetrachloride, 1,1,1-trichloroethane, xylene or an alkane having 5-10 carbon atoms.

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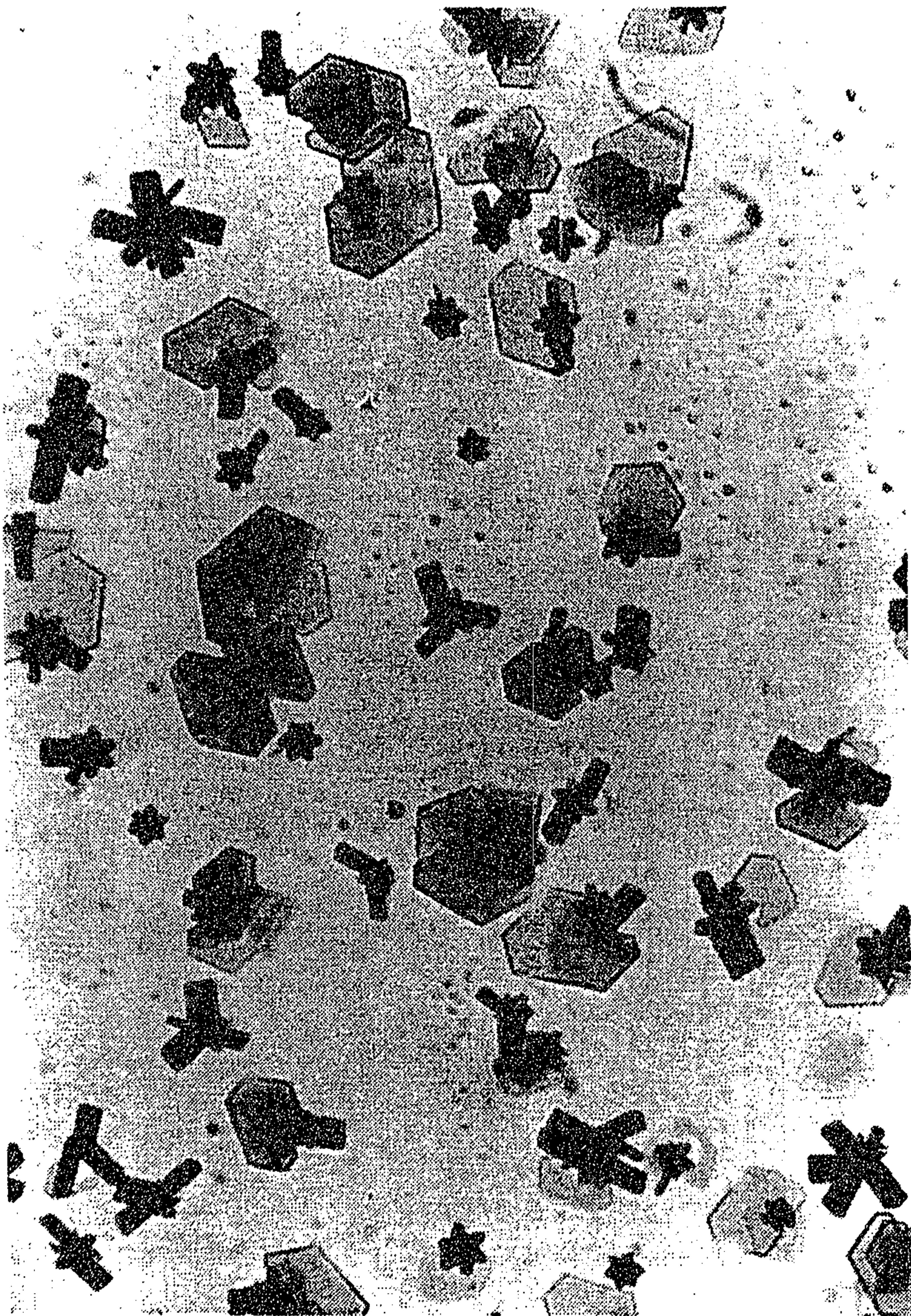


