Russian Chemical Bulletin, International Edition, Vol. 53, No. 12, pp. 2793—2797, December, 2004

Electrochemical synthesis of carbonyl- and phosphoryl-containing methano[60]fullerenes

V. V. Yanilkin,* V. P. Gubskaya, N. V. Nastapova, L. Sh. Berezhnaya, I. E. Ismaev, Yu. Ya. Efremov, and I. A. Nuretdinov

A. E. Arbuzov Institute of Organic and Physical Chemistry,
Kazan Research Center of the Russian Academy of Sciences,
8 ul. Akad. Arbuzova, 420088 Kazan, Russian Federation.
Fax: +7 (843 2) 73 2253. E-mail: yan@iopc.knc.ru

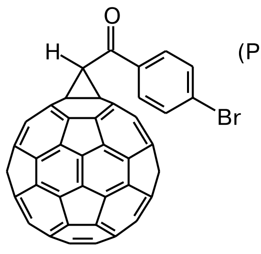
The combined electrochemical reduction of fullerene C_{60} in the presence of *p*-bromobenzoyldibromomethane or bis(diisopropoxyphosphoryl)dibromomethane at a brass electrode in a mixed solvent *o*-dichlorobenzene—MeCN (3 : 1 v/v)/0.05 *M* Bu_4NBF_4 at room temperature affords 61-(*p*-bromobenzoyl)methano[60]fullerene or 61,61-bis(diisopropoxyphosphoryl)methano[60]fullerene, respectively, in ~50% yields.

Key words: electroreduction, synthesis, fullerene, methanofullerene.

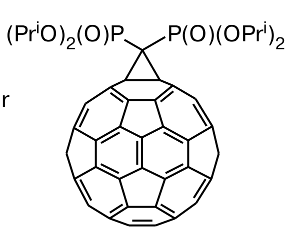
The preparation of functionalized fullerene derivatives and development of methods for their synthesis are significant for the creation of new materials and biologically active compounds based on fullerene. It is known[1—3] that fullerenes in reactions of electrochemical reduction (ER) in aprotic media can reversibly and step-by-step accept up to six electrons per molecule to form hexaanions. Anionic intermediates (radical anions (RA), dianions (DA), radical trianions, *etc.*) manifest properties of bases, nucleophiles, and electron carriers. These properties enhance substantially with an increase in the negative charge of the anion. Therefore, under ER conditions in aprotic media, fullerenes can react as bases, nucleophiles, or *via* an electron transfer mechanism with various substrates, depending on the nature of the latter, which provides wide potentialities of fullerene functionalization. Synthetic prospects of this approach are related to using the electrochemical method that allows one to perform a fine control of the nature of a generated intermediate and its amount by changes in the electrode potential and the amount of passed electricity. Nevertheless, electrosynthesis of fullerene derivatives has not yet been developed properly, and we found only several examples[4—11] of this type in the literature. The methods described for electrosyntheses of fullerene derivatives assume the electrochemical generation of anionic intermediates of fullerene or its derivatives followed by their interaction with halogen-containing organic[4—8] and metal complex[9—11] compounds. In particular, the electrochemical generation of DA of fullerenes and their derivatives affords dialkyl-, alkyl-, and arylfullerenes $C_{60}R_2$ (R = Me, Et, Bu^n, Ph),[4,8,9] alkyl- and arylhydrofullerenes $C_{60}RH$ (R = Me, Ph),[9] tetraalkylfullerenes $C_{60}R_4$ (R = Me, Bu^n, CH_2Ph),[6,8] methanofullerenes $C_{61}RR'$ (R = H, COOEt; R′ = H, COOEt, Bu^t, CN),[5] and exohedral metal complexes of C_{60} and C_{70}.[9—11] Radical trianions[9] and RA of fullerenes[9] are used in the synthetic practice much more rarely. Positive examples for the use of electrochemically generated tetra-, penta-, and hexaanions of fullerenes are not described in the literature. An attempt to use the generation of tetraanions for the synthesis of diarylfullerenes was unsuccessful.[9]

