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Synthesis and structural characterization of polymeric dimethylaluminum phenylthiolate

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This work is dedicated to the outstanding contributions of Professor Stanisław Pasynkiewicz to the field of organoaluminum chemistry during his long career and to the unflagging support he has provided to his many students and co-workers in carrying out this work

Abstract

The reaction of trimethylaluminum with thiophenol forms the compound $[Me_2Al(\mu\text{-}SPh)]_n$ in good yield. The compound has been characterized by ^1H, ^{13}C and variable-temperature NMR (VT-NMR) spectroscopy, mass spectroscopy, and by cryoscopic molecular-weight determination. The solid-state structure has been determined by single-crystal X-ray crystallography and was assigned to the monoclinic space group Pc (no. 7) with cell constants $a = 9.2499(12)$, $b = 7.1344(10)$, $c = 7.1947(11)$ Å, $\beta = 100.328(3)°$ and $Z = 2$. The refinement converged with $R_1 = 7.24\%$ ($wR_2 = 15.4\%$) for 581 ($I \geq 2\sigma(I)$) observed reflections and was described in terms of an infinite chain polymer with phenyl thiol groups and dimethylaluminum groups alternating. The average Al–S distance is 2.36 Å. VT-NMR shows that the compound exhibits some fluxional behavior, which affects the *ortho* protons of the phenyl ring. Cryoscopic molecular-weight measurements show that the aggregation state is a function of concentration, and it has been proposed that two species, dimer and trimer, exist in solution. © 2000 Elsevier Science S.A. All rights reserved.

Keywords: Aluminum; Phenylthiolate; Polymer

1. Introduction

In general, organometallic compounds of the formula $[R_2M(\mu\text{-}ER')]_n$ (M = Al, Ga, In; E = S, Se, Te) are simple oligomers with aggregation states ranging from dimer to tetramer, with the degree of aggregation dependent on the size of the organic groups on the metal and on the chalcogen [1]. In certain cases, solid-state polymeric structures have been reported for Group 13/16 compounds. The first structurally characterized polymeric compound of the class $[R_2M(\mu\text{-}ER')]_n$ was reported by Brauer and Stucky for the structure of dimethylaluminum methylthiolate, which crystallized as an infinite chain polymer [2]. Other polymeric Group 13/16 compounds include $[(Me_2Al)_2S_2C_6H_4]_n$ [3], which has one Me_2Al group coordinated to the two sulfur atoms and a second Me_2Al unit bridging 1,2-benzenedithiolate groups, $[MeIn(SePh)(\mu\text{-}SePh)]_n$ [4], which has both bridging and pendant SePh units in a helical coil, and $[In(\mu\text{-}SePh)_3]_n$ [5], which consists of six-coordinate indium atoms bridged by three SePh units. Our efforts to synthesize other organoaluminum thiolate species have resulted in the structural characterization
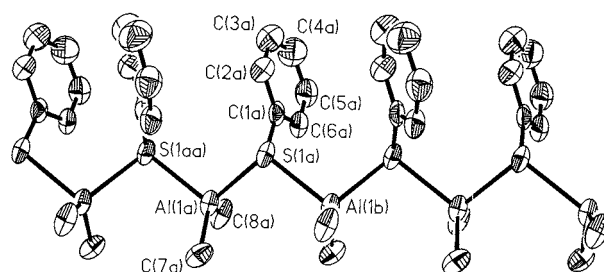

Fig. 1. A portion of the $[Me_2Al(\mu\text{-}SPh)]_n$ chain (50% thermal ellipsoids). Hydrogen atoms have been omitted for clarity.

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Table 1
Selected bond distances (Å) and angles (°) for $[Me_2Al(\mu\text{-SPh})]_n$

Bond distances	
S(1)–C(1)	1.766(13)
S(1)–Al(1)	2.345(3)
S(1)–Al(1) # 1	2.378(3)
Al(1)–C(8)	1.944(10)
Al(1)–C(7)	1.961(11)
Al(1)–S(1) # 2	2.378(3)
Bond angles	
C(1)–S(1)–Al(1)	105.6(4)
C(1)–S(1)–Al(1) # 1	111.9(4)
Al(1)–S(1)–Al(1) # 1	102.27(12)
C(8)–Al(1)–C(7)	122.6(5)
C(8)–Al(1)–S(1)	113.0(4)
C(7)–Al(1)–S(1)	104.3(4)
C(8)–Al(1)–S(1) # 2	100.4(3)
C(7)–Al(1)–S(1) # 2	113.5(4)
S(1)–Al(1)–S(1) # 2	101.10(12)

of the third polymeric aluminum thiolate of the formula of $[R_2M(\mu\text{-ER}')]_n$. The solid-state structure of dimethyl-aluminum phenylthiolate was found to be a chain with alternating dimethylaluminum and phenyl thiol groups. This result was in sharp contrast to the more common solid-state configuration of discrete oligomeric units for diorganoaluminum thiolates.