FIG.1

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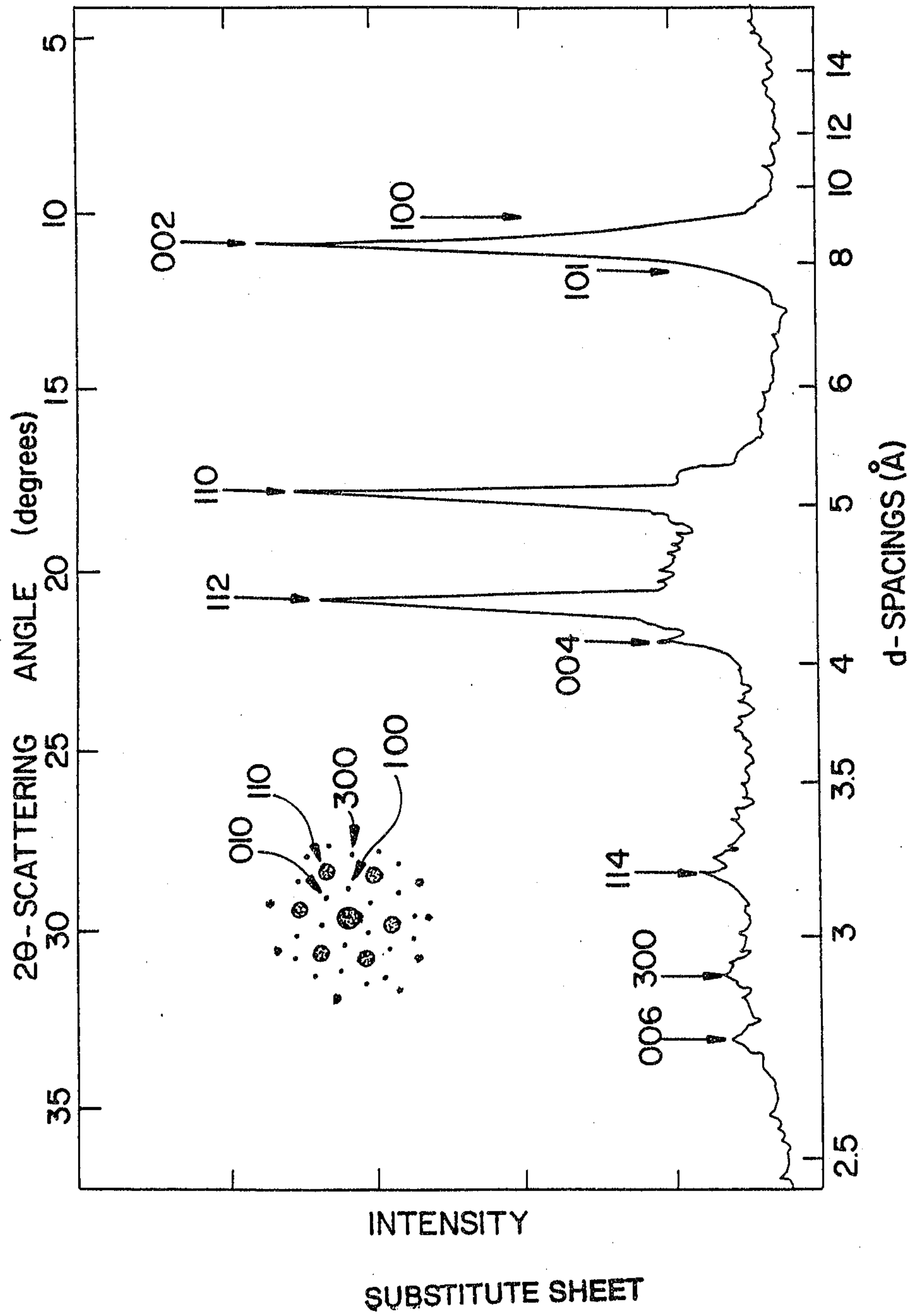