We proposed[12] a one-pot method for the electrosynthesis of methanofullerenes from fullerene C_{60} and geminal dihalides. Electrolysis conditions (potential, ratio of reactants, amount of electricity) were chosen on the basis of analysis of cyclic voltammograms in solutions of these substrates. 61-(Dimethoxyphosphoryl)-61-(methoxycarbonyl)methano[60]fullerene was synthesized by the combined ER of fullerene C_{60} and dibromo(dimethoxyphosphoryl)methoxycarbonylmethane. Electrolysis at potentials of the first step of ER through steps of heterogeneous and homogeneous mediator electron transfer to the dibromosubstituted derivative produces monomethanofullerene. Although the yield of the isolated product was only ~10%, the chromatographic yield was rather high (higher than 50%). The obtained result indicates that the variant proposed for the electrosynthesis of methanofullerenes is attractive. To develop these studies, in this work we studied a possibility of electrosynthesis of monoadducts of fullerene C_{60} **1—5** by the combined ER of fullerene C_{60} and halides **6—10**, respectively.

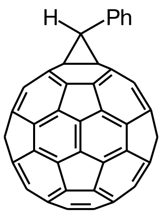
Published in Russian in *Izvestiya Akademii Nauk. Seriya Khimicheskaya,* No. 12, pp. 2681—2685, December, 2004.



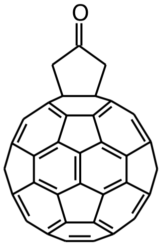
1



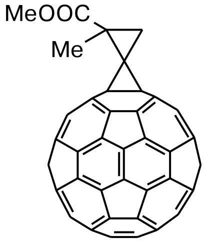
2



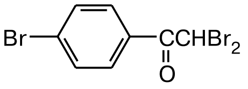
3



4



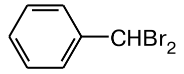
5



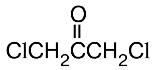
6

$(Pr^iO)_2P(O)CBr_2P(O)(OPr^i)_2$

7

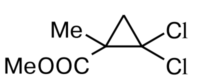


8



$ClCH_2C(O)CH_2Cl$

9



10

Results and Discussion

To estimate the expedience of preparative electrolysis, we preliminarily recorded cyclic voltammograms of fullerene C_{60}, compounds **6—10**, and their mixture at a glassy-carbon electrode in a system *o*-dichlorobenzene (*o*-DCB)—MeCN (3 : 1 vol/vol) containing 0.1 *M* Bu_4NBF_4. The potentials of ER peaks are presented in Table 1. As can be seen, all studied compounds, except for 1,1-dichloro-2-methoxycarbonyl-2-methylcyclopropane (**10**), are reduced under these conditions in an accessible region of potentials. In the case of fullerene solutions, four reversible one-electron reduction peaks are detected, whereas two or three irreversible peaks are observed in solutions of compounds **6—9**. Compounds **6—9** are reduced irreversibly by the first step with the transfer of two electrons, which is typical of the reductive cleavage of the C—Hal bond to form the corresponding carbanions.

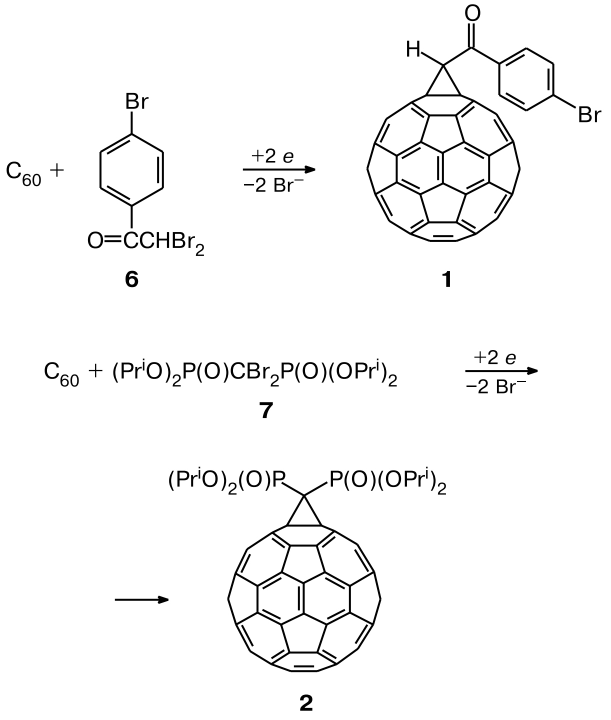
Table 1. Potentials of the reduction peaks (E_p, *vs.* Fc/Fc$^+$) of fullerenes C_{60} and compounds **6—9*** at the glassy-carbon electrode in *o*-DCB—MeCN/0.1 *M* Bu_4NBF_4 ($C_s = 1 \cdot 10^{-3}$ mol L^{-1})

Redox system	$-E_p^1$	$-E_p^2$	$-E_p^3$	$-E_p^4$
	V			
C_{60}	1.06	1.47	1.94	2.41
6	1.13	1.35	2.14	—
7	1.05	1.51	1.99	—
8	1.87	2.22	—	—
9	1.92	2.23	—	—

* Compound **10** is not reduced to the potential −2.50 V.

