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Structures of Polymeric Zinc 3,3-Dimethylacrylate and Zinc 2-Chlorobenzoate

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Abstract. (1): *catena*-Poly[zinc-tris-μ-(3,3-dimethylacrylato-O,O')-zinc-μ-(3,3-dimethylacrylato-O,O')] [$Zn_2\{\mu\text{-}O_2CCH{:}C(CH_3)_2\}_3\{O_2CCH{:}C(CH_3)_2\}$], M_r = 527·2, orthorhombic, *Pbcn*, a = 19·881 (2), b = 14·488 (2), c = 17·049 (2) Å, V = 4910·7 Å^3, Z = 8, D_x = 1·426 Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 2·04 mm^{-1}, $F(000)$ = 2176, T = 295 K, R = 0·0554 for 2272 unique observed reflections. (2): *catena*-Poly[zinc-tris-μ-(2-chlorobenzoato-O,O')-zinc-μ-(2-chlorobenzoato-O,O')] [$Zn_2(\mu\text{-}O_2CC_6H_4Cl)_3(O_2CC_6H_4Cl)$], M_r = 753·0, monoclinic, $P2_1/n$, a = 11·219 (1), b = 13·536 (1), c = 19·790 (2) Å, β = 103·827 (9)°, V = 2918·2 Å^3, Z = 4, D_x = 1·714 Mg m^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, μ = 5·94 mm^{-1}, $F(000)$ = 1504, T = 295 K, R = 0·0447 for 3996 unique observed reflections. Both structures contain polymeric chains in which binuclear Zn_2(carboxylate)$_3^+$ units with three *syn–syn* bridging ligands are connected by single *syn–anti* carboxylate links.

Introduction. Anhydrous zinc carboxylates adopt a variety of structures. Features common to all so far reported are a tetrahedral coordination of Zn atoms and a bridging function for the carboxylate ligands. The acetate and propionate are both known in two different forms, with exclusively *syn–anti* carboxylate bridges which link the Zn atoms into either two-dimensional sheets or a three-dimensional cross-linked network (Capilla & Aranda, 1979; Clegg, Little & Straughan, 1986*b*; Goldschmied, Rae & Stephenson, 1977; Clegg, Little & Straughan, 1987). The benzoate (Guseinov, Musaev, Usubaliev, Amiraslanov & Mamedov, 1984) and crotonate (Clegg, Little & Straughan, 1986*a*) form polymeric chains in which Zn_2(carboxylate)$_3^+$ binuclear units with three *syn–syn* bridges are connected by single *syn–anti* carboxylates. Only *syn–syn* bridges occur in the structure of the 2-chlorobenzoate reported by Nakacho, Misawa, Fujiwara, Wakahara & Tomita (1976), pairs of carboxylates linking the Zn atoms into chains.

As part of a continuing study of zinc carboxylate structures, in an attempt to understand the factors influencing the choice of structure adopted for a particular carboxylate, and to investigate polymorphism in these compounds, we have determined the structure of zinc 3,3-dimethylacrylate and a second form of zinc 2-chlorobenzoate, which are reported here.

Experimental. The compounds were synthesized by refluxing freshly prepared zinc hydroxide with the corresponding carboxylic acids in water and recrystallized from ethanol (1) or acetone (2). Information for (2) is given in square brackets where it differs from that for (1). Crystal size 0·37 × 0·37 × 0·42 mm [0·08 × 0·20 × 0·40 mm], Stoe–Siemens diffractometer, unit-cell parameters from 2θ values of 32 reflections (20–25°) [(20–40°)] measured at $\pm\omega$. Data collection in ω–θ scan mode, scan width 0·765° [0·68°] below α_1 to 0·765° [0·68°] above α_2, scan time 14–56s, $2\theta_{max}$ 50° [130°], index ranges $h0\rightarrow23$, $k0\rightarrow17$, $l0\rightarrow20$ [$h-13\rightarrow13$, $k0\rightarrow15$, $l-23\rightarrow23$], correction for small decay in intensities of three standard reflections, semi-empirical absorption corrections, transmission 0·303–0·352 [0·168–0·327], 4320 [9807] reflections measured, 4320 [4943] unique, 2272 [3996] with $F > 4\sigma(F)$, [R_{int} = 0·0428].