FIG. 2

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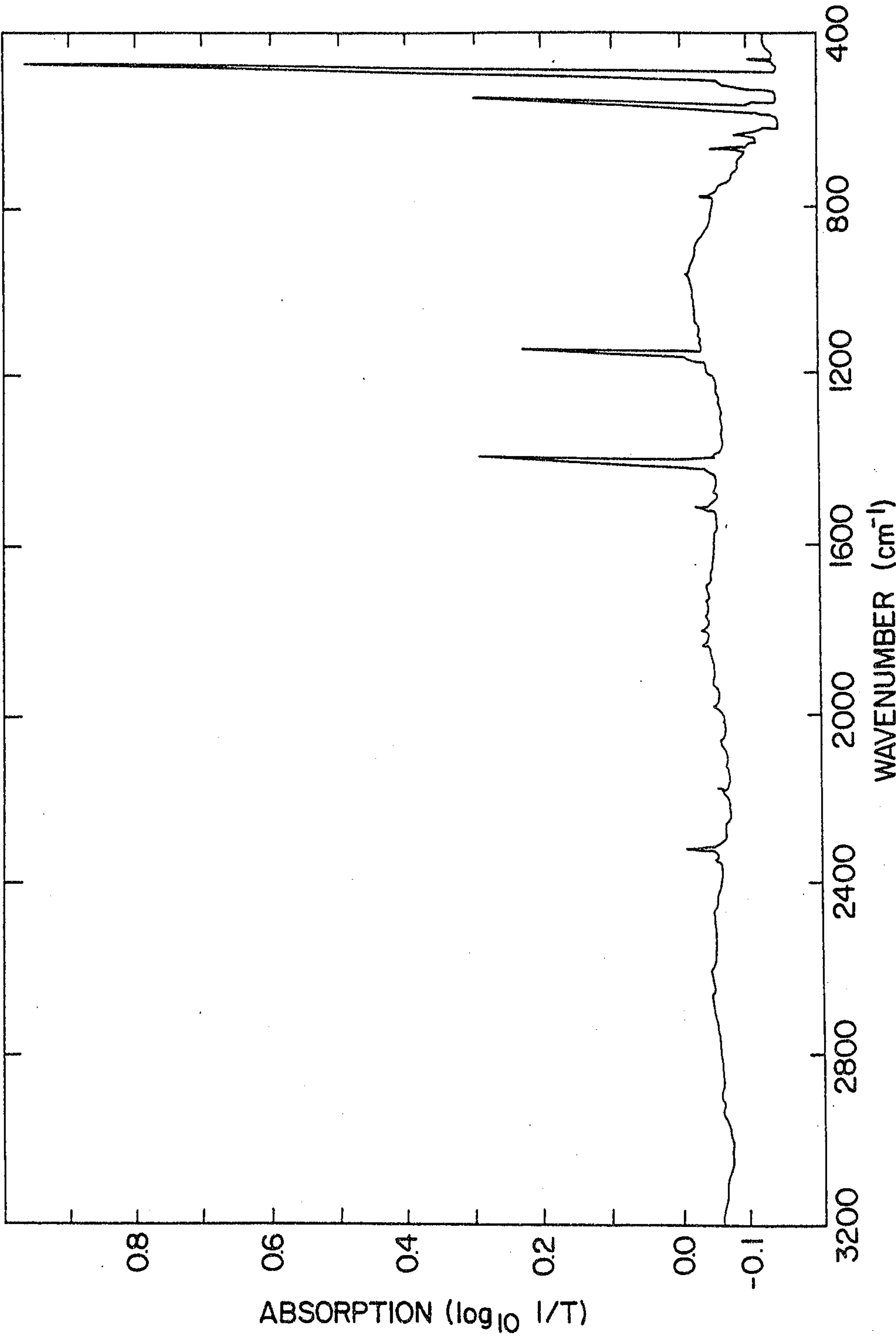


