Preparation and Crystal Structure of Di-μ-acetato-μ-hydroxo-hexa(ethyl acetate)dialuminium Tris(tetrachloroaluminate)†

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The direct reaction between $AlCl_3$ and ethyl acetate in n-hexane yields $[AlCl_3(CH_3CO_2C_2H_5)_2]$ which during crystallization under the influence of moisture forms a complex of formula $[Al_2(\mu\text{-}CH_3CO_2)_2(\mu\text{-}OH)(CH_3CO_2C_2H_5)_6][AlCl_4]_3$. In the homobimetallic di-octahedral cation the Al^{3+} ions are bridged by one OH^- and two $CH_3CO_2^-$ anions. The aluminium–aluminium distance is 3.331(5) Å. The complex crystallizes in space group $P2_1/n$ with a = 14.974(15), b = 19.578(15), c = 20.567(15) Å, and β = 94.40(7)°. The structure was refined to R = 0.0693 for 3 252 reflections.

Polymerization studies have revealed that the interaction of aromatic esters with Ziegler–Natta catalysts supported on $MgCl_2$ results in high isotacticity. Hence, studies on the interaction between ester molecules and each of the catalyst components are very important. It was found that in the reaction between $TiCl_4$ and esters the dimeric[1–3] $[LCl_3Ti(\mu\text{-}Cl)_2TiCl_3L]$ (L = $CH_3CO_2C_2H_5$ or $CH_3OC_6H_4CO_2C_2H_5$) and $[Ti_2\{\mu\text{-}m\text{-}C_6H_4(CO_2Et)_2\}_2Cl_8]$ and monomeric[4,5] *cis*-$[TiCl_4(C_6H_5CO_2C_2H_5)_2]$ or $[TiCl_4\{C_6H_4(CO_2C_2H_5)_2\}]$ compounds are formed. With $AlCl_3$ as catalyst component, ethyl benzoate forms $[AlCl_3(C_6H_5CO_2C_2H_5)]$.[6] In ethyl acetate a compound formulated as $[AlCl_3(CH_3CO_2C_2H_5)_2]$ was formed. During crystallization from CH_2Cl_2 a new crystalline complex, containing $CH_3CO_2^-$ and OH^- anions, was formed which is rather an unexpected result. Therefore studies were undertaken to elucidate its structure. In this paper the crystal structure of $[Al_2(\mu\text{-}CH_3CO_2)_2(\mu\text{-}OH)(CH_3CO_2C_2H_5)_6][AlCl_4]_3$ is described.

Experimental

All reactions were carried out under N_2 using dried solvents and Schlenk-tube techniques. Commercial $AlCl_3$ was sublimed before use. Proton n.m.r. spectra were recorded on a BS-567A TESLA spectrometer, i.r. spectra on a Perkin-Elmer 180 spectrophotometer. Microanalyses were performed at the University of Wrocław.

Di-μ-acetato-μ-hydroxo-hexa(ethyl acetate)dialuminium Tris(tetrachloroaluminate).—To $AlCl_3$ (10 g, 75 mmol), ethyl acetate (30 cm^3) was added slowly, dropwise with stirring. The reaction is strongly exothermic. The brown-yellow solution was filtered and evaporated to dryness *in vacuo*. Thereafter the solid was dissolved in CH_2Cl_2 (50 cm^3). For crystallization, the solution (20 cm^3) was exposed to slow diffusion of n-hexane. After separation of the oil layer, colourless crystals were obtained under the influence of moisture during 1—2 months. The moisture leaked into the crystallization vessel because the solvent had washed out the grease on the stopper. The crystallization process could be accelerated by opening the vessel for *ca.* 1 h to facilitate the penetration of moisture (Found: C, 27.55; H, 4.60; Al, 11.40, Cl, 35.25. $C_{28}H_{55}Al_5Cl_{12}O_{17}$ requires C, 27.50; H, 4.50; Al, 11.00; Cl, 34.75%).