Structure solution by Patterson and difference syntheses, blocked-cascade least-squares refinement on F, weighting $w^{-1} = \sigma^2(F)$, anisotropic thermal parameters for all non-H atoms, H atoms constrained (C—H 0·96 Å, H—C—H 109·5°), aromatic and olefinic H atoms on angle external bisectors, $U(H) = 1{\cdot}2U_{eq}(C)$, extinction parameter x = 1·0 (2) × 10^{-7} [1·7 (1) × 10^{-6}], whereby $F_c' = F_c/(1 + xF_c^2/\sin2\theta)^{1/4}$. [Twofold disorder of orientation for one 2-chlorobenzoate ligand; occupancy factors refined

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Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^3$) *for* (1)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	*y*	*z*	U_{eq}
Zn(1)	2307·9 (3)	6546·9 (5)	3793·1 (4)	51·8 (2)
Zn(2)	3273·4 (3)	8354·8 (5)	3990·4 (4)	56·0 (3)
O(11)	2054 (2)	7181 (3)	2842 (2)	71 (2)
O(12)	2762 (2)	8361 (3)	3023 (2)	68 (2)
C(11)	2317 (3)	7942 (4)	2642 (4)	58 (3)
C(12)	2069 (3)	8319 (4)	1899 (3)	60 (2)
C(13)	2268 (3)	9071 (4)	1525 (3)	58 (2)
C(14)	2808 (3)	9705 (5)	1795 (5)	104 (4)
C(15)	1956 (3)	9337 (5)	758 (3)	81 (3)
O(21)	3213 (2)	6115 (2)	3780 (2)	61 (2)
O(22)	3893 (2)	7297 (2)	4047 (2)	70 (2)
C(21)	3780 (3)	6448 (4)	3918 (3)	52 (2)
C(22)	4374 (3)	5864 (4)	3938 (4)	69 (3)
C(23)	4425 (3)	4953 (4)	3822 (4)	67 (3)
C(24)	3844 (4)	4342 (5)	3667 (6)	114 (4)
C(25)	5092 (4)	4493 (5)	3865 (6)	123 (4)
O(31)	2006 (2)	7242 (3)	4696 (2)	67 (2)
O(32)	2743 (2)	8367 (3)	4940 (2)	74 (2)
C(31)	2235 (3)	7892 (4)	5113 (3)	60 (2)
C(32)	1906 (3)	8098 (4)	5857 (3)	64 (3)
C(33)	1317 (3)	7833 (5)	6122 (4)	72 (3)
C(34)	835 (4)	7256 (6)	5670 (5)	118 (5)
C(35)	1079 (4)	8117 (6)	6919 (4)	118 (4)
O(41)	1855 (2)	5358 (2)	3864 (2)	58 (2)
O(42)	1056 (2)	4326 (2)	3906 (3)	70 (2)
C(41)	1238 (2)	5161 (4)	3838 (4)	54 (2)
C(42)	741 (3)	5900 (4)	3735 (4)	73 (3)
C(43)	93 (3)	5859 (4)	3731 (5)	93 (3)
C(44)	−331 (4)	6717 (5)	3635 (7)	149 (5)
C(45)	−308 (4)	5021 (6)	3814 (9)	242 (8)

