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(3*S*,4*S*)-1-Benzyl-4-(*N*-octylcarbamoyloxy)pyrrolidin-3-yl *N*-octylcarbamate: a low-temperature redetermination

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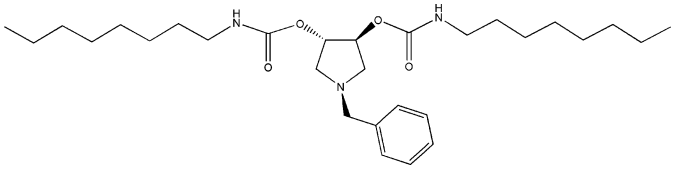
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.003 Å; R factor = 0.039; wR factor = 0.096; data-to-parameter ratio = 16.6.

The title compound, $C_{29}H_{49}N_3O_4$, is a model compound synthesized to obtain information on the intermomecular forces that drive the self-assembly of a similar compound bearing longer aliphatic chains. In the crystal, molecules are arranged in columns due to the formation of two antiparallel strong hydrogen-bonded chains by the carbamate units. The two long aliphatic chains are *trans*.

Related literature

For related literature, see: Cicchi *et al.* (2007); Schoonbeek *et al.* (2000).



Experimental

Crystal data

$C_{29}H_{49}N_3O_4$
M_r = 503.71
Monoclinic, $P2_1$
a = 5.039 (1) Å
b = 15.825 (1) Å
c = 18.330 (1) Å
β = 90.884 (1)°
V = 1461.5 (3) Å^3
Z = 2
Cu $K\alpha$ radiation
μ = 0.60 mm^{-1}
T = 150 (2) K
0.32 × 0.22 × 0.18 mm

Data collection

Oxford Diffraction Xcalibur PX Ultra CCD diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2006)
T_{min} = 0.851, T_{max} = 0.898
23018 measured reflections
5438 independent reflections
4359 reflections with $I > 2\sigma(I)$
R_{int} = 0.053

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.096
S = 1.08
5438 reflections
327 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Absolute structure: Flack (1983), with 2344 Friedel pairs
Flack parameter: −0.02 (16)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—HN2···O2^{i}	0.88	2.01	2.8449 (19)	157
N3—HN3···O4^{ii}	0.88	2.15	3.0211 (19)	172

Symmetry codes: (i) $x+1, y, z$; (ii) $x-1, y, z$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2006); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2057).

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supplementary materials

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(3*S*,4*S*)-1-Benzyl-4-(*N*-octylcarbamoyloxy)pyrrolidin-3-yl *N*-octylcarbamate: a low-temperature redetermination

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Comment

The title compound, (I), was synthesized as a model compound. Its 150 K data collection was useful to suggest a model for the formation of organogels by its homologue, (3*S*, 4S)-Dodecyl-carbamic acid 1-benzyl-4-dodecylcarbamoyl oxy-pyrrolidin-3-yl ester, bearing longer aliphatic chains (C12) (Cicchi *et al.*, 2007). To develop such a model for the formation of the gel it was necessary to obtain information about the more stable conformation of the carbamate units responsable for the self assembling process that give origin to an organogel. Information derived from X-Ray crystallography had already been used to propose suitable models for gel formation (*viz*., Schoonbeek *et al.*, 2000). In the presents case, while C12 did not afford suitable crystals, compound (I) crystallized from diisopropyl ether. The structure showed that the crystal is formed by long columns in which the two carbamates units of each molecule participate in the formation of two antiparallel strong H-bonded chains (Table 2). The long all-*trans* aliphatic chains contributes to the packing of the crystal by Van der Walls interactions. Dimensions are available in the archived CIF.

Experimental

An anhydrous toluene solution (10 ml) of (3*R*,4S)-1-benzylpyrrolidine-3,4-diol (200 mg, 1.04 mmol, 1eq) and octyliso-cianate (321 mg, 2.1eq) were refluxed for 72 h. The crude reaction mixture was concentrated and purified by flash column chromatography (Rf = 1/2, CH2Cl2/AcOEt 1:1) to obtain 323 mg of a white solid (64% yield). The compound was crystallized from diisopropylether to obtain white crystals. *M*.p. 94–95°C. Crystals suitable for X-ray analysis were obtained by slow evaporation of a diluted diisopropyl ether solution.