2. Results and discussion

The solid-state structure of dimethylaluminum phenylthiolate was found to be polymeric with phenyl thiol groups serving as bridges between dimethylaluminum groups. A thermal ellipsoid drawing of the compound is given in Fig. 1. The average Al–S bond distance is 2.36 Å, the average Al–C bond distance is 1.95 Å, and the average S–C bond distance is 1.77 Å, all of which are typical for aluminum thiolates. Selected bond distances and angles are presented in Table 1. Data for a variety of these species are listed in Table 2 for comparison. The position of the phenyl ring bound to sulfur alternates from left to right to left, as viewed down the polymer axis. The pseudo-torsion angle between adjacent phenyl rings is 93.2° (Fig. 2(A)). The S–Al–S and Al–S–Al angles are 101.1 and 102.1°, respectively.

These angles are 5–10° greater than those found in dimeric aluminum thiolate derivatives, where they are constrained to an average of approximately 90°, but nearly identical to those observed in $[Me_2Al(\mu\text{-SMe})]_n$ [2] and in the range observed for trimeric and tetrameric derivatives, where the angles are less restricted.

Another aspect of the structure is seen in Fig. 2(B), which shows a view of the chain perpendicular to its axis. The adjacent phenyl groups are twisted 23° with respect to one another, resulting in the staggered configuration observed. This rotation about the S–C
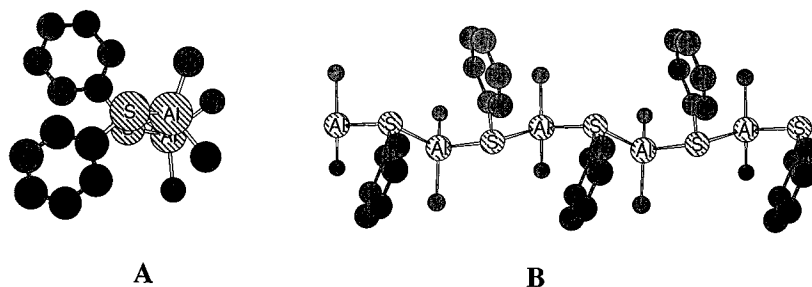
Table 2
Comparison of $[Me_2Al(\mu\text{-SPh})]_n$ with selected aluminum thiolates

Compound	Al–S (Å)	Al–C (Å)	S–C (Å)	Al–S–Al	S–Al–S	Sum@S	Ref.
$[Me_2Al(\mu\text{-SPh})]_n$	2.345	1.961	1.766	102.1	101.1	319	This work
Linear chain	2.378	1.944					
$[Me_2Al(\mu\text{-SMe})]_n$	2.345	1.945	1.806	103.0	100.1	310	[2]
Linear chain	2.351	1.944					
$[Me_2Al(\mu\text{-SMe})]_2$	2.370	1.945	1.811	94.5	85.5	306.7	[6]
Planar dimer [a]							
$[Mes_2Al(\mu\text{-SPh})]_2$	2.395	1.968	1.781	93.6	86.4	319.5	[1]
Planar dimer							
$[Me_2Al(\mu\text{-SC}_6F_5)]_2$	2.413	1.932	1.762	86.88	89.41	300.98	[7]
Butterfly dimer	2.397	1.953		87.31	89.61	308.01	
$\{Me_2Al[\mu\text{-S(2-}t\text{-BuC}_6H_4)]\}_3$	2.353	1.95 (this work)	1.77	115.0	100.6	323.8	[1]
Trimer chair		1.923 [7]		123.2	88.8	338.5	
				114.6	99.1	324.6	
$\{Me_2Al[\mu\text{-S(2-Me}_3\text{Si)C}_6H_4)]\}_3$	2.364	1.953 (this work)	1.776	120.1	100.8	335.7	[1]
Trimer chair		1.936 [7]		113.5	99.2	320.6	
				114.9	89.4	325.4	
$\{i\text{-Bu}_2Al[\mu\text{-S(2-}i\text{-PrC}_6H_4)]\}_3$	2.364	1.939 (this work)	1.803	115.8	99.2	324.6	[1]
Trimer chair		1.929 [7]		114.2	99.9	319.9	
				122.5	101.5	337.1	
$\{i\text{-Bu}_2Al[\mu\text{-S(2,4,6-}i\text{-Pr}_3C_6H_2)]\}_3$	2.37	1.934	1.80	127.6	89.1	360.0	[1]
Twist boat				126.8	93.2	357.0	
				131.5	96.1	356.0	
$\{Me_2Al[\mu\text{-S(2,6-Me}_2C_6H_3)]\}_4$	2.363	1.943	1.789	126.9	96.4	342.8	[1]
Tetramer				130.7	99.5	348.3	