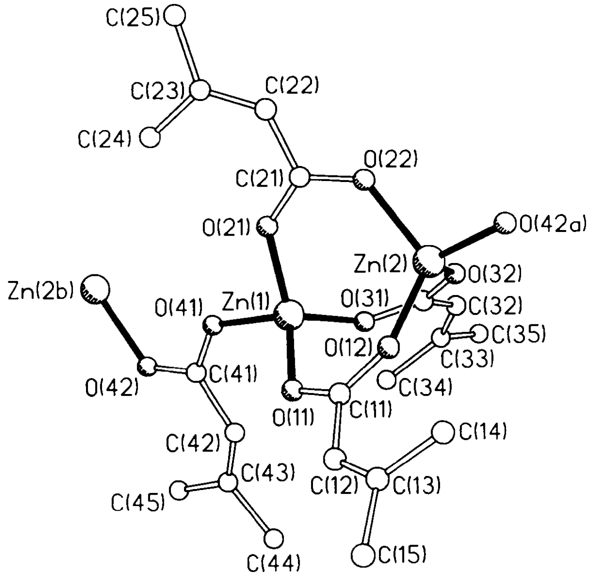

Fig. 1. Structure of the asymmetric unit of (1), with the numbering scheme. H atoms are omitted. Connections to the next units in the chain are shown.

to 0·552:0·448 (2).] $R = 0\cdot0553$ [0·0447], $wR = 0\cdot0384$ [0·0413], for 296 [444] parameters, slope of normal probability plot = 1·59 [1·54], mean $\Delta/\sigma = 0\cdot007$ [0·021], max. $\Delta/\sigma = 0\cdot046$ [0·084], $(\Delta\rho)_{\max} = 0\cdot58$ [0·36], $(\Delta\rho)_{\min} = -0\cdot52$ [$-0\cdot46$] e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974); *SHELXTL* (Sheldrick, 1985) and local computer programs.

Table 2. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^3$) *for* (2)

	x	*y*	*z*	U_{eq}
Zn(1)	4262·5 (4)	2696·9 (4)	2819·5 (2)	46·9 (2)
Zn(2)	2202·8 (4)	951·6 (3)	2585·4 (2)	48·3 (2)
O(11)	5200 (2)	1627 (2)	3356 (1)	58 (1)
O(12)	3663 (2)	550 (2)	3246 (1)	65 (1)
C(11)	4741 (3)	833 (3)	3505 (2)	47 (1)
C(12)	5545 (3)	211 (3)	4053 (2)	45 (1)
C(13)	5317 (3)	−776 (3)	4188 (2)	55 (1)
C(14)	6096 (4)	−1284 (3)	4718 (2)	75 (2)
C(15)	7089 (4)	−833 (4)	5135 (2)	80 (2)
C(16)	7338 (4)	146 (4)	5019 (2)	79 (2)
C(17)	6567 (3)	643 (3)	4487 (2)	65 (2)
Cl(1)	4088 (1)	−1436 (1)	3693 (1)	94 (1)
O(21)	2998 (2)	3037 (2)	3293 (1)	65 (1)
O(22)	1399 (2)	2034 (2)	2937 (1)	62 (1)
C(21)	1895 (3)	2817 (3)	3207 (2)	46 (1)
C(22)	1071 (3)	3557 (3)	3431 (2)	48 (1)
C(23)	1376 (4)	4043 (3)	4066 (2)	56 (1)
C(24)	612 (5)	4752 (3)	4230 (3)	74 (2)
C(25)	−433 (5)	4997 (4)	3760 (4)	95 (3)
C(26)	−764 (4)	4531 (4)	3134 (3)	99 (2)
C(27)	−13 (4)	3788 (3)	2968 (2)	73 (2)
Cl(2)	2682 (1)	3747 (1)	4691 (1)	95 (1)
O(31)	3710 (3)	2430 (2)	1837 (1)	75 (1)
O(32)	2384 (3)	1198 (2)	1657 (1)	73 (1)
C(31)	3015 (4)	1834 (3)	1454 (2)	60 (1)
C(32)	2795 (16)	1830 (18)	653 (13)	52 (5)
C(33)	3630 (6)	2171 (5)	310 (4)	52 (3)
C(34)	3442 (8)	2088 (6)	−397 (3)	69 (3)
C(35)	2384 (7)	1693 (6)	−792 (3)	65 (3)
C(36)	1477 (7)	1352 (6)	−461 (3)	67 (3)
C(37)	1696 (8)	1405 (7)	268 (4)	62 (3)
Cl(3)	5002 (2)	2692 (2)	723 (1)	83 (1)
C(32*x*)	3198 (22)	1997 (24)	742 (18)	64 (8)
C(33*x*)	2355 (9)	1653 (8)	174 (6)	84 (4)
C(34*x*)	2560 (24)	1707 (17)	−507 (6)	110 (8)
C(35*x*)	3638 (14)	2151 (10)	−593 (6)	102 (6)
C(36*x*)	4489 (12)	2520 (11)	−10 (6)	106 (6)
C(37*x*)	4222 (9)	2431 (8)	618 (5)	77 (4)
Cl(3*x*)	957 (3)	1168 (3)	189 (2)	116 (2)
O(41)	5403 (2)	3801 (2)	2848 (1)	55 (1)
O(42)	3806 (2)	4769 (2)	2521 (1)	60 (1)
C(41)	4941 (3)	4649 (3)	2690 (2)	44 (1)
C(42)	5803 (3)	5493 (2)	2727 (2)	46 (1)
C(43)	6322 (3)	5724 (3)	2183 (2)	63 (2)
C(44)	7123 (4)	6497 (3)	2216 (2)	73 (2)
C(45)	7413 (4)	7073 (3)	2806 (2)	78 (2)
C(46)	6892 (4)	6868 (4)	3350 (2)	81 (2)
C(47)	6089 (4)	6085 (3)	3313 (2)	71 (2)
Cl(4)	5945 (1)	5008 (1)	1437 (1)	120 (1)

