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# The Chain Polymeric Structure of Zinc(II) Crotonate

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**Abstract.** *catena*-Crotonato-(*O,O'*)-tri-$\mu$-crotonato-(*O,O'*)-dizinc(II), $[Zn_2(\mu\text{-}O_2CCH{:}CHCH_3)_3(O_2CCH{:}CHCH_3)]$, $M_r = 471{\cdot}1$, orthorhombic, $P2_12_12_1$, $a = 9{\cdot}1120\ (5)$, $b = 13{\cdot}3079\ (9)$, $c = 16{\cdot}9553\ (11)$ Å, $V = 2056{\cdot}03$ Å$^3$, $Z = 4$, $D_x = 1{\cdot}522$ Mg m$^{-3}$, $\lambda$(Mo $K\alpha$) $= 0{\cdot}71073$ Å, $\mu = 2{\cdot}43$ mm$^{-1}$, $F(000) = 960$, $T = 293$ K, $R = 0{\cdot}039$ for 3211 unique observed reflections. Binuclear units with three bridging crotonate ligands $Zn_2$(crotonate)$_3^+$ and a Zn···Zn separation of 3·247 (3) Å are connected by single crotonate links to form a polymeric chain. The conformation of the bridging ligands within each binuclear unit is *syn–syn*, that of the *catena* ligand is *syn–anti*.

**Experimental.** Compound obtained from freshly precipitated $Zn(OH)_2$ and crotonic acid in aqueous solution, recrystallized from ethanol by cooling. Crystal size 0·33 × 0·36 × 0·62 mm, Siemens AED2 diffractometer, cell parameters from $2\theta$ values of 32 reflections ($20 < 2\theta < 25°$), 4828 reflection intensities measured by $\omega/\theta$ scan, scan width 0·875° + $\alpha$-doublet splitting, $2\theta$ 3→50°, $h$ −10→10, $k$ 0→15, $l$ 0→20, and some equivalent reflections with $k < 0$. Semi-empirical absorption corrections based on measurements of equivalent reflections at various azimuthal angles, transmission 0·195–0·272; correction for approx. 6% intensity decay of three standard reflections. 3624 unique reflections ($R_{int} = 0{\cdot}051$), 3211 with $F > 4\sigma(F)$. Structure solved from Patterson and difference syntheses, refined by blocked-cascade least squares to minimize $\sum w\Delta^2$; $w^{-1} = \sigma^2(F) + gF^2$, $g$ refined to 0·00044. Anisotropic thermal parameters for all non-H atoms, H atoms constrained to give C–H = 0·96 Å, rigid methyl groups with freely refined orientation, olefinic C–H on external C–C–C angle, $U(H) = 1{\cdot}2U_{eq}(C)$. Extinction $x = 4{\cdot}6\,(16) \times 10^{-7}$ [$F_c'$

Table 1. *Atomic coordinates* (×10$^4$) *and isotropic thermal parameters* (Å$^2$ ×10$^3$)

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

| | $x$ | $y$ | $z$ | $U_{eq}$ |
|---|---|---|---|---|
| Zn(1) | 3533·1 (6) | 3453·7 (4) | 3197·1 (3) | 52·2 (2) |
| Zn(2) | 5605·7 (6) | 5131·9 (4) | 2365·4 (3) | 52·7 (2) |
| O(11) | 2462 (5) | 4692 (2) | 3406 (3) | 83 (2) |
| O(12) | 3815 (4) | 5755 (3) | 2708 (3) | 75 (1) |
| C(11) | 2692 (6) | 5534 (4) | 3092 (3) | 60 (2) |
| C(12) | 1547 (6) | 6313 (4) | 3182 (4) | 71 (2) |
| C(13) | 1604 (6) | 7182 (4) | 2840 (3) | 65 (2) |
| C(14) | 441 (8) | 7980 (5) | 2863 (4) | 88 (3) |
| O(21) | 3628 (5) | 3272 (3) | 2067 (2) | 73 (1) |
| O(22) | 5381 (5) | 4181 (3) | 1512 (2) | 84 (2) |
| C(21) | 4457 (7) | 3474 (4) | 1507 (3) | 65 (2) |
| C(22) | 4332 (10) | 2867 (5) | 787 (3) | 97 (3) |
| C(23) | 3547 (9) | 2055 (5) | 741 (4) | 92 (3) |
| C(24) | 3468 (15) | 1377 (6) | 29 (5) | 148 (5) |
| O(31) | 5376 (5) | 3431 (3) | 3752 (2) | 80 (2) |
| O(32) | 6810 (4) | 4582 (3) | 3204 (2) | 72 (1) |
| C(31) | 6530 (6) | 3915 (4) | 3703 (3) | 58 (2) |
| C(32) | 7687 (6) | 3659 (4) | 4303 (3) | 62 (2) |
| C(33) | 8945 (6) | 4107 (4) | 4343 (3) | 65 (2) |
| C(34) | 10084 (7) | 3894 (6) | 4945 (3) | 78 (2) |
| O(41) | 2168 (4) | 2485 (2) | 3629 (2) | 57 (1) |
| O(42) | 3489 (4) | 1330 (2) | 3061 (2) | 62 (1) |
| C(41) | 2340 (5) | 1586 (3) | 3423 (3) | 48 (1) |
| C(42) | 1191 (6) | 857 (4) | 3608 (3) | 64 (2) |
| C(43) | −46 (6) | 1096 (4) | 3936 (4) | 69 (2) |
| C(44) | −1299 (8) | 403 (6) | 4107 (5) | 102 (3) |

