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1′,3′-Dicyclohexylspiro[2*H*-1-benzopyran-2,4′-imidazolidine]-2′,4(3*H*),5′-trione, $C_{23}H_{28}N_2O_4$

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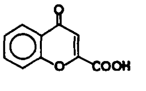
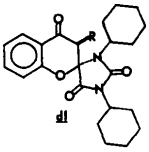
Abstract. $M_r = 396{\cdot}5$, monoclinic, $P2_1/c$, $a = 9{\cdot}935\ (4)$, $b = 6{\cdot}669\ (2)$, $c = 31{\cdot}983\ (9)$ Å, $\beta = 90{\cdot}51\ (3)°$, $V = 2119\ (1)$ Å^3, $Z = 4$, $D_m = 1{\cdot}22\ (2)$ (flotation in aq. $ZnBr_2$), $D_x = 1{\cdot}24$ g cm^{-3}, λ (Mo $K\alpha$) $= 0{\cdot}71069$ Å, $\mu = 0{\cdot}80$ cm^{-1}, $F(000) = 848$, $T = 298$ K, $R = 0{\cdot}053$ for 1934 observed reflections. An unexpected product from the treatment of an α,β-unsaturated acid with *N,N*′-dicyclohexylcarbodiimide and its monochlorination product are shown to be imidazolidine-2,4-diones. There are no unusual bond distances or bond angles.

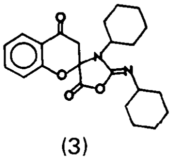
Introduction. During attempts to make esters of acid (1) with dicyclohexylcarbodiimide (DCC), we obtained not esters but instead a crystalline 1:1 adduct of (1) and DCC in good yield. Through elemental analysis and spectral data, we came to the conclusion that the adduct was either (2*a*) or (3). We now report that X-ray results conclusively establish this adduct to be (2*a*) and its monochlorination product to be (2*b*).



(1)



R
(2*a*) H
(2*b*) Cl



(3)

Experimental. *Compound* (2*a*). DCC (2·06 g) in 5 mL dioxane was added in one portion to a hot solution of (1) (1·90 g) in 30 mL dioxane. After refluxing 5h and removing the solvent on a rotary evaporator, the residue was crystallized from absolute ethanol (40 mL), giving 3·0 g (75%) of (2*a*), m.p. 460–462 K. Colorless crystal, 0·70 × 0·30 × 0·35 mm. Automated four-circle Syntex $P2_1$ diffractometer, scan range $4° < 2\theta < 50°$, graphite-monochromated Mo $K\alpha$ radiation, θ–2θ scan technique. 4157 independent reflections, 1934 observed with $I > 3\sigma(I)$. Lp correction but no absorption. Three check reflections collected every 100 data points showed no crystal decay. Direct methods (*MULTAN*80, Main *et al.*, 1980). Positional and anisotropic thermal parameters for all non-H atoms were refined (on *F*) by block-diagonal least squares, H's in calculated positions. $R = 0{\cdot}053$, $R_w = 0{\cdot}060$, $S = 2{\cdot}1$. Weighting scheme of Corfield, Doedens & Ibers (1967) with $p = 0{\cdot}04$. $(\Delta/\sigma)_{\max} = 0{\cdot}3$; maximum height on final difference Fourier map 0·2 e Å^{-3}. Scattering-factor tables from *International Tables for X-ray Crystallography* (1962). No correction for secondary extinction. Refinement program *NUCLS* (Doedens & Ibers); plotter program *ORTEP* (Johnson, 1965).

Compound (2*b*). (2*a*) (10 g) in 100 mL CH_2Cl_2 was treated with 10 mL SO_2Cl_2 in one portion at 298 K. After 3h, the solvent was removed on a rotary evaporator and the residue was heated with 100 mL absolute ethanol on a steam bath; on cooling, 7·7 g

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(71%) of (2*b*), m.p. 484·5–487 K, separated. Chromatography on silica gel, eluting with 2% ethanol in chloroform, gave (2*b*) of m.p. 492–494 K. A crystal of this material, isomorphous with (2*a*), was used for a limited X-ray study to locate the Cl (see *Discussion*).

