Crystal and molecular structures of new fullerides, $(Ph_4P)_2C_{60}Hal$ (Hal = Br or I) and $(Ph_4As)_2C_{60}Cl$

V. V. Gritsenko, O. A. Dyachenko, G. V. Shilov, N. G. Spitsyna, and E. B. Yagubskii*

Institute of Chemical Physics in Chernogolovka, Russian Academy of Sciences,
142432 Chernogolovka, Moscow Region, Russian Federation.
Fax: 007 (096) 576 4009. E-mail: doa@icp.ac.ru

New air-stable fullerides, $(Ph_4P)_2C_{60}Hal$ (Hal = Br or I) and $(Ph_4As)_2C_{60}Cl$, were synthesized, and their crystal structure were determined. A comparative crystal-chemical analysis of the fullerides under study demonstrated that they are isostructural, described by the general formula $(Ph_4X)_2C_{60}Hal$ (X = P or As, and Hal = Cl, Br, or I), and crystallize in the triclinic system. The C_{60}^- and Hal^- anions occupy special centrosymmetric positions. The $(Ph_4P)^+$ and $(Ph_4As)^+$ cations occupy general positions. The principal parameters of the molecular structures are reported.

Key words: fullerides, synthesis, crystal and molecular structure,. X-ray structural analysis.

Radical-anionic salts of C_{60} fullerene are of great interest because of their unique conducting and magnetic properties. In particular, ferromagnets and superconductors were found among the mono- and trianionic salts of fullerene, respectively.[1—3] However, the dependences of the crystal structures of fullerides and their physical properties on the nature and structure of the cations have not been adequately investigated. Single crystals of only four fullerides have been studied by X-ray structural analysis.[4—7]

This is largely because the preparation of suitable single crystals presents difficulties, which arise primarily from their instability in air. However, monoanionic salts of C_{60} with $(Ph_4P)^+$ cations appeared to be rather stable.[4,5,8] As yet, no reasonable explanation has been provided for this exception. The first salt with this cation, $Ph_4PC_{60}(Ph_4PCl)_2$, was prepared by electrocrystallization. Its composition was determined only by elemental analysis.[8] More recently, crystals of fullerides of compositions $(Ph_4P)_2C_{60}I_{0.35}$ [4] and $(Ph_4P)_2C_{60}Cl$ [5] were prepared almost contemporaneously according to an analogous procedure with the use of the Ph_4PI and Ph_4PCl electrolytes, respectively. In this work, we synthesized new air-stable fullerides $(Ph_4P)_2C_{60}Br$ (**1**), $(Ph_4P)_2C_{60}I$ (**2**), and $(Ph_4As)_2C_{60}Cl$ (**3**). Their compositions and structures were determined by X-ray structural analysis. It should be noted that previous attempts to prepare crystals of salts of C_{60} suitable for X-ray structural analysis by crystallization in the presence of the Ph_4PBr or Ph_4AsCl electrolytes were unsuccessful.[9]

Experimental

Synthesis of fullerides. Crystalline samples of **1—3** were prepared by electrocrystallization on a 1-mm disk platinum electrode in the direct current mode (**1**: I = 0.3 μA, dI = 0.5 μA cm^{-2}; **2**: I = 1 μA, dI = 3 μA cm^{-2}; and **3**: I = 1 μA, dI = 2 μA cm^{-2}) at 30 °C. Electrolysis was carried out in a solution of 1,2-dichlorobenzene, which contained C_{60} ($3.7 \cdot 10^{-4}$ mol L^{-1}) fullerene and Ph_4PBr, Ph_4PI, or Ph_4AsCl ($3 \cdot 10^{-3}$ mol L^{-1}), respectively. After 3—4 days, air-stable black irregularly shaped crystals formed on the cathode. The ESR spectra of compounds **1—3** have intense signals belonging to the C_{60} radical anion (one spin per molecule).