The first peak of fullerene C_{60} reduction and peaks of bromide reduction are imposed in the voltammetric curve of a mixture of fullerene with bromides **6** and **7**. Therefore, the generation of RA of fullerene and carbanions of bromophosphonate and bromoacetophenone should be expected for the electrolysis of the mixture at this potential. Methanofullerenes can be formed *via* two directions: interaction of carbanions with fullerene C_{60} similar to the chemical syntheses of methanofullerenes according to Bingel[13] or reactions of fullerene RA with starting bromides **6** and **7**. In the latter case, the fullerene RA acts toward bromides as an electron mediator, reducing bromides to a radical or carbanion, which can, in turn, react with fullerene to form a C—C bond. Both variants finally produce methanofullerenes (Scheme 1).

Scheme 1



In the presence of benzylidenedibromide (**8**) and 1,3-dichloroacetone (**9**), two reversible reduction peaks of fullerene are identical to those observed for fullerene in the absence of dihalides. It follows from this that neither

RA nor DA of C_{60} react with these dihalides with a noticeable rate under the voltammetry conditions. At the potentials of dihalide reduction, fullerene should exist in the near-electrode layer as DA. Naturally, the probability of the reaction of DA with carbanions formed from dihalides is low, which increases the probability of formation of polymeric fullerene products.[12] Therefore, the preparative reduction of fullerene and dihalides **8** and **9** at high cathodic potentials can afford methanofullerenes only in a low yield.

As mentioned above, 1,1-dichlorocyclopropane **10** is not reduced to potentials of background decomposition, and four peaks of fullerene reduction are observed in the presence of **10**. The first three peaks are reversible and completely identical to the peaks in solutions of fullerene C_{60} in the absence of dichloride **10**. This indicates that RA, DA, and radical trianions do not react with dichloride **10**. In turn, the fourth peak increases substantially in the presence of dichloride **10** and becomes irreversible. Evidently, a current increase is caused by the fast homogeneous reduction of dichloride **10** by electrochemically generated fullerene tetraanions. The current increase allows one to draw a qualitative conclusion about a high rate of dichlorocyclopropane reduction by fullerene tetraanions. We believe that this is the first example when the fullerene tetraanion acts as a mediator. Before this discovery, the use of fullerene as a mediator was indicated in the literature only under conditions of generation of RA, DA, and radical trianions.[14—18] As in the above-considered variant, the formation of target methanofullerene, in this case, is complicated by secondary chemical reactions.

An analysis of the voltammetric data shows that the highest yield of monofunctionalized fullerene derivatives can be expected for the combined ER of fullerene C_{60} and compounds **6** and **7**. Therefore, this variant was accomplished at a preparative level (see Experimental). Note that the chemical method of methanofullerene formation through the generation of the monobromophosphonate carbanion affords bismethanofullerenes along with monomethanofullerenes.[19] Since ER proceeds through the generation of carbanions, the formation of a mixture of mono- and bismethanofullerenes by the combined reduction of fullerene and compound **6** can be assumed. To perform the selective synthesis of monomethanofullerenes and avoid a substantial formation of bisadducts, the reduction process should not be brought to the complete conversion of fullerene C_{60}. The optimum conversion implies the maximum ratio of monomethanofullerene to the overall amount of fullerene and bismethanofullerenes. The conversion was determined by HPLC from the dependence of the reaction mixture composition on the amount of passed electricity. The chromatogram of the reaction mixture after preparative C_{60} reduction in the presence of bromide **6** for 40 min is presented in Fig. 1. This duration of electrolysis, which provides a consumption of 1.8 *F* per mole of fullerene, is likely optimum. According to the chromatogram, the main electrolysis product is monomethano[60]fullerene **1**. In addition, bismethanofullerenes are also formed, and unreacted fullerene C_{60} remains in the reaction mixture. All these compounds were isolated by column chromatography, and their preparative yields were the following: monomethano[60]fullerene **1**, 57%; bismethanofullerenes, 12.5% (calculated to reacted fullerene); fullerene, 52.5%.

