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(4′*S*)-4′-Ethyl-3′,10′-dioxo-3′,4′,7′,8′-tetrahydrospiro[1,3-dioxolane-2,6′-1′*H*,6′*H*-pyrano[3,4-*f*]indolizin]-4′-yl *N*-(1-phenylethyl)carbamate

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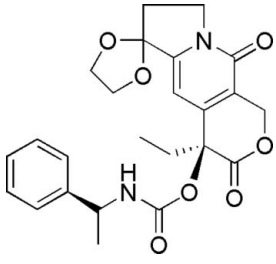
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.034; wR factor = 0.087; data-to-parameter ratio = 8.6.

The title compound, $C_{24}H_{26}N_2O_7$, is an intermediate in the resolution of a tertiary alcohol using a chiral isocyanate reagent. On the basis of the unchanging *S* configuration of the chiral C atom introduced by the isocyanate reagent, the chiral centre on the tricyclo ring system is shown to adopt an *S* configuration. In the crystal structure, N—H···O hydrogen bonds link the molecules into chains.

Related literature

For related literature, see: Duggan & Imagire (1989); Gibbs *et al.* (1989).



Experimental

Crystal data

$C_{24}H_{26}N_2O_7$
M_r = 454.47
Monoclinic, $P2_1$
a = 7.3998 (10) Å
b = 16.383 (2) Å
c = 9.2162 (13) Å
β = 92.322 (2)°
V = 1116.4 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 296 (2) K
0.30 × 0.25 × 0.20 mm

Data collection

Bruker SMART APEX II CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.971, T_{max} = 0.980
7148 measured reflections
2612 independent reflections
2352 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.087
S = 1.06
2612 reflections
302 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O6^{i}	0.88	2.11	2.988 (2)	173

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BI2220).

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supplementary materials

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(4'*S*)-4'-Ethyl-3',10'-dioxo-3',4',7',8'-tetrahydrospiro[1,3-dioxolane-2,6'-1'*H*,6'*H*-pyrano[3,4-*f*]indolizin]-4'-yl *N*-(1-phenylethyl)carbamate

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Comment

The method for resolution of a tertiary alcohol using a chiral isocyanate reagent has been widely applied (Gibbs *et al.*, 1989). The title compound is a key intermediate in the resolution process of alcohol **1** using the (*S*)-isocyanate **2** (Scheme 2).

On the basis of the unchanging *S* configuration of C17, the molecular structure (Figure 1) indicates that the chiral C atom C3 adopts an *S* configuration. The six-membered lactone ring is twisted with an O1/C2/C3/C4 torsion angle of −21.4 (3) °. The pyrrole ring is also twisted, with a N1/C6/C7/C8 torsion angle of 22.3 (2) °. N—H···O hydrogen bonds exist between molecules, linking them into chains along the *c* axis.

Experimental

The (S)-isocyanate **2** (2.0 mmol) was added to a mixture of alcohol **1** (1.0 mmol) and reagent grade CuCl (0.2 mmol) in dry CH_2Cl_2 (12 ml) at room temperature (Scheme 2; Duggan & Imagire, 1989). After stirring for 12 h, the reaction mixture was filtered and the filtrate was diluted with CH_2Cl_2 (20 ml), washed with H_2O (20 ml) and brine (20 ml), dried over $MgSO_4$ and concentrated. The crude product (0.43 g, 94%) was recrystallized from 2-propanol (31 ml) to yield a white powder (m.p. 485 K). Crystals suitable for X-ray analysis were obtained from ethyl acetate.

Refinement

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.98 Å and N—H = 0.88 Å, and with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C) for other H atoms. The methyl groups were allowed to rotate about their local threefold axes. In the absence of significant anomalous scattering effects, Friedel pairs have been merged as equivalent data. The absolute structure was assigned on the basis of the unchanging chiral centre C17, originating from the isocyanate reactant.

