

The 3·5 water molecules which are not bonded to U have been located in two fully-occupied and three half-occupied positions. All 6·5 water molecules of the asymmetric unit appear to be involved in forming hydrogen bonds with each other and with the uranium ions to build up a complex hydrogen-bonding scheme which holds the ions and interstitial water molecules together. A list of thirteen O...O distances in the range 2·60(2)–2·96(5) Å, which appear to be suitable candidates for hydrogen bonding, has been deposited.

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Orthorhombic Anhydrous Zinc(II) Propionate

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Abstract. $\text{Zn}^{2+} \cdot 2\text{C}_3\text{H}_5\text{O}_2^-$, $M_r = 211\cdot5$, orthorhombic, $Pna2_1$, $a = 9\cdot2862$ (9), $b = 4\cdot7937$ (4), $c = 19\cdot0871$ (12) Å, $V = 849\cdot7$ (1) Å³, $Z = 4$, $D_x = 1\cdot653$ Mg m⁻³, $F(000) = 432$, $\lambda(\text{Mo } K\alpha) = 0\cdot71073$ Å, $\mu = 2\cdot93$ mm⁻¹, $T = 293$ K, $R = 0\cdot044$ for 1282 unique observed reflections with $F > 4\sigma(F)$. The structure consists of polymeric sheets in which tetrahedrally coordinated Zn atoms are connected by propionate bridges in a *syn-anti* arrangement.

Introduction. Various polymeric structures have been observed for anhydrous zinc(II) carboxylates. In each case Zn is tetrahedrally coordinated by carboxylate O atoms. The benzoate (Guseinov, Musaev, Usubaliev, Amiraslanov & Mamedov, 1984) and crotonate (Clegg, Little & Straughan, 1986a) form polymeric chains in which $\text{Zn}_2(\text{carboxylate})_2^{\ddagger}$ binuclear units with three *syn-syn* bridges are connected by single *syn-anti* carboxylates. Only *syn-syn* bridges occur in the 2-chlorobenzoate, pairs of carboxylates linking Zn atoms into chains (Nakacho, Misawa, Fujiwara, Wakahara & Tomita, 1976). By contrast, two forms of zinc(II) acetate contain only *syn-anti* bridges, which link the Zn atoms into two-dimensional sheets (Clegg, Little & Straughan, 1986b) or a three-dimensional network (Capilla & Aranda, 1979).

A sheet structure has also been reported for anhydrous zinc(II) propionate (Goldschmied, Rae & Stephenson, 1977). Problems were encountered in this monoclinic structure determination, and were ascribed to a bent crystal and severe crystal decomposition in

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the X-ray beam. We have obtained an orthorhombic form of the compound and report here its structure.

Experimental. Compound obtained from freshly precipitated $\text{Zn}(\text{OH})_2$ and aqueous propionic acid, re-crystallized from ethanol. The factors influencing the form of the crystalline product (monoclinic or orthorhombic) are unclear. Crystal size 0·35 × 0·27 × 0·08 mm, Siemens AED2 diffractometer, cell parameters from 2θ values of 32 reflections measured at $\pm\omega$ ($20 < 2\theta < 25^\circ$). Data collected in ω/θ scan mode, scan width = $1\cdot36^\circ + \alpha$ -doublet splitting, scan time 14–56 s, $2\theta_{\text{max}} 50^\circ$, $h 0 \rightarrow 11$, $k 0 \rightarrow 5$, $l -22 \rightarrow 22$, no significant variation for three standard reflections, semi-empirical absorption correction, transmission 0·388–0·444. 1500 reflections (no equivalents), 1282 with $F > 4\sigma(F)$ for structure determination (Patterson and Fourier recycling methods) and refinement [blocked-cascade minimization of $\sum w\Delta^2$, $\Delta = |F_o| - |F_c|$, $w^{-1} = \sigma^2(F) + 0\cdot00116F^2$]. Anisotropic thermal parameters for non-H atoms, H atoms constrained [$\text{C}-\text{H} = 0\cdot96$ Å, $\text{H}-\text{C}-\text{H} = 109\cdot5^\circ$, $U(\text{H}) = 1\cdot2U_{\text{eq}}(\text{C})$]. Isotropic extinction parameter $x = 2\cdot7$ (5) $\times 10^{-6}$ [$F'_c = F_c/(1 + xF_c^2/\sin 2\theta)^{1/4}$], polar axis direction determined by refinement of $\eta = 1\cdot03$ (9) (Rogers, 1981). Final $R = 0\cdot044$, $wR = 0\cdot053$, max. $\Delta/\sigma = 0\cdot003$, mean = 0·001, slope of normal probability plot = 1·09, $\Delta\rho_{\text{max}} = 1\cdot83 \text{ e } \text{Å}^{-3}$ close to Zn atom, $\Delta\rho_{\text{min}} = -1\cdot04 \text{ e } \text{Å}^{-3}$, scattering factors from *International Tables for X-ray Crystallography* (1974). Programs: *SHELXTL* (Sheldrick, 1985).

