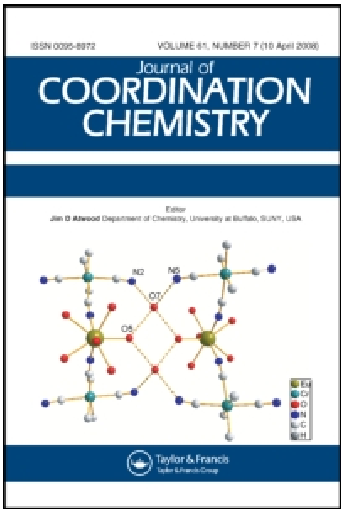

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The Crystal and Molecular Structures of Pb(II) Complexes with Furan-2-Carboxylate and Furan-3-Carboxylate Ligands

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THE CRYSTAL AND MOLECULAR STRUCTURES OF Pb(II) COMPLEXES WITH FURAN-2-CARBOXYLATE AND FURAN-3-CARBOXYLATE LIGANDS

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The crystals of Pb(II) 2-furancarboxylate (title compound I) contain tetrameric structural units Pb_4(2-FCA)$_8$(H_2O_2) in which four Pb(II) ions are bridged by carboxylate oxygen atoms forming a circular moiety. In addition, pairs of Pb(II) ions are bridged by carboxylate oxygen atoms inside this moiety. The molecular pattern observed in Pb(II) 3-furancarboxylate (title compound II) is polymeric. It consists of Pb(3-FCA)$_2$(H_2O) structural units bridged by carboxylate oxygen atoms donated by the furan-3-carboxylate (3-FCA) ligands which are bidentate, using both their carboxylate oxygen atoms for chelation. The coordination around Pb(II) ions is eightfold and ninefold including, apart from carboxylate oxygen atoms, a water oxygen atom and oxygen atoms donated by the furan rings of the ligand molecules. Hydrogen bonds with the water molecule as the donor operate between adjacent ligand molecules. The stereochemical activity of the lone $6s^2$ electron pair on the Pb(II) is observed in title compound II.

Keywords: Furan-2-carboxylic acid; Furan-3-carboxylic acid; Lead(II) complexes; Crystal structure

INTRODUCTION

Heterocyclic carboxylate ligands, apart from exhibiting the ability to act in monodentate and multidentate as well as in bridging modes, frequently act as chelating agents which utilize their hetero-ring atoms such as oxygen or nitrogen to coordinate the metal ions. In particular, this effect is observed when the metal ion is able to accommodate flexible coordination geometries using, for example, its lone electron pair in the coordination process. It is well known [1–3] that the Pb $6s^2$ lone electron pair exhibits strong stereochemical activity and may affect the coordination mode, giving rise to irregular coordination polyhedra with one free site. Therefore, in the course of our studies [4–6] on the interactions of divalent metal ions with furan carboxylate ligands (FCA) we have examined their complexes with Pb(II) ion as

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the coordination center. Consequently, the Pb(II) complexes with the furan-2-carboxylate (2-FCA) and furan-3-carboxylate (3-FCA) ligands have been synthesized and their single crystals grown. The results of an x-ray diffraction study of their crystal and molecular structures are reported in this paper.

EXPERIMENTAL

The title compounds were synthesized by adding of 1 mmol of lead carbonate to 100 mL of aqueous solutions of respective acids (ALDRICH) kept at room temperature. The mixtures were stirred for few hours and then filtered. After a few weeks, well-formed, colorless single crystals deposited in the mother liquid. The dimensions of single crystals used for collecting x-ray data are listed in Table I. X-ray diffraction data are given in Table I. X-ray reflections were measured at room temperature using KUMA KM4 (MoK$_\alpha$ radiation) four circle diffractometer operating in ω–2θ mode. Intensities of two standard reflections were monitored every 200 reflections. Unit cell dimensions and deviations were obtained by least-squares fit to 25 reflections ($15° < 2\theta < 30°$). Reflections were processed using profile analysis and corrected for

TABLE I Crystal data and structural refinement parameters for Pb2-FCA and Pb3-FCA

