Available online at www.sciencedirect.com

ScienceDirect

Mendeleev Commun., 2018, **28**, 76–78

Mendeleev Communications

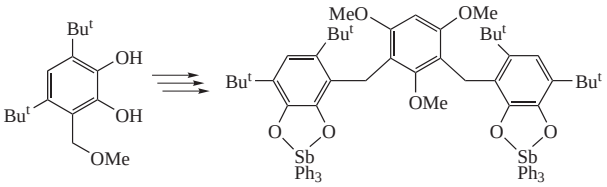
New sterically hindered bis-catechol, bis-*o*-quinone and its bis-triphenylantimony(V) bis-catecholate. 3,5-Di-*tert*-butyl-6-methoxymethylcatechol as alkylating agent

Maxim V. Arsenyev,* Tatyana V. Astaf'eva, Evgeny V. Baranov, Andrey I. Poddel'sky and Sergey A. Chesnokov

G. A. Razuvaev Institute of Organometallic Chemistry, Russian Academy of Sciences, 603950 Nizhny Novgorod, Russian Federation. E-mail: mars@iomc.ras.ru

DOI: 10.1016/j.mencom.2018.01.025

Alkylation of 1,3,5-trimethoxybenzene with 3,5-di-*tert*-butyl-6-methoxymethylcatechol affords new sterically hindered bis-catechol, whose oxidation gives the corresponding bis-*o*-quinone. Its treatment with triphenylstibine leads to bis-triphenylantimony(V) bis-catecholate.

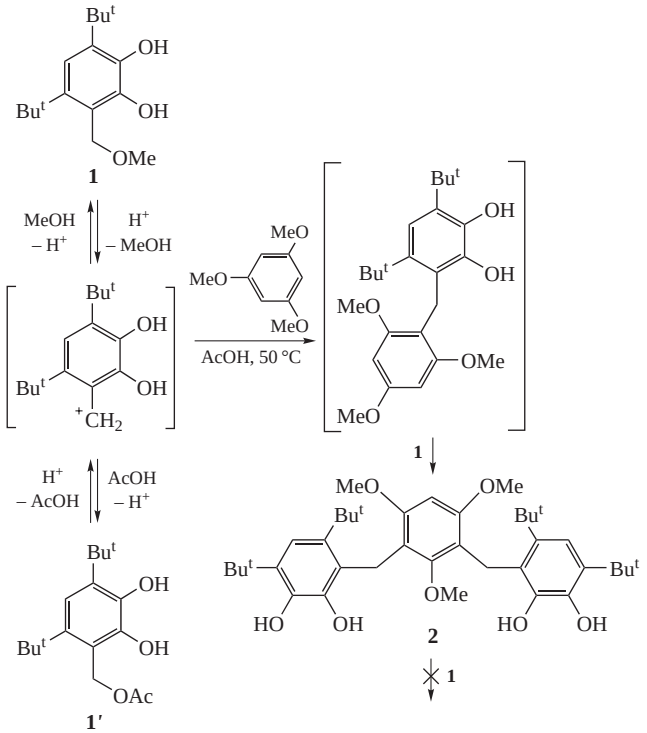


Organoantimony compounds are interesting systems in coordination,[1] organic[2] and bioinorganic[3] chemistry. A special place in organoantimony chemistry is occupied by redox-active catecholate antimony complexes.[4] Among them, triphenylantimony(V) derivatives are capable of reversible binding molecular oxygen with a formation of spiro endo peroxides in solution[5] and polymer materials.[6] Triphenylantimony(V) catecholates also demonstrate antiradical activity.[7] The central antimony(V) atom in catecholate complexes usually exists in a five-coordinate environment and can coordinate various donor substrates as soft Lewis acid. Moving from mononuclear to polynuclear antimony complexes leads to compounds with new structural types and provides access to new molecules-sensors, *e.g.*, for fluoride ions.[4(b)] Binuclear triarylantimony(V) catecholate complexes can be prepared from binuclear organometal compounds and monomeric catechol/quinone or from monomeric triarylantimony and bis-catechol/bis-*o*-quinone ligands.