X-ray structural analysis. The principal data on fullerides **1—3**, including the crystallographic data, the procedures for the measurement of intensities of reflections and their processing, and the methods for the solution and refinement of the structures, are given in Table 1. X-ray absorption in the crystals of **1—3** was ignored. In our opinion, the high value of the *R* factor for compound **3** is due to the slight disorder of fullerene molecules. This is confirmed by the fact that in the difference Fourier synthesis, electron density peaks in the vicinity of the major positions of a number of atoms of C_{60} are observed. The regularity of the arrangement of the additional peaks is absent, which did not allow us to use these peaks in the determination of the structure in spite of the substantial decrease in the *R* factor in this case. The coordinates of nonhydrogen atoms are given in Table 2. The positions of H atoms calculated from geometric considerations and the geometric parameters of the structures may be obtained from the authors. All calculations were carried out on a PC AT-486 computer.

Fullerides **1—3** are isostructural and crystallize in the triclinic system. Initially, the structure of **1** was solved in the noncentrosymmetric space group $P1$. After analysis of the crystal packing, all subsequent calculations were performed in the nonstandard centrosymmetric space group $C\bar{1}$.

Results and Discussion

The crystal structure of fulleride **1** consists of C_{60}^- and Br^- anions and $(Ph_4P)^+$ cations (Fig. 1). Because salts **1—3** are isostructural, fullerides **2** and **3** have similar structures with the only difference that in the

Translated from *Izvestiya Akademii Nauk. Seriya Khimicheskaya,* No. 11, pp. 1982—1986, November, 1997.

structure of **2**, the I^- anions occupy the positions of the Br^- anions, whereas in the structure of **3**, the Cl^- anions and the Ph_4As^+ cations occupy the positions of the Br^- and Ph_4P^+ ions, respectively. Taking into account the aforesaid and to avoid repetitions in the subsequent discussion, the general structural features of fullerides **1—3** will be exemplified by fulleride **1**.

In the structure of **1**, the C_{60}^- and Br^- anions occupy special positions. The C_{60} fullerene molecules are located at the midpoints of the *a* and *b* axes of the unit cell so that the centers of the molecules coincide with the inversion centers (1/2, 0, 0) and (0, 1/2, 0). The Br^- anions occupy the vertices of the unit cells and the centers of the (001) faces, *i.e.*, the inversion centers (0, 0, 0) and (1/2, 1/2, 0). The position of the basis P atom of the $(Ph_4P)^+$ cation is close to (0, 1/4, 1/2). In the space group $C\bar{1}$ in which the structure was solved, this position is general and does not contain symmetry elements.

The average values of the lengths of the C—C single bonds of pentagons (d_{56}) and the C=C double bonds of hexagons (d_{66}) in the C_{60}^- anions of fullerides **1—3** are 1.49 and 1.34 Å (**1**), 1.51 and 1.33 Å (**2**), and 1.52 and 1.30 Å (**3**), respectively. The C_{60} fullerene molecules have the approximately spherical shape. The average radius of C_{60} (R_{av}) in the structure of **1** is 3.53 Å, and the degree of distortion of the sphere is characterized by the range between R_{min} = 3.49 Å and R_{max} = 3.56 Å.

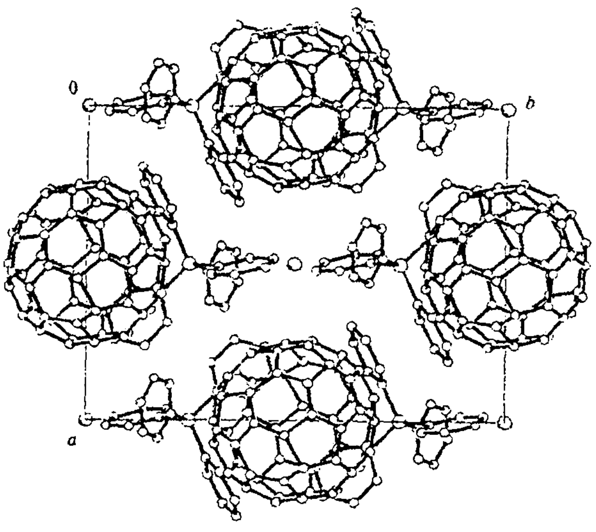

Fig. 1. Projection of the crystal structure of fulleride **1** onto the *ab* plane

Table 1. Principal crystallographic data, the procedures for the measurement of intensities of reflections and their processing, and the methods for the solution and refinement of the structures of **1—3**

