

Table 3. *Inter-plane angles*

The angles refer to the angle between the normals of the respective planes. The P—S—C plane includes the lead C atom of the corresponding phenyl ring.

Plane	$\sigma(\text{plane})$	Atoms in plane	Angle to P—S—C	Angle to hub*
Phenyl 1	0.003 Å	C(1),C(2),C(3),C(5),C(6)	50.3°	43.9°
Phenyl 2	0.006	C(7),C(8),C(9),C(10),C(11),C(12)	55.2	42.1
Phenyl 3	0.005	C(13),C(14),C(15),C(16),C(17),C(18)	10.6	79.4
Phenyl 1'	0.009	C(1'),C(2'),C(3'),C(4'),C(5'),C(6')	50.0	43.4
Phenyl 2'	0.005	C(7'),C(8'),C(9'),C(10'),C(11'),C(12')	57.5	40.0
Phenyl 3'	0.010	C(13'),C(14'),C(15'),C(16'),C(17'),C(18')	12.2	78.1

\* The hub is the plane through the three C atoms bonded to the P atom.

The conformational angles listed in Table 3 indicate a distinct departure from the  $C_3$  symmetry normally assumed to be the ground-state conformation of molecules of this type. However, calculations performed by Andose & Mislow (1974) in their investigation of the 'two-ring flip' mechanism for stereoisomerization of trimesitylmethane suggest that the observed conformation is easily accessible from the ground state. It is interesting to note that their calculations show large variations in the central C(phenyl)—C—C(phenyl) bond angles in the transition conformations although the average angle is the same as in the ground-state conformation. These results are consistent with the idea that the average value of the C—P—C angle in phosphine sulfides is characteristic of the bulk of the substituents on the central atom, while the variation in C—P—C angles is a function of the conformation of the molecule.

The authors would like to thank Dr P. M. Boorman for providing the sample and for helpful discussions

and Mr B. S. Misener for technical assistance. This work was supported by the National Research Council of Canada (Grant A5881 to K. A. Kerr).

#### References

- ANDOSE, J. D. & MISLOW, K. (1974). *J. Am. Chem. Soc.* **96**, 2168–2176.  
 CAMERON, T. S., HOWLETT, K. D., SHAW, R. A. & WOODS, M. (1973). *Phosphorus*, **3**, 71–73.  
 DREISSIG, W. & PLIETH, K. (1972). *Acta Cryst.* **B28**, 3478–3483.  
 DREISSIG, W., PLIETH, K. & ZÄSKE, P. (1972). *Acta Cryst.* **B28**, 3473–3477.  
 GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* **A27**, 368–376.  
*International Tables for X-ray Crystallography* (1974). Vol. IV, p. 99. Birmingham: Kynoch Press.  
 JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory Tennessee.  
 KERR, K. A., BOORMAN, P. M., MISENER, B. S. & VAN ROODE, J. G. H. (1977). *Can. J. Chem.* **55**, 3081–3085.  
 TOLMAN, C. A. (1977). *Chem. Rev.* **77**, 313–348.

*Acta Cryst.* (1978). **B34**, 3787–3790

### *N,N'*-Bis(3,4-dichlorophenyl)urea

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(Received 9 January 1978; accepted 14 July 1978)

**Abstract.**  $C_{13}H_8Cl_4N_2O$ , monoclinic,  $P2_1/n$ ,  $a = 11.713$  (3),  $b = 25.499$  (7),  $c = 9.391$  (5) Å,  $\beta = 92.13$  (4)°,  $V = 2802.87$  Å<sup>3</sup>,  $Z = 8$  (there are two molecules in the asymmetric unit),  $D_c = 1.66$  g cm<sup>-3</sup>.

The structure consists of discrete molecules, linked together by hydrogen bonds and stacked in columns along the  $c$  axis. The columns are connected by van der Waals forces.

