Crystal structure of 2-oxo-1,2-diphenylethyl diisopropylcarbamate

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The title compound, C21H25NO3, crystallized as a racemic twin in the Sohnke space group P21. In the molecular structure of the title compound, both enantiomers show a highly similar conformation with the urethane function and the benzoyl group showing an almost perpendicular arrangement [the dihedral angle is 72.46 (8)° in the S-enantiomer and 76.21 (8)° in the R-enantiomer]. In the crystal structure, molecules of both enantiomers show infinite helical arrangements parallel to the b axis formed by weak C—H · · · O hydrogen bonds between the phenyl ring of the benzoyl group and the carbamate carbonyl group. In case of the R-enantiomer, this helix is additionally stabilized by a bifurcated hydrogen bond between the carbonyl function of the benzoyl group towards both phenyl groups of the molecule.

1. Chemical context
Phenacyl and desyl compounds may act as photoremovable protecting groups (PPGs) and have been a subject of interest for many years (Givens et al., 2012; Kammari et al., 2007; Klán et al., 2013; Sheehan & Umezawa, 1973). In addition to the protection of carboxylic acids, they have also been shown to act as suitable groups for the protection and deprotection of amines (Speckmeier et al., 2018). Besides several carbamate compounds, Lange and co-workers also synthesized the title compound via a CuI-catalysed stereospecific coupling reaction using α-stannylated benzyl carbamates (Lange et al., 2008). We chose a different procedure to synthesize the title compound, according to a synthetic route that has already been reported by Speckmeier et al. (2018). Recently, we reported on the crystal structure of the highly related achiral derivative 2-oxo-2-phenylethyl diisopropylcarbamate (Martens et al., 2021).

2. Structural commentary
The carbamate functional moieties (S-enantiomer: N1A/C3A/O3A/O2A; R-enantiomer: N1B/C3B/O3B/O2B) are essen-
tially planar with the largest deviation for the respective planes being observed for C3A and C3B (in both cases 0.01 Å). The same is true for the benzoyl groups (S-enantiomer: C1A/O1A/C10A–C15A; R-enantiomer: C1B/O1B/C10B–C15B). In case of the S-enantiomer, the carbamate and the benzoyl planes subtend a dihedral angle of 77.46 (8)° whereas for the R-enantiomer an angle of 76.21 (8)° is observed (Fig. 1). These angles show a higher deviation from a perpendicular arrangement than was observed for 2-oxo-2-phenylethyl diisopropylcarbamate (Martens et al., 2021), most probably caused by the enhanced steric requirements of the phenyl substituent at C2A or C2B, respectively. All other bond lengths and angles are of expected values with C3A—

### Table 1

Hydrogen-bond geometry (Å, °).

| D—H···A  | D—H  | H···A  | D···A  | D—H···A |
|----------|------|-------|--------|---------|
| C12A—H12A···O3A' | 0.95 | 2.36  | 3.309 (2) | 174 |
| C14B—H14B···O3B'' | 0.95 | 2.58  | 3.288 (2) | 132 |
| C11B—H11B···O1B'' | 0.95 | 2.69  | 3.553 (2) | 152 |
| C21B—H21B···O1B'' | 0.95 | 2.62  | 3.522 (2) | 158 |

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

N1A [1.354 (7) Å], C3A—O2A [1.360 (7) Å], C3B—N1B [1.350 (7) Å] and C3B—O2B [1.363 (6) Å] being slightly shorter than a typical C—O or C—N single bond due to the partial double-bond character of the respective bonds in a carbamate.

3. Supramolecular features

In the crystal structure, molecules of both enantiomers show infinite helical arrangements parallel to the b axis formed by weak C—H···O hydrogen bonds (Desiraju & Steiner, 2001; Figs. 2 and 3) between the phenyl ring of the benzoyl group and the carbamate carbonyl group (S-enantiomer: C12A—H12A···O3A, R-enantiomer: C14B—H14B···O3B; Table 1). In each of the helices, only one enantiomer is present. Nevertheless, the helices do not act as mirror images because the arrangement of the molecules relative to each other is different. In the case of the R-enantiomer (Fig. 3), the supramolecular helix is additionally stabilized by a bifurcated hydrogen bond between the carbonyl function of the benzoyl group towards both phenyl groups of the molecule (C11B—H12B···O1B and C12B—H12B···O1B; Table 1).

4. Database survey

In the Cambridge Structural Database (CSD; ConQuest Version 2020.3.0; Groom et al., 2016) there is only one carbamate reported with a CH2—C(O)—Ph group attached to the carbamate oxygen atom (NIWQUI; Jiang et al., 2019). This

![Figure 1](image1.png)

**Figure 1**
Molecular structures of both enantiomers of the title compound with displacement ellipsoids drawn at the 50% probability level (R left; S right).

![Figure 2](image2.png)

**Figure 2**
Crystal structure of the S-enantiomer of the title compound showing the helical arrangement of molecules parallel to the b axis built up by C—H···O hydrogen bonds.

