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# Crystal structures of *N*-*tert*-butyl-3-(4-fluorophenyl)-5-oxo-4-[2-(trifluoromethoxy)phenyl]-2,5-dihydrofuran-2-carboxamide and 4-(2*H*-1,3-benzodioxol-5-yl)-*N*-cyclohexyl-5-oxo-3-[4-(trifluoromethyl)phenyl]-2,5-dihydrofuran-2-carboxamide

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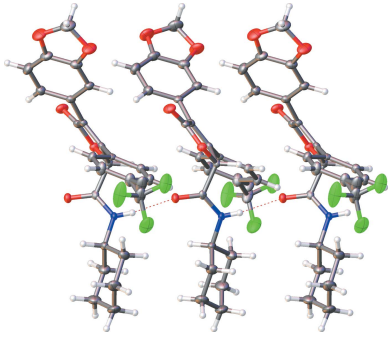
The title compounds, $C_{22}H_{19}F_4NO_4$, (I), and $C_{25}H_{22}F_3NO_5$, (II), each contain a central nearly planar dihydrofuranone ring. The r.m.s. deviation from planarity of these rings is 0.015 Å in (I) and 0.027 Å in (II). The molecules are T-shaped, with the major conformational difference being the O—C—C—O torsion angle [−178.9 (1) in (I) and 37.7 (2)° in (II)]. In the crystal of (I), molecules are linked by N—H···O hydrogen bonds, forming chains along [001] while in (II) molecules are linked by N—H···O hydrogen bonds, forming chains along [010]. In (II), the trifluoromethyl substituent is disordered over two sets of sites, with refined occupancies of 0.751 (3) and 0.249 (3).

## 1. Chemical context

Butenolides, also known as furan-2(5*H*)-ones or furanones, are a recurrent moiety in more than 13,000 natural products (De Souza, 2005) and possess different assorted biological applications, exemplified by cytotoxic (Jung *et al.*, 1990) and antibiotic (Sikorska *et al.*, 2012) activities. Likewise, the butenolide derivative Vioxx® is a potent NSAID (non-steroidal anti-inflammatory drug) used for the relief of pain and inflammation (Prasit *et al.*, 1999) before it was withdrawn from the market in 2004. As a part of our scientific endeavors to access and mimic the complexity and diversity present in naturally occurring molecular scaffolds, the title compounds were synthesized using a Passerini/Knoevenagel sequence and the crystal structures are reported herein. Other multi-component reaction-based approaches towards furanones have been reported, but they use limited starting materials. For example, they use unstable phosphonates (Beck *et al.*, 2001), aliphatic substituents (Bossio *et al.*, 1993, 1994; Marcaccini *et al.*, 2000), or tricarbonyl inputs (Rossbach *et al.*, 2014).



## 2. Structural commentary

The molecular structures of *N*-*tert*-butyl-3-(4-fluorophenyl)-5-oxo-4-[2-(trifluoromethoxy)phenyl]-2,5-dihydrofuran-2-carboxamide (I) (Fig. 1) and 4-(2*H*-1,3-benzodioxol-5-yl)-*N*-cyclohexyl-5-oxo-3-[4-(trifluoromethyl)phenyl]-2,5-dihydrofuran-2-carboxamide (II) (Fig. 2) are similar. The molecules are T-shaped, with the major conformational difference being the O1—C—C—O2 torsion angle. In (I), this torsion angle is −178.9 (1)°, whereas in (II), it is 37.7 (2)°.

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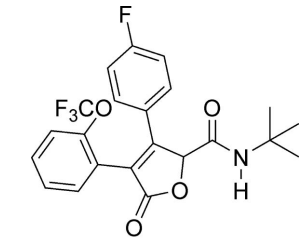
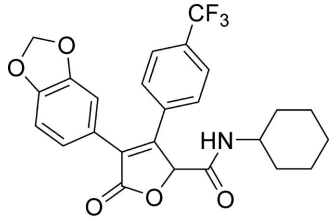



(I)




(II)

In (II), the amide oxygen atom, O1, is tucked between O2 and H1*A*, with contact distances to O2 of 2.738 (1) Å and to H1*A* of 2.54 Å. The central, dihydrofuranone ring is nearly planar in both compounds. The r.m.s. deviation of these central rings is 0.015 Å in (I), and 0.027 Å in (II). In (I), the dihedral angle between the furan ring and the *p*-fluoro substituted benzene ring is 44.66 (4)° and with the trifluoromethoxy-substituted benzene ring it is 48.71 (3)°. In (II), the dihedral angle between the furan ring and the *p*-trifluoromethyl substituted benzene ring is 40.03 (5)° and the dihedral angle with the benzene ring of the benzo[1,3]dioxol-5-yl ring system is 43.06 (6)°. The cyclohexane ring of (II) is in a chair conformation.

The –$CF_3$ substituent of (II) is disordered over two sets of sites. In the major component [occupancy = 0.751 (3)], F2*A* has a close contact to atom O1 [2.772 (2) Å] of a neighbouring molecule, which is lengthened to 3.093 (6) Å in the alternate configuration *i.e.* the minor component. Two hydrogen atoms from symmetry-equivalent molecules flank F3*B* (H2*A*, 2.72 Å, and H23, 2.57 Å) and prevent rotational disorder from alleviating the close contact. In the minor component, the –$CF_3$ group deviates from the plane of the aromatic ring, with C25*B* displaced by 0.36 (1) Å from the mean plane of the aromatic ring.

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

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···O1$^{i}$ | 0.845 (17) | 2.209 (17) | 3.0098 (14) | 158.2 (15) |

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

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

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···O1$^{i}$ | 0.821 (18) | 2.061 (18) | 2.8699 (16) | 168.4 (16) |

Symmetry code: (i) $-x+\frac{3}{2}, y-\frac{1}{2}, z$.

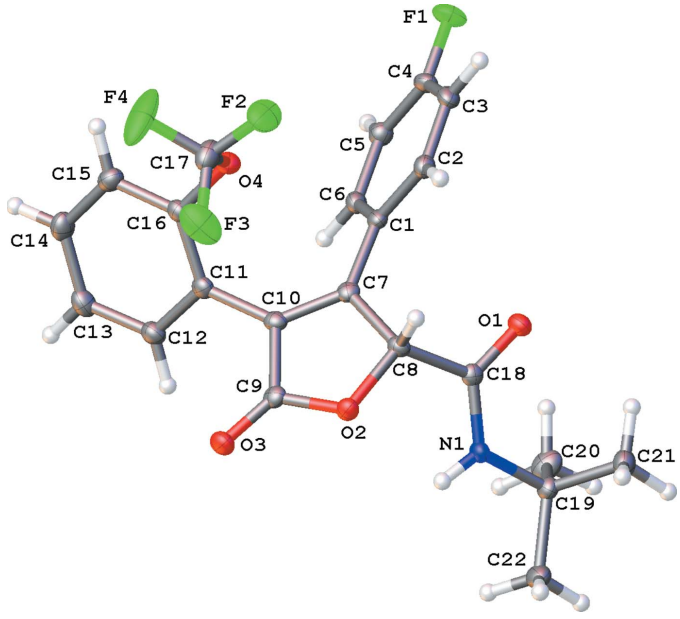



**Figure 1**
The molecular structure of (I). Anisotropically refined atoms are shown as 50% probability displacement ellipsoids.

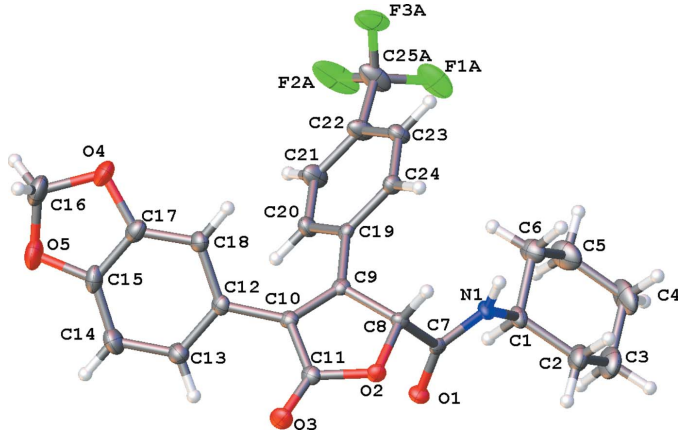



**Figure 2**
The molecular structure of (II). The –$CF_3$ substituent is disordered and only the major component is shown. Anisotropically refined atoms are shown as 50% probability displacement ellipsoids.