Characteristics	**1**	**2**	**3**
Formula	$C_{108}H_{40}BrP_2$	$C_{108}H_{40}IP_2$	$C_{108}H_{40}As_2Cl$
M	1479.36	1526.36	1522.80
System	Triclinic	Triclinic	Triclinic
Space group	$C\bar{1}$	$C\bar{1}$	$C\bar{1}$
a/Å	17.757(5)	17.675(8)	17.830(5)
b/Å	17.766(5)	17.670(7)	17.827(4)
c/Å	13.518(4)	13.751(5)	13.537(4)
α/deg	89.97(2)	90.02(4)	89.99(2)
β/deg	131.03(4)	129.79(6)	131.16(4)
γ/deg	90.00(2)	90.01(3)	90.00(2)
$V/Å^3$	3217(2)	3299(2)	3239(2)
Z	2	2	2
$d_{calc}/g\ cm^{-3}$	1.527	1.536	1.560
F(000)	1506	1542	1542
Crystal size/mm	0.3×0.2×0.1	0.4×0.3×0.1	0.25×0.2×0.2
Scanning range/deg	$4.13<\theta<75.06$	$4.11<\theta<80.32$	$4.12<\theta<80.30$
Range of indices	−22—16, 0—21, 0—16	−22—17, −22—22, 0—13	−22—17, −14—22, 0—17
Number of measured reflection	3075	3907	4597
Number of independent reflections	3071 [*R*(int) = 0.0020]	3704 [*R*(int) = 0.0881]	4505 [*R*(int) = 0.0193]
Number of reflections with $I > 2\sigma(I)$	2442	2831	3304
GOF	0.652	0.776	3.779
R factor [$I > 2\sigma(I)$]	0.0579	0.0572	0.1406
R factor (for all reflections)	0.0751	0.0789	0.1762

Note. Measurements were carried out on an automated four-circle KM-4 diffractometer (KUMA DIFFRACTION, Poland); ω/2θ scanning technique; λ 1.5418. The structures were solved by the direct method (using the SHELX-86 program)[10] and refined by the full-matrix least-squares method based on F^2 (using the SHELXL-93 program).[11] The number of variable parameters was 662.

Table 2. Atomic coordinates in compounds **1** (Hal = Br and X = P), **2** (Hal = I and X = P), and **3** (Hal = Cl and X = As)

