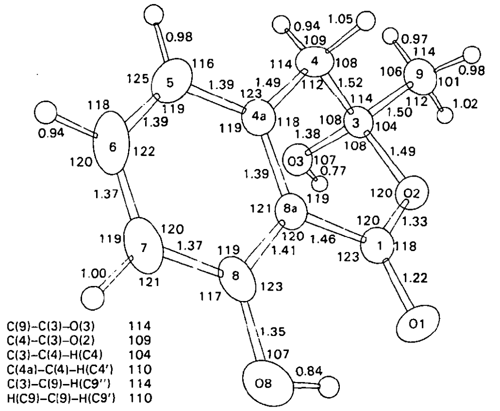


Fig. 1. The molecular structure of (I). Bond lengths (Å) and angles (°) are shown.

The dihydropyran ring of (I) adopts a half-chair form and the hydroxyl group at C(3) is in an axial conformation. Intramolecular hydrogen bonds may be formed between O(1) and H(O8)–O(8) (2·607 Å for O–O and 1·868 Å for O–H). The non-bonding distance of 3·017 Å found between C(9) and O(72) in the molecule of (II) is shorter than that expected. This will cause ring formation at C(9) by attacking the carbonyl group. The aromatic rings of (I) and (II) are almost planar as expected. The molecules are packed together mainly by the hydrogen bonding in both crystals. Non-bonding distances of 2·808 and 2·669 Å are found between O(1) and O(3) in (I) and O(9) and O(71) in (II), respectively.

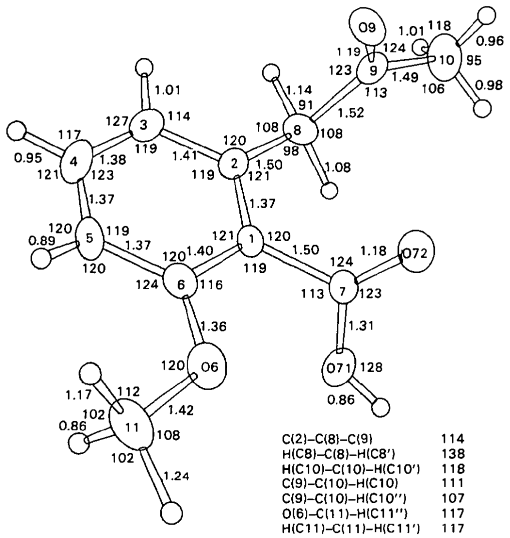


Fig. 2. The molecular structure of (II). Bond lengths (Å) and angles (°) are shown.

References

Germain, G., Main, P. & Woolfson, M. M. (1970). *Acta Cryst.* B**26**, 274–285.

Gripenberg, J. (1974). *Acta Chem. Scand. Ser. B*, **28**, 505–508.

Kameda, K., Aoki, H., Tanaka, H. & Namiki, M. (1973). *Agric. Biol. Chem.* **37**, 2137–2146.

Acta Cryst. (1985). C**41**, 417–419

Structure and Conformation of Two Diastereomers of (±)-1-(2,3-Dichlorophenyl)-3,5,6,7-tetrahydro-3-hydroxy-3,6,6-trimethyl-1*H*-indole-2,4-dione, $C_{17}H_{17}Cl_2NO_3$

By Kuantee Go and Gopinath Kartha

Roswell Park Memorial Institute, 666 Elm Street, Buffalo, New York 14263, USA

and N. Viswanathan

Ciba–Geigy, Research Centre, Goregaon, Bombay 63, India

(*Received* 28 *June* 1984; *accepted* 19 *October* 1984)