## 3. Supramolecular features

In both crystals, N—H···O hydrogen bonds connect the molecules into chains which run, in (I) along the *c*-axis direction (Table 1), and in (II) along the *b*-axis direction (Table 2). The hydrogen-bonding graph set is *C*(4) in both (I) and (II). The partial packing plots of (I) (Fig. 3) and (II) (Fig. 4) illustrate the hydrogen-bonding motifs.

## 4. Database survey

The Cambridge Structural Database (CSD) contains few examples of 3,4,5-substituted furan-2(5*H*)-ones. A search (CSD Version 5.36, November 2014; Groom & Allen, 2014) found only one other structure with an amide attachment at

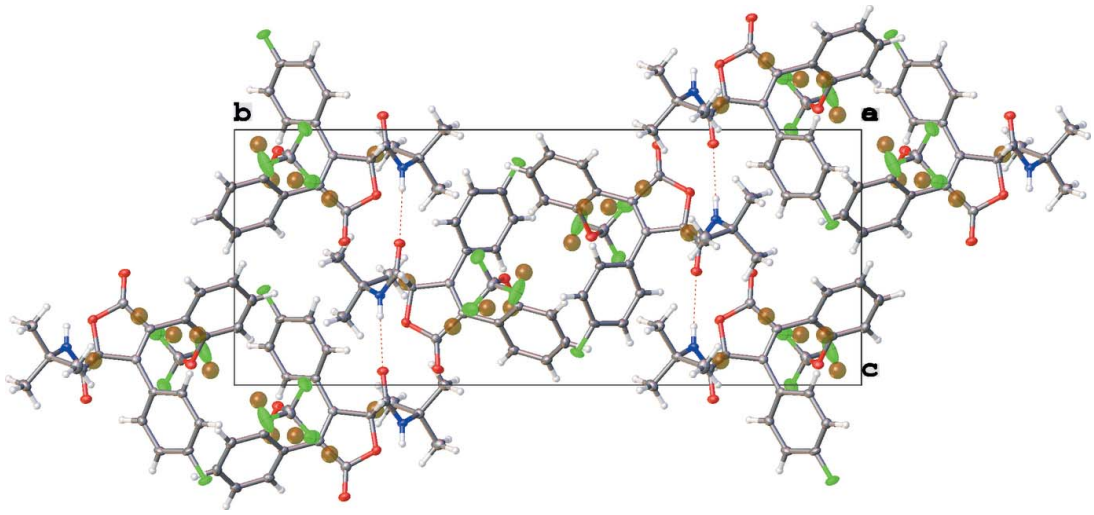



**Figure 3**
Part of the crystal structure of (I) viewed along [100]. The hydrogen bonds linking the molecules into chains along [001] are shown as dotted lines.

the position 5 carbon, TIFXIP (Beck *et. al*, 2001). For TIFXIP, the O1—C—C—O2 torsion angles for the two molecules in the asymmetric unit are −40.1 and 40.4°, similar to that found in (II), and the O1···O2 distances are 2.76 and 2.78 Å. When the search is expanded to include molecules with a second organic substituent on the furan 5-carbon, additional structures are found. In six structures, where only one of the substituents is an amide, the O1—C—C—O2 torsion angle is 180° ± 30° (−150 to 150°); the value of −178.8 (1)° found for (I) falls in this range.

## 5. Synthesis and crystallization

**Compound (I)**: 4-fluorophenylglyoxal (1 eq., 0.5 mmol), trifluoromethoxyphenylacetic acid (1 eq., 0.5 mmol) and *tert*-butyl isocyanide (1eq., 0.5 mmol) were dissolved in DCM (2 mL) and stirred at room temperature for 1 h. After confirming the exclusive formation of the Passerini product (*via* TLC and LC/MS), the solvent was removed and the crude product was dissolved in DMF (2 mL). Diisopropylamine (DIPEA) (2 eq., 1 mmol, 140 µL) was added and the reaction mixture was heated at 393 K using microwave irradiation for 20 minutes. After cooling and verifying reaction completion (TLC and LC/MS), the crude mixture was directly purified by flash chromatography (EtOAc/hexane 0–100%) using an ISCO TM flash chromatography system to afford *N*-*tert*-butyl-3-(4-fluorophenyl)-5-oxo-4-[2-(trifluoromethoxy)phenyl]-2,5-dihydrofuran-2-carboxamide as a beige solid (67% yield).

**Compound (II)**: 4-trifluoromethylphenylglyoxal (1 eq., 0.5 mmol), 3,4-methylenedioxyphenylacetic acid (1eq., 0.5 mmol) and cyclohexyl isocyanide (1eq., 0.5 mmol) were dissolved in DCM (2 mL) and stirred at room temperature for 1 h. After confirming the exclusive formation of the Passerini product (*via* TLC and LC/MS), the solvent was removed and the crude product was dissolved in DMF (2 mL). Diisopropylamine (DIPEA) (2 eq., 1 mmol, 140 µL) was added and the reaction mixture was heated at 393 K using microwave irradiation for 20 minutes. After cooling and verifying reaction completion (TLC and LC/MS), the crude mixture was directly purified by flash chromatography (EtOAc/hexane 0–100%) using an ISCO TM flash chromatography system to afford 4-(2*H*-1,3-benzodioxol-5-yl)-*N*-cyclohexyl-5-oxo-3-[4-(trifluoromethyl)phenyl]-2,5-dihydrofuran-2-carboxamide as a yellow solid (61% yield).

For both compounds, crystals suitable for X-ray structure elucidation were obtained by slow evaporation of a solution of the compound in a mixture of ethyl acetate/hexanes (1:3).

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. Hydrogen atoms were visible in the difference Fourier maps for both structures. The hydrogen

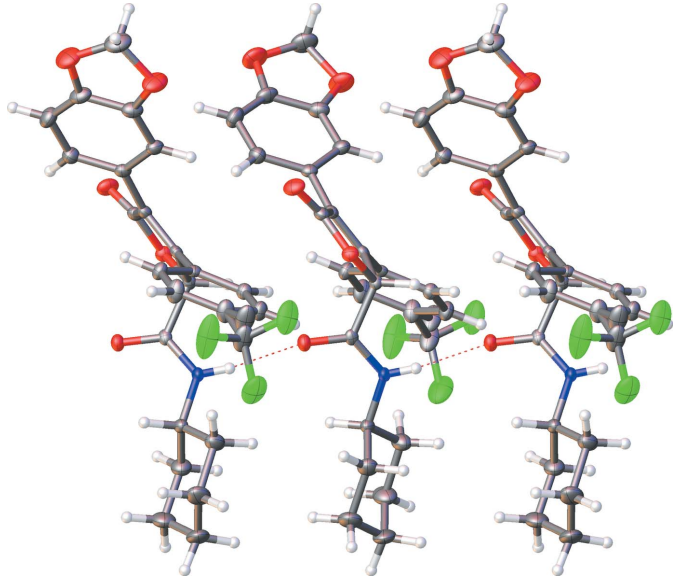


**Figure 4**
A hydrogen-bonded chain of molecules of (II) propagating along [010]. The view is along the [100] direction. Hydrogen bonds are shown as dotted lines.

**Table 3**
Experimental details.

| | (I) | (II) |
|---|---|---|
| Crystal data | | |
| Chemical formula | $C_{22}H_{19}F_4NO_4$ | $C_{25}H_{22}F_3NO_5$ |
| $M_r$ | 437.38 | 473.43 |
| Crystal system, space group | Monoclinic, $P2_1/c$ | Orthorhombic, *Pbca* |
| Temperature (K) | 100 | 100 |
| $a, b, c$ (Å) | 8.0173 (8), 24.900 (2), 10.2186 (9) | 19.2990 (7), 9.5345 (3), 24.2188 (7) |
| $\alpha, \beta, \gamma$ (°) | 90, 96.738 (2), 90 | 90, 90, 90 |
| $V$ (Å$^3$) | 2025.9 (3) | 4456.4 (2) |
| $Z$ | 4 | 8 |
| Radiation type | Mo $K\alpha$ | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.12 | 0.12 |
| Crystal size (mm) | 0.3 × 0.2 × 0.2 | 0.35 × 0.25 × 0.2 |
| Data collection | | |
| Diffractometer | Bruker APEXII CCD | Bruker APEXII CCD |
| Absorption correction | Multi-scan (*SADABS*; Bruker, 2009) | Multi-scan (*SADABS*; Bruker, 2012) |
| $T_{min}$, $T_{max}$ | 0.589, 0.746 | 0.609, 0.745 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 16716, 4139, 3698 | 36729, 3939, 3383 |
| $R_{int}$ | 0.022 | 0.033 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.625 | 0.595 |
| Refinement | | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.034, 0.084, 1.03 | 0.035, 0.086, 1.05 |
| No. of reflections | 4139 | 3939 |
| No. of parameters | 287 | 324 |
| No. of restraints | 0 | 30 |
| 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 |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.31, −0.32 | 0.27, −0.36 |

Computer programs: *APEX2* and *SAINT* (Bruker, 2009, 2012), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *OLEX2* (Dolomanov *et al.*, 2009) and *OLEX.SOLVE* (Bourhis *et al.*, 2015).

atoms bonded to nitrogen atoms which are involved in hydrogen bonding were placed at positions of the electron density peaks and freely refined. All other hydrogen atoms were placed at calculated positions and allowed to ride on their parent atoms: C—H = 0.98 Å for methyl H atoms and 0.95 Å for other H atoms, with $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms and = $1.2U_{eq}(C)$ for other atoms.