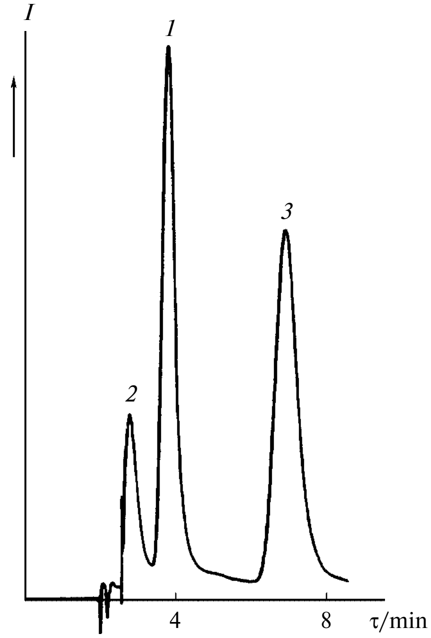
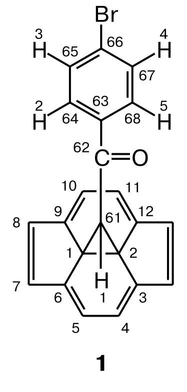


Fig. 1. High-performance liquid chromatogram of the reaction mixture after the preparative reduction of C_{60} in the presence of compound **6** for 40 min (1.8 *F* per moles of fullerene): mono- (*1*), bismethano[60]fullerene (*2*), and fullerene C_{60} (*3*). Electrolysis was carried out at a constant potential of –0.9 V *vs.* Ag/0.01 *M* $AgNO_3$ at the brass cylindrical electrode in *o*-DCB—MeCN (3 : 1)/0.05 *M* Bu_4NBF_4.

The preparative electroreduction of fullerene C_{60} was performed similarly in the presence of dibromide **7** to obtain bis(diisopropoxyphosphoryl)methano[60]fullerene **2** in 50.2% yield.

The structures of monomethanofullerenes **1** and **2** were proved by spectral methods. The molecular weights were determined by electron impact mass spectrometry (**1**) and MALDI-TOF (**2**). The IR spectra contain intense absorption bands of the fullerene core at 526 cm^{-1} (spectra of compounds **1** and **2**), carbonyl group at 1683 cm^{-1} (**1**), and phosphoryl group at 1265 cm^{-1} (**2**). The UV spectra of compounds **1** and **2**



1

contain absorption maxima at 427 and 692 nm, which corresponds to the formation of a monocycloadduct at the closed 6,6-bond.

A singlet at δ 5.603 and two doublets at δ 8.343 and 8.357 appear in the ^{1}H NMR spectra of compound **1**. The ^{13}C NMR spectrum exhibits 20 lines in an interval of 141.02—147.79 ppm of the C_{sp^2} atoms of the fullerene sphere, a signal of the C(61) atom at δ 43.04, a signal at δ 72.07 of two C_{sp^3} atoms of the fullerene sphere, and five lines of the bromobenzoyl moiety with δ 129.979, 130.332, 132.734, 134.659 and 188.770. The chemical shifts of the C and H atoms were assigned on the basis of heteronuclear correlation spectra through long-range HMBC constants. The cross-peaks between the signal of the proton with δ 5.60 and C atoms with δ 72.07, 147.79, and 146.37 indicate explicitly that the signal belongs to the H(1) proton, and the ^{13}C signal with δ 72.07 concerns the sp^3-hybridized C(1) and C(2) atoms of the fullerene shell. The ^{13}C NMR signals at δ 147.79 and 146.37 belong to the sp^2-hybridized C(9), C(12) and C(3), C(6) atoms, respectively. The ^{1}H signal with δ 8.30 has cross-peaks with ^{13}C signals at δ 129.98, 130.32, and 188.77. The ^{1}H signal with δ 7.80 is characterized by cross-peaks with ^{13}C signals at δ 130.30, 132.70, and 134.70. These data make it possible to assign the signals with δ 8.30 and 7.80 to protons H(2), H(6) and H(3), H(5), respectively. The signals in the ^{13}C NMR spectra at δ 188.77, 134.66, 132.73, 130.32, and 129.98 are attributed to the C(62), C(63), C(65), C(67), C(64), C(68), and C(66) atoms.

