

# Supporting Information for

## A Study on the Origin of Radical in Fullerene and Graphene

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## 1. HPLC results of fullerenes

### 1.1 HPLC results of C<sub>60</sub> from TCI with purity of 99.5%



#### Certificate of Analysis

Jun 5, 2017 (JST)

TOKYO CHEMICAL INDUSTRY CO., LTD.  
4-10-1 Nihonbashi-Honcho, Chuo-ku, Tokyo 103-0023 Japan

Chemical Name: Fullerene C60 (pure)		
Product Number: B1641 CAS: 99685-96-8	Lot: L5LYB	
Tests	Results	Specifications
Purity(HPLC)	99.9 area%	min. 99.5 area%

TCI Lot numbers are 4-5 characters in length. Characters listed after the first 4-5 characters are control numbers for internal purpose only.  
The contents of the specifications are subject to change without advance notice. The specification values displayed here are the most up to date values. There may be cases where the product labels display a different specification, however, the product quality still meets the latest specification.

#### Customer service:

TCI (Shanghai) Development Co., Ltd.  
Tel: 021-67121386  
Fax: 021-67121385  
E-mail: Sales-CN@TCIchemicals.com

**Figure S1.** HPLC data for purity of C<sub>60</sub> from TCI.

### 1.2 HPLC results of C<sub>60</sub> from TCI with purity of 99.0%



#### Certificate of Analysis

Jun 5, 2017 (JST)

TOKYO CHEMICAL INDUSTRY CO., LTD.  
4-10-1 Nihonbashi-Honcho, Chuo-ku, Tokyo 103-0023 Japan

Chemical Name: Fullerene C60		
Product Number: B1660 CAS: 99685-96-8	Lot: GG4CL	
Tests	Results	Specifications
Purity(HPLC)	100.0 area%	min. 99.0 area%
Absorbance(E1%1cm)	772	min. 750(335.5nm)

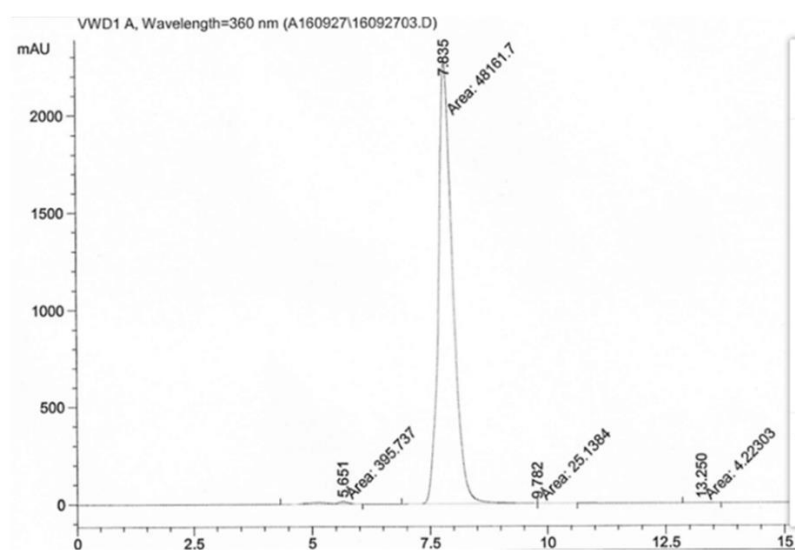
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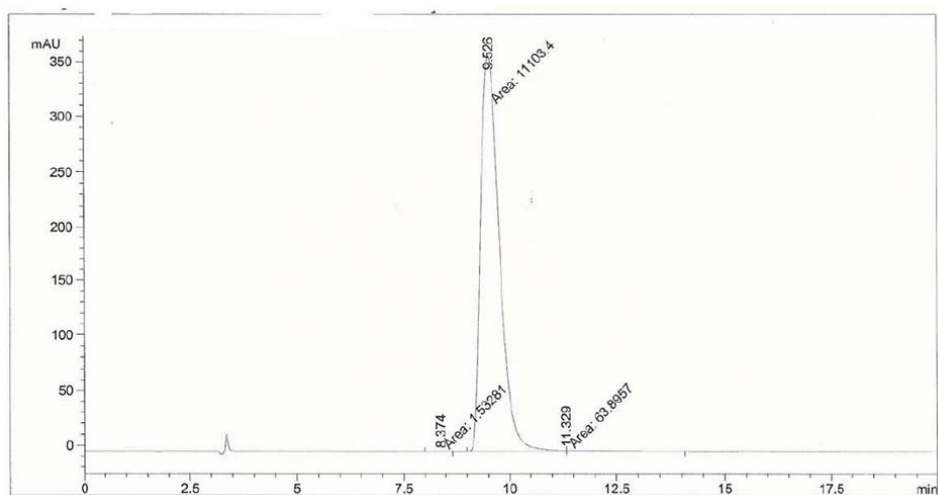
**Figure S2.** HPLC data for purity of C<sub>60</sub> from TCI.