Figures

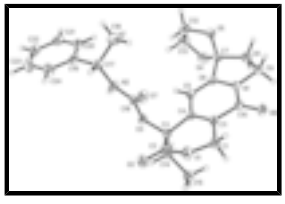

Fig. 1. The molecular structure of the title compound showing displacement ellipsoids at 30% probability for non-H atoms.

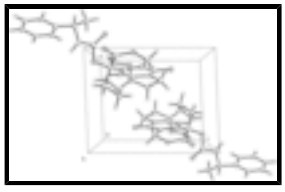

Fig. 2. Unit-cell contents viewed along the *b* axis.

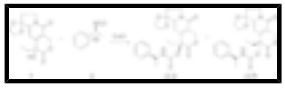

Fig. 3. The formation of the title compound.

(4'*S*)-4'-Ethyl-3',10'-dioxo-3',4',7',8'-tetrahydrospiro[1,3-dioxolane-2,6'- 1'*H*,6'*H*-pyrano[3,4-*f*]indolizin]-4'-yl *N*-(1-phenylethyl)carbamate

Crystal data

$C_{24}H_{26}N_2O_7$	F_{000} = 480
M_r = 454.47	D_x = 1.352 Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2yb	Cell parameters from 2722 reflections
a = 7.3998 (10) Å	θ = 2.2–25.4º
b = 16.383 (2) Å	μ = 0.10 mm^{-1}
c = 9.2162 (13) Å	T = 296 (2) K
β = 92.322 (2)º	Prism, colourless
V = 1116.4 (3) Å^3	0.30 × 0.25 × 0.20 mm
Z = 2	

Data collection

Bruker SMART APEX II CCD diffractometer	2612 independent reflections
Radiation source: fine-focus sealed tube	2352 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.023
T = 296(2) K	θ_{max} = 27.5º
φ and ω scans	θ_{min} = 2.2º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −8→9
T_{min} = 0.971, T_{max} = 0.980	k = −18→21
7148 measured reflections	l = −9→11

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0485P)^2 + 0.0587P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.034	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.087	$\Delta\rho_{max}$ = 0.21 e Å^{-3}
S = 1.06	$\Delta\rho_{min}$ = −0.13 e Å^{-3}

2612 reflections	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
302 parameters	Extinction coefficient: 0.015 (3)
1 restraint	Absolute structure: Friedel pairs merged as equivalent data
Primary atom site location: structure-invariant direct methods	Flack parameter: ?
Secondary atom site location: difference Fourier map	Rogers parameter: ?
Hydrogen site location: inferred from neighbouring sites	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.9756 (3)	0.86809 (10)	0.37052 (17)	0.0497 (4)
O2	1.0016 (3)	0.90832 (11)	0.14679 (19)	0.0571 (5)
O3	0.8961 (2)	0.75590 (10)	0.03780 (15)	0.0361 (3)
O4	0.6197 (2)	0.46524 (11)	0.2027 (2)	0.0518 (4)
O5	0.8933 (2)	0.44686 (10)	0.31575 (17)	0.0438 (4)
O6	0.8228 (2)	0.68765 (11)	0.66980 (16)	0.0445 (4)
O7	1.1799 (2)	0.74245 (12)	0.13801 (17)	0.0483 (4)
N1	0.7652 (2)	0.59843 (11)	0.48365 (19)	0.0342 (4)
N2	1.1043 (3)	0.69448 (13)	−0.0887 (2)	0.0429 (5)
H2	1.0138	0.6919	−0.1538	0.043 (7)*
C1	0.9037 (3)	0.81619 (15)	0.4800 (2)	0.0415 (5)
H1A	0.8013	0.8434	0.5212	0.050*
H1B	0.9953	0.8090	0.5573	0.050*
C2	0.9466 (3)	0.85775 (14)	0.2281 (2)	0.0395 (5)
C3	0.8317 (3)	0.78525 (13)	0.1735 (2)	0.0337 (4)
C4	0.8214 (3)	0.71695 (12)	0.2830 (2)	0.0314 (4)
C5	0.7784 (3)	0.63660 (14)	0.2365 (2)	0.0343 (4)
H5	0.7661	0.6238	0.1383	0.041*
C6	0.7561 (3)	0.57932 (13)	0.3391 (2)	0.0331 (4)
C7	0.7254 (3)	0.48743 (13)	0.3259 (3)	0.0382 (5)
C8	0.6437 (3)	0.46579 (16)	0.4706 (3)	0.0479 (6)
H8A	0.5127	0.4682	0.4625	0.057*
H8B	0.6798	0.4113	0.5012	0.057*