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

| | | | |
|---|---|---|---|
| Zn(1)–O(11) | 1·948 (4) | Zn(1)–O(21) | 1·934 (4) |
| Zn(1)–O(31) | 1·925 (4) | Zn(1)–O(41) | 1·935 (3) |
| Zn(2)–O(12) | 1·920 (4) | Zn(2)–O(22) | 1·934 (4) |
| Zn(2)–O(32) | 1·939 (4) | Zn(2)–O(42$^i$) | 1·935 (3) |
| O(11)–C(11) | 1·257 (6) | O(12)–C(11) | 1·249 (7) |
| C(11)–C(12) | 1·479 (8) | C(12)–C(13) | 1·296 (7) |
| C(13)–C(14) | 1·500 (9) | O(21)–C(21) | 1·242 (7) |
| O(22)–C(21) | 1·262 (7) | C(21)–C(22) | 1·468 (8) |
| C(22)–C(23) | 1·298 (11) | C(23)–C(24) | 1·509 (11) |
| O(31)–C(31) | 1·236 (7) | O(32)–C(31) | 1·252 (6) |
| C(31)–C(32) | 1·504 (8) | C(32)–C(33) | 1·293 (8) |
| C(33)–C(34) | 1·482 (8) | O(41)–C(41) | 1·257 (6) |
| O(42)–C(41) | 1·260 (6) | C(41)–C(42) | 1·462 (7) |
| C(42)–C(43) | 1·296 (8) | C(43)–C(44) | 1·497 (9) |
| O(11)–Zn(1)–O(21) | 108·0 (2) | O(11)–Zn(1)–O(31) | 111·2 (2) |
| O(21)–Zn(1)–O(31) | 116·3 (2) | O(11)–Zn(1)–O(41) | 99·9 (2) |
| O(21)–Zn(1)–O(41) | 108·7 (2) | O(31)–Zn(1)–O(41) | 111·4 (2) |
| O(12)–Zn(2)–O(22) | 114·8 (2) | O(12)–Zn(2)–O(32) | 115·0 (2) |
| O(22)–Zn(2)–O(32) | 111·2 (2) | O(12)–Zn(2)–O(42$^i$) | 96·9 (2) |
| O(22)–Zn(2)–O(42$^i$) | 107·7 (2) | O(32)–Zn(2)–O(42$^i$) | 110·1 (2) |
| Zn(1)–O(11)–C(11) | 126·4 (4) | Zn(2)–O(12)–C(11) | 138·8 (3) |
| O(11)–C(11)–O(12) | 124·6 (5) | O(11)–C(11)–C(12) | 117·6 (5) |
| O(12)–C(11)–C(12) | 117·8 (4) | C(11)–C(12)–C(13) | 123·5 (5) |
| C(12)–C(13)–C(14) | 126·3 (6) | Zn(1)–O(21)–C(21) | 139·2 (4) |
| Zn(2)–O(22)–C(21) | 124·3 (4) | O(21)–C(21)–O(22) | 124·2 (5) |
| O(21)–C(21)–C(22) | 118·0 (6) | O(22)–C(21)–C(22) | 117·8 (5) |
| C(21)–C(22)–C(23) | 123·5 (6) | C(22)–C(23)–C(24) | 124·9 (7) |
| Zn(1)–O(31)–C(31) | 134·5 (4) | Zn(2)–O(32)–C(31) | 130·3 (4) |
| O(31)–C(31)–O(32) | 126·0 (5) | O(31)–C(31)–C(32) | 115·6 (5) |
| O(32)–C(31)–C(32) | 118·3 (5) | C(31)–C(32)–C(33) | 123·6 (5) |
| C(32)–C(33)–C(34) | 124·7 (6) | Zn(1)–O(41)–C(41) | 116·7 (3) |
| C(41)–O(42)–Zn(2$^{ii}$) | 139·4 (3) | O(41)–C(41)–O(42) | 119·8 (4) |
| O(41)–C(41)–C(42) | 118·9 (4) | O(42)–C(41)–C(42) | 121·3 (4) |
| C(41)–C(42)–C(43) | 123·5 (5) | C(42)–C(43)–C(44) | 126·5 (6) |

