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(4*R*,5*S*)-Methyl 1,3-dibenzyl-5-[(*R*)-2-(hydroxydiphenylmethyl)pyrrolidine-1-carbonyl]-2-oxoimidazolidine-4-carboxylate

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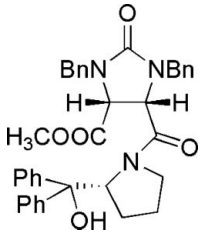
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; R factor = 0.042; wR factor = 0.111; data-to-parameter ratio = 9.0.

The title compound, $C_{37}H_{37}N_3O_5$, was synthesized as a key intermediate in the total synthesis of d-biotin and was crystallized from 2-propanol. It contains one chiral center in the pyrrole ring, where the absolute configuration is known from the synthesis, and from which the two other chiral centers in the imidazolidine ring could be determined. An intramolecular O—H···O and intermolecular C—H···O hydrogen bonds help to construct the three-dimensional network.

Related literature

For related literature, see: Chen *et al.* (2003).



Experimental

Crystal data

$C_{37}H_{37}N_3O_5$
M_r = 603.70
Monoclinic, $P2_1$
a = 12.075 (4) Å
b = 10.907 (4) Å
c = 12.328 (4) Å
β = 101.396 (5)°
V = 1591.5 (10) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 293 (2) K
0.20 × 0.12 × 0.10 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.983, T_{max} = 0.992
8080 measured reflections
3679 independent reflections
2645 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.111
S = 1.02
3679 reflections
411 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.13 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
O5—H5*X*···O4	0.825 (18)	2.00 (2)	2.762 (3)	153 (3)
C4—H4···O1^{i}	0.98	2.57	3.328 (3)	135
C5—H5···O1^{i}	0.98	2.57	3.320 (3)	133
C16—H16···O4^{i}	0.93	2.62	3.383 (4)	139
C17—H17···O5^{i}	0.93	2.80	3.544 (5)	138
C32—H32···O5^{ii}	0.93	2.58	3.435 (5)	153
C37—H37···O1^{iii}	0.93	2.50	3.336 (4)	150

Symmetry codes: (i) $-x, y+\frac{1}{2}, -z$; (ii) $-x+1, y+\frac{1}{2}, -z+1$; (iii) $x, y, z+1$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); 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, 2000); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: EZ2097).

References

Bruker (2000). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chen, F.-E., Yuan, J.-L., Dai, H.-F., Kuang, Y.-Y. & Chu, Y. (2003). *Synthesis*, **14**, 2155–2160.
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supplementary materials

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(4*R*,5*S*)-Methyl 1,3-dibenzyl-5-[(*R*)-2-(hydroxydiphenylmethyl)pyrrolidine-1-carbonyl]-2-oxoimidazolidine-4-carboxylate

J. Liu, F.-E. Chen and M.-Q. Chen

Comment

In our ongoing research on the total synthesis of d-biotin, an efficient and highly diastereoselective approach was developed with (4*R*,5S)-methyl 1,3-dibenzyl-5- ((*R*)-2-(hydroxydiphenylmethyl)pyrrolidine-1-carbonyl)-2- oxoimidazolidine-4-carboxylate(I) prepared as a key intermediate. Determination of the three-dimensional structure would help us to investigate the absolute configurations of the three chiral centers. Herein, we report the crystal structure of the title compound (I).

Fig. 1 shows the molecular structure of (I). Due to the certainty of C24 being *R*-configuration, based on the synthesis, the diffraction analysis indicates that the two other chiral carbon atoms (C4 and C5) have R– and S-configurations respectively. The strong O5—H5X···O4 intramolecular hydrogen bond may enhance the molecular stability of this compound. Intermolecular C—H···O hydrogen bonds lead to a three-dimensional network.

Experimental

To a solution of (4*R*,5S)-1,3-dibenzyl-5-((*R*)-2-(hydroxydiphenylmethyl) pyrrolidine-1-carbonyl)-2- oxoimidazolidine-4-carboxylic acid (8 g, 13.68 mmol) in acetone (100 ml) was added NaOH (1.13 g, 27.36 mmol). After stirring at r.t. for 2 h, Me_2SO_4 (2.57 ml, 27.36 mmol) was added, followed by a catalytic amount of Bu_4NI. Then the reaction mixture was heated under reflux for 10 h. The solvent was evaporated *in vacuo*, 5% aq NH_4Cl (50 ml) was added and extracted with CH_2Cl_2. The combined organic layers were washed with 5% aq $NaHCO_3$ (50 ml), H_2O (50 ml) and brine (50 ml), and dried ($MgSO_4$). Evaporation of the solvent under reduced pressure gave the crude product, which was washed with Et_2O to afford a white powder (6.4 g, 78%), m.p. 444 K. Recrystallization from 2-propanol yielded colorless crystals of the title compound suitable for X-ray diffraction.

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 with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C) for other H atoms. Methyl groups were allowed to rotate freely about the C—C bond.

