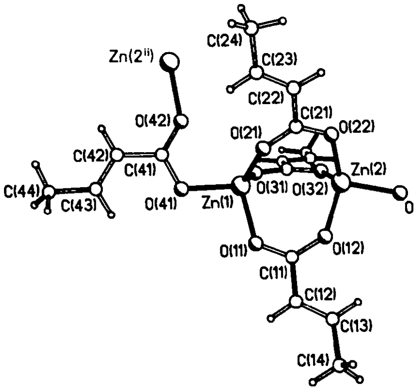

Fig. 1. The $Zn_2(crotonate)_4$ unit, showing the atom-numbering scheme and the connections to the adjoining units.

$= F_c/(1 + xF_c^2/\sin 2\theta)^{1/4}$]. 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.

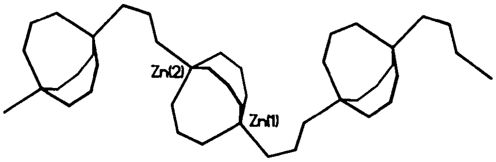

Fig. 2. The backbone of the polymeric chain. Crotonate side chains are not shown.

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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* 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.

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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, $\beta = 100{\cdot}93\ (3)°$, $V = 776{\cdot}0\ (8)\ Å^3$, $Z = 2$, $D_x = 1{\cdot}14\ (1)\ g\ cm^{-3}$, $\lambda(Mo\ K\alpha_1) = 0{\cdot}70930$ 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

compound is a dimer which is located on a crystallographic inversion center. The C—C bond connecting the halves of the dimer is lengthened to 1·598 (1) Å due to crowding of substituents.

Table 1. *Positional and equivalent isotropic thermal parameters with their e.s.d.'s*

$B_{eq} = \frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

	x	y	z	B_{eq}(Å^2)
C(1)	0·6971 (2)	0·0755 (1)	0·4500 (3)	1·52 (3)
C(2)	0·5618 (2)	0·0975 (1)	0·3261 (3)	1·74 (3)
C(3)	0·5537 (2)	0·1329 (1)	0·1282 (3)	1·69 (3)
C(4)	0·6852 (2)	0·1475 (1)	0·0602 (3)	1·66 (3)
C(5)	0·8206 (2)	0·1253 (1)	0·1842 (3)	1·42 (3)
C(6)	0·8307 (2)	0·0873 (1)	0·3816 (2)	1·16 (3)
C(7)	0·9794 (2)	0·0596 (1)	0·5170 (2)	1·14 (3)
C(8)	1·1008 (2)	0·1336 (1)	0·4701 (3)	1·62 (3)
C(9)	0·9713 (2)	0·0795 (1)	0·7405 (3)	1·63 (3)
C(10)	0·4054 (2)	0·1532 (2)	−0·0056 (4)	2·56 (4)

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

C(1)	C(2)	1·389 (1)	C(5)	C(6)	1·397 (1)
C(1)	C(6)	1·400 (1)	C(6)	C(7)	1·535 (1)
C(2)	C(3)	1·392 (2)	C(7)	C(7′)	1·598 (1)
C(3)	C(4)	1·385 (2)	C(7)	C(8)	1·541 (1)
C(3)	C(10)	1·507 (1)	C(7)	C(9)	1·537 (1)
C(4)	C(5)	1·391 (1)			

C(2)	C(1)	C(6)	121·69 (9)	C(1)	C(6)	C(7)	121·46 (8)
C(1)	C(2)	C(3)	121·2 (1)	C(5)	C(6)	C(7)	122·18 (8)
C(2)	C(3)	C(4)	117·76 (9)	C(6)	C(7)	C(7′)	110·2 (1)
C(2)	C(3)	C(10)	120·2 (1)	C(6)	C(7)	C(8)	109·75 (8)
C(4)	C(3)	C(10)	122·0 (1)	C(6)	C(7)	C(9)	109·68 (7)
C(3)	C(4)	C(5)	121·9 (1)	C(7′)	C(7)	C(8)	111·0 (1)
C(4)	C(5)	C(6)	121·94 (9)	C(7′)	C(7)	C(9)	110·7 (1)
C(1)	C(6)	C(5)	116·36 (8)	C(8)	C(7)	C(9)	105·62 (8)