X-Ray Crystal Structure Analysis.—*Crystal data.* $C_{28}H_{55}Al_5Cl_{12}O_{17}$, M = 1 224.1, a = 14.974(15), b = 19.578(15), c = 20.567(15) Å, β = 94.40(7)°, U = 6 012(9) $Å^3$, D_m = 1.366 g cm^{-3}, Z = 4, D_c = 1.352(3) g cm^{-3}, $F(000)$ = 2 512, space group $P2_1/n$, Mo-K_α radiation, λ = 0.710 69 Å, μ = 6.8 cm^{-1}.

A specimen *ca.* 0.6 × 0.7 × 0.4 mm was cut from a large crystal and sealed in a capillary. The space group was determined from Weissenberg photographs. Intensity data were recorded on a Syntex $P2_1$ automated diffractometer using graphite-monochromatized Mo-K_α radiation, at 302(1) K. The intensities of two standard reflections, monitored after every 50 intensity scans, showed no evidence of crystal decay. 4 372 Reflections were collected, of which 3 252 with $I > 3\sigma(I)$ were used for structure analysis.

Structure determination and refinement. The structure was solved by direct methods. The methylene H atoms were introduced in calculated positions. All other H atoms were located from difference Fourier synthesis and refined with the constraints that d(C–H) = 1.08 and d(O–H) = 0.97 Å. An absorption correction was applied following the DIFABS[7] procedure; minimum and maximum values were 0.926 and 1.070, respectively. Blocked full-matrix least-squares refinements were carried out on the positional and anisotropic thermal parameters using the SHELX 76[8] program. Least-squares refinement converged with $R = \Sigma||F_o| - |F_c||/\Sigma|F_o| = 0.0693$ and $R' = \Sigma(w\Delta F^2/\Sigma wF_o^2)^{\frac{1}{2}} = 0.0638$ where $w = 1/\sigma^2(F_o)$. Neutral-atom scattering factors were taken from ref. 9. The scattering factors for Cl, Al, O, and C were corrected for real and imaginary components. For the final cycle of the refinement the maximum Δ/σ was 0.39. Residual electron density in the final difference Fourier map was within −0.38 and 0.48 e $Å^{-3}$.

Additional material available from the Cambridge Crystallographic Data Centre comprises H-atom co-ordinates, thermal parameters, and remaining bond lengths and angles.

Results and Discussion

The addition of ethyl acetate to $AlCl_3$ suspended in n-hexane in a 1:2 ratio gave an air-sensitive product formulated as $[AlCl_3(CH_3CO_2C_2H_5)_2]$. The i.r. spectrum of this compound shows the characteristic ν($AlCl_4^-$) band at 490vs cm^{-1} and ν(C=O) at 1 630vs (br) cm^{-1}. This suggests its ionic character, $[AlCl_2(CH_3CO_2C_2H_5)_4][AlCl_4]$. The structure of this compound is probably similar to that of $[AlCl_2(thf)_4][AlCl_4]$[10] (thf = tetrahydrofuran) which is formed during reaction (2).

$$2AlCl_3 + 4CH_3CO_2C_2H_5 \xrightarrow{\text{n-hexane}} [AlCl_2(CH_3CO_2C_2H_5)_4][AlCl_4] \quad (1)$$

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† *Supplementary data available:* see Instructions for Authors, *J. Chem. Soc., Dalton Trans.*, 1990, Issue 1, pp. xix—xxii.

