

References

- ALBERICCI, M., BRAEKMAN, J. C., DALOZE, D., TURSCH, B., DECLERCQ, J.-P., GERMAIN, G. & VAN MEERSCHE, M. (1978). *Bull. Soc. Chim. Belg.* **87**, 487–492.
- ALLEN, F. H., BELLARD, S., BRICE, M. D., CARTWRIGHT, B. A., DOUBLEDAY, A., HIGGS, H., HUMMELINK, T., HUMMELINK-PETERS, B. G., KENNARD, O., MOTHERWELL, W. D. S., RODGERS, J. R. & WATSON, D. G. (1979). *Acta Cryst.* **B35**, 2331–2339.
- BERNSTEIN, J., SHMUELI, U., ZADOCK, E. & KASHMAN, Y. (1974). *Tetrahedron*, **30**, 2817–2824.
- HALE, R. L., LECLERCQ, J., TURSCH, B., DJERASSI, C., GROSS, R. A. JR, WEINHEIMER, A. J., GUPTA, K. & SCHLEUER, P. J. (1970). *J. Am. Chem. Soc.* **92**, 2179–2180.
- HAMILTON, W. C. (1959). *Acta Cryst.* **12**, 609–610.
- HIRSHFELD, F. L. (1976). *Acta Cryst.* **A32**, 239–244.
- HOSSAIN, M. B., VAN DER HELM, D., MATSON, J. A. & WEINHEIMER, A. J. (1979). *Acta Cryst.* **B35**, 660–666.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
- KARLSSON, R. (1977). *Acta Cryst.* **B33**, 2027–2031.
- MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO78*. A program for drawing crystal and molecular structures. Univ. of Cambridge, England.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
- STROUSE, C. E. (1976). *Rev. Sci. Instrum.* **47**, 871–876.
- TRUEBLOOD, K. N. (1978). *Acta Cryst.* **A34**, 950–954.
- YAO JIA-XING (1981). *Acta Cryst.* **A37**, 642–644.

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Structure of 5,11-Bis(chloroformyl)-2,8-dimethyl-6H,12H-dibenzo[*b,f*][1,5]diazocine at 163 K

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Abstract. $C_{18}H_{16}Cl_2N_2O_2$, $M_r = 363.24$, monoclinic, $P2_1/c$, $a = 10.0742$ (15), $b = 9.9129$ (18), $c = 8.1320$ (12) Å, $\beta = 95.581$ (10)°, $V = 808.2$ (2) Å³, $Z = 2$, $D_x = 1.492$, $D_m(295\text{ K}) = 1.424$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71079$ Å, $\mu = 4.133$ cm⁻¹, $F(000) = 376$, $R = 0.0378$ for 1916 observed reflections. Molecules have a center of symmetry. The eight-membered central ring is in a chair conformation. The planes of the NCOCl groups make angles of 65.79 (4)° with the planes of the benzo bridges such that the Cl and O atoms are in van der Waals contact with the intramolecular H atoms [Cl...H(2), 3.02 (2); O...H(7B), 2.51 (2) Å]. The configuration at N is essentially planar and conjugation is evident in the shortened N–C(benzo) and N–C(carbonyl) bonds [1.439 (2) and 1.347 (2) Å respectively].

Experimental. Title compound prepared by method of Cooper & Partridge (1957). Data crystal obtained by cutting a corner from a large blocky crystal. A summary of data collection and structural refinement is given in Table 1.

Structure solved by *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and Fourier techniques; refined by full-matrix least squares (*SHELX76*, Sheldrick, 1976); all atomic positional parameters refined as well as anisotropic thermal parameters for non-H atoms and isotropic for H atoms. Electron density difference map calculated at $R = 0.06$ revealed H atoms as peaks of 0.67–1.02 e Å⁻³. Scattering factors and anomalous-dispersion corrections for all non-H atoms from *International Tables for X-ray Crystallography* (1974); H scattering factors from Stewart, Davidson & Simpson (1965). Atomic parameters are in Table 2, bond lengths and angles in Table 3.† Atom labeling is shown in Fig. 1 and packing in Fig. 2. Principal computer programs given by Gadol & Davis (1982); program for least-squares-planes' calculations from Cordes (1983).