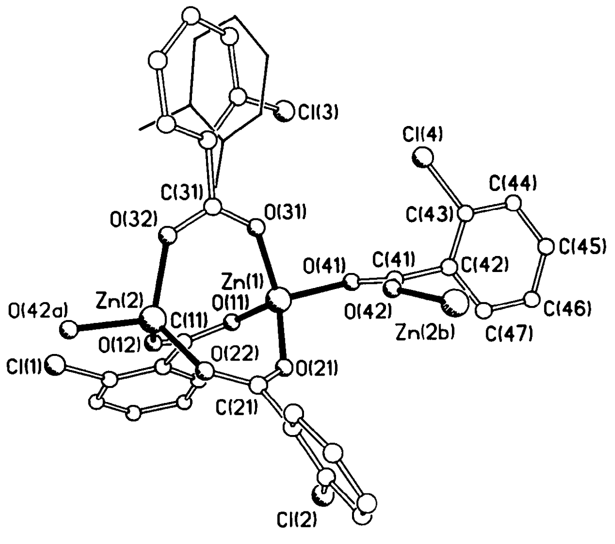

Fig. 2. Structure of the asymmetric unit of (2). All ligands are numbered by a scheme equivalent to that shown for ligand 4 only. The disordered ligand is shown in both orientations.

Table 3. *Bond lengths* (Å) *and angles* (°) *for* (1)