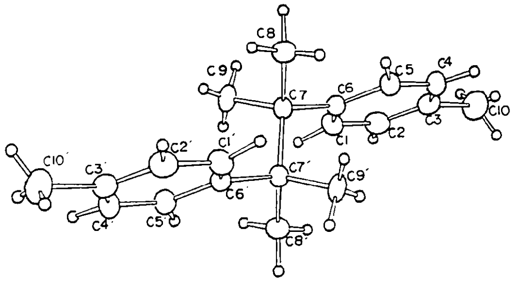

Fig. 1. Plot of the 2,3-dimethyl-2,3-di-*p*-tolylbutane molecule showing atomic-labeling scheme.

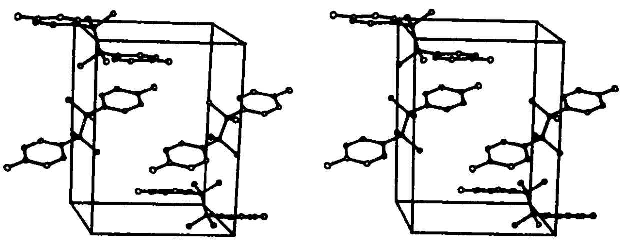

Fig. 2. Stereoview of the molecular packing in the unit cell of the 2,3-dimethyl-2,3-di-*p*-tolylbutane molecule. The axial directions are **a** horizontally to the right, **b** vertically upwards, and **c** out of the page.

Experimental. The compound was obtained as a by-product of the free-radical reaction of the di-*tert*-butyl perester of hexabromo-1,4-cubanedicarboxylic acid in *p*-cymene in the presence of pyridine (Wilberg Decarboxylation Method) and supplied to us by Dr G. W. Griffin. Colorless crystal, approximate dimensions 0·1 × 0·2 × 0·8 mm; Enraf–Nonius CAD-4 diffractometer with graphite-crystal-monochromatized Mo $K\alpha$ radiation; unit-cell dimensions and systematic absences $0k0$, $k = 2n + 1$ and $h0l$, $l = 2n + 1$ uniquely determined space group as $P2_1/c$ with $Z = 2$. Lattice constants determined by least-squares fit of 25 reflections with $35 < 2\theta < 53°$ measured on diffractometer. Three-dimensional intensity data collected in θ:2θ scan mode; total of 2532 reflections collected and merged using *SDP* (Frenz, 1978) to give 2358 unique with 1745 observed having $I > 3\sigma(I)$; $R_{int} = 2·3\%$; $2\theta \leq 60°$; $-12 \leq h \leq 12$, $0 \leq k \leq 18$, $0 \leq l \leq 9$. Data corrected for Lorentz and polarization effects. Three standard reflections measured every 2 h during data collection (011, 302, $\bar{7}30$) showed no significant change in intensity. Absorption as a function of ψ observed to be minimal and therefore not corrected. Structure solved by direct methods using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Initial E map contained ten non-H peaks corresponding to one asymmetric molecular unit. All H atoms located on a difference Fourier map with the exception of the tolyl methyl H atoms which were calculated on the basis of sp^3 geometry and a C—H bond distance of 0·95 Å. All non-H atoms refined anisotropically on F. All H-atom coordinates held fixed with isotropic temperature factors allowed to refine with the exception of the tolyl methyl H atoms for which both coordinates and temperature factors held fixed ($B = 3·5$ Å^2). All refinements carried out using a full-matrix least-squares program. Final $R = 0·060$, $wR = 0·094$ where $w = 1/\sigma(F)^2$ and $\sigma(F)^2 = [\sigma(F)^2_{CS} + (0·04)^2(F^2)^2]$, where $\sigma(F)_{CS}$ is the contribution from counting statistics, $S = 3·01$. In final least-squares cycle $(\Delta/\sigma)_{max} = 0·00$. Comparison of F_o and F_c for strongest reflections gave no indication of secondary extinction. Final difference Fourier map contained no peak larger than 0·47 e Å^{-3} or hole deeper than 0·31 e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974). CAD-4 *SDP* (Frenz, 1978) programs used. Table 1 gives the atomic parameters and Table 2 the bond lengths and angles.* Fig. 1 shows the atomic numbering and Fig. 2 the molecular packing in the unit cell.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42794 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. Other structures exhibiting long C—C bonds due to crowding can be found in Kartt, Beckhaus, Lindner & Ruchart (1983).

