Tableau 6. *Plan moyen* III *et distances à ce plan* (Å)

$0{,}4121X+0{,}7170Y+0{,}5621Z=9{,}5441$

Atomes définissant le plan		Autres atomes	
C(6)	0,0038 (115)	C(1)	0,423 (114)
C(26)	−0,0138 (124)	C(2)	0,0319 (122)
O(27)	0,0025 (85)	C(3)	−0,1168 (112)
O(28)	0,0020 (80)	C(4)	−0,1800 (108)
		C(5)	−0,1100 (117)
		C(7)	0,2692 (115)
		C(8)	−1,0464 (125)
		C(9)	−0,7093 (123)
		C(10)	−1,9235 (124)
		C(11)	−1,5352 (131)
		C(12)	−2,7427 (134)
		C(13)	−2,3055 (131)
		C(14)	−3,4680 (136)
		C(15)	−2,9895 (145)
		C(16)	−4,1315 (138)
		C(17)	−3,6326 (146)
		C(18)	−4,7451(132)
		C(19)	−4,2310(135)
		C(20)	−5,3611 (146)
		C(21)	−4,9070 (157)
		O(23)	−0,1600 (83)
		O(25)	−0,1492 (79)

Tableau 7. *Liaisons hydrogène*

O(23,I)–O(25,IV)	2,85 (5) Å
O(27,I)–O(28,VI)	2,69 (0)
O(27,I)–H(280,VI)	1,97 (7)

Code de symétrie

(I)	x	y	z
(II)	$1-x$	$\frac{1}{2}+y$	$\frac{1}{2}-z$
(III)	$1-x$	$\bar{y}$	$1-z$
(IV)	$2-x$	$\frac{1}{2}+y$	$\frac{1}{2}-z$
(V)	$1+x$	$y-1$	z
(VI)	$2-x$	$\bar{y}-1$	$1-z$

Tableau 8. *Liaisons de type van der Waals*

C(19,I)–O(23,II)	3,48 (6) Å
O(23,I)–C(18,II)	3,45 (5)
O(23,I)–C(20,II)	3,36 (7)
C(21,I)–O(28,III)	3,69 (5)
C(4,I)—O(25,IV)	3,47 (7)
C(3,I)—O(25,IV)	3,57 (3)
C(4,I)—C(4,IV)	3,76 (4)
O(23,I)–C(4,IV)	3,45 (2)
O(23,I)–C(5,IV)	3,55 (3)
O(25,I)–C(21,V)	3,40 (5)
O(27,I)–C(21,V)	3,40 (6)

Conclusion

La structure très originale de cette molécule avec sa chaîne en C(15) entraîne une conformation cristalline très particulière. L'angle dièdre important des plans moyens de la chaîne et du cycle ainsi que le remplissage de la maille sont deux caractéristiques très intéressantes.

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The Crystal Structure of 1-(*N*,*N*-Bis-*p*-chlorobenzoylamino)-4,5-diphenyl-1,2,3-triazole

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(*Received* 10 *March* 1975; *accepted* 23 *April* 1975)

The crystal structure of 1-(*N*,*N*-bis-*p*-chlorobenzoylamino)-4,5-diphenyl-1,2,3-triazole, $C_{28}H_{18}N_4O_2Cl_2$, has been determined with three-dimensional intensities, measured on an automated Philips PW 1100 single-crystal diffractometer (574 independent non-zero reflexions). The structure was essentially solved by direct phase determination combined with Fourier syntheses. The cell constants, obtained by least-squares calculations from direct θ value measurements on the diffractometer, are $a=15{\cdot}0519$ (23), $b=12{\cdot}3312$ (15), $c=13{\cdot}5896$ (32) Å, $\beta=90{\cdot}13$ (2)°, $Z=4$; the space group is $P2_1/n$. The positional and vibrational parameters, with anisotropic temperature factors for the non-hydrogen atoms, were refined by full-matrix least-squares calculations to a final $R=0{\cdot}070$. Correction for anomalous scattering of the Cl atoms was applied. The molecule has a dibenzoylaminotriazole structure. One of the four benzene rings forms with the central triazole ring a roughly coplanar system.

Introduction

In a recent paper on the structure of 1-(α-*o*-bromobenzoyloxy-*o*′-bromobenzylideneamino)-4,5-diphenyl 1,2,3-triazole (Kokkou & Rentzeperis, 1975) reference was made to the triazole derivatives obtained as oxidation products of α-diacylhydrazones and detailed literature on their preparation and various properties

was given. It was also mentioned that several ambiguities in the chemical evidence necessitated exact X-ray crystal structure analysis.

The crystal structure determination of the new compound 1-(*N*,*N*-bis-*p*-chlorobenzoylamino)-4,5-diphenyl-1,2,3-triazole (CBDT; $C_{28}H_{18}N_4O_2Cl_2$), described in the following, was undertaken as part of a systematic programme to elucidate the structures of the triazole derivatives prepared at the Laboratory of Organic Chemistry of the Aristotle University of Thessaloniki.

