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Synthesis and structure of aluminum 2,2'-oxobis(phenolate)s

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ABSTRACT

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1. Introduction

Alkyl aluminum 2,2'-methylene-bis(phenolate)s have been extensively studied in the Ring-Opening Polymerization (ROP) of cyclic esters and cyclohexene oxide, and organic synthesis [1]. The structure of the compounds depends on reaction conditions and the bulkiness of the bis(phenolate) moiety. Reactions of aluminum trialkyls with 2,2'-methylene-4,6-alkylbis(phenol)s in non-coordinative solvents lead to a formation of the dimeric compounds A, whereas, the monomeric complexes B are obtained in the presence of Lewis bases (Scheme) [2]. Recently, a bis(phenolate) ligand variation with an additional functionality has been used as a platform for the preparation of new aluminum complexes. Bridges of the bis(phenolate) moiety containing sulfur and nitrogen atoms act as additional coordination sites and are able to increase the coordination number of aluminum atoms. The monometallic structures C with five-coordinate aluminum atoms are favored in reactions of 2,2'-tiobis(4,6-dialkylphenol)s with trialkyl aluminum in Lewis basic solvents [3]. It was found by Sobota et al. [4] that, in non-coordinative solvents, the aluminum complexes of tridentate (OSO) bis(phenolate) ligands (D) are dimeric. Using various ratios of reagents and reaction conditions, Okuda et al. [3b] and Munoz-Hernandez et al. [3a] obtained unusual dimetallic complexes E with six- and four-coordinate aluminum atoms (Scheme 1).

In contrast to the dimeric aluminum tiobis(phenoxide)s **D**, aluminum complexes with $CH_2N(R')CH_2$ -bridged bis(phenolato) ligands **F** are monomeric in non-coordinative solvents [5]. An

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Two aluminum complexes with OOO-tridentate bis(phenolate) ligands have been synthesized and structurally characterized. Reactions of 2,2'-oxobis(phenol) with ^fBu₃Al and Ph₃Al produced the dimeric compounds [RAl(OC₆H₄OC₆H₄O)]₂ [R = ^fBu (1), R = Ph (2)]. A single crystal X-ray analysis showed that molecules of the compounds contained two 2,2'-oxobis(phenolate) units and two five-coordinate aluminum atoms, each bonded to four oxygen atoms and to one carbon atom.

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application of the bis(phenolato)bis(amine) ligand precursors, bearing an additional nitrogen atom in the dative arm bonded to the bridged nitrogen, resulted in the formation of a series of monomeric aluminum compounds **G** (Scheme 1). A bowl-shaped environment around the metal center prevented complexes from dimerization [6].

In comparison with aluminum tio- and aminobis(phenoxide)s, gallium and indium complexes are rare [7]. Oxygen-linked bis(phenolate) ligands have been not yet used in the chemistry of the group 13 metals. The only tridentate bis(phenolato) ligands of the (OOO)-type, used for synthesis of groups 1 and 4 metal complexes, are ligands with a flexible CH₂OCH₂ link between the phenolato functions [8]. We describe herein the synthesis and characterization of aluminum 2,2'-oxobis(phenolate)s as the complexes supported by (OOO)-tridentate ligand, in which aromatic rings are directly linked by the oxygen atom.

2. Results and discussion

Treatment of 2,2'-oxobis(phenol) with one molar equivalent of R₃Al in toluene produces dimeric compounds $[RAl(OOO)]_2$ [where (OOO) = 2,2'-oxobis(phenolate) unit, R = ^{*t*}Bu (1), R = Ph (2)], as shown in Scheme 2.