FIG. 3

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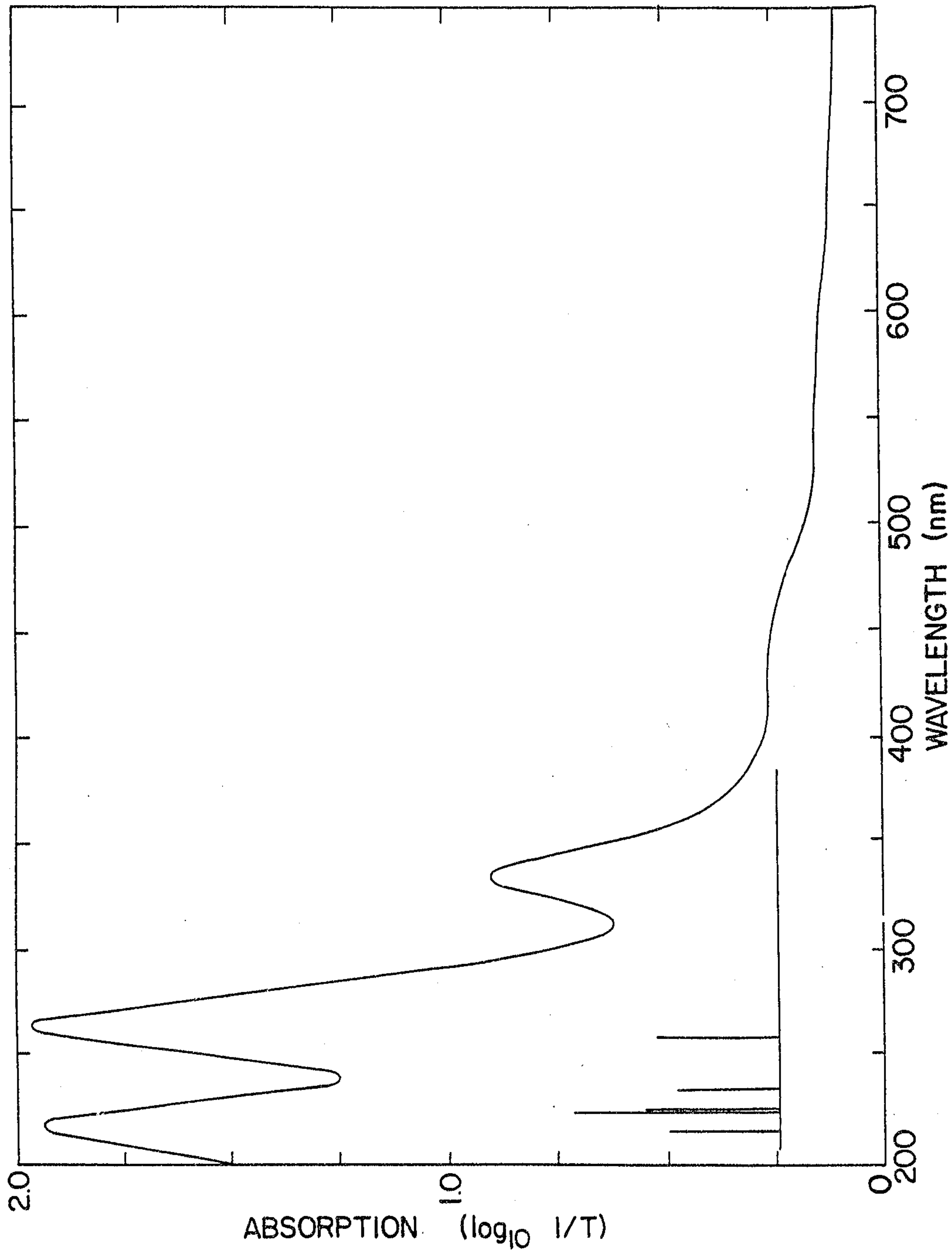


FIG. 4

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