Zn(1)—O(11)	1·930 (4)	Zn(1)—O(21)	1·905 (4)
Zn(1)—O(31)	1·934 (4)	Zn(1)—O(41)	1·947 (4)
Zn(2)—O(12)	1·937 (4)	Zn(2)—O(22)	1·968 (4)
Zn(2)—O(32)	1·931 (4)	Zn(2)—O(42^{i})	1·944 (4)
O(11)—C(11)	1·268 (8)	O(12)—C(11)	1·254 (7)
C(11)—C(12)	1·465 (9)	C(12)—C(13)	1·324 (8)
C(13)—C(14)	1·485 (9)	C(13)—C(15)	1·499 (8)
O(21)—C(21)	1·249 (7)	O(22)—C(21)	1·269 (7)
C(21)—C(22)	1·452 (8)	C(22)—C(23)	1·338 (9)
C(23)—C(24)	1·479 (10)	C(23)—C(25)	1·486 (10)
O(31)—C(31)	1·266 (7)	O(32)—C(31)	1·258 (7)
C(31)—C(32)	1·458 (8)	C(32)—C(33)	1·312 (9)
C(33)—C(34)	1·488 (11)	C(33)—C(35)	1·497 (9)
O(41)—C(41)	1·260 (6)	O(42)—C(41)	1·267 (6)
C(41)—C(42)	1·467 (8)	C(42)—C(43)	1·289 (8)
C(43)—C(44)	1·511 (10)	C(43)—C(45)	1·459 (10)
O(11)—Zn(1)—O(21)	113·2 (2)	O(11)—Zn(1)—O(31)	109·8 (2)
O(21)—Zn(1)—O(31)	118·2 (2)	O(11)—Zn(1)—O(41)	110·6 (2)
O(21)—Zn(1)—O(41)	98·4 (1)	O(31)—Zn(1)—O(41)	105·5 (2)
O(12)—Zn(2)—O(22)	111·9 (2)	O(12)—Zn(2)—O(32)	115·3 (2)
O(22)—Zn(2)—O(32)	107·9 (2)	O(12)—Zn(2)—O(42^{i})	107·1 (2)
O(22)—Zn(2)—O(42^{i})	98·0 (2)	O(32)—Zn(2)—O(42^{i})	115·4 (2)
Zn(1)—O(11)—C(11)	122·1 (4)	Zn(2)—O(12)—C(11)	144·0 (4)
O(11)—C(11)—O(12)	124·9 (6)	O(11)—C(11)—C(12)	114·7 (5)
O(12)—C(11)—C(12)	120·4 (5)	C(11)—C(12)—C(13)	128·5 (5)
C(12)—C(13)—C(14)	125·2 (6)	C(12)—C(13)—C(15)	120·5 (5)
C(14)—C(13)—C(15)	114·2 (5)	Zn(1)—O(21)—C(21)	136·3 (4)
Zn(2)—O(22)—C(21)	129·5 (4)	O(21)—C(21)—O(22)	124·5 (5)
O(21)—C(21)—C(22)	120·9 (5)	O(22)—C(21)—C(22)	114·7 (5)
C(21)—C(22)—C(23)	129·2 (5)	C(22)—C(23)—C(24)	123·8 (6)
C(22)—C(23)—C(25)	120·2 (6)	C(24)—C(23)—C(25)	116·0 (6)
Zn(1)—O(31)—C(31)	136·4 (4)	Zn(2)—O(32)—C(31)	129·1 (4)
O(31)—C(31)—O(32)	124·3 (5)	O(31)—C(31)—C(32)	118·7 (5)
O(32)—C(31)—C(32)	117·0 (5)	C(31)—C(32)—C(33)	129·8 (6)
C(32)—C(33)—C(34)	124·2 (6)	C(32)—C(33)—C(35)	121·0 (6)
C(34)—C(33)—C(35)	114·8 (6)	Zn(1)—O(41)—C(41)	130·4 (3)
C(41)—O(42)—Zn(2^{ii})	120·1 (3)	O(41)—C(41)—O(42)	119·4 (5)
O(41)—C(41)—C(42)	119·6 (5)	O(42)—C(41)—C(42)	121·0 (5)
C(41)—C(42)—C(43)	129·7 (5)	C(42)—C(43)—C(44)	121·3 (6)
C(42)—C(43)—C(45)	125·7 (6)	C(44)—C(43)—C(45)	112·9 (6)

Symmetry operators: (i) $\frac{1}{2}-x, \frac{1}{2}+y,z$; (ii) $\frac{1}{2}-x,-\frac{1}{2}+y,z$.

Table 4. *Bond lengths* (Å) *and angles* (°) *for* (2)