† Tables of anisotropic thermal parameters, hydrogen bond lengths and angles, torsion angles, least-squares planes and structure-factor amplitudes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42575 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. Crystallographic summary for $C_{18}H_{16}Cl_2N_2O_2$

(A) Data collection (163 K) ^{a,b}	
Mode	ω scan
Scan range	Symmetrically over 1.2° about $K\alpha_{1,2}$ maximum
Background	Offset 1.2 and -1.2° in ω from $K\alpha_{1,2}$ maximum
Scan rate ($^\circ \text{ min}^{-1}$)	3.0–6.0
Exposure time (h)	37.1
Stability analysis ^b	
Check reflections	002,020,400, $\bar{2}\bar{2}1$
Computed s	$-0.0003(2)$
t	$0.000005(5)$
Correction range (on I)	$1.000-1.004$
2θ range ($^\circ$)	$4.0-60.0$
Range in hkl , min.	$0,0,-11$
max.	$14,13,11$
Total reflections measured, unique	2357, 2357
Crystal volume (mm^3)	0.0180
Crystal faces, dimensions (mm)	$\{100\}$, 0.24; $\{011\}$, $\{0\bar{1}\bar{1}\}$, 0.23; $\{01\bar{1}\}$, $\{-0\bar{1}1\}$, 0.30
Absorption correction, transmission-factor range	
	0.906–0.921
(B) Structure refinement ^c	
Instability factor p^b	0.04
Reflections used ($F \geq 4\sigma_F$)	1916
No. of variables	141
Goodness of fit, S	1.462
R , wR	0.0378, 0.0455
R for all data	0.0504
Max. shift/e.s.d.	0.011
Max., min. in difference map ($e \text{ \AA}^{-3}$)	0.46, -0.21

Notes: (a) Unit-cell parameters were obtained by least-squares refinement of the setting angles of 45 reflections with $21.4 < 2\theta < 30.6^\circ$. Crystal density was measured by flotation in aqueous $ZnCl_2$. (b) Syntex $P2_1$ auto-diffractometer with a graphite monochromator and a Syntex LT-1 inert-gas (N_2) low-temperature delivery system. Data reduction was carried out as described by Riley & Davis (1976). Crystal and instrument stability were monitored by re-measurement of four check reflections after every 96 reflections. As detailed by Henslee & Davis (1975), these data were analyzed to relate intensity to exposure time by the equation $y = 1.0 + sx + tx^2$ where x is exposure time (h), y is fractional intensity relative to $x = 0$ and s and t are coefficients determined by least-squares fit. (c) Function minimized was $\sum w(F_o - F_c)^2$, where $w = \sigma_F^{-2}$, $\sigma_F = F_o/2I$, and $\sigma_I = [N_{pk} + N_{b1} + N_{b2} + (pI)^2]^{1/2}$.

Table 2. Positions and U/U_{eq} for atoms in $C_{18}H_{16}Cl_2N_2O_2$

	x	y	z	$U^*(\text{\AA}^2)$
Cl	0.82930 (4)	0.29718 (4)	0.44400 (5)	0.02655 (12)
O	0.62255 (12)	0.16746 (12)	0.5270 (2)	0.0296 (4)
N	0.61699 (12)	0.39432 (12)	0.5698 (2)	0.0165 (3)
C(1)	0.68394 (14)	0.52257 (15)	0.5853 (2)	0.0159 (4)
C(2)	0.79629 (15)	0.5373 (2)	0.6974 (2)	0.0183 (4)
C(3)	0.85674 (15)	0.6623 (2)	0.7187 (2)	0.0191 (4)
C(4)	0.80311 (15)	0.77553 (15)	0.6348 (2)	0.0180 (4)
C(5)	0.68992 (15)	0.7584 (2)	0.5238 (2)	0.0174 (4)
C(6)	0.63052 (14)	0.63252 (15)	0.4948 (2)	0.0154 (4)
C(7)	0.48515 (15)	0.3824 (2)	0.6363 (2)	0.0181 (4)
C(8)	0.6710 (2)	0.2777 (2)	0.5232 (2)	0.0203 (4)
C(9)	0.8652 (2)	0.9124 (2)	0.6643 (2)	0.0249 (5)
H(2)	0.831 (2)	0.462 (2)	0.757 (2)	0.022 (5)
H(3)	0.937 (2)	0.673 (2)	0.799 (2)	0.025 (5)
H(5)	0.655 (2)	0.832 (2)	0.463 (2)	0.022 (5)
H(7A)	0.472 (2)	0.463 (2)	0.699 (2)	0.016 (4)
H(7B)	0.486 (2)	0.302 (2)	0.710 (2)	0.016 (4)
H(9A)	0.808 (3)	0.981 (2)	0.632 (3)	0.049 (7)
H(9B)	0.901 (3)	0.923 (3)	0.778 (3)	0.059 (8)
H(9C)	0.945 (3)	0.916 (3)	0.608 (3)	0.056 (7)