Identification code	*Pb2-FCA*	*Pb3-FCA*
Empirical formula	$C_{20}H_{14}O_{13}Pb_2$	$C_{10}H_8O_7Pb$
Colour and habit	colourless parallepiped	colourless parallepiped
Crystal size	0.2 × 0.25 × 0.1 mm	0.15 × 0.2 × 0.1 mm
Crystal system	Triclinic	Triclinic
Space group	$P(-1\bar{p})$	$P(-1\bar{p})$
Unit cell dimensions	$a = 9.031(2)$ Å, $\alpha = 64.11(3)°$ $b = 10.759(3)$ Å, $\beta = 73.56(3)°$ $c = 13.648(3)$ Å, $\gamma = 87.52(3)°$	$a = 6.770(1)$ Å, $\alpha = 79.57(3)°$ $b = 8.170(2)$ Å, $\beta = 77.05(3)°$ $c = 11.090(2)$ Å, $\gamma = 82.70(3)°$
Volume	1139(2) Å^3	585.5(2) Å^3
Z	2	2
$F(000)$	804	412
Density (calculated)	2.556 Mg/m^3	2.537 Mg/m^3
Absorption coefficient	14.83 mm^{-1}	14.431 mm^{-1}
Temperature	273(2) K	273(2) K
Radiation	graphite monochromatized Mo-Kα, $\lambda = 0.71073$ Å	
Scan type	$\omega/2\theta$	
Theta range for data collection	2.0–25°	2.0–25°
Index ranges	$-10 \le h \le 10$, $-12 \le k \le 11$, $-16 \le l \le 13$	$-7 \le h \le 8$, $-9 \le k \le 9$, $-0 \le l \le 13$
Standard reflections (number/interval count/decay)	3/100/2%	3/100/3%
Reflections collected	4824	2161
Independent reflections	4012 [R(int)] = 0.1015	2046 [R(int)] = 0.0701
Observed data $\lvert F_o\rvert \ge 2\sigma\lvert F_o\rvert$	2155	1245
No. of variables/restraints	323/43	165/23
$R_{1(obs)}/R_{1(all)}$	0.0476/0.1384	0.0416/0.1276
$wR_{1(obs)}/wR_{1(all)}$	0.1160/0.1737	0.1066/0.1334
S (goodness of fit)	1.128	1.1018
S_r (Restrained goodness of fit)	1.123	1.1012
A,B (weighting scheme parameters)	0.0996, 0.0	0.0950, 0.48
Absorption correction	Ψ-scans	Ψ-scans
Max transmission T_{max}	0.211	0.194
Min transmission T_{min}	0.066	0.046
Largest diff. peak and hole	2.06 and -1.98 e Å^{-3}	1.41 and -1.84 e Å^{-3}

Lorentz factor and polarization effects. Empirical absorption correction based on Ψ-scan was applied. Non-hydrogen atoms were located by direct methods using SHELXLS program [7] and then hydrogen atoms were found by successive Fourier synthesis. Final least squares refinement on F^2 was done for positional parameters of all atoms, anisotropic temperature factors of all non H-atoms and isotropic temperature factors of hydrogen atoms. A weighting scheme was used in the form: $w = 1/[\sigma^2(F_o^2) + (\mathrm{A}^*\mathrm{P})^2 + \mathrm{B}^*\mathrm{P}]$, where $P = [\mathrm{Max}(F_0^2, 0) + 2F_c^2]/3$. A, B parameters are listed in Table I. The refinement was done using the SHELXL97 program [8]. Final atomic coordinates and equivalent isotropic displacement parameters are listed in Tables II and IV, whereas bond lengths and angles are in Tables III and V, respectively. Listings of the observed and calculated structure factors can be requested from the authors.