Figures

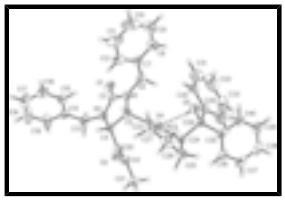


Fig. 1. Molecular structure of the title compound (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme. Hydrogen bonds are indicated by dashed lines.

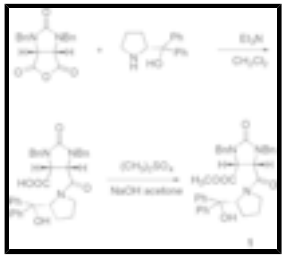


Fig. 2. The formation of the title compound.

(4*R*,5S)-Methyl 1,3-dibenzyl-5-[(*R*)-2-(hydroxydiphenylmethyl)pyrrolidine-1-carbonyl]- 2-oxoimidazolidine-4-carboxylate

Crystal data

$C_{37}H_{37}N_3O_5$	F_{000} = 640
M_r = 603.70	D_x = 1.260 Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2yb	Cell parameters from 793 reflections
a = 12.075 (4) Å	θ = 2.5–21.2º
b = 10.907 (4) Å	μ = 0.08 mm^{-1}
c = 12.328 (4) Å	T = 293 (2) K
β = 101.396 (5)º	Prism, colorless
V = 1591.5 (10) Å^3	0.20 × 0.12 × 0.10 mm
Z = 2	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	3679 independent reflections
Radiation source: fine-focus sealed tube	2645 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.029
T = 293(2) K	θ_{max} = 27.2º
φ and ω scans	θ_{min} = 2.2º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −7→15
T_{min} = 0.983, T_{max} = 0.992	k = −13→13
8080 measured reflections	l = −15→14

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.042$

$wR(F^2) = 0.111$

$S = 1.02$

3679 reflections

411 parameters

2 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0606P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.13$ e Å^{-3}

$\Delta\rho_{min} = -0.13$ e Å^{-3}

Extinction correction: none

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 $F^2 > 2$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	0.01834 (19)	0.4307 (2)	−0.02044 (17)	0.0663 (6)
O2	−0.23633 (19)	0.5563 (3)	0.19449 (19)	0.0850 (7)
O3	−0.16338 (16)	0.7314 (2)	0.27063 (17)	0.0651 (5)
O4	0.03920 (15)	0.54484 (17)	0.32417 (14)	0.0531 (4)
O5	0.24961 (16)	0.56160 (19)	0.46064 (15)	0.0568 (5)
H5X	0.1866 (18)	0.535 (3)	0.432 (2)	0.064 (10)*
N1	0.07821 (18)	0.5786 (2)	0.11061 (18)	0.0528 (5)
C2	0.0000 (2)	0.5168 (3)	0.0374 (2)	0.0494 (6)
N3	−0.10202 (19)	0.5674 (2)	0.03674 (18)	0.0563 (6)
C4	−0.0978 (2)	0.6659 (3)	0.1152 (2)	0.0485 (6)
H4	−0.1218	0.7420	0.0752	0.058*
C5	0.03034 (19)	0.6750 (2)	0.16817 (19)	0.0456 (6)
H5	0.0605	0.7551	0.1527	0.055*
C6	0.1946 (2)	0.5416 (3)	0.1395 (2)	0.0644 (8)
H6A	0.2011	0.4594	0.1113	0.077*