* For non-H atoms, the U value is U_{eq} , calculated as $U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* A_{ij}$ where A_{ij} is the dot product of the i th and j th direct-space unit-cell vectors.

Table 3. Bond lengths (\AA) and angles ($^\circ$) in $C_{18}H_{16}Cl_2N_2O_2$

1	2	3	1–2	1–2–3
C(1)	N	C(7)	1.439 (2)	117.77 (12)
C(7)	N	C(8)	1.487 (2)	115.68 (12)
C(8)	N	C(1)	1.347 (2)	125.74 (13)
C(2)	C(1)	C(6)	1.390 (2)	120.80 (13)
C(2)	C(1)	N		119.70 (13)
C(6)	C(1)	N	1.394 (2)	119.39 (12)
C(3)	C(2)	C(1)	1.384 (2)	119.78 (14)
C(4)	C(3)	C(2)	1.395 (2)	120.96 (13)
C(5)	C(4)	C(9)	1.394 (2)	120.97 (14)
C(5)	C(4)	C(3)		118.17 (14)
C(9)	C(4)	C(3)	1.503 (2)	120.86 (13)
C(6)	C(5)	C(4)	1.394 (2)	121.95 (14)
C(1)	C(6)	C(5)		118.24 (13)
C(1)	C(6)	C(7)		121.93 (13)
C(5)	C(6)	C(7)		119.79 (13)
N	C(7)	C(6)		113.99 (12)
Cl	C(8)	O	1.788 (2)	119.17 (13)
Cl	C(8)	N		114.04 (11)
O	C(8)	N	1.199 (2)	126.8 (2)

Symmetry code: (i) $1 - x, 1 - y, 1 - z$.

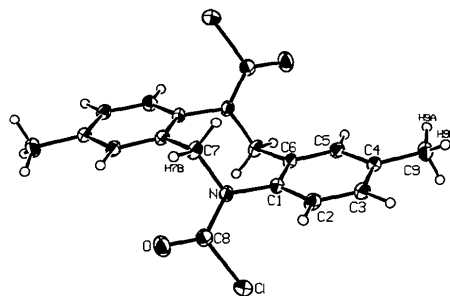


Fig. 1. View of isolated molecule illustrating atom labeling and chair conformation of central eight-membered ring. Plane N, C(7), N(1), C(7) makes an angle of $59.76(6)^\circ$ with the planes of the aromatic rings. Ellipsoids scaled to 50% probability.

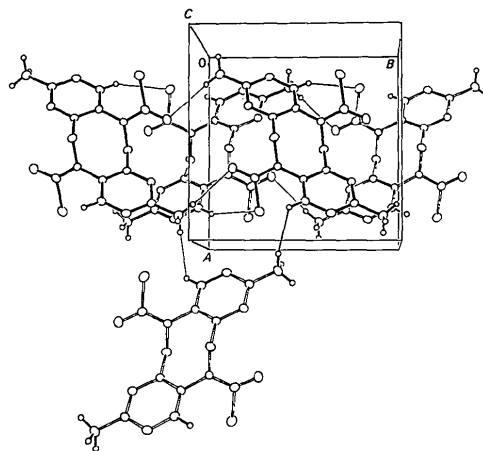


Fig. 2. View of the contents of a unit cell. Closest contacts (indicated by thin lines) based on van der Waals radii are: Cl...H(2)($x, \frac{1}{2} - y, z - \frac{1}{2}$), $2.99(2) \text{ \AA}$ (c direction); O...H(9A)($x, y - 1, z$), $2.71(2) \text{ \AA}$ (b direction); H(2)...H(9C)($2 - x, y - \frac{1}{2}, \frac{1}{2} - z$), $2.46(3) \text{ \AA}$ (a direction). Darkened molecules centered on $z = \frac{1}{2}$ plane; others on $z = 0$ or 1 planes.