Discussion. Atomic parameters for (2*a*) are given in Table 1.* A projection of one of the enantiomers of (2*a*) is shown in Fig. 1 and a packing diagram is shown in Fig. 2. Proposed structure (2*a*) is verified with these results.

The five-membered ring is nearly planar: N(2)–C(9)–C(10)–N(1) = –6·7, C(9)–C(10)–N(1)–C(11) = 6·1, C(10)–N(1)–C(11)–N(2) = –2·9, N(1)–C(11)–N(2)–C(9) = –1·9, and C(11)–N(2)–C(9)–C(10) = 5·3°. All of the atoms in the dihydro-γ-pyrone ring are approximately coplanar except for C(9): O(1)–C(1)–C(6)–C(7) = 3·5, C(1)–C(6)–C(7)–C(8) = 4·0, C(6)–C(7)–C(8)–C(9) = –30·9, C(7)–C(8)–C(9)–O(1) = 51·1, C(8)–C(9)–O(1)–C(1) = –45·3, and C(9)–O(1)–C(1)–C(6) = 18·3°, and the C(10) carbonyl group is pseudoaxial on this ring. The cyclohexane rings are slightly flattened chairs as expected (Bucourt, 1974), with torsion angles ranging from 54·3 to 57·9° [C(12) to C(17) ring; ave. 55·9°] and 52·3 to 58·5° [C(18) to C(23) ring; ave. 55·4°].

As expected, both large substituents on cyclohexane rings are equatorial. The C(18) to C(23) ring is rotated so that H(18)–C(18)–N(2)–C(9) = 1, H(12)–C(12)–N(1)–C(11) = –26°.

Table 1. *Atomic coordinates* (× 10^4) *and isotropic temperature factors* (Å^2) *for* (2*a*)

	x	*y*	*z*	*B**
O(1)	5860 (2)	2886 (4)	8365 (1)	3·7 (2)
O(2)	6207 (3)	–1402 (5)	7462 (1)	6·0 (2)
O(3)	7452 (2)	–902 (4)	8565 (1)	3·7 (2)
O(4)	3963 (3)	415 (5)	9434 (1)	5·4 (2)
N(1)	5942 (3)	–353 (5)	9100 (1)	4·0 (2)
N(2)	4288 (3)	1120 (5)	8737 (1)	4·0 (2)
C(1)	6784 (3)	3098 (6)	8046 (1)	3·3 (2)
C(2)	7507 (4)	4860 (6)	8050 (1)	6·0 (3)
C(3)	8432 (4)	5172 (8)	7739 (2)	6·8 (4)
C(4)	8663 (4)	3761 (10)	7437 (2)	5·7 (3)
C(5)	7931 (4)	2018 (7)	7436 (1)	4·6 (3)
C(6)	6960 (3)	1674 (6)	7739 (1)	3·0 (2)
C(7)	6102 (3)	–110 (6)	7724 (1)	3·7 (2)
C(8)	4993 (3)	–166 (6)	8045 (1)	3·3 (2)
C(9)	5367 (3)	903 (6)	8445 (1)	3·0 (2)
C(10)	6415 (3)	–242 (6)	8700 (1)	3·1 (2)
C(11)	4626 (3)	418 (7)	9127 (1)	4·6 (3)
C(12)	6554 (4)	–1471 (7)	9459 (1)	3·7 (2)
C(13)	8027 (4)	–1047 (7)	9512 (1)	4·6 (3)
C(14)	8561 (4)	–2175 (8)	9898 (1)	4·7 (3)
C(15)	8275 (4)	–4380 (8)	9872 (1)	5·6 (3)
C(16)	6796 (4)	–4796 (7)	9803 (1)	5·7 (3)
C(17)	6259 (4)	–3667 (7)	9418 (1)	5·1 (3)
C(18)	3003 (3)	2169 (6)	8637 (1)	3·1 (2)
C(19)	1836 (4)	731 (6)	8672 (1)	4·6 (3)
C(20)	520 (4)	1789 (8)	8563 (1)	5·9 (3)
C(21)	297 (4)	3547 (9)	8841 (1)	6·1 (3)
C(22)	1446 (5)	5011 (7)	8806 (1)	6·0 (3)
C(23)	2805 (4)	4037 (7)	8901 (1)	4·7 (3)