The compounds were characterized by NMR spectroscopy, elemental analyses and melting point measurements. The molecular structures were determined on the basis of an X-ray diffraction study and are shown in Figs. 1 and 2. Data collection and structure analyses are listed in Table 1. Selected interatomic distances (Å) and angles (°) for molecules are shown in Table 2. Crystals of **1** and **2** suitable for X-ray structure determination were grown from



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n-hexane and toluene solutions at -10 °C, respectively. The molecular studies showed that the molecules of **1** and **2** were dimeric and centrosymmetric. They are composed of two 2,2'-oxobis(phenolate) units, two five-coordinate aluminum atoms and two groups (^tBu or Ph) bonded to the aluminum atoms. The central planar four-membered ring is composed of two aluminum atoms and two bridging oxygen atoms of the bis(phenolate) ligands. This type of bridging has been observed in phenolate aluminum compounds [9]. Each of the metal atoms in **1** and **2** is bonded to one carbon



atom and four oxygen atoms; three of them originating from one bis(phenotate) unit, the fourth is a $O_{phenoxide}$ atom of the second bisphenolate unit. The ^tBu and Ph groups are mutually *cis* with respect to the Al_2O_2 core.

In a cell, crystals of **1** contain two kinds of molecules (**A** and **B**) differing in bond lengths and angles. The angles $[O(1A)-Al(1A)-C(13A) 121.81(6)^{\circ}, O(1A)-Al(A1)-O(2A) 116.93(6)^{\circ}, C(13A)-Al(1A)-O(2A) 119.26(6)^{\circ} and O(3A)-Al(1A)-O(2A)' 149.77(5)^{\circ}$ $[O(2B)-Al(1B)-O(1B) 119.79(6)^{\circ}, O(2B)-Al(1B)-C(13B) 121.79(6)^{\circ}, O(1B)'-Al(1B)-C(13B) 116.96(6)^{\circ}, O(1B)'-Al(1B)-O(3B) 149.67(5)^{\circ}]$ indicate that the coordination sphere geometry of the aluminum atom can be regarded as a distorted trigonal bipyramidal arrangement.

In **1A** molecules, the O(3A) and O(2A)' atoms occupy the axial positions and the O(1A), O(2A) and C(13A) atoms define the equatorial sites of the trigonal bipyramid (Fig. 1). In **1B** molecules, the O(1B)' and O(3B) atoms are situated in the axial positions and the O(1B), O(2B) and C(13B) lie in the equatorial sites. The sums of the angles about the oxygen atoms O(3) { \sum [O(3A)] 330.0°, \sum [O(3B)] 333.8°} are less than 360°, which indicates a steric strain in the molecules. The Al-O_{phenoxide} bond lengths [Al(1A)–O(1A) 1.757(1) Å, Al(1A)–O(2A) 1.866(1) Å, Al(1B)–O(1B) 1.853(1) Å, Al(1B)–O(2B) 1.761(1) Å] are similar to those found in other structures of five-coordinate aluminum aryloxides [3–6].

In the compound **2**, one molecule of toluene is present in the formula unit as a crystalline net stabilizing factor. Similarly to **1**, each metal atom is coordinated in a distorted trigonal bipyramidal fashion by O(3) and O(2)' atoms in axial positions and O(1), O(2) and C(13) atoms in equatorial positions [O(1)-Al(1)-O(2) 122.2(1)°, O(1)-Al(1)-C(13) 119.0(2)°, O(2)-Al(1)-C(13) 117.5(2)°, O(2)'-Al(1)-O(3) 151.4(1)°] (Fig. 2). The sum of the angles about the oxygen atom O(3) { $\sum [O(3)]$ 337.6°} indicates a steric strain in the molecule. Bond lengths of Al(1)-O_{phenoxide} and Al(1)-O(3) [2.157(3) Å] in **2** are similar to those of the compound **1**.