DISCUSSION

The molecular pattern observed in the title compound (I) denoted as Pb2-FCA contains tetrameric structural units composed of two pairs of Pb(II) ions, related by the center of symmetry, and of two 2-FCA ligand molecules per each metal ion. This tetramer, together with the ligand numbering scheme is shown in Fig. 1. The Pb1 ion is coordinated by the ligand molecule denoted as FCA3 *via* its bidentate carboxylate group: Pb1–O31 2.607(19) Å, Pb1–O32 2.561(16) Å. In the same way, the Pb2 ion is coordinated by the FCAI ligand: Pb2–O11 2.685(17) Å, Pb2–O12 2.356(15) Å. The FCA2 ligand donates one carboxylate oxygen atom O22 for the Pb1 ion coordination [Pb1–O22 2.554(15) Å], the other one, O21, is bonded to the Pb2 ion [Pb2–O21 2.526(16) Å]. The FCA4 ligand molecule coordinates the Pb2 ion using only one carboxylate oxygen O41 [Pb2–O41 2415(13) Å]. The Pb1 ion is also coordinated to the O1 oxygen atom of water molecule [Pb1–O1 2.579(13) Å]. The Pb(II) ions within the tetramer are interconnected by a genuine circular bridging system, which proceeds along the pathway: Pb1–O12–Pb2–O32#1–Pb1#1–O12#1–Pb2#1-O32-Pb1 etc.

In the bridging step (Pb1–O12–Pb2), the FCA1 ligand acts in O:O; O bidentate bridging mode. The same bridging mode is maintained in the successive steps (see, Fig. 1). The Pb1–Pb2 distances range form 4.153(5) Å to 4.498(6) Å (see, Table III for details). An additional "internal" bridging pathway operates *via* the FCA2 ligand molecule, which is active in the O:O;O′ bidentate bridging mode (see, Fig. 1). Pb1 and Pb2 ions exhibit different coordination. The Pb1 ion is coordinated by nine oxygen atoms: one donated by a water molecule, six by the carboxylate groups of the 2-FCA ligand molecules and two by their hetero-ring oxygen atoms (see, Table III). On the other hand, the coordination around the Pb2 ion is eightfold. It is accomplished by carboxylate oxygen atoms as well as by the hetero-ring oxygen atoms, which follows from the analysis of the Pb2–$\mathrm{O_{heteroring}}$ distances. Although slightly longer (2.904–3.249 Å) than the observed Pb2–$\mathrm{O_{carboxylate}}$ contacts, they are still in the range of the possible interactions. Weak hydrogen bonds, provided by the water molecule acting as a donor, link it with the carboxylate oxygen atoms of the ligand molecules FCA1 and FCA4 inside the same tertramer (see Table III).

The three-dimensional pattern observed in the crystals of the title compound (II) (code Pb3-FCA) consists of $\mathrm{Pb(3\text{-}FCA)_2(H_2O)}$ structural units bridged by carboxylate

TABLE II Fractional atomic coordinates and equivalent isotropic displacement(Å^3) for Pb2-FCA

