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Synthesis, crystal structure, Hirshfeld analysis and spectroscopic characterization of a norbornene-*N*-hydroxysuccinimide bifunctional linker

Gustavo Porrata-Caraballo, Victoria Y. Soto Díaz, José M. Rivera* and Dalice M. Piñero Cruz

Department of Chemistry, University of Puerto Rico, Rio Piedras Campus, San Juan, 00927, Puerto Rico. *Correspondence e-mail: jose.rivera151@upr.edu

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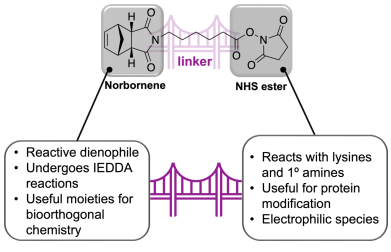
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The title compound, 2,5-dioxopyrrolidin-1-yl 6-[(3a*R*,4*S*,7*R*,7a*S*)-1,3-dioxo-1,3,3a,4,7,7a-hexahydro-2*H*-4,7-methanoisoindol-2-yl]hexanoate, $C_{19}H_{22}N_2O_6$, containing norbornene and *N*-hydroxysuccinimide ester moieties was synthesized by a Diels–Alder reaction followed by an esterification reaction. The compound yielded single crystals upon recrystallization in boiling aqueous solution, which crystallizes in the monoclinic space group $P2_1/n$ with one molecule as the asymmetric unit. According to the Hirshfeld surface analysis, it exhibits several intermolecular interactions including one H···H, one C···O, two C···H, and six O···H interactions. In the extended structure, two norbornene units of neighboring molecules interact *via* C—H···C contacts, while the same norbornene unit interacts through one of the H atoms of the bridge to one in connection to the succinimide. The title compound was also characterized through common spectroscopic methods such as NMR and IR spectroscopy.

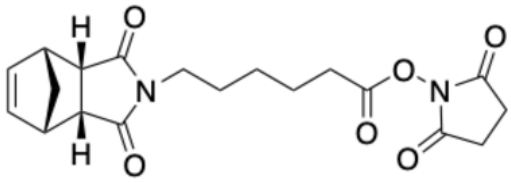
1. Chemical context

Chemical modification of proteins at specific sites is essential for advancing biological and biopharmaceutical applications (Abbas *et al.*, 2024; Haque *et al.*, 2021). The most targeted amino acid residues for protein modification include lysine and cysteine because of their nucleophilic nature (Wan *et al.*, 2024). Among the most widely employed reagents for protein modification are *N*-hydroxysuccinimide (NHS) esters, which readily react with lysine residues and primary amines to form stable amide linkages (Ward *et al.*, 2017). In parallel, norbornene derivatives have emerged as valuable dienophiles in bioorthogonal chemistry (Scinto *et al.*, 2024) because of their reaction with diene moieties. Norbornene-containing compounds efficiently participate in inverse electron-demand Diels–Alder (IEDDA) reactions with tetrazines because of their rapid reaction rates (García-Aznar *et al.*, 2022). This makes norbornene derivatives particularly useful for enabling rapid and selective conjugation processes in complex biological environments. The preparation of a norbornene-containing NHS ester was previously reported for the compound 5-norbornene-2-*N*-hydroxysuccinimide ester *via* an esterification reaction using 5-exo-norbornene carboxylic acid, *N*-hydroxysuccinimide with *N*,*N*′-dicyclohexylcarbodiimide as the coupling reagent in anhydrous dimethylformamide (Kim *et al.*, 2023). The incorporation of both NHS esters and norbornene moieties in a single molecule is therefore attractive for the preparation of bifunctional linkers capable of protein modification followed by subsequent bioorthogonal



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functionalization for numerous applications such as catalysis, protein labeling, and enzyme immobilization.



Herein, we report the crystal structure of the title compound, which bears both norbornene and NHS ester moieties. The crystal structure was determined to confirm the molecular connectivity and examine the structural features associated with the rigid bicyclic alkene and activated succinimidyl ester. The crystal structure and spectral data evidenced the formation of only the endo product for the Diels–Alder reaction, as well as successful synthesis of the succinimidyl ester from the esterification reaction.

2. Structural commentary

The title compound crystallizes in the primitive monoclinic space group $P2_1/n$ with one molecule as the asymmetric unit (Fig. 1). The molecule adopts an extended conformation, with the hexyl chain in an all-trans arrangement to minimize steric repulsion between the norbornene and the succinimidyl moiety. The norbornene is strained due to the rigid bicyclic bridgehead framework caused by the formation of the *endo* isomer in the Diels–Alder reaction, where the C9—C10—C11—C12 torsion angle is 0.2 (2)°, C9—C8—C13—C12 = 1.17 (15)° and C7—C8—C13—C14 of 1.40 (16)°. The five-membered (N2/C16–C19) succinimidyl ring deviates slight from planarity, evidenced by the C16—C17—C18—C19 torsion angle of 6.4 (2)°. This indicates a slightly twist conformation, likely driven by the steric hindrance of the N-substituted aliphatic chain and the crystal packing.

3. Supramolecular features

The title compound exhibits several intermolecular interactions, including one H···H van der Waals, one C···O, two C···H, and six non-classical C—H···O interactions (Table 1, Figs. 2, 3 and 4). A broader comparison with the Cambridge Structural Database (CSD) survey confirms that these non-classical C—H···O and hydrophobic packing features are highly characteristic, recurring motifs for this family of molecules. Structurally, the succinimidyl moiety lies gauche to the norbornene core, while a deviation in the linking carbon chain shifts the succinimidyl off-center. Despite this asymmetric conformation, the elongated molecules pack efficiently into parallel horizontal rows (Fig. 3), along the [010] direction. Two norbornene moieties of neighboring molecules interact *via* C10—H10···C7 and C11—H11···C14 contacts, while the same norbornene group interacts through one of the H atoms of the bridgehead to one in connection to the succinimidyl, C8—H8···H15*B*. Furthermore, the six interactions form a primary non-classical hydrogen-bonding network consolidating the three-dimensional structure. These weak interactions feature distances ranging from 2.46 to 2.71 Å and angles between 136 and 172°, with C5—H5*B*···O3 and C6—H6···O5 exhibiting high linearity (>170°), providing essential directional cohesion to the supramolecular assembly alongside broader van der Waals close contacts (Desiraju *et al.*, 1999). While C—H···O networks typically tolerate significant angular bending in the solid state (frequently falling between 130 and 160°), this distinct near-linear geometry approaches the ideal theoretical limit of 180° (Desiraju *et al.*, 1999).