Refinement

All H atoms were clearly observable in the difference Fourier map but were placed at ideal positions (C—H_3: 0.98) Å, C—H_2: 0.99 Å, C—H: 1.00 Å, N—H: 0.88 Å), and allowed to ride with U(H) = 1.2 × U_{eq}(Host).

Figures

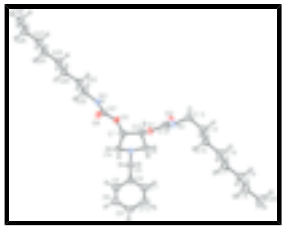

Fig. 1. Molecular structure of (I). Displacement ellipsoids are drawn at the 40% probability level.

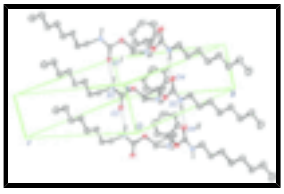

Fig. 2. Packing view of (I) showing the connectivity of molecules *via* N—H···O links (dashed lines). Only nitrogen and oxygen atoms involved in intermolecular hydrogen bonding have been labelled. [Symmetry codes as in Table 2].

(3*S*,4*S*)-1-Benzyl-4-(*N*-octylcarbamoyloxy)pyrrolidin-3-yl *N*-octylcarbamate

Crystal data

$C_{29}H_{49}N_3O_4$	F_{000} = 552
M_r = 503.71	D_x = 1.145 Mg m^{-3}
Monoclinic, $P2_1$	Melting point: 94 K
Hall symbol: P 2yb	Cu $K\alpha$ radiation λ = 1.54184 Å
a = 5.039 (1) Å	Cell parameters from 10467 reflections
b = 15.825 (1) Å	θ = 3.7–74.2º
c = 18.330 (1) Å	μ = 0.60 mm^{-1}
β = 90.884 (1)º	T = 150 (2) K
V = 1461.5 (3) Å^3	Prismatic, colourless
Z = 2	0.32 × 0.22 × 0.18 mm

Data collection

Oxford Diffraction XCALIBUR PX Ultra CCD diffractometer	5438 independent reflections
Radiation source: Enhance (Cu) X-ray Source	4359 reflections with $I > 2\sigma(I)$
Monochromator: Oxford Diffraction Enhance ULTRA assembly	R_{int} = 0.053
Detector resolution: 8.1241 pixels mm^{-1}	θ_{max} = 74.2º
T = 150(2) K	θ_{min} = 3.7º
ω scans	h = −5→6
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2006)	k = −19→19
T_{min} = 0.851, T_{max} = 0.898	l = −22→22
23018 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: difference Fourier map
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.039	$w = 1/[\sigma^2(F_o^2) + (0.0542P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.096	$(\Delta/\sigma)_{max} < 0.001$
S = 1.08	$\Delta\rho_{max}$ = 0.30 e Å^{-3}
5438 reflections	$\Delta\rho_{min}$ = −0.21 e Å^{-3}

327 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983), with how many Friedel pairs?
Primary atom site location: structure-invariant direct methods	Flack parameter: −0.02 (16)
Secondary atom site location: difference Fourier map	

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}
N1	0.4101 (3)	0.30272 (10)	0.04853 (9)	0.0330 (3)
N2	0.9553 (3)	0.00132 (10)	0.10478 (8)	0.0337 (4)
HN2	1.1142	0.0216	0.0960	0.040*
N3	0.4456 (3)	0.20838 (11)	−0.21319 (7)	0.0314 (3)
HN3	0.2770	0.2064	−0.2015	0.038*
O1	0.8120 (2)	0.11802 (8)	0.05040 (7)	0.0319 (3)
O2	0.5140 (2)	0.02369 (9)	0.09573 (8)	0.0401 (3)
O3	0.5166 (2)	0.21265 (9)	−0.09450 (6)	0.0341 (3)
O4	0.8671 (2)	0.22108 (10)	−0.17371 (7)	0.0367 (3)
C1	0.6848 (3)	0.22989 (12)	−0.03276 (9)	0.0291 (4)
H1	0.8768	0.2221	−0.0440	0.035*
C2	0.5931 (3)	0.17090 (12)	0.02901 (9)	0.0296 (4)
H2	0.4416	0.1352	0.0113	0.035*
C3	0.4998 (4)	0.22994 (13)	0.08996 (10)	0.0362 (4)
H3A	0.3533	0.2044	0.1178	0.043*
H3B	0.6472	0.2448	0.1239	0.043*
C4	0.6266 (3)	0.31805 (12)	−0.00332 (9)	0.0320 (4)
H4B	0.7846	0.3423	0.0218	0.038*
H4A	0.5687	0.3567	−0.0430	0.038*
C5	0.3196 (4)	0.37631 (14)	0.09092 (11)	0.0389 (4)
H5A	0.1821	0.3571	0.1252	0.047*
H5B	0.2348	0.4171	0.0569	0.047*
C6	0.5319 (4)	0.42160 (13)	0.13372 (11)	0.0374 (4)
C7	0.6694 (4)	0.48838 (14)	0.10240 (12)	0.0436 (5)
H7	0.6238	0.5058	0.0542	0.052*
C8	0.8694 (5)	0.52962 (15)	0.13954 (14)	0.0521 (6)
H8	0.9604	0.5754	0.1176	0.063*