H6B	0.2160	0.5376	0.2195	0.077*
C7	0.2781 (2)	0.6236 (4)	0.0971 (2)	0.0673 (9)
C8	0.3910 (3)	0.5966 (7)	0.1312 (3)	0.120 (2)
H8	0.4132	0.5337	0.1817	0.144*
C9	0.4715 (4)	0.6629 (10)	0.0905 (5)	0.151 (3)
H9	0.5478	0.6470	0.1159	0.181*
C10	0.4386 (6)	0.7515 (7)	0.0132 (6)	0.145 (3)
H10	0.4931	0.7932	−0.0161	0.174*
C11	0.3266 (5)	0.7808 (4)	−0.0226 (4)	0.1065 (15)
H11	0.3046	0.8425	−0.0745	0.128*
C12	0.2469 (3)	0.7147 (3)	0.0215 (3)	0.0752 (9)
H12	0.1707	0.7333	−0.0012	0.090*
C13	−0.1988 (2)	0.5433 (3)	−0.0507 (2)	0.0642 (8)
H13A	−0.2667	0.5504	−0.0202	0.077*
H13B	−0.1943	0.4593	−0.0753	0.077*
C14	−0.2106 (2)	0.6261 (3)	−0.1498 (2)	0.0553 (7)
C15	−0.1246 (3)	0.6958 (3)	−0.1739 (3)	0.0680 (8)
H15	−0.0548	0.6953	−0.1259	0.082*
C16	−0.1398 (4)	0.7672 (3)	−0.2687 (3)	0.0838 (11)
H16	−0.0810	0.8153	−0.2836	0.101*
C17	−0.2416 (5)	0.7661 (4)	−0.3396 (3)	0.1025 (14)
H17	−0.2527	0.8137	−0.4035	0.123*
C18	−0.3259 (5)	0.6967 (6)	−0.3174 (4)	0.136 (2)
H18	−0.3951	0.6958	−0.3664	0.163*
C19	−0.3110 (4)	0.6273 (4)	−0.2236 (4)	0.1092 (16)
H19	−0.3705	0.5798	−0.2097	0.131*
C20	−0.1737 (2)	0.6415 (3)	0.1980 (2)	0.0556 (7)
C21	−0.2295 (3)	0.7239 (5)	0.3560 (3)	0.0907 (12)
H21A	−0.1970	0.6637	0.4098	0.136*
H21B	−0.3055	0.7007	0.3237	0.136*
H21C	−0.2298	0.8022	0.3914	0.136*
C22	0.05575 (19)	0.6489 (2)	0.2923 (2)	0.0435 (6)
N23	0.09866 (16)	0.7391 (2)	0.36102 (17)	0.0465 (5)
C24	0.1154 (2)	0.7207 (3)	0.4816 (2)	0.0496 (6)
H24	0.0571	0.6641	0.4965	0.060*
C25	0.0911 (3)	0.8475 (3)	0.5232 (3)	0.0693 (9)
H25A	0.1362	0.8622	0.5964	0.083*
H25B	0.0119	0.8555	0.5266	0.083*
C26	0.1229 (3)	0.9373 (3)	0.4395 (3)	0.0792 (10)
H26A	0.0771	1.0108	0.4350	0.095*
H26B	0.2019	0.9602	0.4603	0.095*
C27	0.1008 (3)	0.8700 (3)	0.3306 (3)	0.0633 (8)
H27A	0.0291	0.8948	0.2856	0.076*
H27B	0.1603	0.8859	0.2899	0.076*
C28	0.2328 (2)	0.6636 (3)	0.5283 (2)	0.0471 (6)
C29	0.3256 (2)	0.7552 (3)	0.5229 (2)	0.0548 (7)
C30	0.3740 (2)	0.7603 (4)	0.4306 (3)	0.0726 (9)
H30	0.3523	0.7048	0.3731	0.087*
C31	0.4557 (3)	0.8490 (5)	0.4238 (4)	0.1074 (15)

H31	0.4884	0.8525	0.3616	0.129*
C32	0.4875 (4)	0.9301 (6)	0.5076 (6)	0.130 (2)
H32	0.5432	0.9876	0.5031	0.156*
C33	0.4378 (4)	0.9283 (4)	0.5999 (4)	0.1076 (15)
H33	0.4570	0.9867	0.6554	0.129*
C34	0.3595 (3)	0.8387 (3)	0.6081 (3)	0.0752 (10)
H34	0.3289	0.8340	0.6715	0.090*
C35	0.2425 (2)	0.6118 (3)	0.6452 (2)	0.0516 (7)
C36	0.1531 (3)	0.6007 (3)	0.6997 (2)	0.0658 (9)
H36	0.0834	0.6351	0.6696	0.079*
C37	0.1683 (4)	0.5377 (4)	0.8000 (3)	0.0817 (11)
H37	0.1077	0.5297	0.8357	0.098*
C38	0.2689 (4)	0.4881 (3)	0.8464 (3)	0.0838 (11)
H38	0.2769	0.4454	0.9128	0.101*
C39	0.3598 (3)	0.5011 (4)	0.7949 (3)	0.0831 (10)
H39	0.4299	0.4685	0.8267	0.100*
C40	0.3455 (3)	0.5627 (3)	0.6962 (3)	0.0706 (8)
H40	0.4072	0.5718	0.6621	0.085*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0971 (15)	0.0533 (11)	0.0537 (12)	−0.0013 (11)	0.0272 (10)	−0.0088 (11)
O2	0.0767 (14)	0.106 (2)	0.0775 (15)	−0.0347 (15)	0.0281 (11)	−0.0103 (14)
O3	0.0695 (12)	0.0692 (13)	0.0625 (12)	0.0099 (11)	0.0273 (9)	−0.0043 (12)
O4	0.0664 (11)	0.0444 (11)	0.0489 (10)	−0.0070 (9)	0.0123 (8)	0.0011 (9)
O5	0.0564 (11)	0.0635 (12)	0.0511 (10)	0.0091 (10)	0.0119 (9)	−0.0118 (10)
N1	0.0516 (11)	0.0598 (14)	0.0484 (12)	0.0059 (10)	0.0132 (9)	−0.0049 (11)
C2	0.0643 (16)	0.0521 (16)	0.0343 (13)	−0.0061 (13)	0.0157 (12)	0.0027 (13)
N3	0.0583 (13)	0.0635 (15)	0.0456 (12)	−0.0017 (12)	0.0065 (9)	−0.0038 (11)
C4	0.0532 (14)	0.0468 (14)	0.0451 (14)	0.0035 (12)	0.0088 (11)	0.0043 (12)
C5	0.0517 (13)	0.0424 (14)	0.0447 (14)	−0.0008 (11)	0.0145 (11)	0.0011 (11)
C6	0.0613 (16)	0.080 (2)	0.0540 (16)	0.0163 (16)	0.0176 (12)	0.0009 (16)
C7	0.0549 (15)	0.094 (2)	0.0537 (17)	−0.0020 (16)	0.0129 (13)	−0.0258 (18)
C8	0.059 (2)	0.214 (6)	0.084 (3)	0.000 (3)	0.0053 (18)	−0.009 (3)
C9	0.058 (2)	0.269 (9)	0.125 (4)	−0.015 (4)	0.016 (3)	−0.028 (5)
C10	0.110 (4)	0.177 (7)	0.166 (6)	−0.068 (4)	0.074 (4)	−0.073 (5)
C11	0.134 (4)	0.076 (3)	0.131 (4)	−0.022 (3)	0.078 (3)	−0.026 (3)
C12	0.078 (2)	0.066 (2)	0.091 (2)	−0.0051 (17)	0.0397 (18)	−0.014 (2)
C13	0.0658 (17)	0.073 (2)	0.0515 (15)	−0.0191 (16)	0.0059 (13)	−0.0018 (16)
C14	0.0603 (15)	0.0552 (17)	0.0482 (15)	−0.0049 (13)	0.0051 (12)	−0.0015 (13)
C15	0.0721 (19)	0.070 (2)	0.0630 (18)	0.0012 (15)	0.0174 (15)	0.0069 (16)
C16	0.116 (3)	0.065 (2)	0.081 (2)	0.006 (2)	0.045 (2)	0.0091 (19)
C17	0.159 (4)	0.080 (3)	0.060 (2)	0.008 (3)	0.001 (3)	0.014 (2)
C18	0.125 (4)	0.142 (5)	0.113 (4)	−0.033 (4)	−0.044 (3)	0.052 (4)
C19	0.092 (3)	0.116 (3)	0.102 (3)	−0.031 (3)	−0.025 (2)	0.035 (3)
C20	0.0508 (14)	0.0655 (19)	0.0503 (16)	0.0021 (14)	0.0093 (11)	0.0036 (14)
C21	0.090 (2)	0.118 (3)	0.075 (2)	0.020 (2)	0.0433 (18)	−0.005 (2)