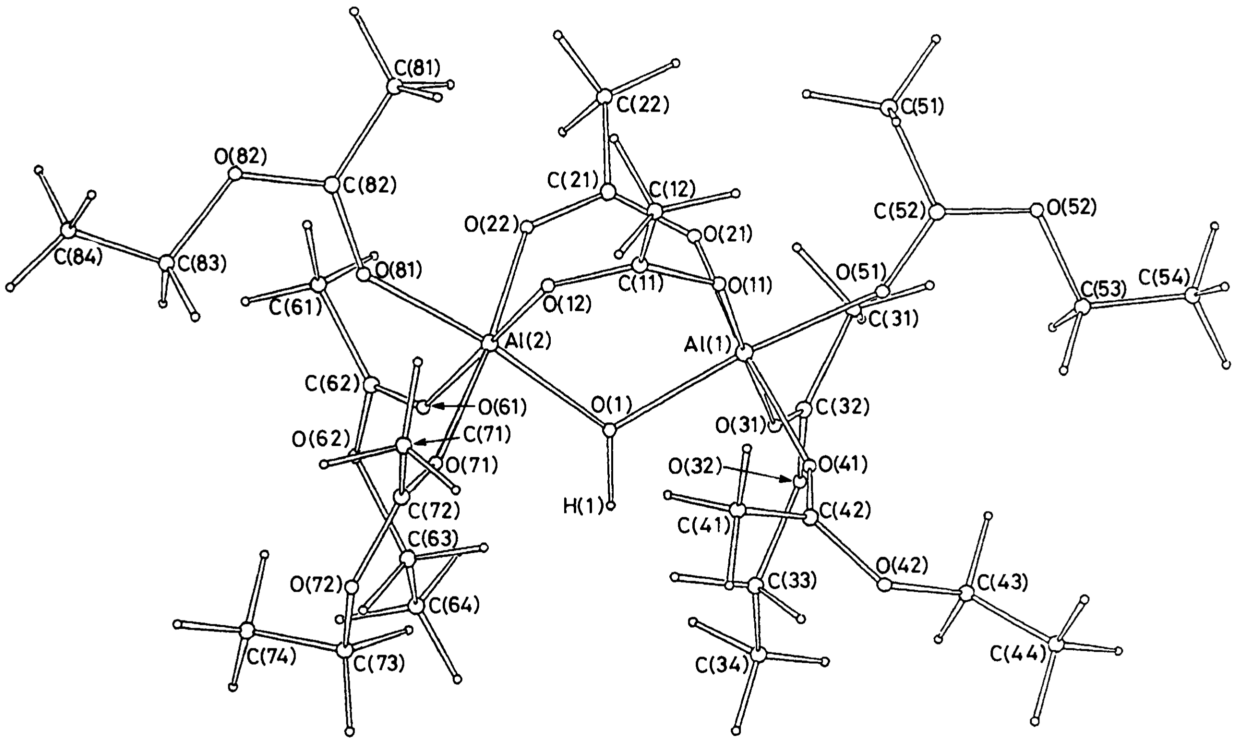

Figure 1. Structure of the $[(CH_3CO_2C_2H_5)_3Al(\mu\text{-}CH_3CO_2)_2(\mu\text{-}OH)Al(CH_3CO_2C_2H_5)_3]^{3+}$ cation

Table 1. Atomic positional parameters with estimated standard deviations in parentheses for di-μ-acetato-μ-hydroxo-hexa(ethyl acetate)dialuminium tris(tetrachloroaluminate)