In (II), the trifluoromethyl substituent is disordered over two sets of sites with refined occupancies of 0.751 (3) and 0.249 (3). The disorder does not correspond to the expected rotational disorder of the –$CF_3$ group, but rather consists of a deviation, in the minor component, of the central carbon atom out of the plane of the aromatic ring.

## Acknowledgements

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# supporting information

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# Crystal structures of *N*-*tert*-butyl-3-(4-fluorophenyl)-5-oxo-4-[2-(trifluoromethoxy)phenyl]-2,5-dihydrofuran-2-carboxamide and 4-(2*H*-1,3-benzodioxol-5-yl)-*N*-cyclohexyl-5-oxo-3-[4-(trifluoromethyl)phenyl]-2,5-dihydrofuran-2-carboxamide

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## Computing details

Data collection: *APEX2* (Bruker, 2009) for (I); *APEX2* (Bruker, 2012) for (II). Cell refinement: *SAINT* (Bruker, 2009) for (I); *SAINT* (Bruker, 2012) for (II). Data reduction: *SAINT* (Bruker, 2009) for (I); *SAINT* (Bruker, 2012) for (II). Program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008) for (I); OLEX.*SOLVE* (Bourhis *et al.*, 2015) for (II). For both compounds, program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: OLEX2 (Dolomanov *et al.*, 2009); software used to prepare material for publication: OLEX2 (Dolomanov *et al.*, 2009).

### (I) *N*-*tert*-Butyl-3-(4-fluorophenyl)-5-oxo-4-[2-(trifluoromethoxy)phenyl]-2,5-dihydrofuran-2-carboxamide

*Crystal data*

$C_{22}H_{19}F_4NO_4$
$M_r = 437.38$
Monoclinic, $P2_1/c$
$a = 8.0173$ (8) Å
$b = 24.900$ (2) Å
$c = 10.2186$ (9) Å
$\beta = 96.738$ (2)°
$V = 2025.9$ (3) Å$^3$
$Z = 4$
$F(000) = 904$
$D_x = 1.434$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9193 reflections
$\theta = 2.6$–$28.2°$
$\mu = 0.12$ mm$^{-1}$
$T = 100$ K
Prism, clear colourless
0.3 × 0.2 × 0.2 mm

*Data collection*

Bruker APEXII CCD diffractometer
Radiation source: sealed tube
Graphite monochromator
Detector resolution: 8 pixels mm$^{-1}$
$\omega$ and $\varphi$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min} = 0.589$, $T_{max} = 0.746$
16716 measured reflections
4139 independent reflections
3698 reflections with $I > 2\sigma(I)$
$R_{int} = 0.022$
$\theta_{max} = 26.4°$, $\theta_{min} = 1.6°$
$h = -9 \rightarrow 10$
$k = -31 \rightarrow 31$
$l = -12 \rightarrow 12$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.084$
$S = 1.03$
4139 reflections
287 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0363P)^2 + 1.005P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.31$ e Å$^{-3}$
$\Delta\rho_{min} = -0.32$ e Å$^{-3}$

*Special details*

**Experimental**. Absorption correction: SADABS-2008/1 (Bruker, 2009) was used for absorption correction. wR2(int) was 0.0543 before and 0.0350 after correction. The Ratio of minimum to maximum transmission is 0.7899. The $\lambda/2$ correction factor is 0.0015.

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

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on $F^2$, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative $F^2$. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on $F^2$ are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| F1 | 0.41272 (11) | 0.45346 (3) | 0.87343 (7) | 0.0292 (2) |
| F2 | −0.18072 (11) | 0.38769 (4) | 0.50333 (9) | 0.0362 (2) |
| O1 | 0.49555 (11) | 0.26340 (4) | 0.55486 (8) | 0.01916 (19) |
| O3 | 0.17815 (11) | 0.32454 (4) | 0.06255 (8) | 0.0216 (2) |
| O4 | 0.01276 (11) | 0.43064 (4) | 0.41800 (8) | 0.0204 (2) |
| F3 | −0.16303 (13) | 0.37653 (4) | 0.29712 (9) | 0.0449 (3) |
| O2 | 0.23911 (11) | 0.27391 (3) | 0.24391 (8) | 0.01759 (19) |
| F4 | −0.25704 (12) | 0.45026 (4) | 0.36505 (14) | 0.0611 (4) |
| N1 | 0.53587 (13) | 0.23207 (4) | 0.35123 (10) | 0.0163 (2) |
| C7 | 0.27384 (14) | 0.34306 (5) | 0.39881 (12) | 0.0153 (2) |
| C3 | 0.27935 (16) | 0.38083 (5) | 0.75683 (12) | 0.0198 (3) |
| H3 | 0.2340 | 0.3693 | 0.8339 | 0.024* |
| C1 | 0.31213 (15) | 0.37069 (5) | 0.52612 (11) | 0.0153 (2) |
| C2 | 0.24707 (15) | 0.35278 (5) | 0.63949 (12) | 0.0175 (3) |
| H2 | 0.1803 | 0.3212 | 0.6360 | 0.021* |
| C5 | 0.44905 (16) | 0.44430 (5) | 0.65007 (12) | 0.0190 (3) |
| H5 | 0.5197 | 0.4750 | 0.6555 | 0.023* |
| C18 | 0.45164 (15) | 0.25822 (5) | 0.43641 (12) | 0.0151 (2) |
| C6 | 0.41299 (15) | 0.41644 (5) | 0.53279 (12) | 0.0167 (2) |
| H6 | 0.4576 | 0.4287 | 0.4560 | 0.020* |
| C9 | 0.21170 (15) | 0.32210 (5) | 0.18045 (12) | 0.0166 (2) |

