atoms, $R = 0{\cdot}045$, $wR = 0{\cdot}037$, $w = 1/\sigma^2(F_o)$ from counting statistics, $S = 2{\cdot}00$; $(\Delta/\sigma)_{\max} = 0{\cdot}002$; $|\Delta\rho|_{\max}$ in final difference Fourier map $= 0{\cdot}16$ e Å^{-3}; all numerical calculations carried out on a Facom M-382 computer in the Science Information Processing Center of the University of Tsukuba; scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. The final atomic parameters are listed in Table 1.* The molecular structure and numbering scheme are illustrated in the *ORTEP* plot (Johnson, 1976) given in Fig. 1. Bond distances and angles are listed in Table 2. The benzyloxy group was found to be *anti* to the bridge C=CH$_2$ group. Ring *A* which bears the benzyloxy group has a chair conformation, resulting in the substituent being axial. This is consistent with the conformation estimated from the ^{1}H NMR spectrum in solution. The bond angles and torsion angles (Table 2) show that ring *A* flattens slightly due to the presence of the axial benzyloxy group. The interatomic distances from O(12) to C(2), C(3) and C(4) are 3·047 (5), 3·029 (5) and 3·017 (5) Å, respectively; all are longer than the estimated values (2·2–2·5 Å) from a Dreiding model. Ring *B* having an α,β-unsaturated carbonyl moiety exists in a half-chair form.

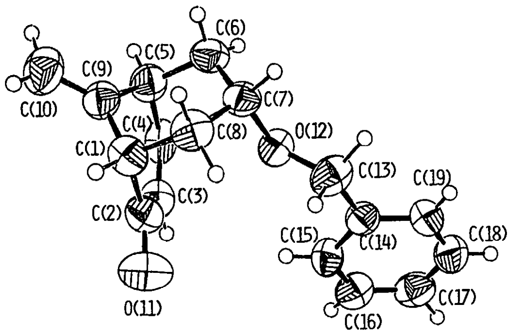

Fig. 1. *ORTEP* drawing (Johnson, 1976) of the molecule with thermal ellipsoids scaled at the 50% probability level and numbering scheme. H atoms are represented by circles of radius 0·1 Å.

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* Lists of structure factors, anisotropic temperature factors, H-atom coordinates and bond distances involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43920 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1987). C**43**, 1601–1603

2-(*p*-Chlorophenyl)-4-phenylindan-1,3-dione

By John C. Barnes* and Wigen Golnazarians

*Chemistry Department, The University, Dundee DD*1 *4HN, Scotland*

and Alexander C. Campbell and James Redpath

*Organon Laboratories Ltd, Newhouse, Lanarkshire ML*1 *5SH, Scotland*

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Abstract. C$_{21}$H$_{13}$ClO$_2$, $M_r = 332{\cdot}9$, monoclinic, $P2_1/n$, $a = 8{\cdot}420$ (2), $b = 10{\cdot}913$ (4), $c = 17{\cdot}899$ (3) Å, $\beta = 91{\cdot}79$ (3)°, $V = 1643{\cdot}9$ Å^3, $Z = 4$, $D_x = 1{\cdot}338$ Mg m^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}71069$ Å, $\mu = 0{\cdot}197$ mm^{-1}, $F(000) = 688$, $T = 293$ K, $R = 0{\cdot}060$ for 2073 reflexions. The molecule has no unusual bond lengths or angles. There is substantial librational motion of the chlorophenyl ring. The indandione system is twisted out of planarity [maximum deviation from benzene plane 0·253 (3) Å of C atom at 2-position].

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* To whom correspondence should be addressed.

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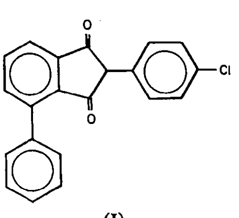
Introduction. The title compound, known by the reference number ORG 7258 (Campbell & Stevenson, 1985), is effective in acute, semi-chronic and chronic models of inflammation. Unlike other 2-phenylindan-1,3-diones, it does not inhibit blood coagulation. ORG 7258 is therefore of pharmacological interest as an agent for the treatment of inflammatory diseases (*e.g.* rheumatoid arthritis), asthma and psoriasis.

The molecular structure (I) has been confirmed by single-crystal structure determination.