[a] Structure determined in gas phase.

Fig. 2. A Chem3D view of [Me$_2$Al(μ-SPh)]$_n$ parallel to the axis of the chain (A) and perpendicular to the axis of the chain (B).

bond may result from a reduced interaction between the π systems on the phenyl rings or may result from improved crystal packing.

Another minor difference between the structures is in the sum of the three angles about the sulfur atom, which is 319.8° for [Me$_2$Al(μ-SPh)]$_n$ and 310.3° for [Me$_2$Al(μ-SMe)]$_n$. The larger sum of angles at sulfur for the phenyl thiolate in comparison to the methyl thiolate is most likely due to the increased steric requirements of the phenyl ring, which forces the sulfur atom into a more planar configuration.

This study further demonstrates the importance of the steric requirements of the ligand on the aggregation state [1]. For the Me$_2$Al derivative with a phenyl substituent on sulfur, a polymer is observed. When a 2,6-dimethylphenyl substituent is present the solid-state structure becomes tetrameric [1], and when larger groups, such as *t*-Bu, *i*-Pr, or –SiMe$_3$, are placed in the 2-position, the molecular unit shifts to trimer in the solid state. Similarly, if one places a bulkier group such as a 2,6-dimethylphenyl group on the aluminum atom, the molecular unit shifts to the dimeric structure. This indicates that changing the steric requirements of the organic group on either the sulfur or aluminum, from a small group to a large group, changes the steric interactions in the molecule and changes the size of the most stable aggregate found in the solid state. Further comparison can be made with the dimethylaluminum pentafluorophenyl thiolate derivative, which was shown to exist as a puckered dimer in the solid state [7]. This shows that changing the electronic properties as well as the steric interactions of the organochalcogen moiety can also have a significant effect on the solid-state structure.

It is not yet completely understood why [Me$_2$Al(μ-SPh)]$_n$ crystallizes as a polymer rather than as a dimer or other oligomer, but it is clear that the differences in energy between the different aggregates — dimer, trimer, tetramer, and polymer — is small. As noted previously, predicting the aggregation state for Group 13/16 compounds remains difficult [1]. Charting the balance between entropy, steric interactions, ring strain, and lattice energy remains the primary obstacle to the successful prediction of the solid-state degree of oligomerization in these compounds.

2.1. NMR studies

The ^{1}H-NMR spectrum of [Me$_2$Al(μ-SPh)]$_n$ shows a single line for the methylaluminum protons in solution even at -90°C. Fig. 3 shows the -50°C ^{1}H-NMR spectrum of [Me$_2$Al(μ-SPh)]$_n$, which displays the expected signals for the methylaluminum protons, the *meta/para* multiplet and the *ortho* doublet of the phenyl thiolate group. However, Fig. 3 also shows that as the temperature is increased the *ortho* doublet broadens and forms a single broad peak at room temperature. As the temperature is increased to 60°C this line continues to broaden, while increasing the temperature to 80°C results in a slight sharpening of the line. All other regions of the spectrum remain unchanged with changes in temperature or concentration.

Normally, the *ortho* proton signal appears as a doublet of doublets, as would be observed in an AB$_2$X$_2$ or ABB'XX' system with three and four bond coupling constants of 7 and 1.5 Hz, respectively. Simulation of the -50°C spectrum for the aromatic region of [Me$_2$Al(μ-SPh)]$_n$ was performed by using the NMR simulation program, WINDNMR [8]. A five-spin model representing the two *ortho*, two *meta* and one *para* proton was used with chemical shifts and coupling constants of: ortho, 7.55 ppm, $^3J_{o-m} = 7.0$ Hz; *meta*, 6.86 ppm, $^3J_{m-o} = 7.0$, $^3J_{m-p} = 7.5$ Hz; *para*, 6.83 ppm, $^3J_{p-m} = 7.5$ Hz; $v_{1/2} = 3.50$ Hz. With such a broad $v_{1/2}$, there was little change in the spectrum when four-bond coupling constants (0.3 Hz) were added.