The ^{31}P NMR spectrum of compound **2** contains one signal with δ 11.74, and the ^{1}H NMR spectrum exhibits two doublets of protons of the Me groups (δ 1.32 and 1.41) and two multiplets of protons of the CHO groups (δ 5.01 and 5.07).

The ^{13}C NMR spectrum contains a triplet of the C(61) atom at δ 41.64 ($J_{C,P}$ = 150.0 Hz), a signal at δ 69.71 ($^2J_{C,P}$ = 4.1 Hz) of two sp^3-hybridized C atoms of the fullerene sphere and 17 lines in an interval of δ 141.39—147.87 for the sp^2-hybridized C atoms of the fullerene sphere. In addition, signals of the C atoms of the fullerene sphere at δ 17.29 (Me) and 64.38 (CHO) are observed. All spectral characteristics agree completely with the published data[19] for compound **2**.

Experimental

Fullerene C_{60} (99.9% purity, available from G. A. Razuvaev Institute of Organometallic Chemistry, Russian Academy of Sciences) and industrial compounds **6**, **8**, and **9** (Soyuzreaktiv, used without additional purification) were used. Compound **7** was synthesized by the bromination of tetraisopropoxydiphosphorylmethane (Lankaster) in the presence of sodium hydride as oil with the following constants: n_D^{20} = 1.4768, d_4^{20} = 1.4278, and $\delta_{^{31}P}$ = −7.2. Compound **10** was synthesized from the corresponding olefin and chloroform using the Makosza method.[20] Acetonitrile was purified by refluxing with $KMnO_4$ and fractional distillation above P_2O_5, and *o*-DCB was purified by vacuum distillation above CaH_2.

Cyclic voltammograms were recorded on a PI-50-1 potentiostat on an N 307/2 two-coordinate recorder. A glassy-carbon disk electrode (*d* = 2 mm) molded into fluoroplastic served as a working electrode. Before each measurement, the electrode was mechanically polished. Potentials were measured relatively to the standard potential of the ferrocene/ferrocenium ion (Fc/Fc^+) redox system using $Ag/AgNO_3$ (0.01 *M*) in MeCN as a reference electrode. Dissolved oxygen was removed by bubbling into a solution of dinitrogen at 20 °C.

The number of electrons was determined by the method of comparing with the one-electron peak of a standard compound (ferrocene).

Preparative electrochemical reduction was carried out using a PI-50-1 potentiostat in a divided (cellulose) glass electrolyzer with a brass cylindrical electrode (S = 16.4 cm^2) at a controlled potential of the first peak of fullerene reduction (E = −0.9 V *vs.* Ag/0.01 *M* $AgNO_3$ in MeCN) in a medium of *o*-DCB—MeCN (3 : 1 vol/vol)/0.05 *M* Bu_4NBF_4 under nitrogen at room (20 °C) temperature. An anode was placed inside a brass cylinder, which was filled with a solution of a supporting electrolyte. A platinum spiral was used as anode.

UV spectra were recorded on a Specord M-40 spectrophotometer in a THF—$CHCl_3$ (10 : 2) mixture. IR spectra were obtained on a Bruker-Vector 22 FTIR spectrometer (KBr pellets). ^{1}H, ^{13}C, and ^{31}P NMR spectra were recorded on Bruker MSL 400 and Bruker Avance 600 spectrometers (100.57 MHz for ^{13}C, 162.00 MHz for ^{31}P, 400.00 and 600.00 MHz for ^{1}H) in $CDCl_3$ (Me_4Si and 85% H_3PO_4 as standards). The mass spectrum of methanofullerene **1** was obtained on a Finnigan MAT-212 instrument; voltage 60 V, current of electron emission 0.1 mA, mass interval m/z 450—1500, direct injection of samples into an ion source, and temperature of the evaporator ~300 °C. The mass spectrum of methanofullerene **2** was recorded on a MALDI-TOF mass spectrometer. HPLC was carried out on a Gilson chromatograph with a UV detector using a column with the C-18 reverse phase (Partisil 5-ODS-3) using a toluene—acetonitrile (2 : 3 vol/vol) mixture as eluent.