C9	0.7175 (3)	0.52940 (15)	0.5779 (3)	0.0442 (6)
H9A	0.6265	0.5453	0.6454	0.053*
H9B	0.8230	0.5090	0.6323	0.053*
C10	0.8130 (3)	0.67425 (14)	0.5379 (2)	0.0342 (4)
C11	0.8446 (3)	0.73440 (13)	0.4266 (2)	0.0326 (4)
C12	0.9214 (4)	0.43939 (19)	0.1647 (3)	0.0581 (7)
H12A	0.9934	0.3915	0.1447	0.070*
H12B	0.9815	0.4873	0.1281	0.070*
C13	0.7343 (5)	0.4315 (2)	0.0987 (3)	0.0685 (9)
H13A	0.7230	0.4613	0.0078	0.082*
H13B	0.7046	0.3746	0.0804	0.082*
C14	0.6397 (3)	0.81741 (16)	0.1294 (2)	0.0432 (5)
H14A	0.6488	0.8518	0.0444	0.052*
H14B	0.5640	0.7712	0.1018	0.052*
C15	0.5467 (3)	0.86541 (17)	0.2454 (3)	0.0510 (6)
H15A	0.6108	0.9155	0.2638	0.076*
H15B	0.5456	0.8338	0.3330	0.076*
H15C	0.4247	0.8773	0.2127	0.076*
C16	1.0725 (3)	0.73094 (14)	0.0376 (2)	0.0357 (5)
C17	1.2862 (3)	0.66796 (18)	−0.1258 (3)	0.0493 (6)
H17	1.3694	0.7138	−0.1083	0.059*
C18	1.3521 (6)	0.5967 (3)	−0.0323 (3)	0.0880 (13)
H18A	1.2645	0.5535	−0.0383	0.132*
H18B	1.3680	0.6143	0.0668	0.132*
H18C	1.4653	0.5774	−0.0662	0.132*
C19	1.2817 (3)	0.64878 (15)	−0.2861 (2)	0.0378 (5)
C20	1.2066 (3)	0.57611 (15)	−0.3382 (2)	0.0424 (5)
H20	1.1624	0.5383	−0.2732	0.051*
C21	1.1968 (4)	0.55937 (18)	−0.4847 (3)	0.0517 (7)
H21	1.1485	0.5100	−0.5175	0.062*
C22	1.2572 (4)	0.6144 (2)	−0.5813 (3)	0.0611 (8)
H22	1.2488	0.6030	−0.6803	0.073*
C23	1.3300 (4)	0.6861 (2)	−0.5336 (3)	0.0696 (9)
H23	1.3713	0.7237	−0.6003	0.084*
C24	1.3436 (4)	0.70394 (18)	−0.3862 (3)	0.0558 (7)
H24	1.3945	0.7531	−0.3547	0.067*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0722 (12)	0.0424 (9)	0.0346 (8)	−0.0192 (9)	0.0029 (8)	−0.0057 (7)
O2	0.0855 (14)	0.0408 (9)	0.0462 (10)	−0.0161 (10)	0.0177 (9)	−0.0003 (8)
O3	0.0421 (8)	0.0407 (8)	0.0254 (7)	0.0030 (7)	0.0020 (6)	−0.0044 (6)
O4	0.0513 (9)	0.0465 (10)	0.0567 (11)	−0.0033 (8)	−0.0082 (8)	−0.0087 (8)
O5	0.0487 (9)	0.0429 (9)	0.0396 (8)	0.0118 (7)	0.0007 (7)	−0.0048 (7)
O6	0.0530 (9)	0.0532 (10)	0.0274 (8)	0.0067 (8)	−0.0001 (7)	−0.0026 (7)
O7	0.0462 (9)	0.0642 (12)	0.0339 (8)	0.0019 (8)	−0.0043 (7)	−0.0097 (8)
N1	0.0365 (9)	0.0356 (10)	0.0306 (9)	0.0042 (8)	0.0044 (7)	0.0025 (7)

