1-Chloro-2-(4-chlorophenyl)-2-(2,4-dichlorophenyl)ethylene*

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Abstract. $C_{14}H_8Cl_4$, orthorhombic, *Pbca*, $a = 21\cdot304$ (6), $b = 22\cdot596$ (7), $c = 5\cdot736$ (2) Å, $Z = 8$, $D_c = 1\cdot53$ g cm^{-3}. The structure was solved by direct methods and refined by full-matrix least squares to $R = 0\cdot072$ for 917 unique reflections. The conformation is similar to that of DDE [1,1-dichloro-2,2-bis(4-chlorophenyl)ethylene].

Introduction. $C_{14}H_8Cl_4$ (I) was investigated as part of a study of analogues and degradation products of DDT. Kerner, Klein & Korte (1972) showed that 1,1-dichloro-2,2-bis(4-chlorophenyl)ethylene (DDE), a degradation product of DDT, is photoisomerized to a

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product which contains a dichlorophenyl group. This product was later found to be a mixture of the *E* and *Z* isomers of 1-chloro-2-(4-chlorophenyl)-2-(2,4-dichlorophenyl)ethylene (Göthe, Wachtmeister, Åkermark, Baeckström, Jansson & Jensen, 1976). One of these isomers was obtained in crystalline form and investigated by X-ray diffraction. The compound is shown to be the *Z* isomer, and not the *E* isomer as was tentatively suggested from UV and NMR spectra (Göthe *et al.*, 1976).

Table 1. *Fractional atomic coordinates* ($\times 10^4$, *for* H $\times 10^3$)

	x	*y*	*z*
Cl(1)	2974 (2)	5574 (2)	13495 (8)
Cl(2)	6997 (2)	3226 (2)	12111 (9)
Cl(3)	4585 (2)	4650 (2)	7299 (7)
Cl(4)	3218 (2)	3043 (2)	7527 (8)
C(1)	3943 (5)	3973 (5)	10397 (22)
C(2)	4056 (5)	4539 (6)	9609 (24)
C(3)	3764 (6)	5036 (6)	10500 (32)
C(4)	3348 (5)	4956 (6)	12283 (28)
C(5)	3213 (6)	4419 (8)	13186 (27)
C(6)	3511 (6)	3914 (6)	12209 (26)
C(7)	4960 (6)	3398 (5)	10090 (24)
C(8)	5377 (6)	3122 (6)	8590 (25)
C(9)	5998 (7)	3074 (6)	9219 (27)
C(10)	6221 (6)	3294 (6)	11291 (29)
C(11)	5814 (7)	3575 (6)	12721 (26)
C(12)	5183 (5)	3622 (6)	12179 (24)
C(13)	4288 (6)	3448 (6)	9474 (22)
C(14)	4003 (6)	3023 (6)	8282 (23)
H(3)	383 (7)	539 (6)	994 (29)
H(5)	292 (6)	429 (7)	1423 (25)
H(6)	350 (5)	351 (6)	1308 (22)
H(8)	531 (6)	295 (6)	701 (23)
H(9)	629 (6)	286 (5)	835 (24)
H(11)	602 (7)	366 (6)	1406 (22)
H(12)	499 (5)	382 (5)	1276 (21)
H(14)	417 (5)	261 (5)	784 (24)

Table 2. *Interatomic distances* (Å) *with estimated standard deviations in parentheses*

Cl(1)–C(4)	1·752 (13)	C(11)–C(12)	1·382 (18)
Cl(2)–C(10)	1·725 (12)	C(12)–C(7)	1·383 (19)
Cl(3)–C(2)	1·758 (13)	C(13)–C(1)	1·492 (16)
Cl(4)–C(14)	1·728 (13)	C(13)–C(7)	1·480 (17)
C(1)–C(2)	1·378 (17)	C(13)–C(14)	1·326 (18)
C(2)–C(3)	1·381 (18)	C(3)–H(3)	0·87 (14)
C(3)–C(4)	1·365 (21)	C(5)–H(5)	0·91 (14)
C(4)–C(5)	1·350 (21)	C(6)–H(6)	1·04 (12)
C(5)–C(6)	1·421 (21)	C(8)–H(8)	1·00 (13)
C(6)–C(1)	1·394 (18)	C(9)–H(9)	0·92 (13)
C(7)–C(8)	1·385 (18)	C(11)–H(11)	0·91 (13)
C(8)–C(9)	1·375 (19)	C(12)–H(12)	0·70 (11)
C(9)–C(10)	1·372 (21)	C(14)–H(14)	1·03 (12)
C(10)–C(11)	1·353 (20)		