Related literature. Structures containing the carbamoyl moiety have been reported by Baggio, Becka, Amzel, Avey & Poljak (1973) and Ganis, Avitabile, Migdal & Goodman (1971).

References

- BAGGIO, S., BECKA, L. N., AMZEL, L. M., AVEY, H. P. & POLJAK, R. J. (1973). *Cryst. Struct. Commun.* **3**, 531–534.
- COOPER, F. C. & PARTRIDGE, M. W. (1957). *J. Chem. Soc.* pp. 2888–2893.
- CORDES, A. W. (1983). Personal communication.
- GADOL, S. M. & DAVIS, R. E. (1982). *Organometallics*, **1**, 1607–1613.
- GANIS, P., AVITABILE, G., MIGDAL, S. & GOODMAN, M. (1971). *J. Am. Chem. Soc.* **93**, 3328–3331.
- HENSLEE, W. H. & DAVIS, R. E. (1975). *Acta Cryst.* **B31**, 1511–1519.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
- MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- RILEY, P. E. & DAVIS, R. E. (1976). *Acta Cryst.* **B32**, 381–386.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

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Stereochemical Studies of Oligomers. XVI.* 2,3-Butanediyl Bis(*m*-chlorobenzoate)

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Abstract. C₁₈H₁₆Cl₂O₄, *M_r* = 367.2, triclinic, *P* $\bar{1}$, *a* = 8.327 (3), *b* = 7.874 (2), *c* = 7.645 (3) Å, α = 61.89 (4), β = 91.46 (3), γ = 93.02 (3)°, *V* = 441.5 (3) Å³, *Z* = 1, *D_x* = 1.38 g cm⁻³, $\lambda(\text{Cu } K\alpha)$ = 1.5418 Å, μ = 35.23 cm⁻¹, *F*(000) = 190, room temperature, *R* = 0.059 for 983 observed reflections. The conformation of the molecule is *trans* in the central part of the aliphatic chain while the rest differs noticeably from this arrangement, the C(7)–O(2)–C(8)–C(8') torsion angle being 149.9 (4)°. The carboxylic groups are tilted by 12.2 (2)° with respect to the aromatic ring planes. The two methyls are *trans* with respect to the C(8)–C(8') bond.

Experimental. Prismatic specimen 0.2 × 0.4 × 0.4 mm, lattice parameters refined by least squares by use of 19 (θ, χ, φ)_{*hkl*} accurate measurements on a Siemens AED single-crystal diffractometer on line to a General Automation Jumbo 220 computer. Intensities collected (2 < θ < 70°) with a modified version (Belletti, Ugozzoli, Cantoni & Pasquinelli, 1979) of the Lehmann & Larsen (1974) procedure, one check reflection recorded every 50 counts, only statistical variation in intensity, 983 observed reflections [*I* >

2 σ (*I*)] retained out of a total of 1486 measured independent reflections, $-10 \leq h \leq 10$, $-7 \leq k \leq 8$, $0 \leq l \leq 8$, data corrected for Lorentz and polarization effects but not for absorption. Direct methods with *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), atomic coordinates of heavy atoms refined by full-matrix least squares first with isotropic temperature factors and then with anisotropic ones using *SHELX76* (Sheldrick, 1976), H atoms found in a difference Fourier map and refined isotropically; *R* = 0.059 and *wR* = 0.061 where *w* = 0.3972/($\sigma^2 F + 0.02812 F^2$), $\sum w(F_o - F_c)^2$ minimized, final difference electron density map did not show peaks > 0.26 e Å⁻³, (Δ/σ)_{max} = 0.288, scattering factors those of *SHELX*.

All calculations performed on a Gould Sel 32/77 computer.†

Table 1 gives the atom parameters and Table 2 bond lengths, angles and selected torsion angles. Fig. 1 shows the molecule and numbering scheme.

† Lists of structure factors, thermal parameters, H coordinates and bond lengths involving H have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42610 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

* Part XV: Bocelli, Ugozzoli & Grenier-Loustalot (1985).