C22	0.0414 (12)	0.0437 (16)	0.0470 (14)	−0.0007 (11)	0.0126 (10)	−0.0006 (12)
N23	0.0476 (11)	0.0422 (12)	0.0491 (12)	0.0006 (9)	0.0083 (9)	−0.0042 (10)
C24	0.0499 (13)	0.0555 (16)	0.0460 (14)	−0.0032 (12)	0.0155 (11)	−0.0104 (13)
C25	0.0664 (18)	0.073 (2)	0.067 (2)	0.0124 (16)	0.0112 (15)	−0.0271 (18)
C26	0.092 (2)	0.0491 (18)	0.088 (3)	0.0115 (17)	−0.0027 (19)	−0.0128 (18)
C27	0.0743 (19)	0.0384 (15)	0.073 (2)	0.0020 (13)	0.0058 (16)	−0.0025 (14)
C28	0.0484 (13)	0.0523 (15)	0.0419 (13)	−0.0010 (12)	0.0122 (10)	−0.0043 (12)
C29	0.0455 (13)	0.0647 (19)	0.0527 (15)	−0.0017 (13)	0.0058 (11)	0.0128 (14)
C30	0.0514 (15)	0.096 (3)	0.0727 (19)	−0.0014 (16)	0.0182 (13)	0.0189 (19)
C31	0.073 (2)	0.140 (4)	0.112 (3)	−0.023 (3)	0.026 (2)	0.042 (3)
C32	0.074 (3)	0.154 (5)	0.148 (4)	−0.053 (3)	−0.013 (3)	0.064 (4)
C33	0.106 (3)	0.094 (3)	0.103 (3)	−0.043 (3)	−0.030 (3)	0.017 (3)
C34	0.072 (2)	0.077 (2)	0.068 (2)	−0.0200 (18)	−0.0070 (16)	0.0021 (18)
C35	0.0585 (16)	0.0525 (16)	0.0451 (14)	−0.0079 (12)	0.0137 (12)	−0.0065 (13)
C36	0.0645 (17)	0.087 (2)	0.0488 (16)	−0.0235 (16)	0.0178 (13)	−0.0123 (16)
C37	0.107 (3)	0.096 (3)	0.0486 (17)	−0.044 (2)	0.0316 (18)	−0.0157 (19)
C38	0.132 (4)	0.070 (2)	0.0475 (18)	−0.019 (2)	0.014 (2)	−0.0023 (17)
C39	0.112 (3)	0.079 (2)	0.0565 (19)	0.020 (2)	0.0122 (19)	0.0123 (18)
C40	0.079 (2)	0.077 (2)	0.0581 (17)	0.0140 (17)	0.0210 (15)	0.0108 (17)

Geometric parameters (Å, °)