The Al(1)–O(3) distances of 2.227(1) Å (1A), 2.177(1) Å (1B) and 2.157(3) Å (2) are significantly longer than those of Al-O_{phenoxide} and longer than typical coordinate Al-O bonds in R₃Al B adducts (where R = alkyl and phenyl groups, B = Lewis base with an oxygen atom) [10] and in aluminum alkoxide complexes with THF, [1i,11]. Since the Al(1)-O(3) distances are much closer to the sum of their covalent radii (1.86 Å) than to the sum of their van der Waals radii (3.57 Å) [12], the Al(1)–O(3) distances are considered to be weak coordinate bonds. Zevaco [1i] found, that the distances between aluminum atoms and non-coordinative carbon atoms linking phenyl rings of a ligand in the dinuclear aluminum para-chloro-bisphenoxide [Al–C 3.796(9) and 3.899(9)Å] (see Scheme 1, structure **A**) are even longer than the sum of their van der Waals radii (3.70 Å). This indicates, that the position of Al(1) and O(3) atoms in 1 and 2 is not due to the arrangement of the phenyl rings and a rigid structure of the 2,2'-oxobis(phenolate) ligand, but instead is forced by the bonding between two atoms. Similar coordinative Al-S bonds were observed in aluminum complexes with sulfide-linked bis(phenolato) ligands [3,4].

The ¹H NMR spectrum of **1** in solution revealed signals of aromatic protons in the range of 7.69–6.71 ppm and a singlet of $(CH_3)_3CAl$ at 0.71 ppm in a 8:9 relative intensity ratio which indicated one bis(phenolate) moiety per one ^tBu group. In the ¹³C NMR spectrum, twelve signals of aromatic carbon atoms and signals of ^tBu group carbon atoms {29.59 [(<u>CH_3)_3C</u>], 15.28 [(CH_3)_3<u>C</u>] ppm} were present. The presence of 12 signals of C_{aromat} indicated a nonequivalence of all carbon atoms of bis(phenolate) unit. It is in agreement with the molecular structure, however, the separated signals of **1A** and **1B** solid-state structures were not observed in the NMR spectra. The NMR spectra of **2** showed the following signals: a multiplet of the aromatic protons (7.73–681 ppm) in the ¹H NMR spectrum and sixteen signals of C_{aromat} atoms [12 signals of



Fig. 1. Molecular structure of [^rBuAl(OOO)]₂: (1A) (top) and (1B) (bottom). Displacement ellipsoids are drawn at the 50% probability level. Hydrogen atoms are omitted for clarity.



Fig. 2. Molecular structure of [PhAl(OOO)]₂ (2). Displacement ellipsoids are drawn at the 50% probability level. Hydrogen atoms and a disordered molecule of toluene are omitted for clarity.

Table 1

Crystal data and data collection parameters for 1 and 2.

	1	$2 \cdot C_7 H_8$
Empirical formula	$C_{32}H_{34}Al_2O_6$	$C_{36}H_{26}Al_2O_6 \cdot C_7H_8$
Formula weight	568.55	700.66
Temperature (K)	100(2)	100(2)
Wavelength (Å)	0.71073	0.71073
Crystal system	Triclinic	Monoclinic
Space group	P-1	P2(1)/n
a (Å)	10.0974(6)	8.962(1)
b (Å)	11.0859(6)	11.911(2)
<i>c</i> (Å)	15.0020(9)	17.386(2)
α(°)	76.813(5)	90
β (°)	76.772(5)	93.83(1)
γ (°)	64.121(6)	90
$V(Å^3)$	1455.0(2)	1851.7(4)
Ζ	2	2
$D_{\rm calc}~({ m g~cm^{-3}})$	1.298	1.257
Absorption coefficient (mm ⁻¹)	0.143	0.126
F (000)	600	732
Crystal size (mm)	$0.2\times0.2\times0.2$	$0.25\times0.10\times0.10$
 Θ range for data collection (°) 	2.66-28.74	2.85-25.00
Index ranges	$-13 \leqslant h \leqslant 13$,	$-10 \leqslant h \leqslant 8$,
	$-14\leqslant k\leqslant 14$,	$-14\leqslant k\leqslant 14$,
	$-20 \leqslant l \leqslant 19$	$-20 \leqslant l \leqslant 20$
Reflections collected	27,433	13,586
Independent reflections	7031 $[R_{int} = 0.0395]$	3255 $[R_{int} = 0.1216]$
Refinement method	Full-matrix least-squares on <i>F</i> ²	Full-matrix least- squares on F ²
Data/restraints/ parameters	7031/0/367	3255/0/211
Goodness-of-fit on F^2	0.646	0.939
Final R indices	$R_1 = 0.0342.$	$R_1 = 0.0589$.
$[I > 2\sigma(I)]$	$wR_2 = 0.0937$	$wR_2 = 0.1396$
R indices (all data)	$R_1 = 0.0626,$	$R_1 = 0.1475$,
. ,	$wR_2 = 0.1072$	$wR_2 = 0.1674$
Max/min of residual	0.297 and -0.272	0.693 and -0.253
electron density		