Zn(1)—O(11)	1·949 (3)	Zn(1)—O(21)	1·933 (3)
Zn(1)—O(31)	1·928 (3)	Zn(1)—O(41)	1·960 (3)
Zn(2)—O(12)	1·915 (3)	Zn(2)—O(22)	1·935 (3)
Zn(2)—O(32)	1·926 (3)	Zn(2)—O(42^{i})	1·942 (3)
O(11)—C(11)	1·257 (4)	O(12)—C(11)	1·255 (4)
C(11)—C(12)	1·493 (4)	C(12)—C(13)	1·399 (5)
C(12)—C(17)	1·386 (5)	C(13)—C(14)	1·378 (5)
C(13)—Cl(1)	1·735 (4)	C(14)—C(15)	1·362 (6)
C(15)—C(16)	1·385 (7)	C(16)—C(17)	1·369 (6)
O(21)—C(21)	1·245 (5)	O(22)—C(21)	1·255 (4)
C(21)—C(22)	1·500 (5)	C(22)—C(23)	1·388 (5)
C(22)—C(27)	1·372 (5)	C(23)—C(24)	1·377 (7)
C(23)—Cl(2)	1·723 (4)	C(24)—C(25)	1·352 (7)
C(25)—C(26)	1·360 (9)	C(26)—C(27)	1·400 (8)
O(31)—C(31)	1·245 (5)	O(32)—C(31)	1·241 (5)
C(31)—C(32)	1·545 (26)	C(31)—C(32*x*)	1·489 (37)
C(32)—C(33)	1·362 (26)	C(32)—C(37)	1·409 (21)
C(33)—C(34)	1·369 (10)	C(33)—Cl(3)	1·714 (7)
C(34)—C(35)	1·364 (10)	C(35)—C(36)	1·413 (12)
C(36)—C(37)	1·405 (10)	C(32*x*)—C(33*x*)	1·367 (30)
C(32*x*)—C(37*x*)	1·363 (31)	C(33*x*)—C(34*x*)	1·422 (20)
C(33*x*)—Cl(3*x*)	1·708 (12)	C(34*x*)—C(35*x*)	1·396 (31)
C(35*x*)—C(36*x*)	1·402 (16)	C(36*x*)—C(37*x*)	1·351 (17)
O(41)—C(41)	1·267 (4)	O(42)—C(41)	1·248 (4)
C(41)—C(42)	1·487 (5)	C(42)—C(43)	1·379 (6)
C(42)—C(47)	1·381 (5)	C(43)—C(44)	1·370 (6)
C(43)—Cl(4)	1·732 (4)	C(44)—C(45)	1·378 (6)
C(45)—C(46)	1·369 (7)	C(46)—C(47)	1·382 (7)
O(11)—Zn(1)—O(21)	106·0 (1)	O(11)—Zn(1)—O(31)	114·3 (1)
O(21)—Zn(1)—O(31)	115·9 (1)	O(11)—Zn(1)—O(41)	106·7 (1)
O(21)—Zn(1)—O(41)	110·8 (1)	O(31)—Zn(1)—O(41)	102·8 (1)
O(12)—Zn(2)—O(22)	111·4 (1)	O(12)—Zn(2)—O(32)	115·7 (1)
O(22)—Zn(2)—O(32)	112·1 (1)	O(12)—Zn(2)—O(42^{i})	102·8 (1)
O(22)—Zn(2)—O(42^{i})	110·7 (1)	O(32)—Zn(2)—O(42^{i})	103·4 (1)
Zn(1)—O(11)—C(11)	124·4 (2)	Zn(2)—O(12)—C(11)	140·9 (3)
O(11)—C(11)—O(12)	125·0 (3)	O(11)—C(11)—C(12)	116·3 (3)
O(12)—C(11)—C(12)	118·7 (3)	C(11)—C(12)—C(13)	124·8 (3)
C(11)—C(12)—C(17)	118·6 (3)	C(13)—C(12)—C(17)	116·6 (3)