| | | | | |
|---|---|---|---|---|
| C12 | 0.25614 (16) | 0.44545 (5) | 0.13706 (12) | 0.0195 (3) |
| H12 | 0.3302 | 0.4254 | 0.0898 | 0.023* |
| C11 | 0.18971 (15) | 0.42177 (5) | 0.24354 (12) | 0.0163 (2) |
| C8 | 0.28161 (15) | 0.28284 (5) | 0.38281 (11) | 0.0156 (2) |
| H8 | 0.1925 | 0.2662 | 0.4304 | 0.019* |
| C10 | 0.22884 (15) | 0.36535 (5) | 0.28033 (12) | 0.0159 (2) |
| C4 | 0.37925 (16) | 0.42599 (5) | 0.75857 (12) | 0.0196 (3) |
| C13 | 0.21580 (17) | 0.49790 (5) | 0.09903 (13) | 0.0221 (3) |
| H13 | 0.2616 | 0.5132 | 0.0260 | 0.027* |
| C16 | 0.08092 (15) | 0.45302 (5) | 0.30855 (12) | 0.0171 (2) |
| C19 | 0.70069 (15) | 0.20563 (5) | 0.38884 (12) | 0.0178 (3) |
| C21 | 0.68968 (18) | 0.16575 (6) | 0.50104 (13) | 0.0257 (3) |
| H21A | 0.6657 | 0.1851 | 0.5802 | 0.038* |
| H21B | 0.7967 | 0.1466 | 0.5194 | 0.038* |
| H21C | 0.5996 | 0.1399 | 0.4755 | 0.038* |
| C15 | 0.04164 (16) | 0.50550 (5) | 0.27394 (13) | 0.0210 (3) |
| H15 | −0.0303 | 0.5260 | 0.3222 | 0.025* |
| C20 | 0.83339 (17) | 0.24854 (6) | 0.42715 (15) | 0.0275 (3) |
| H20A | 0.8465 | 0.2711 | 0.3505 | 0.041* |
| H20B | 0.9407 | 0.2312 | 0.4576 | 0.041* |
| H20C | 0.7981 | 0.2708 | 0.4980 | 0.041* |
| C14 | 0.10909 (17) | 0.52781 (5) | 0.16737 (13) | 0.0231 (3) |
| H14 | 0.0820 | 0.5637 | 0.1413 | 0.028* |
| C17 | −0.14429 (17) | 0.41192 (6) | 0.39516 (14) | 0.0269 (3) |
| C22 | 0.74529 (18) | 0.17542 (6) | 0.26798 (13) | 0.0257 (3) |
| H22A | 0.6615 | 0.1474 | 0.2442 | 0.039* |
| H22B | 0.8564 | 0.1590 | 0.2878 | 0.039* |
| H22C | 0.7467 | 0.2005 | 0.1942 | 0.039* |
| H1 | 0.496 (2) | 0.2324 (6) | 0.2709 (17) | 0.025 (4)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| F1 | 0.0373 (5) | 0.0339 (5) | 0.0166 (4) | −0.0029 (4) | 0.0044 (3) | −0.0100 (3) |
| F2 | 0.0381 (5) | 0.0359 (5) | 0.0385 (5) | −0.0004 (4) | 0.0206 (4) | 0.0091 (4) |
| O1 | 0.0201 (4) | 0.0233 (5) | 0.0138 (4) | 0.0021 (4) | 0.0008 (3) | −0.0012 (3) |
| O3 | 0.0243 (5) | 0.0250 (5) | 0.0144 (4) | 0.0033 (4) | −0.0023 (4) | −0.0031 (4) |
| O4 | 0.0208 (5) | 0.0241 (5) | 0.0172 (4) | 0.0033 (4) | 0.0067 (3) | 0.0007 (4) |
| F3 | 0.0480 (6) | 0.0522 (6) | 0.0339 (5) | −0.0246 (5) | 0.0014 (4) | −0.0021 (4) |
| O2 | 0.0184 (4) | 0.0176 (4) | 0.0157 (4) | 0.0014 (3) | −0.0022 (3) | −0.0030 (3) |
| F4 | 0.0230 (5) | 0.0470 (6) | 0.1169 (10) | 0.0145 (4) | 0.0228 (6) | 0.0425 (6) |
| N1 | 0.0167 (5) | 0.0192 (5) | 0.0124 (5) | 0.0032 (4) | −0.0008 (4) | −0.0003 (4) |
| C7 | 0.0115 (5) | 0.0179 (6) | 0.0169 (6) | 0.0009 (4) | 0.0043 (4) | 0.0000 (5) |
| C3 | 0.0215 (6) | 0.0243 (6) | 0.0143 (6) | 0.0048 (5) | 0.0055 (5) | 0.0030 (5) |
| C1 | 0.0147 (6) | 0.0173 (6) | 0.0140 (6) | 0.0045 (5) | 0.0019 (4) | −0.0002 (4) |
| C2 | 0.0175 (6) | 0.0174 (6) | 0.0179 (6) | 0.0019 (5) | 0.0029 (5) | 0.0025 (5) |
| C5 | 0.0187 (6) | 0.0182 (6) | 0.0198 (6) | 0.0005 (5) | 0.0009 (5) | −0.0015 (5) |
| C18 | 0.0151 (6) | 0.0137 (5) | 0.0162 (6) | −0.0018 (4) | 0.0012 (4) | 0.0007 (4) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C6 | 0.0167 (6) | 0.0195 (6) | 0.0142 (6) | 0.0026 (5) | 0.0028 (5) | 0.0023 (5) |
| C9 | 0.0117 (6) | 0.0195 (6) | 0.0183 (6) | 0.0026 (4) | 0.0004 (4) | −0.0014 (5) |
| C12 | 0.0199 (6) | 0.0233 (6) | 0.0152 (6) | 0.0002 (5) | 0.0014 (5) | −0.0013 (5) |
| C11 | 0.0161 (6) | 0.0189 (6) | 0.0131 (6) | 0.0001 (5) | −0.0017 (4) | −0.0017 (4) |
| C8 | 0.0145 (6) | 0.0186 (6) | 0.0135 (6) | −0.0008 (5) | 0.0010 (4) | −0.0018 (5) |
| C10 | 0.0128 (6) | 0.0202 (6) | 0.0150 (6) | 0.0009 (5) | 0.0030 (4) | −0.0023 (5) |
| C4 | 0.0223 (6) | 0.0233 (6) | 0.0130 (6) | 0.0051 (5) | 0.0001 (5) | −0.0047 (5) |
| C13 | 0.0252 (7) | 0.0234 (7) | 0.0174 (6) | −0.0043 (5) | 0.0007 (5) | 0.0023 (5) |
| C16 | 0.0166 (6) | 0.0211 (6) | 0.0133 (6) | −0.0012 (5) | 0.0002 (5) | −0.0009 (5) |
| C19 | 0.0155 (6) | 0.0203 (6) | 0.0172 (6) | 0.0040 (5) | 0.0009 (5) | 0.0008 (5) |
| C21 | 0.0267 (7) | 0.0273 (7) | 0.0231 (7) | 0.0074 (6) | 0.0034 (5) | 0.0070 (6) |
| C15 | 0.0207 (6) | 0.0199 (6) | 0.0217 (6) | 0.0027 (5) | 0.0002 (5) | −0.0040 (5) |
| C20 | 0.0171 (6) | 0.0282 (7) | 0.0370 (8) | 0.0009 (5) | 0.0020 (6) | −0.0023 (6) |
| C14 | 0.0270 (7) | 0.0169 (6) | 0.0241 (7) | −0.0008 (5) | −0.0025 (5) | 0.0009 (5) |
| C17 | 0.0234 (7) | 0.0245 (7) | 0.0338 (8) | 0.0037 (5) | 0.0074 (6) | 0.0079 (6) |
| C22 | 0.0255 (7) | 0.0302 (7) | 0.0214 (7) | 0.0117 (6) | 0.0030 (5) | −0.0008 (5) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| F1—C4 | 1.3578 (14) | C6—H6 | 0.9500 |
| F2—C17 | 1.3217 (16) | C9—C10 | 1.4791 (17) |
| O1—C18 | 1.2271 (15) | C12—H12 | 0.9500 |
| O3—C9 | 1.2047 (15) | C12—C11 | 1.3968 (17) |
| O4—C16 | 1.4154 (14) | C12—C13 | 1.3900 (18) |
| O4—C17 | 1.3373 (17) | C11—C10 | 1.4784 (17) |
| F3—C17 | 1.3294 (18) | C11—C16 | 1.3942 (17) |
| O2—C9 | 1.3694 (15) | C8—H8 | 1.0000 |
| O2—C8 | 1.4376 (14) | C13—H13 | 0.9500 |
| F4—C17 | 1.3257 (17) | C13—C14 | 1.3836 (19) |
| N1—C18 | 1.3326 (15) | C16—C15 | 1.3804 (18) |
| N1—C19 | 1.4860 (16) | C19—C21 | 1.5272 (17) |
| N1—H1 | 0.845 (17) | C19—C20 | 1.5262 (19) |
| C7—C1 | 1.4720 (16) | C19—C22 | 1.5240 (17) |
| C7—C8 | 1.5104 (16) | C21—H21A | 0.9800 |
| C7—C10 | 1.3423 (17) | C21—H21B | 0.9800 |
| C3—H3 | 0.9500 | C21—H21C | 0.9800 |
| C3—C2 | 1.3855 (18) | C15—H15 | 0.9500 |
| C3—C4 | 1.3795 (19) | C15—C14 | 1.3875 (19) |
| C1—C2 | 1.3982 (17) | C20—H20A | 0.9800 |
| C1—C6 | 1.3940 (17) | C20—H20B | 0.9800 |
| C2—H2 | 0.9500 | C20—H20C | 0.9800 |
| C5—H5 | 0.9500 | C14—H14 | 0.9500 |
| C5—C6 | 1.3856 (17) | C22—H22A | 0.9800 |
| C5—C4 | 1.3768 (18) | C22—H22B | 0.9800 |
| C18—C8 | 1.5358 (17) | C22—H22C | 0.9800 |
| | | | |
| C17—O4—C16 | 116.25 (10) | F1—C4—C5 | 118.14 (12) |
| C9—O2—C8 | 109.78 (9) | C5—C4—C3 | 123.35 (12) |