Atom	*x*	*y*	*z*
Cl(1)	0.956 4(3)	0.429 7(3)	−0.167 7(3)
Cl(2)	1.134 1(3)	0.352 4(3)	−0.078 8(3)
Cl(3)	1.108 2(3)	0.527 7(3)	−0.076 9(3)
Cl(4)	1.169 1(3)	0.447 3(3)	−0.215 8(2)
Cl(5)	0.539 4(3)	0.252 6(3)	−0.141 8(2)
Cl(6)	0.619 3(4)	0.095 4(3)	−0.088 0(3)
Cl(7)	0.657 0(4)	0.235 7(4)	0.007 5(3)
Cl(8)	0.444 3(4)	0.172 4(3)	−0.015 4(3)
Cl(9)	1 045 7(4)	−0.159 5(3)	0.138 0(3)
Cl(10)	0.868 7(4)	−0.087 3(3)	0.208 0(3)
Cl(11)	1.086 0(4)	−0.062 9(3)	0.272 7(3)
Cl(12)	1.010 1(4)	0.013 3(4)	0.133 3(4)
Al(1)	0.871 2(3)	0.215 2(2)	0.165 3(2)
Al(2)	1.084 4(3)	0.243 7(2)	0.138 1(2)
Al(3)	1.092 0(3)	0.439 3(3)	−0.133 7(3)
Al(4)	0.565 1(4)	0.189 4(3)	−0.058 2(3)
Al(5)	1.002 9(4)	−0.075 6(3)	0.188 2(3)
O(1)	0.994 0(5)	0.177 9(4)	0.164 2(3)
O(11)	0.873 5(5)	0.258 0(4)	0.083 9(4)
O(12)	1.016 6(6)	0.271 8(4)	0.063 9(4)
O(21)	0.906 5(6)	0.295 4(4)	0.209 2(4)
O(22)	1.046 5(6)	0.316 3(4)	0.188 5(4)
O(31)	0.865 1(5)	0.168 6(4)	0.244 7(4)
O(32)	0.864 4(6)	0.134 7(5)	0.347 0(4)
O(41)	0.822 4(5)	0.135 1(4)	0.126 0(4)
O(42)	0.764 3(6)	0.049 8(5)	0.067 0(4)
O(51)	0.748 8(5)	0.241 2(5)	0.170 0(4)
O(52)	0.609 9(7)	0.279 4(5)	0.169 8(5)
O(61)	1.159 0(6)	0.212 0(5)	0.210 6(4)
O(62)	1.235 0(6)	0.190 8(7)	0.303 8(5)
O(71)	1.138 4(6)	0.170 6(5)	0.094 7(4)
O(72)	1.215 1(8)	0.096 8(6)	0.041 4(5)
O(81)	1.180 8(6)	0.298 4(5)	0.110 9(4)
O(82)	1.273 4(7)	0.373 6(5)	0.073 6(5)
C(11)	0.933 4(10)	0.275 7(6)	0.048 2(6)
C(12)	0.905 1(8)	0.306 3(7)	−0.017 1(5)
C(21)	0.976 5(10)	0.332 0(7)	0.213 7(6)
C(22)	0.974 6(9)	0.394 7(6)	0.252 9(6)
C(31)	0.797 4(9)	0.239 1(6)	0.327 3(6)
C(32)	0.846 2(8)	0.178 4(7)	0.301 3(7)
C(33)	0.910 9(9)	0.071 3(7)	0.329 2(6)
C(34)	0.924 3(12)	0.028 9(9)	0.387 8(7)
C(41)	0.873 4(9)	0.109 7(8)	0.020 7(7)
C(42)	0.820 7(9)	0.100 5(8)	0.076 4(7)
C(43)	0.703 7(11)	0.039 0(8)	0.117 1(8)
C(44)	0.629 6(12)	−0.003 1(9)	0.090 8(8)
C(51)	0.718 4(9)	0.356 5(7)	0.134 3(7)
C(52)	0.693 6(11)	0.286 9(7)	0.159 0(6)
C(53)	0.584 7(9)	0.214 0(8)	0.194 4(9)
C(54)	0.490 0(10)	0.211 4(8)	0.199 7(9)
C(61)	1.242 5(10)	0.302 3(8)	0.268 8(7)
C(62)	1.207 2(10)	0.232 9(9)	0.256 3(8)
C(63)	1.201 2(11)	0.120 0(9)	0.300 0(7)
C(64)	1.209 3(10)	0.091 6(8)	0.367 8(8)
C(71)	1.146 8(11)	0.180 3(9)	−0.020 8(7)
C(72)	1.163 6(10)	0.150 6(8)	0.045 6(9)
C(73)	1.236 5(8)	0.059 2(9)	0.100 4(7)
C(74)	1.322 2(11)	0.082 3(9)	0.130 3(8)
C(81)	1.130 4(9)	0.415 0(7)	0.090 8(8)
C(82)	1.194 8(10)	0.358 0(8)	0.093 0(7)
C(83)	1.341 3(10)	0.322 4(8)	0.069 1(9)
C(84)	1.420 9(11)	0.352 0(9)	0.048 9(9)