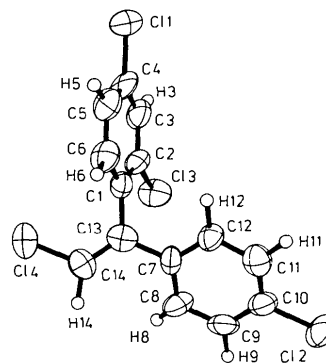

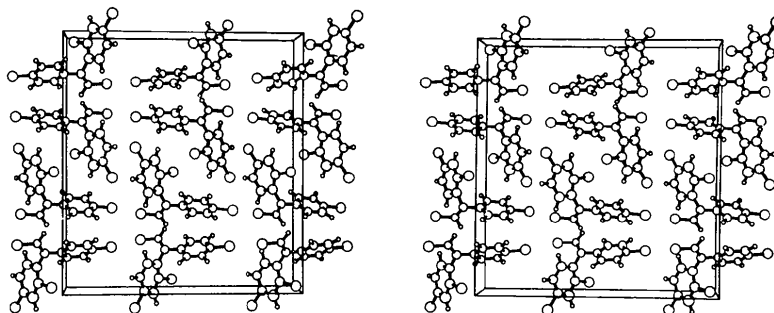Fig. 1. Perspective view of the molecule showing the numbering.

Fig. 2. Stereoview of the packing of (I) along **c** with **a** to the right and **b** up.

Table 3. *Interatomic angles* (°) *with estimated standard deviations in parentheses*

Cl(1)–C(4)–C(3)	119·1 (10)	C(7)–C(8)–C(9)	119·4 (13)
Cl(1)–C(4)–C(5)	117·9 (11)	C(8)–C(9)–C(10)	122·0 (13)
Cl(2)–C(10)–C(9)	122·4 (11)	C(9)–C(10)–C(11)	118·2 (12)
Cl(2)–C(10)–C(11)	119·4 (12)	C(10)–C(11)–C(12)	121·5 (13)
Cl(3)–C(2)–C(1)	119·4 (10)	C(11)–C(12)–C(7)	120·1 (12)
Cl(3)–C(2)–C(3)	116·9 (10)	C(12)–C(7)–C(8)	118·7 (11)
Cl(4)–C(14)–C(13)	123·6 (10)	C(13)–C(1)–C(2)	122·4 (11)
C(1)–C(2)–C(3)	123·7 (12)	C(13)–C(1)–C(6)	120·8 (11)
C(2)–C(3)–C(4)	117·4 (12)	C(13)–C(7)–C(8)	120·5 (12)
C(3)–C(4)–C(5)	123·0 (13)	C(13)–C(7)–C(12)	120·8 (11)
C(4)–C(5)–C(6)	118·4 (13)	C(14)–C(13)–C(1)	122·2 (11)
C(5)–C(6)–C(1)	120·8 (12)	C(14)–C(13)–C(7)	121·8 (11)
C(6)–C(1)–C(2)	116·6 (11)		

Thin needle-shaped crystals were grown from a methanol solution. Systematic absences (0*kl*, *k* odd; *h*0*l*, *l* odd; *hk*0, *h* odd) showed the space group to be *Pbca* (No. 61). A single crystal with dimensions 0·5 × 0·05 × 0·03 mm was mounted on an automatic diffractometer (PW 1100) using graphite-mono-chromatized Cu $K\alpha$ radiation. 2048 unique reflections within $\theta = 60°$ were collected using the $\theta/2\theta$ scanning mode with a scanning speed (θ) of 0·04° s^{-1} and a scan width of 1·50°. The data were corrected for Lorentz and polarization effects, but not for absorption $(\mu = 76·0$ cm$^{-1})$. Accurate cell dimensions were obtained by least-squares refinement of 16 reflections with $\theta > 10°$.

The structure was solved by direct methods using *MULTAN* (Germain, Main & Woolfson, 1971). The four Cl atoms and 11 C atoms were correctly located in the best E map. The positions of the remaining three C atoms were calculated and added before the first two cycles of isotropic refinement, which reduced the R factor from 0·26 to 0·11. After two cycles of aniso-tropic refinement a difference Fourier map revealed all the H atoms. Parameters were refined by full-matrix least squares using anisotropic temperature factors for the non-hydrogen atoms. The H atoms were given the isotropic temperature-factor equivalent of their at-tached C atom, and only their positional parameters were refined. Unit weights were used. The scattering factors were those of *International Tables for X-ray Crystallography* (1974). The final R value was 0·072

for the 917 unique reflections having $\sigma(I)/I < 0·50$.* The final positional parameters are listed in Table 1. The atomic labelling used is indicated in Fig. 1. The packing of the molecule is shown in Fig. 2.

Discussion. Selected interatomic distances and angles are given in Tables 2 and 3. The atomic labelling follows the system used in previous DDT-type struc-tures (Fig. 1). The C–C(aromatic) and Cl–C(aromatic) interatomic distances are in agreement with those found in other DDT analogues (Hovmöller, Smith & Kennard, 1978, and references therein). The length of the double bond C(13)–C(14) is similar to that of DDE – the only DDT analogue so far reported with a C(13)–C(14) double bond (Shields, Kennard & Robinson, 1977). The C(13)–C(14) double bond is delocalized, making the two bonds between C(13) and the atoms closest to it in the phenyl rings 1·492 and 1·480 Å. A similar shortening of the C(13)–C(7) and C(13)–C(1) bonds is observed in DDE [1·485 (10) Å]. The length of the corresponding bonds in the 14 DDT analogues with a C(13)–C(14) single bond is 1·536 (20) Å.