Abstract. The two diastereomers A and B of the title compound ($M_r = 354{\cdot}2$) crystallized in two different crystal systems: (A) monoclinic, $P2_1/c$, $a = 9{\cdot}319$ (1), $b = 16{\cdot}098$ (2), $c = 12{\cdot}013$ (1) Å, $\beta = 109{\cdot}11$ (1)°, $V = 1703$ Å^3, $Z = 4$, $D_x = 1{\cdot}381$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 3{\cdot}603$ mm^{-1}, $F(000) = 736$, room temperature, $R = 0{\cdot}049$ for 2889 observed reflections; (B) triclinic, $P\bar{1}$, $a = 8{\cdot}861$ (1), $b = 9{\cdot}855$ (1), $c = 11{\cdot}020$ (1) Å, $\alpha = 115{\cdot}47$ (1), $\beta = 103{\cdot}05$ (1), $\gamma = 88{\cdot}13$ (1)°, $V = 844$ Å^3, $Z = 2$, $D_x = 1{\cdot}394$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 3{\cdot}636$ mm^{-1}, $F(000) = 368$, room temperature, $R = 0{\cdot}050$ for 3057 observed reflections. The two structures show that diastereomer A has the OH at the asymmetric carbon on the same side as the Cl in the phenyl ring while B has the CH_3 at the asymmetric carbon on the same side as the Cl. In A,

0108-2701/85/030417-03$01.50

Table 1. *Fractional coordinates* (×10^4) *and equivalent isotropic thermal parameters of non*-H *atoms*

	x	y	z	B_{eq}(Å^2)*
Form *A*				
Cl(2′)	1819 (1)	−1500 (1)	4934 (1)	5·8
Cl(3′)	3477 (1)	222 (1)	5637 (1)	6·9
O(2)	3760 (2)	−3301 (1)	6609 (2)	4·2
O(3)	1025 (2)	−4099 (1)	4959 (1)	3·5
O(4)	−2188 (2)	−4358 (1)	5418 (1)	3·7
N(1)	1830 (2)	−2553 (1)	6940 (1)	2·7
C(2)	2463 (2)	−3243 (1)	6574 (2)	2·9
C(3)	1195 (2)	−3902 (1)	6141 (2)	2·6
C(4)	−1735 (2)	−3674 (1)	5835 (1)	2·6
C(5)	−2848 (2)	−3015 (1)	5922 (2)	2·8
C(6)	−2232 (2)	−2411 (1)	6956 (2)	2·9
C(7)	−732 (2)	−2026 (1)	6923 (2)	3·2
C(8)	264 (2)	−2673 (1)	6680 (1)	2·5
C(9)	−163 (2)	−3427 (1)	6219 (1)	2·5
C(10)	1615 (2)	−4649 (1)	6961 (1)	3·6
C(11)	−1967 (2)	−2886 (1)	8108 (2)	4·4
C(12)	−3389 (2)	−1721 (1)	6871 (2)	4·7
C(1′)	2593 (2)	−1774 (1)	7257 (1)	2·5
C(2′)	2647 (2)	−1227 (1)	6385 (2)	3·1
C(3′)	3383 (2)	−468 (1)	6703 (2)	3·4
C(4′)	4054 (2)	−268 (1)	7870 (2)	3·7
C(5′)	3996 (2)	−815 (1)	8727 (2)	3·9
C(6′)	3271 (2)	−1576 (1)	8427 (2)	3·4
Form *B*				
Cl(2′)	5178 (1)	3304 (1)	4844 (1)	4·7
Cl(3′)	5586 (1)	2728 (1)	7477 (1)	5·3
O(2)	4709 (2)	495 (2)	1748 (2)	4·8
O(3)	2541 (2)	222 (2)	−831 (2)	4·0
O(4)	154 (2)	2832 (2)	−942 (2)	5·0
N(1)	2535 (2)	1495 (2)	2513 (1)	3·3
C(2)	3509 (2)	1104 (2)	1620 (2)	3·6
C(3)	2782 (2)	1539 (2)	442 (2)	3·2
C(4)	94 (3)	2762 (2)	121 (2)	3·6
C(5)	−1249 (3)	3323 (3)	797 (2)	4·0
C(6)	−802 (2)	3963 (2)	2391 (2)	3·4
C(7)	10 (2)	2758 (2)	2798 (2)	3·2
C(8)	1218 (2)	2113 (2)	2012 (2)	3·0
C(9)	1285 (2)	2108 (2)	806 (2)	3·2
C(10)	3819 (3)	2745 (3)	461 (2)	4·1
C(11)	−2280 (3)	4304 (3)	2946 (2)	4·7
C(12)	262 (3)	5395 (3)	3006 (2)	4·4
C(1′)	2762 (2)	1211 (2)	3709 (2)	3·1
C(2′)	3954 (2)	2002 (2)	4852 (2)	3·1
C(3′)	4135 (2)	1737 (2)	6022 (2)	3·5
C(4′)	3154 (3)	701 (2)	6036 (2)	4·3
C(5′)	1989 (3)	−87 (2)	4895 (2)	4·6
C(6′)	1785 (3)	172 (2)	3722 (2)	4·0