61-(*p*-Bromobenzoyl)methano[60]fullerene (1). A working solution (30 mL) was prepared by the dissolution of fullerene (30 mg, 0.042 mmol) and tribromoacetophenone **6** (16 mg, 0.05 mmol) in a medium of *o*-DCB—MeCN (3 : 1 v/v)/0.05 *M* Bu_4NBF_4. The solution was magnetically stirred. Electrolysis was monitored by the current value and chromatographic analyses of the reaction mixture. To accumulate a needed amount of monomethanofullerene, the electrolysis was repeated three times more. The total loading of fullerene C_{60} was 120 mg. After electrolysis, the combined solution (120 mL) was concentrated to a minimum volume, the supporting salt was precipitated with hexane, and the solution was supported on a column packed with SiO_2. At first, unreacted fullerene C_{60} (63 mg) was isolated using hexane as eluent, then the main reaction product, monomethano[60]fullerene **1** (42 mg), was eluted with a toluene—hexane mixture, and, finally, a mixture of bismethano[60]fullerenes (11 mg) was obtained using toluene as eluent. The preparative yield of monomethano[60]fullerene **1** (solid powder with m.p. >360 °C) was 57% calculated per reacted fullerene. MS, *m/z* (I_{rel} (%)): 920 (2.2), 919 (3.9), 918 (6.6), 917 (3.6), 916 (6.9), 733 (8), 721 (66), 720 (100). Ions with *m/z* 920, 919, 918, 917, and 916 are molecular, being caused by the natural abundance of isotopes of C, H, O, and Br. The ratio of their intensities corresponds completely to the calculated values for compound **1** of composition $C_{68}H_5OBr$. The precision determination of the weight by the method of peak superposition with a resolution of 10 000 was carried out for the molecular ion with *m/z* 916. The weight determined experimentally was 915.953±0.001, and the calculated weight for $^{12}C_{68}{}^{1}H_5{}^{16}O^{79}Br$ was 915.95239. IR, ν/cm^{-1}: 526, 575, 674, 1003, 1069, 1180, 1214, 1241, 1271, 1317, 1400, 1426, 1581, 1683, 1711, 2851, 2921. UV, λ_{max}/nm: 328, 429, 495, 696. ^{1}H NMR (Me_4Si), δ: 5.603 (s, 1 H); 7.85, 8.35 (both d, 2 H each, *J* = 8 Hz). ^{13}C NMR ($CDCl_3$), δ: 43.818, 72.065, 129.979, 130.321, 132.734, 134.659, 141.024, 141.256, 142.116, 142.191, 142.270, 142.456, 142.827, 143.016, 143.124, 143.341, 143.723, 143.952, 144.423, 144.697, 145.123, 145.233, 145.318, 145.498, 146.368, 147.791, 187.770.

61,61-Bis(diisopropoxyphosphoryl)methano[60]fullerene (2). A working solution (20 mL) was prepared by the dissolution of fullerene (20 mg, 0.028 mmol) and bis(diisopropoxyphosphoryl)dibromomethane **7** (17 mg, 0.034 mmol) in *o*-DCB—MeCN (3 : 1 v/v)/0.05 *M* Bu_4NBF_4. The solution was magnetically stirred. The current was measured and chromatographic analyses of the reaction mixture were carried out during electrolysis similarly to the procedure for the synthesis of compound **1**. To accumulate an appropriate amount of monomethanofullerene, electrolysis was repeated four times. The overall loading of fullerene C_{60} was 100 mg. The combined solution was treated in the same manner as in the previous synthesis. After electrolysis, monomethanofullerene **2** (solid powder with m.p. >360 °C) was obtained in 50.2% yield (51 mg) along with fullerene C_{60} (21 mg). **Compound 2.** MS, *m/z*: 1062.521, 1021.342, 979.575, 938.680, 895.975. The ion with *m/z* 1062 is molecular, and its weight corresponds to the formula $C_{73}H_{28}O_6P_2$. IR, ν/cm^{-1}: 526, 568, 608, 996, 1021, 1103, 1147, 1175, 1200, 1265, 1384, 1427, 1531, 1635, 2890, 2926, 2974. UV, λ_{max}/nm: 326.5, 426.7, 491.3, 692.7. ^{1}H NMR (Me_4Si), δ: 1.32, 1.41 (both d, 12 H each, ^3J = 6.0 Hz); 5.01, 5.07 (both m, 2 H each). ^{31}P NMR ($CDCl_3$), δ: 11.74. ^{13}C NMR ($CDCl_3$), δ: 17.29 (q, 8 Me, $^1J_{C,H}$ = 127.1 Hz); 41.64 (t, C(61), $^1J_{C,P}$ = 150.1 Hz); 64.38 (dd, 4 CHO, $^1J_{C,H}$ = 149.0 Hz, $^2J_{COP}$ = 4.6 Hz); 69.71 (t, 2 C_{sp3}, $^2J_{C,P}$ = 4.1 Hz); 141.39, 141.92, 143.27, 143.91, 144.09, 144.18, 144.30, 145.61, 145.68, 145.84, 145.99, 146.04, 146.31, 146.32, 146.33; 147.72, 147.87 (both d, 2 C each, $^3J_{C,P}$ = 5.0 Hz).