NMR data were collected over a wide range of concentrations. Fig. 4 displays the 25°C NMR spectra for the aromatic region of [Me$_2$Al(μ-SPh)]$_n$ at several concentrations and shows that at concentrations below 0.29 M (based on monomer) only a broad signal for the *ortho* protons was observed; however, at higher concentrations (> 0.60 M) the broad signal sharpened and began to form a doublet. The ^{13}C{^{1}H} signal for the *ortho* position (134 ppm) is also shown to be broad at low concentrations, while increasing the concentration serves to sharpen the signal. One sample of very low concentration (1.7×10^{-2} M in monomer) was examined by ^{1}H-NMR spectroscopy at 80°C, which showed a very broad doublet for the *ortho* proton signal.
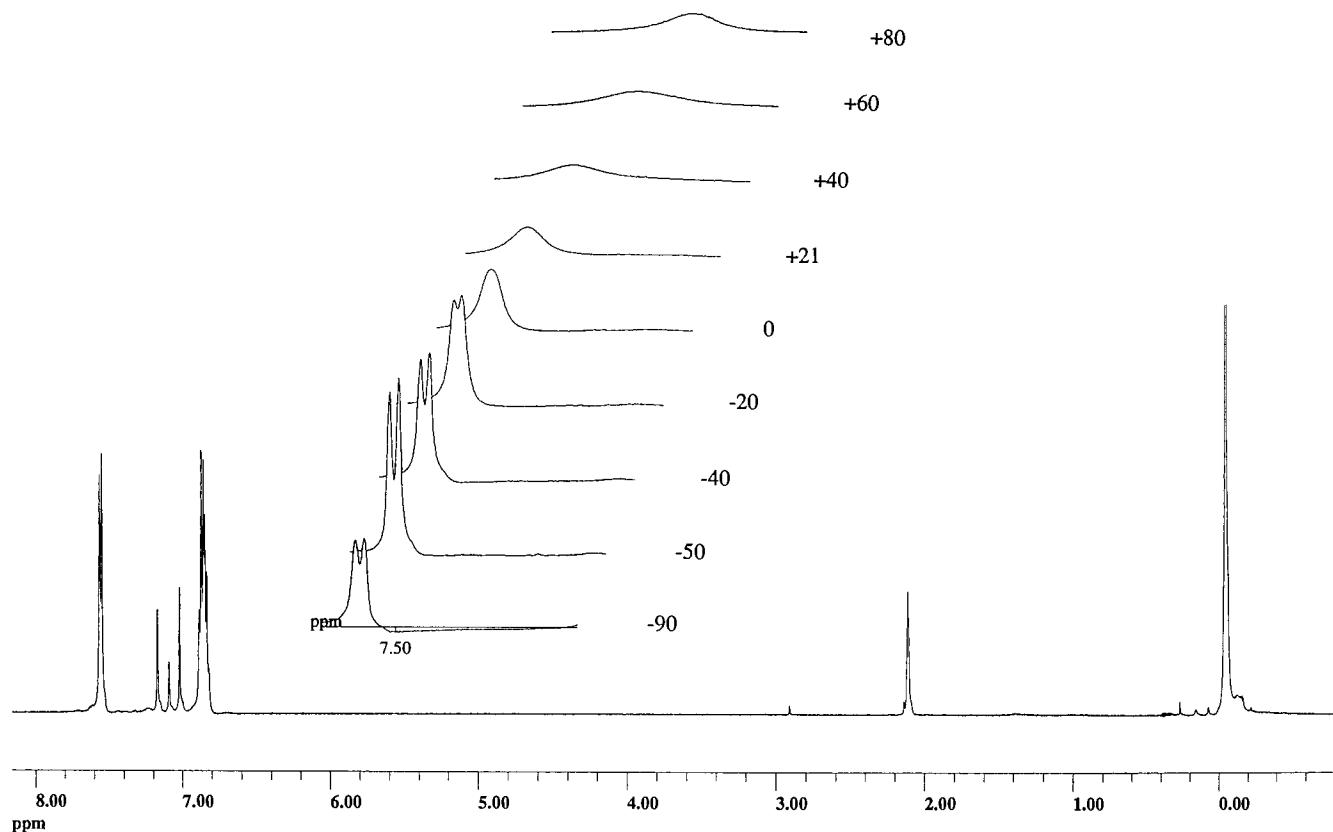
Fig. 3. ^{1}H-NMR spectrum (-50°C, 500 MHz) of [Me$_2$Al(μ-SPh)]$_n$ in toluene-d_8 (0.055 M based on dimer). Inset are the VT spectra (-90 to $+80$°C) for the *ortho* protons on the aromatic ring.