* $B_{eq} = 8\pi^2 U_{eq}$ where $U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*(\mathbf{a}_i.\mathbf{a}_j)$.

the dichlorophenyl ring makes an angle of 88·3 (2)° with the five-membered ring of the indole while in *B*, this angle is 66·6 (3)°. In the crystals, the OH at the asymmetric carbon forms an intermolecular hydrogen bond with the C=O of the six-membered ring in *A*, while in *B*, the intermolecular hydrogen bond is between the OH and the C=O of the five-membered ring.

Introduction. In their studies on the restricted rotation of the N–aryl bond, Viswanathan and co-workers obtained two racemic diastereoisomers in the course of the synthetic studies in dimedone enamines. We have examined the crystals and found that these two diastereomers have interesting conformational differences. We report here our results from X-ray diffraction studies.

Experimental. Crystals obtained from ethanol by slow evaporation. Crystal size for *A* 0·175 × 0·375 × 0·6 mm, for *B* 0·225 × 0·45 × 0·45 mm. Enraf–Nonius CAD-4 automated diffractometer, Ni-filtered Cu $K\alpha$. Lattice dimensions by least-squares fit to a set of 25 reflections in θ range 15–28°. ω–2θ scans and integrated counts with θ_{max} to 77° for both crystals. For *A*, 3600 independent reflections measured, $h \pm 11$, k $0 \rightarrow 20$, l $0 \rightarrow 15$, 2889 with $I > 2\sigma(I)$; for *B* 3567 independent reflections measured, h $0 \rightarrow 11$, k ± 11, l ± 13, 3057 with $I > 2\sigma(I)$. Three standard reflections (overall $\sigma = 0{\cdot}01$ for both *A* and *B*). Lp correction, empirical (one parameter, φ) absorption correction (range 0·95–1·3 for *A*, 0·87–1·4 for *B*). Direct methods (*MULTAN*, Germain, Main & Woolfson, 1971). Anisotropic full-matrix least-squares refinement for 23 non-H atoms and isotropic for H atoms (found from ΔF synthesis). $R = 0{\cdot}049$, $R_w = 0{\cdot}051$ for *A*, $R = 0{\cdot}050$, $R_w = 0{\cdot}053$ for *B* [for reflections with