C9	0.9355 (4)	0.50376 (17)	0.20860 (14)	0.0537 (6)
H9	1.0751	0.5314	0.2346	0.064*
C10	0.8012 (4)	0.43777 (18)	0.24092 (13)	0.0544 (6)
H10	0.8495	0.4203	0.2889	0.065*
C11	0.5966 (4)	0.39690 (15)	0.20387 (11)	0.0464 (5)
H11	0.5021	0.3524	0.2266	0.056*
C12	0.7430 (3)	0.04508 (12)	0.08529 (9)	0.0290 (4)
C13	0.9289 (4)	−0.08052 (13)	0.14085 (10)	0.0349 (4)
H13A	1.0839	−0.1159	0.1285	0.042*
H13B	0.7683	−0.1091	0.1212	0.042*
C14	0.9094 (4)	−0.07582 (13)	0.22323 (11)	0.0362 (4)
H14A	0.7787	−0.0318	0.2360	0.043*
H14B	0.8428	−0.1305	0.2417	0.043*
C15	1.1704 (4)	−0.05602 (14)	0.26068 (10)	0.0371 (4)
H15A	1.2328	0.0002	0.2446	0.045*
H15B	1.3043	−0.0984	0.2462	0.045*
C16	1.1456 (4)	−0.05638 (15)	0.34315 (11)	0.0406 (5)
H16A	1.0220	−0.0109	0.3574	0.049*
H16B	1.0672	−0.1109	0.3583	0.049*
C17	1.4051 (4)	−0.04410 (16)	0.38341 (11)	0.0424 (5)
H17A	1.4807	0.0113	0.3697	0.051*
H17B	1.5311	−0.0885	0.3681	0.051*
C18	1.3776 (4)	−0.04762 (15)	0.46605 (11)	0.0434 (5)
H18A	1.2542	−0.0026	0.4815	0.052*
H18B	1.2991	−0.1026	0.4797	0.052*
C19	1.6352 (4)	−0.03697 (18)	0.50561 (11)	0.0516 (6)
H19A	1.7121	0.0184	0.4925	0.062*
H19B	1.7593	−0.0814	0.4894	0.062*
C20	1.6105 (6)	−0.0417 (2)	0.58814 (12)	0.0639 (7)
H20A	1.5390	−0.0970	0.6017	0.077*
H20B	1.4909	0.0029	0.6048	0.077*
H20C	1.7859	−0.0341	0.6111	0.077*
C21	0.6301 (3)	0.21444 (12)	−0.16218 (9)	0.0273 (4)
C22	0.5237 (3)	0.20502 (15)	−0.28971 (9)	0.0374 (4)
H22B	0.6365	0.1547	−0.2976	0.045*
H22A	0.6292	0.2559	−0.3015	0.045*
C23	0.2871 (3)	0.20081 (14)	−0.33888 (9)	0.0344 (4)
H23A	0.1093	0.1962	−0.3229	0.041*
H23B	0.1878	0.1486	−0.3287	0.041*
C24	0.3689 (4)	0.20496 (14)	−0.41899 (9)	0.0373 (4)
H24A	0.4649	0.2586	−0.4272	0.045*
H24B	0.4933	0.1580	−0.4288	0.045*
C25	0.1414 (4)	0.19961 (14)	−0.47192 (9)	0.0385 (5)
H25A	0.0182	0.2472	−0.4632	0.046*
H25B	0.0434	0.1463	−0.4637	0.046*
C26	0.2326 (4)	0.20255 (15)	−0.55127 (10)	0.0404 (5)
H26B	0.3258	0.2567	−0.5595	0.049*
H26A	0.3615	0.1563	−0.5591	0.049*
C27	0.0124 (4)	0.19438 (15)	−0.60607 (10)	0.0424 (5)