$$2AlCl_3 + 4thf \longrightarrow [AlCl_2(thf)_4][AlCl_4] \qquad (2)$$

The compound $[AlCl_3(CH_3CO_2C_2H_5)_2]$ was dissolved in CH_2Cl_2 and during slow diffusion of n-hexane an oil layer was formed from which after 1—2 months under the influence of moisture colourless crystals were obtained. In the i.r. spectrum of this compound, in addition to ν($AlCl_4^-$) at 490s (br) cm^{-1} and ν(C=O) at 1 630s and 1 690s cm^{-1}, also ν(O–H) frequencies at

Table 2. Principal interatomic distances (Å), bond angles (°), and torsion angles (°) with standard deviations in parentheses

Al(1)–O(1)	1.980(7)	C(42)–O(42)	1.309(16)	Al(2)–O(1)	1.973(7)	C(72)–O(72)	1.313(18)
Al(1)–O(11)	1.873(8)	C(52)–O(52)	1.298(18)	Al(2)–O(12)	1.850(8)	C(82)–O(82)	1.307(18)
Al(1)–O(21)	1.869(8)	O(32)–C(33)	1.483(16)	Al(2)–O(22)	1.873(8)	O(62)–C(63)	1.477(21)
Al(1)–O(31)	1.879(8)	O(42)–C(43)	1.441(17)	Al(2)–O(61)	1.898(9)	O(72)–C(73)	1.433(18)
Al(1)–O(41)	1.885(8)	O(52)–C(53)	1.437(17)	Al(2)–O(71)	1.899(9)	O(82)–C(83)	1.436(17)
Al(1)–O(51)	1.911(8)	C(33)–C(34)	1.465(19)	Al(2)–O(81)	1.914(9)	C(63)–C(64)	1.496(21)
C(11)–O(11)	1.253(14)	C(43)–C(44)	1.453(23)	C(21)–O(21)	1.266(15)	C(73)–C(74)	1.452(20)
C(11)–O(12)	1.265(16)	C(53)–C(54)	1.432(19)	C(21)–O(22)	1.243(15)	C(83)–C(84)	1.416(21)
C(11)–C(12)	1.501(15)	Al(3)–Cl(1)	2.104(6)	C(21)–C(22)	1.469(17)	Al(4)–Cl(5)	2.128(6)
O(31)–C(32)	1.233(16)	Al(3)–Cl(2)	2.111(6)	O(61)–C(62)	1.211(17)	Al(4)–Cl(6)	2.120(7)
O(41)–C(42)	1.223(15)	Al(3)–Cl(3)	2.092(6)	O(71)–C(72)	1.172(19)	Al(4)–Cl(7)	2.063(7)
O(51)–C(52)	1.229(16)	Al(3)–Cl(4)	2.122(6)	O(81)–C(82)	1.247(17)	Al(4)–Cl(8)	2.099(6)
C(32)–C(31)	1.515(17)	Al(5)–Cl(9)	2.068(7)	C(62)–C(61)	1.473(22)	Al(5)–Cl(11)	2.074(7)
C(42)–C(41)	1.451(19)	Al(5)–Cl(10)	2.094(6)	C(72)–C(71)	1.488(22)	Al(5)–Cl(12)	2.082(8)
C(52)–C(51)	1.510(18)	H(1) ··· Cl(12)	2.47(5)	C(82)–C(81)	1.474(20)	O(1) ··· Cl(12)	3.297(9)
C(32)–O(32)	1.284(16)	Al(1) ··· Al(2)	3.331(5)	C(62)–O(62)	1.321(19)		