	x	y	Z	U_{eq}
Pb1	0.46863(7)	0.22524(8)	0.41969(7)	0.0367(3)
Pb2	0.93941(8)	0.09088(8)	0.34944(7)	0.0419(3)
O11	0.929(2)	0.237(2)	0.1343(15)	0.061(4)
O12	0.7361(16)	0.2108(15)	0.2844(13)	0.048(4)
C11	0.810(2)	0.275(3)	0.173(2)	0.047(6)
C12	0.740(2)	0.400(2)	0.104(2)	0.043(4)
C13	0.798(3)	0.489(3)	−0.013(2)	0.060(5)
H13	0.8854	0.4850	−0.0670	0.0715
C14	0.687(3)	0.584(3)	−0.0266(3)	0.060(6)
H14	0.6844	0.6558	−0.0959	0.072
C15	0.590(3)	0.560(3)	0.068(2)	0.060(2)
H15	0.5105	0.6142	0.0786	0.072
O13	0.617(2)	0.4412(17)	0.1533(12)	0.050(4)
O21	0.8497(17)	0.0830(16)	0.5451(13)	0.048(4)
O22	0.613(17)	0.0388(16)	0.5391(14)	0.050(4)
C21	0.706(2)	0.042(2)	0.592(2)	0.036(5)
C22	0.638(2)	−0.0033(2)	0.713(2)	0.041(4)
C23	0.496(3)	−0.031(2)	0.782(3)	0.061(5)
H23	0.4050	−0.0315	0.7625	0.074
C24	0.505(4)	−0.0602(3)	0.887(3)	0.085(9)
H24	0.4202	−0.0785	0.9512	0.102
C25	0.650(4)	−0.058(3)	0.886(3)	0.078(7)
H25	0.6847	−0.0838	0.9490	0.094
O23	0.744(2)	−0.011(2)	0.7733(14)	0.058(4)
O31	0.3838(18)	0.3123(18)	0.5765(15)	0.070(5)
O32	0.2488(17)	0.1286(15)	0.6075(15)	0.049(4)
C31	0.280(2)	0.218(3)	0.636(2)	0.052(6)
C32	0.182(2)	0.208(2)	0.7496(18)	0.040(4)
C33	0.052(3)	0.134(3)	0.820(3)	0.072(8)
H33	−0.0009	0.0675	0.8129	0.087
C34	0.011(4)	0.173(4)	0.906(3)	0.098(10)
H34	−0.0745	0.1349	0.9705	0.117
C35	0.107(4)	0.268(4)	0.885(3)	0.091(9)
H35	0.1023	0.3114	0.9316	0.110
O33	0.222(2)	0.299(2)	0.784(2)	0.080(6)
O41	0.9915(16)	0.3259(14)	0.3179(13)	0.045(6)
O42	1.2116(16)	0.243(5)	0.3428(17)	0.063(5)
C41	1.125(2)	0.333(2)	0.334(17)	0.035(4)
C42	1.172(3)	0.460(2)	0.336(2)	0.044(5)
C43	1.089(3)	0.564(4)	0.340(3)	0.095(12)
H43	0.9880	0.5777	0.3361	0.114
C44	1.196(4)	0.652(3)	0.353(3)	0.098(12)
H44	1.1751	0.7332	0.3606	0.118
C45	1.326(4)	0.594(3)	0.353(3)	0.098(12)
H45	1.4125	0.6273	0.3621	0.117
O43	1.3149(17)	0.4774(16)	0.3382(16)	0.059(4)
O1	0.6807(17)	0.3894(16)	0.4067(18)	0.063(5)
H12	0.76(2)	0.38(2)	0.34(2)	0.09501
H11	0.70(3)	0.48(1)	0.39(2)	0.09501

oxygen atoms donated by the 3-FCA ligands which act in O:O; O′ and O:O′ modes of bridging (see Fig. 2). Three of them, however, are also engaged in bridging adjacent Pb(II) ions: O11–Pb#2, O12–Pb#1, O21–Pb#1 (see Fig. 2 and Table V), forming thus a three-dimensional, highly asymmetric molecular framework. The coordination around the Pb(II) ion is strongly irregular. It consists of four carboxylate oxygen atoms mentioned above: Pb–O11 2.341(11) Å, Pb–O12 2.979(10), Pb–O21 2.774(10)