The conformation of (I) closely resembles that of DDE. Owing to the C(13)–C(14) double bond there are only two conformational parameters with any appreciable degree of freedom. These are the torsional angles C(14)–C(13)–C(1)–C(6) and C(14)–C(13)–C(7)–C(8). For (I) these angles are +69 and +34°; for DDE molecule (1) these are +57 and +52° and for DDE molecule (2) +62 and +48°. The phenyl groups seem to avoid each other as much as possible, making an angle of 86° between the planes through the rings for (I) and 82 and 84° respectively for the two crystallographically independent molecules of DDE.

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tert-Butylimido-λ^4-thio(phenylthio)methyl *p*-Tolyl Sulfone

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Abstract. $C_{18}H_{21}NO_2S_3$, $M_r = 379.5$, monoclinic, $P2_1/n$, $a = 12.275$ (4), $b = 11.010$ (6), $c = 14.722$ (5) Å, $\beta = 96.37$ (2)°, $V = 1977.5$ Å³, $Z = 4$, $D_c = 1.28$ g cm⁻³. Full-matrix least-squares refinement gave $R = 0.051$ and $R_w = 0.053$ for 217 parameters and 1410 significant reflexions $[I > 3\sigma(I)]$. C=S and S=N distances are 1.662 (7) and 1.534 (6) Å respectively; the mean C–S distance is 1.733 (7) Å.

Introduction. Holm, Boerma, Nilsson & Senning (1976) showed that crystalline α-chlorosulfenyl

chlorides react with *tert*-butylamine to give thione-*S*-imides. The title compound with the formula

$$4\text{-}CH_3C_6H_4SO_2{\large\diagdown}_{C_6H_5S}{\Large\diagup}C{=}S{\diagup}{\diagdown}N{-}C(CH_3)_3$$
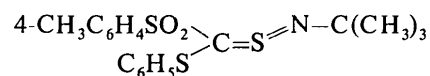
was obtained by this reaction as yellow crystals with m.p. 118–120°C. The crystals were kindly provided by A. Senning.

A crystal of dimensions $1.0 \times 0.6 \times 0.3$ mm was mounted about *b*. Lattice type and space group were established from photographs taken with Cu and Mo radiation.

Intensities were measured out to $2\theta_{max} = 40°$ with a Picker FACS-1 diffractometer. Monochromatic Mo $K\alpha$ radiation was used with a scintillation counter and a pulse-height analyzer. 1410 independent reflexions with $I > 3\sigma(I)$, according to counting statistics, were used in the subsequent calculations. No corrections were made for absorption.

The structure was determined with *MULTAN* (Germain, Main & Woolfson, 1971). Coordinates, anisotropic thermal parameters and a scale factor were refined by full-matrix least-squares calculations. The calculated positions of 18 H atoms were included in the structure-factor calculations but their coordinates and thermal parameters were not refined. The final $R = 0.051$ and $R_w = 0.053$ for 217 parameters. Atomic coordinates are listed in Table 1.*

Discussion. Bond distances are given in Table 2, and angles in Table 3. The numbering of atoms is shown in Fig. 1.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33699 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and their standard deviations*

	x	y	z
S(1)	3115 (1)	4740 (2)	5684 (1)
S(2)	1830 (1)	4598 (2)	7275 (1)
S(3)	4032 (1)	3660 (2)	7498 (1)
O(1)	1516 (4)	5828 (4)	7049 (4)
O(2)	1994 (4)	4248 (5)	8223 (3)
N	5059 (4)	3625 (5)	6991 (4)
C(1)	3036 (4)	4294 (5)	6799 (4)
C(2)	6053 (5)	3015 (7)	7427 (5)
C(3)	6022 (6)	2696 (8)	8419 (6)
C(4)	6975 (6)	3855 (10)	7321 (7)
C(5)	6141 (8)	1836 (9)	6905 (7)
C(6)	850 (4)	3633 (5)	6677 (3)
C(7)	924 (5)	2391 (6)	6820 (5)
C(8)	199 (6)	1625 (7)	6355 (5)
C(9)	4330 (6)	2910 (7)	10716 (5)
C(10)	−708 (6)	3342 (8)	5592 (5)
C(11)	36 (6)	4092 (6)	6069 (5)
C(12)	3508 (6)	3760 (8)	10174 (5)
C(13)	3599 (4)	3428 (5)	5160 (4)
C(14)	3358 (6)	2253 (7)	5414 (5)
C(15)	3759 (6)	1274 (7)	4967 (5)
C(16)	4376 (7)	1461 (10)	4269 (6)
C(17)	4605 (7)	2619 (12)	4007 (6)
C(18)	4217 (6)	3610 (8)	4453 (5)