### 1.3 HPLC results of PC<sub>61</sub>BM from Borun Company Limited



**Figure S3.** HPLC of PC<sub>61</sub>BM

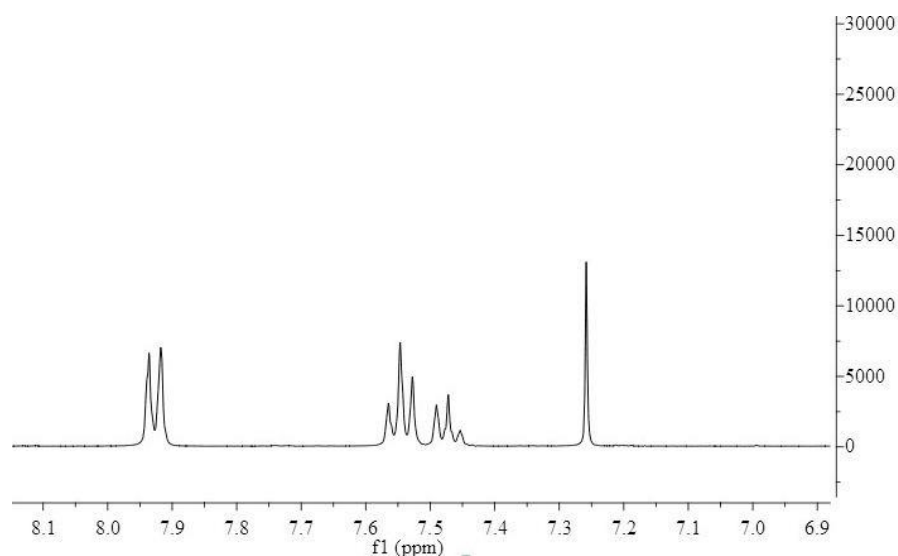
### 1.4 HPLC results of PC<sub>71</sub>BM from Borun Company Limited



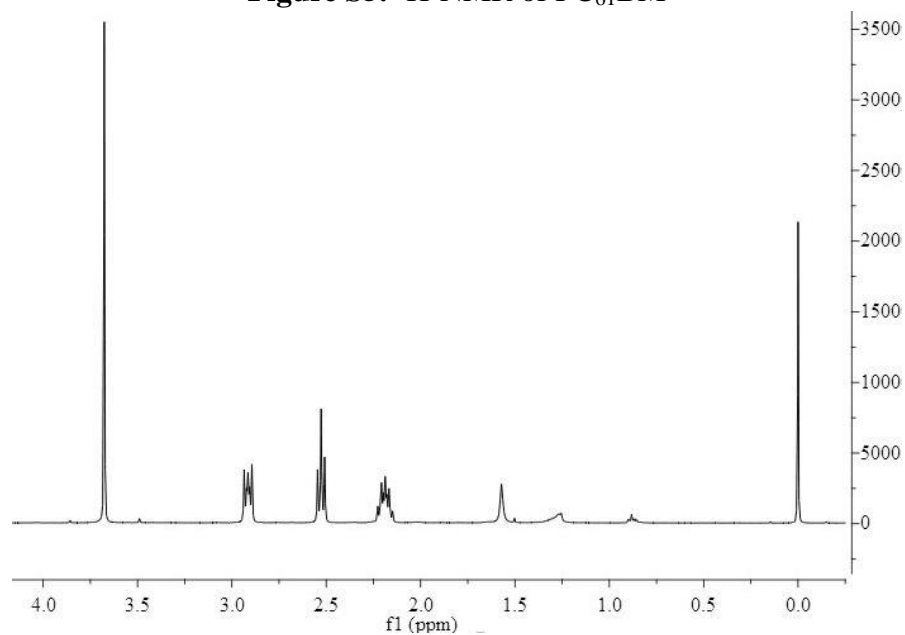
**Figure S4.** HPLC of PC<sub>71</sub>BM