TABLE III Selected bond distances(Å) and angles(°) for Pb2-FCA

Bridging pathway			
Pb1–O32	2.561(16)	O32–Pb1–O12	152.8(5)
Pb1–O12	2.639(14)	O12–Pb2–O32#1	80.9(5)
Pb2–O12	2.356(15)	Pb1–O22–Pb1#1	114.5(5)
Pb2–O32#1	2.717(15)		
Pb1–O22	2.554(15)		
Pb1#1–O22	2.737(15)		
Pb1 coordination			
Pb1–O1	2.579(13)	O1–Pb–O12	70.1(5)
Pb1–O12	2.639(14)	O1–Pb–O31	67.2(5)
Pb1–O22	2.554(15)	O31–Pb1–O32	49.9(5)
Pb1–O22#1	2.737(14)	O32–Pb1–O22	82.8(5)
Pb1–O31	2.607(19)	O22–Pb1–O22#1	65.5(5)
Pb1–O32	2.561(15)	O22#1–Pb1–O12	87.3(5)
Pb1–O13	3.248(18)	O12–Pb1–O13	54.11(5)
Pb1–O43#4	2.905(15)	O13–Pb1–O43#4	64.51(5)
Pb1–O42#4	2.781(17)	O43#4–Pb1–O42#4	56.5(6)
Pb2 coordination			
Pb2–O11	2.685(17)	O11–Pb2–O12	51.2(5)
Pb2–O12	2.356(15)	O12–Pb2–O21	96.6(6)
Pb2–O21	2.526(16)	O21–Pb2–O32#1	98.0(6)
Pb2–O32#1	2.717(14)	O32–Pb2–O41	153.6(6)
Pb2–O41	2.415(13)	O41–Pb2–O42	46.6(5)
Pb2–O42	2.966(15)	O42–Pb2–O23#5	68.5(5)
Pb2–O23#5	2.717(14)	O23#5–Pb2–O21#5	129.5(6)
Pb2–O21#5	2.844(16)	O21#5–Pb2–O11	135.4(5)
Pb-Pb distances			
Pb1–Pb2	4.414(5)		
Pb1–Pb1#2	4.451(5)		
Pb1–Pb2#2	4.498(6)		
Pb2–Pb2#3	4.153(5)		
2-*FCA* 1 *ligand molecule*			
C12–C13	1.410(33)	O13–C12–C13	112(2)
C13–C14	1.391(24)	C12–C13–C14	101(2)
C14–C15	1.259(34)	C13–C14–C15	110(2)
C15–O13	1.368(27)	C14–C15–O13	111(2)
O13–C12	1.291(25)	C15–O13–C12	105(2)
C12–C11	1.495(31)	O11–C11–O12	120(2)
C11–O11	1.203(23)		
C11–O12	1.330(27)		
2-*FCA* 2 *ligand molecule*			
C22–C23	1.321(32)	O23–C22–C23	110(2)
C23–C24	1.359(39)	C22–C23–C24	108(2)
C24–C25	1.306(39)	C23–C24–C25	110(3)
C25–O23	1.398(33)	C24–C25–O23	109(3)
O23–C22	1.407(22)	C25–O23–C22	103(2)
C22–C21	1.444(30)	O21–C21–O22	124(2)
C21–O21	1.274(24)		
C21–O22	1.255(21)		
2-*FCA* 3 *ligand molecule*			
C32–C33	1.308(34)	O33–C32–C33	111(2)
C33–C34	1.372(40)	C32–C33–C34	106(3)
C34–C35	1.245(45)	C33–C34–C35	109(3)
C35–O33	1.385(36)	C34–C35–O33	111(3)
O33–C32	1.343(25)	C35–O33–C32	103(2)
C32–C31	1.510(28)	O31–C31–O32	123(2)
C31–O31	1.228(30)		
C31–O32	1.251(26)		

(*Table Continued*)

TABLE III (Continued)

2-FCA 4 ligand molecule			
C42–C43	1.336(34)	O43–C42–C43	112(2)
C43–C44	1.484(34)	C42–C43–C44	103(2)
C44–C45	1.298(36)	C43–C44–C45	108(2)
C45–O43	1.372(28)	C44–C45–O43	109(2)
O43–C42	1.320(24)	C45–O43–C42	108(2)
C42–C41	1.467(26)	O41–C41–O42	123(2)
C41–O41	1.294(21)		
C41–042	1.190(23)		

Hydrogen bonds

D–H	*d(D–H)*	*d(H···A)*	*∠DHA*	*d(D···A)*	*A*
O1–H12	0.979	2.150	132.97	2.906	O41
O1–H12	0.979	2.296	127.70	2.997	O12

Symmetry codes:
#1 $-x+1, -y, -z+1$; #2 $-x+1, y, z+1$; #3 $-x+2, y, -z+1$; #4 $x-1, y, z$; #5 $-x+2, -y, -z+1$