| | | | |
|---|---|---|---|
| C18—N1—C19 | 123.44 (10) | C12—C13—H13 | 119.9 |
| C18—N1—H1 | 118.0 (11) | C14—C13—C12 | 120.14 (12) |
| C19—N1—H1 | 118.4 (11) | C14—C13—H13 | 119.9 |
| C1—C7—C8 | 123.57 (10) | C11—C16—O4 | 118.55 (11) |
| C10—C7—C1 | 127.58 (11) | C15—C16—O4 | 118.52 (11) |
| C10—C7—C8 | 108.85 (10) | C15—C16—C11 | 122.89 (11) |
| C2—C3—H3 | 120.9 | N1—C19—C21 | 110.75 (10) |
| C4—C3—H3 | 120.9 | N1—C19—C20 | 109.16 (10) |
| C4—C3—C2 | 118.12 (11) | N1—C19—C22 | 107.17 (10) |
| C2—C1—C7 | 121.58 (11) | C20—C19—C21 | 111.26 (11) |
| C6—C1—C7 | 119.01 (10) | C22—C19—C21 | 109.10 (11) |
| C6—C1—C2 | 119.40 (11) | C22—C19—C20 | 109.31 (11) |
| C3—C2—C1 | 120.41 (12) | C19—C21—H21A | 109.5 |
| C3—C2—H2 | 119.8 | C19—C21—H21B | 109.5 |
| C1—C2—H2 | 119.8 | C19—C21—H21C | 109.5 |
| C6—C5—H5 | 121.1 | H21A—C21—H21B | 109.5 |
| C4—C5—H5 | 121.1 | H21A—C21—H21C | 109.5 |
| C4—C5—C6 | 117.87 (12) | H21B—C21—H21C | 109.5 |
| O1—C18—N1 | 125.94 (11) | C16—C15—H15 | 120.6 |
| O1—C18—C8 | 116.73 (10) | C16—C15—C14 | 118.81 (12) |
| N1—C18—C8 | 117.31 (10) | C14—C15—H15 | 120.6 |
| C1—C6—H6 | 119.6 | C19—C20—H20A | 109.5 |
| C5—C6—C1 | 120.82 (11) | C19—C20—H20B | 109.5 |
| C5—C6—H6 | 119.6 | C19—C20—H20C | 109.5 |
| O3—C9—O2 | 121.49 (11) | H20A—C20—H20B | 109.5 |
| O3—C9—C10 | 130.18 (12) | H20A—C20—H20C | 109.5 |
| O2—C9—C10 | 108.33 (10) | H20B—C20—H20C | 109.5 |
| C11—C12—H12 | 119.4 | C13—C14—C15 | 120.10 (12) |
| C13—C12—H12 | 119.4 | C13—C14—H14 | 119.9 |
| C13—C12—C11 | 121.13 (12) | C15—C14—H14 | 119.9 |
| C12—C11—C10 | 120.68 (11) | F2—C17—O4 | 108.01 (12) |
| C16—C11—C12 | 116.91 (11) | F2—C17—F3 | 108.13 (12) |
| C16—C11—C10 | 122.36 (11) | F2—C17—F4 | 108.14 (12) |
| O2—C8—C7 | 104.62 (9) | F3—C17—O4 | 112.54 (11) |
| O2—C8—C18 | 112.83 (9) | F4—C17—O4 | 113.07 (12) |
| O2—C8—H8 | 108.5 | F4—C17—F3 | 106.78 (13) |
| C7—C8—C18 | 113.81 (10) | C19—C22—H22A | 109.5 |
| C7—C8—H8 | 108.5 | C19—C22—H22B | 109.5 |
| C18—C8—H8 | 108.5 | C19—C22—H22C | 109.5 |
| C7—C10—C9 | 108.34 (11) | H22A—C22—H22B | 109.5 |
| C7—C10—C11 | 130.46 (11) | H22A—C22—H22C | 109.5 |
| C11—C10—C9 | 121.11 (11) | H22B—C22—H22C | 109.5 |
| F1—C4—C3 | 118.51 (11) | | |

| | | | |
|---|---|---|---|
| O1—C18—C8—O2 | −178.85 (10) | C12—C13—C14—C15 | −0.2 (2) |
| O1—C18—C8—C7 | 62.13 (14) | C11—C12—C13—C14 | 0.44 (19) |
| O3—C9—C10—C7 | −177.73 (13) | C11—C16—C15—C14 | 1.98 (19) |
| O3—C9—C10—C11 | 5.3 (2) | C8—O2—C9—O3 | 178.20 (11) |

| | | | |
|---|---|---|---|
| O4—C16—C15—C14 | 179.50 (11) | C8—O2—C9—C10 | −2.52 (12) |
| O2—C9—C10—C7 | 3.08 (13) | C8—C7—C1—C2 | −46.08 (17) |
| O2—C9—C10—C11 | −173.85 (10) | C8—C7—C1—C6 | 135.23 (12) |
| N1—C18—C8—O2 | −0.55 (15) | C8—C7—C10—C9 | −2.32 (13) |
| N1—C18—C8—C7 | −119.57 (11) | C8—C7—C10—C11 | 174.22 (12) |
| C7—C1—C2—C3 | −177.29 (11) | C10—C7—C1—C2 | 134.48 (13) |
| C7—C1—C6—C5 | 178.60 (11) | C10—C7—C1—C6 | −44.22 (18) |
| C1—C7—C8—O2 | −178.70 (10) | C10—C7—C8—O2 | 0.83 (12) |
| C1—C7—C8—C18 | −55.10 (15) | C10—C7—C8—C18 | 124.44 (11) |
| C1—C7—C10—C9 | 177.19 (11) | C10—C11—C16—O4 | 3.38 (17) |
| C1—C7—C10—C11 | −6.3 (2) | C10—C11—C16—C15 | −179.09 (12) |
| C2—C3—C4—F1 | −179.44 (11) | C4—C3—C2—C1 | −1.13 (18) |
| C2—C3—C4—C5 | −0.42 (19) | C4—C5—C6—C1 | −1.35 (18) |
| C2—C1—C6—C5 | −0.12 (18) | C13—C12—C11—C10 | 177.90 (11) |
| C18—N1—C19—C21 | 55.13 (15) | C13—C12—C11—C16 | 0.44 (18) |
| C18—N1—C19—C20 | −67.70 (15) | C16—O4—C17—F2 | 173.52 (10) |
| C18—N1—C19—C22 | 174.03 (11) | C16—O4—C17—F3 | 54.24 (15) |
| C6—C1—C2—C3 | 1.39 (18) | C16—O4—C17—F4 | −66.86 (15) |
| C6—C5—C4—F1 | −179.33 (11) | C16—C11—C10—C7 | −48.09 (19) |
| C6—C5—C4—C3 | 1.65 (19) | C16—C11—C10—C9 | 128.08 (13) |
| C9—O2—C8—C7 | 1.13 (12) | C16—C15—C14—C13 | −1.01 (19) |
| C9—O2—C8—C18 | −123.11 (10) | C19—N1—C18—O1 | −1.11 (19) |
| C12—C11—C10—C7 | 134.59 (14) | C19—N1—C18—C8 | −179.23 (10) |
| C12—C11—C10—C9 | −49.24 (16) | C17—O4—C16—C11 | −100.23 (13) |
| C12—C11—C16—O4 | −179.20 (11) | C17—O4—C16—C15 | 82.14 (14) |
| C12—C11—C16—C15 | −1.67 (18) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···O1$^{i}$ | 0.845 (17) | 2.209 (17) | 3.0098 (14) | 158.2 (15) |

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

## (II) 4-(2*H*-1,3-Benzodioxol-5-yl)-*N*-cyclohexyl-5-oxo-3-[4-(trifluoromethyl)phenyl]-2,5-dihydrofuran-2-carboxamide

*Crystal data*

$C_{25}H_{22}F_3NO_5$
$M_r$ = 473.43
Orthorhombic, *Pbca*
*a* = 19.2990 (7) Å
*b* = 9.5345 (3) Å
*c* = 24.2188 (7) Å
*V* = 4456.4 (2) Å$^3$
*Z* = 8
*F*(000) = 1968
$D_x$ = 1.411 Mg m$^{-3}$
Mo *Kα* radiation, λ = 0.71073 Å
Cell parameters from 9996 reflections
θ = 2.5–25.6°
μ = 0.12 mm$^{-1}$
*T* = 100 K
Prism, clear colourless
0.35 × 0.25 × 0.2 mm

*Data collection*

Bruker APEXII CCD diffractometer
Radiation source: sealed tube
Graphite monochromator
Detector resolution: 8 pixels mm$^{-1}$
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2012)
$T_{min}$ = 0.609, $T_{max}$ = 0.745

36729 measured reflections
3939 independent reflections
3383 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.033
$\theta_{max}$ = 25.0°, $\theta_{min}$ = 1.7°
$h$ = −18→22
$k$ = −11→11
$l$ = −28→28

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.086
$S$ = 1.05
3939 reflections
324 parameters
30 restraints
Primary atom site location: iterative

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0329P)^2 + 2.7716P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.27 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.36 e Å$^{-3}$

*Special details*

**Experimental**. SADABS-2012/1 (Bruker,2012) was used for absorption correction. wR2(int) was 0.0555 before and 0.0466 after correction. The Ratio of minimum to maximum transmission is 0.8167. The $\lambda/2$ correction factor is 0.0015.