Table 2

Selected bond distances (Å) and angles (°) for 1 and 2.

	1A	1B	2
Bond length			
Al-O _{phenoxide}	1.757(1)	1.761(1)	1.757(3)
	1.866(1)	1.853(1)	1.839(3)
	1.884(1)	1.890(1)	1.883(3)
Al(1)-O(3)	2.227(1)	2.177(1)	2.157(3)
Al(1)-C(13)	1.967(2)	1.970(2)	1.952(4)
Bond angles			
O(1)-Al(1)-O(2)	116.93(6)	119.79(6)	122.2(1)
O(2)-Al(1)-C(13)	119.26(6)	121.79(6)	117.5(2)
O(1)-Al(1)-C(13)	121.81(6)	116.96(6)	119.0(2)
O(2)'-Al(1)-O(3)	149.77(5)	149.67(5)	151.4(1)
C(12)-O(3)-C(6)	117.5(1)	119.0(1)	121.0(3)
C(12)-O(3)-Al(1)	107.96(8)	106.26(9)	110.7(2)
C(6)-O(3)-Al(1)	104.55(8)	108.52(8)	105.9(2)

carbon atoms of the oxobis(phenolate) unit and four signals of carbon atoms of the PhAl groups], 29.59 [(CH_3)₃C], 15.28 [(CH_3)₃C] ppm in the ¹³C NMR spectrum, which is in accordance with the solid-state structure.

Summarizing, tri-*tert*-butyl- and triphenyl aluminums react with 2,2'-oxobis(phenyl) to yield novel dimeric compounds supported by a tridentate (OOO) ligand. The structural analysis showed a strain in the molecules of **1** and **2** probably due to rigid structure of the ligand in which aromatic rings are directly linked by an oxygen atom. The coordination number of five-coordinate

aluminum atoms is higher in comparison with that of typical aluminum bisphenoxides **A** and **B** (Scheme 1) due to the formation of additional bonds between the oxygen atoms linking aromatic rings in the ligands and aluminum atoms.

3. Experimental

3.1. Materials and instrumentation

All manipulations were carried out using standard Schlenk techniques under an inert gas atmosphere. Solvents were distilled over a blue benzophenone–K complex. tBu3Al·OEt2 and Ph3Al·THF [¹³C NMR (CDCl3) δ 138.24, 128.32, 127.66, 127.22 (C_{aromat}), 72.25, 25.36 (CTHF) ppm] were synthesized as described in the literature [13,14], as well as 2,2'-oxobis(phenol) [15]. ¹H NMR spectra were obtained on a Mercury-400BB spectrometer. Chemical shifts were referenced to the residual proton signals of CDCl₃ (7.26 ppm). Elemental analyses of **1** and **2** were obtained on a Perkin–Elmer 2400 analyzer.

3.2. Synthesis of [^tBuAl(000)]₂ (**1**)

A solution of ${}^{t}Bu_{3}Al \cdot OEt_{2}$ (0.544 g, 2 mmol) in 10 cm³ of Et₂O was injected to a stirred solution of 2,2'-oxobis(phenol) (0.404 g, 2 mmol) in Et₂O (15 cm³) at -76 °C. The reaction mixture was allowed to warm to room temperature. After 24 h a white solid of the pure compound **1** precipitated from the post-reaction mixture (yield 0.505 g, 89%). At 234 °C the compound **1** undergoes the decomposition becoming a gray solid.