In this article we describe the synthesis and structure of new bis-triphenylantimony(V) bis-catecholate based on the sterically hindered bis-*o*-quinone ligand. Generally, the reaction between *o*-quinone and triarylantimony(III) quantitatively affords triarylantimony(V) catecholate complex. Hence, synthesis of binuclear triarylantimony(V) catecholates requires new bis-catecholes/bis-*o*-quinones which can be accessed by nucleophilic addition to quinones[8] or functional catechols,[9] cross-coupling reaction,[10] oxidative dimerization,[10] or polymerization of monomeric quinone.[6,12] Herein, we used alkylation of 1,3,5-trimethoxybenzene with sterically hindered *o*-quinone methide.

Earlier,[13,14] we obtained 3,5-di-*tert*-butyl-6-methoxymethylcatechol and applied it in alkylation of heterocyclic compounds. The active intermediate in such reactions is benzylic carbocation, a protonated form of *o*-quinone methide,[15] generated under acidic conditions. Methoxymethylbenzenes can form *o*-quinone methides under neutral conditions as well.[16] In our case, when catechol **1** is dissolved in AcOH at room temperature, a rapid equilibrium with acetate **1′** is established (Scheme 1). The decreasing intensity of signals at 1.36, 1.39 (Bu^t), 3.48 (OMe), 4.91 (CH_2), 6.88 (C_{Ar}–H) ppm (compound **1**) with simultaneous increasing those at 1.40, 1.41, 5.46, 6.99 ppm (compound **1′**) and at 3.42 ppm (MeOD) in ^{1}H NMR spectrum of catechol **1** in AcOH-d_4 are observed. The equilibrium constant $K = 4.8 \times 10^{-2}$ calculated from the NMR data indicates that the formed ester **1′** is the major alkylating agent in acetic acid (by the analogy with *p*-quinone methide[16]).

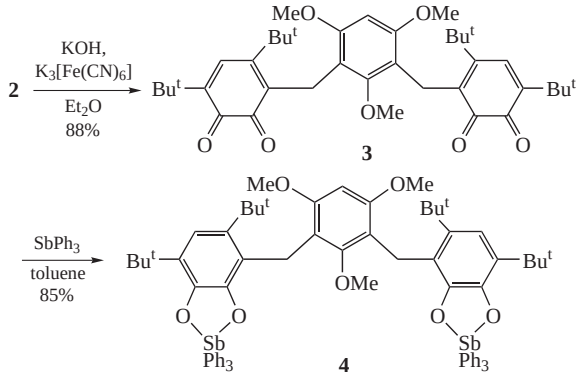
It is known that alkylation of 1,3,5-trimethoxybenzene can give mono-, di- and trisubstituted products.[18] However, tris-adduct was not formed even with the use of excess **1** (three and more equivalents, AcOH, 50 °C), and the main product was bis-catechol **2** (see Scheme 1). Most probably, tris-adduct cannot form due to



Scheme 1

steric factors. To prepare bis-catechol **2**, the use of two equivalents of **1** per trimethoxybenzene was optimal.† In the ^{1}H NMR spectrum of catechol **2**, the phenolic OH groups resonate in the region of 5–6 ppm unlike trialkyl-substituted catechols[13,14,19] including catechol **1**. The shift of signals of OH groups from 12–13 to 5–6 ppm is indicative of weak intramolecular hydrogen bonding. Compounds of similar structures should be good inhibitors of free radical processes.[20]

The oxidation of catechol **2** with $K_3[Fe(CN)_6]$ in alkaline medium leads to new bis-*o*-quinone **3** (Scheme 2).‡ The specific signals of quinonoid systems are observed at 184.80, 183.54 ppm (carbonyl groups) in ^{13}C NMR spectrum and 1678, 1662, 1618 cm^{-1} in IR spectrum. Treatment of bis-*o*-benzoquinone **3** with triphenylstibine gives new bis-triphenylantimony(V) bis-catecholate **4**,§ in which catechol fragments are equal in solution according to



Scheme 2

^{1}H and ^{13}C NMR spectra. The signals of CH_2 and C_{Cat}–H protons are observed at 4.25 and 6.74 ppm, respectively.

