Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Syntheses and structures of five Aib (2-amino-isobutyric acid) derivatives prepared *via* azirine coupling

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Received 4 November 2026
Accepted 5 November 2026

Edited by W. T. A. Harrison, University of Aberdeen, United Kingdom

‡ Experiments carried out as part of a PhD thesis, University of Zurich, 2005.

Keywords: crystal structure; Aib derivatives; Aib molecular conformations; α,α-disubstituted α-amino acids; azirine/oxalone coupling method.

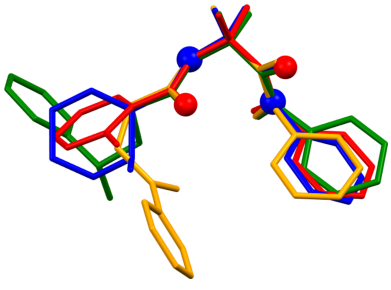
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A series of derivatives of the α,α-disubstituted amino acid Aib (2-amino-isobutyric acid) were prepared *via* the 'azirine coupling' method in which a carboxylic acid reacts with 2,2-dimethyl-3-(*N*-methyl-*N*-phenylamino)-2*H*-azirine to give an *N*-acylated 2-aminoisobutyric amide. For example, salicylic acid and (1*R*,2*S*)-2-hydroxycyclohexanoic acid, respectively, yielded 2-hydroxy-*N*-[1-methyl-1-(*N*-methyl-*N*-phenylcarbamoyl)ethyl]benzamide, $C_{18}H_{20}N_2O_3$, and (1*R*,2*S*)-2-hydroxy-*N*-[1-methyl-1-(*N*-methyl-*N*-phenylcarbamoyl)ethyl]-cyclohexanecarboxamide, $C_{18}H_{26}N_2O_3$. The analogous reactions with 4-hydroxy-4-phenylbutanoic acid and 4-oxo-4-phenylbutanoic acid, respectively, led to *rac*-4-hydroxy-4-phenyl-*N*-[1-methyl-1-(*N*-methyl-*N*-phenylcarbamoyl)ethyl] butanamide, $C_{21}H_{26}N_2O_3$, and *N*-[1-methyl-1-(*N*-methyl-*N*-phenylcarbamoyl)ethyl]-4-oxo-4-phenylbutanamide, $C_{21}H_{24}N_2O_3$. In the case of the reaction with 4-benzyloxy-3,3-dimethylbutanoic acid, the primarily formed 3-benzyloxy-2,2-dimethyl-*N*-[1-methyl-1-(*N*-methyl-*N*-phenylcarbamoyl)ethyl]butanamide was selectively hydrolysed to give 2-[(4-benzyloxy-3,3-dimethylbutanoyl)-amino]-2-methylpropanoic acid, $C_{17}H_{25}NO_4$. The crystal structures of these five compounds have been determined and the conformations of the molecules compared with each other and with those of related structures reported in the literature. In general, the molecular conformations of the backbone segment from the secondary amide group through to the terminal –N(Me)Ph group are very similar in all known structures of molecules of this kind and show a progressive turn between the two amide groups. In contrast, and unusually, the related backbone in the propanoic acid-based molecule, whose structure is reported here, is almost planar.

1. Chemical context

The influence of α,α-disubstituted α-amino acids – the most simple and well-known example is 2-aminoisobutyric acid (also called 2-amino-2-methylpropanoic acid; $C_4H_9NO_2$; Aib) – on the structure of peptides and proteins, *i.e.* the strong preference of the α-helical or 3_{10}-helical conformation, is well known (*e.g.* Nagaraj & Balaram, 1981; Karle & Balaram, 1990; Pavone *et al.*, 1990; Marshall *et al.*, 1990; Toniolo & Benedetti, 1991; De Filippis *et al.*, 1998; Banerjee *et al.*, 2009) and studies of Aib-containing peptides and their role in the formation of ion-channels through membranes are still of current interest (*e.g.* Tsuji *et al.*, 2018; Locarno *et al.*, 2020; Beyer *et al.*, 2020; Zieleniewski *et al.*, 2020; Terada *et al.*, 2020; Law *et al.*, 2024; Yamaguchi *et al.*, 2025; Yuan *et al.*, 2025). A typical class of such peptides are the so-called peptaibiotics, *i.e.* fungal peptide antibiotics with a high proportion of Aib (Toniolo & Brückner, 2009; Stoppacher *et al.*, 2013; Pereira-Dias *et al.*, 2023). A prominent group among them are the peptaibols (*e.g.* **1a**–**e**, Fig. 1), Aib-containing peptides with 5–20 amino acids,



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Ac-Aib-Pro-Aib-Ala-Aib-Ala-Gln-Aib-Val-Aib-Gly-Leu-Aib-Pro-Val-Aib-Aib-Glu-Gln-Pheol

1a

Ac-Aib-Gly-Aib-Leu-Aib-Gln-Aib-Aib-Aib-Ala-Ala- Aib-Pro-Leu-Aib-D-Iva-Gln-Valol

1b

Ac-Phe-Aib-Aib-Aib-D-Iva-Gly-Leu-Aib-Aib-Hyp-Gln-D-Iva-Hyp-Aib-Pro-Pheol

1c

Ac-Trp-Ile-Gln-Aib-Ile-Thr-Aib-Val-Aib-Hyp-Gln-Aib-Hyp-Aib-Pro-Pheol

1d

Ac-Aib-Gln-Val-Val-Aib-Pro-Leu-Leu-Aib-Pro-Leuol

1e

Figure 1
Peptaibols alamethicin F30 (**1a**), trichotoxin A-50(G) (**1b**), antiamoebin I (**1c**), zervamicin II-2 (**1d**) and hypomurocin A1 (**1e**).

with an acylated *N*-terminus, and a C-terminal 2-amino alcohol (Chugh & Wallace, 2001; Whitmore & Wallace, 2006; Das *et al.*, 2018; Hou *et al.*, 2022). The best-known example is alamethicin F30 (**1a**, Fig. 1) (Nagaraj & Balaram, 1981; Mathew & Balaram, 1983; Leitgeb *et al.*, 2007; Nagao *et al.*, 2015).

In our earlier studies on the use of 3-amino-2,2-dimethyl-2*H*-azirines (*e.g.* **2**) as Aib synthons in the preparation of heterocycles and peptides (Heimgartner, 1979, 1986, 1991) it was shown that their reaction with carboxylic acids leads to *N*-acylated Aib-amides **3** (Fig. 2). Their selective hydrolysis yields the corresponding acids **4**, which, *via* the intermediate oxazolones **5**, can be coupled with amino acid esters **6** to give peptides **7**. This reaction sequence is called the 'azirine/oxazolone method'.

Using this methodology, we have prepared a series of peptaibol segments, *e.g.* of alamethicin F30 (Wipf & Heimgartner, 1990), trichotoxin A-50(G) (Altherr *et al.*, 2007), antiamoebin I (Blaser *et al.*, 2013) and zervamicin II-2 (Pradeille & Heimgartner, 2003). Furthermore, the first total syntheses of hypomurocin A1 (Pradeille *et al.*, 2005), A3 and A5 (Pradeille *et al.*, 2012) have been carried out with the 'azirine/oxazolone method'.

Moreover, we have shown in our syntheses of linear and cyclic depsipeptides containing Aib units (Obrecht & Heimgartner, 1990; Villalgordo & Heimgartner, 1997; Koch *et al.*, 2000, 2001; Iliev *et al.*, 2003, 2006; Köttgen *et al.*, 2006, 2009; Linden *et al.*, 2020) that carboxylic acids bearing a free hydroxy group react smoothly with aminoazirines of type **2** to give the corresponding *N*-acylated Aib-amides **3** with *R* being

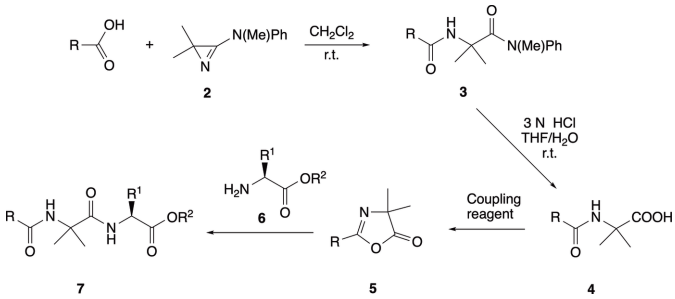


Figure 2
Synthesis of Aib-containing peptides by the 'azirine/oxazolone method'.

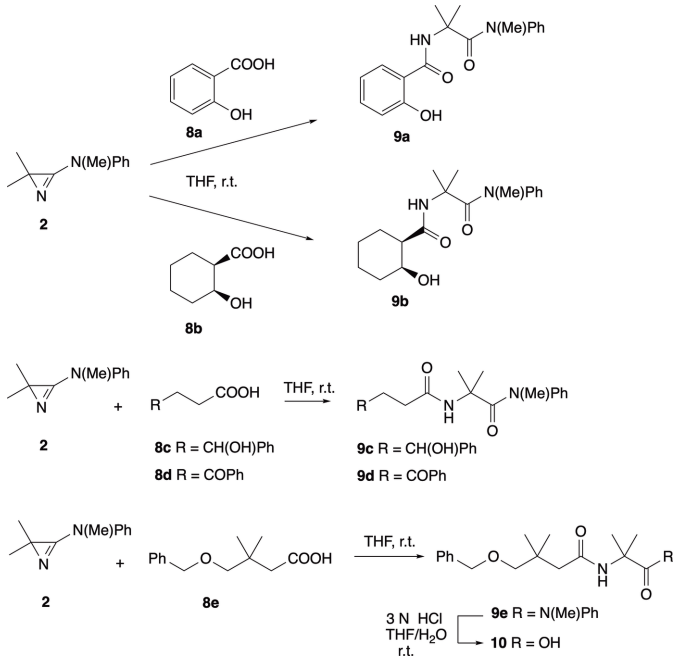


Figure 3
Reactions of carboxylic acids **8** containing oxygen functions with 3-amino-2,2-dimethyl-2*H*-azirine **2**.

an OH-substituted residue exclusively. As model reactions, we transformed salicylic acid (**8a**) and (1*R*,2*S*)-2-hydroxycyclohexanoic acid (**8b**), respectively, by treatment with **2** in tetrahydrofuran (THF) at room temperature (r.t.) into 2-hydroxy-*N*-[1-methyl-1-(*N*-methyl-*N*-phenylcarbamoyl)ethyl]-benzamide (**9a**) (Villalgordo & Heimgartner, 1997; Iliev *et al.*, 2006) and (1*R*,2*S*)-2-hydroxy-*N*-[1-methyl-1-(*N*-methyl-*N*-phenylcarbamoyl)ethyl]cyclohexanecarboxamide (**9b**) (Fig. 3) (Iliev *et al.*, 2006), respectively. Under the same conditions, 4-hydroxy-4-phenylbutanoic acid (**8c**) reacted with the aminoazirine **2** to give the previously unreported *rac*-4-hydroxy-4-phenyl-*N*-[1-methyl-1-(*N*-methyl-*N*-phenylcarbamoyl)ethyl]-butanamide (**9c**).

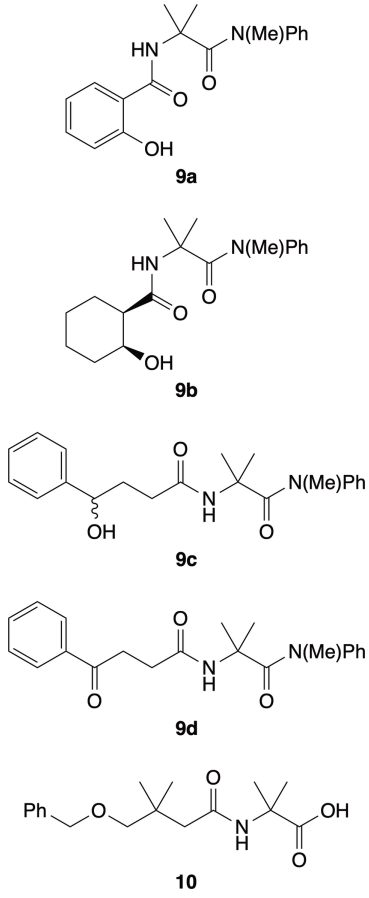
Different approaches to *N*-acylated Aib-amides **3** with a hydroxy group in the residue *R* are the selective reduction of a carbonyl group or the deprotection of a protected hydroxy group, such as a benzyloxy group, of the corresponding precursors. For this reason, we reacted aminoazirine **2** with 4-oxo-4-phenylbutanoic acid (**8d**) under standard conditions (THF, r.t.) and obtained the previously unreported 4-oxo-4-phenyl-*N*-[1-methyl-1-(*N*-methyl-*N*-phenylcarbamoyl)ethyl]-butanamide (**9d**) as a single product in high yield (Fig. 3). Analogously, the reaction of **2** with 4-benzyloxy-3,3-dimethylbutanoic acid (**8e**) yielded 3-benzyloxy-2,2-dimethyl-*N*-[1-methyl-1-(*N*-methyl-*N*-phenylcarbamoyl)ethyl]butanamide (**9e**), which was selectively hydrolysed by treatment with 3N HCl in THF/H_2O at r.t. (*cf.* Iliev *et al.*, 2003) to give 2-methyl-2-[(4-benzyloxy-3,3-dimethyl)butanoyl]amino propanoic acid (**10**).

The crystal structures of **9a**–**9d** and **10** were determined and are discussed here along with a comparison of the molecular backbone conformations of these and other related

Table 1
Selected torsion angles (°) for **9a**–**9d** and **10**.

	9a	9b	9c	9d	10
C1—N2—C3—C4	54.63 (13)	49.6 (2)	46.96 (14)	49.4 (4)	–174.7 (3)
N2—C3—C4—N5	44.81 (13)	49.1 (2)	53.65 (13)	47.0 (3)	
N2—C3—C4—O5					–179.1 (3)
C3—C4—N5—C6	−175.88 (9)	–171.38 (16)	173.10 (9)	–168.1 (2)	
C4—N5—C6—C7	70.39 (13)	60.6 (3)	94.72 (13)	82.5 (3)	
C3—N2—C1—C12	176.84 (9)	178.55 (17)	177.94 (9)	177.5 (3)	177.5 (3)
N2—C1—C12—C13	–165.66 (10)	139.01 (18)	87.36 (13)	160.7 (3)	–81.3 (4)
O1—C1—C12—C13	13.15 (15)	–41.7 (3)	–90.96 (14)	–20.4 (5)	99.2 (3)

compounds whose crystal structures are included in the Cambridge Structural Database (CSD, Version 6.01, update of September 2026; Groom *et al.*, 2016). The patterns of inter- and intramolecular O—H···O and N—H···O hydrogen bonds in these structures are also compared.



2. Structural commentary

The molecules of compounds **9a**–**d** all contain one set of linked secondary and tertiary amide groups with the tertiary amide having the terminal *N*-methyl-*N*-phenyl [–N(Me)Ph] substituent. In their crystal structures, the molecular conformations (Figs. 4 and 5) of the backbone of the molecule from the secondary amide group through to the terminal –N(Me)Ph group are very similar, as exemplified by their overlay shown in Fig. 6. In particular, the adjacent C1—N2—C3—C4 and N2—C3—C4—N5 torsion angles both have the same sign and similar values across all structures (Table 1) with a range of 46.96 (14)–54.63 (13) and 44.81 (13)–53.65 (13)°, respectively. This indicates the presence of a progressive turn between the alignments of the two amide groups. In addition, the plane of the terminal phenyl ring of the –N(Me)Ph group is consistently steeply inclined or even nearly perpendicular to that of its parent amide group. The C4—N5—C6—C7 torsion angles range from 60.6 (3) to 94.72 (13)°. The conformations of the substituents on the other side of the secondary amide group of the molecules of **9a**–**d** vary considerably, partly because of different chain lengths. Even in the structures of **9a** and **9b**, where the only difference is the replacement of the hydroxyphenyl ring in the former by a hydroxycyclohexane ring in the latter, these two rings are oriented quite differently with

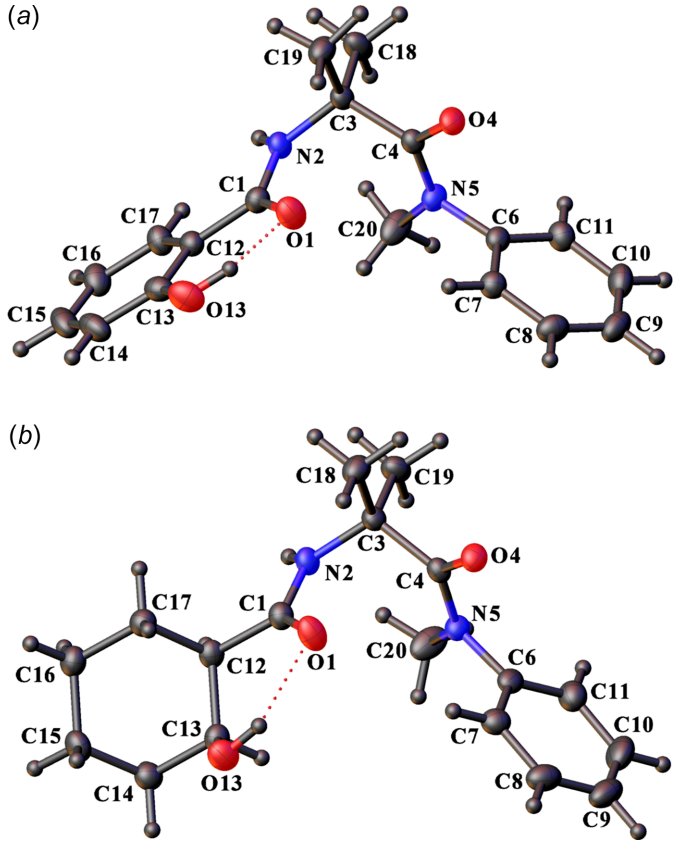


Figure 4
Views of the molecular structures of (*a*) **9a** and (*b*) **9b** with displacement ellipsoids drawn at the 50% probability level. The dotted lines represent intramolecular hydrogen bonds.