Table 1
Short contacts of the title compound (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11···C14^{i}	0.93	2.87	3.407 (3)	118
C10—H10···C7^{i}	0.93	2.81	3.456 (3)	128
C18—H18*B*···O4^{i}	0.97	2.64	3.496 (3)	147
C18—H18*B*···O5^{i}	0.97	2.71	3.564 (3)	147
C11—H11···O3^{ii}	0.93	2.71	3.504 (2)	144
C5—H5*B*···O3^{ii}	0.97	2.46	3.564 (3)	171
C17—H17*A*···O2^{iii}	0.97	2.59	3.354 (2)	136
C6—H6*A*···O5^{iv}	0.97	2.71	3.676 (2)	172
C8—H8···H15*B*—C15^{v}	0.98/0.97^{a}	2.38	3.196 (2)/3.204 (2)^{a}	140/142^{a}
C19···O2^{vi}	-	-	3.124 (2)	

Note: (*a*). The two values correspond to the angles and lengths calculated relative to the individual tetrahedral centers (C8/C15) of the contact. Symmetry codes: (i) $x-1, y, z$; (ii) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$; (iii) $-x+1, -y+1, -z+1$; (iv) $-x+1, -y+2, -z+1$; (v) $-x+1, -y+2, -z+2$; (vi) $x-\frac{1}{2}, -y+\frac{3}{2}, z-\frac{1}{2}$.

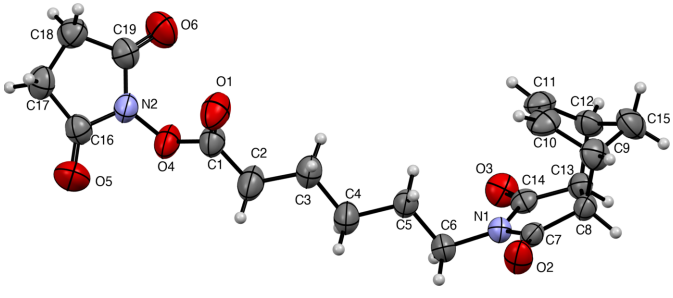


Figure 1
Crystal structure of the title compound with displacement ellipsoids at 50% probability level.

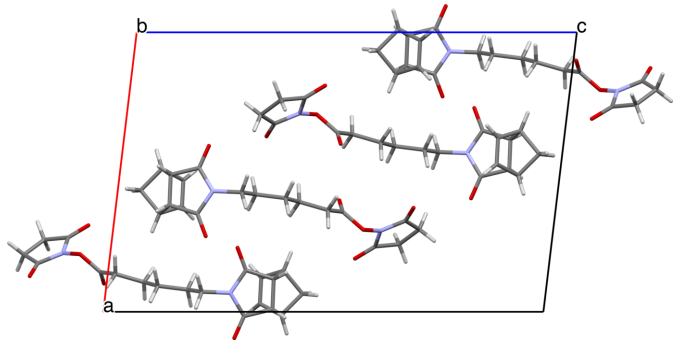


Figure 2
Crystal packing of title compound along the *b* axis.

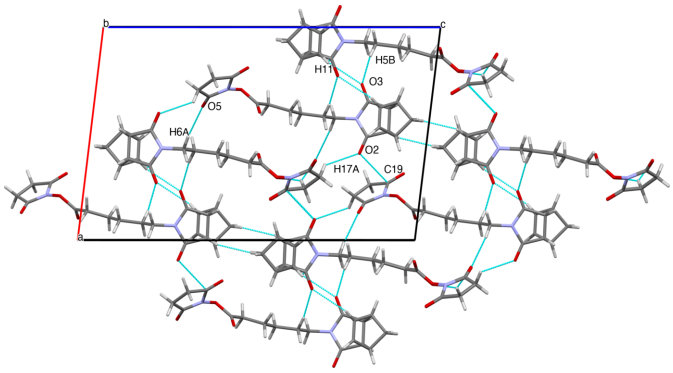


Figure 3
Crystal packing of the title compound, indicating the intermolecular O···H and C···O contacts, viewed along the *b* axis.

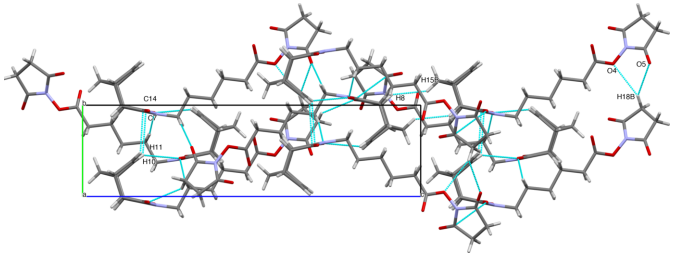


Figure 4
Crystal packing of the title compound, indicating the intermolecular C···H and C···O contacts, viewed along the *a* axis.

4. Hirshfeld surface analysis

The Hirshfeld surface of the title compound (Spackman & Jayatilaka, 2009) mapped over d_{norm} in the color range −0.19 and 1.44 a.u. is shown in Fig. 5 and the two-dimensional fingerprint plots (McKinnon *et al.*, 2007) in Fig. 6. The Hirshfeld surface analysis and fingerprint plots were generated using *TONTO* integrated in *CrystalExplorer17* (Turner *et al.*, 2017). The contributions to the Hirshfeld surface are shown in (Fig. 6*a–h*). The largest contributions to the Hirshfeld surface correspond to the H···H and the O···H interactions, of which the O···H interactions are the most prominent interactions, as shown by red spots in the d_{norm} plot and blue spots in the deformation density (Fig. 7).