Experimental

Pure, colourless CBDT crystals were kindly provided by Professor N. E. Alexandrou and Dr E. D. Micromastoras. The crystals are monoclinic, pseudo-orthorhombic. A transparent single crystal, with dimensions $0{\cdot}15\times0{\cdot}15\times0{\cdot}08$ mm, was selected and centred on a computer-controlled Philips PW1100 four-circle single-crystal diffractometer. With Mo $K\alpha$ radiation ($\lambda=0{\cdot}71609$ Å) the cell constants were determined first by using the PH (Peak Hunting) routine of the instrument. To achieve greater accuracy, the θ angles of 124 strong reflexions with large θ values were directly measured on the diffractometer and subsequently processed with the least-squares program *PARAM* [part of the X-RAY System of Crystallographic Programs (Stewart, Kruger, Ammon, Dickinson & Hall, 1972)]. The final values obtained are given in Table 1. Systematic absences led to the space group $P2_1/n$. The density of the crystals was measured by flotation in a KBr solution.

Table 1. *Crystal data for CBDT*

(Standard errors, given in parentheses, refer to last digit)

$C_{28}H_{18}N_4O_2Cl_2$	F.W. 513·4
Monoclinic	$Z=4$
Space group, $P2_1/n$	$F(000)=1056$
$a=15{\cdot}0519$ (29) Å	$\varrho_{calc}=1{\cdot}352$ g cm^{-3}
$b=12{\cdot}3312$ (19)	$\varrho_{meas}=1{\cdot}354$ g cm^{-3}
$c=13{\cdot}5896$ (32)	m.p. 216°–217°C
$\beta=90{\cdot}13$ (2)°	$\mu=2{\cdot}84$ cm^{-1}
$V=2522{\cdot}33$ Å^3	λ(Mo $K\alpha$)$=0{\cdot}71609$ Å

Three-dimensional intensity data were collected with a scintillation counter on the PW1100 diffractometer in the ω-scan mode, using Mo $K\alpha$ radiation, mono-

Table 2. *Atomic coordinates, anisotropic temperature coefficients* ($\times10^4$) *and equivalent isotropic temperature factors for the non-hydrogen atoms in CBDT, with standard deviations in parentheses*

Anisotropic temperature factor $T=\exp[-(h^2\beta_{11}+k^2\beta_{22}+l^2\beta_{33}+2hk\beta_{12}+2hl\beta_{13}+2kl\beta_{23})]$.