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

	Form *A*	Form *B*		Form *A*	Form *B*		Form *A*	Form *B*
N(1)–C(2)	1·394 (2)	1·378 (2)	C(9)–C(4)	1·441 (1)	1·455 (2)	N(1)–C(1′)	1·430 (2)	1·431 (2)
C(2)–C(3)	1·547 (2)	1·538 (2)	C(4)–C(5)	1·511 (2)	1·511 (3)	C(1′)–C(2′)	1·383 (2)	1·385 (2)
C(2)–O(2)	1·199 (1)	1·213 (2)	C(4)–O(4)	1·227 (2)	1·216 (2)	C(2′)–C(3′)	1·393 (2)	1·396 (2)
C(3)–C(9)	1·508 (2)	1·495 (3)	C(5)–C(6)	1·553 (2)	1·546 (3)	C(2′)–Cl(2′)	1·717 (1)	1·712 (2)
C(3)–O(3)	1·412 (2)	1·420 (2)	C(6)–C(7)	1·541 (2)	1·544 (2)	C(3′)–Cl(3′)	1·719 (2)	1·728 (2)
C(3)–C(10)	1·523 (2)	1·517 (2)	C(6)–C(11)	1·528 (3)	1·536 (3)	C(3′)–C(4′)	1·372 (3)	1·370 (2)
C(9)–C(8)	1·339 (2)	1·341 (2)	C(6)–C(12)	1·528 (2)	1·521 (3)	C(4′)–C(5′)	1·370 (3)	1·369 (3)
C(8)–N(1)	1·402 (1)	1·411 (2)	C(7)–C(8)	1·486 (2)	1·483 (2)	C(5′)–C(6′)	1·389 (2)	1·395 (3)
						C(6′)–C(1′)	1·376 (2)	1·370 (2)
C(8)–N(1)–C(2)	109·7 (2)	109·6 (2)	C(9)–C(8)–C(7)	126·6 (2)	126·5 (2)	C(6)–C(7)–C(8)	110·7 (2)	110·3 (2)
N(1)–C(2)–C(3)	107·2 (2)	107·9 (2)	C(7)–C(8)–N(1)	122·1 (2)	122·7 (2)	N(1)–C(1′)–C(2′)	119·6 (2)	119·9 (2)
N(1)–C(2)–O(2)	125·2 (3)	124·9 (3)	C(8)–N(1)–C(1′)	124·8 (2)	124·9 (2)	N(1)–C(1′)–C(6′)	119·8 (2)	119·7 (2)
C(3)–C(2)–O(2)	127·4 (3)	127·2 (3)	C(2)–N(1)–C(1′)	123·8 (2)	125·2 (2)	C(6′)–C(1′)–C(2′)	120·5 (2)	120·4 (3)
C(2)–C(3)–C(9)	101·2 (2)	101·3 (2)	C(9)–C(4)–C(5)	115·7 (2)	115·7 (3)	C(1′)–C(2′)–C(3′)	119·2 (2)	119·1 (2)
C(2)–C(3)–O(3)	108·3 (2)	108·6 (2)	C(9)–C(4)–O(4)	123·8 (2)	122·4 (3)	C(1′)–C(2′)–Cl(2′)	119·4 (2)	120·6 (2)
C(9)–C(3)–O(3)	110·9 (2)	112·0 (2)	C(5)–C(4)–O(4)	120·4 (2)	121·8 (3)	C(3′)–C(2′)–Cl(2′)	121·4 (2)	120·2 (2)
C(2)–C(3)–C(10)	108·5 (2)	110·3 (2)	C(4)–C(5)–C(6)	114·7 (2)	114·1 (3)	C(2′)–C(3′)–C(4′)	120·3 (2)	120·5 (3)
C(9)–C(3)–C(10)	113·8 (2)	112·5 (2)	C(5)–C(6)–C(7)	109·6 (2)	108·9 (2)	C(2′)–C(3′)–Cl(3′)	120·1 (2)	120·3 (2)
O(3)–C(3)–C(10)	113·2 (2)	111·7 (2)	C(5)–C(6)–C(11)	108·8 (2)	109·4 (3)	C(4′)–C(3′)–Cl(3′)	119·5 (2)	119·1 (2)
C(3)–C(9)–C(8)	110·1 (2)	110·3 (2)	C(5)–C(6)–C(12)	109·7 (2)	110·1 (2)	C(3′)–C(4′)–C(5′)	120·0 (3)	119·9 (3)
C(4)–C(9)–C(8)	121·0 (2)	120·8 (3)	C(7)–C(6)–C(11)	110·2 (2)	107·9 (2)	C(4′)–C(5′)–C(6′)	120·4 (3)	120·3 (3)
C(3)–C(9)–C(4)	128·7 (2)	128·4 (3)	C(7)–C(6)–C(12)	109·4 (2)	110·6 (2)	C(5′)–C(6′)–C(1′)	119·4 (3)	119·7 (3)
C(9)–C(8)–N(1)	111·3 (2)	110·6 (2)	C(11)–C(6)–C(12)	109·0 (2)	109·8 (3)			

$I > 2\sigma(I)$]; $\Delta/\sigma < 0{\cdot}1$ for A, $0{\cdot}2$ for B; max. and min. heights in final ΔF synthesis $0{\cdot}22$ and $-0{\cdot}20$ e Å^{-3} for A, $0{\cdot}64$ and $-0{\cdot}52$ e Å^{-3} for B; $\sum w(|F_o|-|F_c|)^2$ minimized where $w^{-1} = [\sigma^2(F) + (cF)^2]$; f curves from *International Tables for X-ray Crystallography* (1962). Enraf–Nonius (1978) *SDP* and local programs.