Table 2
Hydrogen-bond geometry (Å, °) for **9a**.

D—H···A	D—H	H···A	D···A	D—H···A
O13—H13···O1	0.938 (19)	1.698 (19)	2.5548 (12)	150.1 (18)
N2—H2···O4^{i}	0.884 (17)	2.159 (17)	2.9727 (12)	152.8 (14)

Symmetry code: (i) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$.

Table 3
Hydrogen-bond geometry (Å, °) for **9b**.

D—H···A	D—H	H···A	D···A	D—H···A
O13—H13···O1	0.88 (5)	1.97 (4)	2.729 (2)	143 (4)
N2—H2···O4^{i}	0.86 (3)	2.15 (3)	2.960 (2)	157 (2)

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

respect to the plane of the parent secondary amide group: nearly coplanar for **9a** and roughly perpendicular for **9b**. It is likely that this difference is preferred in order to facilitate the observed intramolecular O—H···O hydrogen bonds between the hydroxy group and the immediately adjacent amide O atom (Tables 2 and 3).

The CSD includes error-free single-crystal data for 22 related purely organic compounds containing the same two amide and –N(Me)Ph terminal groups as **9a**–**d**. Of these, five structures have two molecules in the asymmetric unit (Z' = 2). All of those structure determinations were carried out within the Heimgartner group and most of them are polypeptides of various lengths. The above-described turn between the secondary and tertiary amide groups is evident in all of these structures. The C1—N2—C3—C4 and N2—C3—C4—N5 pairs of torsion angles always have the same sign as each other, and their absolute values fall within the range of 41–66° [mean = 52 (5)°] and 37–62° [mean = 51 (6)°], respectively. The corresponding torsion angles for **9a**–**d** fall well within these ranges. The plane of the phenyl ring of the –N(Me)Ph group in these database structures is also consistently inclined to that of the parent amide group; the range of absolute values of the corresponding C4—N5—C6—C7 torsion angles is 47–121° [mean 89 (21)°].

Compound **9c** has a stereogenic centre at the hydroxy-substituted C atom, C14, of the hydroxymethylphenyl end of the molecule. The hydroxymethylphenyl component is disordered over two configurations, which represent a random distribution of both enantiomers over the same crystallographic sites in the crystal. Although the site occupancies of the disordered components in the model are unequal, the centrosymmetric space group indicates that the compound in the crystal is racemic. The hydroxy group forms an intermolecular O—H···O hydrogen bond with the same neighbouring O atom (Table 4), regardless of which enantiomer is being considered, so the energy environment around each enantiomer must be very similar. Compound **9d**, which is similar to **9c**, but has a carbonyl group instead of a hydroxy group at C14, exhibits similar disorder to that seen in the structure of **9c**, even though the compound is achiral and the disordered carbonyl O atom is not involved in any significant hydrogen bonds (Table 5). The superposition of two enanti-

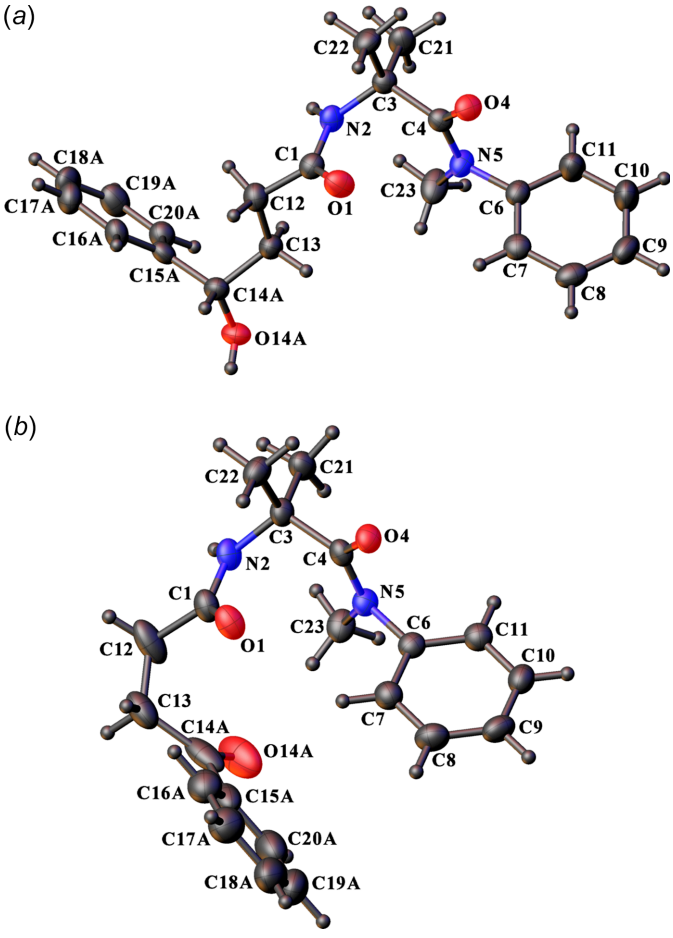


Figure 5
Views of the molecular structures of (*a*) **9c** and (*b*) **9d** with displacement ellipsoids drawn at the 50% probability level. The atoms of the minor disordered component have been omitted in each case for clarity.

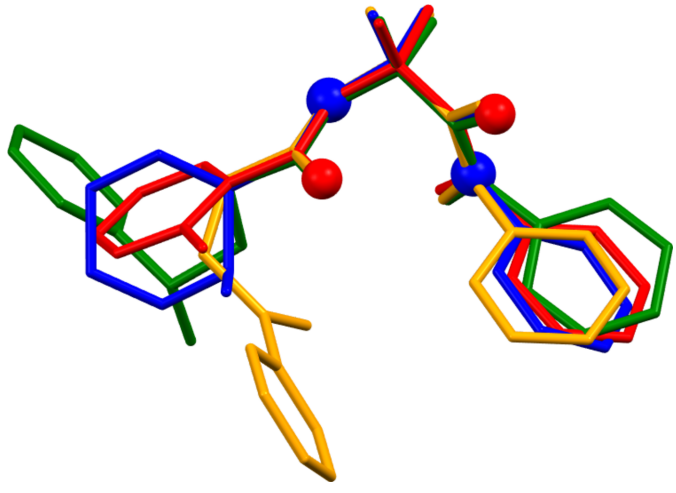


Figure 6
An overlay of the symmetry-unique molecules in the structures of **9a** (red), **9b** (blue), **9c** (green) and **9d** (orange). Representative positions of the amide N and O atoms are indicated by blue and red spheres, respectively. The minor-occupancy disordered regions (*B* component) of **9c** and **9d**, as well as all H atoms, have been omitted for clarity.

Table 4
Hydrogen-bond geometry (Å, °) for **9c**.

D—H···A	D—H	H···A	D···A	D—H···A
O14A—H14A···O1^{i}	0.84	1.95	2.7868 (14)	174
O14B—H14B···O1^{i}	0.84	1.93	2.754 (3)	166
N2—H2···O4^{ii}	0.892 (13)	2.060 (14)	2.9064 (12)	158.1 (12)

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

omers at the same crystallographic site is therefore not the rationale behind the disorder in this case. However, the presence of an unreasonably short H20B···H20B($2-x, -y, z$) contact of 2.04 Å across a crystallographic twofold axis between adjacent molecules with the B disorder conformation suggests that any B conformation molecule must have an A molecule adjacent to it such that the short contact and energetic frustration is avoided. No unreasonably short contacts exist between AA or AB pairs of conformers. As only 28.5 (2)% of the sites in the crystal are occupied by B conformers, BB interactions can be avoided entirely.

The molecular conformation of compound **10** (Fig. 7) in its crystal structure is quite different. The –N(Me)Ph amide group has been replaced by a carboxylic acid function and the backbone of the molecule from the secondary amide group through to the carboxylic acid group is almost planar (Table 1). One might surmise that the planarity arises in order to facilitate a short intramolecular N—H···O hydrogen bond between the amide group and the adjacent carboxylic acid carbonyl O atom, O4 (Table 6). However, this appears to be more of an exception than a rule. The CSD includes error-free single-crystal data for 51 related purely organic compounds

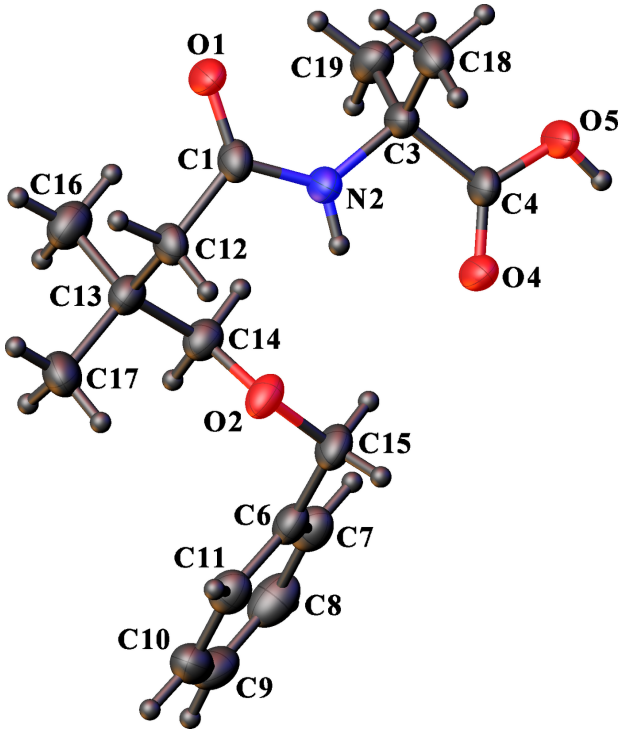


Figure 7
View of the molecular structure of **10** with displacement ellipsoids drawn at the 50% probability level.

Table 5
Hydrogen-bond geometry (Å, °) for **9d**.

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O4^{i}	0.88 (3)	2.01 (3)	2.862 (3)	161 (3)

Symmetry code: (i) $-x+2, -y+\frac{1}{2}, z-\frac{1}{2}$.

Table 6
Hydrogen-bond geometry (Å, °) for **10**.

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5···O1^{i}	0.86 (4)	1.81 (4)	2.622 (3)	157 (4)
N2—H2···O2	0.86 (3)	2.43 (3)	3.140 (3)	139 (3)
N2—H2···O4	0.86 (3)	2.13 (3)	2.606 (3)	115 (2)

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

containing a secondary amide group with an adjacent terminal carboxylic acid group. Of these, 8 structures have $Z' > 1$. Many of these structures are, again, polypeptides. In general, the absolute values of the C1—N2—C3—C4 and N2—C3—C4—O5 torsion angles also display progressive twists within the backbone, similar to those described above. Only two structures have the same degree of planarity and the intramolecular N—H···O hydrogen bond that are observed for **10**. These are those of *N*-*t*-butoxycarbonyl-L-alanyl-2-methylalanine (Bosch *et al.*, 1983) and *N*-(indol-3-ylacetyl)-α-aminoisobutyric acid (Kojić-Prodić *et al.*, 1993). Excluding these two structures, the absolute values of the C1—N2—C3—C4 torsion angles are in the range of 35–68° [mean = 52 (6)°], which is very similar to the range found in the structures of **9a–d** and related compounds described above. The N2—C3—C4—O5 torsion angles of the related carboxylic acid compounds vary more widely, with only seven structures having absolute values > 150°, as in the case of **10**, and three structures having absolute values < 30°, which indicates that the acid hydroxy group is on the same side of the backbone as the amid H atom. The range of N2—C3—C4—O5 torsion angles for the remaining 41 carboxylic acid structures is 30–150°, but the range between the lower and upper quartiles is only 40–50°, which is again quite similar to the corresponding torsion angle ranges found for the –N(Me)Ph compounds discussed above. The larger N2—C3—C4—O5 torsion angle range found across these carboxylic acid structures is likely related to the influence of the propensity of these structures to have intermolecular O—H···O hydrogen bonds emanating from the acid –OH group combined with the requirement for an efficient packing arrangement of the molecule in question.

3. Supramolecular features

The crystal structures of compounds **9a** and **9b** exhibit the same pattern of classical O—H···O and N—H···O hydrogen-bonding interactions. The hydroxy group forms an intramolecular hydrogen bond with the immediately adjacent secondary amide O atom to give a six-membered loop with a graph-set motif of $S(6)$ (Tables 2 and 3; Bernstein *et al.*, 1995). The secondary amide N—H group forms an intermolecular

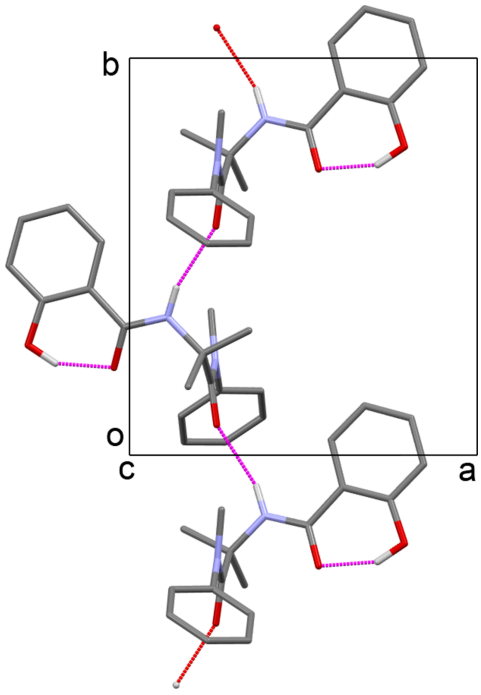


Figure 8
A view down [001] showing a hydrogen-bonded chain of molecules in the crystal structure of **9a**. The intra- and intermolecular hydrogen bonds are shown as dashed lines and uninvolved H atoms have been omitted for clarity.

hydrogen bond with the tertiary amide O atom of a neighbouring molecule and thereby links the molecules into extended chains, which run parallel to the [010] direction and have a graph set motif of $C(5)$ (Fig. 8). The crystal structures of **9c** and **9d** also display the same intermolecular N—H···O interactions, which generate $C(5)$ chains running parallel to the [010] and [001] directions respectively (Tables 4 and 5; Figs. S1 and S2 in the supporting information).

Additionally for **9c**, both orientations of the disordered hydroxy group form an intermolecular O—H···O hydrogen bond with the secondary amide O atom of a different neighbouring molecule. This interaction links pairs of molecules across a centre of inversion into dimeric units which have a graph-set motif of $R_2^2(14)$. The combination of all classic hydrogen bonding interactions links the molecules of **9c** into extended two-dimensional networks which lie perpendicular to the [101] direction (Fig. 9).

For **10**, the carboxylic acid hydroxy group forms an intermolecular O—H···O hydrogen bond with the amide O atom of a neighbouring molecule and thereby links the molecules into extended chains which run parallel to the [001] direction and have a graph-set motif of $C(7)$ (Table 6). The amide N—H group forms bifurcated N—H···(O,O) intramolecular hydrogen bonds with the carboxylic acid carbonyl O atom and with the ether O atom. These interactions form closed loops with graph-set motifs of $S(5)$ and $S(7)$, respectively (Fig. 10).