TABLE IV Fractional atomic coordinates and equivalent isotropic displacements(Å^3) for Pb3-FCA

	x	y	z	U_{eq}
Pb	0.23583(8)	0.35850(7)	0.54323(7)	0.0506(3)
O21	−0.0838(17)	0.2830(12)	0.4503(11)	0.057(3)
022	0.2392(16)	0.1590(12)	0.3917(11)	0.057(3)
C21	0.039(2)	0.1841(18)	0.3937(18)	0.059(4)
C22	−0.029(2)	0.0930(18)	0.3045(16)	0.057(4)
C23	−0.210(2)	0.123(2)	0.2566(16)	0.061(4)
H23	−0.3113	0.2079	0.2754	0.073
C24	−0.216(3)	0.0208(19)	0.1857(17)	0.059(4)
H24	−0.3266	0.0130	0.1510	0.071
O23	−0.023(2)	−0.0838(14)	0.1658(12)	0.067(3)
C25	0.083(3)	−0.0337(18)	0.2318(15)	0.055(4)
H25	0.2170	−0.0740	0.2332	0.066
O12	0.2053(16)	0.5425(15)	0.3095(11)	0.062(3)
O11	0.4950(16)	0.4649(15)	0.3808(11)	0.060(3)
C11	0.401(3)	0.540(2)	0.2841(17)	0.062(4)
C12	0.517(3)	0.607(2)	0.1675(16)	0.063(4)
C13	0.726(3)	0.583(2)	0.1137(17)	0.068(4)
H13	0.8161	0.5128	0.1562	0.082
C14	0.782(3)	0.660(2)	0.0064(18)	0.068(4)
H14	0.9159	0.6709	−0.0361	0.081
O13	0.597(2)	0.7295(16)	−0.0405(11)	0.075(3)
C15	0.448(3)	0.688(2)	−0.0485(18)	0.069(4)
H15	0.3124	0.7048	0.0409	0.83
O1	0.5479(14)	0.1610(12)	0.5682(9)	0.044(2)
H11	0.565(9)	0.078(10)	0.642(5)	0.067
H12 H	0.676(7)	0.151(11)	0.507(2)	0.067

and Pb-O22 2.539(10) Å; water oxygen atom Pb–O1 2.525(9) Å and three bridging carboxylate oxygen atoms contributed by adjacent structural units: Pb–O11#2 2.813(10), Pb–O12#1 3.150(11) and Pb–O21#1 2.987(10) Å. These bond distances fall in the normal range observed in other Pb(II) complexes. There is an evident gap in the coordination geometry in the direction opposite to the molecule FCA1 on Fig. 2. For the comparison of the first coordination spheres of the Pb cations in both title structures see also Fig. 3 which shows more explicitly the distorted neighborhood in structure II. Such distorted coordination is often observed for lead compounds [1–3]. Sometimes it is explained as a result of the lone 6 s electron pair activity [1,2], which occupies the empty space. Larger space occupied by the free electron pair compared to space occupied by bonding Pb–O electrons is in agreement with the VSEPR theory of Gillespie and Nyholm [9,10]. The distance to the nearest hetero-ring oxygen atom lying in this direction O23#3 is rather long-3.693(12) Å and differs significantly from the other Pb–O distances. Also the ring plane orientation of the FCA2#3 molecule discounts inclusion of this ring hetero-atom in Pb coordination. Like in compound Pb2–FCA, the coordinated water molecule acts as a donor of the hydrogen linking it with carboxylate atoms of the ligand molecules (Table V).