For each temperature and concentration, homonuclear decoupling of the *ortho* signal (broad singlet or doublet) resulted in a simplification of the *meta/para* multiplet. Under conditions where a doublet was observed for the *ortho* signal, homonuclear decoupling of the *meta/para* multiplet resulted in a collapse of the doublet to a singlet. However, under conditions where a single broad line was observed for the *ortho* signal, no changes were observed for the broad signal when the *meta/para* multiplet was decoupled. Further evidence of coupling between *ortho* and *meta* protons was shown in a -50°C 2D-COSY NMR experiment, which showed the cross-peaks due to coupling of the *ortho* protons to the *meta* protons.

From the NMR data, it is clear that there is a process occurring in solution, which appears to affect only the line shape of the *ortho* proton signal and is both temperature and concentration dependent. This suggests that an exchange between oligomers may be occurring where one oligomer is favored at high temperatures and low concentrations, while the other is favored at low temperatures and high concentrations.

In order to determine which oligomers were present in solution, cryoscopic molecular-weight studies of [Me$_2$Al(μ-SPh)]$_n$ were performed in benzene. The results gave average molecular weights between dimer

(332.12 g mol^{-1}) and trimer (498.18 g mol^{-1}). Higher sample concentrations resulted in higher molecular weights. This indicates that the compound exists as an equilibrium mixture of oligomers, which are most likely
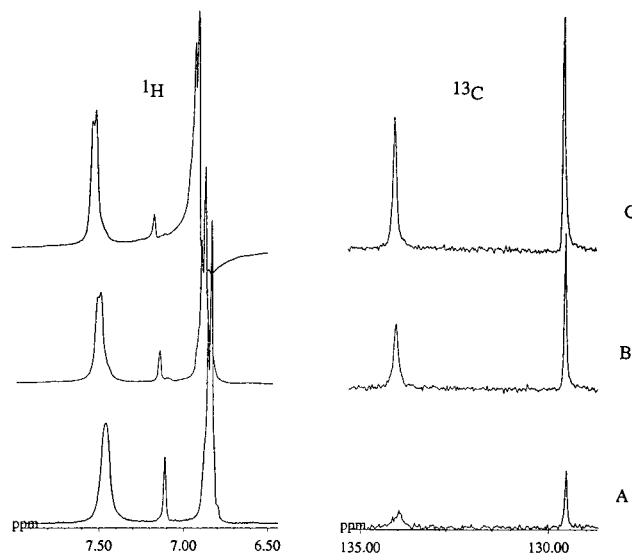

Fig. 4. NMR spectra (25°C ^{1}H and ^{13}C$\{{}^1$H$\}$ 300 and 75 MHz) of the aromatic region of [Me$_2$Al(μ-SPh)]$_n$ as a function of concentration in benzene-d_6.

dimer and trimer, $2[Me_2Al(\mu\text{-}SPh)]_3 = 3[Me_2Al(\mu\text{-}SPh)]_2$ with $K_{eq} = [\text{dimer}]^3[\text{trimer}]^{-2}$. The equilibrium constant calculated for this process from the molecular weight data is 2×10^{-3} M 1^{-1}. Unfortunately, this method does not yield very precise measurements, but it is clearly sufficient to establish the equilibrium process.