N2	0.0400 (10)	0.0571 (13)	0.0313 (9)	0.0084 (9)	−0.0022 (8)	−0.0104 (9)
C1	0.0527 (13)	0.0394 (12)	0.0324 (11)	−0.0061 (11)	0.0010 (10)	−0.0039 (10)
C2	0.0478 (12)	0.0348 (12)	0.0363 (12)	−0.0035 (10)	0.0083 (10)	−0.0043 (10)
C3	0.0405 (11)	0.0331 (10)	0.0274 (10)	0.0012 (9)	0.0020 (8)	−0.0041 (8)
C4	0.0326 (10)	0.0323 (10)	0.0295 (10)	0.0019 (8)	0.0021 (8)	−0.0033 (8)
C5	0.0413 (11)	0.0341 (11)	0.0274 (10)	−0.0003 (9)	−0.0010 (8)	−0.0046 (8)
C6	0.0326 (10)	0.0322 (11)	0.0344 (11)	0.0026 (8)	0.0008 (8)	−0.0044 (9)
C7	0.0394 (11)	0.0310 (11)	0.0441 (13)	0.0014 (9)	0.0010 (9)	0.0002 (9)
C8	0.0508 (13)	0.0394 (13)	0.0545 (15)	0.0010 (11)	0.0133 (11)	0.0048 (11)
C9	0.0528 (14)	0.0399 (12)	0.0408 (13)	0.0058 (11)	0.0130 (10)	0.0095 (10)
C10	0.0339 (10)	0.0393 (11)	0.0295 (10)	0.0065 (9)	0.0019 (8)	−0.0020 (9)
C11	0.0343 (10)	0.0357 (11)	0.0278 (10)	0.0018 (9)	0.0016 (8)	−0.0045 (8)
C12	0.0686 (18)	0.0605 (17)	0.0456 (14)	0.0120 (14)	0.0076 (13)	−0.0118 (13)
C13	0.087 (2)	0.0624 (19)	0.0550 (16)	0.0104 (16)	−0.0080 (15)	−0.0230 (15)
C14	0.0458 (12)	0.0446 (13)	0.0390 (12)	0.0085 (10)	0.0000 (10)	−0.0004 (10)
C15	0.0477 (13)	0.0490 (14)	0.0567 (15)	0.0068 (12)	0.0083 (11)	−0.0009 (12)
C16	0.0439 (11)	0.0357 (11)	0.0276 (10)	0.0011 (9)	0.0021 (9)	−0.0009 (9)
C17	0.0441 (13)	0.0624 (17)	0.0411 (13)	0.0119 (11)	−0.0027 (10)	−0.0115 (12)
C18	0.110 (3)	0.115 (3)	0.0382 (15)	0.065 (3)	−0.0092 (16)	−0.0031 (17)
C19	0.0324 (10)	0.0446 (13)	0.0365 (12)	0.0058 (9)	0.0046 (8)	−0.0024 (10)
C20	0.0445 (12)	0.0438 (13)	0.0390 (12)	0.0037 (10)	0.0025 (10)	0.0011 (10)
C21	0.0482 (14)	0.0552 (15)	0.0512 (15)	0.0099 (12)	−0.0054 (11)	−0.0172 (12)
C22	0.0525 (15)	0.092 (2)	0.0383 (14)	0.0138 (15)	0.0034 (12)	−0.0036 (14)
C23	0.0656 (18)	0.093 (3)	0.0515 (17)	−0.0036 (18)	0.0146 (14)	0.0258 (17)
C24	0.0545 (15)	0.0497 (15)	0.0633 (18)	−0.0088 (12)	0.0033 (13)	0.0010 (13)