O1—C2	1.225 (3)	C18—H18	0.9300
O2—C20	1.194 (4)	C19—H19	0.9300
O3—C20	1.317 (4)	C21—H21A	0.9599
O3—C21	1.443 (4)	C21—H21B	0.9599
O4—C22	1.230 (3)	C21—H21C	0.9599
O5—C28	1.429 (3)	C22—N23	1.335 (3)
O5—H5X	0.825 (18)	N23—C24	1.473 (3)
N1—C2	1.350 (3)	N23—C27	1.477 (4)
N1—C6	1.438 (4)	C24—C25	1.524 (4)
N1—C5	1.451 (3)	C24—C28	1.552 (4)
C2—N3	1.348 (4)	C24—H24	0.9800
N3—C4	1.439 (4)	C25—C26	1.526 (5)
N3—C13	1.449 (4)	C25—H25A	0.9700
C4—C20	1.524 (4)	C25—H25B	0.9700
C4—C5	1.559 (3)	C26—C27	1.506 (5)
C4—H4	0.9800	C26—H26A	0.9700
C5—C22	1.527 (3)	C26—H26B	0.9700
C5—H5	0.9800	C27—H27A	0.9700
C6—C7	1.516 (5)	C27—H27B	0.9700
C6—H6A	0.9700	C28—C29	1.513 (4)
C6—H6B	0.9700	C28—C35	1.531 (4)
C7—C12	1.364 (5)	C29—C30	1.379 (4)
C7—C8	1.377 (5)	C29—C34	1.389 (4)
C8—C9	1.383 (9)	C30—C31	1.396 (6)
C8—H8	0.9300	C30—H30	0.9300
C9—C10	1.360 (10)	C31—C32	1.357 (8)
C9—H9	0.9300	C31—H31	0.9300

C10—C11	1.375 (9)	C32—C33	1.387 (8)
C10—H10	0.9300	C32—H32	0.9300
C11—C12	1.395 (5)	C33—C34	1.377 (5)
C11—H11	0.9300	C33—H33	0.9300
C12—H12	0.9300	C34—H34	0.9300
C13—C14	1.503 (4)	C35—C36	1.384 (4)
C13—H13A	0.9700	C35—C40	1.386 (4)
C13—H13B	0.9700	C36—C37	1.395 (5)
C14—C19	1.364 (5)	C36—H36	0.9300
C14—C15	1.365 (4)	C37—C38	1.349 (6)
C15—C16	1.386 (5)	C37—H37	0.9300
C15—H15	0.9300	C38—C39	1.380 (6)
C16—C17	1.360 (6)	C38—H38	0.9300
C16—H16	0.9300	C39—C40	1.371 (5)
C17—C18	1.339 (7)	C39—H39	0.9300
C17—H17	0.9300	C40—H40	0.9300
C18—C19	1.364 (6)		
C20—O3—C21	117.5 (3)	H21A—C21—H21B	109.5
C28—O5—H5X	107 (2)	O3—C21—H21C	109.5
C2—N1—C6	123.1 (2)	H21A—C21—H21C	109.5
C2—N1—C5	113.3 (2)	H21B—C21—H21C	109.5
C6—N1—C5	123.2 (2)	O4—C22—N23	123.1 (2)
O1—C2—N3	125.4 (2)	O4—C22—C5	118.6 (2)
O1—C2—N1	126.0 (2)	N23—C22—C5	118.3 (2)
N3—C2—N1	108.5 (2)	C22—N23—C24	119.8 (2)
C2—N3—C4	113.0 (2)	C22—N23—C27	125.2 (2)
C2—N3—C13	122.2 (2)	C24—N23—C27	112.5 (2)
C4—N3—C13	123.4 (2)	N23—C24—C25	102.6 (2)
N3—C4—C20	111.9 (2)	N23—C24—C28	111.25 (19)
N3—C4—C5	103.27 (19)	C25—C24—C28	117.5 (2)
C20—C4—C5	114.1 (2)	N23—C24—H24	108.4
N3—C4—H4	109.1	C25—C24—H24	108.4
C20—C4—H4	109.1	C28—C24—H24	108.4
C5—C4—H4	109.1	C24—C25—C26	105.3 (2)
N1—C5—C22	109.6 (2)	C24—C25—H25A	110.7
N1—C5—C4	101.96 (19)	C26—C25—H25A	110.7
C22—C5—C4	113.22 (18)	C24—C25—H25B	110.7
N1—C5—H5	110.6	C26—C25—H25B	110.7
C22—C5—H5	110.6	H25A—C25—H25B	108.8
C4—C5—H5	110.6	C27—C26—C25	105.5 (3)
N1—C6—C7	115.6 (3)	C27—C26—H26A	110.7
N1—C6—H6A	108.4	C25—C26—H26A	110.7
C7—C6—H6A	108.4	C27—C26—H26B	110.7
N1—C6—H6B	108.4	C25—C26—H26B	110.7
C7—C6—H6B	108.4	H26A—C26—H26B	108.8
H6A—C6—H6B	107.4	N23—C27—C26	104.7 (3)
C12—C7—C8	119.1 (4)	N23—C27—H27A	110.8
C12—C7—C6	123.6 (3)	C26—C27—H27A	110.8
C8—C7—C6	117.0 (4)	N23—C27—H27B	110.8