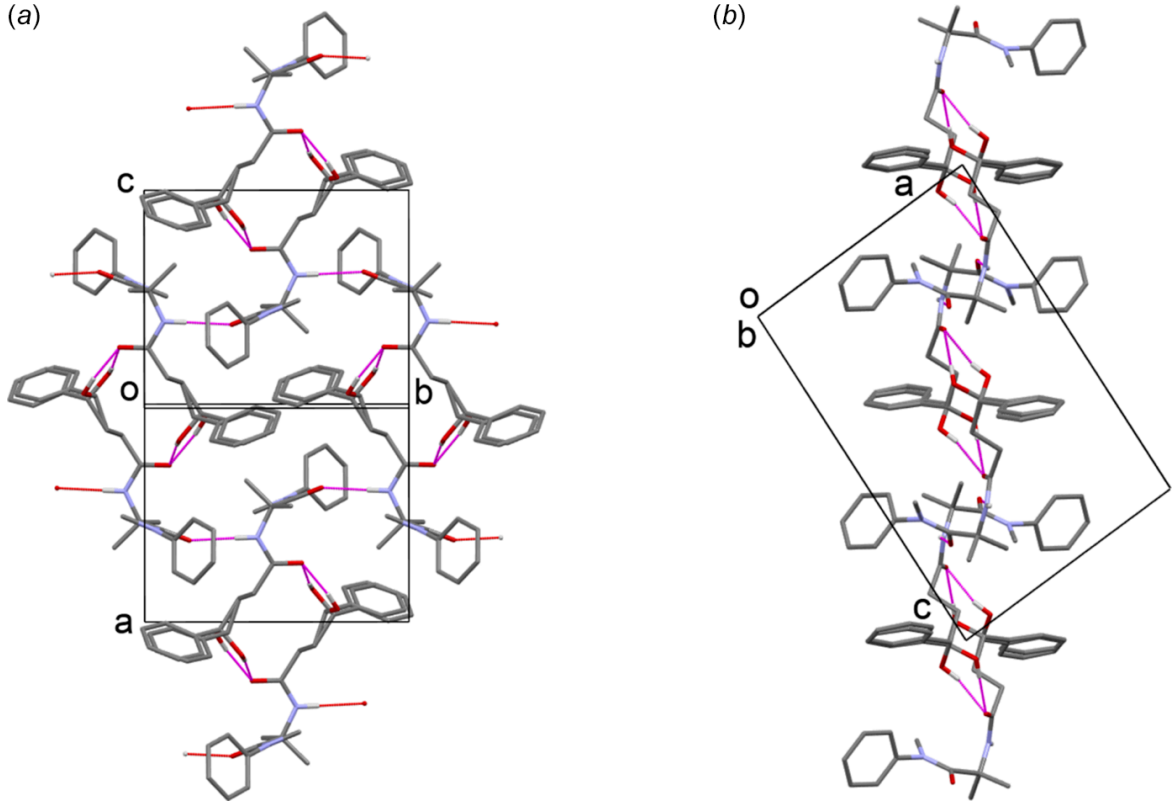


Figure 9
A hydrogen-bonded layer in the crystal structure of **9c**, viewed (*a*) down [101], perpendicular to the plane of the layer, and (*b*) down [010], parallel to the plane of the layer. The intermolecular hydrogen bonds are shown as dashed lines and uninvolved H atoms have been omitted for clarity. Both disordered conformations are displayed in order to highlight the common O—H···O interaction.

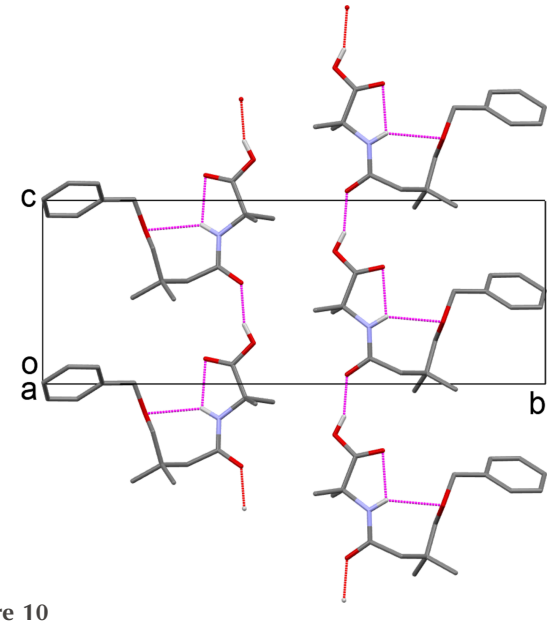


Figure 10
A view down [100] showing two hydrogen-bonded chains of molecules in the crystal structure of **10**. The intra- and intermolecular hydrogen bonds are shown as dashed lines and uninvolved H atoms have been omitted for clarity.

4. Synthesis and crystallization

The synthesis of the Aib-amides **9a** and **9b** and their analytical and spectroscopic data have been described earlier (Iliev *et al.*, 2006). Suitable crystals were obtained by slow crystallization from their solution in a CH_2Cl_2/acetone/ethyl acetate (EtOAc) mixture.

Analogously, the hydroxy acids **8c–e** were reacted with 3-amino-2,2-dimethyl-2*H*-azirine **2**: to a solution of 2–6 mmol of each of the hydroxy acids **8c–e** in 5–20 ml of THF, 1.05 equivalents of the aminoazirine **2** were added dropwise at r.t. The mixture was stirred at r.t. for 12–36 h, then the solvent was evaporated and the remaining solid purified by column chromatography (CC) over silica gel with acetone/CH_2Cl_2 mixtures and dried under high vacuum to give Aib-amides **9c–e** in high yields. Crystallization of **9c** from the mixed solvents of CH_2Cl_2/acetone/hexane and **9d** from CH_2Cl_2/acetonitrile yielded crystals suitable for X-ray diffraction analysis.

The Aib-amide **9e** was hydrolysed according to a standard procedure: to a solution of **9e** in THF, 3N HCl was added dropwise at 273 K and the mixture was left overnight at r.t. The organic solvent was evaporated, the residue was extracted with EtOAc, the organic fraction was washed with brine and dried over $MgSO_4$. After evaporation of the solvent, the crystalline material **10** was washed with diethyl ether/hexane and recrystallized from the mixed solvents of dichloromethane/acetone/hexane/diethyl ether to give suitable crystals for X-ray diffraction analysis.

5. Refinement details

The crystal data, data collection and structure refinement details for all reported structures are summarized in Table 7.

The molecule of **9b** has two stereogenic centres and the compound has crystallised in a chiral space group. The enantiomer used as the model in the refinement of **9b** was chosen to match that of the starting material, (1*R*,2*S*)-2-hydroxycyclohexanoic acid, used in the synthesis. Compounds **9d** and **10** are achiral molecules that have crystallized in non-centrosymmetric achiral space groups. Given that Mo $K\alpha$ radiation was used for all data collections and the compounds are purely organic, Bijvoet pairs were merged during data reduction at the diffractometer for **9b** and **10**. Consequently, the absolute structure parameter for these two structures is not meaningful and is not reported. Although Bijvoet pairs were not merged for **9d**, the absolute structure parameter is too imprecise to allow any conclusions about the absolute structure to be drawn.

For **9c**, the hydroxy group, its parent C atom and its neighbouring phenyl group are disordered over two conformations. Two positions were defined for these atoms and the site occupation factor of the major conformation of the group refined to 0.715 (2). Similarity restraints were applied to the chemically equivalent bond lengths and angles involving all disordered atoms. Furthermore, neighbouring atoms within and between each conformation of the disordered component were restrained to have similar atomic displacement parameters. The disorder indicates that both enantiomers of this chiral compound randomly occupy the same crystallographic site in the structure.

For **9d**, the keto group and its neighbouring phenyl group are disordered over two conformations. Two positions were defined for these atoms and the site occupation factor of the major conformation of the group refined to 0.572 (10). Rigid group constraints were applied to the atoms of each orientation of the disordered phenyl ring, while bond length restraints were applied to the bonds involving all other disordered non-hydrogen atoms [C—C = 1.520 (5), C═O = 1.230 (5) Å]. In addition, pseudo-isotropic restraints were applied to the anisotropic displacement parameters of all disordered atoms.

For **9a–9d**, six, two, three and one low-angle reflections, respectively, whose low intensities suggested that they were partially obscured by the beam stop, were omitted from the final refinement. A secondary extinction parameter was refined for **9b–d** and **10**. Except for the disordered hydroxy group of **9c**, any hydroxy, amide, or carboxylic acid H atoms in the structures were placed in the positions indicated by a difference electron density map and their positions were allowed to refine together with individual isotropic displacement parameters. The methyl H atoms of each structure and the disordered hydroxy H atoms in **9c** were constrained to an ideal geometry (C—H = 0.98, O—H = 0.84 Å) with U_{iso}(H) = 1.5U_{eq}(C,O), but were allowed to rotate freely about the respective parent C—C or C—O bonds. All other H atoms were placed in geometrically idealised positions and constrained to ride on their parent atoms with C—H distances of 0.95 (aromatic), 0.99 (methylene) or 1.00 Å (methine) and U_{iso}(H) = 1.2U_{eq}(C).

Table 7
Experimental details.

	9a	**9b**	**9c**	**9d**	**10**
Crystal data					
Chemical formula	$C_{18}H_{20}N_2O_3$	$C_{18}H_{26}N_2O_3$	$C_{21}H_{26}N_2O_3$	$C_{21}H_{24}N_2O_3$	$C_{17}H_{25}NO_4$
M_r	312.36	318.41	354.44	352.42	307.38
Crystal system, space group	Monoclinic, $P2_1/n$	Monoclinic, $C2$	Monoclinic, $P2_1/n$	Orthorhombic, $Aea2$	Orthorhombic, $Pca2_1$
Temperature (K)	160	160	160	160	160
a, b, c (Å)	10.4492 (2), 11.4071 (2), 14.2774 (3)	31.3330 (11), 5.9603 (2), 9.0440 (3)	10.6878 (2), 11.1971 (2), 16.1449 (3)	13.0433 (4), 25.8095 (8), 11.3381 (3)	10.1569 (5), 21.3548 (10), 7.7969 (3)
α, β, γ (°)	90, 107.0562 (11), 90	90, 94.416 (2), 90	90, 93.7334 (6), 90	90, 90, 90	90, 90, 90
V (Å^3)	1626.95 (6)	1683.99 (10)	1928.00 (6)	3816.87 (19)	1691.14 (13)
Z	4	4	4	8	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.09	0.09	0.08	0.08	0.09
Crystal size (mm)	0.27 × 0.27 × 0.20	0.45 × 0.15 × 0.05	0.25 × 0.25 × 0.15	0.30 × 0.18 × 0.10	0.45 × 0.32 × 0.07
Data collection					
Diffractometer	Nonius KappaCCD area-detector diffractometer	Nonius KappaCCD area-detector diffractometer	Nonius KappaCCD diffractometer	Nonius KappaCCD diffractometer	Nonius KappaCCD area-detector diffractometer
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	42366, 4756, 3746	13467, 1626, 1559	53347, 5616, 3368	26178, 3258, 2823	21591, 1614, 1306
R_{int}	0.045	0.052	0.068	0.069	0.091
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.704	0.595	0.704	0.595	0.595
Refinement					
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.045, 0.123, 1.05	0.031, 0.082, 1.08	0.049, 0.134, 1.01	0.039, 0.092, 1.06	0.038, 0.094, 1.11
No. of reflections	4756	1626	5616	3258	1614
No. of parameters	219	220	318	292	212
No. of restraints	0	1	258	103	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.23, −0.26	0.18, −0.19	0.26, −0.23	0.15, −0.15	0.14, −0.15
Absolute structure	–	Bijvoet pairs were merged during data reduction, so the absolute structure parameter is not meaningful. The enantiomer used in the refinement was chosen to match that of the chiral starting material used in the synthesis.	–	Flack x determined using 1135 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013). The compound in the crystal is itself achiral.	Bijvoet pairs were merged during data reduction, so the absolute structure parameter was not meaningful. The compound in the crystal is itself achiral.
Absolute structure parameter	–	–	–	0.1 (7)	–

Computer programs: *COLLECT* (Nonius, 2000), *DENZO-SMN* and *SCALEPACK* (Otwinowski & Minor, 1997), *SIR92* (Altomare *et al.*, 1994), *SHELXL2025* (Sheldrick, 2015), *OLEX2* (Dolomanov *et al.*, 2009), *Mercury* (Macrae *et al.*, 2020), *publCIF* (Westrip, 2010) and *PLATON* (Spek, 2020).

Acknowledgements

We thank the *Swiss National Science Foundation* and *F. Hoffmann-La Roche AG*, Basel for financial support.

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supporting information

Acta Cryst. (2026). E**82** [https://doi.org/10.1107/S2056989026011552]

Syntheses and structures of five Aib (2-aminoisobutyric acid) derivatives prepared *via* azirine coupling

Anthony Linden, Boyan Iliev and Heinz Heimgartner

Computing details

2-Hydroxy-*N*-[1-methyl-1-(*N*-methyl-*N*-phenylcarbamoyl)ethyl]benzamide (9a)

Crystal data

$C_{18}H_{20}N_2O_3$
M_r = 312.36
Monoclinic, $P2_1/n$
a = 10.4492 (2) Å
b = 11.4071 (2) Å
c = 14.2774 (3) Å
β = 107.0562 (11)°
V = 1626.95 (6) Å^3
Z = 4
$F(000)$ = 664
D_x = 1.275 Mg m^{-3}
Melting point: 420 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4987 reflections
θ = 2.0–30.0°
μ = 0.09 mm^{-1}
T = 160 K
Prism, colourless
0.27 × 0.27 × 0.20 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Radiation source: Nonius FR590 sealed tube generator
Horizontally mounted graphite crystal monochromator
Detector resolution: 9 pixels mm^{-1}
φ and ω scans with κ offsets
42366 measured reflections
4756 independent reflections
3746 reflections with $I > 2\sigma(I)$
R_{int} = 0.045
θ_{max} = 30.0°, θ_{min} = 2.3°
h = 0→14
k = 0→16
l = −20→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.123
S = 1.05
4756 reflections
219 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0574P)^2 + 0.3575P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Special details

Experimental. Solvent used: dichloromethane / acetone / ethyl acetate Cooling Device: Oxford Cryosystems Cryostream 700 Crystal mount: glued on a glass fibre Mosaicity (deg.): 0.471 (1) Frames collected: 348 Seconds exposure per frame: 26 Degrees rotation per frame: 2.0 Crystal-Detector distance (mm): 30.0

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}
O1	−0.04433 (8)	0.22043 (7)	0.17802 (6)	0.0325 (2)
O4	0.25118 (8)	0.07638 (6)	0.22469 (6)	0.02592 (18)
O13	−0.29167 (9)	0.27689 (8)	0.11887 (7)	0.0371 (2)
H13	−0.2136 (19)	0.2312 (17)	0.1351 (13)	0.068 (6)*
N2	0.10958 (9)	0.34736 (8)	0.26484 (6)	0.02244 (19)
H2	0.1344 (15)	0.4204 (15)	0.2813 (11)	0.044 (4)*
N5	0.24687 (9)	0.23663 (7)	0.13253 (6)	0.0236 (2)
C1	−0.01384 (11)	0.32286 (9)	0.20677 (8)	0.0232 (2)
C3	0.21123 (10)	0.25546 (9)	0.29914 (7)	0.0223 (2)
C4	0.23455 (10)	0.18313 (9)	0.21408 (7)	0.0205 (2)
C6	0.25935 (11)	0.16289 (9)	0.05357 (8)	0.0224 (2)
C7	0.14939 (11)	0.09913 (10)	0.00003 (8)	0.0262 (2)
H7	0.068671	0.100384	0.017863	0.031*
C8	0.15766 (13)	0.03326 (10)	−0.07999 (9)	0.0326 (3)
H8	0.082843	−0.011185	−0.116686	0.039*
C9	0.27519 (14)	0.03272 (10)	−0.10590 (9)	0.0353 (3)
H9	0.280829	−0.011822	−0.160775	0.042*
C10	0.38463 (13)	0.09676 (11)	−0.05225 (9)	0.0347 (3)
H10	0.464845	0.096457	−0.070771	0.042*
C11	0.37779 (12)	0.16162 (10)	0.02871 (9)	0.0289 (2)
H11	0.453380	0.204442	0.066379	0.035*
C12	−0.11409 (10)	0.41884 (9)	0.17892 (8)	0.0235 (2)
C13	−0.24975 (11)	0.38883 (10)	0.13755 (8)	0.0276 (2)
C14	−0.34673 (12)	0.47699 (12)	0.11565 (9)	0.0356 (3)
H14	−0.438459	0.456805	0.089036	0.043*
C15	−0.31058 (13)	0.59270 (12)	0.13229 (9)	0.0360 (3)
H15	−0.377694	0.651695	0.117382	0.043*
C16	−0.17700 (13)	0.62438 (10)	0.17065 (9)	0.0332 (3)
H16	−0.152352	0.704603	0.180607	0.040*
C17	−0.08026 (11)	0.53752 (10)	0.19418 (9)	0.0282 (2)
H17	0.011019	0.558914	0.221257	0.034*
C18	0.34362 (12)	0.31360 (10)	0.35488 (9)	0.0324 (3)
H181	0.371075	0.368121	0.311331	0.049*
H182	0.412567	0.253320	0.377754	0.049*
H183	0.332027	0.356589	0.411265	0.049*
C19	0.16867 (13)	0.17267 (10)	0.36918 (9)	0.0315 (3)