Geometric parameters (Å, °)

O1—C2	1.333 (3)	C9—H9A	0.970
O1—C1	1.438 (3)	C9—H9B	0.970
O2—C2	1.199 (3)	C10—C11	1.448 (3)
O3—C16	1.369 (3)	C12—C13	1.495 (5)
O3—C3	1.439 (2)	C12—H12A	0.970
O4—C7	1.400 (3)	C12—H12B	0.970
O4—C13	1.419 (4)	C13—H13A	0.970
O5—C7	1.416 (3)	C13—H13B	0.970
O5—C12	1.421 (3)	C14—C15	1.515 (3)
O6—C10	1.235 (3)	C14—H14A	0.970
O7—C16	1.210 (3)	C14—H14B	0.970
N1—C6	1.368 (3)	C15—H15A	0.960
N1—C10	1.380 (3)	C15—H15B	0.960
N1—C9	1.478 (3)	C15—H15C	0.960
N2—C16	1.337 (3)	C17—C19	1.510 (3)
N2—C17	1.468 (3)	C17—C18	1.519 (4)
N2—H2	0.88	C17—H17	0.980
C1—C11	1.487 (3)	C18—H18A	0.960
C1—H1A	0.970	C18—H18B	0.960
C1—H1B	0.970	C18—H18C	0.960
C2—C3	1.534 (3)	C19—C24	1.382 (4)

C3—C4	1.511 (3)	C19—C20	1.391 (3)
C3—C14	1.553 (3)	C20—C21	1.377 (3)
C4—C11	1.358 (3)	C20—H20	0.930
C4—C5	1.417 (3)	C21—C22	1.356 (4)
C5—C6	1.347 (3)	C21—H21	0.930
C5—H5	0.930	C22—C23	1.359 (5)
C6—C7	1.527 (3)	C22—H22	0.930
C7—C8	1.528 (3)	C23—C24	1.390 (4)
C8—C9	1.523 (4)	C23—H23	0.930
C8—H8A	0.970	C24—H24	0.930
C8—H8B	0.970		
C2—O1—C1	124.53 (18)	O5—C12—C13	103.7 (2)
C16—O3—C3	116.79 (16)	O5—C12—H12A	111.0
C7—O4—C13	108.6 (2)	C13—C12—H12A	111.0
C7—O5—C12	105.57 (19)	O5—C12—H12B	111.0
C6—N1—C10	124.12 (18)	C13—C12—H12B	111.0
C6—N1—C9	113.22 (18)	H12A—C12—H12B	109.0
C10—N1—C9	122.67 (18)	O4—C13—C12	105.1 (2)
C16—N2—C17	122.03 (19)	O4—C13—H13A	110.7
C16—N2—H2	117.3	C12—C13—H13A	110.7
C17—N2—H2	120.3	O4—C13—H13B	110.7
O1—C1—C11	114.38 (17)	C12—C13—H13B	110.7
O1—C1—H1A	108.7	H13A—C13—H13B	108.8
C11—C1—H1A	108.7	C15—C14—C3	115.36 (19)
O1—C1—H1B	108.7	C15—C14—H14A	108.4
C11—C1—H1B	108.7	C3—C14—H14A	108.4
H1A—C1—H1B	107.6	C15—C14—H14B	108.4
O2—C2—O1	118.8 (2)	C3—C14—H14B	108.4
O2—C2—C3	121.9 (2)	H14A—C14—H14B	107.5
O1—C2—C3	119.10 (18)	C14—C15—H15A	109.5
O3—C3—C4	111.14 (17)	C14—C15—H15B	109.5
O3—C3—C2	110.17 (16)	H15A—C15—H15B	109.5
C4—C3—C2	113.39 (17)	C14—C15—H15C	109.5
O3—C3—C14	102.54 (16)	H15A—C15—H15C	109.5
C4—C3—C14	110.84 (18)	H15B—C15—H15C	109.5
C2—C3—C14	108.18 (18)	O7—C16—N2	127.0 (2)
C11—C4—C5	120.59 (19)	O7—C16—O3	123.32 (19)
C11—C4—C3	119.16 (18)	N2—C16—O3	109.65 (18)
C5—C4—C3	120.17 (18)	N2—C17—C19	107.78 (18)
C6—C5—C4	117.85 (19)	N2—C17—C18	111.8 (3)
C6—C5—H5	121.1	C19—C17—C18	112.9 (2)
C4—C5—H5	121.1	N2—C17—H17	108.1
C5—C6—N1	121.5 (2)	C19—C17—H17	108.1
C5—C6—C7	130.8 (2)	C18—C17—H17	108.1
N1—C6—C7	107.74 (18)	C17—C18—H18A	109.5
O4—C7—O5	106.74 (19)	C17—C18—H18B	109.5
O4—C7—C6	113.41 (19)	H18A—C18—H18B	109.5
O5—C7—C6	109.87 (18)	C17—C18—H18C	109.5
O4—C7—C8	114.8 (2)	H18A—C18—H18C	109.5