C7—C8—C9	120.1 (6)	C26—C27—H27B	110.8
C7—C8—H8	119.9	H27A—C27—H27B	108.9
C9—C8—H8	119.9	O5—C28—C29	107.3 (2)
C10—C9—C8	119.8 (5)	O5—C28—C35	105.6 (2)
C10—C9—H9	120.1	C29—C28—C35	111.5 (2)
C8—C9—H9	120.1	O5—C28—C24	108.7 (2)
C9—C10—C11	121.5 (5)	C29—C28—C24	110.4 (2)
C9—C10—H10	119.3	C35—C28—C24	113.04 (19)
C11—C10—H10	119.3	C30—C29—C34	119.2 (3)
C10—C11—C12	117.7 (5)	C30—C29—C28	120.1 (3)
C10—C11—H11	121.2	C34—C29—C28	120.7 (3)
C12—C11—H11	121.2	C29—C30—C31	119.8 (4)
C7—C12—C11	121.7 (4)	C29—C30—H30	120.1
C7—C12—H12	119.2	C31—C30—H30	120.1
C11—C12—H12	119.2	C32—C31—C30	120.2 (4)
N3—C13—C14	115.4 (2)	C32—C31—H31	119.9
N3—C13—H13A	108.4	C30—C31—H31	119.9
C14—C13—H13A	108.4	C31—C32—C33	120.8 (4)
N3—C13—H13B	108.4	C31—C32—H32	119.6
C14—C13—H13B	108.4	C33—C32—H32	119.6
H13A—C13—H13B	107.5	C34—C33—C32	119.0 (5)
C19—C14—C15	117.6 (3)	C34—C33—H33	120.5
C19—C14—C13	118.5 (3)	C32—C33—H33	120.5
C15—C14—C13	123.9 (3)	C33—C34—C29	120.9 (4)
C14—C15—C16	121.1 (3)	C33—C34—H34	119.5
C14—C15—H15	119.4	C29—C34—H34	119.5
C16—C15—H15	119.4	C36—C35—C40	117.5 (3)
C17—C16—C15	119.3 (4)	C36—C35—C28	124.6 (2)
C17—C16—H16	120.3	C40—C35—C28	117.6 (2)
C15—C16—H16	120.3	C35—C36—C37	119.7 (3)
C18—C17—C16	120.0 (4)	C35—C36—H36	120.2
C18—C17—H17	120.0	C37—C36—H36	120.2
C16—C17—H17	120.0	C38—C37—C36	121.6 (3)
C17—C18—C19	120.6 (4)	C38—C37—H37	119.2
C17—C18—H18	119.7	C36—C37—H37	119.2
C19—C18—H18	119.7	C37—C38—C39	119.6 (3)
C14—C19—C18	121.4 (4)	C37—C38—H38	120.2
C14—C19—H19	119.3	C39—C38—H38	120.2
C18—C19—H19	119.3	C40—C39—C38	119.1 (4)
O2—C20—O3	125.3 (3)	C40—C39—H39	120.4
O2—C20—C4	124.9 (3)	C38—C39—H39	120.4
O3—C20—C4	109.7 (3)	C39—C40—C35	122.5 (3)
O3—C21—H21A	109.5	C39—C40—H40	118.8
O3—C21—H21B	109.5	C35—C40—H40	118.8
C6—N1—C2—O1	−7.2 (4)	C4—C5—C22—O4	66.3 (3)
C5—N1—C2—O1	−179.1 (2)	N1—C5—C22—N23	131.0 (2)
C6—N1—C2—N3	174.1 (2)	C4—C5—C22—N23	−116.0 (2)
C5—N1—C2—N3	2.2 (3)	O4—C22—N23—C24	−7.5 (3)
O1—C2—N3—C4	178.9 (2)	C5—C22—N23—C24	174.9 (2)