H191	0.145885	0.218671	0.420028	0.047*
H192	0.242505	0.119206	0.399801	0.047*
H193	0.090433	0.127221	0.332494	0.047*
C20	0.23505 (15)	0.36197 (10)	0.10908 (10)	0.0377 (3)
H201	0.142397	0.380102	0.071889	0.057*
H202	0.294658	0.382014	0.069760	0.057*
H203	0.260186	0.407630	0.169893	0.057*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0294 (4)	0.0218 (4)	0.0424 (5)	−0.0024 (3)	0.0044 (4)	−0.0066 (3)
O4	0.0329 (4)	0.0169 (4)	0.0283 (4)	0.0044 (3)	0.0096 (3)	0.0014 (3)
O13	0.0280 (5)	0.0352 (5)	0.0420 (5)	−0.0065 (4)	0.0008 (4)	0.0007 (4)
N2	0.0229 (4)	0.0172 (4)	0.0266 (5)	0.0013 (3)	0.0063 (4)	−0.0019 (3)
N5	0.0307 (5)	0.0167 (4)	0.0251 (4)	−0.0008 (3)	0.0109 (4)	−0.0012 (3)
C1	0.0247 (5)	0.0211 (5)	0.0245 (5)	−0.0005 (4)	0.0081 (4)	−0.0017 (4)
C3	0.0237 (5)	0.0185 (5)	0.0230 (5)	0.0027 (4)	0.0043 (4)	−0.0013 (4)
C4	0.0190 (5)	0.0183 (5)	0.0233 (5)	−0.0002 (4)	0.0049 (4)	−0.0014 (4)
C6	0.0271 (5)	0.0174 (4)	0.0233 (5)	0.0025 (4)	0.0083 (4)	0.0012 (4)
C7	0.0273 (5)	0.0240 (5)	0.0276 (5)	−0.0005 (4)	0.0085 (4)	0.0000 (4)
C8	0.0424 (7)	0.0256 (6)	0.0273 (6)	−0.0025 (5)	0.0064 (5)	−0.0028 (4)
C9	0.0564 (8)	0.0257 (6)	0.0277 (6)	0.0060 (5)	0.0182 (5)	−0.0006 (4)
C10	0.0397 (7)	0.0322 (6)	0.0399 (7)	0.0075 (5)	0.0234 (5)	0.0045 (5)
C11	0.0272 (6)	0.0272 (5)	0.0340 (6)	0.0006 (4)	0.0114 (5)	0.0013 (4)
C12	0.0229 (5)	0.0248 (5)	0.0233 (5)	0.0013 (4)	0.0076 (4)	0.0007 (4)
C13	0.0254 (5)	0.0325 (6)	0.0243 (5)	−0.0007 (4)	0.0061 (4)	0.0037 (4)
C14	0.0232 (5)	0.0463 (7)	0.0362 (6)	0.0054 (5)	0.0068 (5)	0.0122 (5)
C15	0.0335 (6)	0.0393 (7)	0.0383 (7)	0.0140 (5)	0.0154 (5)	0.0159 (5)
C16	0.0374 (7)	0.0257 (6)	0.0405 (7)	0.0071 (5)	0.0176 (5)	0.0081 (5)
C17	0.0262 (5)	0.0245 (5)	0.0350 (6)	0.0020 (4)	0.0107 (5)	0.0019 (4)
C18	0.0270 (6)	0.0286 (6)	0.0345 (6)	0.0027 (4)	−0.0018 (5)	−0.0079 (5)
C19	0.0439 (7)	0.0266 (6)	0.0268 (5)	0.0064 (5)	0.0148 (5)	0.0025 (4)
C20	0.0643 (9)	0.0185 (5)	0.0379 (7)	−0.0003 (5)	0.0267 (6)	0.0013 (5)

Geometric parameters (Å, º)

O1—C1	1.2484 (13)	C10—C11	1.3918 (17)
O4—C4	1.2330 (12)	C10—H10	0.9500
O13—C13	1.3509 (15)	C11—H11	0.9500
O13—H13	0.938 (19)	C12—C17	1.4000 (15)
N2—C1	1.3421 (14)	C12—C13	1.4086 (15)
N2—C3	1.4703 (13)	C13—C14	1.3964 (17)
N2—H2	0.884 (17)	C14—C15	1.3743 (19)
N5—C4	1.3538 (13)	C14—H14	0.9500
N5—C6	1.4428 (13)	C15—C16	1.3894 (19)
N5—C20	1.4654 (14)	C15—H15	0.9500
C1—C12	1.4866 (15)	C16—C17	1.3846 (16)

C3—C18	1.5301 (15)	C16—H16	0.9500
C3—C19	1.5331 (15)	C17—H17	0.9500
C3—C4	1.5455 (14)	C18—H181	0.9800
C6—C11	1.3841 (16)	C18—H182	0.9800
C6—C7	1.3850 (15)	C18—H183	0.9800
C7—C8	1.3911 (16)	C19—H191	0.9800
C7—H7	0.9500	C19—H192	0.9800
C8—C9	1.3826 (19)	C19—H193	0.9800
C8—H8	0.9500	C20—H201	0.9800
C9—C10	1.3835 (19)	C20—H202	0.9800
C9—H9	0.9500	C20—H203	0.9800

C13—O13—H13	105.6 (12)	C17—C12—C13	118.51 (10)
C1—N2—C3	121.78 (9)	C17—C12—C1	123.00 (10)
C1—N2—H2	121.0 (10)	C13—C12—C1	118.47 (10)
C3—N2—H2	117.2 (10)	O13—C13—C14	117.68 (11)
C4—N5—C6	117.54 (8)	O13—C13—C12	122.72 (10)
C4—N5—C20	127.84 (9)	C14—C13—C12	119.59 (11)
C6—N5—C20	114.28 (9)	C15—C14—C13	120.55 (11)
O1—C1—N2	120.29 (10)	C15—C14—H14	119.7
O1—C1—C12	120.63 (10)	C13—C14—H14	119.7
N2—C1—C12	119.07 (9)	C14—C15—C16	120.76 (11)
N2—C3—C18	108.59 (8)	C14—C15—H15	119.6
N2—C3—C19	109.70 (9)	C16—C15—H15	119.6
C18—C3—C19	108.58 (9)	C17—C16—C15	119.14 (11)
N2—C3—C4	112.57 (8)	C17—C16—H16	120.4
C18—C3—C4	108.25 (9)	C15—C16—H16	120.4
C19—C3—C4	109.05 (8)	C16—C17—C12	121.41 (11)
O4—C4—N5	120.35 (9)	C16—C17—H17	119.3
O4—C4—C3	118.67 (9)	C12—C17—H17	119.3
N5—C4—C3	120.79 (9)	C3—C18—H181	109.5
C11—C6—C7	120.82 (10)	C3—C18—H182	109.5
C11—C6—N5	119.94 (10)	H181—C18—H182	109.5
C7—C6—N5	119.14 (9)	C3—C18—H183	109.5
C6—C7—C8	119.71 (10)	H181—C18—H183	109.5
C6—C7—H7	120.1	H182—C18—H183	109.5
C8—C7—H7	120.1	C3—C19—H191	109.5
C9—C8—C7	119.75 (11)	C3—C19—H192	109.5
C9—C8—H8	120.1	H191—C19—H192	109.5
C7—C8—H8	120.1	C3—C19—H193	109.5
C8—C9—C10	120.28 (11)	H191—C19—H193	109.5
C8—C9—H9	119.9	H192—C19—H193	109.5
C10—C9—H9	119.9	N5—C20—H201	109.5
C9—C10—C11	120.34 (11)	N5—C20—H202	109.5
C9—C10—H10	119.8	H201—C20—H202	109.5
C11—C10—H10	119.8	N5—C20—H203	109.5
C6—C11—C10	119.09 (11)	H201—C20—H203	109.5
C6—C11—H11	120.5	H202—C20—H203	109.5

C10—C11—H11	120.5

C3—N2—C1—O1	−1.98 (16)	C6—C7—C8—C9	−0.53 (17)
C3—N2—C1—C12	176.84 (9)	C7—C8—C9—C10	0.40 (18)
C1—N2—C3—C18	174.48 (10)	C8—C9—C10—C11	0.48 (18)
C1—N2—C3—C19	−66.99 (12)	C7—C6—C11—C10	1.09 (16)
C1—N2—C3—C4	54.63 (13)	N5—C6—C11—C10	−175.23 (10)
C6—N5—C4—O4	9.26 (15)	C9—C10—C11—C6	−1.21 (18)
C20—N5—C4—O4	−177.85 (11)	O1—C1—C12—C17	−168.50 (11)
C6—N5—C4—C3	−175.88 (9)	N2—C1—C12—C17	12.69 (16)
C20—N5—C4—C3	−2.99 (17)	O1—C1—C12—C13	13.15 (15)
N2—C3—C4—O4	−140.25 (10)	N2—C1—C12—C13	−165.66 (10)
C18—C3—C4—O4	99.71 (11)	C17—C12—C13—O13	179.05 (10)
C19—C3—C4—O4	−18.25 (13)	C1—C12—C13—O13	−2.53 (16)
N2—C3—C4—N5	44.81 (13)	C17—C12—C13—C14	−1.87 (16)
C18—C3—C4—N5	−75.23 (12)	C1—C12—C13—C14	176.55 (10)
C19—C3—C4—N5	166.80 (10)	O13—C13—C14—C15	−179.55 (11)
C4—N5—C6—C11	−113.22 (11)	C12—C13—C14—C15	1.32 (18)
C20—N5—C6—C11	72.93 (14)	C13—C14—C15—C16	0.36 (19)
C4—N5—C6—C7	70.39 (13)	C14—C15—C16—C17	−1.45 (19)
C20—N5—C6—C7	−103.46 (12)	C15—C16—C17—C12	0.86 (18)
C11—C6—C7—C8	−0.23 (16)	C13—C12—C17—C16	0.79 (17)
N5—C6—C7—C8	176.12 (10)	C1—C12—C17—C16	−177.56 (11)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O13—H13···O1	0.938 (19)	1.698 (19)	2.5548 (12)	150.1 (18)
N2—H2···O4^{i}	0.884 (17)	2.159 (17)	2.9727 (12)	152.8 (14)

Symmetry code: (i) −*x*+1/2, *y*+1/2, −*z*+1/2.

(1*R*,2*S*)-2-Hydroxy-*N*-[1-methyl-1-(*N*-methyl-*N*-phenylcarbamoyl)ethyl]cyclohexanecarboxamide (9b)

Crystal data

$C_{18}H_{26}N_2O_3$
M_r = 318.41
Monoclinic, *C*2
a = 31.3330 (11) Å
b = 5.9603 (2) Å
c = 9.0440 (3) Å
β = 94.416 (2)°
V = 1683.99 (10) Å^3
Z = 4
F(000) = 688

D_x = 1.256 Mg m^{-3}
Melting point: 445 K
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 1626 reflections
θ = 2.0–25.0°
μ = 0.09 mm^{-1}
T = 160 K
Plate, colourless
0.45 × 0.15 × 0.05 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Radiation source: Nonius FR590 sealed tube generator
Horizontally mounted graphite crystal monochromator
Detector resolution: 9 pixels mm^{-1}
ω scans with κ offsets

13467 measured reflections
1626 independent reflections
1559 reflections with $I > 2\sigma(I)$
$R_{int} = 0.052$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.3°$
$h = 0 \rightarrow 36$
$k = 0 \rightarrow 7$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.082$
$S = 1.08$
1626 reflections
220 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0486P)^2 + 0.627P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}
Extinction correction: SHELXL-2025 (Sheldrick, 2015), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.012 (3)
Absolute structure: Bijvoet pairs were merged during data reduction, so the absolute structure parameter is not meaningful. The enantiomer used in the refinement was chosen to match that of the chiral starting material used in the synthesis.

Special details

Experimental. Solvent used: dichloromethane / acetone / ethyl acetate Cooling Device: Oxford Cryosystems Cryostream 700 Crystal mount: glued on a glass fibre Mosaicity (deg.): 0.792 (2) Frames collected: 240 Seconds exposure per frame: 150 Degrees rotation per frame: 2.0 Crystal-Detector distance (mm): 30.0

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.10121 (6)	0.1017 (3)	0.56826 (16)	0.0326 (4)
O4	0.09861 (4)	0.4219 (3)	0.27865 (15)	0.0255 (4)
O13	0.14385 (5)	−0.0854 (3)	0.81239 (17)	0.0316 (4)
H13	0.1347 (13)	0.027 (9)	0.755 (4)	0.087 (13)*
N2	0.07545 (5)	−0.1270 (3)	0.38359 (18)	0.0227 (4)
H2	0.0744 (7)	−0.264 (5)	0.353 (2)	0.021 (6)*
N5	0.14406 (5)	0.1335 (3)	0.25330 (18)	0.0220 (4)
C1	0.09256 (6)	−0.0902 (4)	0.5230 (2)	0.0239 (4)
C3	0.06567 (6)	0.0580 (4)	0.2784 (2)	0.0228 (5)
C4	0.10433 (6)	0.2191 (4)	0.2714 (2)	0.0207 (4)
C6	0.18031 (6)	0.2819 (4)	0.2679 (2)	0.0248 (5)
C7	0.19012 (6)	0.3928 (4)	0.4014 (2)	0.0281 (5)
H7	0.171485	0.381584	0.479110	0.034*

C8	0.22726 (7)	0.5199 (4)	0.4208 (3)	0.0359 (6)
H8	0.234037	0.595896	0.511915	0.043*
C9	0.25439 (7)	0.5359 (5)	0.3078 (3)	0.0410 (6)
H9	0.279755	0.623205	0.321406	0.049*
C10	0.24481 (7)	0.4263 (5)	0.1760 (3)	0.0427 (6)
H10	0.263704	0.437023	0.099008	0.051*
C11	0.20728 (7)	0.2986 (4)	0.1544 (3)	0.0336 (5)
H11	0.200446	0.224215	0.062672	0.040*
C12	0.10007 (6)	−0.2933 (4)	0.6211 (2)	0.0234 (5)
H12	0.100901	−0.429133	0.556477	0.028*
C13	0.14334 (7)	−0.2741 (4)	0.7138 (2)	0.0251 (5)
H131	0.166758	−0.256559	0.645529	0.030*
C14	0.15148 (7)	−0.4832 (4)	0.8066 (2)	0.0280 (5)
H141	0.178885	−0.466477	0.867717	0.034*
H142	0.154148	−0.613636	0.740180	0.034*
C15	0.11560 (7)	−0.5274 (4)	0.9079 (2)	0.0292 (5)
H151	0.116171	−0.409773	0.985368	0.035*
H152	0.120600	−0.673781	0.957955	0.035*
C16	0.07129 (7)	−0.5294 (4)	0.8223 (2)	0.0288 (5)
H161	0.068665	−0.665560	0.759631	0.035*
H162	0.048976	−0.536864	0.893970	0.035*
C17	0.06352 (6)	−0.3225 (4)	0.7243 (2)	0.0264 (5)
H171	0.061742	−0.187584	0.787447	0.032*
H172	0.035945	−0.338567	0.664073	0.032*
C18	0.02794 (6)	0.1940 (4)	0.3289 (3)	0.0315 (5)
H181	0.003204	0.094989	0.335966	0.047*
H182	0.020576	0.313321	0.256895	0.047*
H183	0.035904	0.260566	0.426209	0.047*
C19	0.05320 (7)	−0.0408 (4)	0.1247 (2)	0.0308 (5)
H191	0.076979	−0.130699	0.092481	0.046*
H192	0.046798	0.081221	0.053810	0.046*
H193	0.027861	−0.136093	0.129512	0.046*
C20	0.15500 (7)	−0.0980 (5)	0.2200 (3)	0.0399 (6)
H201	0.131212	−0.196703	0.241150	0.060*
H202	0.180750	−0.142132	0.281523	0.060*
H203	0.160398	−0.110582	0.115046	0.060*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0514 (10)	0.0190 (9)	0.0269 (8)	−0.0011 (7)	−0.0006 (7)	−0.0018 (7)
O4	0.0271 (7)	0.0181 (9)	0.0315 (8)	0.0020 (7)	0.0042 (5)	0.0004 (7)
O13	0.0399 (8)	0.0227 (9)	0.0313 (8)	−0.0033 (8)	−0.0035 (6)	−0.0045 (8)
N2	0.0287 (8)	0.0160 (10)	0.0235 (9)	−0.0001 (7)	0.0026 (6)	0.0007 (8)
N5	0.0219 (9)	0.0179 (9)	0.0264 (8)	0.0018 (7)	0.0032 (6)	0.0004 (7)
C1	0.0271 (9)	0.0203 (11)	0.0246 (10)	0.0013 (10)	0.0044 (7)	−0.0002 (10)
C3	0.0228 (10)	0.0200 (11)	0.0255 (10)	0.0014 (9)	0.0003 (7)	0.0032 (9)
C4	0.0252 (10)	0.0184 (11)	0.0182 (9)	0.0011 (9)	0.0008 (7)	0.0002 (8)