O5—C7—C8	109.14 (19)	H18B—C18—H18C	109.5
C6—C7—C8	102.82 (19)	C24—C19—C20	117.9 (2)
C9—C8—C7	105.3 (2)	C24—C19—C17	121.5 (2)
C9—C8—H8A	110.7	C20—C19—C17	120.6 (2)
C7—C8—H8A	110.7	C21—C20—C19	120.9 (2)
C9—C8—H8B	110.7	C21—C20—H20	119.6
C7—C8—H8B	110.7	C19—C20—H20	119.6
H8A—C8—H8B	108.8	C22—C21—C20	120.4 (3)
N1—C9—C8	103.28 (19)	C22—C21—H21	119.8
N1—C9—H9A	111.1	C20—C21—H21	119.8
C8—C9—H9A	111.1	C21—C22—C23	119.9 (3)
N1—C9—H9B	111.1	C21—C22—H22	120.0
C8—C9—H9B	111.1	C23—C22—H22	120.0
H9A—C9—H9B	109.1	C22—C23—C24	120.7 (3)
O6—C10—N1	121.4 (2)	C22—C23—H23	119.6
O6—C10—C11	124.9 (2)	C24—C23—H23	119.6
N1—C10—C11	113.73 (18)	C19—C24—C23	120.1 (3)
C4—C11—C10	121.9 (2)	C19—C24—H24	119.9
C4—C11—C1	122.42 (19)	C23—C24—H24	119.9
C10—C11—C1	115.64 (17)		
C2—O1—C1—C11	19.3 (3)	C6—N1—C9—C8	−9.5 (2)
C1—O1—C2—O2	174.3 (2)	C10—N1—C9—C8	170.22 (18)
C1—O1—C2—C3	−1.0 (3)	C7—C8—C9—N1	23.0 (2)
C16—O3—C3—C4	−68.2 (2)	C6—N1—C10—O6	−178.39 (19)
C16—O3—C3—C2	58.3 (2)	C9—N1—C10—O6	1.9 (3)
C16—O3—C3—C14	173.30 (18)	C6—N1—C10—C11	3.0 (3)
O2—C2—C3—O3	38.1 (3)	C9—N1—C10—C11	−176.68 (18)
O1—C2—C3—O3	−146.7 (2)	C5—C4—C11—C10	−5.0 (3)
O2—C2—C3—C4	163.4 (2)	C3—C4—C11—C10	171.69 (18)
O1—C2—C3—C4	−21.4 (3)	C5—C4—C11—C1	175.1 (2)
O2—C2—C3—C14	−73.2 (3)	C3—C4—C11—C1	−8.2 (3)
O1—C2—C3—C14	102.0 (2)	O6—C10—C11—C4	−176.1 (2)
O3—C3—C4—C11	150.41 (18)	N1—C10—C11—C4	2.5 (3)
C2—C3—C4—C11	25.7 (3)	O6—C10—C11—C1	3.8 (3)
C14—C3—C4—C11	−96.2 (2)	N1—C10—C11—C1	−177.63 (18)
O3—C3—C4—C5	−32.9 (3)	O1—C1—C11—C4	−14.4 (3)
C2—C3—C4—C5	−157.67 (18)	O1—C1—C11—C10	165.72 (18)
C14—C3—C4—C5	80.4 (2)	C7—O5—C12—C13	−31.7 (3)
C11—C4—C5—C6	2.0 (3)	C7—O4—C13—C12	−3.3 (3)
C3—C4—C5—C6	−174.58 (19)	O5—C12—C13—O4	21.5 (3)
C4—C5—C6—N1	3.3 (3)	O3—C3—C14—C15	−170.4 (2)
C4—C5—C6—C7	−174.3 (2)	C4—C3—C14—C15	70.9 (3)
C10—N1—C6—C5	−6.0 (3)	C2—C3—C14—C15	−54.0 (3)
C9—N1—C6—C5	173.7 (2)	C17—N2—C16—O7	−4.6 (4)
C10—N1—C6—C7	172.05 (18)	C17—N2—C16—O3	174.3 (2)
C9—N1—C6—C7	−8.3 (2)	C3—O3—C16—O7	−10.1 (3)
C13—O4—C7—O5	−16.4 (3)	C3—O3—C16—N2	170.89 (18)
C13—O4—C7—C6	104.7 (2)	C16—N2—C17—C19	−166.5 (2)
C13—O4—C7—C8	−137.5 (2)	C16—N2—C17—C18	68.9 (3)