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Structure of 1-Iodo-3,5-diphenyl-1λ^4,2,4,6-thiatriazine

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Abstract. $C_{14}H_{10}IN_3S$, $M_r = 379{\cdot}2$, monoclinic, $C2/c$, $a = 24{\cdot}527$ (3), $b = 5{\cdot}117$ (2), $c = 22{\cdot}495$ (3) Å, $\beta = 93{\cdot}85$ (1)°, $V = 2817$ (2) Å^3, $Z = 8$, $D_x = 1{\cdot}79$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 23{\cdot}8$ cm^{-1}, $F(000) = 1472$, $T = 293$ K, $R = 0{\cdot}031$ for 1524 unique observed reflections. The N_3C_2 ring segment is planar within 0·048 (5) Å and the S atom is displaced 0·256 (1) Å from this plane. All of the atoms of the molecule except the I atom are planar within 0·139 (4) Å. The S—I bond distance of 2·665 (2) Å is 0·3 Å longer than value for an S—I single bond.

Experimental. Compound prepared by the reaction of elemental iodine with the dimer of 3,5-diphenyl-1,2,4,6-thiatriazine. Crystals obtained from acetonitrile solutions. Black platelet data crystal 0·08 × 0·26 × 0·58 mm mounted on a glass fiber. Density not measured. Intensities measured with an Enraf–Nonius CAD-4 diffractometer using variable-speed (3 to 17° min^{-1}) ω–2θ scans. Unit cell determined from least squares of angle data for 25 reflections with $16 < 2\theta < 24°$. Analytical absorption correction based on crystal shape varied from 0·66 to 1·00. Data collected to $\sin\theta/\lambda$ of 0·60 Å^{-1}, $0 \le h \le 29$, $-6 \le k \le 0$, $-26 \le l \le 26$. Four standard reflections (0,0,$\overline{10}$, 10,0,0, 0,0,10, 0$\bar{2}$0) varied 4·4% over 26·9 h of data collection; anisotropic-drift correction applied. 3152 reflections measured, 3006 unique ($R_{\rm int} = 0{\cdot}03$), 1555 reflections with $I < 3\sigma(I)$ considered unobserved. Solved by Patterson and Fourier methods. Full-matrix least squares minimized $\sum w(\Delta F)^2$. H atoms constrained to idealized (C—H = 0·95 Å) positions with isotropic $B = 1{\cdot}2 \times B$ of bonded C atom. All non-H atoms refined anisotropically for a total of 172 variables. $R = 0{\cdot}031$, $wR = 0{\cdot}038$, and $S = 1{\cdot}22$, where non-Poisson $w^{-1} = [\sigma^2(I) + 0{\cdot}0016I^2]/4F^2$. Final $(\Delta/\sigma)_{\max} < 0{\cdot}01$, $\Delta\rho_{\max} = 0{\cdot}56$ (5) and $\Delta\rho_{\min} = -0{\cdot}52$ (5) e Å^{-3} on final difference map. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974); programs used those of Enraf–Nonius (1982) *SDP* package. Table 1 gives atom coordinates and Table 2 gives selected bond distances and angles.* Fig. 1 gives the atom numbering.

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