## 2. $^1\text{H}$ -NMR results of fullerenes

### 2.1 $^1\text{H}$ -NMR results of $\text{PC}_{61}\text{BM}$

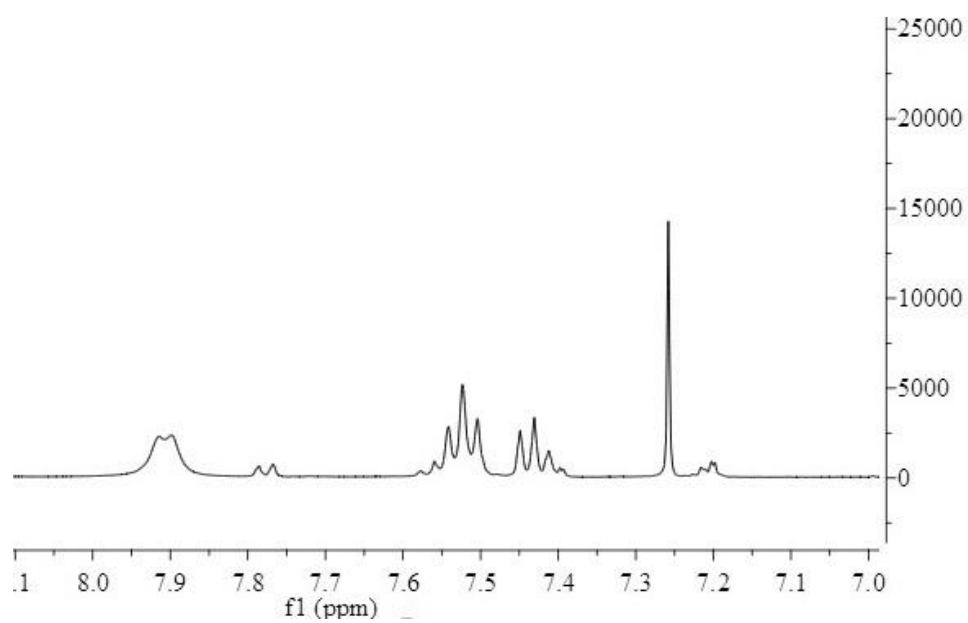


**Figure S5.**  $^1\text{H}$ -NMR of  $\text{PC}_{61}\text{BM}$

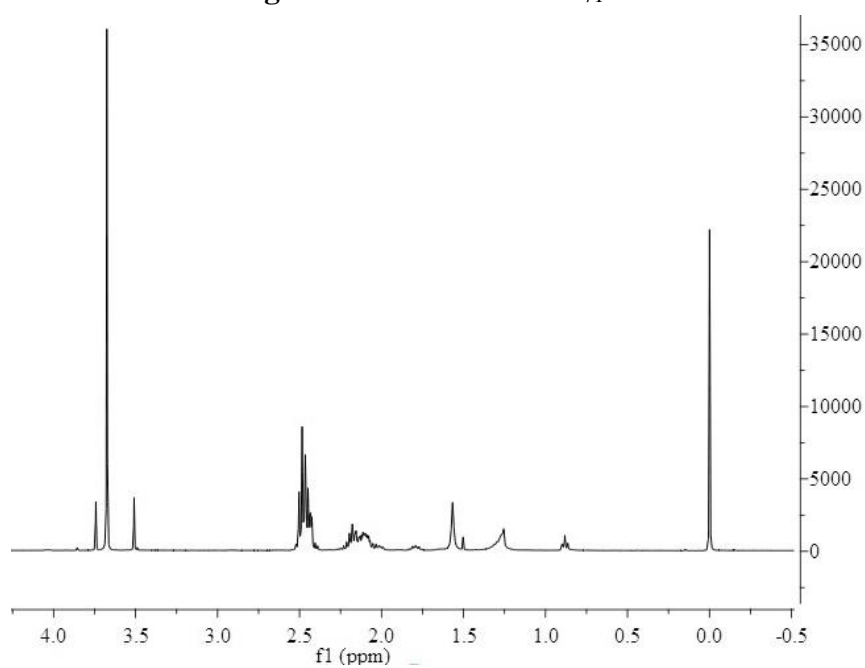


**Figure S6.**  $^1\text{H}$ -NMR of  $\text{PC}_{61}\text{BM}$

## 2.2 $^1\text{H}$ -NMR results of $\text{PC}_{71}\text{BM}$

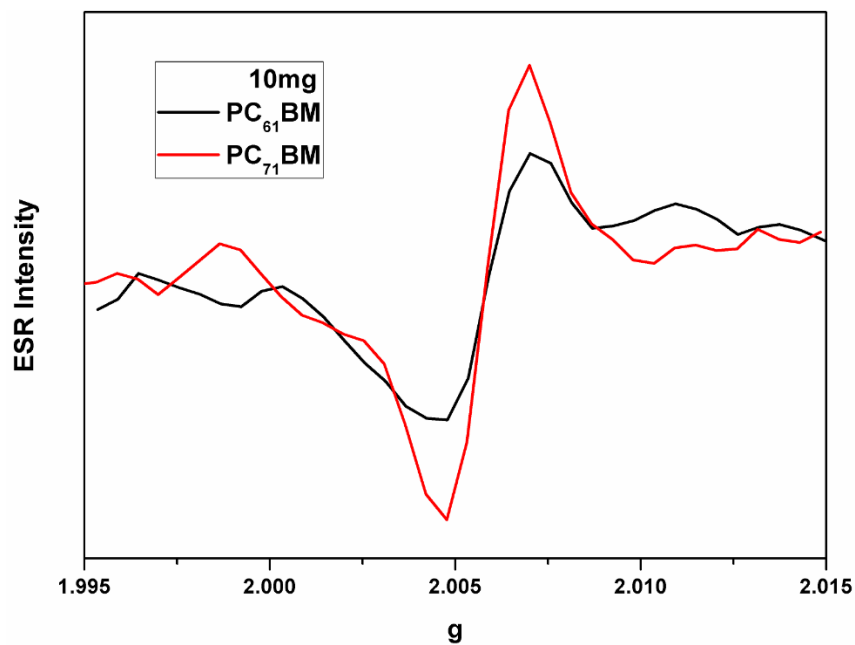