Al(1)–O(1)–Al(2)	114.8(4)	Al(1)–O(11)–C(11)	135.3(8)	O(12)–Al(2)–O(71)	93.6(4)	Al(1)–O(31)–C(32)–O(32)	−168.2(21)
O(1)–Al(1)–O(11)	94.2(4)	Al(1)–O(21)–C(21)	135.4(8)	O(12)–Al(2)–O(81)	88.3(4)	Al(1)–O(41)–C(42)–O(42)	−170.4(26)
O(1)–Al(1)–O(21)	95.0(4)	Al(1)–O(31)–C(32)	140.8(9)	O(22)–Al(2)–O(61)	89.9(4)	Al(1)–O(51)–C(52)–O(52)	177.9(18)
O(1)–Al(1)–O(31)	86.4(4)	Al(1)–O(41)–C(42)	143.2(9)	O(22)–Al(2)–O(71)	171.8(4)	C(33)–O(32)–C(32)–O(31)	0.9(19)
O(1)–Al(1)–O(41)	91.0(4)	Al(1)–O(51)–C(52)	145.0(9)	O(22)–Al(2)–O(81)	90.3(4)	C(43)–O(42)–C(42)–O(41)	1.7(20)
O(1)–Al(1)–O(51)	173.4(4)	C(32)–O(32)–C(33)	117.0(10)	O(61)–Al(2)–O(71)	82.9(4)	C(53)–O(52)–C(52)–O(51)	−1.1(20)
O(11)–Al(1)–O(21)	91.9(4)	C(42)–O(42)–C(43)	116.0(11)	O(61)–Al(2)–O(81)	90.1(4)	C(34)–C(33)–O(32)–C(32)	−179.6(17)
O(11)–Al(1)–O(31)	176.9(4)	C(52)–O(52)–C(53)	116.4(11)	O(71)–Al(2)–O(81)	85.8(4)	C(44)–C(43)–O(42)–C(42)	162.4(20)
O(11)–Al(1)–O(41)	91.3(4)	O(1)–H(1) ··· Cl(12)	143(6)	Al(2)–O(12)–C(11)	134.1(8)	C(54)–C(53)–O(52)–C(52)	−176.0(17)
O(11)–Al(1)–O(51)	90.6(4)	O(1)–Al(2)–O(12)	94.2(4)	Al(2)–O(22)–C(21)	136.1(8)	Al(2)–O(61)–C(62)–O(62)	167.3(32)
O(21)–Al(1)–O(31)	91.0(4)	O(1)–Al(2)–O(22)	95.8(4)	Al(2)–O(61)–C(62)	141.1(10)	Al(2)–O(71)–C(72)–O(72)	166.2(39)
O(21)–Al(1)–O(41)	173.0(4)	O(1)–Al(2)–O(61)	87.0(4)	Al(2)–O(71)–C(72)	146.4(10)	Al(2)–O(81)–C(82)–O(82)	−175.2(29)
O(21)–Al(1)–O(51)	89.3(4)	O(1)–Al(2)–O(71)	87.8(4)	Al(2)–O(81)–C(82)	139.3(9)	C(63)–O(62)–C(62)–O(61)	−3.2(29)
O(31)–Al(1)–O(41)	85.7(4)	O(1)–Al(2)–O(81)	173.2(4)	C(62)–O(62)–C(63)	117.3(12)	C(73)–O(72)–C(72)–O(71)	3.6(25)
O(31)–Al(1)–O(51)	88.5(4)	O(12)–Al(2)–O(22)	93.4(4)	C(72)–O(72)–C(73)	116.8(12)	C(83)–O(82)–C(82)–O(81)	3.6(32)
O(41)–Al(1)–O(51)	84.4(4)	O(12)–Al(2)–O(61)	176.3(4)	C(82)–O(82)–C(83)	121.0(12)	C(64)–C(63)–O(62)–C(62)	−157.2(22)
						C(74)–C(73)–O(72)–C(72)	−95.4(26)
						C(84)–C(83)–O(82)–C(82)	−178.4(27)