* From last isotropic refinement.

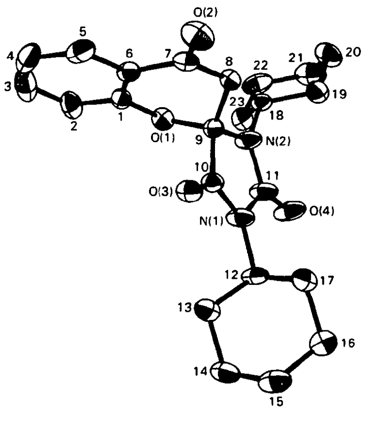

Fig. 1. (2*a*), with 50% probability ellipsoids.

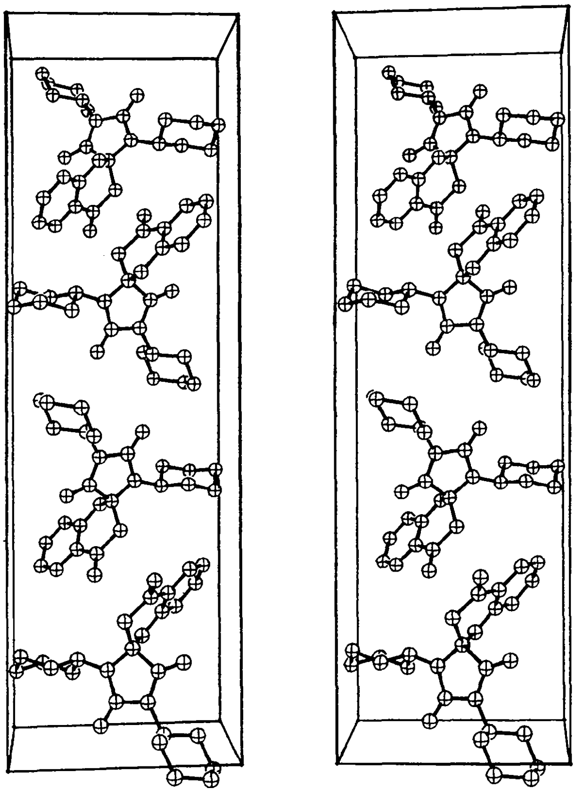

Fig. 2. Stereoscopic illustration of a unit cell of (2*a*); **a** horizontal, **c** vertical.

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* Lists of bond lengths and angles, anisotropic thermal parameters and H-atom coordinates for (2*a*) and structure factors for (2*a*) and (2*b*) have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38959 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

An analogous compound (2*b*) containing a Cl atom was obtained by chlorinating (2*a*). (2*b*) gave smaller crystals than (2*a*), but as it appeared to be isomorphous with (2*a*) ($P2_1/c$, $a = 10{\cdot}38$, $b = 6{\cdot}72$, $c = 31{\cdot}96$ Å, $\beta = 90{\cdot}2°$), an attempt was made to collect a data set in order to locate the Cl atom. Refinement of Cl coordinates and temperature factor using the 135 largest observed structure factors for (2*b*) and structure factors calculated from the atomic coordinates of (2*a*) (isotropic temperature factors, H atoms included) and a calculated pseudoaxial Cl gave increasingly large Cl temperature factors. However, using a calculated pseudoequatorial Cl [configuration depicted in (2*b*)] gave convergence at $x = 0{\cdot}464$ (4), $y = -0{\cdot}246$ (7), $z = 0{\cdot}822$ (1) Å, and $B = 5$ (1) Å^2, with $R = 0{\cdot}118$, clearly indicating the Cl position.