Atom	**1**			**2**			**3**		
	x	*y*	*z*	*x*	*y*	*z*	*x*	*y*	*z*
Hal	0	0	0	0	0	0	0	0	0
X	−0.0001(2)	0.2500(2)	0.5002(3)	0.0001(2)	0.2499(1)	0.5006(3)	0.0001(2)	0.2499(1)	0.5002(2)
C(1)	0.121(1)	0.468(1)	0.876(2)	0.1183(1)	0.463(1)	0.876(2)	0.121(3)	0.464(4)	0.881(4)
C(2)	0.022(1)	0.457(1)	0.766(2)	0.026(1)	0.457(1)	0.774(2)	0.020(3)	0.462(3)	0.766(4)
C(3)	−0.023(1)	0.389(1)	0.766(2)	−0.024(2)	0.387(1)	0.771(2)	−0.026(3)	0.389(2)	0.763(3)
C(4)	0.038(1)	0.344(1)	0.875(2)	0.041(1)	0.3452(9)	0.880(2)	0.045(3)	0.347(2)	0.881(3)
C(5)	0.136(1)	0.369(1)	0.978(2)	0.134(1)	0.369(1)	0.970(3)	0.134(5)	0.372(3)	0.973(7)
C(6)	0.184(1)	0.417(1)	1.007(2)	0.185(1)	0.411(2)	1.016(3)	0.183(4)	0.415(4)	1.027(7)
C(7)	−0.124(1)	0.399(1)	0.703(2)	−0.120(2)	0.400(1)	0.713(2)	0.121(4)	0.397(2)	0.707(3)
C(8)	−0.150(2)	0.476(2)	0.661(3)	−0.153(3)	0.474(3)	0.669(3)	0.159(6)	0.471(4)	0.657(4)
C(9)	−0.047(1)	0.521(1)	0.702(2)	−0.042(2)	0.521(2)	0.711(2)	−0.046(3)	0.521(3)	0.703(3)
C(10)	0.155(1)	0.540(1)	0.930(2)	0.158(1)	0.536(2)	0.935(2)	0.150(3)	0.538(3)	0.924(4)
C(11)	0.089(1)	0.603(1)	0.869(2)	0.088(1)	0.602(1)	0.866(2)	0.091(3)	0.599(3)	0.873(4)
C(12)	0.078(3)	0.670(2)	0.960(5)	0.079(3)	0.677(2)	0.966(5)	0.069(6)	0.670(4)	0.964(7)
C(13)	0.142(3)	0.656(2)	1.054(5)	0.146(3)	0.652(2)	1.038(5)	0.148(5)	0.649(4)	1.040(9)
C(14)	0.218(1)	0.602(1)	1.126(3)	0.216(1)	0.602(1)	1.118(3)	0.217(3)	0.603(3)	1.117(6)
C(15)	0.226(1)	0.540(1)	1.070(2)	0.224(1)	0.540(1)	1.061(3)	0.225(3)	0.541(3)	1.072(5)
C(16)	−0.009(1)	0.593(1)	0.761(2)	−0.006(1)	0.590(2)	0.766(2)	−0.008(2)	0.588(1)	0.765(3)
C(17)	0.243(1)	0.467(1)	1.123(2)	0.240(1)	0.464(2)	1.120(3)	0.239(3)	0.469(4)	1.111(4)
C(18)	0.230(1)	0.592(1)	1.239(2)	0.226(1)	0.592(2)	1.231(3)	0.229(2)	0.591(3)	1.243(4)
C(19)	0.249(1)	0.520(1)	1.297(3)	0.244(1)	0.521(2)	1.283(3)	0.250(3)	0.525(3)	1.285(5)
C(20)	0.258(1)	0.458(1)	1.236(3)	0.255(1)	0.456(2)	1.237(3)	0.257(2)	0.463(3)	1.246(6)
C(21)	0.213(1)	0.388(1)	1.243(2)	0.208(1)	0.387(1)	1.228(3)	0.210(3)	0.390(2)	1.236(4)
C(22)	0.172(1)	0.399(1)	1.295(2)	0.168(2)	0.397(2)	1.293(3)	0.173(3)	0.397(3)	1.298(4)
C(23)	0.191(2)	0.478(2)	1.343(3)	0.180(3)	0.473(3)	1.332(3)	0.179(6)	0.473(4)	1.337(4)
C(24)	−0.160(1)	0.363(1)	0.760(2)	−0.159(1)	0.362(1)	0.764(2)	−0.157(3)	0.365(2)	0.758(5)
C(25)	−0.095(1)	0.3180(9)	0.873(2)	−0.096(1)	0.3194(9)	0.873(2)	−0.085(4)	0.316(2)	0.894(5)
C(26)	0.003(1)	0.309(1)	0.928(2)	0.003(1)	0.3081(8)	0.934(2)	−0.004(4)	0.312(2)	0.918(5)
C(27)	0.075(1)	0.310(1)	1.068(2)	0.073(1)	0.3099(8)	1.064(2)	0.078(3)	0.312(2)	1.071(6)