The X-ray suitable crystals of **4** were grown as the water solvate **4**·H_2O by slow crystallization from *n*-hexane in air at 4 °C (Figure 1).¶ Antimony atoms in crystals of **4**·H_2O are not equal in contrast to solution. Atom Sb(1) has a pentacoordinated environment. The parameter τ applied for the description of the geometry in pentacoordinated compounds[21] was calculated to be 0.18. This value testifies to the tetragonal pyramidal environment of Sb(1). The base of pyramid is formed by atoms O(1), O(2), C(40) and C(52); the bond angles O(1)–Sb(1)–C(40) and O(2)–Sb(1)–C(52) are 144.83(2)° and 155.31(2)°, respectively. The water molecule is coordinated to Sb(2), so the latter adopts the distorted octahedral environment. Aromatic cycles C(1)–C(6),

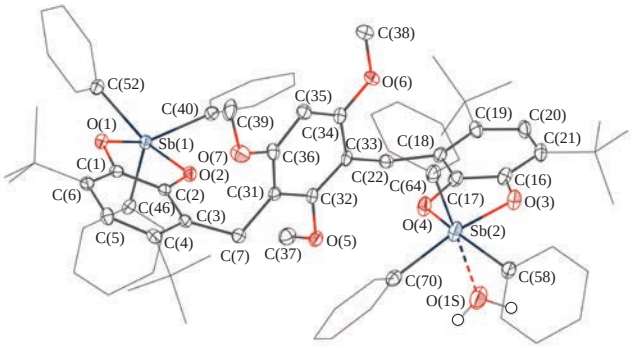


Figure 1 Molecular structure of bis-catecholate **4**·H_2O. Thermal ellipsoids are given with 30% probability. Hydrogens are not shown (excluding H_2O).

† *6,6'-[(2,4,6-Trimethoxy-1,3-phenylene)bis(methylene)]bis(3,5-di-tert-butylcatechol)* **2**. 3,5-Di-*tert*-butyl-6-methoxymethylcatechol (2.68 g, 10 mmol) and 1,3,5-trimethoxybenzene (0.84 g, 5 mmol) were dissolved in AcOH (10 ml), and the mixture was stirred at 50 °C for 4 h. Water (5 ml) was added and the precipitate was filtered, dried *in vacuo* and recrystallized from hexane to afford 2.48 g (78%) of white crystals, mp 165–167 °C. ^{1}H NMR (200 MHz, $CDCl_3$) δ: 6.93 (s, 2H, C_{Ar}–H), 6.34 (s, 1H, C_{Ar2}–H), 6.11 (s, 2H, OH), 5.87 (s, 2H, OH), 4.19 (s, 4H, CH_2), 3.70 (s, 6H, OMe), 3.21 (s, 3H, OMe), 1.48 and 1.37 (2s, 18H, Bu^t). ^{13}C NMR (50 MHz, $CDCl_3$) δ: 157.71, 142.59, 142.32, 138.10, 131.76, 123.67, 116.66, 114.91, 93.75, 62.33, 55.89, 36.22, 34.69, 32.05, 29.53, 24.35. IR (Nujol, ν/cm^{-1}): 3518, 3507, 3443, 3330 (m), 1601 (s), 1588 (m), 1489 (m), 1483 (m), 1414 (s), 1400 (m), 1372 (s), 1365 (m), 1319 (w), 1296 (m), 1286 (s), 1260 (m), 1240 (s), 1217 (m), 1208 (m), 1184 (w), 1173 (w), 1164 (w), 1122 (w), 1095 (s), 1036 (w), 1022 (m), 973 (m), 933 (w), 924 (w), 888 (w), 868 (m), 810 (m), 805 (w), 784 (w), 760 (w), 744 (w), 716 (m), 678 (m), 639 (w), 603 (w), 565 (w), 514 (m), 453 (w). Found (%): C, 73.61; H, 8.83. Calc. for $C_{39}H_{56}O_7$ (%): C, 73.55; H, 8.86.