One possible explanation for the unusual NMR observations for $[Me_2Al(\mu\text{-}SPh)]_n$ may be that there is a fast interconversion between the dimer and trimer species. For the methylaluminum signal, this exchange may lead to an averaged signal at all temperatures. For the *meta* and *para* protons distant from the ring, the signals may not be significantly affected by the aggregation state. The *ortho* protons, however, are close to the metal–chalogen ring and may be substantially affected by the change in environment. At low temperature or high concentration, it is likely that a single aggregate, the trimer, dominates and the *ortho–meta* coupling can be observed. The location of the *ortho* protons would subject them to the greatest change in chemical environment upon conversion from dimer to trimer compared to the *meta* and *para* protons. Cooling of the solution or increasing the concentration may slow the interconversion process or allow one oligomer (trimer) to predominate such that the coupling to the *ortho* protons can be observed. Conversely, heating the solution or lowering the concentration may increase the rate of interconversion or shift the equilibrium in favor of the other oligomer (dimer).

Because the dimer–trimer exchange is fast, the signal observed for the methylaluminum protons would be the time-averaged signal for the dimeric and trimeric species and would appear unaffected by changes in temperature and concentration except for small changes in chemical shift. Close examination of the methylaluminum peak at various temperatures and concentrations does show small changes in the chemical shift of this peak.

Simulation of the most concentrated 25°C 300 MHz ^{1}H-NMR spectrum of $[Me_2Al(\mu\text{-}SPh)]_n$ gave chemical shifts and coupling constants of: *ortho*, $\delta = 7.468$ ppm $^3J_{o-m} = 7.0$ Hz; *meta*, $\delta = 6.865$ ppm, $^3J_{m-o} = 7.0$, $J_{m-p} = 7.5$ Hz; *para*, 6.842 ppm, $^3J_{p-m} = 7.5$ Hz. As in the 500 MHz simulation, including the four-bond coupling constants in the simulation had little effect on the spectrum. Comparison between the spectrum determined at 25°C and high concentration with the simulated spectrum showed that while a good fit was obtained for the *meta–para* multiplet, the simulated *ortho* doublet was sharper than the signal determined experimentally. This broadening indicates that exchange is still taking place in the sample. Based on molecular-weight studies, at these relatively high concentrations, the trimeric oligomer makes up approximately 90–95% of the species present in solution and so the amount of dimer exchanging with trimer is greatly reduced, thereby allowing the coupling to be observed.

3. Experimental

3.1. General procedures

All manipulations were performed in the absence of water and air using standard Schlenk line and dry box techniques [9]. Argon was purified by passing it through a series of columns containing Deox catalyst (Alfa), calcium sulfate and phosphorous pentaoxide. All solvents used were dried using standard techniques, and all glassware was oven-dried. Trimethylaluminum (2.0 M solution in hexane) was obtained from Aldrich and used as received. Thiophenol (Aldrich) was freshly distilled prior to use. Special care should be taken when handling any compounds containing chalcogen as they are foul smelling and toxic. ^{1}H- and ^{13}C-NMR spectra were recorded on Gemini-300 and Varian-U500 NMR spectrometers. The chemical shifts were referenced to the residual proton line from benzene-d_6 ($\delta = 7.15$ ppm for ^{1}H; $\delta = 128.0$ ppm for ^{13}C). Variable-temperature NMR spectra were obtained on U-500 and GN-300 spectrometers in toluene-d_8 (referenced to the residual proton at 2.09 ppm). Mass spectra were obtained on a Kratos MS-80 mass spectrum analyzer in EI mode. Elemental analysis was performed by Galbraith Laboratories, Knoxville, TN. Melting points were measured on a Haake–Buchler melting point apparatus and are uncorrected. Solution molecular weight studies were performed cryoscopically in benzene. Temperatures were measured every 10 s using a Keithley model 197A microvoltmeter equipped with a mV to °C converter.