**Introduction.** The intensities of 3350 independent non-zero reflections with  $I > 2\sigma(I)$  were collected on a Siemens AED single-crystal diffractometer using Cu  $K\alpha$  radiation. To calculate structure factors, only the Lorentz-polarization corrections were taken into account, omitting other corrections such as those for absorption. The structure was determined by direct methods using the *SHELX* 76 program (Sheldrick, 1976). The *E* map showed the positions of all non-hydrogen atoms. Eight of them were taken as Cl atoms assuming two independent molecules. A few cycles of refinement showed that this was correct. All H atoms were found in the difference Fourier map, but the positional parameters of 12 phenyl-ring H atoms were computed assuming suitable geometries and were not included in the refinement. Only their contributions were taken into account. The refinement was carried out using full-matrix least-squares techniques. The final conventional *R* factor, obtained for the observed reflections after anisotropic refinement for non-hydrogen atoms and isotropic refinement for four H atoms belonging to the urea groups, was 0.042.

Since the chemical formula was unknown at the start of the investigation, it was possible that O atoms were in the positions of the N atoms. To resolve this dilemma we recorded the infrared spectrum of the crystal (Fig. 1) from which it can be seen that the urea group is present. The strong band  $6.46 \mu\text{m}$  ( $1548 \text{ cm}^{-1}$ ) is due to the H atom in the *trans* position to the carbonyl. The band at  $2.97 \mu\text{m}$  ( $3367 \text{ cm}^{-1}$ ) is due to N-H stretching (Pouchert, 1970).

Tables 1 and 2 give positional parameters of the non-hydrogen atoms and the H atoms of the urea groups

respectively. Table 3 gives the bond distances and angles. Fig. 2 is a view of the two independent molecules.\*

**Discussion.** The atoms of each molecule lie in three planes. These are indicated in Table 4, together with the largest atomic distances from the planes. The angle between the urea planes of the two independent molecules *A* and *B* is  $40.87^\circ$ .

The molecules are linked together by N-H...O hydrogen bonds of 2.87 and 2.92 Å (Fig. 3), forming columns along the *c* axis. Each column is connected to the others by van der Waals forces. We found very short van der Waals contacts between two Cl atoms of 3.31, 3.46 and 3.50 Å. The arrangement of the molecules viewed along the *c* axis is shown in Fig. 4.

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33777 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

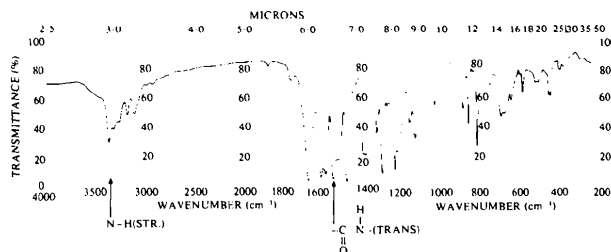


Fig. 1. Infrared spectrum of *N,N'*-bis(3,4-dichlorophenyl)urea.

Table 1. Final positional parameters for non-hydrogen atoms ( $\times 10^4$ ) in molecules *A* and *B* with standard deviations in parentheses