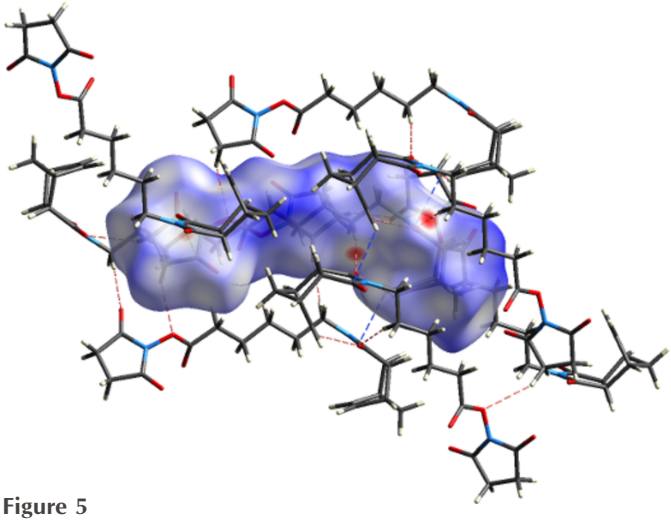


Figure 5
Hirshfeld surface mapped over d_{norm} with the three main intermolecular interactions in the crystal shown: H···H (green), O···H (red) and C···H (blue).

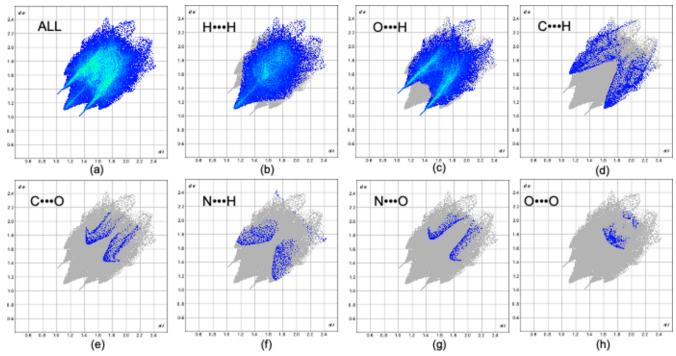


Figure 6
(*a*) The full two-dimensional fingerprint plot calculated for the title compound and those delineated into (*b*) H···H, (*c*) O···H/H···O, (*d*) C···H/H···C, (*e*) C···O/O···C, (*f*) N···H/H···N, (*g*) N···O/O···N and O···O contacts.

5. Database survey

A search of the Cambridge Structural Database (CSD Version 6.01, Nov. 2025, Feb. and May 2026 updates; Groom *et al.*, 2016) for the title compound revealed no hits. Two searches were done of the individual moieties with no ions, applying only organics and only single-crystal structure filters for which we obtained 18 hits for *N*-methyl-5-norbornene-2,3-dicarboximide and seven for the *N*-acetoxysuccinimide, including: *N*-methylbicyclo(2.2.1)hept-2-ene-endo-5,6-dicarboximide, which crystallizes in the *Pnma* space group (IFILIS; Birney *et al.* 2002) and 1-(acetyloxy) pyrrolidine-2,5-dione ($P2_1/c$, MODDAN; Lis 2019). The torsion angles exhibited by IFILIS deviate by almost 1°, showing more torsion than the title compound, while MODDAN shows no torsion due to not having any substituents. From both searches, 18 compounds crystallize in a monoclinic space group and nine of those have structurally similar substituents, *N*-(2-{[N6-(*t*-butoxycarbonyl)adeninyl]methylcarbonylamino}ethyl)-5-norbornene-endo-

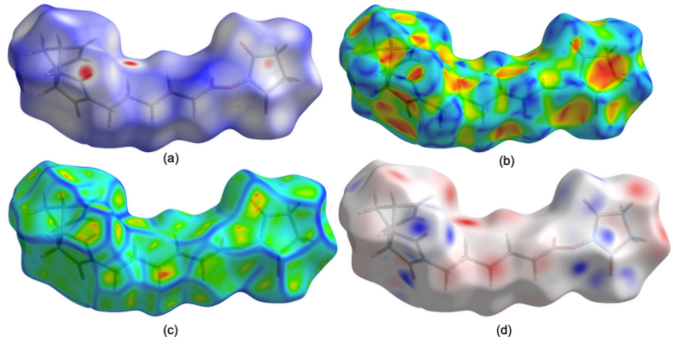


Figure 7
Hirshfeld surface for title compound mapped over (*a*) d_{norm}, (*b*) shape-index, (*c*) curvedness and (*d*) deformation density.

2,3-dicarboximide (ACUHOV; Davies, *et al.*, 2001), *N*-[7-(2-{[2-(3,5-dioxo-4-azatricyclo[5.2.1.02,6]dec-8-en-4-yl)ethyl]-amino}-2-oxoethyl)-6-oxo-6,7-dihydro-1*H*-purin-2-yl]-2-methylpropanamide (TIHNIJ; North *et al.*, 1998), (3-carbamoyl-1,4,5,6,7-pentamethyl-2-oxa-3-azabicyclo[2.2.1]hept-5-en-7-yl)methyl(3,5-dioxo-4-azatricyclo[5.2.1.02,6]dec-8-en-4-yl)-acetate and (3-carbamoyl-1,4,5,6,7-pentamethyl-2-oxa-3-azabicyclo[2.2.1]hept-5-en-7-yl)methyl(3,5-dioxo-4-azatricyclo-[5.2.1.02,6]dec-8-en-4-yl)acetate (WADVOP and WADVUV; Kensy *et al.*, 2016), 4,4′-ethane-1,2-diylbis(4-azatricyclo-[5.2.1.02,6]dec-8-ene-3,5-dione) (XUWWES; Fernández *et al.*, 2010), *N*-(*t*-butyloxycarbonyl)gabapentin succinimidyl ester (AWUXOF: Ananda *et al.*, 2003), 1,1′-(1,6-dioxohexane-1,6-diyl)bis(oxy)dipyrrolidine-2,5-dione (WALPEH; Zhang *et al.*, 2014) The first five compounds correspond to the norbornene moiety analogs, and the last two are *N*-acetoxysuccinimide analogs. ACUHOV is the only norbornene analog to show comparable torsion angles to the title compound, and WALPEH is the only *N*-acetoxysuccinimide analog. The norbornene analogs all showed at least one H···H short contact, specifically with the H atoms of the bridgehead, although the O···H short contacts were the most abundant. In the *N*-acetoxysuccinimide analogs, no H···H interactions were present, while the most predominant short contact was for the O···H substituent. Both groups of analogs exhibit similar short-contact behaviour to the title compound where the H···H interaction is at the norbornene bridgehead, and O···H interactions consolidate the crystal structure.

