Five-Coordinate Aluminum Amides

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In the present manuscript we will detail the first use of the Salen(^{t}Bu)H_2 ligands with the group 13 elements. This includes the compounds of formula Salen(^{t}Bu)AlCl (where Salen = Salen(*N*,*N*′-ethylenebis(3,5-di-*tert*-butylsalicylideneimine)) (**1**), Salpen (*N*,*N*′-propylenebis-(3,5-di-*tert*-butylsalicylideneimine)) (**2**), Salophen (*N*,*N*′-benzenylidenebis(3,5-di-*tert*-butyl-salicylideneamine)) (**3**), and Salomphen (*N*,*N*′-(3,4-dimethylbenzenylidene)bis(3,5-di-*tert*-butylsalicylideneimine)) (**4**)) and the novel five-coordinate monomeric amides of formula LAlNRR′ (where L = Acen (*N*,*N*′-ethylenebis((^{t}Bu)salicylidene(methyl)imine); R, R′ = H, Ph (**5**); R = R′ = $SiMe_3$ (**6**)) and L = Salen (R, R′ = H, ^{t}Bu (**7**), H, Ph (**8**), and H, Dipp (2,6-diisopropylphenyl) (**9**); R = R′ = Me (**10**), Et (**11**), and $SiMe_3$ (**12**))). Additionally the unique monomeric hydroxy complex Salen(^{t}Bu)AlOH (**13**) and its condensation product, (Salen(^{t}Bu)Al)$_2$O (**14**), will be described. All of the compounds were characterized by spectroscopic (^{1}H, ^{13}C, and ^{27}Al NMR, IR) and physical (mp, analyses) techniques. X-ray crystallographic data are presented for **14**.

Introduction

The Salen[1] class of ligands have been used extensively to support transition metal bonding schemes[2] and to a much lesser extent those of the main group elements. Some group 13 examples include those incorporating aluminum[3] and gallium[4] alkyls, aluminum alkoxides,[5] and aluminum cations.[6] One considerable limitation in using the Salen ligands lies in the fact that they are generally insoluble when bound in a neutral complex. Occasionally, this problem can be tempered somewhat by use of the Acen ligand (Figure 1b). In order to avoid solubility problems entirely we have begun investigating derivatives that feature pendant alkyl groups. One such Salen derivative is Salen(^{t}Bu)H_2, which possesses tertiary butyl groups at two positions on each of the phenol rings (Figure 1c). Another consequence of the resulting steric encumberance on the ligand is to favor monomeric complexes. We envisioned this ligand as being a soluble "platform" upon which unique group 13 chemistry may be conducted. Indeed, these ligands have had some degree of success in the realm of transition metal chemistry.[7] Perhaps the most systematically studied of these complexes are those used as epoxidation catalysts.[8]

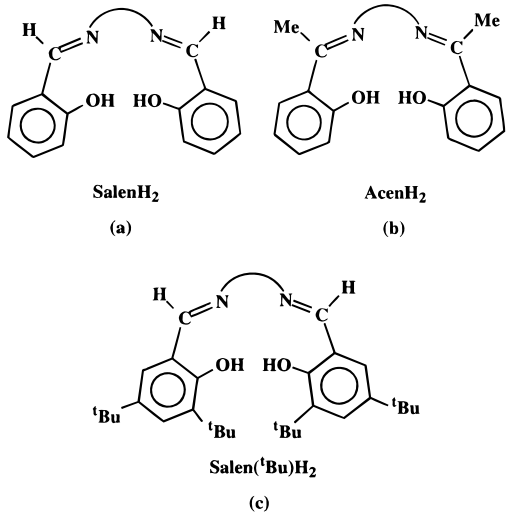

Figure 1. General depiction of the types of Salen ligands mentioned in the text.

In the present manuscript we will describe the compounds of formula Salen(^{t}Bu)AlCl (where Salen = Salen (**1**), Salpen (**2**), Salophen (**3**), Salomphen (**4**)) and the novel five-coordinate monomeric amides of formula LAlNRR′ (where L = Acen (R, R′ = H, Ph (**5**); R = R′ = $SiMe_3$ (**6**)) and L = Salen(^{t}Bu) (R, R′ = H, ^{t}Bu (**7**), H, Ph (**8**), and H, Dipp (2,6-diisopropylphenyl) (**9**); R = R′ = Me (**10**), Et (**11**), and $SiMe_3$ (**12**))). Additionally, the unique monomeric hydroxy complex Salen(^{t}Bu)AlOH (**13**) and its condensation product, (Salen(^{t}Bu)Al)$_2$O (**14**), will also be reported.

Results and Discussion

Synthesis and Characterization of 1−4. The monomeric aluminum chloride derivatives can be pre-

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(1) "Salen" is the name that has historically been used to describe the entire class of such ligands possessing various diamino backbones. However, it is also the specific name of the ethyl derivative, SalenH_2.

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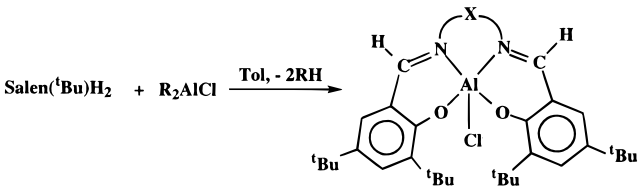
Table 1. Selected Spectroscopic Data (ppm) for Compounds 1−14