![Figure 3](image3.png)

**Figure 3**
Crystal structure of the R-enantiomer of the title compound showing the helical arrangement of molecules parallel to the b axis built up by C—H···O hydrogen bonds.
compound shows a diethylamino group and a p-chlorophenyl substituent instead of the disopropylamino group and the non-substituted phenyl group as in the title compound. Contrary to the title compound, the carbamate plane and the benzoyl plane are almost coplanar. The carbonyl oxygen atoms show numerous short contacts towards different C—H groups of neighbouring molecules, leading to a dense three-dimensional network. In addition, we recently reported a structure, in which there also is a CH2—C(O)—Ph group instead of the CH(Ph)—C(O)—Ph unit in the title compound (Martens et al., 2021). In this structure, a layered arrangement is realized by all three oxygen atoms acting as hydrogen-bond acceptor sites. Moreover, there is one structure reported in the literature that is identical to the title compound with the exception of one bromine substituent at the 4-position of the phenyl ring attached to the Cl=O carbonyl group (DOKMAS; Lange et al., 2008). In the latter case, the enantiopure S-enantiomer was crystallized. The supramolecular structure of this compound shows the same bifurcated hydrogen bond as is observed for the R-enantiomer of the title compound. On the other hand, the analogue of O3 is not engaged in a C—H ⋯O interaction but shows a short oxygen—bromine contact (3.139 Å). These two interactions lead to a double-strand arrangement of molecules parallel to the a axis.

5. Synthesis and crystallization
Disopropylamine (0.05 mol, 5.05 g) and one equivalent of caesium carbonate (0.05 mol, 16.55 g) were placed in a Schlenk tube and dissolved in anhydried DMSO (150 ml). The tube was sealed with a septum, and two balloons filled with CO2 were bubbled through the reaction mixture within one h while stirring. After the addition of CO2, 1.1 equivalents of 2-bromo-1,2-diphenylethan-1-one (0.055 mol, 15.13 g) dissolved in a small amount of DMSO were added in one portion. The consumption of the 2-bromo-1,2-diphenylethan-1-one was thus monitored by TLC, and after 30 min the reaction mixture was poured onto ice to quench the reaction. After extraction with dichloromethane (3 × 40 ml), the combined organic phases were washed with brine, separated and dried over Na2SO4. The solvent was removed in vacuo and the crude product was recrystallized from n-hexane/ethylacetate (4:1, v/v) to afford the title compound (16.12 g; 95%) as a colourless crystalline solid. M.p. 485 K; 1H NMR (500 MHz, CDCl3) [ppm]: δ = 7.96 (dd, 2H), 7.50–7.47 (m, 3H), 7.39–7.32 (m, 5H), 6.88 (s, 1H), 4.05 (s, 1H), 3.86 (s, 1H), 1.28 (d, 12H); 13C NMR (126 MHz, CDCl3) [ppm]: δ = 195.4 (C=O), 154.8 (NC=O), 135.2, 134.5, 133.3, 129.0, 129.0, 128.9, 128.7, 128.7 (Cphenyl), 77.7 (C benzyllic), 46.8, 45.9 [(H3C)2CH—], 21.6, 21.4 [(H3C)2CH—].

6. Refinement
Crystal data, data collection and structure refinement details are summarized in Table 2. All hydrogen atoms were placed in idealized positions (C—H = 0.95–0.98 Å) and refined using a riding model with isotropic displacement parameters calculated as \( U_{iso}(H) = 1.2(C) \) for methine and hydrogen atoms of the phenyl group or \( 1.5 \times U_{eq}(C) \) for methyl groups. The crystal studied was refined as a two-component twin with fractions of 29% vs 71%.

| Crystal data | Chemical formula |
|--------------|------------------|
| C22H35NO3 |   |
| M, | 339.42 |
| Crystal system, space group | Monoclinic, P21 |
| Temperature (K) | 133 |
| a, b, c (Å) | 15.7976 (5), 5.9184 (3), 19.5340 (8) |
| β (°) | 90.310 (2) |
| V (Å³) | 1826.33 (13) |
| Z | 4 |
| Radiation type | Mo Kα |
| μ (mm⁻¹) | 0.08 |
| Crystal size (mm) | 0.11 × 0.10 × 0.09 |

| Data collection | Absorption correction |
|-----------------|-----------------------|
| Nonius KappaCCD | Multi-scan (SADABS; Krause et al., 2015) |
| Tmin, Tmax | 0.0659, 0.746 |
| No. of measured, independent and observed [I > 2σ(I)] reflections | 18477, 8221, 7092 |
| Rint | 0.051 |
| (sin θ/λ)max (Å⁻¹) | 0.649 |

| Refined | R[F² > 2σ(F²)], wR(F²), S |
|---------|--------------------------|
| | 0.069, 0.140, 1.09 |
| No. of reflections | 8221 |
| No. of parameters | 460 |
| No. of restraints | 1 |
| H-atom treatment | H-atom parameters constrained |
| Δρmax, Δρmin (e Å⁻³) | 0.27, −0.25 |
| Absolute structure | Twinning involves inversion, so Flack parameter cannot be determined |

Table 2

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Crystal structure of 2-oxo-1,2-diphenylethyl diisopropylcarbamate

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Computing details
Data collection: COLLECT (Nonius 1998); cell refinement: DENZO (Otwinowski & Minor, 1997); data reduction: DENZO (Otwinowski & Minor, 1997); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: SHELXL (Sheldrick, 2015b); molecular graphics: ORTEP-3 for Windows (Farrugia, 2012); software used to prepare material for publication: Mercury (Macrae et al., 2020).