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}	B
Cl(1)	0·7018 (7)	0·1282 (7)	0·4629 (8)	110 (7)	124 (8)	200 (12)	−33 (7)	−40 (8)	−47 (9)	10·79 Å^2
Cl(2)	0·9227 (7)	0·0340 (7)	0·6074 (7)	150 (9)	78 (7)	125 (9)	27 (7)	−11 (7)	7 (7)	9·23
O(1)	0·4294 (14)	0·0606 (14)	0·7119 (14)	107 (18)	84 (18)	75 (20)	−32 (14)	−33 (14)	6 (14)	6·80
O(2)	0·3279 (11)	0·5527 (14)	0·7301 (12)	73 (14)	79 (15)	49 (15)	−26 (12)	16 (12)	−12 (13)	5·05
N(1)	0·0134 (14)	0·3968 (16)	0·6860 (16)	65 (16)	56 (18)	45 (17)	6 (14)	18 (14)	39 (16)	4·22
N(2)	0·0784 (12)	0·3814 (16)	0·6228 (15)	14 (20)	72 (35)	44 (29)	7 (21)	17 (20)	−30 (25)	2·99
N(3)	0·1176 (13)	0·2890 (15)	0·6510 (16)	33 (25)	41 (29)	97 (38)	37 (23)	−17 (26)	−25 (24)	4·25
N(4)	0·9624 (14)	0·4947 (15)	0·6898 (16)	31 (25)	41 (31)	58 (30)	−34 (26)	13 (24)	−3 (23)	3·23
C(1)	0·0817 (17)	0·2513 (19)	0·7327 (20)	41 (18)	48 (24)	88 (28)	−23 (18)	43 (18)	14 (19)	4·34
C(2)	0·0122 (16)	0·3206 (18)	0·7548 (17)	59 (33)	13 (28)	3 (30)	−9 (25)	10 (27)	0 (26)	2·16
C(3)	0·4876 (22)	0·0863 (21)	0·7698 (23)	74 (52)	48 (43)	62 (67)	−2 (36)	−16 (42)	−48 (36)	4·77
C(4)	0·8675 (20)	0·4852 (20)	0·6830 (19)	82 (41)	49 (38)	21 (35)	−43 (33)	33 (34)	−29 (27)	4·04
C(5)	0·8282 (15)	0·3956 (17)	0·6249 (20)	13 (27)	37 (33)	78 (42)	−12 (21)	17 (27)	12 (30)	3·10
C(6)	0·8672 (17)	0·3608 (24)	0·5391 (22)	27 (32)	117 (51)	69 (55)	−7 (33)	43 (39)	−11 (42)	4·93
C(7)	0·8249 (22)	0·2750 (24)	0·4894 (22)	82 (26)	153 (39)	81 (32)	−21 (26)	−4 (23)	−60 (28)	7·63
C(8)	0·7506 (23)	0·2306 (19)	0·5278 (15)	75 (43)	30 (33)	96 (57)	−16 (31)	−28 (39)	−35 (35)	5·28
C(9)	0·7181 (24)	0·2676 (24)	0·6135 (24)	130 (52)	96 (52)	47 (48)	12 (45)	17 (42)	−61 (37)	7·06
C(10)	0·7521 (18)	0·3490 (23)	0·6704 (18)	62 (21)	113 (30)	37 (24)	−14 (19)	21 (19)	0 (21)	5·10
C(11)	0·5182 (15)	0·1947 (17)	0·8014 (20)	8 (30)	7 (28)	34 (37)	3 (23)	−11 (31)	0 (32)	1·24
C(12)	0·4912 (18)	0·2810 (24)	0·7428 (19)	65 (22)	75 (26)	26 (23)	37 (19)	−2 (18)	21 (19)	4·16
C(13)	0·5115 (20)	0·3839 (21)	0·7741 (27)	49 (45)	29 (59)	109 (60)	21 (35)	−17 (44)	37 (44)	4·81
C(14)	0·5560 (18)	0·4002 (27)	0·8599 (26)	29 (22)	148 (39)	78 (32)	−26 (21)	−23 (21)	−26 (25)	5·81
C(15)	0·5783 (20)	0·3167 (20)	0·9200 (24)	95 (42)	4 (32)	111 (57)	−5 (36)	17 (36)	−16 (41)	5·71
C(16)	0·5611 (16)	0·2136 (19)	0·8903 (21)	31 (37)	66 (54)	45 (45)	−15 (29)	−27 (36)	−18 (35)	3·42
C(17)	0·1187 (20)	0·1542 (19)	0·7814 (21)	88 (24)	16 (22)	66 (26)	−2 (17)	−21 (22)	−5 (18)	4·63
C(18)	0·1735 (21)	0·0871 (24)	0·7243 (21)	117 (28)	83 (28)	65 (28)	1 (24)	44 (23)	8 (24)	6·87
C(19)	0·2161 (27)	0·0057 (40)	0·7712 (37)	93 (31)	237 (68)	145 (49)	7 (35)	53 (32)	62 (41)	11·19
C(20)	0·1919 (35)	0·9767 (28)	0·8649 (36)	105 (31)	49 (28)	170 (50)	53 (25)	6 (30)	7 (33)	8·37
C(21)	0·1325 (27)	0·0323 (23)	0·9202 (27)	137 (48)	13 (32)	76 (43)	−31 (33)	−33 (37)	12 (33)	6·30
C(22)	0·0980 (23)	0·1265 (23)	0·8758 (20)	118 (22)	72 (24)	51 (23)	−16 (21)	38 (19)	46 (19)	6·30
C(23)	0·9466 (18)	0·3034 (18)	0·8358 (18)	71 (36)	11 (29)	27 (37)	19 (31)	−14 (27)	4 (26)	3·10
C(24)	0·8819 (19)	0·2232 (19)	0·8333 (20)	56 (31)	8 (30)	50 (38)	−8 (24)	5 (29)	−18 (25)	3·13
C(25)	0·8225 (19)	0·2199 (31)	0·9148 (27)	17 (19)	138 (37)	101 (36)	−19 (23)	15 (22)	78 (27)	5·82
C(26)	0·8185 (35)	0·2976 (44)	0·9850 (31)	113 (33)	177 (47)	48 (28)	102 (32)	59 (37)	16 (29)	8·21
C(27)	0·8861 (27)	0·3692 (33)	0·9820 (34)	70 (30)	125 (37)	134 (47)	−23 (29)	−22 (31)	−69 (40)	7·99
C(28)	0·9457 (23)	0·3808 (29)	0·9050 (26)	59 (24)	92 (33)	88 (33)	12 (25)	47 (22)	4 (27)	5·86

chromatized with a graphite monochromator. The intensities of 2025 independent reflexions up to $2\theta = 46{\cdot}5°$ (max. $h,k,l = 14,11,\pm 10$ respectively) were examined and 574 of them, with top intensities, I_{top}, such that $I_{top} - 2\sqrt{I_{top}} > I_{bck}$, where I_{bck} is the intensity of the background, were considered as observed and included in all subsequent computations. The remaining 1451 reflexions were treated as weak, by applying to them the Hamilton (1955) correction.

Integrated intensities were converted to $|F_o|$ values in the usual way, with a modified version of the special measurement treatment program by Hornstra & Stubbe (1972). No absorption correction was applied. For all subsequent computations, the programs of the X-RAY System were used.

