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Stereochemistry of a Hetero-Diels–Alder Product

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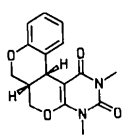
Abstract. Racemic *cis*-2,4-dimethyl-1,2,3,4,6a,12b-hexahydro-6*H*,7*H*-2,4-diazabenzo[*b*]pyrano[3,4-*c*]-benzo[*b*]pyran-1,3-dione, $C_{16}H_{16}N_2O_4$, $M_r = 300{\cdot}3$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}779$ (6), $b = 9{\cdot}332$ (3), $c = 19{\cdot}627$ (9) Å, $V = 1425$ Å^3, $Z = 4$, $D_x = 1{\cdot}400$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}095$ mm^{-1}, $F(000) = 632$, $T = 298$ K, $R = 0{\cdot}045$ for 2184 unique reflections. The title compound is the main product of an intramolecular Diels–Alder reaction. The structure was investigated in order to determine whether the ring junction is *cis* or *trans*. The sample was racemic; although the space group is non-centrosymmetric, the absolute structure was not determined. The ring conformations are as expected for six-membered rings.

Experimental. (I): crystal 0·5 × 0·4 × 0·3 mm. Stoe–Siemens four-circle diffractometer, monochromated Mo $K\alpha$ radiation, profile-fitting mode involving variable scan width and speed (Clegg, 1981). 3050 reflections measured, $2\theta_{max}$ 50°, $+h+k+l$, $-h+k+l$ and some $-h-k-l$, three check reflections with no intensity change. 2500 unique data ($R_{int} = 0{\cdot}028$), of which 2184 with $F > 3\sigma(F)$ (Friedel opposites not merged) used for all calculations (program system *SHELXTL*, Sheldrick, 1978). Index ranges $|h| \le 11$, $|k| \le 15$, $|l| \le 23$. Cell constants refined from $\pm 2\theta$ values of 32 reflections in range 20–25°. Absorption and extinction corrections not necessary.



(I)

Structure solution by multisolution direct methods. Refinement on F to $R = 0{\cdot}054$, $wR = 0{\cdot}055$; all non-H atoms anisotropic, H atoms included using a riding model [C—H 0·96 Å, $U(H) = 1{\cdot}2U_{eq}(C)$], 205 parameters, $S = 1{\cdot}15$, weighting scheme $w^{-1} = \sigma^2(F) + 0{\cdot}00112F^2$ which gave a featureless analysis of variance in terms of F_o and $\sin\theta$, max. $\Delta/\sigma = 0{\cdot}04$, max. and min. height in final $\Delta\rho$ map 0·23 and −0·30 e Å^{-3} respectively. Atomic scattering factors from *SHELXTL*.

Atomic parameters are given in Table 1, bond distances and angles in Table 2.* Fig. 1 shows the atom numbering.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43055 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic thermal parameters* (Å^2 × 10^3)

	x	y	z	U_{eq}*
C(1)	9025 (3)	299 (2)	3316 (1)	44 (1)
N(2)	9608 (3)	670 (2)	3969 (1)	49 (1)
C(3)	11173 (4)	1274 (3)	4108 (1)	54 (1)
N(4)	12241 (3)	1519 (2)	3554 (1)	52 (1)
C(4a)	11737 (3)	1142 (2)	2908 (1)	45 (1)
O(5)	13001 (2)	1341 (2)	2455 (1)	59 (1)
C(6)	12586 (3)	1097 (3)	1745 (1)	61 (1)
C(6a)	11329 (3)	−123 (3)	1648 (1)	55 (1)
C(7)	10915 (4)	−298 (3)	900 (1)	70 (1)
O(8)	10190 (3)	965 (2)	615 (1)	72 (1)
C(8a)	8879 (4)	1587 (3)	985 (1)	54 (1)
C(9)	7876 (4)	2590 (3)	644 (1)	67 (1)
C(10)	6579 (4)	3279 (3)	974 (2)	67 (1)
C(11)	6248 (3)	2996 (3)	1649 (2)	59 (1)
C(12)	7241 (3)	1992 (3)	1987 (1)	49 (1)
C(12a)	8578 (3)	1266 (2)	1671 (1)	44 (1)
C(12b)	9683 (3)	177 (2)	2047 (1)	44 (1)
C(12c)	10160 (3)	614 (2)	2766 (1)	41 (1)
O(13)	7612 (2)	−261 (2)	3250 (1)	61 (1)
C(14)	8498 (4)	358 (4)	4557 (1)	71 (1)
O(15)	11612 (3)	1603 (2)	4684 (1)	74 (1)
C(16)	13947 (4)	2160 (4)	3669 (2)	80 (1)

* U_{eq} defined as one third of the trace of the orthogonalized U_{ij} tensor.