C(12)—C(13)—C(14)	120·6 (3)	C(12)—C(13)—Cl(1)	122·8 (3)
C(14)—C(13)—Cl(1)	116·5 (3)	C(13)—C(14)—C(15)	121·0 (4)
C(14)—C(15)—C(16)	119·8 (4)	C(15)—C(16)—C(17)	118·8 (4)
C(12)—C(17)—C(16)	123·1 (4)	Zn(1)—O(21)—C(21)	134·2 (2)
Zn(2)—O(22)—C(21)	126·6 (2)	O(21)—C(21)—O(22)	125·8 (4)
O(21)—C(21)—C(22)	117·6 (3)	O(22)—C(21)—C(22)	116·6 (3)
C(21)—C(22)—C(23)	123·2 (3)	C(21)—C(22)—C(27)	117·7 (3)
C(23)—C(22)—C(27)	119·0 (4)	C(22)—C(23)—C(24)	120·7 (4)
C(22)—C(23)—Cl(2)	121·6 (3)	C(24)—C(23)—Cl(2)	117·6 (3)
C(23)—C(24)—C(25)	119·7 (5)	C(24)—C(25)—C(26)	121·1 (5)
C(25)—C(26)—C(27)	119·9 (5)	C(22)—C(27)—C(26)	119·5 (4)
Zn(1)—O(31)—C(31)	137·6 (3)	Zn(2)—O(32)—C(31)	128·8 (2)
O(31)—C(31)—O(32)	125·2 (4)	O(31)—C(31)—C(32)	123·4 (9)
O(32)—C(31)—C(32)	111·4 (9)	O(31)—C(31)—C(32*x*)	105·8 (12)
O(32)—C(31)—C(32*x*)	128·8 (12)	C(31)—C(32)—C(33)	123·4 (14)
C(31)—C(32)—C(37)	117·5 (17)	C(33)—C(32)—C(37)	119·0 (19)
C(32)—C(33)—C(34)	121·8 (11)	C(32)—C(33)—Cl(3)	123·2 (11)
C(34)—C(33)—Cl(3)	115·0 (6)	C(33)—C(34)—C(35)	121·2 (8)
C(34)—C(35)—C(36)	119·1 (6)	C(35)—C(36)—C(37)	119·4 (7)
C(32)—C(37)—C(36)	119·5 (13)	C(31)—C(32*x*)—C(33*x*)	120·2 (21)
C(31)—C(32*x*)—C(37*x*)	123·2 (21)	C(33*x*)—C(32*x*)—C(37*x*)	116·5 (27)
C(32*x*)—C(33*x*)—C(34*x*)	121·7 (19)	C(32*x*)—C(33*x*)—Cl(3*x*)	125·1 (17)
C(34*x*)—C(33*x*)—Cl(3*x*)	113·2 (12)	C(33*x*)—C(34*x*)—C(35*x*)	118·6 (14)
C(34*x*)—C(35*x*)—C(36*x*)	119·5 (12)	C(35*x*)—C(36*x*)—C(37*x*)	118·0 (12)
C(32*x*)—C(37*x*)—C(36*x*)	125·8 (17)	Zn(1)—O(41)—C(41)	117·2 (2)
C(41)—O(42)—Zn(2^{ii})	131·9 (2)	O(41)—C(41)—O(42)	121·1 (3)
O(41)—C(41)—C(42)	117·4 (3)	O(42)—C(41)—C(42)	121·5 (3)
C(41)—C(42)—C(43)	121·8 (3)	C(41)—C(42)—C(47)	120·3 (3)
C(43)—C(42)—C(47)	117·8 (4)	C(42)—C(43)—C(44)	122·1 (4)
C(42)—C(43)—Cl(4)	118·5 (3)	C(44)—C(43)—Cl(4)	119·4 (3)
C(43)—C(44)—C(45)	119·4 (4)	C(44)—C(45)—C(46)	119·5 (4)
C(45)—C(46)—C(47)	120·6 (4)	C(42)—C(47)—C(46)	120·5 (4)