3 600w and at 1 590m cm^{-1} and a band at 1 330 cm^{-1} due to the carboxyl group are seen. The ^{1}H n.m.r. spectrum recorded in CD_3OD solution revealed signals typical for ethyl acetate and acetic acid molecules in a 3:1 ratio. In this compound, as shown by *X*-ray diffraction studies, $[Al_2(\mu\text{-}CH_3CO_2)_2(\mu\text{-}OH)(CH_3CO_2C_2H_5)_6]^{3+}$ cations and $[AlCl_4]^-$ anions are present in a 1:3 ratio. The structure of the di-μ-acetato-μ-hydroxo-hexa(ethyl acetate)dialuminium cation is presented in Figure 1. Atomic parameters are shown in Table 1 and principal interatomic distances, bond angles, and torsion angles in Table 2.

In this homobimetallic dioctahedral aluminium cation the aluminium atoms are surrounded by two bridging $CH_3CO_2^-$ and one OH^- groups and three ethyl acetate molecules. The average Al–O(H) bridge distance is 1.976(7) Å, similar to that in $[Al(OH)(O_2CC_2H_5)_2]$.[11] The average Al–O bridge distance involving the two carboxyl groups is 1.866(9) Å comparable with the value of 1.868(8) Å in $[Al_2(\mu\text{-}CH_3CO_2)(CH_3)_6]^-$.[12] The ethyl acetate molecules co-ordinate to aluminium through the carbonyl oxygen atoms. The average Al–O(carbonyl) distance is 1.895(9) Å and, as may be expected, is longer than the Al–O distance, 1.761(3) Å, in tetrahedral $[AlCl_3(C_6H_5CO_2C_2H_5)]$.[6]

In the crystal of $[Al_2(\mu\text{-}CH_3CO_2)_2(\mu\text{-}OH)(CH_3CO_2C_2H_5)_6][AlCl_4]_3$ there are short intermolecular contacts between chlorine atoms and the carbon atoms of the co-ordinated carbonyl groups of the ethyl acetate molecules. The atoms C(32), C(62), C(52), and C(42) are at 3.324(13), 3.404(15), 3.278(13), and 3.437(15) Å from Cl(5), Cl(5), Cl(7), and Cl(12) respectively. This type of C ··· Cl interaction has been observed earlier, *e.g.* in $[Ti_2\{\mu\text{-}m\text{-}C_6H_4(CO_2Et)_2\}_2Cl_8]$.[3]

The methyl groups C(34), C(54), and C(84) are in *trans* position to C(32), C(52), and C(82) and C(44), C(64) are in anticlinal positions to C(42) and C(62). However, the methyl group C(74) is perpendicular to the ester group. Atoms C(83), C(73), C(63), C(53), C(43), and C(33) are coplanar with corresponding ester groups (see torsion angles in Table 2).

The hydroxyl group O(1)H forms a hydrogen bond with atom Cl(12) (Table 1 and Figure 2). It is rather weak, the observed sharp i.r. band being at 3 600 cm^{-1}.

The data available indicate that $AlCl_3$ in ethyl acetate and thf forms ionic compounds [equations (1) and (2)]. Instead, with ethyl benzoate the molecular compound $[AlCl_3(C_6H_5CO_2Et)]$ is obtained. This observation could be of great importance in the explanation of the different behaviour in ethylene and propylene polymerization processes displayed by the compounds obtained in CH_3CO_2Et, thf, and in aromatic esters.