H27A	−0.1113	0.2424	−0.6003	0.051*
H27B	−0.0872	0.1418	−0.5963	0.051*
C28	0.1096 (4)	0.19258 (17)	−0.68477 (11)	0.0487 (6)
H28A	0.1948	0.2473	−0.6960	0.058*
H28B	0.2457	0.1479	−0.6893	0.058*
C29	−0.1075 (5)	0.17669 (19)	−0.73927 (12)	0.0578 (6)
H29C	−0.2419	0.2212	−0.7356	0.069*
H29B	−0.1890	0.1217	−0.7296	0.069*
H29A	−0.0342	0.1768	−0.7885	0.069*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0300 (8)	0.0340 (9)	0.0349 (8)	0.0031 (6)	−0.0037 (6)	−0.0025 (7)
N2	0.0230 (7)	0.0401 (9)	0.0379 (8)	−0.0018 (6)	−0.0030 (6)	0.0054 (7)
N3	0.0233 (7)	0.0444 (9)	0.0263 (7)	−0.0013 (7)	−0.0036 (5)	−0.0014 (7)
O1	0.0224 (5)	0.0334 (7)	0.0397 (7)	−0.0003 (5)	−0.0040 (5)	0.0058 (6)
O2	0.0221 (6)	0.0453 (8)	0.0528 (8)	−0.0030 (5)	−0.0049 (6)	0.0140 (7)
O3	0.0249 (6)	0.0529 (8)	0.0242 (6)	−0.0067 (6)	−0.0049 (5)	−0.0028 (6)
O4	0.0250 (6)	0.0522 (8)	0.0329 (6)	0.0017 (6)	−0.0039 (5)	−0.0010 (6)
C1	0.0228 (8)	0.0371 (10)	0.0271 (8)	−0.0016 (7)	−0.0091 (7)	0.0000 (7)
C2	0.0224 (8)	0.0348 (10)	0.0314 (9)	0.0015 (7)	−0.0049 (7)	0.0015 (8)
C3	0.0375 (9)	0.0385 (11)	0.0327 (10)	0.0018 (8)	0.0034 (8)	0.0024 (8)
C4	0.0305 (9)	0.0338 (10)	0.0315 (9)	−0.0003 (8)	−0.0060 (7)	0.0017 (8)
C5	0.0296 (9)	0.0422 (12)	0.0448 (11)	0.0062 (8)	−0.0025 (8)	−0.0038 (9)
C6	0.0345 (9)	0.0366 (11)	0.0409 (10)	0.0077 (8)	−0.0015 (8)	−0.0086 (9)
C7	0.0474 (11)	0.0338 (11)	0.0495 (12)	0.0070 (9)	−0.0061 (9)	−0.0049 (9)
C8	0.0486 (12)	0.0356 (12)	0.0721 (16)	−0.0019 (9)	−0.0035 (11)	−0.0132 (11)
C9	0.0410 (11)	0.0564 (15)	0.0635 (15)	0.0015 (11)	−0.0079 (11)	−0.0294 (12)
C10	0.0497 (13)	0.0732 (18)	0.0401 (11)	0.0109 (12)	−0.0083 (10)	−0.0189 (11)
C11	0.0457 (12)	0.0535 (14)	0.0399 (11)	0.0030 (10)	0.0022 (9)	−0.0080 (10)
C12	0.0237 (8)	0.0333 (10)	0.0299 (9)	0.0004 (7)	−0.0056 (7)	0.0018 (7)
C13	0.0299 (9)	0.0346 (10)	0.0400 (10)	0.0011 (8)	−0.0060 (8)	0.0054 (8)
C14	0.0286 (8)	0.0389 (11)	0.0411 (10)	−0.0012 (8)	−0.0028 (8)	0.0036 (9)
C15	0.0306 (9)	0.0458 (12)	0.0348 (10)	−0.0003 (8)	−0.0034 (7)	0.0006 (9)
C16	0.0385 (10)	0.0458 (12)	0.0374 (10)	0.0000 (9)	−0.0021 (8)	0.0009 (9)
C17	0.0372 (10)	0.0530 (13)	0.0369 (11)	−0.0007 (9)	−0.0035 (8)	0.0009 (9)
C18	0.0455 (11)	0.0514 (13)	0.0334 (10)	−0.0029 (9)	−0.0008 (9)	0.0004 (9)
C19	0.0458 (12)	0.0713 (17)	0.0375 (12)	−0.0054 (11)	−0.0052 (9)	−0.0016 (11)
C20	0.0699 (16)	0.083 (2)	0.0387 (13)	−0.0004 (14)	−0.0107 (11)	−0.0018 (12)
C21	0.0247 (8)	0.0272 (9)	0.0300 (8)	−0.0003 (7)	−0.0039 (6)	0.0002 (8)
C22	0.0298 (8)	0.0549 (13)	0.0275 (8)	0.0007 (9)	−0.0021 (7)	−0.0023 (9)
C23	0.0286 (8)	0.0460 (12)	0.0286 (8)	−0.0033 (8)	−0.0036 (7)	−0.0016 (8)
C24	0.0317 (9)	0.0488 (13)	0.0314 (9)	−0.0035 (9)	−0.0043 (7)	0.0000 (9)
C25	0.0348 (9)	0.0504 (13)	0.0301 (9)	−0.0016 (9)	−0.0061 (7)	−0.0036 (9)
C26	0.0381 (9)	0.0513 (13)	0.0318 (9)	−0.0038 (9)	−0.0049 (7)	0.0006 (9)
C27	0.0399 (10)	0.0546 (14)	0.0326 (9)	−0.0008 (9)	−0.0065 (8)	0.0007 (9)
C28	0.0488 (12)	0.0638 (16)	0.0334 (10)	−0.0009 (10)	−0.0079 (9)	0.0017 (10)