6. Synthesis and crystallization

The synthesis of the title compound was carried out by a two-step synthetic route (Fig. 8). The first step was a Diels–Alder reaction between 6-maleimidohexanoic acid with cyclopentadiene dissolved in toluene at 373 K for 1 h. The solvent was removed under reduced pressure to yield the norbornene carboxylic acid intermediate in an 86% yield. The second step was an esterification reaction between the norbornene carboxylic acid intermediate, *N*-hydroxysuccinimide with EDC·HCl [1-ethyl-3-(3-dimethylaminopropyl) carbodiimide hydrochloride] as the coupling reagent and 4-dimethylaminopyridine (DMAP) as the catalyst in dichloromethane at 298 K for 3 h. The solvent was removed under reduced pressure and the mixture was transferred to a separatory funnel and extracted using ethyl acetate as the organic phase. The organic phase was extracted with nanopure water (2 × 25 mL), 10% $NaHCO_3$ (2 × 25 mL), and brine (2 × 25 mL). The organic phase was collected and dried with anhydrous Na_2SO_4. The organic crude product was purified with flash chromatography using silica as the stationary phase and a

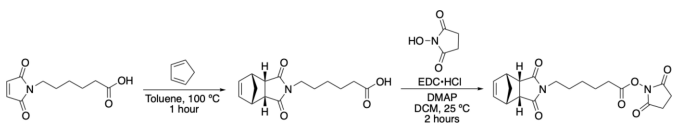


Figure 8
Synthetic route used for the title compound.

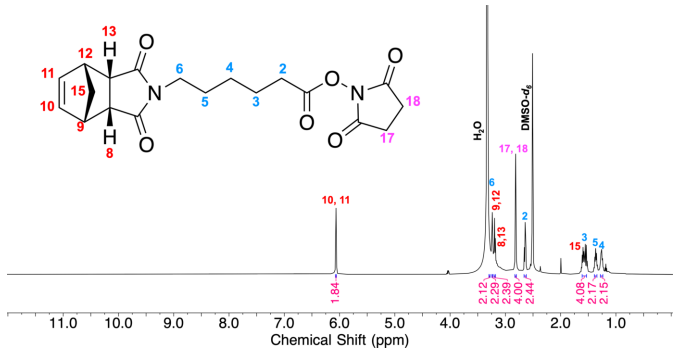


Figure 9
^{1}H NMR (500 MHz, DMSO-d_6, 298 K) of the title compound (10 m*M*).

mobile phase of hexanes/ethyl acetate. The compound eluted at 65% ethyl acetate and was recovered as a white powder. The title compound was recrystallized with boiling aqueous solution; cooling of the solution afforded white crystals (41% yield).

7. Spectroscopic characterization

NMR analyses were performed on a Bruker AV-500 MHz spectrometer using dimethyl sulfoxide-d_6 (DMSO-d_6) as the solvent. The solvent signals at 2.50 ppm and 39.5 ppm were used as internal standards for ^{1}H NMR (Fig. 9) and ^{13}C APT (Fig. 10), respectively. The characterization of the title compound is the following: white solid with a melting point of (380–384 K) ^{1}H NMR (500 MHz, DMSO-d_6) δ 6.06 (*s*, *J* = 1.9 Hz, 2H), 3.30 (*s*, 2H), 3.24 (*s*, 2H), 3.20 (*t*, *J* = 7.0 Hz, 2H), 2.81 (*s*, 4H), 2.64 (*t*, *J* = 7.3 Hz, 2H), 1.63–1.51 (*m*, 4H), 1.37 (*p*, *J* = 7.1 Hz, 2H), 1.26 (*ddd*, *J* = 15.2, 9.0, 6.0 Hz, 2H). ^{13}C NMR (126 MHz, DMSO) δ 177.91, 170.72, 169.36, 134.79, 52.25, 45.64, 44.71, 37.68, 30.49, 27.22, 25.91, 25.71, 24.21. The title compound is characterized by the appearance of a sharp singlet peak corresponding to the methylene protons of the *N*-hydroxysuccinimide moiety at 2.8 ppm. IR peaks (Fig. 11) at 3061.6 and 2943 cm^{-1} correspond to the ═C—H stretch of the alkene double bond in the norbornene moiety, and to the

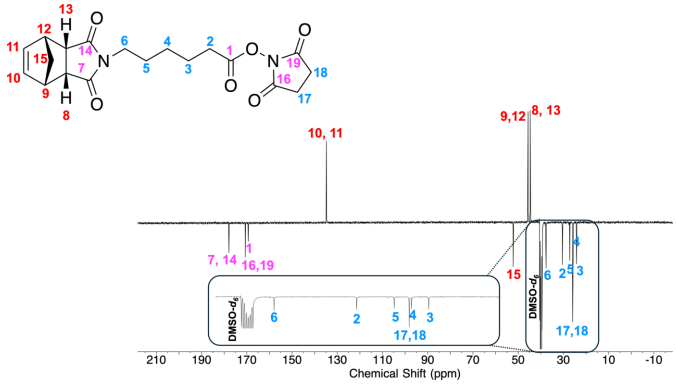


Figure 10
^{13}C APT NMR (126 MHz, DMSO-d_6, 298 K) of the title compound (10 m*M*).