It is interesting that the bridge unit $Al_2(\mu\text{-}CH_3CO_2)_2(\mu\text{-}OH)$ in the present compound is similar to $Fe_2(\mu\text{-}RCO_2)_2(\mu\text{-}OH)$ postulated for haemerythrine.[13]

Acknowledgements

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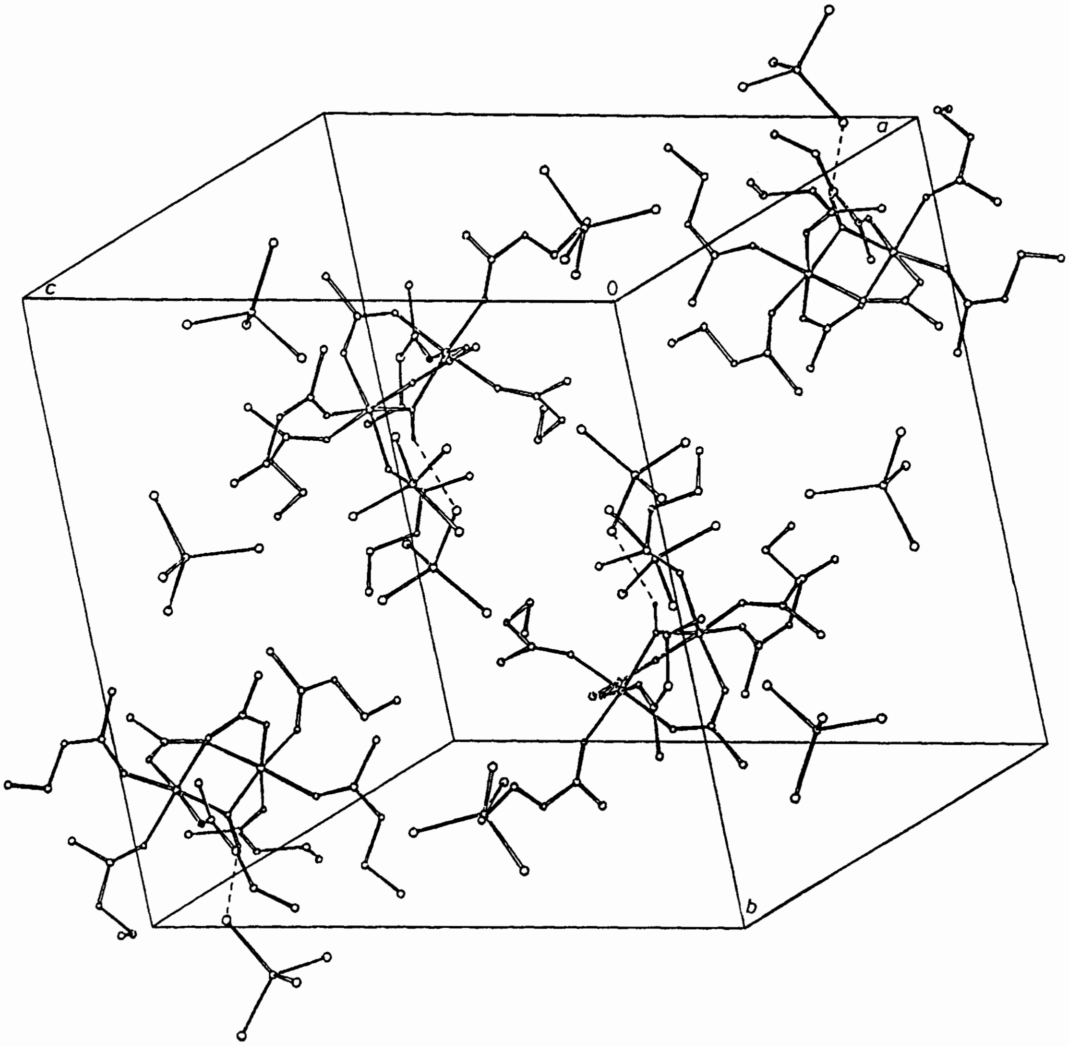

Figure 2. The packing arrangement in the $[Al_2(\mu\text{-}CH_3CO_2)_2(\mu\text{-}OH)(CH_3CO_2C_2H_5)_6][AlCl_4]_3$ crystal. Dashed lines show hydrogen bonds

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