	Molecule <i>A</i>			Molecule <i>B</i>		
	<i>x</i>	<i>y</i>	<i>z</i>	<i>x</i>	<i>y</i>	<i>z</i>
Cl(1)	5092 (1)	5602 (1)	6488 (2)	4715 (1)	1887 (0)	1237 (1)
Cl(2)	4668 (1)	6271 (0)	9260 (2)	4005 (1)	1149 (0)	3750 (1)
Cl(3)	-193 (1)	2506 (0)	2901 (1)	184 (1)	5079 (0)	8275 (1)
Cl(4)	419 (1)	1397 (0)	1651 (1)	1110 (1)	6152 (0)	7112 (1)
O(1)	2273 (2)	3886 (1)	764 (3)	2356 (2)	3615 (1)	5790 (2)
N(1)	2756 (3)	4139 (1)	8526 (3)	2558 (3)	3369 (1)	3467 (3)
N(2)	2024 (3)	3330 (1)	8861 (4)	1940 (3)	4187 (1)	3957 (3)
C(1)	4301 (3)	5348 (2)	7846 (4)	3895 (3)	2124 (1)	2594 (4)
C(2)	4130 (3)	5640 (2)	9058 (5)	3575 (3)	1798 (1)	3695 (4)
C(3)	3501 (4)	5428 (2)	124 (5)	2922 (3)	2001 (1)	4748 (4)
C(4)	3035 (4)	4932 (2)	9996 (4)	2583 (3)	2522 (1)	4727 (4)
C(5)	3218 (3)	4636 (1)	8780 (4)	2917 (3)	2846 (1)	3638 (4)
C(6)	3865 (3)	4848 (2)	7709 (4)	3580 (3)	2642 (1)	2564 (4)
C(7)	583 (3)	2456 (2)	1379 (4)	936 (3)	5096 (1)	6732 (4)
C(8)	837 (3)	1967 (2)	822 (4)	1341 (3)	5572 (1)	6224 (4)
C(9)	1436 (4)	1940 (2)	9576 (5)	1930 (4)	5579 (1)	4976 (4)
C(10)	1825 (4)	2392 (2)	8961 (4)	2138 (4)	5120 (1)	4264 (4)
C(11)	1598 (3)	2884 (1)	9544 (4)	1743 (3)	4648 (1)	4777 (4)
C(12)	951 (3)	2912 (1)	758 (4)	1117 (3)	4633 (1)	6001 (4)
C(25)	2344 (3)	2793 (1)	9488 (4)	2288 (3)	3715 (1)	4518 (4)

Table 2. Final positional parameters for hydrogen atoms ( $\times 10^3$ ) belonging to the urea groups with standard deviations in parentheses

	Molecule A			Molecule B		
	x	y	z	x	y	z
H(1)	279 (5)	402 (2)	777 (6)	263 (14)	335 (7)	277 (17)
H(2)	207 (5)	329 (6)	812 (6)	183 (16)	410 (8)	323 (20)

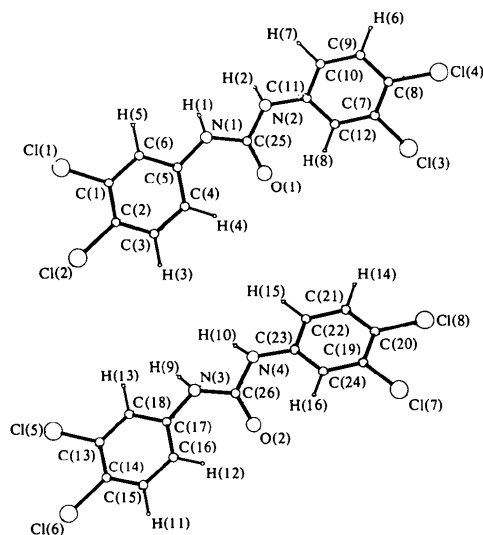


Fig. 2. A view of the two independent molecules A and B.

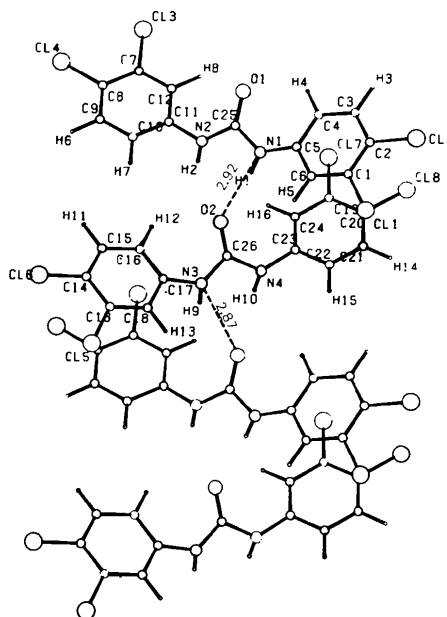


Fig. 3. Hydrogen bonds in the crystal.