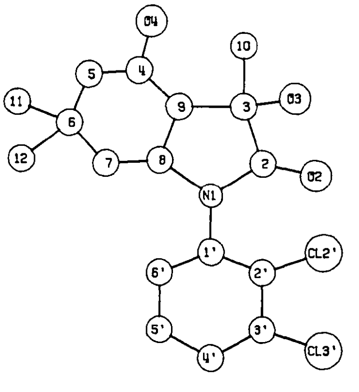
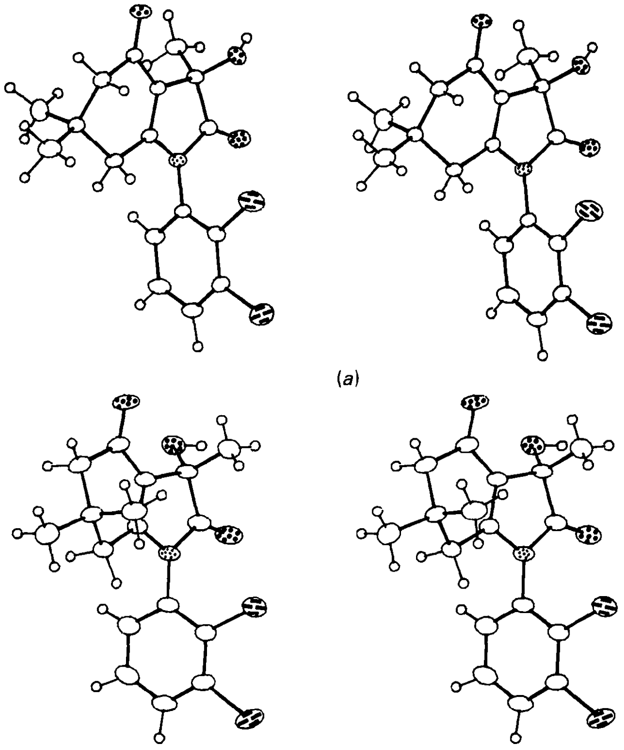


Fig. 1. Labelling of atoms.


(*a*)

(*b*)

Fig. 2. Stereoscopic view: (*a*) form *A*; (*b*) form *B*. Atoms shaded by dashes, large dots and small dots indicate Cl, O and N atoms, respectively.

Table 3. *Intermolecular hydrogen bonds*

Diastereomer	*A*	*B*
Donor	O(3)	O(3)
Acceptor	O(4)	O(2)
O–H···O distance (Å)	2·805 (2)	2·801 (2)
O–H distance (Å)	0·75 (3)	0·89 (4)
H···O distance (Å)	2·06 (3)	1·98 (4)
O–H···O angle (°)	170 (3)	152 (3)
Symmetry operator of the second molecule	$-x$, $(1+y)$, $1-z$	$1-x$, $-y$, $-z$

Discussion. The final parameters are given in Table 1;* the standard deviations were calculated using Cruickshank's (1965) expressions.

The two diastereomers *A* and *B* differ in their conformations and configurations around the asymmetric carbon, C(3). Stereoscopic views for *A* and *B* are shown in Figs. 2(*a*) and (*b*) respectively (labelling of atoms in Fig. 1). *A* has the OH at C(3) on the same side as the Cl atoms in the dichlorophenyl ring; the phenyl ring makes as angle of 88·3 (2)° with the five-membered ring. *B* has the CH_3 at C(3) on the same side as the Cl atoms; the phenyl ring makes an angle of 66·6 (3)° with the five-membered ring. With these differences, molecules pack differently in the crystals and form different intermolecular hydrogen bonds. The OH at C(3) forms an intermolecular hydrogen bond with O(4) in *A*, while in *B*, the hydrogen bond is with O(2). There are slight variations in ring puckering as indicated by the torsion angles.* It is seen that the main conformational differences for these two diastereomers arise from the rotation about the N(1)–C(1′) bond (the aryl bond). Table 2 gives the bond lengths and angles. Values for the two structures agree fairly closely within experimental errors. The hydrogen bonds are given in Table 3.

This work was supported by the New York State Department of Health.

References

Cruickshank, D. W. J. (1965). In *Computing Methods in Crystallography*. Oxford: Pergamon Press.

Enraf–Nonius (1978). *Structure Determination Package*. Enraf–Nonius, Delft.

Germain, G., Main, P. & Woolfson, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

International Tables for X-ray Crystallography (1962). Vol. III. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

---

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and torsion angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39847 (42 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.