C29	0.0607 (14)	0.0780 (17)	0.0344 (11)	0.0084 (13)	−0.0121 (10)	0.0003 (11)

Geometric parameters (Å, °)

N1—C3	1.448 (3)	C14—H14B	0.9900
N1—C5	1.476 (3)	C15—C16	1.519 (3)
N1—C4	1.478 (2)	C15—H15A	0.9900
N2—C12	1.319 (2)	C15—H15B	0.9900
N2—C13	1.461 (2)	C16—C17	1.504 (3)
N2—HN2	0.8800	C16—H16A	0.9900
N3—C21	1.312 (2)	C16—H16B	0.9900
N3—C22	1.463 (2)	C17—C18	1.524 (3)
N3—HN3	0.8800	C17—H17A	0.9900
O1—C12	1.367 (2)	C17—H17B	0.9900
O1—C2	1.434 (2)	C18—C19	1.486 (3)
O2—C12	1.220 (2)	C18—H18A	0.9900
O3—C21	1.374 (2)	C18—H18B	0.9900
O3—C1	1.4295 (19)	C19—C20	1.522 (3)
O4—C21	1.2206 (19)	C19—H19A	0.9900
C1—C4	1.526 (3)	C19—H19B	0.9900
C1—C2	1.544 (3)	C20—H20A	0.9800
C1—H1	1.0000	C20—H20B	0.9800
C2—C3	1.536 (3)	C20—H20C	0.9800
C2—H2	1.0000	C22—C23	1.485 (2)
C3—H3A	0.9900	C22—H22B	0.9900
C3—H3B	0.9900	C22—H22A	0.9900
C4—H4B	0.9900	C23—C24	1.533 (2)
C4—H4A	0.9900	C23—H23A	0.9500
C5—C6	1.499 (3)	C23—H23B	0.9858
C5—H5A	0.9900	C24—C25	1.493 (2)
C5—H5B	0.9900	C24—H24A	0.9900
C6—C11	1.378 (3)	C24—H24B	0.9900
C6—C7	1.392 (3)	C25—C26	1.533 (3)
C7—C8	1.372 (3)	C25—H25A	0.9900
C7—H7	0.9500	C25—H25B	0.9900
C8—C9	1.367 (4)	C26—C27	1.491 (3)
C8—H8	0.9500	C26—H26B	0.9900
C9—C10	1.383 (4)	C26—H26A	0.9900
C9—H9	0.9500	C27—C28	1.531 (3)
C10—C11	1.386 (3)	C27—H27A	0.9900
C10—H10	0.9500	C27—H27B	0.9900
C11—H11	0.9500	C28—C29	1.491 (3)
C13—C14	1.517 (3)	C28—H28A	0.9900
C13—H13A	0.9900	C28—H28B	0.9900
C13—H13B	0.9900	C29—H29C	0.9800
C14—C15	1.507 (3)	C29—H29B	0.9800
C14—H14A	0.9900	C29—H29A	0.9800
C3—N1—C5	116.61 (15)	C17—C16—H16A	108.8
C3—N1—C4	103.91 (14)	C15—C16—H16A	108.8