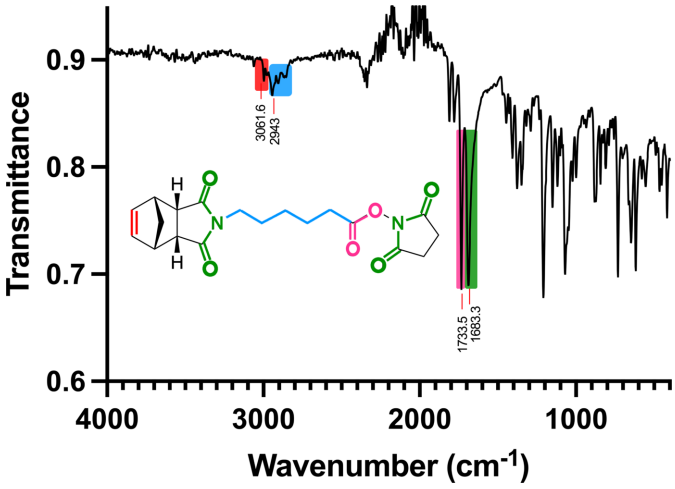


Figure 11
Infrared spectrum with diamond-attenuated total reflection (ATR-FTIR) of the title compound with 32 scans.

C—H bond stretch of sp^3 methylene carbon atoms between the norbornene and the NHS ester, respectively. IR peak at 1734 cm^{-1} corresponds to the *N*-hydroxysuccinimide ester bond formed in the esterification reaction and the peak at 1683 cm^{-1} corresponds to the two cyclic amides for both moieties.

8. Refinement

Crystal data, data collection and structure refinement details for the title compound are summarized in Table 2. H atoms were positioned geometrically (–H = 0.93–0.98 Å) and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C)$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{19}H_{22}N_2O_6$
M_r	374.38
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	298
a, b, c (Å)	14.1628 (3), 5.95502 (13), 22.2452 (5)
β (°)	96.859 (2)
V (Å^3)	1862.73 (8)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.84
Crystal size (mm)	0.13 × 0.05 × 0.04
Data collection	
Diffractometer	SuperNova, Single source at offset/far, HyPix3000
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2025)
T_{min}, T_{max}	0.951, 0.967
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8970, 3419, 2626
R_{int}	0.024
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.603
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.041, 0.118, 1.07
No. of reflections	3389
No. of parameters	244
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.18, −0.18

Computer programs: *CrysAlis PRO* (Rigaku OD, 2025), *SHELXS* (Sheldrick, 2008), *SHELXL2018/3* (Sheldrick, 2015) and *OLEX2* (Dolomanov *et al.*, 2009).

Acknowledgements

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Synthesis, crystal structure, Hirshfeld analysis and spectroscopic characterization of a norbornene-*N*-hydroxysuccinimide bifunctional linker

Gustavo Porrata-Caraballo, Victoria Y. Soto Díaz, José M. Rivera and Dalice M. Piñero Cruz

Computing details

2,5-Dioxopyrrolidin-1-yl 6-[(3a*R*,4*S*,7*R*,7a*S*)-1,3-dioxo-1,3,3a,4,7,7a-hexahydro-2*H*-4,7-methanoisoindol-2-yl]hexanoate

Crystal data

$C_{19}H_{22}N_2O_6$
M_r = 374.38
Monoclinic, $P2_1/n$
a = 14.1628 (3) Å
b = 5.95502 (13) Å
c = 22.2452 (5) Å
β = 96.859 (2)°
V = 1862.73 (8) Å^3
Z = 4
$F(000)$ = 792
D_x = 1.335 Mg m^{-3}
Melting point = 380.15–384.15 K
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 3918 reflections
θ = 3.5–68.1°
μ = 0.84 mm^{-1}
T = 298 K
Needle, clear colourless
0.13 × 0.05 × 0.04 mm

Data collection

SuperNova, Single source at offset/far, HyPix3000 diffractometer
Radiation source: micro-focus sealed X-ray tube, SuperNova (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.0000 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2025)
T_{min} = 0.951, T_{max} = 0.967
8970 measured reflections
3419 independent reflections
2626 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 68.3°, θ_{min} = 3.5°
h = −16→17
k = −7→3
l = −26→26

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.118
S = 1.07
3389 reflections
244 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0566P)^2 + 0.2435P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.43206 (9)	1.0920 (2)	0.78357 (6)	0.0419 (3)
O4	0.29365 (10)	0.5479 (2)	0.43138 (6)	0.0637 (4)
O2	0.59368 (8)	1.0621 (3)	0.80492 (6)	0.0636 (4)
O3	0.27093 (8)	1.0832 (2)	0.78773 (6)	0.0619 (4)
C7	0.51516 (11)	1.0558 (3)	0.82122 (7)	0.0430 (4)
N2	0.28981 (11)	0.3656 (3)	0.39232 (6)	0.0526 (4)
C13	0.38197 (11)	1.0189 (3)	0.87842 (7)	0.0445 (4)
H13	0.360668	1.135720	0.904744	0.053*
C14	0.35115 (11)	1.0686 (3)	0.81296 (7)	0.0432 (4)
C8	0.49067 (11)	1.0064 (3)	0.88385 (7)	0.0446 (4)
H8	0.520237	1.114889	0.913451	0.054*
O1	0.39885 (11)	0.3474 (3)	0.49381 (6)	0.0764 (4)
C1	0.35792 (14)	0.5190 (4)	0.48303 (8)	0.0573 (5)
C6	0.42811 (14)	1.1262 (3)	0.71837 (7)	0.0523 (4)
H6A	0.484975	1.204086	0.709503	0.063*
H6B	0.373579	1.218789	0.704296	0.063*
O6	0.19049 (11)	0.1520 (3)	0.44258 (7)	0.0822 (5)
C3	0.39375 (14)	0.7041 (4)	0.58446 (8)	0.0602 (5)
H3A	0.452880	0.620471	0.590554	0.072*
H3B	0.345607	0.617596	0.601742	0.072*
C16	0.32616 (13)	0.3812 (3)	0.33793 (8)	0.0535 (4)
C11	0.37733 (14)	0.6223 (3)	0.84969 (9)	0.0571 (5)
H11	0.334414	0.547199	0.821782	0.069*
C10	0.47119 (15)	0.6109 (3)	0.85378 (10)	0.0617 (5)
H10	0.506027	0.526175	0.829068	0.074*
C19	0.23388 (13)	0.1806 (3)	0.39998 (8)	0.0548 (4)
C5	0.42044 (13)	0.9026 (3)	0.68542 (7)	0.0504 (4)
H5A	0.477706	0.816135	0.697190	0.061*
H5B	0.367134	0.819218	0.697719	0.061*
C17	0.29518 (14)	0.1723 (4)	0.30439 (8)	0.0608 (5)
H17A	0.349868	0.089716	0.293781	0.073*
H17B	0.254304	0.208299	0.267508	0.073*
O5	0.37210 (12)	0.5367 (3)	0.32315 (7)	0.0835 (5)
C12	0.35285 (13)	0.7793 (3)	0.89857 (8)	0.0546 (4)
H12	0.288729	0.764881	0.910510	0.066*
C9	0.51169 (13)	0.7584 (3)	0.90527 (8)	0.0595 (5)
H9	0.577133	0.725859	0.922648	0.071*
C4	0.40697 (16)	0.9288 (4)	0.61716 (8)	0.0636 (5)
H4A	0.462002	1.004509	0.604585	0.076*