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

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| O1 | 0.75295 (5) | 0.55806 (10) | 0.49296 (4) | 0.0185 (2) | |
| O2 | 0.80402 (5) | 0.43446 (10) | 0.39940 (4) | 0.0172 (2) | |
| O3 | 0.81211 (6) | 0.58478 (12) | 0.32882 (4) | 0.0255 (3) | |
| O4 | 0.51011 (6) | 0.45837 (13) | 0.21882 (5) | 0.0367 (3) | |
| F3B | 0.4051 (13) | 0.082 (2) | 0.4558 (11) | 0.0458 (10) | 0.249 (3) |
| O5 | 0.52165 (7) | 0.69044 (14) | 0.19298 (5) | 0.0384 (3) | |
| N1 | 0.72446 (7) | 0.34318 (13) | 0.52641 (5) | 0.0165 (3) | |
| F1B | 0.4084 (4) | 0.2249 (7) | 0.5233 (3) | 0.0499 (6) | 0.249 (3) |
| C7 | 0.74426 (7) | 0.43101 (14) | 0.48714 (6) | 0.0140 (3) | |
| C19 | 0.61606 (7) | 0.35359 (15) | 0.41728 (5) | 0.0144 (3) | |
| C8 | 0.74979 (7) | 0.36625 (15) | 0.42917 (5) | 0.0145 (3) | |
| H8 | 0.7576 | 0.2627 | 0.4312 | 0.017* | |
| C9 | 0.68437 (7) | 0.40034 (14) | 0.39745 (6) | 0.0144 (3) | |
| C24 | 0.60782 (8) | 0.21905 (15) | 0.43879 (6) | 0.0173 (3) | |
| H24 | 0.6460 | 0.1561 | 0.4393 | 0.021* | |
| C11 | 0.77610 (8) | 0.51128 (15) | 0.35706 (6) | 0.0175 (3) | |
| C20 | 0.55997 (8) | 0.44519 (15) | 0.41735 (6) | 0.0199 (3) | |
| H20 | 0.5651 | 0.5371 | 0.4027 | 0.024* | |
| C12 | 0.65547 (8) | 0.54436 (15) | 0.31161 (6) | 0.0173 (3) | |

| | | | | | |
|---|---|---|---|---|---|
| C10 | 0.70054 (7) | 0.48558 (15) | 0.35506 (6) | 0.0156 (3) | |
| C17 | 0.56398 (8) | 0.51889 (17) | 0.24760 (6) | 0.0244 (4) | |
| C15 | 0.57085 (9) | 0.65775 (18) | 0.23231 (6) | 0.0268 (4) | |
| C23 | 0.54458 (8) | 0.17701 (16) | 0.45938 (6) | 0.0217 (3) | |
| H23 | 0.5390 | 0.0847 | 0.4734 | 0.026* | |
| C14 | 0.61954 (9) | 0.74357 (18) | 0.25555 (7) | 0.0295 (4) | |
| H14 | 0.6239 | 0.8392 | 0.2451 | 0.035* | |
| C22 | 0.48935 (8) | 0.26896 (17) | 0.45959 (7) | 0.0261 (4) | |
| C13 | 0.66266 (8) | 0.68364 (16) | 0.29549 (6) | 0.0230 (3) | |
| H13 | 0.6978 | 0.7394 | 0.3120 | 0.028* | |
| C1 | 0.69989 (8) | 0.38852 (15) | 0.58066 (6) | 0.0198 (3) | |
| H1A | 0.6828 | 0.4871 | 0.5770 | 0.024* | |
| C2 | 0.75650 (10) | 0.38768 (19) | 0.62406 (6) | 0.0313 (4) | |
| H2A | 0.7944 | 0.4515 | 0.6127 | 0.038* | |
| H2B | 0.7759 | 0.2920 | 0.6276 | 0.038* | |
| C21 | 0.49677 (8) | 0.40313 (17) | 0.43866 (7) | 0.0273 (4) | |
| H21 | 0.4586 | 0.4661 | 0.4389 | 0.033* | |
| C18 | 0.60503 (8) | 0.45830 (16) | 0.28706 (6) | 0.0209 (3) | |
| H18 | 0.5997 | 0.3627 | 0.2973 | 0.025* | |
| C6 | 0.63867 (9) | 0.29746 (18) | 0.59757 (6) | 0.0304 (4) | |
| H6A | 0.6535 | 0.1981 | 0.5992 | 0.036* | |
| H6B | 0.6016 | 0.3053 | 0.5695 | 0.036* | |
| C5 | 0.61007 (11) | 0.3418 (2) | 0.65399 (7) | 0.0414 (5) | |
| H5A | 0.5894 | 0.4366 | 0.6511 | 0.050* | |
| H5B | 0.5731 | 0.2759 | 0.6654 | 0.050* | |
| C25B | 0.4193 (8) | 0.2164 (15) | 0.4674 (5) | 0.0359 (9) | 0.249 (3) |
| C3 | 0.72705 (11) | 0.4350 (2) | 0.67971 (7) | 0.0413 (5) | |
| H3A | 0.7639 | 0.4307 | 0.7081 | 0.050* | |
| H3B | 0.7113 | 0.5336 | 0.6768 | 0.050* | |
| C4 | 0.66675 (12) | 0.3431 (2) | 0.69726 (7) | 0.0461 (5) | |
| H4A | 0.6835 | 0.2462 | 0.7034 | 0.055* | |
| H4B | 0.6476 | 0.3784 | 0.7326 | 0.055* | |
| C16 | 0.49374 (10) | 0.5578 (2) | 0.17675 (7) | 0.0384 (5) | |
| H16A | 0.4429 | 0.5649 | 0.1722 | 0.046* | |
| H16B | 0.5141 | 0.5281 | 0.1411 | 0.046* | |
| F2B | 0.3664 (3) | 0.2895 (6) | 0.4465 (4) | 0.0671 (8) | 0.249 (3) |
| C25A | 0.4240 (2) | 0.2222 (4) | 0.48835 (13) | 0.0359 (9) | 0.751 (3) |
| F1A | 0.43368 (11) | 0.1921 (2) | 0.54261 (8) | 0.0499 (6) | 0.751 (3) |
| F2A | 0.37403 (9) | 0.31660 (19) | 0.48644 (14) | 0.0671 (8) | 0.751 (3) |
| F3A | 0.4005 (4) | 0.1032 (6) | 0.4662 (3) | 0.0458 (10) | 0.751 (3) |
| H1 | 0.7263 (8) | 0.2588 (19) | 0.5199 (6) | 0.016 (4)* | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0213 (6) | 0.0130 (5) | 0.0212 (5) | −0.0007 (4) | 0.0009 (4) | −0.0008 (4) |
| O2 | 0.0135 (5) | 0.0206 (5) | 0.0175 (5) | −0.0016 (4) | 0.0016 (4) | 0.0006 (4) |
| O3 | 0.0224 (6) | 0.0336 (6) | 0.0206 (6) | −0.0079 (5) | 0.0041 (5) | 0.0053 (5) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O4 | 0.0344 (7) | 0.0465 (8) | 0.0292 (6) | −0.0087 (6) | −0.0167 (5) | 0.0072 (6) |
| F3B | 0.0203 (12) | 0.034 (2) | 0.083 (3) | −0.0092 (14) | 0.0007 (16) | 0.0106 (14) |
| O5 | 0.0378 (7) | 0.0447 (8) | 0.0327 (7) | 0.0069 (6) | −0.0141 (6) | 0.0109 (6) |
| N1 | 0.0240 (7) | 0.0106 (6) | 0.0149 (6) | 0.0013 (5) | −0.0005 (5) | −0.0006 (5) |
| F1B | 0.0493 (13) | 0.0453 (11) | 0.0550 (13) | 0.0014 (9) | 0.0319 (10) | 0.0147 (8) |
| C7 | 0.0101 (7) | 0.0137 (7) | 0.0182 (7) | 0.0018 (6) | −0.0030 (6) | −0.0005 (6) |
| C19 | 0.0149 (7) | 0.0175 (7) | 0.0108 (6) | −0.0009 (6) | −0.0018 (6) | −0.0013 (5) |
| C8 | 0.0132 (7) | 0.0139 (7) | 0.0164 (7) | −0.0011 (6) | 0.0023 (6) | 0.0003 (6) |
| C9 | 0.0165 (7) | 0.0127 (7) | 0.0140 (7) | 0.0001 (6) | −0.0005 (6) | −0.0041 (6) |
| C24 | 0.0149 (7) | 0.0178 (7) | 0.0191 (7) | 0.0017 (6) | 0.0001 (6) | 0.0002 (6) |
| C11 | 0.0205 (8) | 0.0181 (7) | 0.0137 (7) | −0.0008 (6) | 0.0021 (6) | −0.0024 (6) |
| C20 | 0.0198 (8) | 0.0172 (7) | 0.0226 (8) | 0.0012 (6) | 0.0005 (6) | 0.0042 (6) |
| C12 | 0.0185 (8) | 0.0209 (8) | 0.0126 (7) | 0.0003 (6) | 0.0023 (6) | −0.0001 (6) |
| C10 | 0.0185 (8) | 0.0151 (7) | 0.0133 (7) | −0.0013 (6) | 0.0011 (6) | −0.0028 (6) |
| C17 | 0.0214 (8) | 0.0334 (9) | 0.0185 (7) | −0.0024 (7) | −0.0018 (6) | −0.0002 (7) |
| C15 | 0.0261 (9) | 0.0344 (9) | 0.0201 (8) | 0.0080 (7) | −0.0027 (7) | 0.0059 (7) |
| C23 | 0.0209 (8) | 0.0170 (8) | 0.0273 (8) | −0.0013 (6) | 0.0031 (7) | 0.0030 (6) |
| C14 | 0.0353 (10) | 0.0230 (8) | 0.0303 (9) | 0.0041 (7) | −0.0007 (8) | 0.0073 (7) |
| C22 | 0.0163 (8) | 0.0231 (8) | 0.0388 (9) | −0.0007 (7) | 0.0068 (7) | 0.0018 (7) |
| C13 | 0.0271 (9) | 0.0214 (8) | 0.0204 (8) | −0.0020 (7) | −0.0011 (7) | −0.0003 (6) |
| C1 | 0.0297 (9) | 0.0151 (7) | 0.0144 (7) | 0.0025 (6) | 0.0009 (6) | 0.0002 (6) |
| C2 | 0.0414 (11) | 0.0311 (9) | 0.0215 (8) | 0.0004 (8) | −0.0080 (8) | 0.0002 (7) |
| C21 | 0.0175 (8) | 0.0232 (8) | 0.0412 (10) | 0.0052 (7) | 0.0038 (7) | 0.0036 (7) |
| C18 | 0.0241 (9) | 0.0228 (8) | 0.0157 (7) | −0.0019 (7) | −0.0003 (6) | 0.0022 (6) |
| C6 | 0.0400 (10) | 0.0301 (9) | 0.0211 (8) | −0.0063 (8) | 0.0055 (7) | −0.0023 (7) |
| C5 | 0.0561 (13) | 0.0432 (11) | 0.0248 (9) | −0.0085 (10) | 0.0160 (9) | −0.0013 (8) |
| C25B | 0.0282 (13) | 0.0283 (11) | 0.051 (3) | 0.0034 (9) | 0.023 (2) | 0.008 (2) |
| C3 | 0.0624 (14) | 0.0431 (11) | 0.0183 (8) | 0.0009 (10) | −0.0093 (8) | −0.0028 (8) |
| C4 | 0.0841 (16) | 0.0385 (11) | 0.0156 (8) | 0.0002 (11) | 0.0074 (9) | 0.0039 (8) |
| C16 | 0.0328 (10) | 0.0533 (12) | 0.0290 (9) | 0.0027 (9) | −0.0123 (8) | 0.0072 (9) |
| F2B | 0.0218 (7) | 0.0383 (9) | 0.141 (2) | 0.0111 (7) | 0.0384 (13) | 0.0238 (13) |
| C25A | 0.0282 (13) | 0.0283 (11) | 0.051 (3) | 0.0034 (9) | 0.023 (2) | 0.008 (2) |
| F1A | 0.0493 (13) | 0.0453 (11) | 0.0550 (13) | 0.0014 (9) | 0.0319 (10) | 0.0147 (8) |
| F2A | 0.0218 (7) | 0.0383 (9) | 0.141 (2) | 0.0111 (7) | 0.0384 (13) | 0.0238 (13) |
| F3A | 0.0203 (12) | 0.034 (2) | 0.083 (3) | −0.0092 (14) | 0.0007 (16) | 0.0106 (14) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C7 | 1.2310 (16) | C23—C22 | 1.380 (2) |
| O2—C8 | 1.4276 (17) | C14—H14 | 0.9500 |
| O2—C11 | 1.3704 (17) | C14—C13 | 1.398 (2) |
| O3—C11 | 1.2008 (18) | C22—C21 | 1.383 (2) |
| O4—C17 | 1.3784 (19) | C22—C25B | 1.455 (16) |
| O4—C16 | 1.427 (2) | C22—C25A | 1.507 (4) |
| F3B—C25B | 1.342 (17) | C13—H13 | 0.9500 |
| O5—C15 | 1.3807 (19) | C1—H1A | 1.0000 |
| O5—C16 | 1.429 (2) | C1—C2 | 1.516 (2) |
| N1—C7 | 1.3234 (18) | C1—C6 | 1.522 (2) |