Determination of the structure and refinement

The structure was obtained by combining direct phase determination with Fourier syntheses. With the program *PHASE* of the X-RAY System the phases of 108 strong reflexions were determined. On the resulting *E* map it was possible to locate the positions of the two Cl atoms and one of the two benzoylamino groups of the asymmetric unit. The rest of the non-hydrogen atoms were located by successive Fourier syntheses. Structure-factor calculation at this stage gave $R = 0{\cdot}306$.

Refinement of the structure was carried out by full-matrix least-squares calculations, using the program *CRYLSQ* of the X-RAY System. The atomic scattering factors for Cl, N, O and C were obtained from Cromer & Waber (1965), and for H from Stewart, Davidson & Simpson (1965). Anomalous dispersion corrections for Cl were taken from *International Tables for X-ray Crystallography* (1968). A single scale factor was used for the whole set of reflexion data.

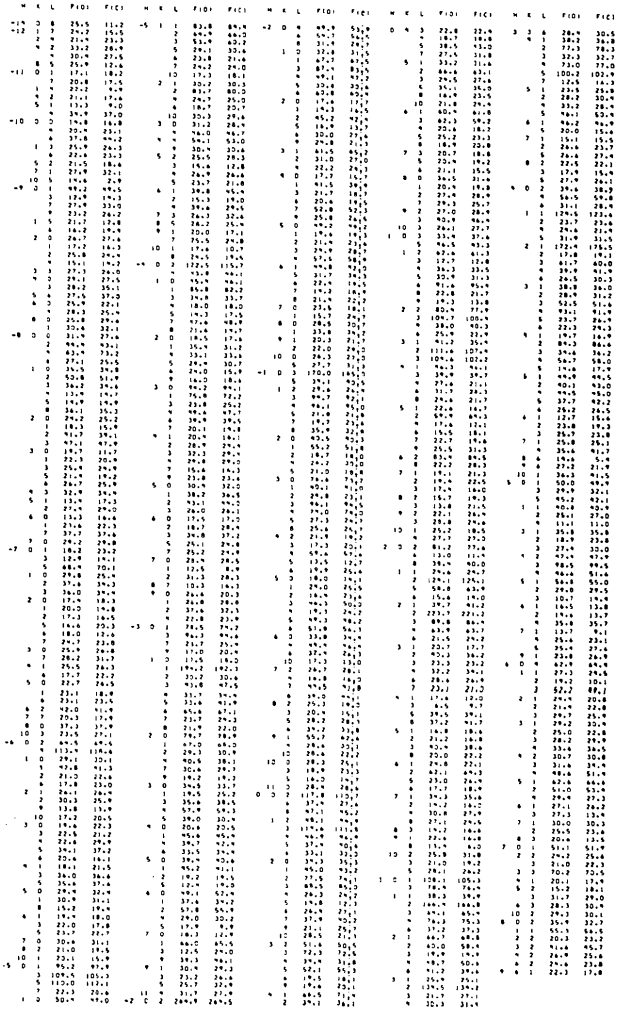
With isotropic temperature factors and unit weights, refinement reduced R to 0·112 in six cycles. Eight further cycles with anisotropic temperature coefficients reduced R to 0·071. The H atoms were then located on a difference Fourier synthesis and included in further refinement cycles with isotropic temperature factors assigned to them, equal to those of the corresponding C atoms to which they are bonded. At this stage the weighting function $w = 1/(A + B|F_o| + C|F_o|^2)$ (Cruickshank, Pilling, Bujosa, Lovell & Truter, 1961) was introduced and a systematic weight analysis was carried out with the programs *WTANAL* and *WTLSSQ* of the X-RAY System. This led to essentially unit weights and consequently the original weighting scheme was kept. Convergence was reached at $R = 0{\cdot}070$.

The final positional parameters and anisotropic temperature coefficients for the non-hydrogen atoms are given in Table 2. The final coordinates and isotropic temperature factors for the hydrogen atoms are shown in Table 3. Comparison between $|F_o|$ and $|F_c|$ values, obtained with the parameters in Tables 2 and

Table 3. *Atomic coordinates and isotropic temperature factors for the hydrogen atoms in CBDT*

	x	*y*	*z*	*B*
H(C6)	0·929 (12)	0·395 (14)	0·513 (13)	5·2 Å²
H(C7)	0·855 (13)	0·245 (15)	0·424 (15)	7·0
H(C9)	0·661 (12)	0·219 (15)	0·640 (14)	7·9
H(C10)	0·717 (9)	0·372 (13)	0·732 (13)	4·3
H(C12)	0·455 (10)	0·266 (11)	0·676 (11)	1·7
H(C13)	0·494 (12)	0·454 (15)	0·731 (14)	5·9
H(C15)	0·607 (12)	0·336 (14)	0·939 (14)	5·8
H(C16)	0·584 (12)	0·148 (14)	0·929 (14)	8·2
H(C18)	0·188 (12)	0·098 (15)	0·647 (15)	6·4
H(C19)	0·256 (18)	0·042 (24)	0·737 (21)	8·4
H(C20)	0·217 (17)	0·092 (22)	0·893 (18)	6·3
H(C21)	0·118 (15)	0·017 (18)	0·002 (19)	9·5
H(C22)	0·042 (13)	0·167 (15)	0·905 (14)	6·0
H(C24)	0·892 (12)	0·165 (15)	0·778 (13)	3·6
H(C25)	0·775 (16)	0·156 (19)	0·901 (18)	5·6
H(C26)	0·764 (14)	0·283 (17)	0·037 (17)	8·8
H(C27)	0·885 (16)	0·418 (20)	0·045 (19)	4·9
H(C28)	0·981 (15)	0·449 (18)	0·912 (16)	2·9