C(28)	0.166(1)	0.346(1)	1.127(2)	0.165(1)	0.346(1)	1.122(2)	0.163(3)	0.347(2)	1.123(4)
C(29)	0.077(1)	0.361(1)	1.237(2)	0.076(2)	0.362(1)	1.233(3)	0.088(4)	0.368(3)	1.244(5)
C(30)	0.031(1)	0.318(1)	1.127(2)	0.024(1)	0.318(1)	1.119(3)	0.026(5)	0.316(2)	1.105(5)
C(31)	−0.0921(7)	0.3211(7)	0.399(1)	−0.0909(6)	0.3219(5)	0.402(1)	−0.102(2)	0.319(1)	0.394(2)
C(32)	−0.0734(8)	0.3954(8)	0.444(1)	−0.0725(7)	0.3959(6)	0.449(1)	−0.081(2)	0.399(1)	0.440(2)
C(33)	−0.1468(9)	0.4490(9)	0.372(2)	−0.1435(8)	0.4505(6)	0.378(1)	−0.152(2)	0.458(2)	0.374(3)
C(34)	−0.2390(9)	0.4306(9)	0.252(2)	−0.2314(8)	0.4340(7)	0.263(1)	−0.248(2)	0.429(2)	0.246(3)
C(35)	−0.2591(9)	0.3576(9)	0.207(2)	−0.2510(8)	0.3615(7)	0.215(1)	−0.262(2)	0.361(1)	0.200(2)
C(36)	−0.1856(8)	0.3029(8)	0.280(1)	−0.1815(7)	0.3052(6)	0.285(1)	−0.187(2)	0.309(1)	0.273(2)
C(37)	−0.0080(8)	0.1787(8)	0.399(1)	−0.0064(6)	0.1776(5)	0.403(1)	−0.004(2)	0.181(1)	0.389(2)
C(38)	0.019(1)	0.105(1)	0.445(2)	0.0220(9)	0.1043(7)	0.451(1)	0.023(2)	0.103(1)	0.440(2)
C(39)	0.019(1)	0.050(1)	0.372(2)	0.0233(9)	0.0492(7)	0.380(2)	0.024(2)	0.043(2)	0.374(3)
C(40)	−0.0094(9)	0.069(1)	0.253(2)	−0.0083(9)	0.0671(7)	0.261(1)	−0.007(2)	0.071(2)	0.240(3)
C(41)	−0.034(1)	0.142(1)	0.206(2)	−0.0337(9)	0.1393(8)	0.215(1)	−0.037(2)	0.139(1)	0.199(2)
C(42)	−0.0351(8)	0.197(1)	0.280(1)	−0.0337(7)	0.1952(7)	0.287(1)	−0.040(2)	0.192(1)	0.271(2)
C(43)	0.1213(7)	0.2920(7)	0.600(1)	0.1203(6)	0.2913(5)	0.598(1)	0.124(2)	0.300(1)	0.609(2)
C(44)	0.1727(8)	0.2965(9)	0.555(1)	0.1716(8)	0.2968(7)	0.552(1)	0.179(2)	0.302(2)	0.558(3)
C(45)	0.2638(9)	0.332(1)	0.629(1)	0.2609(8)	0.3330(9)	0.620(1)	0.270(2)	0.338(2)	0.625(3)
C(46)	0.3040(8)	0.3656(8)	0.746(1)	0.3029(8)	0.3625(7)	0.739(1)	0.306(2)	0.371(2)	0.756(4)
C(47)	0.2537(9)	0.3614(9)	0.792(1)	0.2532(7)	0.3579(7)	0.785(1)	0.256(2)	0.363(1)	0.799(2)
C(48)	0.1634(8)	0.3253(8)	0.721(1)	0.1626(7)	0.3235(7)	0.715(1)	0.173(2)	0.324(2)	0.727(2)
C(49)	−0.0206(7)	0.2076(7)	0.602(1)	−0.0225(6)	0.2073(5)	0.599(1)	−0.016(2)	0.201(1)	0.613(2)
C(50)	−0.1170(9)	0.203(1)	0.557(1)	−0.1207(8)	0.2022(7)	0.551(1)	−0.120(2)	0.198(2)	0.559(2)
C(51)	−0.135(1)	0.166(1)	0.629(2)	−0.1392(10)	0.1653(9)	0.624(1)	−0.145(2)	0.161(2)	0.628(3)
C(52)	−0.057(1)	0.135(1)	0.748(1)	−0.0633(10)	0.1372(8)	0.738(1)	−0.051(3)	0.129(2)	0.756(3)
C(53)	0.038(1)	0.139(1)	0.790(2)	0.0302(9)	0.1407(7)	0.784(1)	0.039(2)	0.139(1)	0.797(2)
C(54)	0.0577(9)	0.1750(9)	0.720(1)	0.0518(8)	0.1760(6)	0.715(1)	0.058(2)	0.176(2)	0.730(2)