C6	0.0221 (10)	0.0186 (11)	0.0334 (11)	0.0033 (8)	0.0004 (8)	0.0045 (9)
C7	0.0260 (9)	0.0250 (12)	0.0327 (11)	0.0031 (10)	−0.0003 (8)	−0.0001 (10)
C8	0.0295 (11)	0.0276 (13)	0.0492 (14)	0.0009 (10)	−0.0068 (10)	−0.0013 (11)
C9	0.0246 (11)	0.0307 (14)	0.0672 (16)	−0.0015 (10)	−0.0003 (11)	0.0066 (13)
C10	0.0297 (11)	0.0438 (16)	0.0563 (15)	0.0010 (12)	0.0148 (10)	0.0131 (14)
C11	0.0310 (11)	0.0345 (13)	0.0359 (11)	0.0034 (10)	0.0065 (9)	0.0051 (11)
C12	0.0290 (10)	0.0183 (10)	0.0229 (10)	−0.0020 (9)	0.0031 (8)	−0.0030 (9)
C13	0.0281 (10)	0.0205 (11)	0.0270 (10)	−0.0013 (9)	0.0041 (8)	−0.0003 (9)
C14	0.0296 (11)	0.0238 (12)	0.0303 (11)	0.0009 (10)	0.0003 (9)	0.0007 (10)
C15	0.0378 (11)	0.0278 (13)	0.0223 (10)	0.0012 (10)	0.0036 (8)	0.0025 (9)
C16	0.0313 (10)	0.0275 (13)	0.0283 (10)	−0.0024 (9)	0.0062 (8)	0.0004 (9)
C17	0.0264 (9)	0.0255 (13)	0.0274 (10)	0.0001 (9)	0.0028 (8)	0.0000 (9)
C18	0.0247 (10)	0.0268 (13)	0.0434 (12)	0.0013 (10)	0.0049 (9)	0.0041 (11)
C19	0.0322 (11)	0.0291 (14)	0.0296 (11)	−0.0027 (10)	−0.0063 (8)	0.0014 (10)
C20	0.0299 (11)	0.0202 (12)	0.0703 (16)	0.0035 (11)	0.0074 (10)	−0.0082 (13)

Geometric parameters (Å, º)

O1—C1	1.238 (3)	C12—C13	1.542 (3)
O4—C4	1.225 (3)	C12—C17	1.543 (3)
O13—C13	1.435 (3)	C12—H12	1.0000
O13—H13	0.88 (5)	C13—C14	1.513 (3)
N2—C1	1.349 (3)	C13—H131	1.0000
N2—C3	1.473 (3)	C14—C15	1.527 (3)
N2—H2	0.86 (3)	C14—H141	0.9900
N5—C4	1.367 (3)	C14—H142	0.9900
N5—C6	1.438 (3)	C15—C16	1.537 (3)
N5—C20	1.458 (3)	C15—H151	0.9900
C1—C12	1.509 (3)	C15—H152	0.9900
C3—C18	1.532 (3)	C16—C17	1.527 (3)
C3—C19	1.532 (3)	C16—H161	0.9900
C3—C4	1.551 (3)	C16—H162	0.9900
C6—C11	1.383 (3)	C17—H171	0.9900
C6—C7	1.390 (3)	C17—H172	0.9900
C7—C8	1.388 (3)	C18—H181	0.9800
C7—H7	0.9500	C18—H182	0.9800
C8—C9	1.382 (3)	C18—H183	0.9800
C8—H8	0.9500	C19—H191	0.9800
C9—C10	1.372 (4)	C19—H192	0.9800
C9—H9	0.9500	C19—H193	0.9800
C10—C11	1.402 (4)	C20—H201	0.9800
C10—H10	0.9500	C20—H202	0.9800
C11—H11	0.9500	C20—H203	0.9800
C13—O13—H13	104 (3)	O13—C13—H131	109.2
C1—N2—C3	121.97 (19)	C14—C13—H131	109.2
C1—N2—H2	117.2 (15)	C12—C13—H131	109.2
C3—N2—H2	120.0 (15)	C13—C14—C15	112.00 (18)

C4—N5—C6	118.66 (18)	C13—C14—H141	109.2
C4—N5—C20	127.48 (18)	C15—C14—H141	109.2
C6—N5—C20	113.86 (16)	C13—C14—H142	109.2
O1—C1—N2	121.4 (2)	C15—C14—H142	109.2
O1—C1—C12	121.76 (17)	H141—C14—H142	107.9
N2—C1—C12	116.8 (2)	C14—C15—C16	112.17 (17)
N2—C3—C18	109.28 (16)	C14—C15—H151	109.2
N2—C3—C19	108.91 (18)	C16—C15—H151	109.2
C18—C3—C19	108.99 (17)	C14—C15—H152	109.2
N2—C3—C4	111.42 (15)	C16—C15—H152	109.2
C18—C3—C4	107.93 (18)	H151—C15—H152	107.9
C19—C3—C4	110.27 (16)	C17—C16—C15	112.55 (18)
O4—C4—N5	120.88 (19)	C17—C16—H161	109.1
O4—C4—C3	119.40 (18)	C15—C16—H161	109.1
N5—C4—C3	119.69 (19)	C17—C16—H162	109.1
C11—C6—C7	120.3 (2)	C15—C16—H162	109.1
C11—C6—N5	119.9 (2)	H161—C16—H162	107.8
C7—C6—N5	119.53 (18)	C16—C17—C12	110.48 (17)
C8—C7—C6	119.8 (2)	C16—C17—H171	109.6
C8—C7—H7	120.1	C12—C17—H171	109.6
C6—C7—H7	120.1	C16—C17—H172	109.6
C9—C8—C7	120.1 (2)	C12—C17—H172	109.6
C9—C8—H8	119.9	H171—C17—H172	108.1
C7—C8—H8	119.9	C3—C18—H181	109.5
C10—C9—C8	120.2 (2)	C3—C18—H182	109.5
C10—C9—H9	119.9	H181—C18—H182	109.5
C8—C9—H9	119.9	C3—C18—H183	109.5
C9—C10—C11	120.3 (2)	H181—C18—H183	109.5
C9—C10—H10	119.8	H182—C18—H183	109.5
C11—C10—H10	119.8	C3—C19—H191	109.5
C6—C11—C10	119.3 (2)	C3—C19—H192	109.5
C6—C11—H11	120.4	H191—C19—H192	109.5
C10—C11—H11	120.4	C3—C19—H193	109.5
C1—C12—C13	110.56 (17)	H191—C19—H193	109.5
C1—C12—C17	110.92 (17)	H192—C19—H193	109.5
C13—C12—C17	109.98 (16)	N5—C20—H201	109.5
C1—C12—H12	108.4	N5—C20—H202	109.5
C13—C12—H12	108.4	H201—C20—H202	109.5
C17—C12—H12	108.4	N5—C20—H203	109.5
O13—C13—C14	107.90 (16)	H201—C20—H203	109.5
O13—C13—C12	111.30 (18)	H202—C20—H203	109.5
C14—C13—C12	110.13 (18)		

C3—N2—C1—O1	−0.7 (3)	C6—C7—C8—C9	−0.1 (4)
C3—N2—C1—C12	178.55 (17)	C7—C8—C9—C10	−0.1 (4)
C1—N2—C3—C18	−69.6 (2)	C8—C9—C10—C11	0.5 (4)
C1—N2—C3—C19	171.48 (17)	C7—C6—C11—C10	0.7 (3)
C1—N2—C3—C4	49.6 (2)	N5—C6—C11—C10	−173.6 (2)

C6—N5—C4—O4	10.4 (3)	C9—C10—C11—C6	−0.8 (4)
C20—N5—C4—O4	−169.8 (2)	O1—C1—C12—C13	−41.7 (3)
C6—N5—C4—C3	−171.38 (16)	N2—C1—C12—C13	139.01 (18)
C20—N5—C4—C3	8.5 (3)	O1—C1—C12—C17	80.6 (2)
N2—C3—C4—O4	−132.59 (19)	N2—C1—C12—C17	−98.7 (2)
C18—C3—C4—O4	−12.6 (3)	C1—C12—C13—O13	62.9 (2)
C19—C3—C4—O4	106.3 (2)	C17—C12—C13—O13	−59.9 (2)
N2—C3—C4—N5	49.1 (2)	C1—C12—C13—C14	−177.47 (17)
C18—C3—C4—N5	169.11 (17)	C17—C12—C13—C14	59.7 (2)
C19—C3—C4—N5	−71.9 (2)	O13—C13—C14—C15	64.5 (2)
C4—N5—C6—C11	−125.1 (2)	C12—C13—C14—C15	−57.1 (2)
C20—N5—C6—C11	55.0 (3)	C13—C14—C15—C16	52.6 (3)
C4—N5—C6—C7	60.6 (3)	C14—C15—C16—C17	−50.8 (3)
C20—N5—C6—C7	−119.3 (2)	C15—C16—C17—C12	53.5 (2)
C11—C6—C7—C8	−0.3 (3)	C1—C12—C17—C16	179.61 (17)
N5—C6—C7—C8	174.1 (2)	C13—C12—C17—C16	−57.8 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O13—H13···O1	0.88 (5)	1.97 (4)	2.729 (2)	143 (4)
N2—H2···O4^{i}	0.86 (3)	2.15 (3)	2.960 (2)	157 (2)

Symmetry code: (i) *x*, *y*−1, *z*.

rac-4-Hydroxy-4-phenyl-*N*-[1-methyl-1-(*N*-methyl-*N*-phenylcarbamoyl)ethyl]butanamide (9c)

Crystal data

$C_{21}H_{26}N_2O_3$
M_r = 354.44
Monoclinic, $P2_1/n$
a = 10.6878 (2) Å
b = 11.1971 (2) Å
c = 16.1449 (3) Å
β = 93.7334 (6)°
V = 1928.00 (6) Å^3
Z = 4
$F(000)$ = 760
D_x = 1.221 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5890 reflections
θ = 2.0–30.0°
μ = 0.08 mm^{-1}
T = 160 K
Prism, colourless
0.25 × 0.25 × 0.15 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: Nonius FR591 sealed tube generator
Horizontally mounted graphite crystal monochromator
Detector resolution: 9 pixels mm^{-1}
φ and ω scans with κ offsets
53347 measured reflections
5616 independent reflections
3368 reflections with $I > 2\sigma(I)$
R_{int} = 0.068
θ_{max} = 30.0°, θ_{min} = 2.5°
h = 0→15
k = 0→15
l = −22→22

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.134$
$S = 1.01$
5616 reflections
318 parameters
258 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0695P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}
Extinction correction: SHELXL-2025 (Sheldrick, 2015), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.021 (3)

Special details

Experimental. Solvent used: dichloromethane / acetone / hexane Cooling Device: Oxford Cryosystems Cryostream 700 Crystal mount: glued on a glass fibre Mosaicity (deg.): 0.457 (1) Frames collected: 370 Seconds exposure per frame: 60 Degrees rotation per frame: 2.0 Crystal-Detector distance (mm): 30.0

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.

Refinement. The hydroxy group, its parent C-atom and its neighbouring phenyl group are disordered over two conformations. Two positions were defined for these atoms and the site occupation factor of the major conformation of the group refined to 0.715 (2). Similarity restraints were applied to the chemically equivalent bond lengths and angles across the two disordered conformations, including both sets of the disordered C13–C14 bond. Furthermore, neighbouring atoms within and between each conformation of the disordered component were restrained to have similar atomic displacement parameters.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	−0.08985 (8)	0.59404 (7)	0.18426 (5)	0.0408 (2)	
O4	−0.34556 (7)	0.66901 (7)	0.27544 (5)	0.0346 (2)	
N2	−0.14431 (8)	0.42600 (9)	0.24942 (6)	0.0319 (3)	
H2	−0.1356 (11)	0.3471 (12)	0.2548 (8)	0.044 (4)*	
N5	−0.38855 (8)	0.53079 (8)	0.17708 (6)	0.0338 (3)	
C1	−0.07456 (10)	0.48552 (10)	0.19728 (7)	0.0327 (3)	
C3	−0.23823 (10)	0.48289 (9)	0.29894 (7)	0.0310 (3)	
C4	−0.32556 (10)	0.56912 (9)	0.24778 (7)	0.0285 (3)	
C6	−0.47901 (11)	0.61137 (10)	0.13763 (7)	0.0337 (3)	
C7	−0.44612 (11)	0.68537 (10)	0.07467 (7)	0.0368 (3)	
H7	−0.362703	0.684839	0.057620	0.044*	
C8	−0.53455 (12)	0.76056 (11)	0.03617 (8)	0.0435 (3)	
H8	−0.511632	0.812843	−0.006636	0.052*	
C9	−0.65597 (13)	0.75948 (12)	0.06004 (9)	0.0505 (4)	
H9	−0.717134	0.810072	0.032942	0.061*	
C10	−0.68878 (13)	0.68568 (15)	0.12275 (10)	0.0609 (4)	
H10	−0.772423	0.685652	0.139379	0.073*	
C11	−0.60007 (12)	0.61114 (13)	0.16188 (9)	0.0528 (4)	
H11	−0.622733	0.559938	0.205369	0.063*	

C12	0.01982 (11)	0.41337 (11)	0.15407 (8)	0.0384 (3)	
H121	0.052573	0.349105	0.191613	0.046*	
H122	0.091056	0.465348	0.141460	0.046*	
C13	−0.03638 (11)	0.35769 (11)	0.07335 (7)	0.0371 (3)	
H131	−0.090644	0.289560	0.086575	0.045*	0.715 (2)
H132	−0.088987	0.417592	0.042336	0.045*	0.715 (2)
H133	−0.072767	0.422553	0.037743	0.045*	0.285 (2)
H134	−0.106066	0.304521	0.087107	0.045*	0.285 (2)
O14A	0.00564 (10)	0.27073 (10)	−0.05408 (7)	0.0379 (4)	0.715 (2)
H14A	0.032562	0.306677	−0.094979	0.057*	0.715 (2)
C14A	0.0666 (4)	0.3145 (3)	0.0194 (2)	0.0340 (8)	0.715 (2)
H141	0.121540	0.383185	0.006089	0.041*	0.715 (2)
C15A	0.1441 (5)	0.2198 (3)	0.0653 (3)	0.0322 (7)	0.715 (2)
C16A	0.2597 (5)	0.2469 (4)	0.1056 (5)	0.0396 (8)	0.715 (2)
H16A	0.292893	0.325323	0.102182	0.047*	0.715 (2)
C17A	0.3268 (5)	0.1598 (4)	0.1507 (5)	0.0480 (10)	0.715 (2)
H17A	0.404467	0.179994	0.179260	0.058*	0.715 (2)
C18A	0.2824 (5)	0.0447 (4)	0.1547 (5)	0.0488 (11)	0.715 (2)
H18A	0.330010	−0.015243	0.184029	0.059*	0.715 (2)
C19A	0.1674 (5)	0.0180 (4)	0.1153 (4)	0.0489 (10)	0.715 (2)
H19A	0.134890	−0.060796	0.118163	0.059*	0.715 (2)
C20A	0.0992 (4)	0.1047 (4)	0.0716 (4)	0.0385 (9)	0.715 (2)
H20A	0.019809	0.084838	0.045411	0.046*	0.715 (2)
O14B	0.1358 (3)	0.3661 (3)	−0.0167 (2)	0.0441 (10)	0.285 (2)
H14B	0.109226	0.376948	−0.066281	0.066*	0.285 (2)
C14B	0.0556 (10)	0.2845 (7)	0.0218 (7)	0.039 (2)	0.285 (2)
H142	0.004409	0.242326	−0.023252	0.047*	0.285 (2)
C15B	0.1334 (12)	0.1912 (9)	0.0696 (8)	0.0286 (17)	0.285 (2)
C16B	0.2518 (13)	0.2173 (9)	0.1066 (12)	0.0389 (19)	0.285 (2)
H16B	0.286730	0.294859	0.101771	0.047*	0.285 (2)
C17B	0.3185 (13)	0.1278 (10)	0.1509 (13)	0.045 (2)	0.285 (2)
H17B	0.399167	0.145306	0.176225	0.054*	0.285 (2)
C18B	0.2700 (14)	0.0156 (11)	0.1586 (13)	0.046 (2)	0.285 (2)
H18B	0.316981	−0.044782	0.187875	0.055*	0.285 (2)
C19B	0.1521 (12)	−0.0086 (8)	0.1232 (11)	0.0398 (18)	0.285 (2)
H19B	0.117382	−0.086260	0.127821	0.048*	0.285 (2)
C20B	0.0845 (11)	0.0790 (8)	0.0812 (9)	0.0346 (18)	0.285 (2)
H20B	0.001712	0.061877	0.059524	0.042*	0.285 (2)
C21	−0.31830 (12)	0.38582 (11)	0.33570 (9)	0.0431 (3)	
H211	−0.355330	0.335381	0.290946	0.065*	
H212	−0.385235	0.423012	0.365429	0.065*	
H213	−0.265708	0.336774	0.374304	0.065*	
C22	−0.17027 (12)	0.55309 (12)	0.36904 (8)	0.0423 (3)	
H221	−0.112001	0.500193	0.400831	0.063*	
H222	−0.231591	0.585302	0.405695	0.063*	
H223	−0.123472	0.618948	0.345704	0.063*	
C23	−0.37513 (12)	0.41763 (11)	0.13297 (9)	0.0484 (4)	
H231	−0.337857	0.432647	0.080078	0.073*	