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

C(1)–N(2)	1·402 (3)	C(1)–C(12c)	1·426 (3)
C(1)–O(13)	1·224 (3)	N(2)–C(3)	1·369 (3)
N(2)–C(14)	1·470 (3)	C(3)–N(4)	1·387 (3)
C(3)–O(15)	1·221 (3)	N(4)–C(4a)	1·373 (3)
N(4)–C(16)	1·474 (4)	C(4a)–O(5)	1·338 (3)
C(4a)–C(12c)	1·351 (3)	O(5)–C(6)	1·448 (3)
C(6)–C(6a)	1·513 (4)	C(6a)–C(7)	1·512 (4)
C(6a)–C(12b)	1·527 (3)	C(7)–O(8)	1·421 (3)
O(8)–C(8a)	1·380 (3)	C(8a)–C(9)	1·389 (4)
C(8a)–C(12a)	1·400 (3)	C(9)–C(10)	1·360 (4)
C(10)–C(11)	1·375 (4)	C(11)–C(12)	1·385 (4)
C(12)–C(12a)	1·388 (3)	C(12a)–C(12b)	1·521 (4)
C(12b)–C(12c)	1·516 (3)		

N(2)–C(1)–C(12c)	116·2 (2)	N(2)–C(1)–O(13)	119·5 (2)
C(12c)–C(1)–O(13)	124·3 (2)	C(1)–N(2)–C(3)	124·8 (2)
C(1)–N(2)–C(14)	118·6 (2)	C(3)–N(2)–C(14)	116·6 (2)
N(2)–C(3)–N(4)	116·4 (2)	N(2)–C(3)–O(15)	122·5 (2)
N(4)–C(3)–O(15)	121·1 (2)	C(3)–N(4)–C(4a)	120·7 (2)
C(3)–N(4)–C(16)	119·1 (2)	C(4a)–N(4)–C(16)	120·2 (2)
N(4)–C(4a)–O(5)	111·6 (2)	N(4)–C(4a)–C(12c)	122·9 (2)
O(5)–C(4a)–C(12c)	125·5 (2)	C(4a)–O(5)–C(6)	117·0 (2)
O(5)–C(6)–C(6a)	112·6 (2)	C(6)–C(6a)–C(7)	109·9 (2)
C(6)–C(6a)–C(12b)	109·9 (2)	C(7)–C(6a)–C(12b)	109·8 (2)
C(6a)–C(7)–O(8)	112·2 (2)	C(7)–O(8)–C(8a)	115·8 (2)
O(8)–C(8a)–C(9)	116·4 (2)	O(8)–C(8a)–C(12a)	122·7 (2)
C(9)–C(8a)–C(12a)	120·9 (2)	C(8a)–C(9)–C(10)	120·4 (2)
C(9)–C(10)–C(11)	120·4 (3)	C(10)–C(11)–C(12)	119·2 (2)
C(11)–C(12)–C(12a)	122·3 (2)	C(8a)–C(12a)–C(12)	116·8 (2)
C(8a)–C(12a)–C(12b)	121·0 (2)	C(12)–C(12a)–C(12b)	122·2 (2)
C(6a)–C(12b)–C(12a)	110·4 (2)	C(6a)–C(12b)–C(12c)	108·7 (2)
C(12a)–C(12b)–C(12c)	114·2 (2)	C(1)–C(12c)–C(4a)	118·8 (2)
C(1)–C(12c)–C(12b)	119·9 (2)	C(42)–C(12c)–C(12b)	120·8 (2)

Related literature. For the preparation of the compound *via* an intramolecular hetero-Diels–Alder reaction see Tietze, Stegelmeir, Harms & Brumby (1982). For structures of related *trans*-fused Diels–Alder products see Tietze, von Kiedrowski, Harms, Clegg & Sheldrick (1980), Harms, Jones & Sheldrick (1980) and Clegg, Harms, Sheldrick, von Kiedrowski & Tietze (1980).

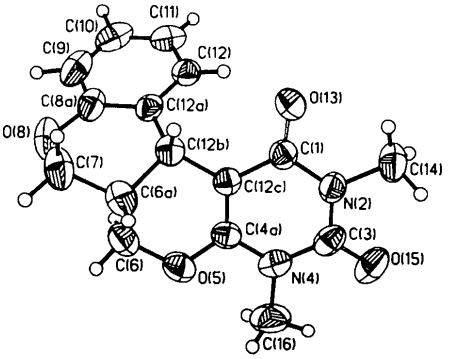

Fig. 1. The asymmetric unit of the title compound, showing the atom-numbering scheme.

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Structure of an Indolochinolizine Derivative

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Abstract. Racemic (2*S*,3*S*,12b*R*)-2-(1,3-dimethyl-2,4,6-trioxo-1,3-diazacyclohexyl)-3-ethyl-1,2,3,4,5,6-hexahydro-12b*H*-indolo[2,3-*a*]quinolizinium chloride, $C_{23}H_{29}N_4O_3^+.Cl^-$, $M_r = 445{\cdot}0$, orthorhombic, *Pbca*, $a = 14{\cdot}462\ (2)$, $b = 17{\cdot}405\ (1)$, $c = 17{\cdot}299\ (2)$ Å, $V = 4354$ Å^3, $Z = 8$, $D_x = 1{\cdot}357$ Mg m^{-3}, λ(Mo $K\alpha$) =

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