The corresponding parameters of C_{60} in the structure of 2 are 3.53, 3.49, and 3.61 Å, respectively. In the structure of 3, these parameters are 3.49, 3.43, and 3.56 Å, respectively. The distances between the centers of the adjacent fullerene molecules located on the *a* and *b* axes are 12.559 (**1**), 12.495 (**2**), and 12.606 Å (**3**).

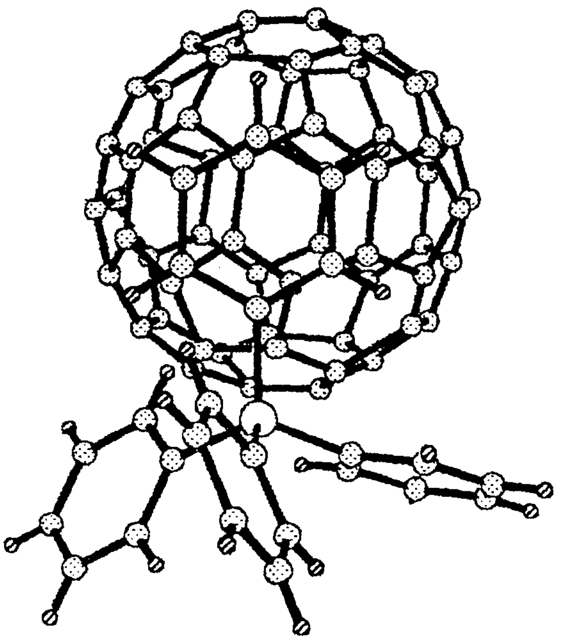

Fig. 2. View of the C_{60}^- anion along the normal to the plane of the phenyl ring of the $(Ph_4X)^+$ cation

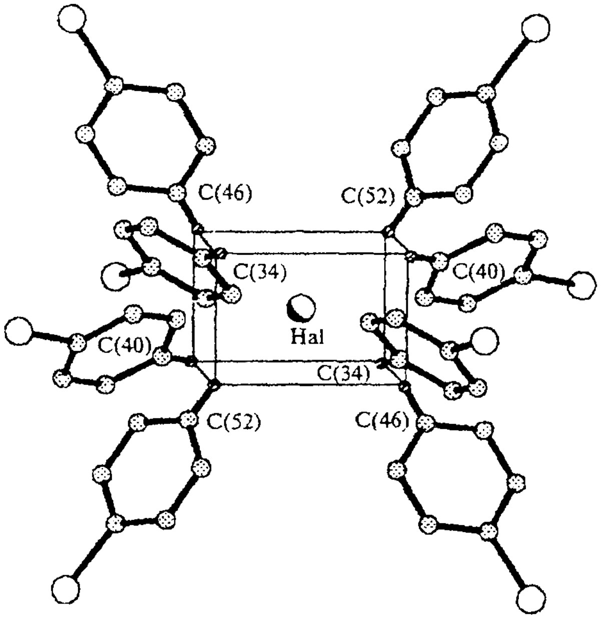

Fig. 3. Environment about the Hal^- anion in the structures of **1**—**3**

Table 3. Parameters and volumes of the unit cells of fullerides **1**, **2**, **4**, and **5** (for convenience of comparison, the unit cell parameters of **1** and **2** are given in the tetragonal system)

Compound	*a*,*b*/Å	*c*/Å	*V*/Å^3
$(PhP)_2C_{60}Br$ (**1**)	12.559(2)	20.388(4)	3215(2)
$(PhP)_2C_{60}I$ (**2**)	12.496(8)	21.131(5)	3299(2)
$(PhP)_2C_{60}Cl$(**4**)^5	12.5731(9)	20.1415(25)	3184.0
$(PhP)_2C_{60}I_{0.35}$(**5**)^4	12.588(2)	20.134(2)	3190.5(7)

The C_{60}^- fullerene anions are surrounded by eight phenyl rings of the $[Ph_4P]^+$ or $[Ph_4As]^+$ cations so that the planes of the phenyl rings are projected onto the C=C double bonds of the C_{60} fullerene molecules with the excessive electron density (Fig. 2). There are no shortened intermolecular contacts between the $[Ph_4P]^+$ and $[Ph_4As]^+$ cations and the C_{60}^-, Br^-, I^-, and Cl^- anions. In the structures of **1**—**3**, the halogen atoms are surrounded by eight H atoms of the tetraphenylphosphonium or tetraphenylarsonium groups, which form slightly distorted tetragonal prisms (Fig. 3).