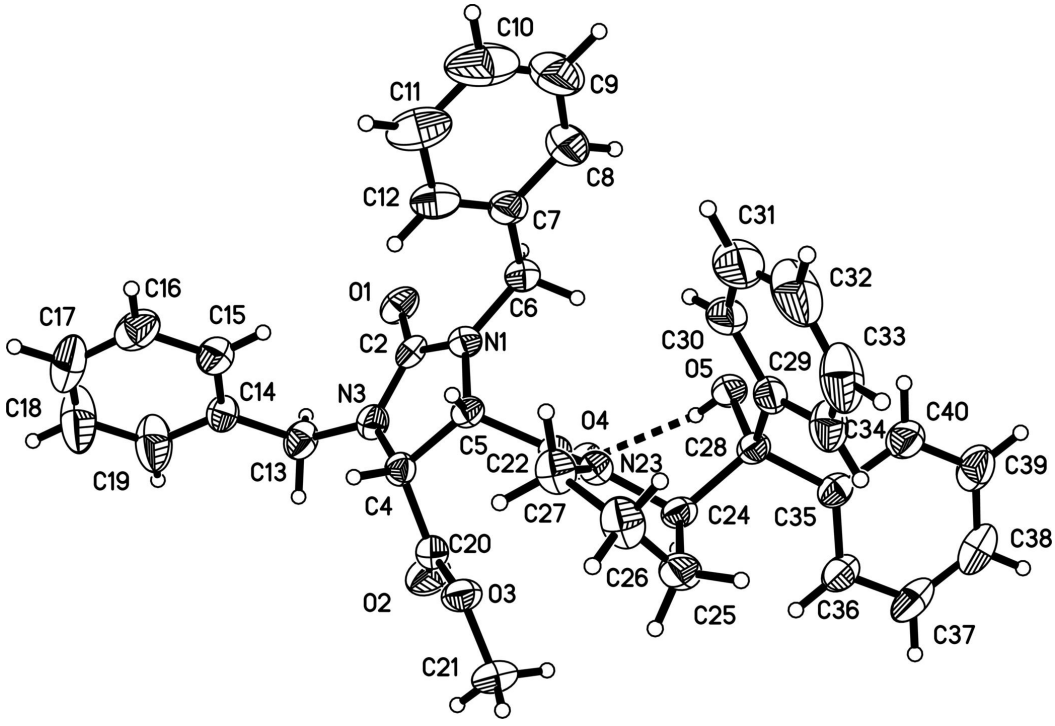
N1—C2—N3—C4	−2.3 (3)	O4—C22—N23—C27	−168.1 (3)
O1—C2—N3—C13	−14.3 (4)	C5—C22—N23—C27	14.4 (3)
N1—C2—N3—C13	164.5 (2)	C22—N23—C24—C25	−145.6 (2)
C2—N3—C4—C20	−121.7 (2)	C27—N23—C24—C25	17.3 (3)
C13—N3—C4—C20	71.7 (3)	C22—N23—C24—C28	88.0 (3)
C2—N3—C4—C5	1.5 (3)	C27—N23—C24—C28	−109.2 (3)
C13—N3—C4—C5	−165.1 (2)	N23—C24—C25—C26	−28.8 (3)
C2—N1—C5—C22	119.0 (2)	C28—C24—C25—C26	93.6 (3)
C6—N1—C5—C22	−52.8 (3)	C24—C25—C26—C27	30.6 (3)
C2—N1—C5—C4	−1.2 (3)	C22—N23—C27—C26	163.1 (2)
C6—N1—C5—C4	−173.0 (2)	C24—N23—C27—C26	1.3 (3)
N3—C4—C5—N1	−0.2 (2)	C25—C26—C27—N23	−19.6 (3)
C20—C4—C5—N1	121.5 (2)	N23—C24—C28—O5	−47.9 (3)
N3—C4—C5—C22	−117.8 (2)	C25—C24—C28—O5	−165.8 (2)
C20—C4—C5—C22	3.9 (3)	N23—C24—C28—C29	69.5 (3)
C2—N1—C6—C7	109.6 (3)	C25—C24—C28—C29	−48.4 (3)
C5—N1—C6—C7	−79.4 (3)	N23—C24—C28—C35	−164.9 (2)
N1—C6—C7—C12	−10.7 (4)	C25—C24—C28—C35	77.3 (3)
N1—C6—C7—C8	174.6 (3)	O5—C28—C29—C30	27.7 (3)
C12—C7—C8—C9	1.2 (7)	C35—C28—C29—C30	142.9 (3)
C6—C7—C8—C9	176.1 (5)	C24—C28—C29—C30	−90.5 (3)
C7—C8—C9—C10	−2.7 (10)	O5—C28—C29—C34	−155.2 (2)
C8—C9—C10—C11	2.8 (10)	C35—C28—C29—C34	−40.0 (3)
C9—C10—C11—C12	−1.2 (8)	C24—C28—C29—C34	86.5 (3)
C8—C7—C12—C11	0.4 (5)	C34—C29—C30—C31	−0.2 (5)
C6—C7—C12—C11	−174.2 (3)	C28—C29—C30—C31	176.9 (3)
C10—C11—C12—C7	−0.4 (6)	C29—C30—C31—C32	0.0 (6)
C2—N3—C13—C14	−87.1 (3)	C30—C31—C32—C33	−1.5 (7)
C4—N3—C13—C14	78.2 (4)	C31—C32—C33—C34	3.3 (7)
N3—C13—C14—C19	−165.8 (4)	C32—C33—C34—C29	−3.6 (6)
N3—C13—C14—C15	17.9 (4)	C30—C29—C34—C33	2.0 (5)
C19—C14—C15—C16	1.4 (5)	C28—C29—C34—C33	−175.0 (3)
C13—C14—C15—C16	177.8 (3)	O5—C28—C35—C36	−110.4 (3)
C14—C15—C16—C17	−1.0 (5)	C29—C28—C35—C36	133.4 (3)
C15—C16—C17—C18	0.1 (7)	C24—C28—C35—C36	8.3 (4)
C16—C17—C18—C19	0.5 (8)	O5—C28—C35—C40	63.6 (3)
C15—C14—C19—C18	−0.9 (7)	C29—C28—C35—C40	−52.6 (4)
C13—C14—C19—C18	−177.4 (5)	C24—C28—C35—C40	−177.7 (3)
C17—C18—C19—C14	−0.1 (9)	C40—C35—C36—C37	−2.4 (4)
C21—O3—C20—O2	2.9 (4)	C28—C35—C36—C37	171.6 (3)
C21—O3—C20—C4	180.0 (3)	C35—C36—C37—C38	0.8 (5)
N3—C4—C20—O2	−5.5 (4)	C36—C37—C38—C39	0.9 (6)
C5—C4—C20—O2	−122.3 (3)	C37—C38—C39—C40	−1.1 (6)
N3—C4—C20—O3	177.4 (2)	C38—C39—C40—C35	−0.5 (6)
C5—C4—C20—O3	60.5 (3)	C36—C35—C40—C39	2.3 (5)
N1—C5—C22—O4	−46.7 (3)	C28—C35—C40—C39	−172.1 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5X···O4	0.825 (18)	2.00 (2)	2.762 (3)	153 (3)
C4—H4···O1^{i}	0.98	2.57	3.328 (3)	135
C5—H5···O1^{i}	0.98	2.57	3.320 (3)	133
C16—H16···O4^{i}	0.93	2.62	3.383 (4)	139
C17—H17···O5^{i}	0.93	2.80	3.544 (5)	138
C32—H32···O5^{ii}	0.93	2.58	3.435 (5)	153
C37—H37···O1^{iii}	0.93	2.50	3.336 (4)	150