**Figure S7.**  $^1\text{H}$ -NMR of  $\text{PC}_{71}\text{BM}$

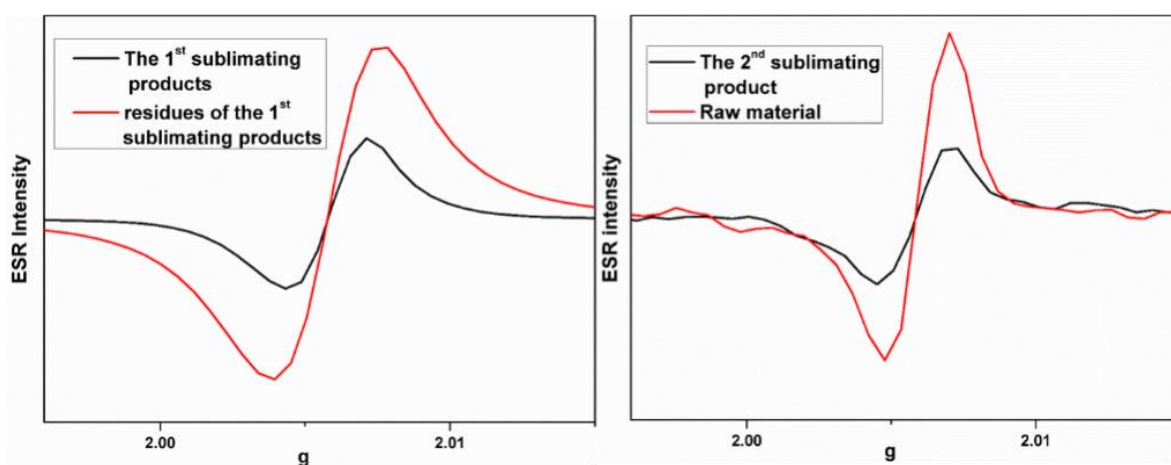


**Figure S8.**  $^1\text{H}$ -NMR of  $\text{PC}_{71}\text{BM}$

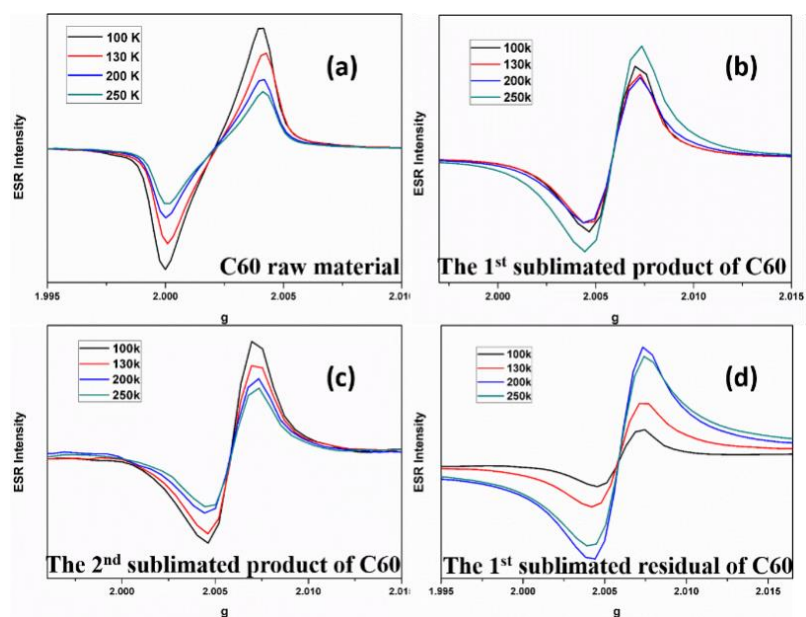
### 3. Electron Spin Resonance (ESR) results of fullerenes



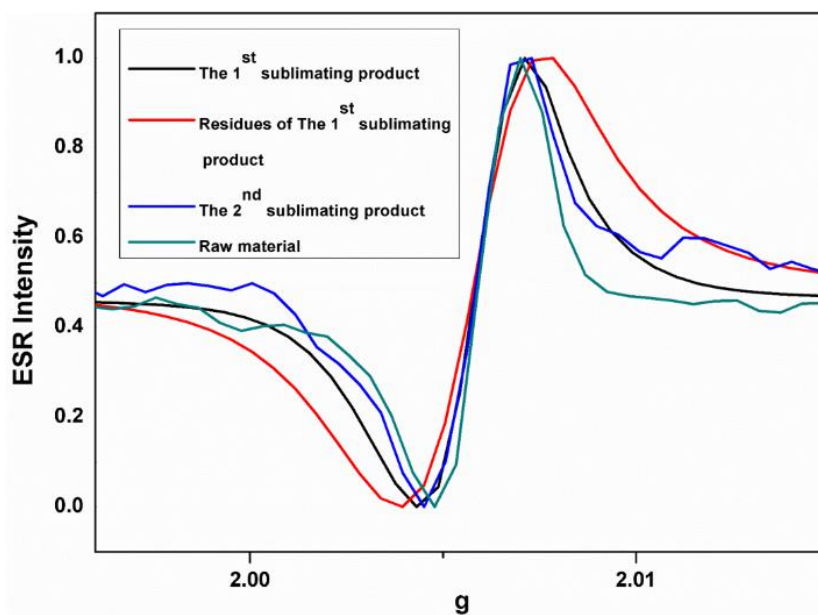
**Figure S9.** ESR spectra of PC<sub>61</sub>BM and PC<sub>71</sub>BM with 10 mg of each sample.