TABLE V Selected bond distances (Å) and angles (°) for Pb3-FCA

Pb coordination			
Pb–O1	2.525 (9)	O1–Pb–O11	77.5 (5)
Pb–O11	2.341 (11)	O11–Pb–O12	51.2 (6)
Pb–O12	2.979 (10)	O12–Pb–O21	66.3 (5)
Pb–O21	2.774 (10)	O21–Pb–O22	51.0 (5)
Pb–O22	2.539 (10)	O22–Pb–O11#2	140.0 (5)
Pb–O11#2	2.813 (10)	O11#2–Pb–O12#1	107.1 (6)
Pb–O12#1	3.150 (11)	O12#1–Pb–O21#1	59.3 (5)
Pb–O21#1	2.987 (10)	O21#1–Pb–O23#3	112.3 (5)
Pb–O23#3	3.693 (12)	O23#3–Pb–O1	80.4 (5)
Pb–Pb distances			
Pb–Pb#1	3.893 (1)		
Pb–Pb#2	4.338 (1)		
3–FCA 1 ligand molecule			
C12–C13	1.41 (3)	C15–C12–C13	96.9 (15)
C13–C14	1.24 (2)	C12–C13–C14	116.3 (17)
C14–C13	1.47 (2)	C13–C14–O13	107.0 (16)
O13–C15	1.28 (2)	C14–O13–C15	105.9 (13)
C15–C12	1.51 (2)	O13–C15–C12	112.0 (16)
C12–C11	1.41 (2)	O11–C11–O12	115.0 (14)
C11–C11	1.38 (2)		
C11–C12	1.29 (2)		
3–FCA 2 ligand molecule			
C22–C23	1.42 (2)	C25–C22–C23	100.3 (14)
C23–C24	1.26 (2)	C22–C23–C24	111.7 (17)
C24–O23	1.46 (2)	C23–C24–O23	110.0 (15)
O23–C25	1.28 (2)	C24–O23–C25	104.3 (13)
C25–C22	1.46 (2)	O23–C25–C22	113.2 (13)
C22–C21	1.52 (2)	O21–C21–O22	126.5 (15)
C21–O21	1.23 (2)		
C21–O22	1.34 (1)		

Hydrogen bonds Pb2-FCA

D–H	d(D–H)	d(H···A)	∠DHA	d(D···A)	A	Symmetry code of A
O1–H12	0.979	2.150	132.97	2.906	O41	
O1–H12	0.979	2.296	127.70	2.997	O12	
O1–H11	0.960	2.449	133.07	3.183	O33	#2
O1–H11	0.960	2.452	151.99	3.330	O31	#2

Symmetry codes:
#1 $-x, -y+1, -z+1$; #2 $-x+1, -y+1, -z+1$; #3 $-x, -y, -z+1$; #4 $-x+1, -y, -z+1$, #5 $x+1, y, z$

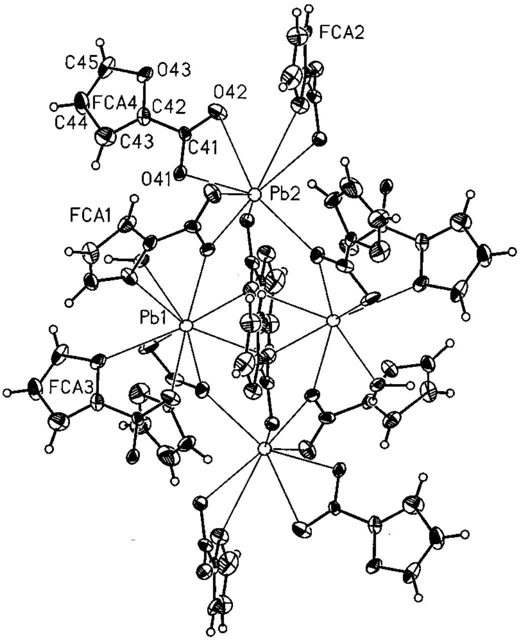

FIGURE 1 The view of Pb2-FCA motif showing Pb1 and Pb2 coordination. The coordination bonds are shown as the thin lines.