We wish to thank the University of Arizona Computer Center for computer time.

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Structure d'Agents Mutagènes. III. Le Méthoxy-8 Nitro-2 Naphto[2,3-*b*]furanne, $C_{13}H_9NO_4$

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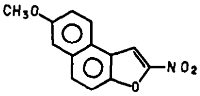
(*Reçu le* 25 *avril* 1983, *accepté le* 13 *octobre* 1983)

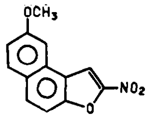
Abstract. $M_r = 243{\cdot}2$, triclinic, $P\bar{1}$, $a = 7{\cdot}175$ (1), $b = 7{\cdot}411$ (1), $c = 11{\cdot}340$ (2) Å, $\alpha = 89{\cdot}10$ (1), $\beta = 107{\cdot}58$ (1), $\gamma = 108{\cdot}63$ (1)°, $V = 542{\cdot}6$ Å^3, $Z = 2$, $D_m = 1{\cdot}48$, $D_x = 1{\cdot}489$ g cm^{-3}, Cu $K\bar{\alpha}$, $\lambda = 1{\cdot}5418$ Å, $\mu = 9{\cdot}5$ cm^{-1}, $F(000) = 252$, room temperature, final $R = 0{\cdot}047$ for 1435 observed reflections. The title compound is an isomer of the 7-methoxy analog (R7000), the most effective mutagenic agent; geometrical parameters are very similar in the two compounds. Molecules are stacked in columns with a quasi-regular spacing of 3·35 Å. Cohesion between columns results in CH···O interactions.

Introduction. Dans la série des naphtofurannes de synthèse (Royer & Buisson, 1980; Cavier, Buisson, Lemoine & Royer, 1981) le méthoxy-7 nitro-2 naphto-[2,1-*b*]furanne (R7000), dont la structure cristalline a été déterminée récemment (Bravic, Bideau & Courseille, 1982), semble être un agent mutagène particulièrement efficace (Weill-Thévenet, Buisson, Royer & Hofnung, 1981).

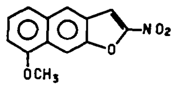
Dans le but d'établir des relations structure–activité nous avons entrepris l'étude structurale d'une série de molécules homologues du R7000, possédant un noyau naphtofuranne et qui présentent par ailleurs des propriétés antibactériennes et protozoocides remarquables. Nous présentons ici la structure cristalline d'un isomère du R7000, le R7105 que nous comparerons également avec un autre isomère, le R6998 (Bideau, Bravic, Cotrait & Courseille, 1983).



R 7000



R 6998



R 7105

Partie expérimentale. Prismes de couleur orangée (solution benzène + acétone). Monocristal 0,3 × 0,4 × 0,5 mm, $\mu R = 0{,}42$. D_m par flottation. Diffractomètre Enraf–Nonius CAD-4, monochromateur en graphite. Optimisation des paramètres cristallins par minimisation des angles d'Euler de 12 réflexions. Balayage ω–2θ, $\Delta\theta = (0{,}8 + 0{,}14\mathrm{tg}\theta)°$, ouverture du détecteur: $(1{,}3 + 1{,}0\mathrm{tg}\theta)$ mm, $2\theta < 120°$, $\frac{1}{2}$ Ewald sphère. Correction Lp, absorption négligée. Pas de décroissance des intensités de références (040, 320, 006) avec le temps. 1604 réflexions mesurées dont 1435 avec $I > 3\sigma(I)$. Méthodes directes, programme *MULTAN*78 (Main,

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