C12—O5—C7—O4	30.5 (3)	N2—C17—C19—C24	99.6 (3)
C12—O5—C7—C6	−92.8 (2)	C18—C17—C19—C24	−136.5 (3)
C12—O5—C7—C8	155.1 (2)	N2—C17—C19—C20	−77.4 (3)
C5—C6—C7—O4	−35.3 (3)	C18—C17—C19—C20	46.5 (3)
N1—C6—C7—O4	146.85 (19)	C24—C19—C20—C21	0.9 (4)
C5—C6—C7—O5	84.0 (3)	C17—C19—C20—C21	177.9 (2)
N1—C6—C7—O5	−93.8 (2)	C19—C20—C21—C22	−1.3 (4)
C5—C6—C7—C8	−159.9 (2)	C20—C21—C22—C23	0.8 (4)
N1—C6—C7—C8	22.3 (2)	C21—C22—C23—C24	0.0 (5)
O4—C7—C8—C9	−151.2 (2)	C20—C19—C24—C23	0.0 (4)
O5—C7—C8—C9	89.0 (2)	C17—C19—C24—C23	−177.1 (3)
C6—C7—C8—C9	−27.6 (2)	C22—C23—C24—C19	−0.4 (5)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O6^{i}	0.88	2.11	2.988 (2)	173

Symmetry codes: (i) *x*, *y*, *z*−1.