3.1.1. Synthesis of [Me2Al(μ-SPh)]n

Thiophenol (1.06 g, 9.6 mmol) was dissolved in pentane (20 ml). To this was added 4.8 ml of 2.0 M Me_3Al dropwise at room temperature (r.t.). The reaction mixture was stirred until evolution of gas was no longer observed. The resulting solution was placed in the freezer for 72 h, during which time a colorless powder deposited. The remaining solution was decanted off and the solid dried under vacuum, giving $[Me_2Al(\mu\text{-}SPh)]_n$ in 90% yield. Crystals of the compound were grown at r.t. from a saturated pentane solution yielding colorless plates. X-ray crystallography shows that the compound crystallizes as a polymer chain. M.p. 126–128°C. Anal. Calc.: C, 57.81; H, 6.67. Found: C, 54.51; H, 6.56%. ^{1}H in C_6D_6 (7.113) 7.46 (broad s, 2H, SC$_6H_5$), 6.82 (m, 3H, SC$_6H_5$), -0.192 (s, 6H AlMe_2) ^{13}C[^{1}H] 134.0, 129.65 (SC_6H_5), -7.427 (Al(CH_3)$_2$. Mass spec. Frag., Mass (% rel. int.) $Me_6Al_4S_3Ph_3$, 525(0.3); $Me_6Al_3S_3Ph_3$, 498(0.2); $Me_5Al_3S_3Ph_3$, 483(3.8); $Me_4Al_3S_3Ph_3$, 468(0.5); $Al_3S_3Ph_3$, 408(0.3); $Me_5Al_3S_3Ph_2$, 405(0.6); $Me_4Al_3S_3Ph_2$, 391(2.4); $Me_4Al_2S_2Ph_2$, 333(4.2), 332(0.1); $Me_3Al_2S_2Ph_2$, 317(93.7); $Me_2Al_2S_2Ph_2$, 303(5.1), 302(3.8), 301(22.7); $Al_2S_2Ph_2$, 272(1.2); $Me_4Al_2S_2Ph$, 255(2.6); $Me_3Al_2S_2Ph$, 240(0.7),

Table 3
Selected experimental parameters for [Me$_2$Al(μ-SPh)]$_n$

Empirical formula	C$_8$H$_{11}$AlS
Formula weight (amu)	166.21
Crystal system	Monoclinic
Space group	*Pc* (no. 7)
Unit cell dimensions	
a (Å)	9.2499(12)
b (Å)	7.1344(10)
c (Å)	7.1947(11)
β (°)	100.328(3)
V (Å^3)	467.10(11)
Z	2
$D_{calc.}$ (Mg m^{-3})	1.182
$F(000)$ (electrons)	176
Final R indices [$I>2\sigma(I)$]	$R_1 = 0.0724$,
	$wR_2 = 0.1547$
R indices (all data)	$R_1 = 0.1166$,
	$wR_2 = 0.1697$
Goodness-of-fit on F^2	0.962

239(6.5); Me$_2$Al$_2$S$_2$Ph, 225(10.7); Me$_4$Al$_2$SPh, 223(20.1); Me$_3$Al$_2$SPh, 209(8.4), 208(0.7), 207(9.1); Me$_2$AlSPh, 166(61.9); MeAlSPh, 151(100); AlSPh, 137(10.5), 136(1.9); Al$_2$S$_2$, 118(0.4); Me$_2$AlS, 89(2.1); MeAlS, 74(5.6); AlS, 59(1.5); Me$_2$Al, 57(92.3).

3.2. X-ray structure determination of [Me$_2$Al(μ-SPh)]$_n$

Crystals of [Me$_2$Al(μ-SPh)]$_n$ were obtained as described in the Section 3. Data for [Me$_2$Al(μ-SPh)]$_n$ were collected at r.t. on a Siemens P4/CCD SMART/SAINT system [10]. Frames were collected at a distance of 5 cm at 10 s/frame. Unit cell constants were refined in SAINT. Structure solution of [Me$_2$Al(μ-SPh)]$_n$ was performed using direct methods from SHELXS-90 [11], which located the heavy atoms as well as some of the carbon atoms. Structure refinement (full-matrix least squares) was obtained using SHELXL-93 [12], which allowed for the anisotropic refinement of the remaining non-hydrogen atoms. Hydrogen atoms were placed in calculated positions with a C–H bond distance of 0.95 Å and were assigned isotropic thermal parameters based on the thermal parameters of the carbon atoms to which they were bound. [Me$_2$Al(μ-SPh)]$_n$ crystallizes in the monoclinic space group *Pc* (no. 7). The structure converged with final R indices of $R_1 = 7.24\%$, $wR_2 = 15.47\%$ for ($I>2\sigma(I)$) and $R_1 = 11.66\%$, $wR_2 = 16.97\%$ for all data. Selected crystallographic parameters are given in Table 3.

4. Supplementary material

Complete crystal data, bond distances and angles, anisotropic displacement parameters for all non-hydrogen atoms, atomic coordinates and isotropic thermal parameters for all non-hydrogen atoms, and hydrogen atom coordinates and isotropic displacement parameters have been deposited with the Cambridge Crystallographic Data Center (CCDC). These data can be obtained free of charge from The Director, Cambridge Crystallographic Data Center, 12 Union Road, Cambridge, CB2 1EZ, UK (Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam. ac.uk), citing the deposition number CCDC 135310, the authors and the reference.

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