C5—N1—C4	116.49 (15)	C17—C16—H16B	108.8
C12—N2—C13	120.59 (14)	C15—C16—H16B	108.8
C12—N2—HN2	119.7	H16A—C16—H16B	107.7
C13—N2—HN2	119.7	C16—C17—C18	113.03 (17)
C21—N3—C22	119.20 (14)	C16—C17—H17A	109.0
C21—N3—HN3	120.4	C18—C17—H17A	109.0
C22—N3—HN3	120.4	C16—C17—H17B	109.0
C12—O1—C2	114.85 (13)	C18—C17—H17B	109.0
C21—O3—C1	117.45 (12)	H17A—C17—H17B	107.8
O3—C1—C4	109.87 (14)	C19—C18—C17	112.86 (18)
O3—C1—C2	106.55 (14)	C19—C18—H18A	109.0
C4—C1—C2	103.44 (15)	C17—C18—H18A	109.0
O3—C1—H1	112.2	C19—C18—H18B	109.0
C4—C1—H1	112.2	C17—C18—H18B	109.0
C2—C1—H1	112.2	H18A—C18—H18B	107.8
O1—C2—C3	113.52 (14)	C18—C19—C20	113.3 (2)
O1—C2—C1	108.41 (14)	C18—C19—H19A	108.9
C3—C2—C1	105.31 (15)	C20—C19—H19A	108.9
O1—C2—H2	109.8	C18—C19—H19B	108.9
C3—C2—H2	109.8	C20—C19—H19B	108.9
C1—C2—H2	109.8	H19A—C19—H19B	107.7
N1—C3—C2	101.48 (14)	C19—C20—H20A	109.5
N1—C3—H3A	111.5	C19—C20—H20B	109.5
C2—C3—H3A	111.5	H20A—C20—H20B	109.5
N1—C3—H3B	111.5	C19—C20—H20C	109.5
C2—C3—H3B	111.5	H20A—C20—H20C	109.5
H3A—C3—H3B	109.3	H20B—C20—H20C	109.5
N1—C4—C1	103.00 (14)	O4—C21—N3	124.57 (16)
N1—C4—H4B	111.2	O4—C21—O3	125.44 (14)
C1—C4—H4B	111.2	N3—C21—O3	109.99 (14)
N1—C4—H4A	111.2	N3—C22—C23	111.00 (14)
C1—C4—H4A	111.2	N3—C22—H22B	109.4
H4B—C4—H4A	109.1	C23—C22—H22B	109.4
N1—C5—C6	115.41 (15)	N3—C22—H22A	109.4
N1—C5—H5A	108.4	C23—C22—H22A	109.4
C6—C5—H5A	108.4	H22B—C22—H22A	108.0
N1—C5—H5B	108.4	C22—C23—C24	110.76 (15)
C6—C5—H5B	108.4	C22—C23—H23A	124.6
H5A—C5—H5B	107.5	C24—C23—H23A	124.6
C11—C6—C7	119.19 (19)	C22—C23—H23B	109.2
C11—C6—C5	120.62 (19)	C24—C23—H23B	111.2
C7—C6—C5	120.18 (18)	H23A—C23—H23B	52.6
C8—C7—C6	121.5 (2)	C25—C24—C23	113.90 (15)
C8—C7—H7	119.2	C25—C24—H24A	108.8
C6—C7—H7	119.2	C23—C24—H24A	108.8
C7—C8—C9	118.9 (2)	C25—C24—H24B	108.8
C7—C8—H8	120.5	C23—C24—H24B	108.8
C9—C8—H8	120.5	H24A—C24—H24B	107.7
C8—C9—C10	120.6 (2)	C24—C25—C26	112.11 (15)