Fig. 1

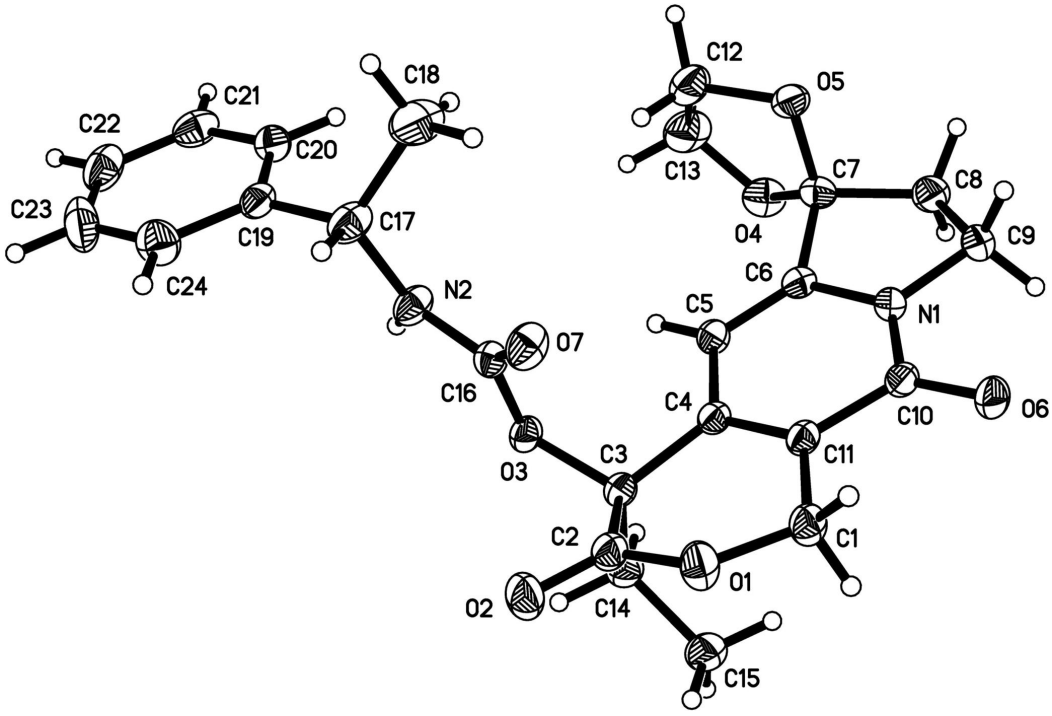
C12
O5
C18
C21
C22
C20
C13
C7
C8
C23
C19
C17
O4
C9
C24
C6
N2
C5
N1
O7
C16
C4
C10
O6
O3
C3
C11
C2
C1
O1
O2
C14
C15

Fig. 2

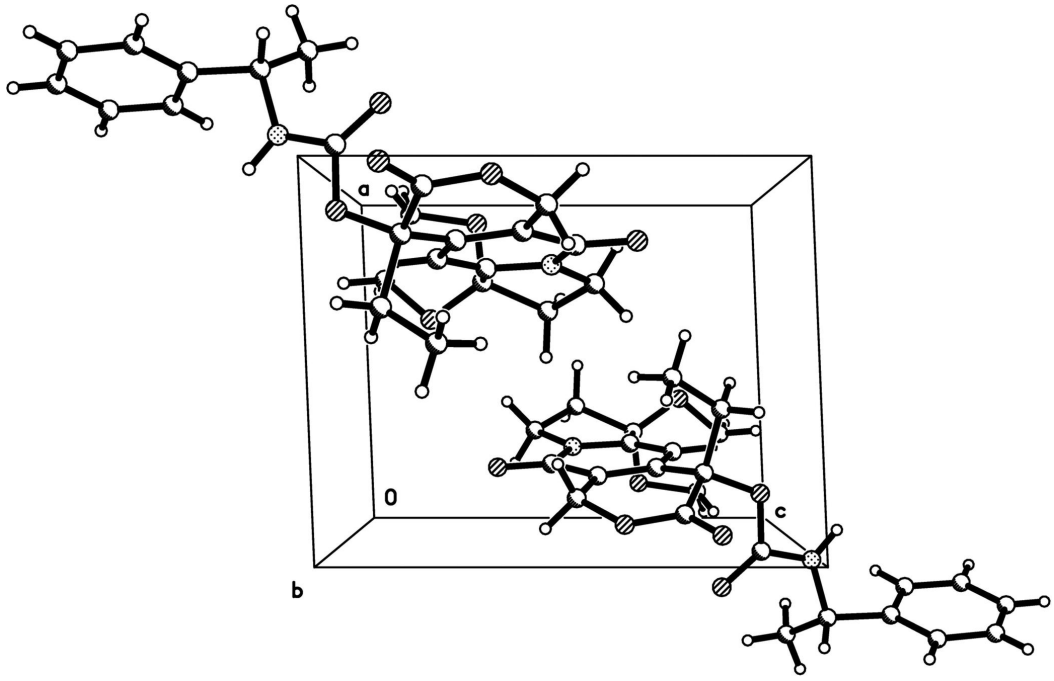

Fig. 3