2-Oxo-1,2-diphenylethyl diisopropylcarbamate

Crystal data
C21H25NO3  
Mr = 339.42  
Monoclinic, P21  
\(a = 15.7976\) (5) Å  
\(b = 5.9184\) (3) Å  
\(c = 19.5340\) (8) Å  
\(\beta = 90.310\) (2°)  
\(V = 1826.33\) (13) Å³  
Z = 4

Data collection
Nonius KappaCCD diffractometer  
\(\theta = 1.7–27.5°\)  
\(\mu = 0.08\) mm⁻¹  
\(T = 133\) K  
Prism, colourless  
\(0.11 \times 0.10 \times 0.09\) mm

8221 independent reflections
7092 reflections with \(I > 2\sigma(I)\)
\(R_{int} = 0.051\)
\(\theta_{max} = 27.5°, \theta_{min} = 1.7°\)
\(h = -20\rightarrow20\)
\(k = -7\rightarrow7\)
\(l = -25\rightarrow25\)

Refinement
Refinement on \(F^2\)  
Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full  
H-atom parameters constrained
\(R[F^2 > 2\sigma(F^2)] = 0.069\)  
\(wR(F^2) = 0.140\)  
\(S = 1.09\)  
8221 reflections  
460 parameters  
1 restraint

Primary atom site location: structure-invariant direct methods  
\(\Delta/\sigma)_{max} < 0.001\)  
\(\Delta p_{max} = 0.27\) e Å⁻³  
\(\Delta p_{min} = -0.25\) e Å⁻³

Absolute structure: Twinning involves inversion, so Flack parameter cannot be determined
**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.** Refined as a two-component inversion twin.