**Figure S10.** ESR spectra of C<sub>60</sub> raw material, sublimated product and residual of C<sub>60</sub> raw material in Ar (weight 33mg)

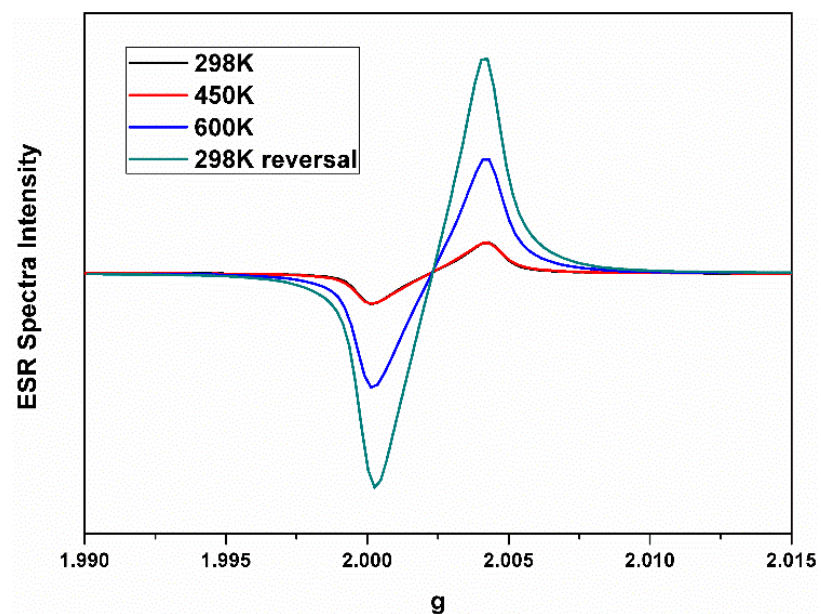


**Figure S11.** VT-ESR of C<sub>60</sub> raw material, the sublimated product and residual

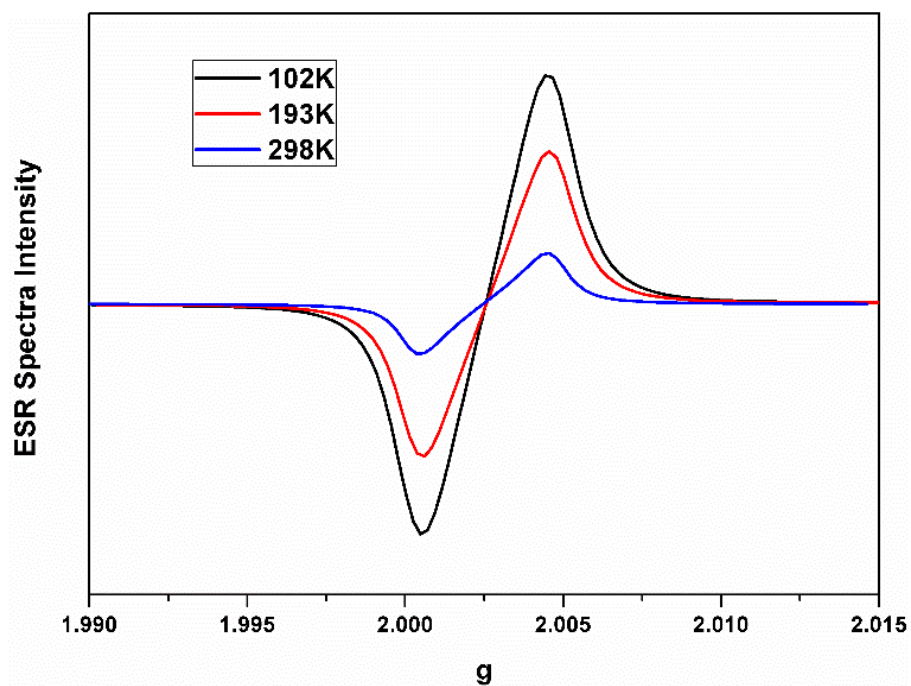


**Figure S12.** Normalized ESR spectra of C<sub>60</sub> raw material, sublimated product and residual of C<sub>60</sub> raw material in Ar.