‡ *6,6'-[(2,4,6-Trimethoxy-1,3-phenylene)bis(methylene)]bis(3,5-di-tert-butyl-o-benzoquinone)* **3**. The mixture of solutions of compound **2** (1.92 g, 3 mmol) in Et_2O (50 ml) and $K_3[Fe(CN)_6]$ (6.8 g, 20 mmol) with KOH (0.67 g, 12 mmol) in water (100 ml) was vigorously stirred for 60 min. Then the ether layer was washed with water (3×100 ml) and the extract was dried with Na_2SO_4. The solvent was evaporated and the product was crystallized from hexane solution (200 ml). The powder was filtered, washed with cold hexane and dried *in vacuo* to afford 1.67 g (88%) of red crystals, mp 191–193 °C. ^{1}H NMR (200 MHz, $CDCl_3$) δ: 7.02 (s, 2H, C_q–H), 6.09 (s, 1H, C_{Ar2}–H), 3.96 (s, 4H, 2CH_2), 3.56 (s, 9H, 3OMe), 1.24 and 1.17 (2s, 18H, Bu^t). ^{13}C NMR (50 MHz, $CDCl_3$) δ: 184.80, 183.54, 156.65, 156.37, 150.50, 145.51, 139.19, 139.00, 115.35, 93.12, 60.48, 55.65, 37.62, 34.92, 29.86, 29.15, 22.16. IR (Nujol, ν/cm^{-1}): 1678 (s), 1662 (s), 1618 (w), 1594 (m), 1582 (m), 1565 (w), 1398 (w), 1377 (s), 1368 (s), 1329 (w), 1316 (w), 1278 (m), 1240 (m), 1212 (m), 1196 (m), 1183 (m), 1150 (w), 1115 (s), 1090 (s), 1019 (m), 980 (m), 973 (m), 937 (w), 929 (w), 918 (w), 907 (m), 901 (m), 856 (w), 848 (w), 800 (m), 746 (w), 676 (w), 610 (m), 601 (m), 490 (w). Found (%): C, 74.07; H, 8.20. Calc. for $C_{39}H_{52}O_7$ (%): C, 74.02; H, 8.28.

§ *Bis-triphenylantimony(V) bis-catecholate* **4**. A solution of triphenylstibine (353 mg, 1 mmol) in toluene (30 ml) was added dropwise with stirring to a solution of *o*-benzoquinone **3** (320 mg, 0.5 mmol) in toluene (15 ml) until the solution colour turned completely yellow. Toluene was evaporated under reduced pressure and the residue was dissolved in *n*-hexane (30 ml). Its storage at –18 °C for a day provided bright yellow crystalline powder of complex **4**, yield 572 mg (85%). ^{1}H NMR (200 MHz, $CDCl_3$) δ: 7.52–7.62 (m, 18H, Ph), 7.34–7.46 (m, 18H, Ph), 6.74 (s, 2H, C_{Cat}–H), 5.93 (s, 1H, C_{Ar}–H), 4.25 (s, 4H, CH_2), 3.11 (s, 6H, 2OMe), 2.95 (s, 3H, OMe), 1.46 (s, 36H, 4Bu^t). ^{13}C NMR (50 MHz, $CDCl_3$): 158.36, 156.72, 146.96, 141.69, 138.06, 136.23, 135.31, 130.65, 129.59, 128.85, 123.48, 118.74, 111.88, 96.36, 59.44, 56.04, 36.01, 34.49, 32.42, 29.85, 26.37. IR (Nujol, ν/cm^{-1}): 1595 (m), 1581 (m), 1477 (s), 1432 (s), 1404 (s), 1377 (s), 1365 (m), 1330 (w), 1316 (w), 1290 (m), 1262 (m), 1240 (m), 1203 (m), 1182 (w), 1171 (w), 1118 (m), 1089 (m), 1071 (m), 1061 (w), 1050 (m), 1022 (m), 997 (m), 983 (m), 937 (w), 883 (w), 853 (w), 845 (w), 800 (m), 743 (s), 735 (s), 693 (s), 677 (w), 665(w), 617 (m), 611 (m), 592 (w), 550 (w), 515 (w), 482 (m), 452 (s). Found (%): C, 67.32; H, 6.14; Sb, 18.10. Calc. for $C_{75}H_{82}O_7Sb_2$ (%): C, 67.28; H, 6.17; Sb, 18.19.

¶ *X-ray diffraction analysis*. The crystals suitable for X-ray analysis (solvate with H_2O) were grown from hexane. The X-ray data were collected on a Bruker AXS SMART APEX diffractometer (graphite-monochromated MoKα radiation, λ = 0.71073 Å, ω-scan technique) at 100 K. The intensity data were integrated by SAINT[23] program. SADABS[24] was used to perform area-detector scaling and absorption corrections. The structure was solved by a direct method with a dual-space algorithm using SHELXT program[25] and was refined on F^2 using SHELXTL[26] package. All non-hydrogen atoms were found from Fourier synthesis of electron density and were refined anisotropically. The hydrogen atoms were placed in calculated positions and refined in the riding model except for those of the coordinated water molecule, which were located from difference Fourier synthesis and refined isotropically. One OMe group of the bridging 2,4,6-trimethoxy-1,3-phenylene fragment is disordered over two positions.