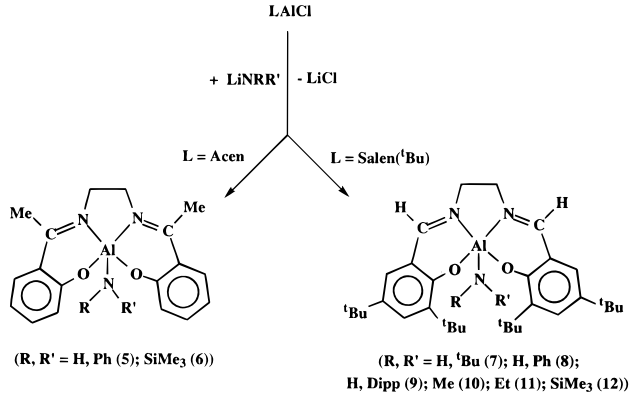
compd	Ph−^{t}Bu	backbone	NC−R	^{27}Al ($W_{1/2}$)
Salen(^{t}Bu)AlCl (**1**)	1.29 (s)	3.74 (m)	8.37 (s)	57 (5000)
	1.53 (s)	4.15 (m)		
Salpen(^{t}Bu)AlCl (**2**)	1.29 (s)	2.19 (m)	8.27 (s)	43 (5300)
	1.50 (s)	3.64 (m)		
		4.05 (m)		
Salophen(^{t}Bu)AlCl (**3**)	1.34 (s)	7.66 (d)	8.97 (s)	52 (5300)
	1.59 (s)	7.77 (m)		
Salomphen(^{t}Bu)AlCl (**4**)	1.34 (s)	2.35 (s)	8.90 (s)	48 (6500)
	1.59 (s)	7.23 (d)	R = Me	
AcenAlNHPh (**5**)		3.73−3.90(m)	2.53 (s)	42 (1212)
AcenAlN($SiMe_3$)$_2$ (**6**)		3.76 (m)	2.48 (s)	43 (2600)
		4.30 (m)		
			R = H	
Salen(^{t}Bu)AlNHtBu (**7**)	1.32 (s)	3.76 (m)	8.40 (s)	56 (6800)
	1.56 (s)	4.16 (m)		
Salen(^{t}Bu)AlNHPh (**8**)	1.35 (s)	3.64 (m)	8.37 (s)	42 (1100)
	1.52 (s)	3.97 (m)		
Salen(^{t}Bu)AlNHDipp (**9**)	1.35 (s)	3.53 (m)	8.28 (s)	43 (3000)
	1.58 (s)			
Salen(^{t}Bu)AlNMe$_2$ (**10**)	1.28 (s)	4.01 (m)	8.37 (s)	45 (3500)
	1.51 (s)			
Salen(^{t}Bu)AlNEt$_2$ (**11**)	1.28 (s)	3.92 (m)	8.38 (s)	55 (6500)
	1.55 (s)			
Salen(^{t}Bu)AlN($SiMe_3$)$_2$ (**12**)	1.29 (s)	3.49 (m)	8.10 (s)	45 (2100)
	1.45 (s)	4.35 (m)		
Salen(^{t}Bu)AlOH (**13**)	1.30 (s)	3.15 (m)	7.86 (s)	41 (2500)
	1.32 (s)	3.30 (m)		
[Salen(^{t}Bu)Al]$_2$O (**14**)	1.52 (s)	4.30 (m)		

Scheme 1. General Synthetic Pathway for the Preparation of 1−4



(X = Et (1); Pr (2); Ph (3); 3,4-Me_2Ph (4))

Scheme 2. General Synthesis of the Acen and Salen(^{t}Bu) Amides



(R, R' = H, Ph (5); $SiMe_3$ (6))

(R, R' = H, ^{t}Bu (7); H, Ph (8); H, Dipp (9); Me (10); Et (11); $SiMe_3$ (12))

pared by combining the ligand with R_2AlCl in a 1:1 ratio in toluene (Scheme 1). The compounds are soluble and can be isolated by precipitation after concentration of the solution or crystallization after cooling to −30 °C for a few days. This contrasts with the non-*tert*-butylated Salen syntheses in which the SalenAlCl derivatives precipitate from toluene immediately. Indeed, the insolubility of these latter complexes, although nice with regards to obtaining high yields, has made access to NMR data problematic.[6c] For compounds **1**−**4**, however, this is not a difficulty. They are readily soluble in benzene and chloroform. The ^{1}H NMR data shows no complexity that may indicate a dimeric or "open" structure in solution (Table 1). Moreover, the ^{27}Al NMR shifts in the range δ 43−57 ppm are consistent with the presence of five-coordinate aluminum.[9] A monomeric formulation is further supported by cryoscopic molecular weight determinations conducted in benzene. Although it may be intuitive to consider that the Salen(^{t}Bu) ligand would adopt a planar, monomeric geometry (this property is generally why the ligand is used in the first place), this is not always the case. For example, the SalenAlOMe derivatives form dimeric methoxy-bridged structures in which the aluminum is six-coordinate and the ligand is in a bent conformation.[6b] Moreover, neutral group 13 chloride reagents will, in the absence of bridging heteroatoms or steric encumberance, adopt chloride-bridged structures.[10] Compounds **1**−**4** fulfill both exceptions; the presence of the ^{t}Bu groups negates the possibility of either chloride or heteroatom bridge-bonding. These suppositions have been somewhat verified in the crystal structure of Salcen(^{t}Bu)AlCl (Salcen = (*R*, *R*)-*N*,*N*′-cyclohexylenebis-(3,5-di-*tert*-butylsalicylideneimine)).[11]

Synthesis and Characterization of 5−12. The amides are prepared by the addition of lithium amide to either AcenAlCl[6c] or Salen(^{t}Bu)AlCl (**1**) in toluene (Scheme 2). They can be isolated, after filtration and solvent removal, as bright orange or yellow solids in nearly quantitative yield. They are extremely air sensitive and will undergo protonolysis to form **13**. The level of sensitivity to this process can be correlated to the degree of steric bulk on the nitrogen. Thus, the least

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sterically encumbered, **10**, is the most sensitive (reacting instantaneously in air) while those incorporating the Dipp ligand (**9**) and the $SiMe_3$ groups (**12**) are the least air sensitive (decomposing over the course of minutes). Thus, **7**−**12** are more air sensitive than the SalenAlR complexes.[3] This difference may be attributed to the increased steric bulk of the Salen(^{t}Bu) ligands combined with the increased steric requirements of an Al−NR_2 group compared to an Al−R group.