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

|     | x           | y           | z           | Uiso*/Ueq |
|-----|-------------|-------------|-------------|-----------|
| O1A | 0.8121 (3)  | 0.6853 (7)  | −0.0240 (2) | 0.0288 (10) |
| O2A | 0.6870 (3)  | 0.4176 (7)  | 0.01863 (19)| 0.0190 (9) |
| O3A | 0.7822 (3)  | 0.2958 (8)  | 0.0975 (2)  | 0.0261 (10) |
| N1A | 0.6619 (3)  | 0.4852 (9)  | 0.1293 (2)  | 0.0199 (11) |
| C1A | 0.8205 (4)  | 0.4889 (10) | −0.0388 (3) | 0.0194 (12) |
| C2A | 0.7421 (3)  | 0.3312 (10) | −0.0333 (3) | 0.0170 (11) |
| H2A | 0.759703    | 0.172549    | −0.022792   | 0.020*     |
| C3A | 0.7164 (4)  | 0.3938 (10) | 0.0838 (3)  | 0.0201 (12) |
| C4A | 0.6799 (4)  | 0.4475 (11) | 0.2031 (3)  | 0.0224 (13) |
| H4A | 0.731816    | 0.351395    | 0.206096    | 0.027*     |
| C5A | 0.6988 (4)  | 0.6660 (12) | 0.2408 (3)  | 0.0337 (16) |
| H5A | 0.717040    | 0.631644    | 0.287699    | 0.051*     |
| H5AB| 0.647681    | 0.759697    | 0.242005    | 0.051*     |
| C6A | 0.743922    | 0.747829    | 0.217106    | 0.051*     |
| H6A | 0.6084 (4)  | 0.3161 (12) | 0.2359 (3)  | 0.0286 (14) |
| H6AA| 0.625444    | 0.268598    | 0.281976    | 0.043*     |
| H6AB| 0.595400    | 0.182491    | 0.208083    | 0.043*     |
| H6AC| 0.557999    | 0.412251    | 0.238758    | 0.043*     |
| C7A | 0.5909 (3)  | 0.6403 (10) | 0.1106 (3)  | 0.0186 (12) |
| H7A | 0.563719    | 0.685615    | 0.154630    | 0.022*     |
| C8A | 0.6230 (4)  | 0.8573 (11) | 0.0779 (3)  | 0.0267 (13) |
| H8A | 0.6644 (4)  | 0.925211    | 0.107349    | 0.040*     |
| H8AB| 0.575809    | 0.963387    | 0.072233    | 0.040*     |
| H8AC| 0.647270    | 0.822968    | 0.033024    | 0.040*     |
| C9A | 0.5215 (4)  | 0.5267 (11) | 0.0676 (3)  | 0.0218 (12) |
| H9A | 0.471118    | 0.623236    | 0.066660    | 0.033*     |
| H9AB| 0.507155    | 0.380250    | 0.087855    | 0.033*     |
| H9AC| 0.541918    | 0.504067    | 0.020820    | 0.033*     |
| C10A| 0.9019 (4)  | 0.3949 (9)  | −0.0646 (3) | 0.0172 (11) |
| C11A| 0.9726 (4)  | 0.5377 (11) | −0.0640 (3) | 0.0238 (13) |
| C11A| 0.967778    | 0.685807    | −0.045748   | 0.029*     |
| C12A| 1.0490 (4)  | 0.4657 (12) | −0.0897 (3) | 0.0303 (15) |
| H12A| 1.096641    | 0.563704    | −0.088757   | 0.036*     |
| C13A| 1.0564 (4)  | 0.2506 (13) | −0.1167 (3) | 0.0327 (16) |
| H13A| 1.108798    | 0.202773    | −0.135411   | 0.039*     |
| C14A| 0.9876 (4)  | 0.1038 (11) | −0.1168 (3) | 0.0297 (15) |
| H14A| 0.993042    | −0.044172   | −0.135173   | 0.036*     |
| C15A| 0.9104 (3)  | 0.1759 (10) | −0.0897 (3) | 0.0213 (12) |
| Atom   | x      | y      | z      | Tm (iso) | Tm (aniso) |
|--------|--------|--------|--------|----------|------------|
| H15A   | 0.863640 | 0.075140 | -0.088342 | 0.026*   |
| C16A   | 0.6918 (3) | 0.3416 (10) | -0.0996 (3) | 0.0184 (12) |
| C17A   | 0.6459 (4) | 0.5366 (11) | -0.1152 (3) | 0.0253 (13) |
| H17A   | 0.647648 | 0.661921 | -0.084779 | 0.030*   |
| C18A   | 0.5975 (4) | 0.5490 (12) | -0.1749 (3) | 0.0276 (14) |
| H18A   | 0.565708 | 0.681533 | -0.184613 | 0.033*   |
| C19A   | 0.5955 (4) | 0.3686 (12) | -0.2201 (3) | 0.0294 (15) |
| H19A   | 0.562904 | 0.377176 | -0.261009 | 0.035*   |