Crystal data for compound **4**. $C_{75}H_{82}O_7Sb_2·H_2O$, M = 1356.92, crystal size 0.22×0.16×0.09 mm, monoclinic, space group $C2/c$, at 100 K: a = 35.710(7), b = 12.827(2) and c = 31.647(5) Å, β = 115.685(4)°, V = = 13063(4) Å^3, Z = 8, d_{calc} = 1.380 g cm^{-3}, μ = 0.883 mm^{-1}, F(000) = 5600, 2θ = 52°, R_{int} = 0.1026 (12824), R_1 = 0.0580 [$I > 2\sigma(I)$], wR_2 (all data) = = 0.1149, largest diff. electron density, peak and hole, 2.639 / –2.459 e Å^{-3}.

CCDC 1560909 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre *via* http://www.ccdc.cam.ac.uk.

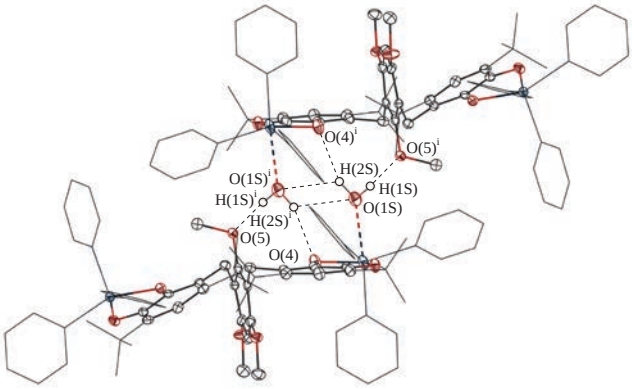


Figure 2 Dimeric fragment in the crystal of **4**·H_2O.

C(31)–C(26) and C(16)–C(21) are not coplanar. The breakage of structure is carried out along the methylene groups [atoms C(7) and C(22)], and the catecholate fragments [C(1)–C(6) and C(31)–C(26)] located on the opposite sides relative to the plane of the trimethoxybenzene fragment [C(16)–C(21)]. The antimony atoms are not located in the plane of chelating catecholate ligands. The central atoms Sb(1) and Sb(2) are shifted by 0.468 and 0.104 Å apart from the catecholato planes. The bend angles of the chelating metallocycles along the lines O(1)···O(2) and O(5)···O(6) are 16.9° and 4.4°, respectively. The geometrical characteristics of redox active ligands correspond to catecholato form of both O,O′-chelating fragments. The bonds O(1)–C(1) and O(2)–C(2) [1.367(6) and 1.376(6) Å, respectively], and O(3)–C(16) and O(4)–C(17) [1.379(6) and 1.366(6) Å, respectively] fall in the range of typical values for ordinary O–C bonds in different catecholates.[22]

The formation of dimer structures of **4**·H_2O observed in crystal is due to the two types of intermolecular hydrogen bonds involving water [H_2O(S) and H_2O(S)′] coordinated to the atoms Sb(2) and Sb(2)′ (Figure 2), *viz.*, two strong two-centered H-bonds with participation of methoxy groups O(5)C(37) and O(5)^{i}C(37)^{i} [the distances O(5)···H(1S)^{i} and H(1S)···O(5)^{i} are 1.80(2) Å] and four weak three-centered H-bonds with participation of oxygen atoms O(4)^{i}, O(4) (catecholic fragments) and O(1S)^{i}, O(1S) (coordinated water). The distances H(2S)^{i}···O(4), H(2S)···O(4)^{i} and H(2S)^{i}···O(1S), H(2S)···O(1S)^{i} are 2.32(3) and 2.45(5) Å, respectively.

Thus, the results obtained show that catechol **1** can be used as alkylating agent for activated aromatic compounds in the synthesis of oligomeric sterically shielded trialkyl substituted catechols. New bis-*o*-benzoquinone **3** and bis-catecholate of triphenylantimony(V) **4** were synthesized. Catecholate **4** crystallizes as a monohydrate in air and dimeric structure of **4**·H_2O in the crystal is formed due to intermolecular hydrogen bonding with water molecules.

The equipment of the center for collective use ‘Analytical Center IOMC RAS’ (Nizhny Novgorod) was used in this work.