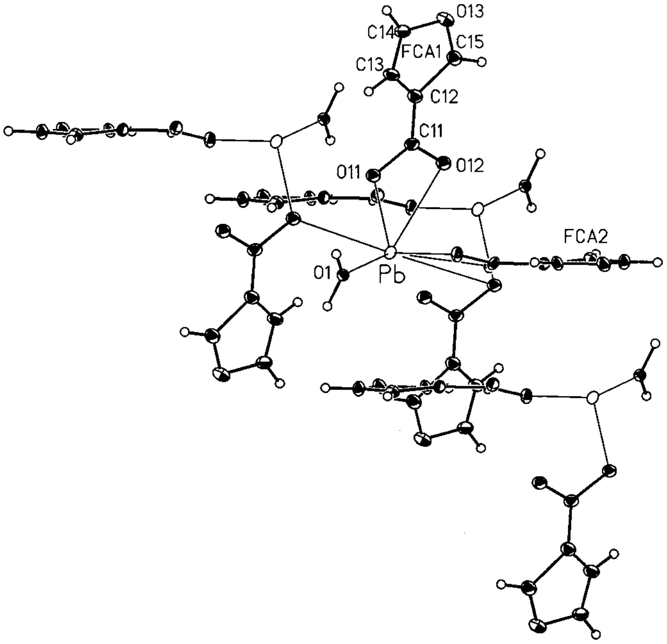

FIGURE 2 The view of Pb3-FCA motif showing Pb coordination. The full coordination is shown only for one Pb cation depicted on the drawing. The coordination bonds are shown as the thin lines.

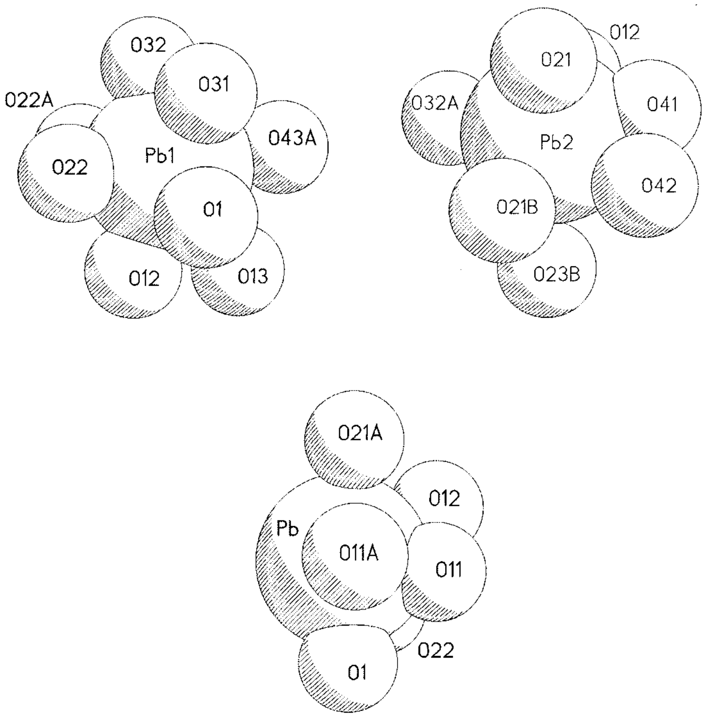

FIGURE 3 The comparison of the first coordination spheres for Pb2-FC (top) and Pb3-FCA (bottom). In all three drawings one of the oxygen atoms is completely shaded by the Pb central atom and therefore not shown on the drawing.

References

[1] G. Svensson, S. Olson and J. Albertsson (1998). *Acta Chem. Scand.*, **52**, 868.
[2] M.A. Malik, P. O'Brien, M. Motevalli, A.C. Jones and T. Leedham (1999). *Polyhedron*, **18**, 1641.
[3] S. Norberg, G. Svensson and J. Albertsson (1999). *Acta Cryst.*, **C55**, 356.
[4] B. Paluchowska, J.K. Maurin and J. Leciejewicz (1996). *Acta Cryst.*, **C52**, 347.
[5] B. Paluchowska, J.K. Maurin and J. Leciejewicz (1996). *Polish J. Chem.*, **70**, 1402.
[6] B. Paluchowska, J.K. Maurin and J. Leciejewicz (1997). *Acta Cryst.*, **C53**, 287.
[7] G.M. Sheldrick (1990). *Acta Cryst.*, **A46**, 467.
[8] G.M. Sheldrick (1997). Program for crystal structure refinement. University of Göttingen.
[9] R.J. Gillespie and R.S. Nyholm (1957). *Q. Rev. Chem. Soc.*, **11**, 239.
[10] R.F.W. Bader, R.J. Gillespie and P.J. MacDougall (1988). *J. Am. Chem. Soc.*, **110**, 7329.