| C20A   | 0.6413 (4) | 0.1766 (12) | -0.2050 (3) | 0.0302 (15) |
| H20A   | 0.639871 | 0.052366 | -0.235793 | 0.036*   |
| C21A   | 0.6895 (4) | 0.1619 (11) | -0.1456 (3) | 0.0230 (13) |
| H21A   | 0.721040 | 0.028715 | -0.136229 | 0.028*   |
| O1B    | 0.6967 (3) | 0.7307 (7) | 0.4533 (2) | 0.0271 (10) |
| O2B    | 0.8103 (3) | 0.4526 (7) | 0.51374 (18) | 0.0194 (9) |
| O3B    | 0.7098 (3) | 0.3500 (8) | 0.5898 (2) | 0.0264 (10) |
| N1B    | 0.8295 (3) | 0.5399 (9) | 0.6247 (2) | 0.0196 (10) |
| C1B    | 0.6829 (4) | 0.5301 (10) | 0.4479 (3) | 0.0200 (12) |
| C2B    | 0.7551 (3) | 0.3627 (10) | 0.4613 (3) | 0.0183 (12) |
| H2B    | 0.732304 | 0.212046 | 0.475242 | 0.022*   |
| C3B    | 0.7777 (4) | 0.4423 (10) | 0.5781 (3) | 0.0189 (12) |
| C4B    | 0.8101 (4) | 0.5094 (11) | 0.6976 (3) | 0.0258 (14) |
| H4B    | 0.756939 | 0.418041 | 0.700236 | 0.031*   |
| C5B    | 0.7933 (5) | 0.7329 (14) | 0.7337 (3) | 0.0413 (19) |
| C5B    | 0.846412 | 0.817367 | 0.738191 | 0.062*   |
| C6B    | 0.770122 | 0.703504 | 0.779273 | 0.062*   |
| H5BC   | 0.752597 | 0.821747 | 0.706896 | 0.062*   |
| C6B    | 0.8799 (5) | 0.3745 (13) | 0.7337 (3) | 0.0386 (18) |
| H6BA   | 0.863578 | 0.346188 | 0.781268 | 0.058*   |
| H6BB   | 0.932837 | 0.460894 | 0.732922 | 0.058*   |
| H6BC   | 0.888072 | 0.230129 | 0.710081 | 0.058*   |
| C7B    | 0.9040 (4) | 0.6810 (10) | 0.6067 (3) | 0.0216 (12) |
| H7B    | 0.928159 | 0.734045 | 0.651270 | 0.026*   |
| C8B    | 0.8777 (4) | 0.8945 (11) | 0.5683 (3) | 0.0278 (14) |
| H8BA   | 0.833198 | 0.972502 | 0.593895 | 0.042*   |
| H8BB   | 0.856216 | 0.853703 | 0.522793 | 0.042*   |
| H8BC   | 0.926743 | 0.994531 | 0.563576 | 0.042*   |
| C9B    | 0.9745 (4) | 0.5502 (11) | 0.5720 (3) | 0.0272 (14) |
| H9BA   | 0.986965 | 0.413125 | 0.598401 | 0.041*   |
| H9BB   | 1.025404 | 0.644571 | 0.569792 | 0.041*   |
| H9BC   | 0.956746 | 0.508404 | 0.525597 | 0.041*   |
| C10B   | 0.5976 (3) | 0.4428 (10) | 0.4260 (3) | 0.0165 (11) |
| C11B   | 0.5695 (4) | 0.2237 (10) | 0.4395 (3) | 0.0206 (12) |
| H11B   | 0.606805 | 0.115534 | 0.459205 | 0.025*   |
| C12B   | 0.4860 (3) | 0.1654 (11) | 0.4238 (3) | 0.0223 (12) |
| H12B   | 0.466538 | 0.016660 | 0.433074 | 0.027*   |
| C13B   | 0.4313 (4) | 0.3211 (12) | 0.3951 (3) | 0.0260 (14) |
| H13B   | 0.374611 | 0.278911 | 0.384894 | 0.031*   |
| C14B   | 0.4588 (4) | 0.5393 (11) | 0.3809 (3) | 0.0232 (13) |
| H14B  | 0.421022 | 0.646333 | 0.361192 | 0.028* |
|-------|----------|----------|----------|--------|
| C15B  | 0.5419 (4) | 0.5997 (10) | 0.3959 (3) | 0.0221 (13) |
| H15B  | 0.561242 | 0.747893 | 0.385732 | 0.027* |
| C16B  | 0.8076 (3) | 0.3419 (10) | 0.3967 (3) | 0.0185 (11) |
| C17B  | 0.8615 (4) | 0.5147 (11) | 0.3770 (3) | 0.0220 (12) |
| H17B  | 0.866476 | 0.646580 | 0.404392 | 0.026* |
| C18B  | 0.9080 (4) | 0.4954 (11) | 0.3172 (3) | 0.0235 (13) |
| H18B  | 0.944589 | 0.614749 | 0.303810 | 0.028* |
| C19B  | 0.9015 (4) | 0.3042 (12) | 0.2771 (3) | 0.0286 (15) |
| H19B  | 0.933510 | 0.292162 | 0.236173 | 0.034* |
| C20B  | 0.8484 (4) | 0.1302 (11) | 0.2965 (3) | 0.0259 (14) |
| H20B  | 0.843657 | −0.001200 | 0.268859 | 0.031* |
| C21B  | 0.8016 (4) | 0.1475 (10) | 0.3570 (3) | 0.0217 (12) |
| H21B  | 0.765911 | 0.026904 | 0.370783 | 0.026* |