Table 4. *Observed and calculated structure factors for CBDT*



3 is made in Table 4, in which for brevity the unobserved reflexions are omitted. Interatomic distances and bond angles are given in Tables 5 and 6.

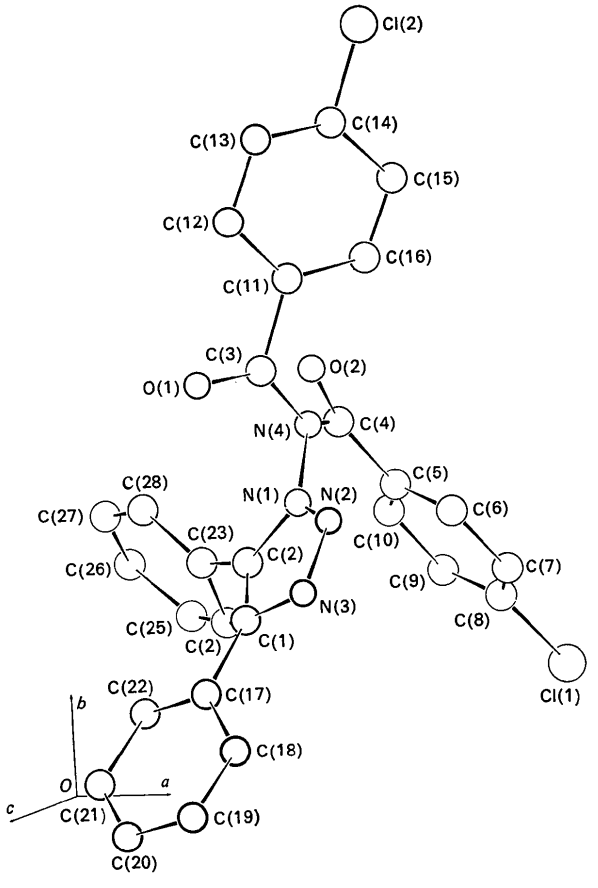


Fig. 1. Clinographic projection of the CBDT molecule, showing its conformation.

Table 5. *Interatomic distances* (Å) *in CBDT, with their standard deviations in parentheses*

N(1)—N(2)	1·32 (3)	C(19)–C(20)	1·37 (7)
N(2)—N(3)	1·34 (3)	C(20)–C(21)	1·36 (6)
N(3)—C(1)	1·32 (3)	C(21)–C(22)	1·40 (4)
C(1)—C(2)	1·38 (3)	C(22)–C(17)	1·36 (4)
C(2)—N(1)	1·33 (3)	C(2)—C(23)	1·49 (4)
N(1)—N(4)	1·43 (3)	C(23)–C(24)	1·39 (4)
N(4)—C(3)	1·46 (4)	C(24)–C(25)	1·43 (4)
C(3)—O(1)	1·22 (4)	C(25)–C(26)	1·35 (6)
N(4)—C(4)	1·44 (4)	C(26)–C(27)	1·35 (7)
C(4)—O(2)	1·21 (3)	C(27)–C(28)	1·38 (6)
C(4)—C(5)	1·48 (4)	C(28)–C(23)	1·35 (4)
C(5)—C(6)	1·38 (4)	C(6)—H(C6)	1·1 (2)
C(6)—C(7)	1·41 (4)	C(7)—H(C7)	1·1 (2)
C(7)—C(8)	1·35 (5)	C(9)—H(C9)	1·1 (2)
C(8)—C(9)	1·34 (5)	C(10)–H(C10)	1·0 (2)
C(9)—C(10)	1·37 (4)	C(12)–H(C12)	1·1 (2)
C(10)–C(5)	1·42 (4)	C(13)–H(C13)	1·1 (2)
C(8)—Cl(1)	1·71 (3)	C(15)–H(C15)	1·1 (2)
C(3)—C(11)	1·48 (4)	C(16)–H(C16)	1·0 (2)
C(11)–C(12)	1·39 (4)	C(18)–H(C18)	1·1 (2)
C(12)–C(13)	1·37 (4)	C(19)–H(C19)	1·0 (3)
C(13)–C(14)	1·36 (5)	C(20)–H(C20)	1·0 (3)
C(14)–C(15)	1·36 (4)	C(21)–H(C21)	1·2 (3)
C(15)–C(16)	1·36 (4)	C(22)–H(C22)	1·1 (2)
C(16)–C(11)	1·39 (4)	C(24)–H(C24)	1·0 (2)
C(14)–Cl(2)	1·74 (3)	C(25)–H(C25)	1·1 (2)
C(1)—C(17)	1·48 (4)	C(26)–H(C26)	1·1 (2)
C(17)–C(18)	1·40 (4)	C(27)–H(C27)	1·1 (2)
C(18)–C(19)	1·35 (6)	C(28)–H(C28)	1·0 (2)
Distances of special interest			
O(1)–O(2)	3·74 (3)	Cl(1)–Cl(2)	11·817 (13)