H4B	0.351730	1.022344	0.605525	0.076*
C15	0.43522 (15)	0.7330 (4)	0.94790 (9)	0.0711 (6)
H15A	0.433110	0.583244	0.964858	0.085*
H15B	0.440508	0.844497	0.979914	0.085*
C18	0.24188 (15)	0.0352 (3)	0.34614 (9)	0.0613 (5)
H18A	0.179308	−0.004516	0.326417	0.074*
H18B	0.276297	−0.101611	0.358068	0.074*
C2	0.36440 (17)	0.7336 (4)	0.51750 (8)	0.0705 (6)
H2A	0.302998	0.807731	0.511642	0.085*
H2B	0.409976	0.830900	0.501148	0.085*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0466 (7)	0.0435 (7)	0.0357 (6)	−0.0034 (6)	0.0059 (5)	−0.0027 (5)
O4	0.0825 (9)	0.0574 (7)	0.0464 (7)	0.0115 (7)	−0.0115 (6)	−0.0094 (6)
O2	0.0457 (7)	0.0918 (10)	0.0552 (7)	−0.0134 (6)	0.0139 (6)	−0.0105 (7)
O3	0.0466 (7)	0.0674 (9)	0.0699 (8)	0.0131 (6)	−0.0005 (6)	0.0030 (6)
C7	0.0436 (8)	0.0444 (8)	0.0412 (8)	−0.0078 (7)	0.0060 (6)	−0.0099 (7)
N2	0.0632 (9)	0.0540 (8)	0.0395 (7)	−0.0012 (7)	0.0012 (6)	−0.0075 (6)
C13	0.0487 (9)	0.0449 (8)	0.0419 (8)	−0.0018 (7)	0.0135 (7)	−0.0076 (7)
C14	0.0448 (9)	0.0356 (8)	0.0496 (9)	0.0039 (6)	0.0066 (7)	−0.0044 (7)
C8	0.0455 (8)	0.0514 (9)	0.0364 (8)	−0.0083 (7)	0.0028 (6)	−0.0072 (7)
O1	0.0905 (10)	0.0689 (9)	0.0637 (8)	0.0186 (8)	−0.0164 (7)	−0.0089 (7)
C1	0.0651 (11)	0.0623 (11)	0.0425 (9)	0.0044 (9)	−0.0027 (8)	−0.0040 (8)
C6	0.0655 (11)	0.0546 (10)	0.0366 (8)	−0.0071 (8)	0.0058 (7)	0.0005 (7)
O6	0.0842 (10)	0.1017 (12)	0.0668 (9)	−0.0088 (9)	0.0339 (8)	−0.0021 (8)
C3	0.0656 (11)	0.0702 (12)	0.0442 (9)	−0.0014 (10)	0.0037 (8)	−0.0094 (9)
C16	0.0511 (9)	0.0624 (11)	0.0463 (9)	0.0003 (8)	0.0035 (7)	0.0034 (8)
C11	0.0645 (11)	0.0379 (8)	0.0659 (11)	−0.0080 (8)	−0.0052 (9)	0.0041 (8)
C10	0.0694 (12)	0.0404 (9)	0.0735 (13)	0.0084 (8)	0.0008 (9)	0.0013 (8)
C19	0.0521 (10)	0.0623 (11)	0.0503 (10)	0.0035 (8)	0.0074 (8)	0.0020 (8)
C5	0.0516 (9)	0.0585 (10)	0.0412 (9)	−0.0070 (8)	0.0055 (7)	−0.0061 (8)
C17	0.0606 (11)	0.0709 (12)	0.0524 (10)	0.0026 (9)	0.0131 (8)	−0.0098 (9)
O5	0.0919 (11)	0.0795 (11)	0.0816 (10)	−0.0235 (9)	0.0206 (9)	0.0067 (8)
C12	0.0533 (10)	0.0591 (11)	0.0520 (10)	−0.0121 (8)	0.0080 (8)	0.0070 (8)
C9	0.0520 (10)	0.0667 (11)	0.0561 (10)	0.0019 (9)	−0.0086 (8)	0.0114 (9)
C4	0.0784 (13)	0.0701 (12)	0.0413 (9)	−0.0011 (10)	0.0029 (9)	−0.0059 (9)
C15	0.0772 (13)	0.0818 (14)	0.0521 (10)	−0.0140 (11)	−0.0016 (9)	0.0219 (10)
C18	0.0636 (11)	0.0590 (11)	0.0622 (11)	−0.0023 (9)	0.0112 (9)	−0.0086 (9)
C2	0.0918 (15)	0.0670 (12)	0.0487 (10)	0.0078 (11)	−0.0080 (10)	−0.0103 (9)