H232	−0.457793	0.380768	0.122381	0.073*
H233	−0.320771	0.363775	0.166933	0.073*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0523 (5)	0.0272 (5)	0.0438 (5)	0.0005 (4)	0.0108 (4)	0.0040 (4)
O4	0.0435 (5)	0.0239 (4)	0.0363 (5)	0.0034 (3)	0.0018 (4)	−0.0040 (3)
N2	0.0336 (5)	0.0222 (5)	0.0399 (6)	0.0024 (4)	0.0025 (4)	0.0024 (4)
N5	0.0359 (5)	0.0284 (5)	0.0363 (6)	0.0050 (4)	−0.0045 (4)	−0.0062 (4)
C1	0.0334 (6)	0.0287 (6)	0.0357 (7)	−0.0015 (5)	−0.0005 (5)	−0.0007 (5)
C3	0.0349 (6)	0.0257 (6)	0.0323 (6)	0.0010 (5)	0.0024 (5)	0.0012 (5)
C4	0.0321 (6)	0.0235 (6)	0.0302 (6)	−0.0025 (4)	0.0048 (5)	0.0001 (5)
C6	0.0355 (6)	0.0332 (6)	0.0320 (7)	0.0044 (5)	−0.0018 (5)	−0.0036 (5)
C7	0.0391 (7)	0.0368 (7)	0.0342 (7)	−0.0021 (5)	−0.0004 (5)	−0.0054 (5)
C8	0.0590 (8)	0.0390 (7)	0.0317 (7)	0.0016 (6)	−0.0030 (6)	−0.0003 (6)
C9	0.0541 (9)	0.0558 (9)	0.0403 (8)	0.0211 (7)	−0.0068 (6)	−0.0004 (7)
C10	0.0426 (8)	0.0794 (11)	0.0616 (10)	0.0212 (7)	0.0093 (7)	0.0155 (9)
C11	0.0440 (8)	0.0628 (9)	0.0525 (9)	0.0130 (7)	0.0107 (7)	0.0184 (7)
C12	0.0339 (6)	0.0371 (7)	0.0444 (8)	0.0023 (5)	0.0040 (5)	−0.0008 (6)
C13	0.0356 (6)	0.0419 (7)	0.0339 (7)	0.0082 (5)	0.0034 (5)	0.0038 (5)
O14A	0.0449 (7)	0.0415 (7)	0.0277 (7)	−0.0021 (5)	0.0057 (5)	−0.0010 (5)
C14A	0.0341 (13)	0.0381 (16)	0.0301 (12)	0.0004 (12)	0.0056 (10)	0.0045 (11)
C15A	0.0342 (15)	0.0333 (17)	0.0304 (12)	0.0033 (13)	0.0124 (11)	−0.0009 (13)
C16A	0.0307 (13)	0.0385 (18)	0.0501 (14)	0.0033 (13)	0.0075 (12)	0.0022 (18)
C17A	0.0351 (14)	0.055 (2)	0.0538 (15)	0.0078 (15)	0.0010 (12)	0.000 (2)
C18A	0.050 (2)	0.041 (3)	0.0565 (16)	0.0191 (19)	0.0078 (16)	0.011 (2)
C19A	0.053 (2)	0.0337 (19)	0.0622 (18)	0.0034 (16)	0.0174 (15)	0.0030 (16)
C20A	0.0372 (17)	0.037 (2)	0.0414 (17)	0.0024 (14)	0.0057 (14)	0.0002 (15)
O14B	0.0513 (19)	0.0391 (18)	0.0432 (19)	0.0023 (14)	0.0136 (15)	0.0083 (15)
C14B	0.040 (3)	0.044 (4)	0.036 (3)	−0.005 (3)	0.016 (2)	0.010 (3)
C15B	0.026 (3)	0.031 (4)	0.029 (3)	0.000 (3)	0.004 (2)	−0.001 (3)
C16B	0.037 (3)	0.033 (4)	0.048 (3)	−0.012 (3)	0.011 (3)	−0.002 (4)
C17B	0.033 (3)	0.044 (5)	0.057 (3)	0.010 (3)	−0.001 (3)	0.001 (4)
C18B	0.049 (3)	0.037 (4)	0.052 (4)	0.001 (3)	0.007 (3)	0.001 (4)
C19B	0.038 (3)	0.025 (3)	0.057 (4)	−0.003 (3)	0.007 (3)	−0.001 (3)
C20B	0.032 (3)	0.028 (3)	0.046 (4)	−0.006 (2)	0.015 (3)	−0.009 (3)
C21	0.0423 (7)	0.0357 (7)	0.0521 (8)	0.0022 (5)	0.0091 (6)	0.0128 (6)
C22	0.0446 (7)	0.0464 (7)	0.0348 (7)	0.0040 (6)	−0.0066 (6)	−0.0021 (6)
C23	0.0473 (8)	0.0396 (7)	0.0565 (9)	0.0083 (6)	−0.0111 (7)	−0.0193 (6)

Geometric parameters (Å, º)

O1—C1	1.2421 (13)	C15A—C20A	1.382 (3)
O4—C4	1.2283 (13)	C15A—C16A	1.392 (3)
N2—C1	1.3380 (15)	C16A—C17A	1.388 (3)
N2—C3	1.4687 (14)	C16A—H16A	0.9500
N2—H2	0.892 (13)	C17A—C18A	1.377 (4)

N5—C4	1.3566 (15)	C17A—H17A	0.9500
N5—C6	1.4402 (14)	C18A—C19A	1.380 (4)
N5—C23	1.4652 (15)	C18A—H18A	0.9500
C1—C12	1.4995 (16)	C19A—C20A	1.379 (3)
C3—C22	1.5229 (16)	C19A—H19A	0.9500
C3—C21	1.5271 (16)	C20A—H20A	0.9500
C3—C4	1.5443 (15)	O14B—C14B	1.423 (8)
C6—C7	1.3748 (17)	O14B—H14B	0.8400
C6—C11	1.3760 (18)	C14B—C15B	1.514 (6)
C7—C8	1.3827 (17)	C14B—H142	1.0000
C7—H7	0.9500	C15B—C20B	1.378 (6)
C8—C9	1.3776 (19)	C15B—C16B	1.395 (6)
C8—H8	0.9500	C16B—C17B	1.400 (7)
C9—C10	1.370 (2)	C16B—H16B	0.9500
C9—H9	0.9500	C17B—C18B	1.368 (7)
C10—C11	1.3844 (19)	C17B—H17B	0.9500
C10—H10	0.9500	C18B—C19B	1.376 (7)
C11—H11	0.9500	C18B—H18B	0.9500
C12—C13	1.5316 (17)	C19B—C20B	1.370 (6)
C12—H121	0.9900	C19B—H19B	0.9500
C12—H122	0.9900	C20B—H20B	0.9500
C13—C14A	1.526 (3)	C21—H211	0.9800
C13—C14B	1.561 (7)	C21—H212	0.9800
C13—H131	0.9900	C21—H213	0.9800
C13—H132	0.9900	C22—H221	0.9800
C13—H133	0.9900	C22—H222	0.9800
C13—H134	0.9900	C22—H223	0.9800
O14A—C14A	1.403 (4)	C23—H231	0.9800
O14A—H14A	0.8400	C23—H232	0.9800
C14A—C15A	1.510 (3)	C23—H233	0.9800
C14A—H141	1.0000		

C1—N2—C3	123.71 (9)	C20A—C15A—C16A	118.1 (2)
C1—N2—H2	119.7 (8)	C20A—C15A—C14A	120.6 (2)
C3—N2—H2	116.6 (8)	C16A—C15A—C14A	121.3 (3)
C4—N5—C6	117.13 (9)	C17A—C16A—C15A	120.3 (3)
C4—N5—C23	128.59 (9)	C17A—C16A—H16A	119.8
C6—N5—C23	114.26 (9)	C15A—C16A—H16A	119.8
O1—C1—N2	121.41 (11)	C18A—C17A—C16A	120.9 (3)
O1—C1—C12	122.24 (11)	C18A—C17A—H17A	119.5
N2—C1—C12	116.33 (10)	C16A—C17A—H17A	119.5
N2—C3—C22	108.58 (9)	C17A—C18A—C19A	118.8 (3)
N2—C3—C21	108.84 (9)	C17A—C18A—H18A	120.6
C22—C3—C21	109.32 (10)	C19A—C18A—H18A	120.6
N2—C3—C4	113.00 (9)	C20A—C19A—C18A	120.5 (3)
C22—C3—C4	108.49 (9)	C20A—C19A—H19A	119.7
C21—C3—C4	108.56 (9)	C18A—C19A—H19A	119.7
O4—C4—N5	120.35 (10)	C19A—C20A—C15A	121.3 (3)

O4—C4—C3	119.23 (10)	C19A—C20A—H20A	119.3
N5—C4—C3	120.12 (9)	C15A—C20A—H20A	119.3
C7—C6—C11	120.13 (11)	C14B—O14B—H14B	109.5
C7—C6—N5	120.53 (10)	O14B—C14B—C15B	109.9 (6)
C11—C6—N5	119.32 (11)	O14B—C14B—C13	108.3 (6)
C6—C7—C8	119.94 (11)	C15B—C14B—C13	115.7 (9)
C6—C7—H7	120.0	O14B—C14B—H142	107.5
C8—C7—H7	120.0	C15B—C14B—H142	107.5
C9—C8—C7	119.85 (12)	C13—C14B—H142	107.5
C9—C8—H8	120.1	C20B—C15B—C16B	118.3 (5)
C7—C8—H8	120.1	C20B—C15B—C14B	119.9 (6)
C10—C9—C8	120.21 (12)	C16B—C15B—C14B	121.7 (6)
C10—C9—H9	119.9	C15B—C16B—C17B	119.1 (5)
C8—C9—H9	119.9	C15B—C16B—H16B	120.4
C9—C10—C11	120.01 (13)	C17B—C16B—H16B	120.4
C9—C10—H10	120.0	C18B—C17B—C16B	121.4 (6)
C11—C10—H10	120.0	C18B—C17B—H17B	119.3
C6—C11—C10	119.85 (13)	C16B—C17B—H17B	119.3
C6—C11—H11	120.1	C17B—C18B—C19B	119.1 (6)
C10—C11—H11	120.1	C17B—C18B—H18B	120.5
C1—C12—C13	112.14 (10)	C19B—C18B—H18B	120.5
C1—C12—H121	109.2	C20B—C19B—C18B	120.2 (5)
C13—C12—H121	109.2	C20B—C19B—H19B	119.9
C1—C12—H122	109.2	C18B—C19B—H19B	119.9
C13—C12—H122	109.2	C19B—C20B—C15B	121.9 (5)
H121—C12—H122	107.9	C19B—C20B—H20B	119.1
C14A—C13—C12	110.96 (19)	C15B—C20B—H20B	119.1
C12—C13—C14B	116.2 (5)	C3—C21—H211	109.5
C14A—C13—H131	109.4	C3—C21—H212	109.5
C12—C13—H131	109.4	H211—C21—H212	109.5
C14A—C13—H132	109.4	C3—C21—H213	109.5
C12—C13—H132	109.4	H211—C21—H213	109.5
H131—C13—H132	108.0	H212—C21—H213	109.5
C12—C13—H133	108.2	C3—C22—H221	109.5
C14B—C13—H133	108.2	C3—C22—H222	109.5
C12—C13—H134	108.2	H221—C22—H222	109.5
C14B—C13—H134	108.2	C3—C22—H223	109.5
H133—C13—H134	107.4	H221—C22—H223	109.5
C14A—O14A—H14A	109.5	H222—C22—H223	109.5
O14A—C14A—C15A	112.3 (3)	N5—C23—H231	109.5
O14A—C14A—C13	106.3 (3)	N5—C23—H232	109.5
C15A—C14A—C13	109.6 (3)	H231—C23—H232	109.5
O14A—C14A—H141	109.5	N5—C23—H233	109.5
C15A—C14A—H141	109.5	H231—C23—H233	109.5
C13—C14A—H141	109.5	H232—C23—H233	109.5
C3—N2—C1—O1	−3.73 (17)	C1—C12—C13—C14B	177.8 (3)
C3—N2—C1—C12	177.94 (9)	C12—C13—C14A—O14A	−177.22 (16)

C1—N2—C3—C22	−73.44 (13)	C12—C13—C14A—C15A	61.1 (3)
C1—N2—C3—C21	167.65 (10)	O14A—C14A—C15A—C20A	−41.0 (6)
C1—N2—C3—C4	46.96 (14)	C13—C14A—C15A—C20A	76.9 (6)
C6—N5—C4—O4	−0.62 (15)	O14A—C14A—C15A—C16A	141.9 (5)
C23—N5—C4—O4	177.67 (12)	C13—C14A—C15A—C16A	−100.1 (6)
C6—N5—C4—C3	173.10 (9)	C20A—C15A—C16A—C17A	−0.1 (9)
C23—N5—C4—C3	−8.62 (18)	C14A—C15A—C16A—C17A	177.0 (6)
N2—C3—C4—O4	−132.56 (10)	C15A—C16A—C17A—C18A	1.8 (11)
C22—C3—C4—O4	−12.11 (14)	C16A—C17A—C18A—C19A	−2.2 (11)
C21—C3—C4—O4	106.59 (11)	C17A—C18A—C19A—C20A	1.0 (10)
N2—C3—C4—N5	53.65 (13)	C18A—C19A—C20A—C15A	0.7 (9)
C22—C3—C4—N5	174.10 (10)	C16A—C15A—C20A—C19A	−1.1 (9)
C21—C3—C4—N5	−67.19 (13)	C14A—C15A—C20A—C19A	−178.2 (5)
C4—N5—C6—C7	94.72 (13)	C12—C13—C14B—O14B	−73.5 (7)
C23—N5—C6—C7	−83.80 (13)	C12—C13—C14B—C15B	50.3 (7)
C4—N5—C6—C11	−87.21 (14)	O14B—C14B—C15B—C20B	−151.9 (12)
C23—N5—C6—C11	94.26 (14)	C13—C14B—C15B—C20B	85.1 (15)
C11—C6—C7—C8	0.64 (18)	O14B—C14B—C15B—C16B	31.6 (18)
N5—C6—C7—C8	178.68 (10)	C13—C14B—C15B—C16B	−91.4 (15)
C6—C7—C8—C9	−1.18 (17)	C20B—C15B—C16B—C17B	3 (2)
C7—C8—C9—C10	1.1 (2)	C14B—C15B—C16B—C17B	179.2 (16)
C8—C9—C10—C11	−0.5 (2)	C15B—C16B—C17B—C18B	0 (3)
C7—C6—C11—C10	0.0 (2)	C16B—C17B—C18B—C19B	−1 (3)
N5—C6—C11—C10	−178.11 (12)	C17B—C18B—C19B—C20B	0 (3)
C9—C10—C11—C6	0.0 (2)	C18B—C19B—C20B—C15B	3 (2)
O1—C1—C12—C13	−90.96 (14)	C16B—C15B—C20B—C19B	−4 (2)
N2—C1—C12—C13	87.36 (13)	C14B—C15B—C20B—C19B	179.2 (14)
C1—C12—C13—C14A	164.51 (16)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O14*A*—H14*A*···O1^{i}	0.84	1.95	2.7868 (14)	174
O14*B*—H14*B*···O1^{i}	0.84	1.93	2.754 (3)	166
N2—H2···O4^{ii}	0.892 (13)	2.060 (14)	2.9064 (12)	158.1 (12)

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

N-[1-Methyl-1-(*N*-methyl-*N*-phenylcarbamoyl)ethyl]-4-oxo-4-phenylbutanamide (9d)

Crystal data

$C_{21}H_{24}N_2O_3$
M_r = 352.42
Orthorhombic, *Aea*2
a = 13.0433 (4) Å
b = 25.8095 (8) Å
c = 11.3381 (3) Å
V = 3816.87 (19) Å^3
Z = 8
F(000) = 1504

D_x = 1.227 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 1904 reflections
θ = 2.0–25.0°
μ = 0.08 mm^{-1}
T = 160 K
Tablet, colourless
0.30 × 0.18 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: Nonius FR591 sealed tube generator
Horizontally mounted graphite crystal monochromator
Detector resolution: 9 pixels mm^{-1}
φ and ω scans with κ offsets

26178 measured reflections
3258 independent reflections
2823 reflections with $I > 2\sigma(I)$
$R_{int} = 0.069$
$\theta_{max} = 25.0°$, $\theta_{min} = 3.1°$
$h = -15 \rightarrow 15$
$k = -30 \rightarrow 30$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.092$
$S = 1.06$
3258 reflections
292 parameters
103 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0423P)^2 + 1.3647P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.15$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}
Extinction correction: SHELXL-2025 (Sheldrick, 2015), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0043 (7)
Absolute structure: Flack *x* determined using 1135 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013). The compound in the crystal is itself achiral.
Absolute structure parameter: 0.1 (7)

Special details

Experimental. Solvent used: dichloromethane / acetonitrile Cooling Device: Oxford Cryosystems Cryostream 700 Crystal mount: glued on a glass fibre Mosaicity (deg.): 0.809 (2) Frames collected: 204 Seconds exposure per frame: 100 Degrees rotation per frame: 2.0 Crystal-Detector distance (mm): 30.0

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.