This work was financially supported by the Russian Foundation for Basic Research (Project Nos. 04-03-32287 and 02-03-32900).

References

1. Q. Xie, E. Perez-Cordero, and L. Echegoyen, *J. Am. Chem. Soc.*, 1992, **114**, 3978.
2. Y. Ohsawa and T. Saji, *J. Chem. Soc., Chem. Commun.*, 1992, 781.
3. F. Zhou, C. Jeboulet, and A. J. Bard, *J. Am. Chem. Soc.*, 1992, **114**, 11004.
4. C. Caron, R. Subramanian, F. D´Souza, J. Kim, W. Kutner, M. T. Jones, and K. M. Kadish, *J. Am. Chem. Soc.*, 1993, **115**, 8505.
5. P. L. Boulas, Y. Zuo, and L. Echegoyen, *Chem. Commun.*, 1996, 1547.
6. K. M. Kadish, X. Gao, E. Van Caemelbecke, T. Suenobu, and S. Fukuzumi, *J. Am. Chem. Soc.*, 2000, **122**, 563.
7. K. M. Kadish, X. Gao, E. Van Caemelbecke, T. Hirasaka, T. Suenobu, and S. Fukuzumi, *J. Phys. Chem.*, 1998, **102**, 3898.
8. K.-M. Mangold, W. Kutner, L. Dunsch, and J. Frohner, *Synth. Methods*, 1996, **77**, 73.
9. T. V. Magdesieva, V. V. Bashilov, D. N. Kravchuk, V. I. Sokolov, and K. P. Butin, *Elektrokhimiya*, 1999, **35**, 1125 [*Russ. J. Electrochem.*, 1999, **35**, 1117 (Engl. Transl.)].
10. V. V. Bashilov, T. V. Magdesieva, D. N. Kravchuk, P. V. Petrovskii, A. Ginzburg, K. P. Butin, and V. I. Sokolov, *J. Organomet. Chem.*, 2000, **599**, 37.
11. T. V. Magdesieva, D. N. Kravchuk, V. V. Bashilov, I. V. Kuznetsova, V. I. Sokolov, and K. P. Butin, *Izv. Akad. Nauk, Ser. Khim.*, 2002, 1459 [*Russ. Chem. Bull., Int. Ed.*, 2002, **51**, 1588].
12. V. V. Yanilkin, V. P. Gubskaya, and I. A. Nuretdinov, *Mendeleev Commun.*, 2003, 13.
13. C. Bingel, *Chem. Ber.*, 1993, **126**, 1957.
14. T. Fuchigami, M. Kasuga, and A. Konno, *J. Electroanal. Chem.*, 1996, **411**, 115.
15. Y. Huang and D. D. Wayner, *J. Am. Chem. Soc.*, 1993, **115**, 367.
16. F. D´Souza, J.-P. Choi, Y.-Y. Hsieh, K. Shriver, and W. Kutner, *J. Phys. Chem. B*, 1998, **102**, 212.
17. F. D´Souza, J.-P. Choi, and W. Kutner, *J. Phys. Chem. B*, 1998, **102**, 4247.
18. F. D´Souza, J-P. Choi, and W. Kutner, *J. Phys. Chem. B*, 1999, **103**, 2892.
19. I. A. Nuretdinov, V. P. Gubskaya, N. I. Shishikina, G. M. Fazleeva, L. Sh. Berezhnaya, I. P. Karaseva, F. G. Sibgatullina, and V. V. Zverev, *Izv. Akad. Nauk, Ser. Khim.*, 2002, 250 [*Russ. Chem. Bull., Int. Ed.*, 2002, **51**, 263].
20. M. Makosza and M. Wawrzyniewicz, *Tetrahedron Lett.*, 1969, **53**, 4659.

Received May 12, 2004;
in revised form September 6, 2004