C8—C9—H9	119.7	C24—C25—H25A	109.2
C10—C9—H9	119.7	C26—C25—H25A	109.2
C11—C10—C9	120.5 (2)	C24—C25—H25B	109.2
C11—C10—H10	119.7	C26—C25—H25B	109.2
C9—C10—H10	119.7	H25A—C25—H25B	107.9
C6—C11—C10	119.2 (2)	C27—C26—C25	113.93 (16)
C6—C11—H11	120.4	C27—C26—H26B	108.8
C10—C11—H11	120.4	C25—C26—H26B	108.8
O2—C12—N2	125.23 (17)	C27—C26—H26A	108.8
O2—C12—O1	123.74 (15)	C25—C26—H26A	108.8
N2—C12—O1	111.02 (14)	H26B—C26—H26A	107.7
N2—C13—C14	114.50 (16)	C26—C27—C28	113.02 (17)
N2—C13—H13A	108.6	C26—C27—H27A	109.0
C14—C13—H13A	108.6	C28—C27—H27A	109.0
N2—C13—H13B	108.6	C26—C27—H27B	109.0
C14—C13—H13B	108.6	C28—C27—H27B	109.0
H13A—C13—H13B	107.6	H27A—C27—H27B	107.8
C15—C14—C13	113.22 (15)	C29—C28—C27	113.10 (19)
C15—C14—H14A	108.9	C29—C28—H28A	109.0
C13—C14—H14A	108.9	C27—C28—H28A	109.0
C15—C14—H14B	108.9	C29—C28—H28B	109.0
C13—C14—H14B	108.9	C27—C28—H28B	109.0
H14A—C14—H14B	107.7	H28A—C28—H28B	107.8
C14—C15—C16	111.59 (16)	C28—C29—H29C	109.5
C14—C15—H15A	109.3	C28—C29—H29B	109.5
C16—C15—H15A	109.3	H29C—C29—H29B	109.5
C14—C15—H15B	109.3	C28—C29—H29A	109.5
C16—C15—H15B	109.3	H29C—C29—H29A	109.5
H15A—C15—H15B	108.0	H29B—C29—H29A	109.5
C17—C16—C15	113.81 (16)		
C21—O3—C1—C4	105.90 (18)	C7—C6—C11—C10	−1.5 (3)
C21—O3—C1—C2	−142.69 (16)	C5—C6—C11—C10	177.36 (19)
C12—O1—C2—C3	84.66 (19)	C9—C10—C11—C6	1.3 (3)
C12—O1—C2—C1	−158.70 (14)	C13—N2—C12—O2	1.1 (3)
O3—C1—C2—O1	120.31 (15)	C13—N2—C12—O1	−177.95 (15)
C4—C1—C2—O1	−123.87 (14)	C2—O1—C12—O2	2.6 (2)
O3—C1—C2—C3	−117.88 (15)	C2—O1—C12—N2	−178.32 (15)
C4—C1—C2—C3	−2.06 (17)	C12—N2—C13—C14	−87.7 (2)
C5—N1—C3—C2	−176.27 (15)	N2—C13—C14—C15	−74.3 (2)
C4—N1—C3—C2	−46.63 (17)	C13—C14—C15—C16	−177.03 (17)
O1—C2—C3—N1	147.80 (14)	C14—C15—C16—C17	175.20 (18)
C1—C2—C3—N1	29.35 (17)	C15—C16—C17—C18	−178.06 (19)
C3—N1—C4—C1	46.10 (16)	C16—C17—C18—C19	179.0 (2)
C5—N1—C4—C1	175.81 (14)	C17—C18—C19—C20	−179.1 (2)
O3—C1—C4—N1	87.93 (16)	C22—N3—C21—O4	3.2 (3)
C2—C1—C4—N1	−25.51 (15)	C22—N3—C21—O3	−177.17 (16)
C3—N1—C5—C6	68.1 (2)	C1—O3—C21—O4	7.8 (3)
C4—N1—C5—C6	−55.2 (2)	C1—O3—C21—N3	−171.84 (16)
N1—C5—C6—C11	−88.4 (2)	C21—N3—C22—C23	−178.32 (17)