Geometric parameters (Å, º)

N1—C7	1.377 (2)	C16—C17	1.490 (3)
N1—C14	1.392 (2)	C16—O5	1.200 (2)
N1—C6	1.459 (2)	C11—H11	0.9300
O4—N2	1.3875 (19)	C11—C10	1.323 (3)

O4—C1	1.389 (2)	C11—C12	1.506 (3)
O2—C7	1.2107 (19)	C10—H10	0.9300
O3—C14	1.209 (2)	C10—C9	1.502 (3)
C7—C8	1.505 (2)	C19—C18	1.493 (3)
N2—C16	1.373 (2)	C5—H5A	0.9700
N2—C19	1.379 (2)	C5—H5B	0.9700
C13—H13	0.9800	C5—C4	1.516 (2)
C13—C14	1.499 (2)	C17—H17A	0.9700
C13—C8	1.532 (2)	C17—H17B	0.9700
C13—C12	1.565 (2)	C17—C18	1.505 (3)
C8—H8	0.9800	C12—H12	0.9800
C8—C9	1.569 (3)	C12—C15	1.528 (3)
O1—C1	1.185 (2)	C9—H9	0.9800
C1—C2	1.488 (3)	C9—C15	1.530 (3)
C6—H6A	0.9700	C4—H4A	0.9700
C6—H6B	0.9700	C4—H4B	0.9700
C6—C5	1.517 (2)	C15—H15A	0.9700
O6—C19	1.202 (2)	C15—H15B	0.9700
C3—H3A	0.9700	C18—H18A	0.9700
C3—H3B	0.9700	C18—H18B	0.9700
C3—C4	1.524 (3)	C2—H2A	0.9700
C3—C2	1.508 (3)	C2—H2B	0.9700

H11···C14	2.869	H5B···O3	2.461
H10···C7	2.81	O2···H17A	2.588
H18B···O4	2.642	H6A···O5	2.713
H18B···O5	2.708	H8···H15B	2.383
H11···O3	2.709	C19···O2	3.124

C7—N1—C14	112.97 (13)	O6—C19—N2	124.49 (18)
C7—N1—C6	123.69 (14)	O6—C19—C18	130.18 (19)
C14—N1—C6	123.00 (14)	C6—C5—H5A	109.0
N2—O4—C1	113.19 (14)	C6—C5—H5B	109.0
N1—C7—C8	108.59 (13)	H5A—C5—H5B	107.8
O2—C7—N1	124.18 (15)	C4—C5—C6	112.76 (16)
O2—C7—C8	127.22 (15)	C4—C5—H5A	109.0
C16—N2—O4	120.54 (15)	C4—C5—H5B	109.0
C16—N2—C19	116.36 (15)	C16—C17—H17A	110.4
C19—N2—O4	121.85 (14)	C16—C17—H17B	110.4
C14—C13—H13	111.4	C16—C17—C18	106.45 (15)
C14—C13—C8	105.05 (12)	H17A—C17—H17B	108.6
C14—C13—C12	113.71 (13)	C18—C17—H17A	110.4
C8—C13—H13	111.4	C18—C17—H17B	110.4
C8—C13—C12	103.29 (13)	C13—C12—H12	116.2
C12—C13—H13	111.4	C11—C12—C13	105.54 (13)
N1—C14—C13	108.38 (13)	C11—C12—H12	116.2
O3—C14—N1	123.73 (16)	C11—C12—C15	100.63 (16)
O3—C14—C13	127.87 (15)	C15—C12—C13	99.53 (14)

C7—C8—C13	104.96 (13)	C15—C12—H12	116.2
C7—C8—H8	111.5	C8—C9—H9	116.2
C7—C8—C9	114.14 (14)	C10—C9—C8	106.12 (14)
C13—C8—H8	111.5	C10—C9—H9	116.2
C13—C8—C9	102.83 (13)	C10—C9—C15	100.40 (16)
C9—C8—H8	111.5	C15—C9—C8	99.13 (15)
O4—C1—C2	108.28 (16)	C15—C9—H9	116.2
O1—C1—O4	122.06 (17)	C3—C4—H4A	109.1
O1—C1—C2	129.65 (18)	C3—C4—H4B	109.1
N1—C6—H6A	109.6	C5—C4—C3	112.43 (17)
N1—C6—H6B	109.6	C5—C4—H4A	109.1
N1—C6—C5	110.48 (14)	C5—C4—H4B	109.1
H6A—C6—H6B	108.1	H4A—C4—H4B	107.9
C5—C6—H6A	109.6	C12—C15—C9	94.19 (14)
C5—C6—H6B	109.6	C12—C15—H15A	112.9
H3A—C3—H3B	107.9	C12—C15—H15B	112.9
C4—C3—H3A	109.2	C9—C15—H15A	112.9
C4—C3—H3B	109.2	C9—C15—H15B	112.9
C2—C3—H3A	109.2	H15A—C15—H15B	110.3
C2—C3—H3B	109.2	C19—C18—C17	105.79 (16)
C2—C3—C4	111.90 (17)	C19—C18—H18A	110.6
N2—C16—C17	105.43 (15)	C19—C18—H18B	110.6
O5—C16—N2	124.46 (18)	C17—C18—H18A	110.6
O5—C16—C17	130.11 (17)	C17—C18—H18B	110.6
C10—C11—H11	126.3	H18A—C18—H18B	108.7
C10—C11—C12	107.36 (17)	C1—C2—C3	113.70 (18)
C12—C11—H11	126.3	C1—C2—H2A	108.8
C11—C10—H10	125.9	C1—C2—H2B	108.8
C11—C10—C9	108.14 (18)	C3—C2—H2A	108.8
C9—C10—H10	125.9	C3—C2—H2B	108.8
N2—C19—C18	105.33 (15)	H2A—C2—H2B	107.7