Description of the structure and discussion

The structural and conformational features of the CBDT molecule are shown in the clinographic projection of Fig. 1. The compound has clearly a dibenzoylaminotriazole structure and not a triazolylisoimide structure, as in the case of 1-(α-*o*-bromobenzoyloxy-*o'*-bromobenzylideneamino)-4,5-diphenyl-1,2,3-triazole

Table 6. *Bond angles in CBDT, with their standard deviations in parentheses*

N(1)–N(2)–N(3)	105·3 (1·8)°	C(5)—C(6)—C(7)	116·6 (2·5)°	C(17)–C(18)–C(19)	117·2 (3·1)°
N(2)–N(3)–C(1)	111·1 (1·9)	C(6)—C(7)—C(8)	119·5 (2·8)	C(18)–C(19)–C(20)	120·3 (3·8)
N(3)–C(1)–C(2)	106·0 (2·1)	C(7)—C(8)—C(9)	120·0 (2·9)	C(19)–C(20)–C(21)	123·8 (3·7)
C(1)–C(2)–N(1)	105·9 (2·1)	C(8)—C(9)—C(10)	127·1 (3·1)	C(20)–C(21)–C(22)	115·2 (3·3)
C(2)–N(1)–N(2)	111·6 (2·0)	C(9)—C(10)–C(5)	110·5 (2·5)	C(21)–C(22)–C(17)	121·7 (2·9)
N(2)–N(1)–N(4)	123·0 (1·9)	C(10)–C(5)—C(6)	126·0 (2·3)	C(22)–C(17)–C(18)	120·5 (2·5)
C(2)–N(1)–N(4)	124·3 (2·0)	C(10)–C(5)—C(4)	113·0 (2·3)	C(1)—C(17)–C(18)	116·8 (2·5)
N(1)–C(2)–C(23)	129·0 (2·2)	C(6)—C(5)—C(4)	120·9 (2·3)	C(1)—C(17)–C(22)	122·6 (2·5)
C(1)–C(2)–C(23)	124·9 (2·1)	C(7)—C(8)—Cl(1)	117·1 (2·6)	C(23)–C(24)–C(25)	116·2 (2·5)
C(2)–C(1)–C(17)	133·4 (2·4)	C(9)—C(8)—Cl(1)	122·8 (2·6)	C(24)–C(25)–C(26)	123·8 (3·5)
N(3)–C(1)–C(17)	120·5 (2·3)	C(11)–C(12)–C(13)	117·8 (2·5)	C(25)–C(26)–C(27)	114·1 (4·1)
N(1)–N(4)–C(3)	112·9 (2·0)	C(12)–C(13)–C(14)	120·8 (2·7)	C(26)–C(27)–C(28)	125·7 (4·1)
N(1)–N(4)–C(4)	117·6 (1·8)	C(13)–C(14)–C(15)	121·7 (2·9)	C(27)–C(28)–C(23)	117·6 (3·3)
N(4)–C(3)–O(1)	114·3 (2·3)	C(14)–C(15)–C(16)	119·0 (3·0)	C(28)–C(23)–C(24)	120·9 (2·6)
N(4)–C(4)–O(2)	113·7 (2·2)	C(15)–C(16)–C(11)	120·2 (2·4)	C(2)—C(23)–C(24)	123·1 (2·2)
N(4)–C(3)–C(11)	115·4 (2·5)	C(16)–C(11)–C(12)	120·3 (2·1)	C(2)—C(23)–C(28)	115·3 (2·4)
N(4)–C(4)–C(5)	119·4 (2·2)	C(12)–C(11)–C(3)	115·6 (2·4)		
C(3)–N(4)–C(4)	126·7 (2·1)	C(16)–C(11)–C(3)	123·3 (2·2)		
O(1)–C(3)–C(11)	130·2 (2·6)	C(13)–C(14)–Cl(2)	116·8 (2·4)		
O(2)–C(4)–C(5)	126·9 (2·6)	C(15)–C(14)–Cl(2)	121·4 (2·6)		

[BBDT; see Kokkou & Rentzeperis (1975)]. The characteristic helical chain of BBDT is missing in CBDT.