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

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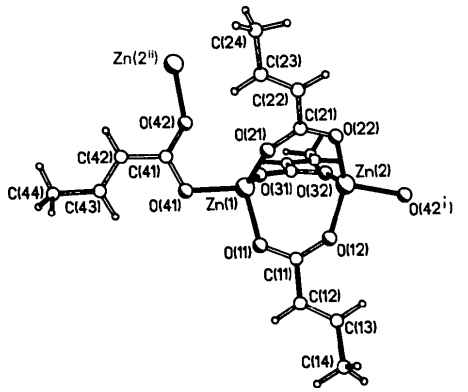


Fig. 1. The $Zn_2$(crotonate)$_4$ unit, showing the atom-numbering scheme and the connections to the adjoining units.

$= F_c/(1 + xF_c^2/\sin 2\theta)^{1/4}$]. Chirality of the individual crystal by refinement of $\eta = 1{\cdot}12$ (4) (Rogers, 1981). $R = 0{\cdot}039$, $wR = 0{\cdot}050$, slope of normal probability plot = 1·44, $(\Delta/\sigma)_{max} = 0{\cdot}022$, $(\Delta/\sigma)_{mean} = 0{\cdot}004$, $(\Delta\rho)_{max} = 0{\cdot}90$ e Å$^{-3}$ close to Zn atoms, $(\Delta\rho)_{max} = 0{\cdot}26$ e Å$^{-3}$ remote from Zn, $(\Delta\rho)_{min} = -0{\cdot}43$ e Å$^{-3}$. Scattering factors from *International Tables for X-ray Crystallography* (1974). Programs: *SHELXTL* (Sheldrick, 1978). Table 1* gives the atom parameters and Table 2 bond lengths and angles. Fig. 1 shows the binuclear asymmetric unit together with its connections to the adjacent units, Fig. 2 shows the polymeric chain structure.

* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42790 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

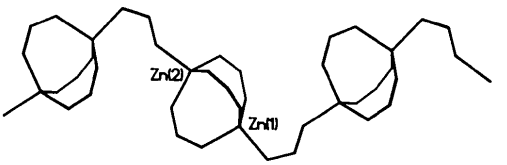


Fig. 2. The backbone of the polymeric chain. Crotonate side chains are not shown.

**Related literature.** With additional donors, zinc crotonate forms discrete binuclear complexes of the type $Zn_2$(crotonate)$_4$(donor)$_2$, with four bridging crotonates and two axial ligands (Clegg, Little & Straughan, 1986), as is commonly observed for carboxylates of divalent metal ions. The triply bridged $[Zn_2(acetate)_3]^+$ cation, closely related to the $Zn_2$(crotonate)$_3$ unit described here, has been observed (Birnbaum, Cotton, Dori & Kapon, 1984). Three crotonate bridges between a pair of Zn atoms are found in $Zn_3$(crotonate)$_6$(quinoline)$_2$, but displaying two different modes of bridging (Clegg, Little & Straughan, 1985).

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# Structure of 2,3-Dimethyl-2,3-di-*p*-tolylbutane

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**Abstract.** $C_{20}H_{26}$, $M_r = 266{\cdot}4$, monoclinic, $P2_1/c$, $a = 9{\cdot}207$ (4), $b = 12{\cdot}791$ (3), $c = 6{\cdot}711$ (2) Å, $\beta = 100{\cdot}93$ (3)°, $V = 776{\cdot}0$ (8) Å$^3$, $Z = 2$, $D_x = 1{\cdot}14$ (1) g cm$^{-3}$, $\lambda$(Mo $K\alpha_1$) = 0·70930 Å, $\mu = 0{\cdot}69$ cm$^{-1}$, $F(000) = 292$, $T = 100$ (2) K, final $R = 0{\cdot}060$ for 1745 observed unique reflections. The title

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