The ^{27}Al NMR for **5**−**12** is consistent with the presence of monomeric, five-coordinate complexes in solution (Table 1). The observed chemical shifts in the range δ 40−57 ppm correspond to that of five-coordinate aluminum.[9] Additionally, cryoscopic molecular weight determinations support a monomeric formulation. By comparison, six-coordinate Salen−aluminum complexes demonstrate chemical shifts in the range δ 3−9 ppm.[6c] The primary amides, **5** and **7**−**9**, display an NH stretching frequency in the expected region of the spectrum (≈3300 cm^{-1}).

The ^{1}H NMR data for compounds **1**−**4** and **7**−**14** have two features in common. They demonstrate two singlets for the Ph−^{t}Bu groups in the range δ 1.28−1.59 ppm. Moreover, only one singlet is observed for the hydrogen on the imine carbon. These fall in the relatively broad range δ 7.86−8.97 ppm. Similarly, compounds **5** and **6** display singlets at δ 2.53 and 2.48 ppm, respectively. Compound **10** has two resonances in the ^{1}H NMR spectrum for the N−Me groups. This might be interpreted in terms of hindered rotation about the Al−N bond due to π bonding. Unfortunately, attempts to heat the compound to observe coalescence of these two peaks led to decomposition of the compound. Moreover, NMR spectra obtained to −63 °C were identical to that obtained at ambient temperature. None of the other compounds displayed inequivalence of the group on nitrogen. Further work in this area is aimed at obtaining crystal structures of these compounds for comparison to the more traditional three-coordinate[12] and four-coordinate aluminum amides.[13]

Synthesis and Characterization of 13 and 14. Compounds **7**−**12** are fairly air sensitive and undergo protonolysis with a limited amount of adventitious water to form a compound that can be characterized as Salen(^{t}Bu)AlOH (**13**) (Scheme 3). This compound reacts with an additional 1 mol of amide to form (Salen(^{t}Bu)Al)$_2$O (**14**). Upon further hydrolysis **14**, in turn, converts to **13** which is indefinitely stable in air at 25 °C. Like the amides, it is also very soluble and could be characterized fully. The ^{1}H NMR data for **13** are consistent with the other monomeric compounds. Additionally, it possesses a ν_{O-H} IR stretch centered at ν 3474 cm^{-1}. Compound **13** is a rare example of a group

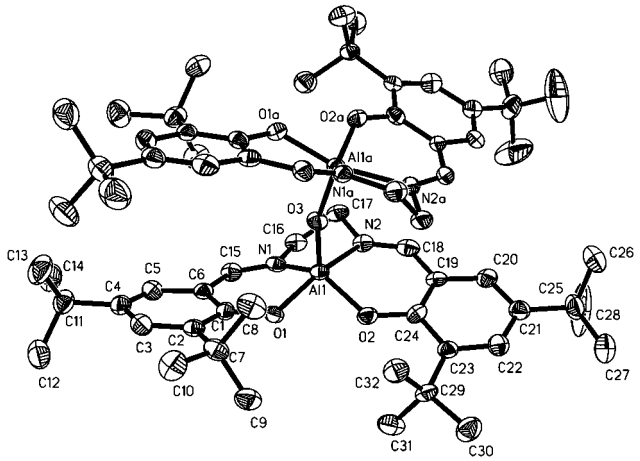
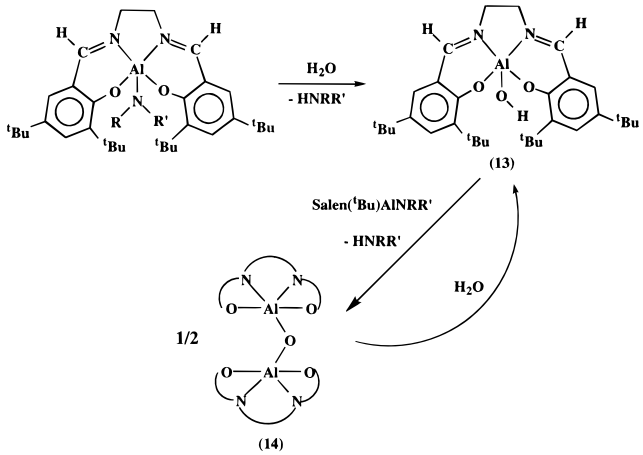


Figure 2. Molecular structure and atom-numbering scheme for [Salen(^{t}Bu)Al]$_2$O (**14**).

Scheme 3. Reactions Leading to the Formation of 13 and 14



13 complex with an nonbridging hydroxyl group. One other example is ^{t}Bu$_2$Ga(THF)OH, which has an OH stretch of 3285 cm^{-1}.[14] It is isolated by the hydrolysis of Ga^{t}Bu$_3$ in coordinating solvents (THF). In contrast, the presence of a monomeric Ga−OH complex is not observed in the hydrolysis of Mes_3Ga.[15]

Crystal Structure of 14. The X-ray structure of **14** confirms the dimeric arrangement (Figure 2). In the structure two Salen(^{t}Bu)Al units are held together by a bridging oxygen atom. A C_2 rotational axis makes these two units equivalent. The overall morphology of **14** is consistent with that observed for (SalenAl)$_2$O[5] and (SalenFe)$_2$O.[16] However, the Al−O−Al bond angle in **14** (159.5(5)°) (Table 2) is larger than that observed in either the Al (152.0(3)°) or Fe (139.1−144.6°) systems. The larger angle can be attributed to the increase in steric interactions on going from the Salen to the Salen(^{t}Bu) ligand. The Al−O distance in **14** is actually shorter (1.696(3) Å) than that observed in the Salen complex (1.705(5) Å). It is also marginally shorter than the analogous distance in Salen(^{t}Bu)AlOSiPh$_3$ (1.719(14) Å).[17]

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Table 2. Selected Bond Lengths (Å) and Angles (deg) for [Salen(ᵗBu)Al]₂O (14)