The triazole and the four benzene rings of the molecule are planar to a good approximation. In Table 7 are listed the various least-squares planes of the CBDT molecule, together with the dihedral angles between them. The benzene ring *B*3, attached to C(1), Fig. 1, forms with the triazole ring, *T*, a roughly coplanar system, the angle $T \wedge B3$ being 16.1°. In contrast to BBDT, the benzene ring *B*2 does not belong to this coplanar system ($T \wedge B2 = 72{\cdot}0°$ and $B2 \wedge B3 = 56{\cdot}6°$). The two benzene rings *B*1 and *B*4 are almost parallel ($B1 \wedge B4 = 5{\cdot}6°$) and form almost equal angles with the triazole ring (73·9° and 68·4° respectively). In the bifurcated main chain, the two carbonyl groups form two planes, *P*1 and *P*2 with adjacent atoms: the first is formed by the atoms N(4)–O(2)–C(4)–C(5) and the second by N(4)–O(1)–C(3)–C(11). Both are roughly perpendicular to the triazole ring ($T \wedge P1 = 82{\cdot}3°$ and $T \wedge P2 = 87{\cdot}2°$). Plane *P*1, inclined at an angle of 37·2°, traces the benzene ring *B*1 along the line formed by the atoms C(4)–C(5)–C(8)–Cl(1). Similarly, plane *P*2, inclined at an angle of 22·1°, traces the benzene ring *B*2 along the line C(3)–C(11)–C(14)–Cl(2). Each of the two conjugated systems, O=C–Ar may be considered as roughly planar.

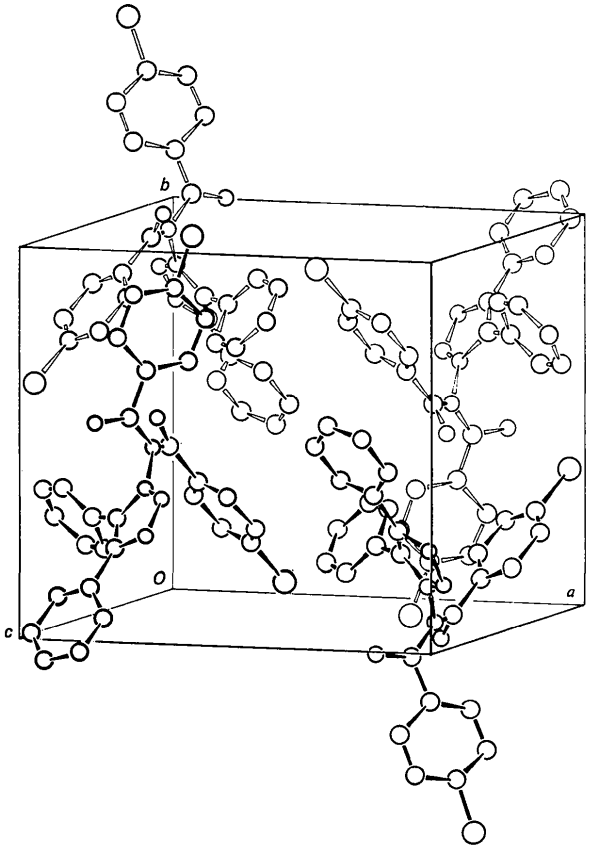


Fig. 2. Clinographic projection of the structure of CBDT, showing the molecular packing.

The two Cl–C_{ar} bond lengths, 1·71 and 1·74 Å respectively, are normal. The geometrical features of the triazole and the benzene rings are in good agreement with the usually accepted values. Mean bond lengths in

Table 7. *Least-squares planes in CBDT, with displacements of atoms from the planes* (Å)

The equation for a plane is in the form $AX + BY + CZ = D$ and refers to orthogonal axes. The coordinates *X*, *Y*, *Z* are expressed in Å; *D* is the distance of the plane from the origin. Asterisks indicate atoms not included in the calculation of the plane. The mean estimated standard deviations of the atoms defining a plane are given in parentheses attached to the distance of the first atom.

(*T*) Triazole ring

$0{\cdot}62310X + 0{\cdot}54380Y + 0{\cdot}56216Z = 17{\cdot}40060$

N(1)	−0·008 (16)	C(17)*	0·080
N(2)	0·018	C(23)*	−0·118
N(3)	−0·020	N(4)*	0·199
C(1)	0·015		
C(2)	−0·005		

(*B*1) Benzene ring 1 [attached to C(4)]

$0{\cdot}56575X - 0{\cdot}65507Y + 0{\cdot}50081Z = 8{\cdot}12001$

C(5)	−0·021 (14)	C(4)*	−0·016
C(6)	0·010	Cl(1)*	−0·036
C(7)	0·005		
C(8)	−0·008		
C(9)	−0·003		
C(10)	0·017		

(*B*2) Benzene ring 2 [attached to C(3)]

$0{\cdot}88051X + 0{\cdot}04619Y - 0{\cdot}47178Z = 8{\cdot}92562$

C(11)	−0·016 (17)	C(3)*	0·124
C(12)	0·014	Cl(2)*	0·018
C(13)	0·005		
C(14)	−0·023		
C(15)	0·012		
C(16)	−0·002		

(*B*3) Benzene ring 3 [attached to C(1)]