Carrying out the synthesis of bis-catechol and bis-quinone was supported by the President of the Russian Federation (grant no. MK-1951.2017.3). A.I.P. is grateful to the Russian Foundation for Basic Research (grant no. 16-33-60157mol_a_dk).

References

1 (*a*) W. Levason and G. Reid, in *Comprehensive Coordination Chemistry II*, eds. J. A. McCleverty and T. J. Meyer, Elsevier, 2003, vol. 3, pp. 465–544; (*b*) P. V. V. N. Kishore and V. Baskar, *Inorg. Chem.*, 2014, **53**, 6737; (*c*) A. K. Jami, M. S. R. Prabhu and V. Baskar, *Organometallics*, 2010, **29**, 1137.

2 Y. Huang, *Acc. Chem. Res.*, 1992, **25**, 182.

3 (*a*) N. Farrell, in *Comprehensive Coordination Chemistry II*, eds. J. A. McCleverty and T. J. Meyer, Elsevier, 2003, vol. 9, pp. 809–840; (*b*) R.-C. Liu, Y.-Q. Ma, L. Yu, J.-S. Li, J.-R. Cui and R.-Q. Wang, *Appl. Organomet. Chem.*, 2003, **17**, 662.

4 (*a*) J. Qiu, D. K. Unruh and A. F. Cozzolino, *J. Phys. Chem. A*, 2016, **120**, 9257; (*b*) M. Hirai and F. P. Gabbaï, *Chem. Sci.*, 2014, **5**, 1886.

5 G. A. Abakumov, A. I. Poddel’sky, E. V. Grunova, V. K. Cherkasov, G. K. Fukin, Y. A. Kurskii and L. G. Abakumova, *Angew. Chem. Int. Ed.*, 2005, **44**, 2767.

6 (*a*) M. V. Arsenyev, M. P. Shurygina, A. I. Poddel’sky, N. O. Druzhkov, S. A. Chesnokov, G. K. Fukin, V. K. Cherkasov and G. A. Abakumov, *J. Polym. Res.*, 2013, **20**, 98; (*b*) S. A. Chesnokov, N. A. Lenshina, M. V. Arsenyev, R. S. Kovylin, M. A. Baten’kin, A. I. Poddel’sky and G. A. Abakumov, *Appl. Organomet. Chem.*, 2017, **31**, e3553; (*c*) N. A. Lenshina, M. P. Shurygina, M. V. Arsenyev, A. I. Poddel’sky, S. D. Zaitsev, S. A. Chesnokov and G. A. Abakumov, *J. Coord. Chem.*, 2015, **68**, 4159.

7 I. V. Smolyaninov, A. I. Poddel’sky, E. O. Korchagina, S. A. Smolyaninova and N. T. Berberova, *Dokl. Phys. Chem.*, 2015, **460**, 45 (*Dokl. Akad. Nauk*, 2015, **460**, 561).

8 G. Poneti, M. Mannini, B. Cortigiani, L. Poggini, L. Sorace, E. Otero, P. Sainctavit, R. Sessoli and A. Dei, *Inorg. Chem.*, 2013, **52**, 11798.

9 (*a*) D. A. Shultz, S. H. Bodnar, R. K. Kumar, H. Lee and J. W. Kampf, *Inorg. Chem.*, 2001, **40**, 546; (*b*) M. V. Arsenyev, E. V. Baranov, S. A. Chesnokov, V. K. Cherkasov and G. A. Abakumov, *Russ. Chem. Bull., Int. Ed.*, 2013, **62**, 2394 (*Izv. Akad. Nauk, Ser. Khim.*, 2013, 2394); (*c*) M. V. Arsenyev, E. V. Baranov, A. Yu. Fedorov, S. A. Chesnokov and G. A. Abakumov, *Mendeleev Commun.*, 2015, **25**, 312; (*d*) M. V. Arsenyev, N. M. Khamaletdinova, E. V. Baranov, S. A. Chesnokov and V. K. Cherkasov, *Russ. Chem. Bull., Int. Ed.*, 2016, **65**, 1805 (*Izv. Akad. Nauk, Ser. Khim.*, 2016, 1805); (*e*) N. O. Druzhkov, E. N. Egorova, M. V. Arsen’ev, E. V. Baranov and V. K. Cherkasov, *Russ. Chem. Bull., Int. Ed.*, 2016, **65**, 2855 (*Izv. Akad. Nauk, Ser. Khim.*, 2016, 2855).