Al(1)−O(1)	1.821(6)	O(2)−C(24)	1.352(10)
Al(1)−O(2)	1.793(6)	O(3)−Al(1A)	1.696(3)
Al(1)−O(3)	1.696(3)	N(1)−C(15)	1.284(11)
Al(1)−N(1)	2.008(5)	N(1)−C(16)	1.473(9)
Al(1)−N(2)	2.035(7)	N(2)−C(17)	1.460(9)
O(1)−C(1)	1.311(10)	N(2)−C(18)	1.274(11)
O(1)−Al(1)−O(2)	90.2(3)	Al(1)−N(1)−C(15)	124.2(5)
O(1)−Al(1)−O(3)	104.2(3)	Al(1)−N(1)−C(16)	117.8(5)
O(2)−Al(1)−O(3)	117.7(3)	C(15)−N(1)−C(16)	117.5(5)
O(1)−Al(1)−N(1)	88.0(3)	Al(1)−N(2)−C(17)	112.6(5)
O(2)−Al(1)−N(1)	130.8(3)	Al(1)−N(2)−C(18)	126.8(5)
O(3)−Al(1)−N(1)	110.3(3)	C(17)−N(2)−C(18)	120.5(7)
O(1)−Al(1)−N(2)	158.2(3)	O(1)−C(1)−C(2)	121.4(6)
O(2)−Al(1)−N(2)	86.9(3)	O(1)−C(1)−C(6)	119.7(7)
O(3)−Al(1)−N(2)	96.2(3)	N(1)−C(15)−C(6)	125.4(6)
N(1)−Al(1)−N(2)	77.7(3)	N(1)−C(16)−C(17)	106.1(6)
Al(1)−O(1)−C(1)	129.1(5)	N(2)−C(17)−C(16)	103.8(7)
Al(1)−O(2)−C(24)	132.7(6)	N(2)−C(18)−C(19)	123.5(8)
Al(1)−O(3)−Al(1A)	159.5(5)	O(2)−C(24)−C(19)	120.7(6)
		O(2)−C(24)−C(23)	118.7(7)

Conclusion

We have demonstrated that the Salen(ᵗBu) ligands are useful in the isolation of unique bonding schemes for aluminum. Two such complexes were described. The first are the unique five-coordinate aluminum amides that were the principal subject of this manuscript. The second unique complex is the monomeric aluminum hydroxide. Very few of this type of complex exist in the literature. It should be a useful starting material to other monomeric bimetallic complexes possessing an Al−O−metal linkage.

Experimental Section

General Considerations. All manipulations were conducted using Schlenk techniques in conjunction with an inert-atmosphere glovebox. All solvents were rigorously dried prior to use. NMR data were obtained on JEOL-GSX-400 and -270 instruments at 270.17 (^{1}H) and 62.5 (^{13}C) MHz. Chemical shifts are reported relative to $SiMe_4$ and are in ppm. Elemental analyses were obtained on a Perkin-Elmer 2400 analyzer. Infrared data were recorded as KBr pellets on a Matheson Instruments 2020 Galaxy Series spectrometer and are reported in cm^{-1}. 3,5-Di-*tert*-butyl-2-hydroxybenzaldehyde[18] and Salen(ᵗBu)H$_2$[19] were prepared according to the literature. Cryoscopic molecular weight determinations were conducted by following an established procedure.[20]

Salen(ᵗBu)AlCl (1). Salen(ᵗBu)H$_2$ (9.268 g, 18.70 mmol) was dissolved in 50 mL of toluene and cooled to −78 °C, and then a cooled solution (−78 °C) of dimethylaluminum chloride (1.96 g, 21.07 mmol) in 50 mL of toluene was added. The solution was gradually warmed to 25 °C over 2 h and stirred for an additional 10 h, and then the volatiles were removed under reduced pressure to give 10.37 g of a pale yellow solid (99%): Mp 309−310 °C; ^{1}H NMR (270 MHz, $CDCl_3$) δ 1.29 (s, 18H, CCH_3), 1.53 (s, 18H, CCH_3), 3.74 (m, 2H, NCH_2), 4.15 (m, 2H, NCH_2), 7.03 (d, 2H, Ph-*H*), 7.55 (d, 2H, Ph-*H*), 8.37 (d, 2H, NC*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 29.5 (CCH_3), 31.1 (CCH_3), 33.8 (*C*CH_3), 35.4 (*C*CH_3), 54.6 (NCH_2), 118.4 (Ph), 127.4, 131.6, 139.2, 141.5, 163.3, 170.7 (N*C*H); ^{27}Al NMR (104.15 MHz, $CDCl_3$) δ 57 ($W_{1/2}$ = 5000 Hz); IR (KBr) 2953 (m), 1624 (vs), 1543 (w), 1398 (w), 1259 (s), 1094 (m), 816 (w), 608 (s), 444 (m) cm^{-1}. Anal. Calcd for $C_{32}H_{46}N_2O_2AlCl$: C, 69.50; H, 7.91; N, 5.07. Found: C, 69.18; H, 8.02; N, 5.56.

Salpen(ᵗBu)AlCl (2). A solution of dimethylaluminum chloride (0.841 g, 9.191 mmol) in 50 mL of toluene was cooled to −78 °C and combined with a rapidly stirring solution of Salpan(ᵗBu)H$_2$ (4.655 g, 9.186 mmol) in 50 mL of toluene also cooled to −78 °C. The exothermic reaction was allowed to proceed at −78 °C for 1 h. The solution was brought to room temperature slowly and stirred overnight. A precipitate formed, and the solvent was removed under vacuum giving 4.74 g of pale yellow solid (91%): Mp 282−284 °C (dec); ^{1}H NMR (270 MHz, $CDCl_3$) δ 1.29 (s, 18H, CCH_3), 1.50 (s, 18H, CCH_3), 2.17−2.21 (m, 2H, CH_2CH_2), 3.62−3.67 (m, 2H, NCH_2), 4.03−4.08 (m, 2H, NCH_2), 7.05 (d, 2H, Ph*H*), 7.54 (d, 2H, Ph*H*), 8.27 (s, 2H, NC*H*); ^{27}Al NMR (104.15 MHz, $CDCl_3$) δ 43 ($W_{1/2}$ = 5300 Hz); IR (KBr) 2955, 2905, 2866, 1626, 1557, 1464, 1420, 1391, 1352, 1314, 1260, 1181, 1098, 862, 849, 785, 762, 600, 438. Anal. Calcd for $C_{33}H_{48}N_2O_2AlCl$: C, 69.88; H, 8.53. Found: C, 69.61; H, 8.16.