**Atomic displacement parameters (Å²)**

|    | U¹¹ | U¹² | U¹³ | U¹² | U¹³ | U¹³ |
|----|-----|-----|-----|-----|-----|-----|
| O1A | 0.026 (2) | 0.018 (2) | 0.042 (3) | −0.0023 (19) | 0.005 (2) | −0.007 (2) |
| O2A | 0.019 (2) | 0.022 (2) | 0.0166 (18) | 0.0056 (17) | 0.0047 (16) | −0.0037 (16) |
| O3A | 0.023 (2) | 0.029 (3) | 0.027 (2) | 0.0088 (19) | −0.0014 (18) | 0.0011 (18) |
| N1A | 0.017 (2) | 0.026 (3) | 0.017 (2) | 0.004 (2) | −0.0009 (18) | −0.001 (2) |
| C1A | 0.023 (3) | 0.015 (3) | 0.020 (3) | −0.001 (2) | −0.002 (2) | −0.002 (2) |
| C2A | 0.020 (3) | 0.015 (3) | 0.016 (2) | 0.005 (2) | 0.002 (2) | 0.000 (2) |
| C3A | 0.022 (3) | 0.015 (3) | 0.023 (3) | −0.003 (2) | −0.002 (2) | 0.001 (2) |
| C4A | 0.024 (3) | 0.024 (3) | 0.019 (3) | 0.006 (3) | −0.003 (2) | −0.003 (2) |
| C5A | 0.037 (4) | 0.040 (4) | 0.025 (3) | −0.008 (3) | −0.009 (3) | −0.007 (3) |
| C6A | 0.039 (4) | 0.026 (3) | 0.021 (3) | −0.005 (3) | 0.003 (3) | 0.000 (3) |
| C7A | 0.017 (3) | 0.023 (3) | 0.015 (3) | 0.006 (2) | 0.000 (2) | 0.001 (2) |
| C8A | 0.032 (3) | 0.018 (3) | 0.029 (3) | 0.002 (3) | −0.003 (3) | −0.002 (3) |
| C9A | 0.018 (3) | 0.025 (3) | 0.022 (3) | 0.003 (3) | 0.002 (2) | 0.001 (3) |
| C10A| 0.023 (3) | 0.013 (3) | 0.015 (3) | −0.001 (2) | 0.000 (2) | 0.003 (2) |
| C11A| 0.026 (3) | 0.021 (3) | 0.024 (3) | −0.004 (3) | 0.000 (2) | −0.001 (3) |
| C12A| 0.024 (3) | 0.037 (4) | 0.030 (3) | −0.009 (3) | 0.000 (3) | 0.002 (3) |
| C13A| 0.019 (3) | 0.045 (4) | 0.034 (4) | 0.008 (3) | 0.008 (3) | 0.010 (3) |
| C14A| 0.035 (3) | 0.026 (3) | 0.029 (3) | 0.010 (3) | 0.006 (3) | 0.008 (3) |
| C15A| 0.017 (3) | 0.024 (3) | 0.023 (3) | −0.002 (2) | 0.005 (2) | 0.006 (2) |
| C16A| 0.016 (3) | 0.020 (3) | 0.019 (3) | −0.007 (2) | 0.008 (2) | −0.001 (2) |
| C17A| 0.029 (3) | 0.021 (3) | 0.026 (3) | −0.001 (3) | 0.003 (3) | −0.002 (3) |
| C18A| 0.026 (3) | 0.030 (4) | 0.026 (3) | 0.005 (3) | 0.000 (3) | −0.003 (3) |
| C19A| 0.023 (3) | 0.044 (4) | 0.020 (3) | 0.000 (3) | 0.000 (2) | −0.004 (3) |
| C20A| 0.029 (3) | 0.032 (4) | 0.030 (3) | −0.006 (3) | 0.004 (3) | −0.012 (3) |
| C21A| 0.022 (3) | 0.023 (3) | 0.024 (3) | 0.004 (3) | 0.003 (2) | −0.002 (2) |
| O1B | 0.026 (2) | 0.016 (2) | 0.039 (3) | −0.0019 (18) | −0.003 (2) | 0.0013 (18) |
| O2B | 0.019 (2) | 0.022 (2) | 0.0173 (18) | −0.0015 (17) | 0.0029 (16) | 0.0019 (16) |
| O3B | 0.023 (2) | 0.029 (2) | 0.027 (2) | −0.0082 (19) | 0.0045 (17) | 0.0007 (19) |
| N1B | 0.020 (2) | 0.022 (3) | 0.017 (2) | −0.002 (2) | 0.0021 (19) | 0.000 (2) |
| C1B | 0.024 (3) | 0.018 (3) | 0.018 (3) | −0.003 (3) | 0.003 (2) | 0.002 (2) |
C2B  0.019 (3)  0.014 (3)  0.022 (3)  −0.002 (2)  −0.007 (2)  0.001 (2)
C3B  0.018 (3)  0.015 (3)  0.024 (3)  0.004 (2)  0.000 (2)  0.006 (2)
C4B  0.025 (3)  0.035 (4)  0.017 (3)  −0.009 (3)  0.004 (2)  0.001 (3)
C5B  0.047 (4)  0.047 (5)  0.030 (4)  0.004 (4)  0.015 (3)  −0.007 (3)
C6B  0.054 (5)  0.039 (5)  0.023 (3)  −0.004 (4)  0.000 (3)  0.011 (3)
C7B  0.025 (3)  0.021 (3)  0.020 (3)  −0.005 (2)  0.001 (2)  −0.003 (2)
C8B  0.034 (3)  0.021 (3)  0.028 (3)  −0.004 (3)  0.000 (3)  0.004 (3)
C9B  0.024 (3)  0.028 (4)  0.030 (3)  −0.002 (3)  0.003 (3)  −0.002 (3)
C10B 0.017 (3)  0.019 (3)  0.013 (2)  0.001 (2)  0.002 (2)  −0.003 (2)
C11B 0.019 (3)  0.021 (3)  0.022 (3)  −0.001 (2)  0.001 (2)  0.004 (2)
C12B 0.020 (3)  0.021 (3)  0.026 (3)  −0.006 (2)  0.002 (2)  −0.003 (2)
C13B 0.019 (3)  0.038 (4)  0.021 (3)  −0.004 (3)  0.002 (2)  −0.006 (3)
C14B 0.023 (3)  0.027 (3)  0.020 (3)  0.006 (3)  −0.006 (2)  0.000 (3)
C15B 0.024 (3)  0.020 (3)  0.022 (3)  0.004 (2)  0.003 (2)  −0.003 (2)
C16B 0.018 (3)  0.018 (3)  0.019 (3)  0.000 (2)  −0.004 (2)  0.003 (2)
C17B 0.023 (3)  0.021 (3)  0.022 (3)  0.003 (3)  −0.001 (2)  0.000 (2)
C18B 0.022 (3)  0.024 (3)  0.024 (3)  −0.001 (3)  0.001 (2)  0.007 (3)
C19B 0.025 (3)  0.037 (4)  0.024 (3)  0.004 (3)  0.005 (3)  0.003 (3)
C20B 0.029 (3)  0.025 (3)  0.024 (3)  0.007 (3)  −0.001 (3)  −0.004 (3)
C21B 0.020 (3)  0.019 (3)  0.026 (3)  0.000 (2)  −0.007 (2)  0.002 (2)