| | | | |
|---|---|---|---|
| N1—C1 | 1.4624 (18) | C2—H2A | 0.9900 |
| N1—H1 | 0.821 (18) | C2—H2B | 0.9900 |
| F1B—C25B | 1.371 (11) | C2—C3 | 1.531 (2) |
| C7—C8 | 1.5375 (19) | C21—H21 | 0.9500 |
| C19—C9 | 1.472 (2) | C18—H18 | 0.9500 |
| C19—C24 | 1.394 (2) | C6—H6A | 0.9900 |
| C19—C20 | 1.391 (2) | C6—H6B | 0.9900 |
| C8—H8 | 1.0000 | C6—C5 | 1.533 (2) |
| C8—C9 | 1.5131 (19) | C5—H5A | 0.9900 |
| C9—C10 | 1.346 (2) | C5—H5B | 0.9900 |
| C24—H24 | 0.9500 | C5—C4 | 1.515 (3) |
| C24—C23 | 1.378 (2) | C25B—F2B | 1.336 (14) |
| C11—C10 | 1.480 (2) | C3—H3A | 0.9900 |
| C20—H20 | 0.9500 | C3—H3B | 0.9900 |
| C20—C21 | 1.384 (2) | C3—C4 | 1.518 (3) |
| C12—C10 | 1.476 (2) | C4—H4A | 0.9900 |
| C12—C13 | 1.391 (2) | C4—H4B | 0.9900 |
| C12—C18 | 1.405 (2) | C16—H16A | 0.9900 |
| C17—C15 | 1.381 (2) | C16—H16B | 0.9900 |
| C17—C18 | 1.369 (2) | C25A—F1A | 1.358 (4) |
| C15—C14 | 1.367 (2) | C25A—F2A | 1.321 (4) |
| C23—H23 | 0.9500 | C25A—F3A | 1.335 (6) |

| | | | |
|---|---|---|---|
| C11—O2—C8 | 109.46 (11) | N1—C1—C6 | 108.95 (12) |
| C17—O4—C16 | 104.46 (13) | C2—C1—H1A | 107.7 |
| C15—O5—C16 | 104.43 (13) | C2—C1—C6 | 111.71 (13) |
| C7—N1—C1 | 123.52 (12) | C6—C1—H1A | 107.7 |
| C7—N1—H1 | 118.0 (11) | C1—C2—H2A | 109.7 |
| C1—N1—H1 | 118.5 (11) | C1—C2—H2B | 109.7 |
| O1—C7—N1 | 125.44 (13) | C1—C2—C3 | 109.95 (15) |
| O1—C7—C8 | 119.35 (12) | H2A—C2—H2B | 108.2 |
| N1—C7—C8 | 114.96 (12) | C3—C2—H2A | 109.7 |
| C24—C19—C9 | 120.19 (13) | C3—C2—H2B | 109.7 |
| C20—C19—C9 | 120.48 (13) | C20—C21—H21 | 120.1 |
| C20—C19—C24 | 119.25 (13) | C22—C21—C20 | 119.73 (14) |
| O2—C8—C7 | 109.22 (11) | C22—C21—H21 | 120.1 |
| O2—C8—H8 | 111.3 | C12—C18—H18 | 121.6 |
| O2—C8—C9 | 104.91 (10) | C17—C18—C12 | 116.73 (14) |
| C7—C8—H8 | 111.3 | C17—C18—H18 | 121.6 |
| C9—C8—C7 | 108.63 (11) | C1—C6—H6A | 109.4 |
| C9—C8—H8 | 111.3 | C1—C6—H6B | 109.4 |
| C19—C9—C8 | 121.10 (12) | C1—C6—C5 | 111.21 (14) |
| C10—C9—C19 | 129.72 (13) | H6A—C6—H6B | 108.0 |
| C10—C9—C8 | 108.88 (12) | C5—C6—H6A | 109.4 |
| C19—C24—H24 | 119.9 | C5—C6—H6B | 109.4 |
| C23—C24—C19 | 120.27 (14) | C6—C5—H5A | 109.4 |
| C23—C24—H24 | 119.9 | C6—C5—H5B | 109.4 |
| O2—C11—C10 | 108.87 (12) | H5A—C5—H5B | 108.0 |