Salophen(ᵗBu)AlCl (3). A solution of diethylaluminum chloride (0.108 g, 0.896 mmol) in 20 mL of toluene was added to a solution of Salophen(ᵗBu)H$_2$ (0.500 g, 0.925 mmol) in 50 mL of toluene. The reaction was refluxed for 2 h. The solution was then cooled to −30 °C for 2 weeks. At this time a precipitate had formed so the solvent was removed under vacuum yielding 0.421 g of a bright yellow solid (78%): Mp 322 °C (dec); ^{1}H NMR (270 MHz, $CDCl_3$) δ 1.34 (s, 18H, CCH_3), 1.59 (s, 18H, CCH_3), 7.24 (d, 2H, Ph*H*), 7.41 (m, 2H, Ph*H*), 7.66 (d, 2H, Ph*H*), 7.77 (m, 2H, PhH), 8.97 (s, 2H, NC*H*); ^{27}Al NMR (104.15 MHz, $CDCl_3$) δ 52 ($W_{1/2}$ = 5300 Hz); IR (KBr) 2959, 2907, 2868, 1618, 1586, 1541, 1470, 1397, 1362, 1262, 1202, 1111, 847, 808, 756, 598, 459. Anal. Calcd for $C_{36}H_{46}N_2O_2AlCl$: C, 71.92; H, 7.71. Found: C, 71.60; H, 7.46.

Salomphen(ᵗBu)AlCl (4). A solution of dimethylaluminum chloride (0.161 g, 1.76 mmol) in 25 mL of toluene was added to a rapidly stirring solution of Salomphen(ᵗBu)H$_2$ (1.00 g, 1.76 mmol) in 50 mL of toluene. The reaction was allowed to stir overnight. A bright orange precipitate was formed. The solution was filtered, and the solvent was removed from the precipitate under vacuum, yielding 0.78 g of a bright yellow solid (71%): Mp 330 °C (dec); ^{1}H NMR (270 MHz, $CDCl_3$) δ 1.34 (s, 18H, CCH_3), 1.59 (s, 18H, CCH_3), 2.35 (s, 6H, PhCH_3), 7.23 (d, 2H, Ph*H*), 7.94 (s, 2H, Ph*H*), 7.64 (d, 2H, Ph*H*), 8.90 (s, 2H, NC*H*); ^{27}Al NMR (104.15 MHz, $CDCl_3$) δ 48 ($W_{1/2}$ = 6500 Hz); IR (KBr) 2955, 2907, 2868, 1620, 1591, 1553, 1470, 1360, 1256, 1181, 849, 787, 770, 602, 444 cm^{-1}. Anal. Calcd for $C_{38}H_{50}N_2O_2AlCl$: C, 72.53; H, 8.00. Found: C, 72.70; H, 7.93.

AcenAlNHPh (5). Aniline (0.500 mL, 5.49 mmol) was dissolved in 30 mL of dry THF and cooled to −78 °C. *n*-BuLi (2.10 mL, 2.58 M, 5.42 mmol) was added dropwise to the aniline solution and the reaction stirred at −78 °C for 5 min and then warmed to 0 °C over 20 min. The solution was cannulated cold into a stirring suspension of AcenAlCl (1.942 g, 5.45 mmol) in 30 mL of THF. The reaction was stirred at 25 °C for 12 h, after which the THF was filtered off to leave a yellow solid. The solid was extracted in CH_2Cl_2, the solution filtered, and the solvent removed to give 1.02 g of a pale yellow solid (46%): Mp 208 °C; ^{1}H NMR (270 MHz, $CDCl_3$) δ 2.53 (s, 6H, CH_3), 3.73−3.90 (m, 4H, NCH_2), 6.19 (d, J = 8 Hz, 2H, Ph-*H*), 6.33 (app t, 1H, Ph-*H*), 6.70−6.83 (m, 4H, Ph-*H*), 7.12 (d, J = 9 Hz, 2H, Ph-*H*), 7.38 (app t, 2H, Ph-*H*), 7.62 (d, J = 9 Hz, 2H, Ph-*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.1 (*C*H$_3$), 48.2 (NCH_2), 113.7 (Ph), 115.0 (Ph), 116.5 (Ph), 116.6 (Ph), 118.4 (Ph), 120.0 (Ph), 123.9 (Ph), 128.5 (Ph), 129.3 (Ph), 134.9 (Ph), 153.2 (Ph), 164.7 (Ph), 176.4 (*C*═N); ^{27}Al NMR (104.19 MHz, $CDCl_3$) δ 42 (1212); IR (KBr) 3352 (w), 3028 (m), 2361 (m), 1604 (vs), 1545 (vs), 1334 (vs), 750 (vs), 472 (s) cm^{-1}. Anal. Calcd for $C_{24}H_{24}N_3O_2Al$: C, 69.35; H, 5.94. Found: C, 69.72; H, 5.85.