$0{\cdot}76755X + 0{\cdot}55449Y + 0{\cdot}32156Z = 17{\cdot}34522$

C(17)	0·030 (35)	C(1)*	0·025
C(18)	−0·044		
C(19)	0·016		
C(20)	0·025		
C(21)	−0·038		
C(22)	−0·011		

(*B*4) Benzene ring 4 [attached to C(2)]

$0{\cdot}61663X - 0{\cdot}57925Y + 0{\cdot}53314Z = 12{\cdot}63687$

C(23)	0·021 (40)	C(2)*	−0·077
C(24)	−0·025		
C(25)	0·037		
C(26)	−0·048		
C(27)	0·046		
C(28)	−0·033		

(*P*1) Plane 1 of the main chain

$0{\cdot}00759X - 0{\cdot}59343Y + 0{\cdot}80485Z = 4{\cdot}03086$

N(4)	0·003 (8)	C(8)*	0·140
O(2)	0·004	Cl(1)*	0·175
C(4)	−0·012		
C(5)	0·004		

(*P*2) Plane 2 of the main chain

$-0{\cdot}64551X + 0{\cdot}03979Y + 0{\cdot}76272Z = -1{\cdot}94893$

N(4)	0·005 (11)	C(14)*	−0·134
O(1)	0·007	Cl(2)*	−0·201
C(3)	−0·017	Cl(1)	−0·001
C(11)	0·005		

Table 7 (*cont.*)

Dihedral angles between planes (°)

$T \wedge B1$	73·9	$B1 \wedge B2$	76·6	$B2 \wedge B3$	56·6	$B3 \wedge B4$	71·1	$B4 \wedge P1$	39·0	$P1 \wedge P2$	54·2
$T \wedge B2$	72·0	$B1 \wedge B3$	76·6	$B2 \wedge B4$	74·7	$B3 \wedge P1$	86·3	$B4 \wedge P2$	89·2		
$T \wedge B3$	16·1	$B1 \wedge B4$	5·6	$B2 \wedge P1$	66·4	$B3 \wedge P2$	76·8				
$T \wedge B4$	68·4	$B1 \wedge P1$	37·2	$B2 \wedge P2$	22·1						
$T \wedge P1$	82·3	$B1 \wedge P2$	89·5								
$T \wedge P2$	87·2										

the four benzene rings $B1$, $B2$, $B3$ and $B4$ are 1·38, 1·37, 1·38 and 1·37 Å respectively. The bond lengths in the two carbonyl groups, 1·22 and 1·21 Å are quite satisfactory.

The intramolecular distance O(1)–O(2) = 3·74 Å is much longer than the sum of the van der Waals radii of the corresponding atoms. Other distances are within expected values.

Fig. 2 is a clinographic projection of the structure showing the molecular packing of CBDT in the unit cell. The various intermolecular distances are normal. It is of interest to note that the shortest Cl–Cl intermolecular distance is 3·83 Å, slightly over two van der Waals radii.

Thanks are due to Professor N. E. Alexandrou and Dr E. D. Micromastoras, Laboratory of Organic Chemistry, Aristotle University of Thessaloniki, Greece, for providing the CBDT crystals. We also thank the University of Thessaloniki for the use of its UNIVAC 1106 computer.

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The Crystal Structure of *O*-(Phenyl-cyano-nitromethyl)-benzoyl-cyanoxime

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(*Received* 20 *March* 1975; *accepted* 23 *April* 1975)

The crystal structure of *O*-(phenyl-cyano-nitromethyl)-benzoyl-cyanoxime, $C_{16}H_{10}N_4O_3$, has been determined from three-dimensional intensities, measured with an automated Philips PW 1100 single-crystal diffractometer (3094 independent reflexions). The structure was solved by direct phase determination. The cell constants, obtained by least-squares calculations from direct θ-value measurements on the diffractometer, are $a = 13{\cdot}0346$ (14), $b = 9{\cdot}8186$ (7), $c = 12{\cdot}0830$ (12) Å, $\beta = 101{\cdot}60$ (1)°, $Z = 4$; the space group is $P2_1/n$. The positional and vibrational parameters, with anisotropic temperature factors for the non-hydrogen atoms, were refined by full-matrix least-squares calculations to a final $R = 0{\cdot}078$. The molecule of the compound has the *O*-cyanoxime structure. All the atoms of the main chain lie on a plane, which forms a small angle with one of the benzene rings. An interesting peculiarity of the structure is the rather long C–NO_2 distance of 1·617 Å.

Introduction

The silver salt of phenylnitroacetonitrile, $[C_6H_5C(CN)(NO_2)]Ag$, reacts with CS_2 (Alexandrou, 1965) or with some alkylation reagents (Lianis, 1975) and yields a compound with the molecular formula $C_{16}H_{10}N_4O_3$. From chemical and spectroscopic data (Alexandrou & Lianis, 1975) it could not be decided whether it contained an open chain or a heterocyclic ring. This necessitated an X-ray structure analysis.