N1—C5—C6—C7	90.5 (2)	N3—C22—C23—C24	175.15 (16)
C11—C6—C7—C8	0.6 (3)	C22—C23—C24—C25	179.04 (18)
C5—C6—C7—C8	−178.28 (19)	C23—C24—C25—C26	−179.07 (17)
C6—C7—C8—C9	0.5 (3)	C24—C25—C26—C27	177.99 (19)
C7—C8—C9—C10	−0.7 (3)	C25—C26—C27—C28	−176.83 (19)
C8—C9—C10—C11	−0.2 (3)	C26—C27—C28—C29	174.8 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—HN2···O2^{i}	0.88	2.01	2.8449 (19)	157
N3—HN3···O4^{ii}	0.88	2.15	3.0211 (19)	172

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

Fig. 1

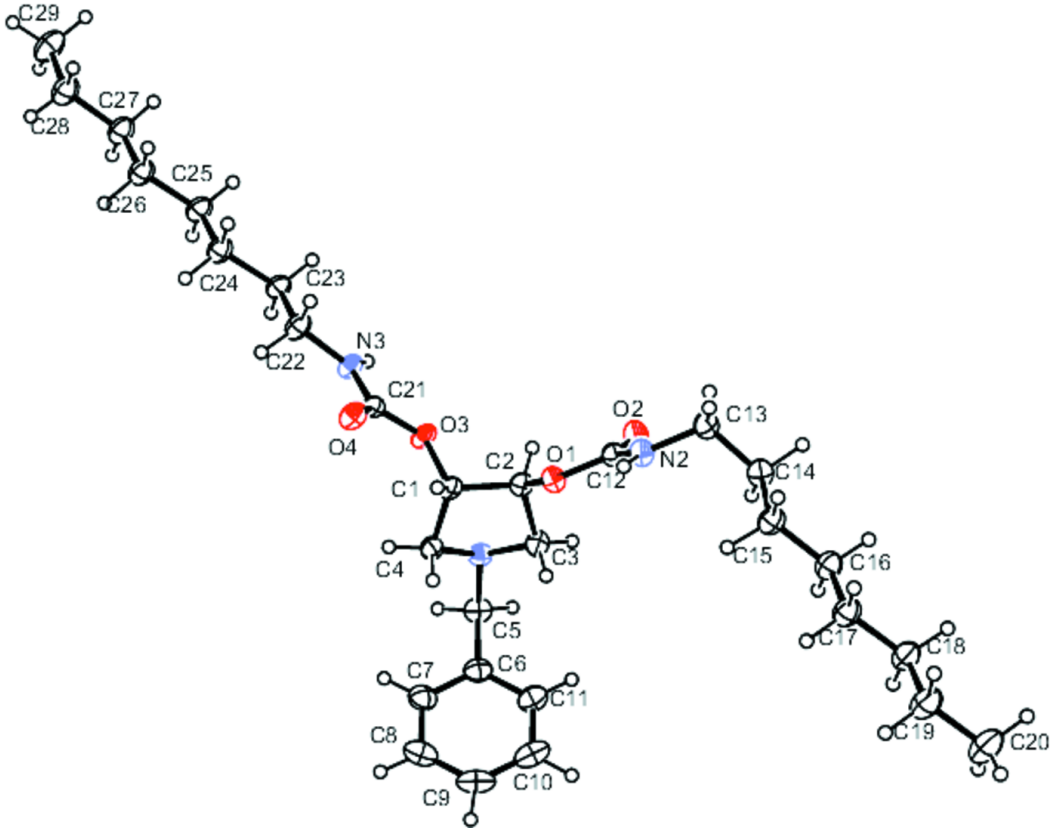
C29
C27
C28
C25
C26
C24
C23
N3
C22
C21
O4
O3
C2
O1
C12
O2
N2
C13
C14
C1
C4
C3
C15
C16
C5
C17
C18
C7
C6
C11
C8
C19
C20
C9
C10

Fig. 2