N1—C7—C8—C13	−0.19 (17)	C8—C9—C15—C12	59.45 (16)
N1—C7—C8—C9	111.62 (15)	O1—C1—C2—C3	−27.2 (4)
N1—C6—C5—C4	174.73 (15)	C1—O4—N2—C16	−109.14 (19)
O4—N2—C16—C17	−170.63 (14)	C1—O4—N2—C19	84.1 (2)
O4—N2—C16—O5	8.8 (3)	C6—N1—C7—O2	4.7 (3)
O4—N2—C19—O6	−6.2 (3)	C6—N1—C7—C8	−174.72 (14)
O4—N2—C19—C18	174.53 (15)	C6—N1—C14—O3	−3.0 (2)
O4—C1—C2—C3	154.03 (18)	C6—N1—C14—C13	175.72 (14)
O2—C7—C8—C13	−179.56 (17)	C6—C5—C4—C3	−176.77 (16)
O2—C7—C8—C9	−67.7 (2)	O6—C19—C18—C17	172.8 (2)
C7—N1—C14—O3	−176.56 (15)	C16—N2—C19—O6	−173.45 (18)
C7—N1—C14—C13	2.19 (17)	C16—N2—C19—C18	7.3 (2)
C7—N1—C6—C5	87.73 (19)	C16—C17—C18—C19	6.4 (2)
C7—C8—C9—C10	−47.17 (19)	C11—C10—C9—C8	−70.26 (19)
C7—C8—C9—C15	−150.87 (14)	C11—C10—C9—C15	32.5 (2)
N2—O4—C1—O1	−5.6 (3)	C11—C12—C15—C9	49.18 (17)

N2—O4—C1—C2	173.30 (16)	C10—C11—C12—C13	70.28 (18)
N2—C16—C17—C18	−2.3 (2)	C10—C11—C12—C15	−32.85 (19)
N2—C19—C18—C17	−8.0 (2)	C10—C9—C15—C12	−48.93 (18)
C13—C8—C9—C10	65.92 (17)	C19—N2—C16—C17	−3.2 (2)
C13—C8—C9—C15	−37.78 (15)	C19—N2—C16—O5	176.21 (19)
C13—C12—C15—C9	−58.76 (17)	O5—C16—C17—C18	178.3 (2)
C14—N1—C7—O2	178.15 (16)	C12—C13—C14—N1	−114.37 (15)
C14—N1—C7—C8	−1.24 (18)	C12—C13—C14—O3	64.3 (2)
C14—N1—C6—C5	−85.11 (19)	C12—C13—C8—C7	120.83 (14)
C14—C13—C8—C7	1.40 (16)	C12—C13—C8—C9	1.17 (15)
C14—C13—C8—C9	−118.27 (14)	C12—C11—C10—C9	0.2 (2)
C14—C13—C12—C11	45.24 (18)	C4—C3—C2—C1	175.54 (18)
C14—C13—C12—C15	149.19 (15)	C2—C3—C4—C5	170.56 (17)
C8—C13—C14—N1	−2.15 (17)	C9—C10—C11—C12	0.2 (2)
C8—C13—C14—O3	176.52 (16)	C9—C8—C13—C12	1.17 (15)
C8—C13—C12—C11	−68.03 (16)	C7—C8—C13—C14	1.40 (16)
C8—C13—C12—C15	35.91 (16)	C16—C17—C18—C19	6.4 (2)

Hydrogen-bond geometry (Å, º)

Note: (*a*). The two values correspond to the angles and lengths calculated relative to the individual tetrahedral centers (C8/C15) of the contact.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11···C14^{i}	0.93	2.87	3.407 (3)	118
C10—H10···C7^{i}	0.93	2.81	3.456 (3)	128
C18—H18*B*···O4^{i}	0.97	2.64	3.496 (3)	147
C18—H18*B*···O5^{i}	0.97	2.71	3.564 (3)	147
C11—H11···O3^{ii}	0.93	2.71	3.504 (2)	144
C5—H5*B*···O3^{ii}	0.97	2.46	3.564 (3)	171
C17—H17*A*···O2^{iii}	0.97	2.59	3.354 (2)	136
C6—H6*A*···O5^{iv}	0.97	2.71	3.676 (2)	172
C8—H8···C15^{v}	0.98	2.38	3.196 (2)	140
C19···O2^{vi}	-	-	3.124 (2)	?

Symmetry codes: (i) *x*−1, *y*, *z*; (ii) −*x*+1/2, *y*−1/2, −*z*+3/2; (iii) −*x*+1, −*y*+1, −*z*+1; (iv) −*x*+1, −*y*+2, −*z*+1; (v) −*x*+1, −*y*+2, −*z*+2; (vi) *x*−1/2, −*y*+3/2, *z*−1/2.

Short contacts of the title compound (Å)

Contact	**Length (Å)**	**Symm. op. 1**	**Symm. op. 2**
C11—H11···C14	2.869	*x*, *y*, *z*	*x*, -1 + *y*, *z*
C10—H10···C7	2.810	*x*, *y*, *z*	*x*, -1 + *y*, *z*
C18—H18*B*···O4	2.642	*x*, *y*, *z*	*x*, -1 + *y*, *z*
C18—H18*B*···O5	2.708	*x*, *y*, *z*	*x*, -1 + *y*, *z*
C11—H11···O3	2.709	*x*, *y*, *z*	1/2 - *x*, -1/2 + *y*, 3/2 - *z*
C5—H5*B*···O3	2.461	*x*, *y*, *z*	1/2 - *x*, -1/2 + *y*, 3/2 - *z*
C17—H17*A*···O2	2.588	*x*, *y*, *z*	1 - *x*, 1 - *y*, 1 - *z*
C6—H6*A*···O5	2.713	*x*, *y*, *z*	1 - *x*, 2 - *y*, 1 - *z*
C8—H8···H15*B*—C15	2.383	*x*, *y*, *z*	1 - *x*, 2 - *y*, 2 - *z*
C19···O2	3.124	*x*, *y*, *z*	-1/2 + *x*, 3/2 - *y*, -1/2 + *z*