Refinement. The keto group and its neighbouring phenyl group are disordered over two conformations. Two positions were defined for these atoms and the site occupation factor of the major conformation of the group refined to 0.572 (10). Rigid group constraints were applied to each orientation of the disordered phenyl ring, while bond length restraints were applied to both sets of the disordered C13–C14, C14–O14 and C14–C15 bonds. In addition, pseudo-isotropic restraints were applied to the anisotropic displacement parameters of all disordered atoms.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.80609 (15)	0.19224 (8)	0.45049 (18)	0.0420 (5)	
O4	0.95872 (13)	0.23387 (7)	0.66415 (16)	0.0336 (5)	
N2	0.90974 (18)	0.25104 (10)	0.3632 (2)	0.0335 (5)	
H2	0.939 (2)	0.2599 (11)	0.296 (3)	0.033 (8)*	
N5	1.04461 (16)	0.19148 (9)	0.52324 (19)	0.0305 (5)	
C1	0.8409 (2)	0.21248 (12)	0.3601 (3)	0.0372 (7)	
C3	0.9483 (2)	0.27344 (11)	0.4735 (2)	0.0324 (6)	
C4	0.9838 (2)	0.23098 (10)	0.5596 (2)	0.0291 (6)	
C6	1.0565 (2)	0.14857 (10)	0.6042 (2)	0.0311 (6)	

C7	0.9814 (2)	0.11081 (11)	0.6066 (3)	0.0384 (7)	
H7	0.923665	0.113319	0.555835	0.046*	
C8	0.9907 (2)	0.06926 (12)	0.6833 (3)	0.0466 (8)	
H8	0.939288	0.043221	0.685266	0.056*	
C9	1.0749 (3)	0.06575 (11)	0.7568 (3)	0.0444 (8)	
H9	1.080903	0.037577	0.810296	0.053*	
C10	1.1504 (2)	0.10335 (11)	0.7524 (3)	0.0400 (7)	
H10	1.208880	0.100541	0.801891	0.048*	
C11	1.1411 (2)	0.14493 (11)	0.6766 (3)	0.0359 (7)	
H11	1.192764	0.170870	0.674294	0.043*	
C12	0.8070 (3)	0.19600 (16)	0.2384 (3)	0.0606 (11)	
H121	0.866898	0.196186	0.184759	0.073*	
H122	0.756470	0.221276	0.207879	0.073*	
C13	0.7594 (3)	0.14244 (13)	0.2386 (3)	0.0555 (9)	
H131	0.745808	0.132428	0.155813	0.067*	0.572 (10)
H132	0.692319	0.144673	0.279154	0.067*	0.572 (10)
H133	0.706857	0.139767	0.301343	0.067*	0.428 (10)
H134	0.726483	0.135196	0.161711	0.067*	0.428 (10)
O14A	0.9170 (4)	0.0939 (3)	0.2773 (8)	0.078 (2)	0.572 (10)
C14A	0.8228 (5)	0.0979 (3)	0.2979 (8)	0.059 (2)	0.572 (10)
C15A	0.7787 (4)	0.05687 (18)	0.3830 (5)	0.048 (2)	0.572 (10)
C16A	0.6861 (4)	0.06490 (16)	0.4407 (5)	0.056 (2)	0.572 (10)
H16A	0.648186	0.095751	0.426956	0.067*	0.572 (10)
C17A	0.6488 (5)	0.02779 (19)	0.5185 (4)	0.065 (2)	0.572 (10)
H17A	0.585461	0.033277	0.557962	0.078*	0.572 (10)
C18A	0.7042 (6)	−0.01735 (17)	0.5387 (5)	0.074 (3)	0.572 (10)
H18A	0.678762	−0.042716	0.591865	0.088*	0.572 (10)
C19A	0.7969 (5)	−0.02538 (16)	0.4810 (6)	0.073 (4)	0.572 (10)
H19A	0.834790	−0.056235	0.494763	0.088*	0.572 (10)
C20A	0.8342 (4)	0.0117 (2)	0.4032 (6)	0.069 (2)	0.572 (10)
H20A	0.897518	0.006239	0.363757	0.082*	0.572 (10)
O14B	0.9259 (5)	0.1117 (3)	0.2172 (8)	0.061 (2)	0.428 (10)
C14B	0.8431 (6)	0.1056 (4)	0.2604 (10)	0.049 (3)	0.428 (10)
C15B	0.8115 (5)	0.0634 (2)	0.3414 (6)	0.042 (3)	0.428 (10)
C16B	0.7297 (6)	0.0654 (2)	0.4198 (6)	0.043 (3)	0.428 (10)
H16B	0.686173	0.094872	0.421320	0.052*	0.428 (10)
C17B	0.7114 (7)	0.0241 (3)	0.4959 (6)	0.063 (3)	0.428 (10)
H17B	0.655446	0.025511	0.549440	0.075*	0.428 (10)
C18B	0.7750 (8)	−0.0190 (2)	0.4937 (7)	0.072 (5)	0.428 (10)
H18B	0.762546	−0.047206	0.545671	0.087*	0.428 (10)
C19B	0.8569 (7)	−0.0210 (2)	0.4153 (8)	0.070 (3)	0.428 (10)
H19B	0.900376	−0.050561	0.413782	0.084*	0.428 (10)
C20B	0.8751 (5)	0.0202 (2)	0.3392 (7)	0.056 (3)	0.428 (10)
H20B	0.931105	0.018800	0.285660	0.068*	0.428 (10)
C21	1.0374 (2)	0.31005 (12)	0.4448 (3)	0.0400 (7)	
H211	1.016562	0.334152	0.382514	0.060*	
H212	1.096529	0.289779	0.417969	0.060*	
H213	1.056210	0.329613	0.515688	0.060*	

C22	0.8626 (2)	0.30456 (12)	0.5314 (3)	0.0413 (7)
H221	0.804407	0.281674	0.548138	0.062*
H222	0.840613	0.332312	0.478138	0.062*
H223	0.887635	0.319687	0.605301	0.062*
C23	1.0850 (2)	0.18181 (13)	0.4036 (2)	0.0399 (7)
H231	1.085634	0.214309	0.358945	0.060*
H232	1.041232	0.156538	0.363307	0.060*
H233	1.154940	0.168162	0.409254	0.060*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0387 (12)	0.0570 (13)	0.0303 (11)	−0.0107 (10)	0.0026 (9)	−0.0003 (10)
O4	0.0384 (10)	0.0390 (11)	0.0234 (11)	0.0030 (9)	−0.0002 (9)	−0.0006 (8)
N2	0.0337 (13)	0.0467 (14)	0.0203 (12)	−0.0005 (11)	0.0013 (11)	0.0026 (10)
N5	0.0326 (12)	0.0351 (13)	0.0238 (12)	0.0018 (10)	0.0028 (9)	0.0009 (9)
C1	0.0303 (14)	0.0509 (17)	0.0304 (16)	−0.0030 (13)	−0.0021 (13)	0.0003 (14)
C3	0.0352 (15)	0.0375 (15)	0.0244 (14)	0.0016 (12)	−0.0025 (12)	0.0017 (12)
C4	0.0252 (13)	0.0354 (15)	0.0266 (15)	−0.0043 (11)	−0.0014 (11)	0.0000 (11)
C6	0.0337 (15)	0.0316 (14)	0.0279 (14)	0.0027 (11)	0.0038 (12)	−0.0020 (12)
C7	0.0376 (15)	0.0379 (15)	0.0398 (15)	0.0014 (13)	−0.0013 (13)	−0.0023 (14)
C8	0.0510 (18)	0.0351 (16)	0.054 (2)	−0.0037 (14)	0.0028 (17)	0.0022 (15)
C9	0.063 (2)	0.0310 (15)	0.0396 (17)	0.0055 (15)	0.0008 (16)	0.0041 (13)
C10	0.0455 (16)	0.0401 (16)	0.0344 (16)	0.0092 (14)	−0.0055 (14)	−0.0012 (14)
C11	0.0363 (15)	0.0367 (15)	0.0348 (16)	0.0009 (12)	0.0026 (13)	−0.0013 (13)
C12	0.053 (2)	0.099 (3)	0.0303 (18)	−0.028 (2)	−0.0017 (15)	−0.0070 (18)
C13	0.0436 (18)	0.076 (2)	0.0469 (19)	−0.0053 (18)	−0.0065 (16)	−0.0223 (17)
O14A	0.038 (3)	0.096 (5)	0.101 (6)	−0.004 (3)	0.005 (3)	−0.037 (4)
C14A	0.036 (3)	0.076 (5)	0.066 (5)	−0.006 (4)	0.006 (4)	−0.048 (4)
C15A	0.046 (4)	0.047 (4)	0.052 (5)	0.014 (3)	−0.013 (4)	−0.022 (3)
C16A	0.058 (5)	0.054 (4)	0.055 (4)	0.008 (3)	−0.003 (4)	−0.008 (3)
C17A	0.080 (5)	0.061 (4)	0.054 (4)	0.003 (3)	−0.007 (3)	−0.002 (3)
C18A	0.099 (5)	0.060 (4)	0.063 (4)	0.004 (4)	−0.032 (4)	−0.002 (3)
C19A	0.085 (6)	0.060 (5)	0.075 (6)	0.015 (4)	−0.034 (5)	−0.004 (4)
C20A	0.060 (4)	0.069 (4)	0.076 (5)	0.011 (3)	−0.023 (4)	−0.022 (4)
O14B	0.059 (4)	0.061 (4)	0.064 (5)	0.004 (3)	0.020 (4)	0.005 (3)
C14B	0.039 (5)	0.057 (5)	0.050 (6)	−0.007 (4)	0.005 (4)	−0.018 (4)
C15B	0.041 (5)	0.046 (5)	0.038 (5)	−0.005 (4)	−0.013 (4)	−0.009 (4)
C16B	0.049 (6)	0.037 (4)	0.044 (5)	0.003 (4)	0.002 (4)	−0.001 (3)
C17B	0.077 (6)	0.061 (5)	0.050 (5)	−0.016 (4)	−0.005 (4)	0.011 (4)
C18B	0.088 (7)	0.060 (6)	0.069 (7)	−0.002 (5)	−0.025 (5)	−0.001 (5)
C19B	0.083 (6)	0.055 (5)	0.072 (6)	−0.004 (4)	−0.029 (5)	0.003 (4)
C20B	0.062 (5)	0.055 (5)	0.052 (5)	0.007 (4)	−0.017 (4)	−0.003 (4)
C21	0.0466 (18)	0.0387 (16)	0.0348 (16)	−0.0059 (13)	−0.0066 (14)	0.0042 (13)
C22	0.0500 (18)	0.0402 (16)	0.0337 (17)	0.0104 (14)	−0.0013 (13)	0.0038 (13)
C23	0.0372 (16)	0.0516 (19)	0.0310 (16)	0.0087 (14)	0.0058 (13)	0.0005 (13)

Geometric parameters (Å, º)

O1—C1	1.237 (4)	C15A—C16A	1.3900
O4—C4	1.232 (3)	C15A—C20A	1.3900
N2—C1	1.341 (4)	C16A—C17A	1.3900
N2—C3	1.467 (4)	C16A—H16A	0.9500
N2—H2	0.88 (3)	C17A—C18A	1.3900
N5—C4	1.356 (3)	C17A—H17A	0.9500
N5—C6	1.447 (3)	C18A—C19A	1.3900
N5—C23	1.476 (4)	C18A—H18A	0.9500
C1—C12	1.510 (4)	C19A—C20A	1.3900
C3—C22	1.526 (4)	C19A—H19A	0.9500
C3—C21	1.533 (4)	C20A—H20A	0.9500
C3—C4	1.539 (4)	O14B—C14B	1.196 (6)
C6—C11	1.379 (4)	C14B—C15B	1.484 (6)
C6—C7	1.382 (4)	C15B—C16B	1.3900
C7—C8	1.386 (4)	C15B—C20B	1.3900
C7—H7	0.9500	C16B—C17B	1.3900
C8—C9	1.381 (5)	C16B—H16B	0.9500
C8—H8	0.9500	C17B—C18B	1.3900
C9—C10	1.383 (4)	C17B—H17B	0.9500
C9—H9	0.9500	C18B—C19B	1.3900
C10—C11	1.380 (4)	C18B—H18B	0.9500
C10—H10	0.9500	C19B—C20B	1.3900
C11—H11	0.9500	C19B—H19B	0.9500
C12—C13	1.515 (5)	C20B—H20B	0.9500
C12—H121	0.9900	C21—H211	0.9800
C12—H122	0.9900	C21—H212	0.9800
C13—C14B	1.468 (6)	C21—H213	0.9800
C13—C14A	1.567 (6)	C22—H221	0.9800
C13—H131	0.9900	C22—H222	0.9800
C13—H132	0.9900	C22—H223	0.9800
C13—H133	0.9900	C23—H231	0.9800
C13—H134	0.9900	C23—H232	0.9800
O14A—C14A	1.255 (6)	C23—H233	0.9800
C14A—C15A	1.544 (6)		
C1—N2—C3	123.0 (3)	C16A—C15A—C20A	120.0
C1—N2—H2	117.1 (19)	C16A—C15A—C14A	121.0 (4)
C3—N2—H2	119.3 (19)	C20A—C15A—C14A	119.0 (4)
C4—N5—C6	116.5 (2)	C15A—C16A—C17A	120.0
C4—N5—C23	128.0 (2)	C15A—C16A—H16A	120.0
C6—N5—C23	114.5 (2)	C17A—C16A—H16A	120.0
O1—C1—N2	122.5 (3)	C18A—C17A—C16A	120.0
O1—C1—C12	122.0 (3)	C18A—C17A—H17A	120.0
N2—C1—C12	115.4 (3)	C16A—C17A—H17A	120.0
N2—C3—C22	108.9 (2)	C17A—C18A—C19A	120.0
N2—C3—C21	108.8 (2)	C17A—C18A—H18A	120.0

C22—C3—C21	108.8 (2)	C19A—C18A—H18A	120.0
N2—C3—C4	111.3 (2)	C18A—C19A—C20A	120.0
C22—C3—C4	108.8 (2)	C18A—C19A—H19A	120.0
C21—C3—C4	110.2 (2)	C20A—C19A—H19A	120.0
O4—C4—N5	119.5 (2)	C19A—C20A—C15A	120.0
O4—C4—C3	119.2 (2)	C19A—C20A—H20A	120.0
N5—C4—C3	121.3 (2)	C15A—C20A—H20A	120.0
C11—C6—C7	120.5 (3)	O14B—C14B—C13	121.1 (6)
C11—C6—N5	121.1 (2)	O14B—C14B—C15B	126.9 (7)
C7—C6—N5	118.4 (2)	C13—C14B—C15B	112.0 (5)
C6—C7—C8	119.7 (3)	C16B—C15B—C20B	120.0
C6—C7—H7	120.1	C16B—C15B—C14B	125.5 (5)
C8—C7—H7	120.1	C20B—C15B—C14B	114.4 (5)
C9—C8—C7	119.9 (3)	C15B—C16B—C17B	120.0
C9—C8—H8	120.0	C15B—C16B—H16B	120.0
C7—C8—H8	120.0	C17B—C16B—H16B	120.0
C8—C9—C10	119.8 (3)	C18B—C17B—C16B	120.0
C8—C9—H9	120.1	C18B—C17B—H17B	120.0
C10—C9—H9	120.1	C16B—C17B—H17B	120.0
C11—C10—C9	120.4 (3)	C17B—C18B—C19B	120.0
C11—C10—H10	119.8	C17B—C18B—H18B	120.0
C9—C10—H10	119.8	C19B—C18B—H18B	120.0
C6—C11—C10	119.6 (3)	C20B—C19B—C18B	120.0
C6—C11—H11	120.2	C20B—C19B—H19B	120.0
C10—C11—H11	120.2	C18B—C19B—H19B	120.0
C1—C12—C13	112.1 (3)	C19B—C20B—C15B	120.0
C1—C12—H121	109.2	C19B—C20B—H20B	120.0
C13—C12—H121	109.2	C15B—C20B—H20B	120.0
C1—C12—H122	109.2	C3—C21—H211	109.5
C13—C12—H122	109.2	C3—C21—H212	109.5
H121—C12—H122	107.9	H211—C21—H212	109.5
C14B—C13—C12	106.6 (5)	C3—C21—H213	109.5
C12—C13—C14A	117.0 (4)	H211—C21—H213	109.5
C12—C13—H131	108.1	H212—C21—H213	109.5
C14A—C13—H131	108.1	C3—C22—H221	109.5
C12—C13—H132	108.1	C3—C22—H222	109.5
C14A—C13—H132	108.1	H221—C22—H222	109.5
H131—C13—H132	107.3	C3—C22—H223	109.5
C14B—C13—H133	110.4	H221—C22—H223	109.5
C12—C13—H133	110.4	H222—C22—H223	109.5
C14B—C13—H134	110.4	N5—C23—H231	109.5
C12—C13—H134	110.4	N5—C23—H232	109.5
H133—C13—H134	108.6	H231—C23—H232	109.5
O14A—C14A—C15A	115.1 (6)	N5—C23—H233	109.5
O14A—C14A—C13	119.8 (6)	H231—C23—H233	109.5
C15A—C14A—C13	125.1 (5)	H232—C23—H233	109.5
C3—N2—C1—O1	−1.3 (5)	C1—C12—C13—C14A	−51.9 (5)

C3—N2—C1—C12	177.5 (3)	C12—C13—C14A—O14A	−44.4 (10)
C1—N2—C3—C22	−70.5 (3)	C12—C13—C14A—C15A	135.3 (7)
C1—N2—C3—C21	171.0 (3)	O14A—C14A—C15A—C16A	160.7 (6)
C1—N2—C3—C4	49.4 (4)	C13—C14A—C15A—C16A	−19.1 (10)
C6—N5—C4—O4	14.1 (3)	O14A—C14A—C15A—C20A	−18.1 (10)
C23—N5—C4—O4	−178.4 (3)	C13—C14A—C15A—C20A	162.1 (6)
C6—N5—C4—C3	−168.1 (2)	C20A—C15A—C16A—C17A	0.0
C23—N5—C4—C3	−0.5 (4)	C14A—C15A—C16A—C17A	−178.9 (6)
N2—C3—C4—O4	−135.1 (2)	C15A—C16A—C17A—C18A	0.0
C22—C3—C4—O4	−15.2 (3)	C16A—C17A—C18A—C19A	0.0
C21—C3—C4—O4	104.1 (3)	C17A—C18A—C19A—C20A	0.0
N2—C3—C4—N5	47.0 (3)	C18A—C19A—C20A—C15A	0.0
C22—C3—C4—N5	167.0 (2)	C16A—C15A—C20A—C19A	0.0
C21—C3—C4—N5	−73.8 (3)	C14A—C15A—C20A—C19A	178.9 (6)
C4—N5—C6—C11	−98.2 (3)	C12—C13—C14B—O14B	−38.9 (13)
C23—N5—C6—C11	92.6 (3)	C12—C13—C14B—C15B	139.7 (8)
C4—N5—C6—C7	82.5 (3)	O14B—C14B—C15B—C16B	157.5 (11)
C23—N5—C6—C7	−86.7 (3)	C13—C14B—C15B—C16B	−21.0 (13)
C11—C6—C7—C8	0.7 (4)	O14B—C14B—C15B—C20B	−19.0 (16)
N5—C6—C7—C8	−180.0 (3)	C13—C14B—C15B—C20B	162.5 (6)
C6—C7—C8—C9	0.0 (5)	C20B—C15B—C16B—C17B	0.0
C7—C8—C9—C10	−0.9 (5)	C14B—C15B—C16B—C17B	−176.4 (9)
C8—C9—C10—C11	1.3 (5)	C15B—C16B—C17B—C18B	0.0
C7—C6—C11—C10	−0.4 (4)	C16B—C17B—C18B—C19B	0.0
N5—C6—C11—C10	−179.7 (2)	C17B—C18B—C19B—C20B	0.0
C9—C10—C11—C6	−0.6 (4)	C18B—C19B—C20B—C15B	0.0
O1—C1—C12—C13	−20.4 (5)	C16B—C15B—C20B—C19B	0.0
N2—C1—C12—C13	160.7 (3)	C14B—C15B—C20B—C19B	176.8 (8)
C1—C12—C13—C14B	−70.5 (6)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O4^{i}	0.88 (3)	2.01 (3)	2.862 (3)	161 (3)

Symmetry code: (i) −*x*+2, −*y*+1/2, *z*−1/2.