AcenAlN(SiMe$_3$)$_2$ (6). AcenAlCl (5.00 g, 14.04 mmol) and LiN(SiMe$_3$)$_2$ (2.50 g, 14.51 mmol) were placed in a 250 mL

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round bottom flask, and 100 mL of dry THF was added. The solution was stirred at 25 °C for 12 h, after which the solvents were removed in vacuo to give a dark tan solid. The solid was extracted in CH_2Cl_2, the solution was filtered, and CH_2Cl_2 was removed in vacuo to give a yellow-orange solid (5.86 g, 87%): Mp 277−279 °C (dec); ^{1}H NMR (270 MHz, $CDCl_3$) δ −0.07 (s, 18H, SiCH_3), 2.48 (s, 6H, CH_3), 3.76 (m, 2H, NCH_2), 4.30 (m, 2H, NCH_2), 6.64−7.57 (m, 8H, Ph-*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 2.5 (SiCH_3), 4.9 (Si*C*H$_3$), 17.2 (*C*H$_3$), 48.0 (N*C*H$_2$), 115.8 (Ph), 120.3 (Ph), 123.9 (Ph), 129.2 (Ph), 134.7 (Ph), 165.2 (Ph), 174.6 (*C*═N); ^{27}Al NMR (104.15 MHz, $CDCl_3$) δ 43 ($W_{1/2}$ = 2600); IR (KBr) 2953 (m), 1606 (vs), 1447 (s), 1248 (s), 937 (s), 885 (vs), 833 (vs), 754 (vs), 474 (s) cm^{-1}. Anal. Calcd for $C_{24}H_{36}N_3O_2AlSi_2$: C, 60.20; H, 6.93; N, 8.42. Found: C, 59.88; H, 7.48; N, 8.73.

Salen(ᵗBu)AlNHᵗBu (7). *tert*-Butylamine (1.0 mL, 9.45 mmol) was dissolved in 20 mL of THF and stirred at −78 °C, and then ᵗBuLi (5.5 mL, 1.72 M in pentane, 9.46 mmol) was added. The solution was warmed to 25 °C, stirred for 4 h, and then cannulated onto a suspension of Salen(ᵗBu)AlCl (4.95 g, 8.95 mmol) in 50 mL of toluene. The solution changed from a yellow to orange during the course of the addition and was stirred for 12 h. The volatiles were removed in vacuo, the orange solid was extracted in CH_2Cl_2, the solution was filtered, and the solvent was removed under reduced pressure to provide **7** as an orange solid (4.38 g, 83%): Mp 258−261 °C; ^{1}H NMR (270 MHz, $CDCl_3$) δ 1.16 (s, 9H, NCCH_3), 1.32 (s, 18H, CCH_3), 1.56 (s, 18H, CCH_3), 3.76 (m, 2H, NCH_2), 4.16 (m, 2H, NCH_2), 7.07 (d, 2H, Ph-*H*), 7.58 (d, 2H, Ph-*H*), 8.40 (s, 2H, NC*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 29.8 (C*C*H$_3$), 31.5 (C*C*H$_3$), 32.6 (NC*C*H$_3$), 34.1 (*C*CH$_3$), 35.7 (*C*CH$_3$), 54.8 (N*C*H$_2$), 118.3 (Ph), 127.3, 131.5, 139.0, 141.3, 163.0, 170.5 (N*C*H); ^{27}Al NMR (104.15 MHz, $CDCl_3$) δ 56 ($W_{1/2}$ = 6800 Hz); IR (KBr) 3243, 2857, 1633, 1622, 1446, 1301, 1168, 1055, 873, 754, 598, 498. Anal. Calcd for $C_{36}H_{57}N_3O_2Al$: C, 73.22; H, 9.66; N, 7.11. Found: C, 72.74; H, 9.73; N, 7.41.

Salen(ᵗBu)AlNHPh (8). Aniline (0.174 mL, 1.91 mmol) was combined with 20 mL of hexane and stirred at 25 °C, and then ᵗBuLi (1.1 mL, 1.80 M in pentane, 1.98 mmol) was added to produce a milky white suspension, which was stirred for 5 min and then cannulated onto a suspension of Salen(ᵗBu)AlCl (1.06 g, 1.92 mmol) in 20 mL of toluene. The solution changed from a yellow to orange during the course of the addition and was stirred for 3 h and filtered, and the volatiles were removed in vacuo to provide **8** as a pale yellow solid (1.13 g, 97%): Mp, turned orange at 190 °C and then melted while turning dark red at 254−256 °C; ^{1}H NMR (270 MHz, $CDCl_3$) δ 1.35 (s, 18H, CCH_3), 1.52 (s, 18H, CCH_3), 3.01 (br s, 1H, N*H*), 3.64 (m, 2H, NCH_2), 3.97 (m, 2H, NCH_2), 6.07 (d, 2H, Ph-*H*), 6.28 (t, 1H, Ph-*H*), 6.77 (t, 2H, Ph-*H*), 7.09 (d, 2H, Ph-*H*), 7.58 (d, 2H, Ph-*H*), 8.37 (s, 2H, NC*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 29.5 (C*C*H$_3$), 31.2 (C*C*H$_3$), 33.9 (*C*CH$_3$), 35.4 (*C*CH$_3$), 55.4 (N*C*H$_2$), 113.2 (Ph-amide), 116.4 (Ph-amide), 116.5 (Ph-amide), 118.4 (Ph-ligand), 127.1 (Ph-ligand), 128.8 (Ph-amide), 131.1 (Ph-ligand), 138.6 (Ph-ligand), 141.5 (Ph-ligand), 154.1 (Ph-amide), 163.4 (Ph-ligand), 170.2 (N*C*H); ^{27}Al NMR (104.15 MHz, $CDCl_3$) δ 42 ($W_{1/2}$ = 1100 Hz); IR (KBr) 3360, 2859, 1649, 1622, 1442, 1301, 1177, 1056, 874, 755, 755, 598, 498, 434 cm^{-1}. Anal. Calcd for $C_{38}H_{52}N_3O_2Al$: C, 74.88; H, 8.54; N, 6.90. Found: C, 74.66; H, 8.51; N, 7.22.