Symmetry codes: (i) −*x*, *y*+1/2, −*z*; (ii) −*x*+1, *y*+1/2, −*z*+1; (iii) *x*, *y*, *z*+1.

Fig. 1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C8
C12
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C31
C32
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O1
C6
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C30
C33
O5
C29
C18
C14
N3
C5
O4
C34
C40
C28
C19
C13
C22
N23
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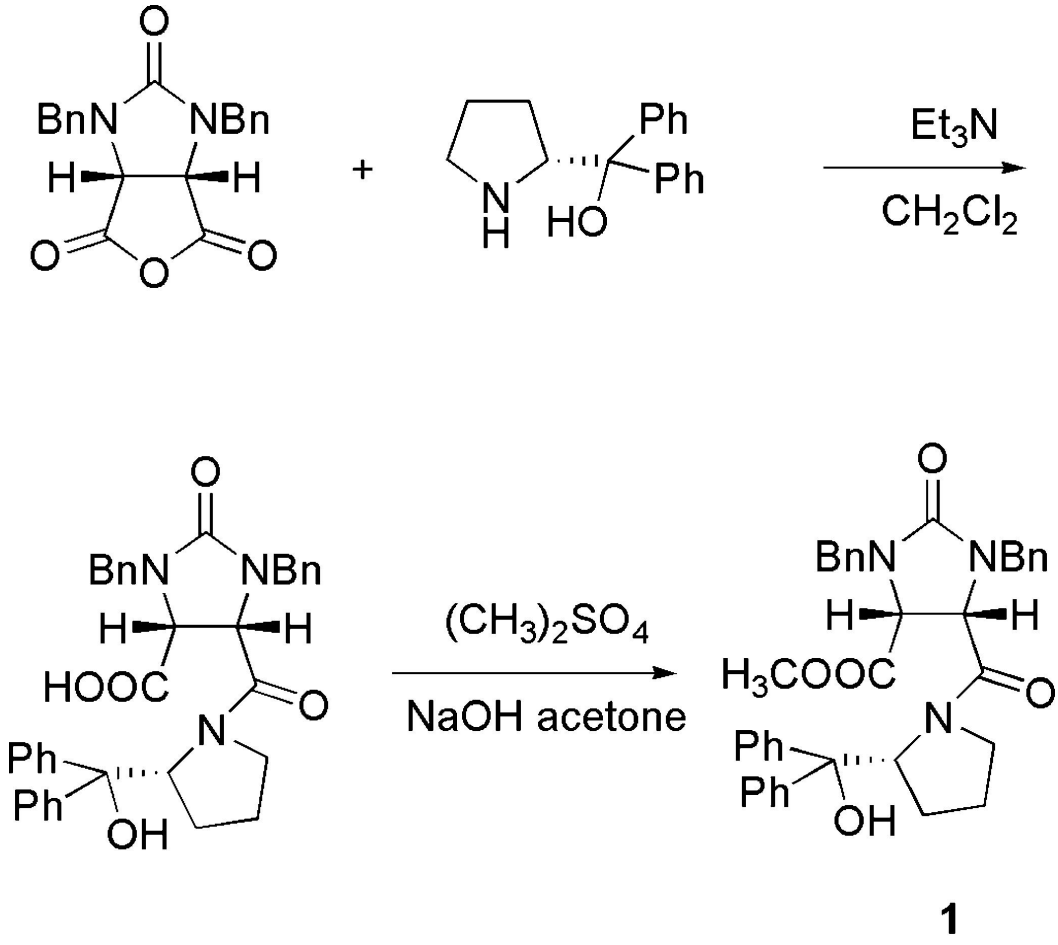
C37
C21

Fig. 2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HOOC
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NaOH acetone
H3COOC
OH

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