¹H NMR (CDCl₃) δ 7.69 (dd, ³*J*_{H-H} = 9,9 Hz, 4*J*_{H-H} = 1,8 Hz, 2H, H_{aromat}), 7.54 (dd, 2H, ³*J*_{H-H} = 9,4 Hz, 4*J*_{H-H} = 1,2 Hz, H_{aromat}), 7.43 (dd, 2H, ³*J*_{H-H} = 9,9 Hz, 4*J*_{H-H} = 1,8 Hz, H_{aromat}), 7.34–7.28 (m, 2H, H_{aromat}), 7.16–7.00 (m, 6H, H_{aromat}), 6.85–6.71 (m, 2H, H_{aromat}), 0.71 (s, 18H, (C<u>H</u>₃)₃Al). ¹³C NMR (CDCl₃) δ 151.60, 147.28, 145.86, 145.40, 127.37, 126.75, 122.56, 120.73, 118.67, 118.20, 117.47, 115.78 (C_{aromat}), 29.59 [(<u>C</u>H₃)₃C], 15.28 [(CH₃)₃C] ppm.

Elemental anal.: Calcd. for $C_{32}H_{34}Al_2O_6$: C, 67.54; H, 5.98. Found: C, 67.20; H, 6.30 wt.%.

3.3. Synthesis of [PhAl(000)]₂ (2)

A solution of Ph₃Al·THF (0.660 g, 2 mmol) in 20 cm³ of Et₂O was injected to a stirred solution of 2,2'-oxobis(phenol) (0.404 g, 2 mmol) in Et₂O (15 cm³) at -76 °C. The reaction mixture was allowed to warm to room temperature. The solvent was removed *in vacuo* from the post-reaction mixture. A white solid of the compound **2** was quantitatively obtained. Mp.: >300 °C.

 ^{1}H NMR (CDCl₃) δ 7.73–6.81 (H_{aromat}, m) ppm. ^{13}C NMR (CDCl₃) δ 117.21, 117.62, 118.02, 118.95, 119.81, 122.82, 127.34, 127.62, 128.33, 128.68, 128.87, 137.43, 145.91, 146.12, 147.93, 151.87 (C_{aromat}) ppm.

Elemental anal.: Calcd. for C₃₆H₂₆Al₂O₆: C, 71.05; H, 4.28. Found: C, 70.71; H, 4.49 wt.%.

Colorless crystals of $2 \cdot C_7 H_8$ for X-ray diffraction studies were obtained from a toluene solution at -10 °C.

3.4. X-ray crystal structure determinations

Determination of the crystal structures was performed on a KUMA CCD κ -axis diffractometer with graphite-monochromated Mo K α radiation. A crystal of **1** was positioned at 62.25 mm from the KM4CCD camera and 2000 frames were measured at 0.6° intervals with a counting time of 15 s. A crystal of **2** was positioned at 62.25 mm from the KM4CCD camera and 600 frames were measured at 1.0° intervals with a counting time of 35 s. Data reduction

and analysis was carried out with the Kuma Diffraction programs. The data were corrected for Lorentz and polarization effects and multi-scan absorption correction was applied [16]. The structures were solved by direct methods [17] and refined using SHELXL [18]. The non-hydrogen atoms were refined anisotropically. The hydrogen atoms were located from a difference map and refined isotropically. The atomic scattering factors were taken from the International Tables [19].

The X-ray structures were measured in the Crystallography Unit of the Physical Chemistry Laboratory at the Chemistry Department of the University of Warsaw.

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Appendix A. Supplementary material

CCDC 805949 and 805950 contain the supplementary crystallographic data for 1 and 2. These data can be obtained free of charge from The Cambridge Crystallographic Data Center via www.ccdc.cam.ac.uk/data_request/cif.

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