(I)

Experimental. Crystals of the title compound were supplied by Organon Laboratories Ltd as pale-yellow clustered needles. Preliminary photographs showed that the crystals were monoclinic and of poor quality, leading to the high value of R_{int} given below. Unit-cell dimensions were refined from 25 reflexions with $\theta \simeq 12{\cdot}5°$ using an Enraf–Nonius CAD-4F diffractometer. Data were collected for one quadrant of a crystal 0·2 × 0·2 × 0·4 mm, to a θ limit of 25°, 3157 observed reflexions gave 2884 unique reflexions of which 2073 reflexions with $F_o > 2\sigma(F_o)$ were used in the refinement ($R_{\text{int}} = 0{\cdot}14$). Ranges of indices, $-10 \leq h \leq 10$, $0 \leq k \leq 12$, $0 \leq l \leq 21$. The intensities of two standard reflexions were checked every hour, and the orientation of the crystal verified every 400 reflexions. Data were corrected for Lorentz and polarization terms and for absorption using a ψ-scan routine during data collection, maximum and minimum transmission coefficients 0·84 and 1·09. No drift in intensity was observed during data collection.

All calculations were performed on the Dundee University DEC 10 computer using *SHELX*76 (Sheldrick, 1976), *XANADU* (Roberts & Sheldrick, 1975) and *PLUTO* (Motherwell & Clegg, 1978). Atomic scattering parameters were taken from *International Tables for X-ray Crystallography* (1974).

The structure was solved by direct-methods routine *EEES* of *SHELX*76. Structure developed by difference synthesis and least-squares refinement, first with isotropic thermal parameters and then anisotropic thermal parameters for all non-H atoms. The model refined to convergence at $R = 0{\cdot}060$ with H atoms included at calculated positions. Isotropic temperature factors refined for H atoms. Before this model was accepted as the best solution an attempt was made to investigate the large anisotropic thermal parameters of Cl(24). A rigid-body calculation on the chlorophenyl group alone gave $RG = \sum|\Delta U_{ij}|/\sum|U_{ij}| = 0{\cdot}023$

Table 1. *Coordinates* (×10⁴) *for non-*H *atoms and equivalent isotropic temperature factors* (Å²×10³) *with e.s.d.'s in parentheses*

$$U_{\text{eq}} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
C(1)	9085 (3)	3828 (2)	4236 (1)	48 (1)
C(2)	8159 (3)	4834 (2)	4041 (1)	41 (1)
C(3)	8862 (3)	5917 (2)	3780 (1)	44 (1)
C(4)	10509 (3)	5922 (3)	3763 (1)	58 (1)
C(5)	11421 (3)	4907 (3)	3961 (2)	63 (1)
C(6)	10721 (3)	3830 (3)	4196 (2)	57 (1)
C(7)	8081 (3)	2813 (2)	4511 (2)	54 (1)
C(8)	6388 (3)	3296 (2)	4548 (1)	49 (1)
C(9)	6478 (3)	4585 (2)	4207 (1)	46 (1)
O(10)	8527 (3)	1794 (2)	4676 (2)	83 (1)
O(11)	5360 (2)	5253 (2)	4116 (1)	63 (1)
C(12)	7941 (3)	6996 (2)	3515 (1)	49 (1)
C(13)	8295 (4)	8157 (3)	3786 (2)	64 (1)
C(14)	7453 (5)	9164 (3)	3522 (2)	81 (1)
C(15)	6280 (5)	9022 (4)	2989 (3)	88 (1)
C(16)	5914 (4)	7871 (4)	2716 (2)	80 (1)
C(17)	6741 (3)	6862 (3)	2974 (2)	61 (1)
C(18)	5136 (3)	2473 (2)	4206 (1)	47 (1)
C(19)	4388 (3)	1600 (3)	4636 (2)	60 (1)
C(20)	3279 (4)	810 (3)	4335 (2)	68 (1)
C(21)	2900 (3)	907 (3)	3580 (2)	67 (1)
C(22)	3579 (4)	1773 (3)	3145 (2)	70 (1)
C(23)	4715 (4)	2545 (3)	3455 (2)	62 (1)
Cl(24)	1510 (1)	−89 (1)	3181 (1)	115 (1)

Table 2. *Interatomic distances* (Å)

C(2)—C(1)	1·385 (3)	C(13)—C(12)	1·385 (4)
C(6)—C(1)	1·382 (3)	C(17)—C(12)	1·386 (4)
C(7)—C(1)	1·487 (4)	C(14)—C(13)	1·383 (5)
C(3)—C(2)	1·407 (3)	C(15)—C(14)	1·361 (6)
C(9)—C(2)	1·481 (3)	C(16)—C(15)	1·378 (6)
C(4)—C(3)	1·388 (4)	C(17)—C(16)	1·375 (5)
C(12)—C(3)	1·480 (4)	C(19)—C(18)	1·388 (4)
C(5)—C(4)	1·387 (4)	C(23)—C(18)	1·381 (4)
C(6)—C(5)	1·386 (4)	C(20)—C(19)	1·370 (4)
C(8)—C(7)	1·523 (4)	C(21)—C(20)	1·384 (5)
O(10)—C(7)	1·207 (3)	C(22)—C(21)	1·361 (5)
C(9)—C(8)	1·536 (3)	Cl(24)—C(21)	1·734 (3)
C(18)—C(8)	1·501 (3)	C(23)—C(22)	1·378 (4)
O(11)—C(9)	1·198 (3)		