Comparison of bond lengths and angles of this compound with those of the urea molecule (De Villiers & Boeyens, 1975) shows that the C–O length and N–C–N angle are smaller, whereas C–N and

Table 3. Bond distances (Å) and angles ( $^\circ$ )

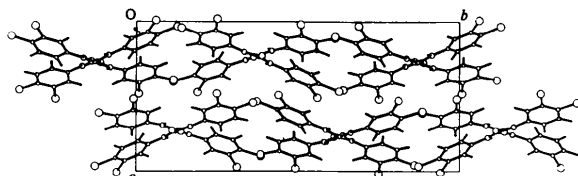
	A	B		A	B
Cl(1)–C(1)	1.730 (4)	1.733 (4)	Cl(1)–C(1)–C(2)	120.2 (3)	120.7 (3)
Cl(2)–C(2)	1.737 (4)	1.730 (4)	Cl(1)–C(1)–C(6)	119.0 (3)	118.5 (3)
Cl(3)–C(7)	1.727 (4)	1.724 (4)	Cl(2)–C(2)–C(1)	121.8 (3)	120.6 (3)
Cl(4)–C(8)	1.730 (4)	1.725 (4)	Cl(2)–C(2)–C(3)	119.1 (3)	120.6 (3)
C(1)–C(2)	1.381 (6)	1.390 (5)	Cl(3)–C(7)–C(8)	120.4 (3)	120.0 (3)
C(2)–C(3)	1.376 (6)	1.373 (6)	Cl(3)–C(7)–C(12)	118.1 (3)	119.2 (3)
C(3)–C(4)	1.382 (6)	1.385 (5)	Cl(4)–C(8)–C(7)	121.1 (3)	121.3 (3)
C(4)–C(5)	1.391 (5)	1.383 (5)	Cl(4)–C(8)–C(9)	119.8 (3)	119.4 (3)
C(5)–C(6)	1.391 (5)	1.395 (5)	C(6)–C(1)–C(2)	120.8 (4)	120.8 (3)
C(6)–C(1)	1.376 (6)	1.371 (5)	C(1)–C(2)–C(3)	119.1 (4)	118.8 (3)
C(7)–C(8)	1.387 (5)	1.393 (5)	C(2)–C(3)–C(4)	121.1 (4)	121.2 (4)
C(8)–C(9)	1.388 (6)	1.382 (6)	C(3)–C(4)–C(5)	119.6 (4)	119.6 (3)
C(9)–C(10)	1.376 (6)	1.376 (5)	C(4)–C(5)–C(6)	119.3 (4)	119.5 (3)
C(10)–C(11)	1.397 (5)	1.381 (5)	C(5)–C(6)–C(1)	120.1 (4)	120.0 (3)
C(11)–C(12)	1.394 (5)	1.387 (5)	C(12)–C(7)–C(8)	121.6 (4)	120.8 (3)
C(12)–C(7)	1.378 (5)	1.386 (5)	C(7)–C(8)–C(9)	119.1 (4)	119.3 (3)
O(1)–C(25)	1.227 (4)	1.222 (4)	C(8)–C(9)–C(10)	119.8 (4)	120.2 (4)
N(1)–C(5)	1.397 (5)	1.407 (4)	C(9)–C(10)–C(11)	121.2 (4)	120.3 (4)
N(1)–C(25)	1.364 (5)	1.369 (4)	C(10)–C(11)–C(12)	119.0 (3)	120.5 (3)
N(2)–C(25)	1.364 (5)	1.369 (5)	C(11)–C(12)–C(7)	119.3 (3)	118.8 (3)
N(2)–C(11)	1.407 (5)	1.429 (5)	N(1)–C(5)–C(4)	124.1 (3)	124.0 (3)
N(1)–H(1)	0.78 (6)	0.67 (16)	N(1)–C(5)–C(6)	116.6 (3)	116.2 (3)
N(2)–H(2)	0.71 (6)	0.72 (19)	C(5)–N(1)–C(25)	128.3 (3)	127.3 (3)
			N(2)–C(11)–C(10)	118.2 (3)	117.7 (3)
			N(2)–C(11)–C(12)	122.9 (3)	121.6 (3)
			C(11)–N(2)–C(25)	126.8 (3)	124.6 (3)
			N(1)–C(25)–N(2)	111.8 (3)	111.3 (3)
			N(1)–C(25)–O(1)	124.1 (3)	124.1 (3)
			N(2)–C(25)–O(1)	124.1 (3)	124.6 (3)