Discussion. Final atomic parameters, bond lengths and angles are given in Tables 1 and 2.* The structure consists of polymeric two-dimensional sheets, with *syn-anti* propionate bridges connecting tetrahedrally coordinated Zn atoms (Fig. 1). The arrangement within the sheets is the same as in the monoclinic form of the propionate (Goldschmied, Rae & Stephenson, 1977), the difference being in the stacking of these sheets, between which there is no covalent bonding; the ethyl side chains protrude from each side of the sheets (Fig. 2). In the determination of the orthorhombic structure, no problems were encountered similar to those affecting the monoclinic form.

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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 43470 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^4$)

$U_{eq} = \frac{1}{3}(\text{trace of the orthogonalized } U_{ij} \text{ matrix}).$

	x	y	z	U_{eq}
Zn	6413 (1)	7832 (1)	5000*	375 (2)
O(11)	4365 (5)	7689 (9)	5248 (3)	455 (15)
O(12)	2413 (5)	8516 (11)	5849 (3)	481 (17)
C(11)	3741 (7)	8742 (15)	5769 (4)	412 (21)
C(12)	4585 (8)	10406 (18)	6293 (4)	601 (29)
C(13)	3743 (10)	11930 (30)	6834 (9)	759 (51)
O(21)	6905 (6)	5654 (10)	4178 (3)	467 (16)
O(22)	6593 (6)	1760 (9)	4754 (3)	504 (16)
C(21)	7100 (7)	3053 (13)	4246 (4)	405 (20)
C(22)	8021 (10)	1569 (16)	3716 (4)	562 (26)
C(23)	8701 (13)	3409 (33)	3182 (9)	871 (59)

* Fixed to define the origin along the polar axis.

Table 2. Bond lengths (\AA) and angles ($^\circ$)

Zn—O(11)	1.961 (5)	Zn—O(21)	1.939 (6)
Zn—O(12)	1.977 (6)	Zn—O(22 ^b)	1.948 (5)
O(11)—C(11)	1.256 (9)	O(12)—C(11)	1.248 (8)
C(11)—C(12)	1.500 (11)	C(12)—C(13)	1.486 (1)
O(21)—C(21)	1.267 (8)	O(22)—C(21)	1.243 (9)
C(21)—C(22)	1.504 (11)	C(22)—C(23)	1.488 (18)
O(11)—Zn—O(21)	113.9 (2)	O(11)—Zn—O(12 ^b)	104.2 (2)
O(21)—Zn—O(12 ^b)	112.1 (2)	O(11)—Zn—O(22 ^b)	100.1 (2)
O(21)—Zn—O(22 ^b)	107.8 (2)	O(12 ^b)—Zn—O(22 ^b)	118.3 (2)
Zn—O(11)—C(11)	128.6 (4)	C(11)—O(12)—Zn ⁱⁱⁱ	113.1 (5)
O(11)—C(11)—O(12)	121.2 (7)	O(11)—C(11)—C(12)	120.0 (6)
O(12)—C(11)—C(12)	118.7 (7)	C(11)—C(12)—C(13)	116.7 (7)
Zn—O(21)—C(21)	118.7 (5)	C(21)—O(22)—Zn ^{iv}	134.6 (5)
O(21)—C(21)—O(22)	121.1 (6)	O(21)—C(21)—C(22)	118.6 (6)
O(22)—C(21)—C(22)	120.3 (6)	C(21)—C(22)—C(23)	114.9 (8)

Symmetry operators: (i) $\frac{1}{2} + x, \frac{3}{2} - y, z$; (ii) $x, 1 + y, z$; (iii) $-\frac{1}{2} + x, \frac{3}{2} - y, z$; (iv) $x, -1 + y, z$.

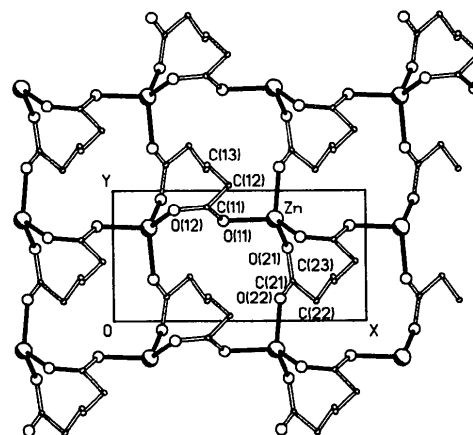


Fig. 1. The structure of one two-dimensional sheet, seen in projection along the *c* axis, with the numbering scheme.

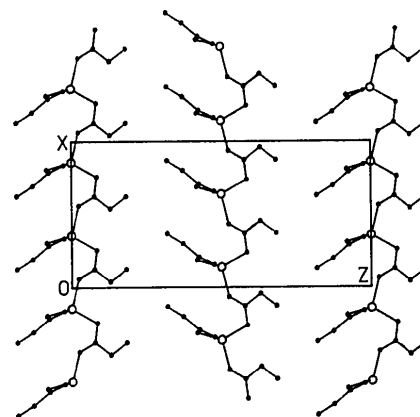


Fig. 2. View along the *b* axis, showing the arrangement of sheets in the unit cell.

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