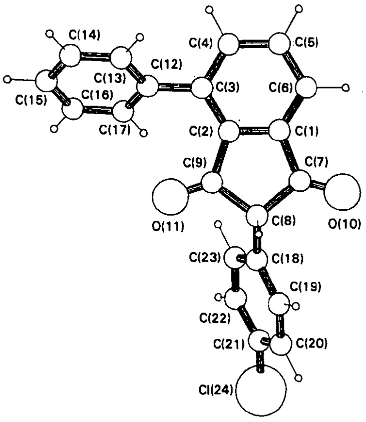


Fig. 1. The molecule viewed normal to the plane C(1), C(3), C(5).

compared with 0·245 for the whole molecule, suggesting that the thermal parameters of Cl(24) represent a librational motion of the whole ring wagging about C(8) and not a disordered structure. This was confirmed by least-squares refinement of a model with the Cl atom divided between two sites. This model refined to $R = 0·058$ but gave very unsatisfactory positional and thermal parameters for Cl(24).

Final refinement (minimizing $\sum w|F_o - |F_c||^2$): 231 refined parameters in two blocks, $R = 0·060$, $wR = 0·098$, $w = 0·1950/[\sigma^2(F) + 0·0299F^2]$, mean shift/e.s.d. = 0·064, max. shift/e.s.d. = 0·297, max. difference peak = 0·33 e Å^{-3}, max. negative peak = 0·38 e Å^{-3}.

Discussion. Atomic coordinates for the molecule of (I) are given in Table 1,* with bond lengths and angles in Table 2. The molecule is shown in Fig. 1.

The five-membered ring of the indandione system is twisted so that C(7), C(8) and C(9) are 0·069 (3), 0·253 (3) and 0·146 (2) Å from the plane of the benzene ring. O(11) is raised 0·200 (2) Å above the plane of the benzene ring whereas O(10) is 0·007 (3) Å below the plane. The normals to the planes of the phenyl groups C(12)–C(17) and C(18)–C(23) make angles of 127·5 (1) and 67·4 (1)° to the normal to the plane of C(1)–C(6). These features avoid approaches of the ring protons to O(10) and O(11).

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43970 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1987). C**43**, 1603–1607

Structure of (3*R*,6*R*)-3-Isobutyl-1,4-diazabicyclo[4.2.0]octane-2,5-dione and (3*S*,6*R*)-3-Isobutyl-1,4-diazabicyclo[4.4.0]decane-2,5-dione

By Jindřich Symerský

Institute of Organic Chemistry and Biochemistry, Czechoslovak Academy of Sciences, 166 10 *Praha* 6, *Czechoslovakia*

Karel Huml

Institute of Macromolecular Chemistry, Czechoslovak Academy of Sciences, 162 02 *Praha* 6, *Czechoslovakia*

and Václav Petříček

Institute of Physics, Czechoslovak Academy of Sciences, 180 40 *Praha* 8, *Czechoslovakia*

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Abstract. (I): $C_{10}H_{16}N_2O_2$, $M_r = 196·25$, orthorhombic, $P2_12_12_1$, $a = 9·097$ (4), $b = 17·682$ (6), $c = 6·559$ (5) Å, $V = 1055$ (1) Å^3, $Z = 4$, $D_x = 1·24$ (1) Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0·71069$ Å, $\mu = 0·081$ mm^{-1}, $F(000) = 424$, $T = 295$ K. Final $R = 0·046$ for 1575 unique observed reflections. (II): $C_{12}H_{20}N_2O_2$, $M_r = 224·30$, orthorhombic, $P2_12_12_1$, $a = 10·04$ (3), $b = 20·22$ (6), $c = 6·35$ (1) Å, $V = 1290$ (6) Å^3, $Z = 4$, $D_x = 1·16$ (1) Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0·71069$ Å, $\mu = 0·074$ mm^{-1}, $F(000) = 488$, $T = 295$ K. Final $R = 0·054$ for 1317 unique observed reflections. The 2,5-piperazinedione ring in a twist-boat conformation becomes more puckered as the side-ring size decreases. There is a pronounced non-planarity of bonds at N(7) as a consequence of the side-ring constraints.

Introduction. 2,5-Piperazinediones can serve as model compounds for an investigation of mutual spatial relations between two homoconjugated peptide groups

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