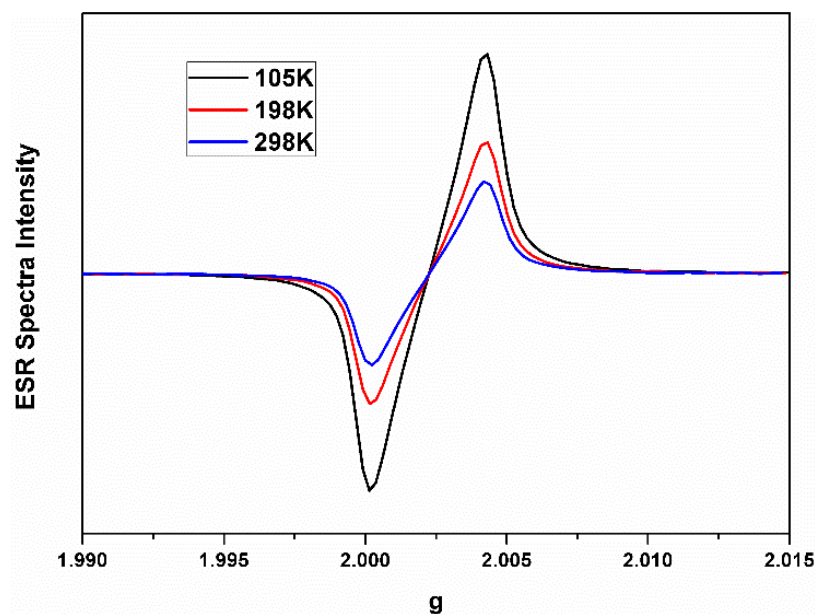




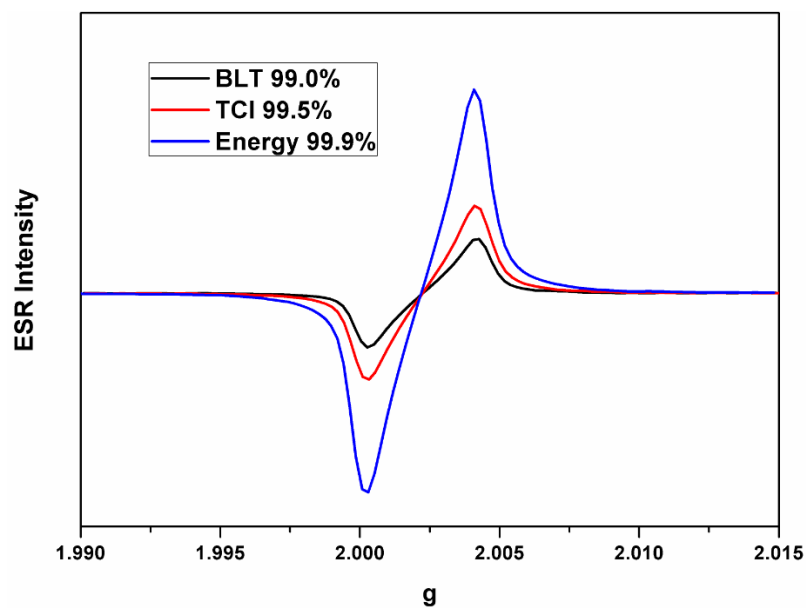
**Figure S13.** Variable temperature ESR spectra of C<sub>60</sub> (containing self-polymerized C<sub>60</sub>) in air (Purity 99.9%; weight 33mg)



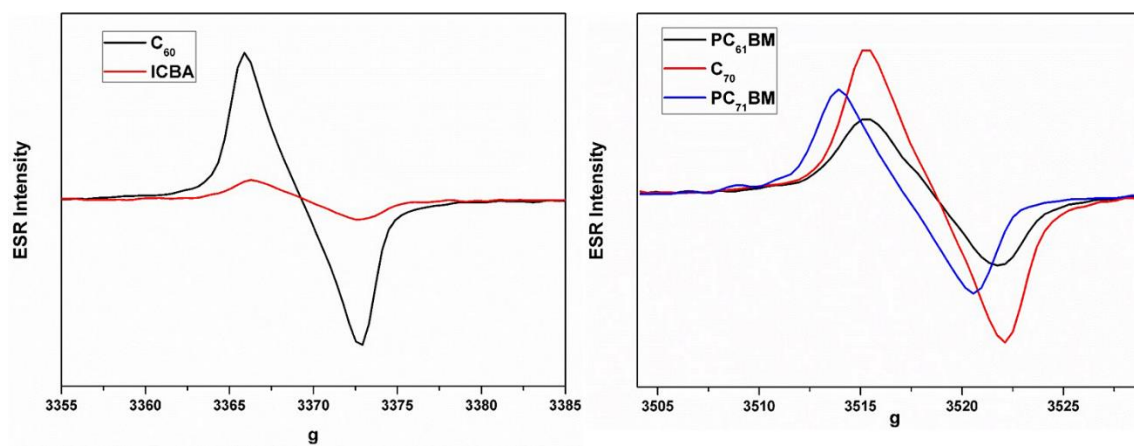
**Figure S14.** Variable temperature ESR spectra of oxidized graphene in air (1mg) with intrinsic radical.