10 (*a*) D. A. Shultz, A. K. Boal, D. J. Driscoll, J. R. Kitchin and G. N. Tew, *J. Org. Chem.*, 1995, **60**, 3578; (*b*) A. Bencini, C. A. Daul, A. Dei, F. Mariotti, H. Lee, D. A. Shultz and L. Sorace, *Inorg. Chem.*, 2001, **40**, 1582; (*c*) A. Caneschi, A. Dei, C. P. Mussari, D. A. Shultz, L. Sorace and K. E. Vostrikova, *Inorg. Chem.*, 2002, **41**, 1086; (*d*) J. C. Sloop, D. A. Shultz, T. Coote, B. Shepler, U. Sullivan, J. W. Kampf and P. D. Boyle, *J. Phys. Org. Chem.*, 2012, **25**, 314.

11 A. I. Poddel’sky, A. V. Piskunov, N. O. Druzhkov, G. K. Fukin, V. K. Cherkasov and G. A. Abakumov, *Z. Anorg. Allg. Chem.*, 2009, **635**, 2563.

12 A. V. Safronova, L. N. Bochkarev, N. O. Druzhkov, Yu. A. Kurskii, E. V. Baranov and G. A. Abakumov, *Russ. J. Gen. Chem.*, 2012, **82**, 294 (*Zh. Obshch. Khim.*, 2012, **82**, 299).

13 M. V. Arsenyev, E. V. Baranov, M. P. Shurygina, S. A. Chesnokov and G. A. Abakumov, *Mendeleev Commun.*, 2016, **26**, 552.

14 A. I. Poddel’sky, M. V. Arsenyev, T. V. Astaf’eva, S. A. Chesnokov, G. K. Fukin and G. A. Abakumov, *J. Organomet. Chem.*, 2017, **835**, 17.

15 (*a*) R. Dalpozzo, G. Bartoli, L. Sambri and P. Melchiorre, *Chem. Rev.*, 2010, **110**, 3501; (*b*) M. S. Singh, A. Nagaraju, N. Anand and S. Chowdhury, *RSC Adv.*, 2014, **4**, 55924; (*c*) D. V. Osipov, V. A. Osyanin and Yu. N. Klimochkin, *Russ. Chem. Rev.*, 2017, **86**, 625.

16 W. Iwanek, K. Stefańska, A. Szumna and M. Wierzbicki, *Tetrahedron*, 2015, **71**, 2222.

17 S. V. Bukharov, G. N. Nugumanova, N. A. Mukmeneva, A. R. Burilov, E. M. Kasymova, M. A. Pudovik and A. I. Konovalov, *Zh. Org. Khim.*, 2004, **40**, 327 (in Russian).

18 K. Tangdenpaisal, W. Phakhodee, S. Ruchirawat and P. Ploypradith, *Tetrahedron*, 2013, **69**, 933.

19 Yu. A. Sayapin, I. O. Tupaeva, V. V. Tkachev and G. V. Shilov, *Russ. J. Org. Chem.*, 2016, **52**, 214 (*Zh. Org. Khim.*, 2016, **52**, 231).

20 (*a*) L. Sobczyk, S. J. Grabowski and T. M. Krygowski, *Chem. Rev.*, 2005, **105**, 3513; (*b*) T. Schaefer, *J. Phys. Chem.*, 1975, **79**, 1888; (*c*) E. T. Denisov and T. G. Denisova, *Russ. Chem. Rev.*, 2009, **78**, 1047 (*Usp. Khim.*, 2009, **78**, 1129).

21 A. W. Addison, T. N. Rao, J. Reedijk, J. van Rijn and G. C. Verschoor, *J. Chem. Soc., Dalton Trans.*, 1984, 1349.

22 A. I. Poddel’sky, E. V. Baranov, G. K. Fukin, V. K. Cherkasov and G. A. Abakumov, *J. Organomet. Chem.*, 2013, **733**, 44.

23 *SAINT, v. 8.27B*, Bruker AXS, Madison, WI, USA, 2012.

24 *SADABS, v. 2014/2*, Bruker AXS, Madison, WI, USA, 2014.

25 G. M. Sheldrick, *Acta Crystallogr.*, 2015, **A71**, 3.

26 G. M. Sheldrick, *SHELXTL, v. 6.14*, Bruker AXS, Madison, WI, 2003.

Received: 14th July 2017; Com. 17/5308