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|---|---|---|---|
| O3—C11—O2 | 120.71 (13) | C4—C5—C6 | 111.02 (17) |
| O3—C11—C10 | 130.42 (14) | C4—C5—H5A | 109.4 |
| C19—C20—H20 | 119.9 | C4—C5—H5B | 109.4 |
| C21—C20—C19 | 120.30 (14) | F3B—C25B—F1B | 103.5 (14) |
| C21—C20—H20 | 119.9 | F3B—C25B—C22 | 119.5 (14) |
| C13—C12—C10 | 120.26 (13) | F1B—C25B—C22 | 104.5 (9) |
| C13—C12—C18 | 120.51 (14) | F2B—C25B—F3B | 105.2 (16) |
| C18—C12—C10 | 119.23 (13) | F2B—C25B—F1B | 103.1 (10) |
| C9—C10—C11 | 107.66 (13) | F2B—C25B—C22 | 118.8 (11) |
| C9—C10—C12 | 129.54 (14) | C2—C3—H3A | 109.4 |
| C12—C10—C11 | 122.78 (13) | C2—C3—H3B | 109.4 |
| O4—C17—C15 | 109.76 (14) | H3A—C3—H3B | 108.0 |
| C18—C17—O4 | 127.80 (15) | C4—C3—C2 | 111.16 (15) |
| C18—C17—C15 | 122.41 (15) | C4—C3—H3A | 109.4 |
| O5—C15—C17 | 109.59 (15) | C4—C3—H3B | 109.4 |
| C14—C15—O5 | 128.44 (15) | C5—C4—C3 | 111.39 (15) |
| C14—C15—C17 | 121.96 (15) | C5—C4—H4A | 109.4 |
| C24—C23—H23 | 120.0 | C5—C4—H4B | 109.4 |
| C24—C23—C22 | 120.04 (14) | C3—C4—H4A | 109.4 |
| C22—C23—H23 | 120.0 | C3—C4—H4B | 109.4 |
| C15—C14—H14 | 121.6 | H4A—C4—H4B | 108.0 |
| C15—C14—C13 | 116.70 (15) | O4—C16—O5 | 107.95 (13) |
| C13—C14—H14 | 121.6 | O4—C16—H16A | 110.1 |
| C23—C22—C21 | 120.41 (15) | O4—C16—H16B | 110.1 |
| C23—C22—C25B | 120.0 (6) | O5—C16—H16A | 110.1 |
| C23—C22—C25A | 117.36 (19) | O5—C16—H16B | 110.1 |
| C21—C22—C25B | 117.6 (5) | H16A—C16—H16B | 108.4 |
| C21—C22—C25A | 121.96 (19) | F1A—C25A—C22 | 113.3 (3) |
| C12—C13—C14 | 121.67 (15) | F2A—C25A—C22 | 113.2 (3) |
| C12—C13—H13 | 119.2 | F2A—C25A—F1A | 106.1 (3) |
| C14—C13—H13 | 119.2 | F2A—C25A—F3A | 108.4 (5) |
| N1—C1—H1A | 107.7 | F3A—C25A—C22 | 110.5 (4) |
| N1—C1—C2 | 112.81 (13) | F3A—C25A—F1A | 104.8 (4) |

| | | | |
|---|---|---|---|
| O1—C7—C8—O2 | 37.65 (17) | C10—C12—C18—C17 | 179.02 (13) |
| O1—C7—C8—C9 | −76.25 (16) | C17—O4—C16—O5 | −19.09 (18) |
| O2—C8—C9—C19 | −176.88 (12) | C17—C15—C14—C13 | 0.5 (2) |
| O2—C8—C9—C10 | −2.55 (15) | C15—O5—C16—O4 | 18.95 (18) |
| O2—C11—C10—C9 | 2.98 (15) | C15—C17—C18—C12 | −0.3 (2) |
| O2—C11—C10—C12 | −175.42 (12) | C15—C14—C13—C12 | −1.3 (2) |
| O3—C11—C10—C9 | −176.73 (15) | C23—C22—C21—C20 | 0.0 (3) |
| O3—C11—C10—C12 | 4.9 (2) | C23—C22—C25B—F3B | −22.7 (17) |
| O4—C17—C15—O5 | −0.23 (19) | C23—C22—C25B—F1B | 92.3 (9) |
| O4—C17—C15—C14 | 178.50 (15) | C23—C22—C25B—F2B | −153.5 (8) |
| O4—C17—C18—C12 | −178.10 (15) | C23—C22—C25A—F1A | 59.2 (3) |
| O5—C15—C14—C13 | 178.93 (16) | C23—C22—C25A—F2A | −179.9 (2) |
| N1—C7—C8—O2 | −147.74 (12) | C23—C22—C25A—F3A | −58.1 (5) |
| N1—C7—C8—C9 | 98.36 (14) | C13—C12—C10—C9 | 137.71 (16) |

| | | | |
|---|---|---|---|
| N1—C1—C2—C3 | −179.57 (14) | C13—C12—C10—C11 | −44.3 (2) |
| N1—C1—C6—C5 | −179.20 (14) | C13—C12—C18—C17 | −0.6 (2) |
| C7—N1—C1—C2 | −95.91 (17) | C1—N1—C7—O1 | 9.6 (2) |
| C7—N1—C1—C6 | 139.43 (14) | C1—N1—C7—C8 | −164.62 (13) |
| C7—C8—C9—C19 | −60.19 (16) | C1—C2—C3—C4 | 56.9 (2) |
| C7—C8—C9—C10 | 114.14 (13) | C1—C6—C5—C4 | −54.2 (2) |
| C19—C9—C10—C11 | 173.54 (14) | C2—C1—C6—C5 | 55.50 (19) |
| C19—C9—C10—C12 | −8.2 (2) | C2—C3—C4—C5 | −56.8 (2) |
| C19—C24—C23—C22 | −1.1 (2) | C21—C22—C25B—F3B | 141.0 (15) |
| C19—C20—C21—C22 | −0.5 (2) | C21—C22—C25B—F1B | −103.9 (8) |
| C8—O2—C11—O3 | 175.07 (13) | C21—C22—C25B—F2B | 10.3 (12) |
| C8—O2—C11—C10 | −4.67 (14) | C21—C22—C25A—F1A | −114.8 (3) |
| C8—C9—C10—C11 | −0.15 (15) | C21—C22—C25A—F2A | 6.1 (4) |
| C8—C9—C10—C12 | 178.09 (13) | C21—C22—C25A—F3A | 127.9 (4) |
| C9—C19—C24—C23 | 177.42 (13) | C18—C12—C10—C9 | −41.9 (2) |
| C9—C19—C20—C21 | −176.65 (14) | C18—C12—C10—C11 | 136.14 (15) |
| C24—C19—C9—C8 | −41.76 (19) | C18—C12—C13—C14 | 1.4 (2) |
| C24—C19—C9—C10 | 145.21 (15) | C18—C17—C15—O5 | −178.43 (15) |
| C24—C19—C20—C21 | 0.1 (2) | C18—C17—C15—C14 | 0.3 (3) |
| C24—C23—C22—C21 | 0.7 (3) | C6—C1—C2—C3 | −56.43 (18) |
| C24—C23—C22—C25B | 164.0 (5) | C6—C5—C4—C3 | 55.1 (2) |
| C24—C23—C22—C25A | −173.4 (2) | C25B—C22—C21—C20 | −163.6 (5) |
| C11—O2—C8—C7 | −111.87 (12) | C16—O4—C17—C15 | 11.93 (18) |
| C11—O2—C8—C9 | 4.42 (14) | C16—O4—C17—C18 | −170.00 (17) |
| C20—C19—C9—C8 | 134.97 (14) | C16—O5—C15—C17 | −11.56 (18) |
| C20—C19—C9—C10 | −38.1 (2) | C16—O5—C15—C14 | 169.81 (18) |
| C20—C19—C24—C23 | 0.6 (2) | C25A—C22—C21—C20 | 173.8 (2) |
| C10—C12—C13—C14 | −178.21 (14) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···O1$^{i}$ | 0.821 (18) | 2.061 (18) | 2.8699 (16) | 168.4 (16) |

Symmetry code: (i) −*x*+3/2, *y*−1/2, *z*.