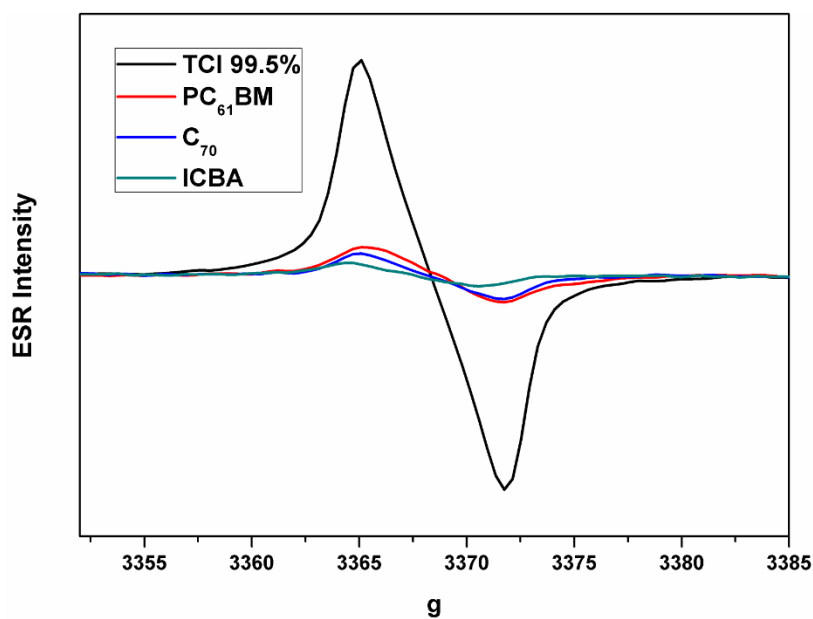
**Figure S15.** Variable temperature ESR spectra of TCI C<sub>60</sub> in Ar containing self-polymerized C<sub>60</sub> (weight 33mg).



**Figure S16.** ESR spectra of C<sub>60</sub> (containing self-polymerized C<sub>60</sub>) from different suppliers with the weight of 33mg at 298K.



**Figure S17.** ESR spectra of fullerene derivatives with 0.046 mmol in air of each sample (containing self-polymerized  $C_{60}$ ).

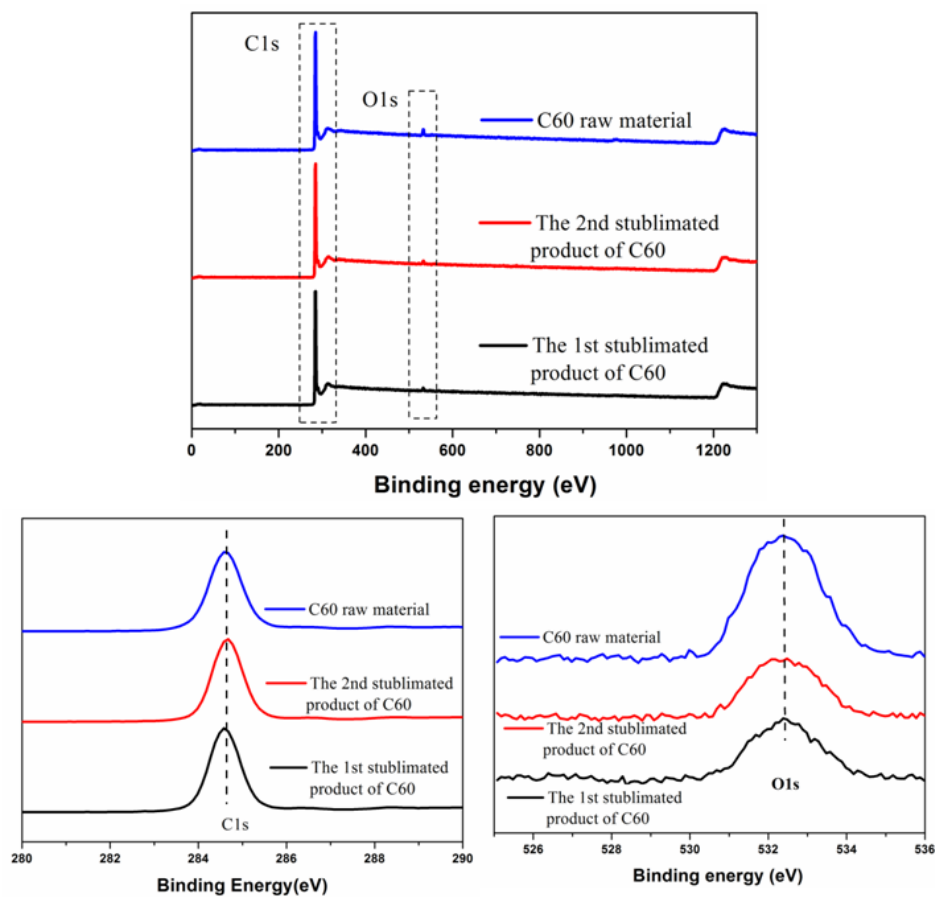


**Figure S18.** ESR spectra of fullerene derivatives with 0.046 mmol of each sample in Ar (containing self-polymerized  $C_{60}$ ).