Geometric parameters (Å, °)

| Bond          | Distance (Å) | Angle (°)   |
|---------------|--------------|-------------|
| O1A—C1A 1.205(7) | O1B—C1B 1.212(7) |  |
| O2A—C3A 1.360(7) | O2B—C3B 1.350(7) |  |
| O2A—C2A 1.435(6) | O2B—C2B 1.469(7) |  |
| O3A—C3A 1.219(7) | O3B—C3B 1.227(7) |  |
| N1A—C3A 1.354(7) | N1B—C3B 1.350(7) |  |
| N1A—C4A 1.484(7) | N1B—C4B 1.469(7) |  |
| N1A—C7A 1.493(7) | N1B—C7B 1.486(7) |  |
| C1A—C10A 1.491(8) | C1B—C10B 1.503(8) |  |
| C1A—C2A 1.555(8) | C1B—C2B 1.533(8) |  |
| C2A—C16A 1.516(8) | C2B—C16B 1.519(8) |  |
| C2A—H2A 1.0000 | C2B—H2B 1.0000 |  |
| C4A—C6A 1.516(8) | C4B—C5B 1.524(10) |  |
| C4A—C5A 1.518(9) | C4B—C6B 1.531(9) |  |
| C4A—H4A 1.0000 | C4B—H4B 1.0000 |  |
| C5A—H5AA 0.9800 | C5B—H5BA 0.9800 |  |
| C5A—H5AB 0.9800 | C5B—H5BB 0.9800 |  |
| C5A—H5AC 0.9800 | C5B—H5BC 0.9800 |  |
| C6A—H6AA 0.9800 | C6B—H6BA 0.9800 |  |
| C6A—H6AB 0.9800 | C6B—H6BB 0.9800 |  |
| C6A—H6AC 0.9800 | C6B—H6BC 0.9800 |  |
| C7A—C8A 1.523(8) | C7B—C9B 1.519(8) |  |
| C7A—C9A 1.532(8) | C7B—C8B 1.526(8) |  |
| C7A—H7A 1.0000 | C7B—H7B 1.0000 |  |
| C8A—H8AA 0.9800 | C8B—H8BA 0.9800 |  |
| C8A—H8AB 0.9800 | C8B—H8BB 0.9800 |  |
C8A—H8AC 0.9800 C8B—H8BC 0.9800
C9A—H9AA 0.9800 C9B—H9BA 0.9800
C9A—H9AB 0.9800 C9B—H9BB 0.9800
C9A—H9AC 0.9800 C9B—H9BC 0.9800
C10A—C15A 1.392 (8) C10B—C11B 1.396 (8)
C10A—C11A 1.400 (8) C10B—C15B 1.405 (8)
C11A—C12A 1.377 (8) C11B—C12B 1.396 (8)
C11A—H11A 0.9500 C11B—H11B 0.9500
C12A—C13A 1.384 (10) C12B—C13B 1.380 (9)
C12A—H12A 0.9500 C12B—H12B 0.9500
C13A—C14A 1.391 (9) C13B—C14B 1.391 (9)
C13A—H13A 0.9500 C13B—H13B 0.9500
C14A—C15A 1.400 (8) C14B—C15B 1.391 (8)
C14A—H14A 0.9500 C14B—H14B 0.9500
C15A—H15A 0.9500 C15B—H15B 0.9500
C16A—C21A 1.392 (8) C16B—C17B 1.386 (8)
C16A—C17A 1.396 (9) C16B—C21B 1.391 (8)
C17A—C18A 1.393 (8) C17B—C18B 1.388 (8)
C17A—H17A 0.9500 C17B—H17B 0.9500
C18A—C19A 1.386 (9) C18B—C19B 1.380 (9)
C18A—H18A 0.9500 C18B—H18B 0.9500
C19A—C20A 1.378 (10) C19B—C20B 1.383 (9)
C19A—H19A 0.9500 C19B—H19B 0.9500
C20A—C21A 1.388 (9) C20B—C21B 1.400 (8)
C20A—H20A 0.9500 C20B—H20B 0.9500
C21A—H21A 0.9500 C21B—H21B 0.9500