Table 4. Deviations (Å) of atoms from least-squares planes

Least-squares plane through the indicated atoms	Greatest deviations	Angle made with the urea plane (°)
Molecule A		
Urea N(1), N(2), O(1), C(25)	C(25) -0.001	
Phenyl I C(1)-C(6)	C(2) -0.013	16.32
Phenyl II C(7)-C(12)	C(7) 0.033	29.53
Molecule B		
Urea N(3), N(4), O(2), C(26)	C(26) -0.002	
Phenyl I C(13)-C(18)	C(15) -0.013	29.73
Phenyl II C(19)-C(24)	C(20) 0.021	39.67

N—C—O are larger, presumably as a result of the influence of the 3,4-dichlorophenyl substituents.

The authors are greatly indebted to Professors L.

Fig. 4. The arrangement of the molecules viewed along *c*.

Cavalca, M. Nardelli and B. Ribàr for their interest and to Dr A. Nikolić for the infrared spectra.

#### References

- DE VILLIERS, J. P. R. & BOEYENS, J. C. A. (1975). *J. Cryst. Mol. Struct.* **5**, 215–226.  
 POUCHERT, C. J. (1970). *The Altrich Library of Infrared Spectra*. Altrich Chem. Co.  
 SHELDRIK, G. M. (1976). *SHELX 76*. Program for crystal structure determination. Univ. of Cambridge, England.

*Acta Cryst.* (1978). **B34**, 3790–3792

## Perchloro-*p*-xylene

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(Received 2 May 1978; accepted 5 September 1978)

**Abstract.**  $C_8Cl_{10}$ ,  $M_r = 450.62$ ,  $F(000) = 1744$ , orthorhombic,  $Pbca$ ,  $a = 16.436$  (9),  $b = 18.600$  (5),  $c = 9.792$  (7) Å,  $V = 2993$  (3) Å<sup>3</sup>,  $Z = 8$ ,  $D_c = 1.999$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.7107$  Å,  $\mu = 18.2$  cm<sup>-1</sup>. The structure was solved by direct methods and refined to  $R = 0.056$  for 1749 observed reflections. Both the benzene ring and the trichloromethyl groups are distorted due to steric repulsions among the Cl atoms.

**Introduction.**  $C_8Cl_{10}$  was synthesized by Ballester, Molinet & Castañer (1960), and was tentatively assigned a molecular conformation based on the UV-visible absorption spectrum (Ballester & Castañer, 1960).

These authors provided us with samples of perchloro-*p*-xylene in the form of pale greenish-yellow prismatic crystals obtained by slow evaporation of an ether solution.

A crystal of dimensions 0.2 × 0.2 × 0.4 mm was used for the data collection on a Nonius CAD-4 diffractometer. The cell dimensions were obtained by a least-squares refinement of the setting angles of 22 reflections. Mo  $K\alpha$  radiation was used, mono-

chromatized by reflection from a graphite crystal, at a take-off angle of 5°. The  $\omega$ - $2\theta$  technique was used with scan interval  $\Delta\omega = 0.70 + 0.35 \tan \theta$ . The background was measured before and after each reflection. The maximum scan time was 80 s, with 53.3 s used for the peak scanning and 13.3 s for each background. The intensity of 523 was chosen as a standard and it was measured every hour. Three reflections were selected for orientation control and measured every 100 reflections. 2723 independent reflections were collected within the range  $1 < \theta \leq 30^\circ$ , 1749 of which were considered as observed, by the criterion  $I > 2\sigma(I)$ . The data were collected with the crystal mounted along the *c* axis. No absorption corrections were made ( $\mu r < 0.5$ ).

The structure was solved by the *MULTAN* system of computer programs (Main, Woolfson, Lessinger, Germain & Declercq, 1974), and refined by means of a block-diagonal least-squares procedure with a modified version of *SFLS* (Ahmed, Hall, Pippy & Huber, 1966), written by X. Solans. The function minimized was  $w\{|F_o| - |F_c|\|^2$ , where  $w = (8.0 + |F_o| + 0.01|F_o|^2)^{-1/2}$ . The refinement was terminated at  $R = 0.056$  for all