**Table S1** Different field of each fullerene derivative sample in ESR spectra.

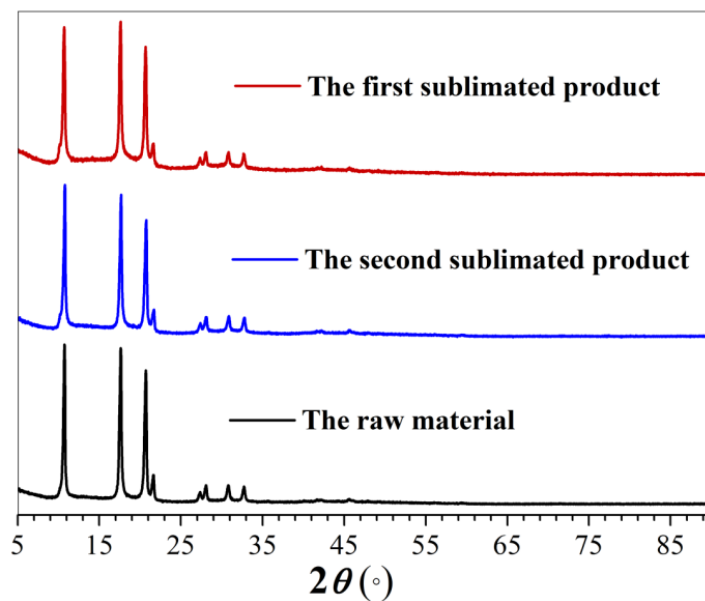
Fullerene derivatives	Different field of each fullerene derivative sample
$C_{60}$	3365.884
PC <sub>61</sub> BM	3515.478
$C_{70}$	3515.088
PC <sub>71</sub> BM	3513.916
ICBA	3366.275

#### 4. X-ray photoelectron spectroscopy (XPS) results of fullerenes



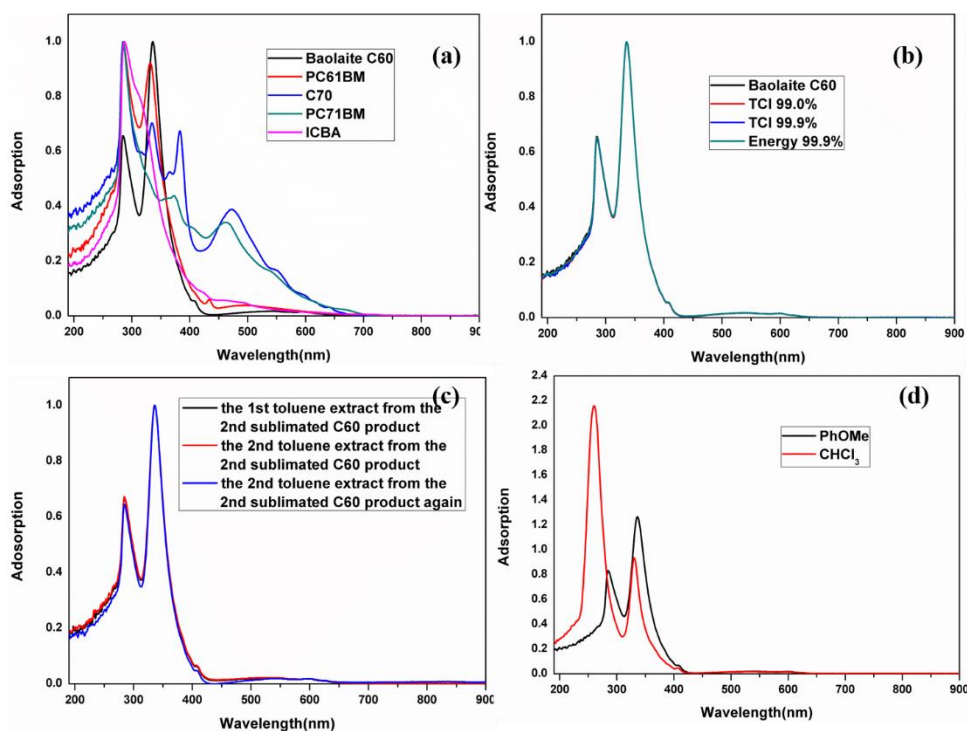
**Figure S19.** XPS spectra of C<sub>60</sub> raw material, sublimated product by sublimation of C<sub>60</sub> raw material

## 5. X-ray diffraction (XRD) results of fullerenes



**Figure S20.** XRD spectra of  $C_{60}$  raw material, the first sublimated product and the second sublimated product by sublimation of  $C_{60}$  raw material (all three samples contain self-polymerized  $C_{60}$ ).

## 6. UV-vis absorption spectra results of fullerenes and derivatives



**Figure S21.** The UV-vis absorption spectra of (a) fullerene derivatives in toluene, (b) raw materials from different company and purity in toluene, (c) the toluene extract from the 2<sup>nd</sup> sublimated C<sub>60</sub> in toluene and (d) raw material in toluene and CHCl<sub>3</sub> respectively. (C<sub>60</sub> contains none of self-polymerized C<sub>60</sub> as the polymerized C<sub>60</sub> is not soluble in toluene and it was removed from toluene solution for UV-vis absorption test).