Salen(ᵗBu)AlNHDipp (9). 2,6-Diisopropylaniline (0.380 mL, 2.02 mmol) was combined with 20 mL of hexane and stirred at 25 °C, and then ⁿBuLi (0.85 mL, 2.5 M in hexane, 2.13 mmol) was added to produce a milky white suspension which was stirred for 10 min and then cannulated onto a suspension of Salen(ᵗBu)AlCl (1.134 g, 2.05 mmol) in 20 mL of toluene. The solution changed from a yellow to orange during the course of the addition and was stirred for 10 h and volatiles removed in vacuo. Dissolution of the residue in 40 mL of toluene, followed by filtration, and removal of volatiles in vacuo provided **9** as a pale yellow solid (1.326 g, 95%): Mp 283−289 °C (dec); ^{1}H NMR (270 MHz, $CDCl_3$) δ 0.81 (d, 12H, CHCH_3), 1.35 (s, 18H, CCH_3), 1.58 (s, 18H, CCH_3), 2.42 (br s, 1H, N*H*), 2.81 (m, 2H, C*H*CH$_3$), 3.53 (m, 4H,NCH_2), 6.62 (t, 1H, Ph-*H*), 6.83 (d, 2H, Ph-*H*), 7.06 (d, 2H, Ph-*H*), 7.59 (d, 2H, Ph-*H*), 8.28 (s, 2H, NC*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 22.9 (CH*C*H$_3$), 27.5 (*C*HCH$_3$), 29.8 (C*C*H$_3$), 31.5 (C*C*H$_3$), 34.1 (*C*CH$_3$), 35.7 (*C*CH$_3$), 55.9 (N*C*H$_2$), 117.1 (Dipp), 118.7 (Ph-ligand), 122.0 (Dipp), 127.0 (Ph-ligand), 130.0 (Ph-ligand), 138.3 (Ph-ligand), 139.4 (Dipp), 141.3 (Ph-ligand), 147.4 (Dipp), 163.3 (Ph-ligand), 169.5 (N*C*H); ^{27}Al NMR (104.15 MHz, $CDCl_3$) δ 43 ($W_{1/2}$ = 3000 Hz); IR (KBr) 3360, 2953, 1626, 1554, 1460, 1390, 1261, 1177, 839, 754, 583 cm^{-1}. Anal. Calcd for $C_{44}H_{64}N_3O_2Al$: C, 76.19; H, 9.24; N, 6.06. Found: C, 75.93; H, 8.99; N, 5.34.

Salen(ᵗBu)AlNMe$_2$ (10). Salen(ᵗBu)AlCl (0.500 g, 0.91 mmol) was suspended in 10 mL of toluene and stirred at 25 °C, and then lithium dimethylamide (0.048 g, 0.94 mmol) was added as a solid. The solution was stirred at 35 °C for 24 h and filtered, and the volatiles were removed under reduced pressure to give 0.496 g of an orange solid (98%): Mp 172 °C (dec); ^{1}H NMR (270 MHz, $CDCl_3$) δ 1.28 (s, 18H, CCH_3), 1.51 (s, 18H, CCH_3), 2.09 (br s, 3H, NCH_3), 2.61 (s, 3H, NCH_3), 4.01 (m, 4H, NCH_2), 7.03 (s, 2H, Ph-*H*), 7.52 (s, 2H, Ph-*H*), 8.37 (s, 2H, NC*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 29.7 (C*C*H$_3$), 31.4 (C*C*H$_3$), 34.1 (*C*CH$_3$), 35.6 (*C*CH$_3$), 39.1 (N*C*H$_3$), 43.3 (N*C*H$_3$), 54.2 (N*C*H$_2$), 118.5 (Ph), 127.9, 131.5, 138.8, 140.7, 163.0, 170.8 (N*C*H); ^{27}Al NMR (104.15 MHz, $CDCl_3$) δ 45 ($W_{1/2}$ = 4500 Hz); IR (KBr) 2955, 1651, 1628, 1541, 1442, 1257, 1175, 1026, 839, 599, 498 cm^{-1}. Anal. Calcd for $C_{34}H_{52}N_3O_2Al$: C, 72.73; H, 9.27. Found: C, 72.74; H, 9.36.

Salen(ᵗBu)AlNEt$_2$ (11). Salen(ᵗBu)AlCl (1.703 g, 3.08 mmol) was suspended in 20 mL of toluene and stirred at 25 °C, and then lithium diethylamide (0.279 g, 3.36 mmol) was added as a solid. The solution was stirred at 35 °C for 48 h and filtered, and the volatiles were removed under reduced pressure to give 1.632 g of an orange solid (90%): Mp 262−264 °C (dec); ^{1}H NMR (270 MHz, $CDCl_3$) δ 1.10 (t, 6H, CH$_2$CH_3), 1.28 (s, 18H, CCH_3), 1.55 (s, 18H, CCH_3), 2.64 (br q, 4H, NCH_2CH$_3$), 3.92 (br s, 4H, NCH_2), 7.05 (s, 2H, Ph-*H*), 7.56 (s, 2H, Ph-*H*), 8.38 (s, 2H, NC*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 15.5 (CH$_2$*C*H$_3$), 29.8 (C*C*H$_3$), 31.4 (C*C*H$_3$), 34.1 (*C*CH$_3$), 35.7 (*C*CH$_3$), 44.0 (NCH$_2$CH$_3$), 54.8 (N*C*H$_2$), 118.3 (Ph), 127.3, 131.4, 139.0, 141.3, 163.0, 170.4 (N*C*H); ^{27}Al NMR (104.15 MHz, $CDCl_3$) δ 55 ($W_{1/2}$ = 6500 Hz); IR (KBr) 2953, 2908, 2868, 1622, 1541, 1439, 1314, 1258, 1175, 1018, 854, 752, 578 cm^{-1}. Anal. Calcd for $C_{36}H_{56}N_3O_2Al$: C, 73.34; H, 9.51; N, 7.13. Found: C, 73.67; H, 9.43; N, 6.89.