C3A—O2A—C2A 114.8 (4) C3B—O2B—C2B 114.1 (4)
C3A—N1A—C4A 117.2 (5) C3B—N1B—C4B 118.1 (5)
C3A—N1A—C7A 124.4 (5) C3B—N1B—C7B 124.0 (4)
C4A—N1A—C7A 118.0 (4) C4B—N1B—C7B 117.9 (5)
O1A—C1A—C10A 122.5 (5) O1B—C1B—C10B 121.5 (6)
O1A—C1A—C1A 118.3 (5) O1B—C1B—C2B 118.9 (5)
C10A—C1A—C2A 119.2 (5) C10B—C1B—C2B 119.5 (5)
O2A—C2A—C16A 105.8 (4) O2B—C2B—C16B 106.8 (4)
O2A—C2A—C1A 108.7 (5) O2B—C2B—C1B 109.2 (5)
C16A—C2A—C1A 109.4 (4) C16B—C2B—C1B 108.6 (4)
O2A—C2A—H2A 110.9 O2B—C2B—H2B 110.7
C16A—C2A—H2A 110.9 C16B—C2B—H2B 110.7
C1A—C2A—H2A 110.9 C1B—C2B—H2B 110.7
O3A—C3A—N1A 126.2 (5) O3B—C3B—N1B 126.4 (5)
O3A—C3A—O2A 122.8 (5) O3B—C3B—O2B 121.8 (5)
N1A—C3A—O2A 110.9 (5) N1B—C3B—O2B 111.8 (5)
N1A—C4A—C6A 110.4 (5) N1B—C4B—C5B 112.3 (5)
N1A—C4A—C5A 112.3 (5) N1B—C4B—C6B 110.9 (5)
C6A—C4A—C5A 112.1 (5) C5B—C4B—C6B 111.4 (6)
N1A—C4A—H4A 107.2 N1B—C4B—H4B 107.3
C6A—C4A—H4A 107.2 C5B—C4B—H4B 107.3
C5A—C4A—H4A 107.2  C6B—C4B—H4B 107.3
C4A—C5A—H5AA 109.5  C4B—C5B—H5BA 109.5
C4A—C5A—H5AB 109.5  C4B—C5B—H5BB 109.5
H5AA—C5A—H5AB 109.5  H5BA—C5B—H5BB 109.5
C4A—C5A—H5AC 109.5  C4B—C5B—H5BC 109.5
H5AA—C5A—H5AC 109.5  H5BA—C5B—H5BC 109.5
C4A—C5A—H5AD 109.5  H5BB—C5B—H5BC 109.5
C4A—C6A—H6AA 109.5  C4B—C6B—H6BA 109.5
C4A—C6A—H6AB 109.5  C4B—C6B—H6BB 109.5
H6AA—C6A—H6AB 109.5  C4B—C6B—H6BC 109.5
C4A—C6A—H6AC 109.5  H6BA—C6B—H6BC 109.5
H6AB—C6A—H6AC 109.5  H6BB—C6B—H6BC 109.5
N1A—C7A—C8A 111.7 (5)  N1B—C7B—C8B 113.7 (5)
N1A—C7A—C9A 113.5 (5)  N1B—C7B—C9B 111.6 (5)
C8A—C7A—C9A 112.2 (5)  C9B—C7B—C9A 111.7 (5)
N1A—C7A—H7A 106.3  N1B—C7B—H7B 105.6
C8A—C7A—H7A 106.3  C9B—C7B—H7B 105.6
N1A—C7A—H7A 106.3  C8B—C7B—H7B 105.6
N2A—C7A—C8AA 109.5  N2B—C7B—C8BA 109.5
N2A—C7A—H8AA 109.5  N2B—C7B—H8BB 109.5
C7A—C8A—H8BB 109.5  N2B—C7B—H8BC 109.5
C7A—C8A—H8AC 109.5  H8AA—C8A—H8AC 109.5
H8AA—C8A—H8AC 109.5  C8B—C7B—H8BC 109.5
C7A—C9A—H9AA 109.5  H8AB—C8A—H8AC 109.5
C7A—C9A—H9AB 109.5  C7B—C9B—H9AA 109.5
H9AA—C9A—H9AB 109.5  C7B—C9B—H9AB 109.5
C7A—C9A—H9AC 109.5  C9B—C9B—H9AB 109.5
C7A—C9A—H9AC 109.5  C7B—C9B—H9BC 109.5
H9AA—C9A—H9AC 109.5  H9BA—C9B—H9BC 109.5
C15A—C10A—C11A 119.1 (5)  C11B—C10B—C15B 119.6 (5)
C15A—C10A—C1A 123.5 (5)  C11B—C10B—C1B 123.4 (5)
C11A—C10A—C1A 117.5 (5)  C15B—C10B—C1B 116.7 (5)
C12A—C11A—C10A 120.8 (6)  C10B—C11B—C12B 119.3 (6)
C12A—C11A—H11A 119.6  C10B—C11B—H11B 120.4
C10A—C11A—H11A 119.6  C12B—C11B—H11B 120.4
C11A—C12A—C13A 119.9 (6)  C13B—C12B—C11B 120.8 (6)
C11A—C12A—H12A 120.0  C13B—C12B—H12B 119.6
C13A—C12A—H12A 120.0  C11B—C12B—H12B 119.6
C12A—C13A—C14A 120.5 (6)  C12B—C13B—C14B 120.4 (6)
C12A—C13A—H13A 119.7  C12B—C13B—H13B 119.8
C14A—C13A—H13A 119.7  C14B—C13B—H13B 119.8
C13A—C14A—C15A 119.4 (6)  C13B—C14B—C15B 119.5 (6)
C13A—C14A—H14A 120.3  C13B—C14B—H14B 120.3
C15A—C14A—H14A 120.3  C15B—C14B—H14B 120.3
C10A—C15A—C14A 120.2 (6)  C14B—C15B—C10B 120.4 (6)
C10A—C15A—H15A 119.9  C14B—C15B—H15B 119.8
C14A—C15A—H15A 119.9  C10B—C15B—H15B 119.8
C21A—C16A—C17A 118.6 (5)  C17B—C16B—C21B 119.7 (5)
C21A—C16A—C2A  122.1 (5)  C17B—C16B—C2B  120.7 (5)
C17A—C16A—C2A  119.3 (5)  C21B—C16B—C2B  119.7 (5)
C18A—C17A—C16A 120.5 (6)  C16B—C17B—C18B 120.1 (6)
C18A—C17A—H17A 119.7  C16B—C17B—H17B 119.9
C16A—C17A—H17A 119.7  C18B—C17B—H17B 119.9
C19A—C18A—C17A 120.2 (6)  C19B—C18B—C17B 120.5 (6)
C19A—C18A—H18A 119.9  C19B—C18B—H18B 119.8
C17A—C18A—H18A 119.9  C17B—C18B—H18B 119.8
C20A—C19A—C18A 119.3 (6)  C18B—C19B—C20B 119.8 (6)
C20A—C19A—H19A 120.4  C18B—C19B—H19B 120.1
C18A—C19A—H19A 120.4  C20B—C19B—H19B 120.1
C19A—C20A—C21A 121.0 (6)  C19B—C20B—C21B 120.1 (6)
C19A—C20A—H20A 119.5  C19B—C20B—H20B 120.0
C21A—C20A—H20A 119.5  C21B—C20B—H20B 120.0
C20A—C21A—C16A 120.3 (6)  C16B—C21B—C20B 119.8 (6)
C20A—C21A—H21A 119.8  C16B—C21B—H21B 120.1
C16A—C21A—H21A 119.8  C20B—C21B—H21B 120.1

Hydrogen-bond geometry (Å, °)

| D—H···A     | D—H  | H···A  | D···A   | D—H···A |
|-------------|-------|--------|---------|---------|
| C12A···O3A  | 0.95  | 2.36   | 3.309 (2)| 174     |
| C14B···O3B  | 0.95  | 2.58   | 3.288 (2)| 132     |
| C11B···O1B  | 0.95  | 2.69   | 3.553 (2)| 152     |
| C21B···O1B  | 0.95  | 2.62   | 3.522 (2)| 158     |

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