Symmetry operators: (i) $\frac{1}{2}-x,-\frac{1}{2}+y,\frac{1}{2}-z$; (ii) $\frac{1}{2}-x,\frac{1}{2}+y,\frac{1}{2}-z$.

Atomic coordinates and equivalent isotropic thermal parameters are given in Tables 1 and 2, bond lengths and angles in Tables 3 and 4.*

Discussion. The basic structural unit of each polymer is shown in Figs. 1 and 2, and the polymeric chains in Figs. 3 and 4. Both structures are of the same type as that already observed for zinc crotonate (Clegg, Little & Straughan, 1986*a*), with one carboxylate ligand performing a different bridging function from the other three within each Zn_2(carboxlyate)$_4$ unit. This is in contrast to the previously reported structure of a different crystalline form of zinc 2-chlorobenzoate (Nakacho *et al.*, 1976), where all the carboxylates are of the same type, acting as *syn–syn* bridges between pairs of Zn atoms. Since the recrystallization was carried out from acetone in both cases, there is no clear explanation for the production of two quite different structures. The structure found for both of these compounds in this work appears to be a relatively common one among zinc carboxylates with unsaturated side-chain substituents, and is also adopted by some mixed-ligand systems (Clegg, Harbron & Straughan, 1990).

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52522 (43 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

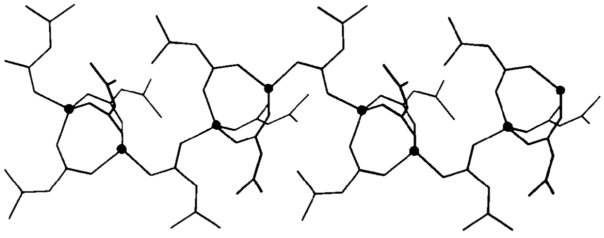

Fig. 3. The polymeric chain structure of (1).

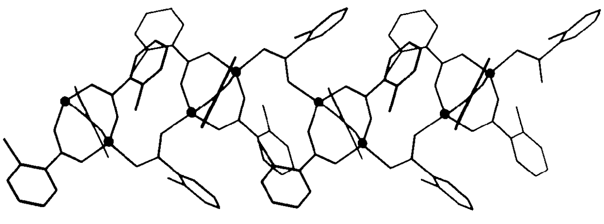

Fig. 4. The polymeric chain structure of (2). Only one component of the disordered ligand is shown.

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Structure of Oxotris(8-quinolinolato-*N*,*O*)niobium(V) Chloroform Solvate

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Abstract. [Nb($C_9H_6NO)_3O$].$CHCl_3$, $M_r = 660{\cdot}74$, triclinic, $P\bar{1}$, $a = 9{\cdot}142\,(2)$, $b = 9{\cdot}693\,(2)$, $c = 16{\cdot}523\,(6)$ Å, $\alpha = 84{\cdot}01\,(2)$, $\beta = 73{\cdot}18\,(2)$, $\gamma = 73{\cdot}42\,(2)^\circ$, $V = 1342{\cdot}9\,(7)$ Å^3, $Z = 2$, $D_x = 1{\cdot}63$ Mg m^{-3}, Mo $K\alpha$ radiation (graphite monochromator, $\lambda = 0{\cdot}71073$ Å), $\mu = 0{\cdot}769$ mm^{-1}, $T = 293$ K, $F(000) = 664$. Final conventional R factor = 0·042 for 3501 observed reflections and 428 variables. No unusual geometric features are present in the molecule. The central niobium is heptacoordinated, with one oxo and three 8-quinolinol ligands. The Nb—O(1) distance, 1·719 (3) Å, indicates a double bond. The average distance Nb—O(8-quinolinol) is 2·053 (3) Å. The Nb—N average distance is 2·385 (3) Å and the average bite angle, N—Nb—O, is 70·8 (1)°.

Introduction. In the course of our structural researches on Ni^{II} complexes we carried out the determination of the crystal structures of complexes containing 8-quinolinol derivatives as ligands (García-Granda & Gómez-Beltrán, 1986; García-Granda, Beurskens, Behm & Gómez-Beltrán, 1987; García-Granda, Jansen, Beurskens, Behm & Gómez-Beltrán, 1988). The present paper extends such investigations to a niobium complex with 8-quinolinol. It is well known that organic reagents, particularly the 8-hydroxyquinoline or its dihalogen derivatives, are of major importance in the analytical chemistry of niobium. This analytical chemistry includes separations (Gibalo, 1970), quantitative gravimetric analysis (Sanz-Medel, 1973), spectrophotometric (Motojima & Hashitani, 1961; Sanz-Medel & Díaz-García, 1981), fluorimetric (Sanz-Medel, Fernández de la Campa & García Alonso, 1987) and phosphorimetric (Kirkbright, Thompson & West, 1970; Sanz-Medel, Martínez García & Díaz García, 1987) methods for the determination of the element.

This structural investigation was mainly undertaken in order to study the influence of the central positive ion (*e.g.* Ni^{2+} or NbO^{3+}) on the coordination distances and bond angles with the organic ligand 8-hydroxyquinoline. However, additional

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