Analysis of the structural features of fullerides **1**—**3** and those of the $(Ph_4P)_2C_{60}Cl$ (**4**)^5 and $(Ph_4P)_2C_{60}I_{0.35}$ (**5**)^4 compounds reported previously showed that all the salts are described by the general formula $(Ph_4X)_2C_{60}Hal$

	1	**2**	**3**	**4**	**5**
X	P	P	As	P	P
Hal	Br	I	Cl	Cl	$I_{0.35}$

and belong to the same structural type. This conclusion is confirmed by the crystal-chemical changes in the unit cell parameters of fullerides **4**, **1**, and **2** in which halides are replaced in the order Cl→Br→I. These parameters are given in Table 3. For convenience of comparison with the published data,^{4,5} the unit cell parameters of fullerides **1** and **2** are given in the tetragonal system. As can be seen from Table 3, when the halogen atoms are replaced in the order Cl→Br→I, the expected increase in the volume (V) of the unit cell in the direction **4**→**1**→**2** occurs as a result of the increase in the c parameter against the background of the slight decrease in the a and b parameters. It should be noted that the a, b, and c unit cell parameters and the volume V of fulleride **5**, unlike the parameters of fulleride **2**, are inconsistent with the observed tendency of these parameters to change in the order Cl→Br→I, but they virtually coincide with the corresponding parameters of fulleride **4**. On the one hand, the virtually identical unit cells of fullerides **4** and **5** and on the other hand, the identical electron scattering abilities of the $I_{0.35}$ atom and the Cl^- ion (in both cases, they are equal to 18) suggest that fulleride **5** is chloride of composition **4** rather than iodide.*

It should be also noted that, in our opinion, the triclinic space group $C\bar{1}$ (within the framework of which the structures of **1**—**3** were solved) is more adequate to

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* When this paper was in press, our suggestion that the composition of fulleride **5** may be in error, which we have made exclusively based on the results of a comparative crystal-chemical analysis of fullerides **1**, **2**, **4**, and **5**, was confirmed by the data reported in Ref. 14. The authors believed that fulleride **5** is a mixed radical-anionic salt of composition $[Ph_4P]^+C_{60}^{\dot{-}}Cl_{1-x}I_x$ (x = 0.10—0.15) rather than a system with a deficiency of iodine.

fullerides **1—4** than the tetragonal space group *I4/m*, in which the structures of **4** and **5** were solved[4,5] and which is consistent only with the positions of the I, Br, As, Cl, and P atoms, *i.e.*, with the "heavy" atoms of these salts. An inconsistency of the group *I4/m* has also been noted previously.[4] In particular, it was found that in this group, the C_{60}^- anions surround the *4/m* centers in spite of the fact that the point symmetry group I_h of the C_{60} fullerene does not contain a rotation fourfold axis.

The crystal-chemical study of the radical-anionic salts of C_{60} fullerene, **1—3**, allows us to reveal a number of their structural features. Primarily, these structures are characterized by the absence of shortened intermolecular contacts between C_{60} molecules and the Ph_4P or Ph_4As groups. The absence of these contacts and their associated intermolecular contacts, which prevent the rotation of the fullerene and, hence, enhance the stability of fullerene complexes,[12,13] suggests that salts **1—3** are stabilized either through electrostatic interactions or through interactions between the *p*-orbitals of the carbon atoms at the double bonds of the fullerenes and the correspondingly oriented phenyl rings of the cations. It should be also noted that in spite of the substantial differences in the C—C and C=C bond lengths in the C_{60}^- anions of salts **1—3**, their average values suggest that the single and double bonds alternate in these anions.

This work was supported by the Russian Scientific and Technical Program "Actual Directions in the Physics of Condensed Media," the Direction "Fullerenes and Atomic Clusters," and by the Russian Foundation for Basic Research (Project No. 96-03-32685a).

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Received November 26, 1996;
in revised form May 13, 1997