Salen(ᵗBu)AlN(SiMe$_3$)$_2$ (12). Salen(ᵗBu)AlCl (0.738 g, 1.38 mmol) was suspended in 10 mL of toluene and stirred at 25 °C, and then lithium hexamethyldisilazane (0.252 g, 1.51 mmol) was added as a solid. The solution was stirred at 35 °C for 48 h and filtered, and the volatiles were removed under reduced pressure to give 0.944 g of a fluorescent yellow solid (99%): Mp 201−210 °C (dec); ^{1}H NMR (270 MHz, $CDCl_3$) δ −0.03 (s, 18H, SiCH_3), 1.29 (s, 18H, CCH_3), 1.45 (s, 18H, CCH_3), 3.49 (m, 2H, NCH_2), 4.35 (m, 2H, NCH_2), 6.94 (d, 2H, Ph-*H*), 7.48 (d, 2H, Ph-*H*), 8.10 (s, 2H, NC*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 5.09 (Si*C*H$_3$), 29.9 (C*C*H$_3$), 31.2 (C*C*H$_3$), 33.7 (*C*CH$_3$), 35.2 (*C*CH$_3$), 53.1 (N*C*H$_2$), 118.9 (Ph), 127.0, 130.9, 137.8, 140.9, 163.6, 169.5 (N*C*H); ^{27}Al NMR (104.15 MHz, $CDCl_3$) δ 45 ($W_{1/2}$ = 2100 Hz); ^{29}Si NMR ($CDCl_3$) δ −3.5; IR (KBr) 2961, 1655, 1624, 1421, 1259, 916, 885, 754, 573 cm^{-1}. Anal. Calcd for $C_{38}H_{64}N_3O_2AlSi_2$: C, 67.16; H, 9.43; N, 6.19. Found: C, 66.76; H, 8.96; N, 6.34.

Salen(ᵗBu)AlOH (13). Compound **13** can be prepared by allowing a solution of **7**−**12** to stir, without heating, in air for 12 h. It is isolated as a pale yellow solid after removal of the solvent under vacuum: Mp 310 °C (dec); ^{1}H NMR (270 MHz, $CDCl_3$) δ 1.30 (s, 18H, CCH_3), 1.32 (s, 18H, CCH_3), 1.52 (br s, 1H, O*H*), 3.15 (m, 2H, NCH_2), 3.30 (m, 2H, NCH_2), 6.87 (d, 2H, Ph-*H*), 7.34 (d, 2H, Ph-*H*), 7.86 (s, 2H, NC*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 29.6 (C*C*H$_3$), 31.6 (C*C*H$_3$), 34.0 (*C*CH$_3$), 35.6 (*C*CH$_3$), 54.9 (N*C*H$_2$), 118.7 (Ph), 126.2, 129.0, 136.8,

140.8, 163.9, 167.1 (N*C*H); ^{27}Al NMR (104.15 MHz, $CDCl_3$) δ 41 ($W_{1/2}$ = 2500 Hz); IR (KBr) 3474, 2955, 1631, 1442, 1259, 1174, 1003, 837, 750, 579 cm^{-1}. Anal. Calcd for $C_{32}H_{47}N_2O_3Al$: C, 71.91; H, 8.80. Found: C, 71.64; H, 8.71.

[Salen(ᵗBu)Al]$_2$O (14). A solution of Salen(ᵗBu)AlNHᵗBu (2.02 g, 3.42 mmol) in 40 mL of CH_3CN, 40 mL of CH_2Cl_2, and 40 mL of THF was heated open to the atmosphere until all of the solid had dissolved and then allowed to stand uncovered for 3 days until prisms formed (1.355 g, 76%): Mp 305−306 °C (dec); ^{1}H NMR (270 MHz, $CDCl_3$) δ 1.29 (s, 18H, CC*H*$_3$), 1.52 (s, 18H, CC*H*$_3$), 3.70−4.30 (v br s, 4H, NC*H*$_2$), 7.02 (d, 2H, Ph-*H*), 7.51 (d, 2H, Ph-*H*), 8.35 (s, 2H, NC*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 29.7 (C*C*H$_3$), 31.4 (C*C*H$_3$), 34.1 (*C*CH$_3$), 35.7 (*C*CH$_3$), 55.5 (N*C*H$_2$), 118.3 (Ph), 127.2, 130.7, 138.2, 140.9, 163.4, 169.8, (N*C*H); ^{27}Al NMR (104.15 MHz, $CDCl_3$) δ 40 ($W_{1/2}$ = 1800 Hz); IR (KBr) 2955, 2904, 2868, 1662, 1632, 1552, 1475, 1445, 1314, 1175, 1003, 837, 750, 577, 498 cm^{-1}. Anal. Calcd for $C_{64}H_{92}N_4O_5Al_2$: C, 73.14; H, 8.76; N, 5.33. Found: C, 72.83; H, 8.42; N, 5.29.

X-ray Experimental Section

Details of the crystal data and a summary of data collection parameters for the complexes are given in Table 3. Data were collected on a Siemens P4 diffractometer using graphite-monochromated Mo Kα (0.710 73 Å) radiation. In each case the check reflections indicated a less than 5% decrease in intensity over the course of data collection, and hence, no correction was applied. All calculations were performed on a personal computer using the Siemens software package, SHELXTL-Plus. The structures were solved by direct methods and successive interpretation of difference Fourier maps, followed by least-squares refinement. All non-hydrogen atoms were refined anisotropically. The hydrogen atoms were included in the refinement in calculated positions using fixed isotropic parameters.

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Table 3. Crystal Data for [Salen(ᵗBu)Al]$_2$O (14)

compd	**14**
formula	$C_{32}H_{46}AlN_2O_{2.5}$
fw	525.7
cryst system	monoclinic
space group	*C*2/*c*
a (Å)	24.915(3)
b (Å)	17.401(3)
c (Å)	17.573(1)
β (°)	122.78(1)
V (Å^3)	6405.2(14)
Z	8
D_{calc} (g/cm^3)	1.090
cryst size (mm)	0.6 × 0.5 × 0.4
temp (K)	298
2θ range (deg)	3.5−45
scan type	2θ−θ
scan speed (deg/min)	8−60
scan range (deg)	0.29
reflcns collcd	4885
indp reflcns	4133
obsd reflcns	1893 ($F > 4.0\sigma(F)$)
no. of params	339
R	0.0632
R_w	0.0647
GOF	4.30
lar diff peak (e/Å^3)	0.23

Supporting Information Available: Tables of X-ray parameters, positional and thermal parameters, and bond distances and angles and a unit cell diagram (10 pages). Ordering information is given on any current masthead page.

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