2-[(4-Benzyloxy-3,3-dimethylbutanoyl)amino]-2-methylpropanoic acid (10)

Crystal data

$C_{17}H_{25}NO_4$
M_r = 307.38
Orthorhombic, $Pca2_1$
a = 10.1569 (5) Å
b = 21.3548 (10) Å
c = 7.7969 (3) Å
V = 1691.14 (13) Å^3
Z = 4
F(000) = 664

D_x = 1.207 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1757 reflections
θ = 2.0–25.0°
μ = 0.09 mm^{-1}
T = 160 K
Plate, colourless
0.45 × 0.32 × 0.07 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Radiation source: Nonius FR590 sealed tube generator
Horizontally mounted graphite crystal monochromator
Detector resolution: 9 pixels mm^{-1}
φ and ω scans with κ offsets

21591 measured reflections
1614 independent reflections
1306 reflections with $I > 2\sigma(I)$
$R_{int} = 0.091$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.2°$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 25$
$l = 0 \rightarrow 9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.094$
$S = 1.11$
1614 reflections
212 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0475P)^2 + 0.0521P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.14$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}
Extinction correction: SHELXL-2025 (Sheldrick, 2015), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.016 (3)
Absolute structure: Bijvoet pairs were merged during data reduction, so the absolute structure parameter was not meaningful. The compound in the crystal is itself achiral.

Special details

Experimental. Solvent used: dichloromethane / acetone / hexane / diethyl ether Cooling Device: Oxford Cryosystems Cryostream 700 Crystal mount: glued on a glass fibre Mosaicity (deg.): 0.791 (2) Frames collected: 295 Seconds exposure per frame: 25 Degrees rotation per frame: 1.4 Crystal-Detector distance (mm): 32.2

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.9617 (2)	0.39458 (9)	0.5515 (3)	0.0428 (6)
O2	0.92737 (19)	0.20388 (10)	0.8381 (3)	0.0401 (5)
O4	0.9173 (2)	0.32388 (9)	1.1370 (3)	0.0435 (6)
O5	0.9778 (2)	0.41924 (10)	1.2227 (3)	0.0518 (7)
H5	0.977 (4)	0.4009 (18)	1.321 (6)	0.072 (13)*
N2	0.9239 (3)	0.35057 (12)	0.8109 (3)	0.0347 (7)
H2	0.903 (3)	0.3168 (14)	0.866 (4)	0.040 (10)*
C1	0.9285 (3)	0.34893 (14)	0.6396 (4)	0.0356 (7)
C3	0.9615 (3)	0.40335 (13)	0.9229 (4)	0.0358 (8)
C4	0.9492 (3)	0.37728 (15)	1.1058 (4)	0.0348 (7)
C6	1.0277 (3)	0.11840 (13)	1.0044 (4)	0.0351 (7)
C7	1.1444 (3)	0.10925 (14)	1.0913 (4)	0.0428 (8)
H7	1.183597	0.143365	1.150588	0.051*

C8	1.2055 (3)	0.05136 (16)	1.0938 (4)	0.0538 (9)
H8	1.286151	0.045773	1.153655	0.065*
C9	1.1480 (4)	0.00178 (15)	1.0082 (4)	0.0509 (9)
H9	1.188112	−0.038379	1.011869	0.061*
C10	1.0338 (4)	0.00998 (14)	0.9185 (5)	0.0468 (8)
H10	0.995957	−0.024006	0.857258	0.056*
C11	0.9736 (3)	0.06816 (14)	0.9172 (4)	0.0410 (8)
H11	0.893887	0.073672	0.855371	0.049*
C12	0.8889 (3)	0.28694 (12)	0.5606 (4)	0.0355 (7)
H121	0.860135	0.294462	0.441042	0.043*
H122	0.812405	0.270191	0.624610	0.043*
C13	0.9986 (3)	0.23688 (13)	0.5594 (4)	0.0374 (7)
C14	1.0408 (3)	0.21885 (13)	0.7395 (4)	0.0382 (7)
H141	1.100577	0.182273	0.734856	0.046*
H142	1.088719	0.254080	0.793479	0.046*
C15	0.9599 (3)	0.18142 (15)	1.0056 (4)	0.0430 (8)
H151	0.878225	0.178229	1.074342	0.052*
H152	1.018140	0.212277	1.062457	0.052*
C16	1.1198 (3)	0.26040 (16)	0.4630 (5)	0.0514 (9)
H161	1.154083	0.297949	0.519765	0.077*
H162	1.095413	0.270603	0.344615	0.077*
H163	1.187574	0.227750	0.462680	0.077*
C17	0.9428 (4)	0.17882 (15)	0.4706 (5)	0.0503 (9)
H171	1.012662	0.147532	0.457108	0.075*
H172	0.908585	0.190466	0.357512	0.075*
H173	0.871533	0.161259	0.540269	0.075*
C18	0.8661 (3)	0.45760 (13)	0.9016 (4)	0.0459 (9)
H181	0.866515	0.471548	0.781835	0.069*
H182	0.893093	0.492279	0.976014	0.069*
H183	0.777241	0.443960	0.933144	0.069*
C19	1.1039 (3)	0.42274 (15)	0.8920 (4)	0.0477 (9)
H191	1.161778	0.386617	0.909827	0.072*
H192	1.127881	0.456156	0.972304	0.072*
H193	1.113544	0.437935	0.774089	0.072*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0729 (16)	0.0311 (12)	0.0242 (12)	−0.0031 (10)	0.0010 (11)	0.0044 (10)
O2	0.0484 (13)	0.0443 (13)	0.0277 (11)	0.0100 (10)	0.0062 (10)	0.0089 (9)
O4	0.0714 (16)	0.0327 (13)	0.0264 (11)	−0.0088 (10)	0.0002 (11)	0.0044 (10)
O5	0.0981 (19)	0.0352 (12)	0.0223 (12)	−0.0159 (11)	−0.0040 (13)	0.0001 (12)
N2	0.0522 (18)	0.0287 (14)	0.0231 (16)	−0.0016 (12)	−0.0022 (12)	0.0012 (11)
C1	0.0457 (19)	0.0367 (18)	0.0246 (18)	0.0063 (14)	−0.0039 (14)	−0.0014 (14)
C3	0.053 (2)	0.0296 (16)	0.0245 (17)	−0.0058 (14)	0.0005 (14)	−0.0011 (13)
C4	0.048 (2)	0.0345 (18)	0.0214 (16)	−0.0013 (14)	−0.0029 (14)	−0.0017 (13)
C6	0.0397 (18)	0.0414 (17)	0.0242 (15)	−0.0026 (14)	0.0020 (13)	0.0045 (13)
C7	0.0431 (19)	0.0508 (19)	0.0345 (18)	−0.0070 (15)	−0.0042 (15)	0.0039 (14)

C8	0.044 (2)	0.072 (2)	0.046 (2)	0.0100 (17)	−0.0008 (17)	0.0198 (17)
C9	0.060 (2)	0.043 (2)	0.049 (2)	0.0128 (16)	0.0136 (18)	0.0161 (16)
C10	0.059 (2)	0.0360 (19)	0.045 (2)	−0.0045 (16)	0.0091 (17)	−0.0022 (16)
C11	0.0404 (18)	0.0460 (19)	0.0364 (18)	−0.0035 (15)	−0.0014 (14)	0.0027 (16)
C12	0.0470 (18)	0.0344 (16)	0.0252 (16)	0.0025 (14)	−0.0059 (14)	−0.0035 (13)
C13	0.0462 (17)	0.0359 (17)	0.0301 (16)	0.0017 (14)	0.0058 (14)	0.0030 (14)
C14	0.0428 (17)	0.0387 (17)	0.0330 (17)	0.0053 (13)	0.0030 (16)	0.0054 (16)
C15	0.057 (2)	0.046 (2)	0.0255 (18)	0.0080 (16)	0.0024 (15)	0.0056 (14)
C16	0.058 (2)	0.053 (2)	0.043 (2)	0.0066 (17)	0.0114 (17)	0.0079 (16)
C17	0.075 (3)	0.037 (2)	0.039 (2)	0.0064 (16)	0.0063 (18)	−0.0060 (15)
C18	0.072 (2)	0.0337 (17)	0.0318 (17)	0.0056 (16)	−0.0024 (16)	0.0010 (14)
C19	0.061 (2)	0.0473 (19)	0.0346 (18)	−0.0145 (16)	0.0002 (16)	0.0019 (15)

Geometric parameters (Å, º)

O1—C1	1.239 (4)	C11—H11	0.9500
O2—C14	1.421 (3)	C12—C13	1.545 (4)
O2—C15	1.430 (4)	C12—H121	0.9900
O4—C4	1.210 (4)	C12—H122	0.9900
O5—C4	1.310 (4)	C13—C14	1.518 (5)
O5—H5	0.86 (4)	C13—C16	1.527 (5)
N2—C1	1.337 (4)	C13—C17	1.529 (5)
N2—C3	1.476 (4)	C14—H141	0.9900
N2—H2	0.86 (3)	C14—H142	0.9900
C1—C12	1.515 (4)	C15—H151	0.9900
C3—C18	1.519 (4)	C15—H152	0.9900
C3—C19	1.523 (4)	C16—H161	0.9800
C3—C4	1.536 (4)	C16—H162	0.9800
C6—C7	1.380 (4)	C16—H163	0.9800
C6—C11	1.384 (4)	C17—H171	0.9800
C6—C15	1.512 (4)	C17—H172	0.9800
C7—C8	1.384 (4)	C17—H173	0.9800
C7—H7	0.9500	C18—H181	0.9800
C8—C9	1.381 (5)	C18—H182	0.9800
C8—H8	0.9500	C18—H183	0.9800
C9—C10	1.365 (5)	C19—H191	0.9800
C9—H9	0.9500	C19—H192	0.9800
C10—C11	1.385 (4)	C19—H193	0.9800
C10—H10	0.9500		

C14—O2—C15	112.5 (2)	C14—C13—C17	108.5 (2)
C4—O5—H5	108 (3)	C16—C13—C17	110.0 (3)
C1—N2—C3	127.0 (3)	C14—C13—C12	111.9 (2)
C1—N2—H2	119 (2)	C16—C13—C12	110.9 (2)
C3—N2—H2	114 (2)	C17—C13—C12	107.2 (3)
O1—C1—N2	122.9 (3)	O2—C14—C13	109.2 (2)
O1—C1—C12	122.3 (3)	O2—C14—H141	109.8
N2—C1—C12	114.8 (3)	C13—C14—H141	109.8

N2—C3—C18	110.7 (3)	O2—C14—H142	109.8
N2—C3—C19	111.1 (2)	C13—C14—H142	109.8
C18—C3—C19	112.4 (3)	H141—C14—H142	108.3
N2—C3—C4	104.6 (2)	O2—C15—C6	113.5 (3)
C18—C3—C4	109.0 (3)	O2—C15—H151	108.9
C19—C3—C4	108.8 (3)	C6—C15—H151	108.9
O4—C4—O5	124.3 (3)	O2—C15—H152	108.9
O4—C4—C3	123.4 (3)	C6—C15—H152	108.9
O5—C4—C3	112.3 (3)	H151—C15—H152	107.7
C7—C6—C11	118.2 (3)	C13—C16—H161	109.5
C7—C6—C15	120.9 (3)	C13—C16—H162	109.5
C11—C6—C15	120.9 (3)	H161—C16—H162	109.5
C6—C7—C8	121.3 (3)	C13—C16—H163	109.5
C6—C7—H7	119.4	H161—C16—H163	109.5
C8—C7—H7	119.4	H162—C16—H163	109.5
C9—C8—C7	119.2 (3)	C13—C17—H171	109.5
C9—C8—H8	120.4	C13—C17—H172	109.5
C7—C8—H8	120.4	H171—C17—H172	109.5
C10—C9—C8	120.6 (3)	C13—C17—H173	109.5
C10—C9—H9	119.7	H171—C17—H173	109.5
C8—C9—H9	119.7	H172—C17—H173	109.5
C9—C10—C11	119.6 (3)	C3—C18—H181	109.5
C9—C10—H10	120.2	C3—C18—H182	109.5
C11—C10—H10	120.2	H181—C18—H182	109.5
C6—C11—C10	121.1 (3)	C3—C18—H183	109.5
C6—C11—H11	119.4	H181—C18—H183	109.5
C10—C11—H11	119.4	H182—C18—H183	109.5
C1—C12—C13	114.6 (3)	C3—C19—H191	109.5
C1—C12—H121	108.6	C3—C19—H192	109.5
C13—C12—H121	108.6	H191—C19—H192	109.5
C1—C12—H122	108.6	C3—C19—H193	109.5
C13—C12—H122	108.6	H191—C19—H193	109.5
H121—C12—H122	107.6	H192—C19—H193	109.5
C14—C13—C16	108.2 (3)		

C3—N2—C1—O1	−2.9 (5)	C7—C6—C11—C10	0.8 (4)
C3—N2—C1—C12	177.5 (3)	C15—C6—C11—C10	−178.7 (3)
C1—N2—C3—C18	68.0 (4)	C9—C10—C11—C6	0.5 (5)
C1—N2—C3—C19	−57.5 (4)	O1—C1—C12—C13	99.2 (3)
C1—N2—C3—C4	−174.7 (3)	N2—C1—C12—C13	−81.3 (4)
N2—C3—C4—O4	1.3 (4)	C1—C12—C13—C14	62.9 (3)
C18—C3—C4—O4	119.7 (3)	C1—C12—C13—C16	−58.0 (4)
C19—C3—C4—O4	−117.4 (3)	C1—C12—C13—C17	−178.2 (3)
N2—C3—C4—O5	−179.1 (3)	C15—O2—C14—C13	175.2 (2)
C18—C3—C4—O5	−60.7 (3)	C16—C13—C14—O2	173.1 (2)
C19—C3—C4—O5	62.1 (3)	C17—C13—C14—O2	−67.5 (3)
C11—C6—C7—C8	−0.9 (4)	C12—C13—C14—O2	50.6 (3)
C15—C6—C7—C8	178.6 (3)	C14—O2—C15—C6	−67.5 (3)

C6—C7—C8—C9	−0.3 (5)	C7—C6—C15—O2	128.5 (3)
C7—C8—C9—C10	1.7 (5)	C11—C6—C15—O2	−52.0 (4)
C8—C9—C10—C11	−1.7 (5)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5···O1^{i}	0.86 (4)	1.81 (4)	2.622 (3)	157 (4)
N2—H2···O2	0.86 (3)	2.43 (3)	3.140 (3)	139 (3)
N2—H2···O4	0.86 (3)	2.13 (3)	2.606 (3)	115 (2)

Symmetry code: (i) *x*, *y*, *z*+1.