# organic compounds

# Methyl 3-(4-{6-methyl-4-[3-(trifluoromethyl)phenyl]pyridazin-3-yloxy}-phenyl)propanoate

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In the title compound, $C_{22}H_{19}F_3N_2O_3$, the benzene rings of the trifluoromethylphenyl and benzoylphenyl groups form dihedral angles of 41.89 (10) and 67.44 (10)°, respectively, with the pyridazine ring. The methylpropanoate group is nearly coplanar with the attached benzene ring [dihedral angle = 3.9 (2)°]. The trifluoromethyl group is disordered over two positions; the site-occupancy factors are *ca* 0.64 and 0.36. In the crystal structure, inversion-related molecules are linked through C—H···O hydrogen bonds and C—H···$\pi$ interactions.

## Related literature

For the biological activities of pyridazine derivatives, see: Heinisch & Kopelent (1992); Kolar & Tisler (1999).
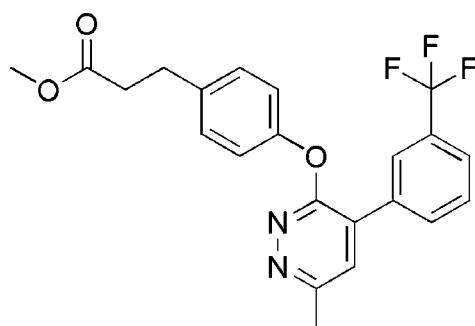


# Experimental

### Crystal data

| | |
|---|---|
| $C_{22}H_{19}F_3N_2O_3$ | $\gamma = 78.92\ (3)°$ |
| $M_r = 416.39$ | $V = 950.6\ (4)$ Å³ |
| Triclinic, $P\bar{1}$ | $Z = 2$ |
| $a = 9.4916\ (19)$ Å | Mo $K\alpha$ radiation |
| $b = 10.232\ (2)$ Å | $\mu = 0.12$ mm$^{-1}$ |
| $c = 11.022\ (2)$ Å | $T = 113\ (2)$ K |
| $\alpha = 81.70\ (3)°$ | $0.20 \times 0.16 \times 0.06$ mm |
| $\beta = 65.12\ (3)°$ | |

### Data collection

| | |
|---|---|
| Rigaku Saturn CCD area-detector diffractometer | 5529 measured reflections |
| | 3319 independent reflections |
| Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005) | 1862 reflections with $I > 2\sigma(I)$ |
| | $R_{int} = 0.054$ |
| $T_{min} = 0.977$, $T_{max} = 0.993$ | |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)] = 0.055$ | 72 restraints |
| $wR(F^2) = 0.147$ | H-atom parameters constrained |
| $S = 1.06$ | $\Delta\rho_{max} = 0.40$ e Å$^{-3}$ |
| 3319 reflections | $\Delta\rho_{min} = -0.39$ e Å$^{-3}$ |
| 301 parameters | |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C11—H11$B$···O2$^i$ | 0.96 | 2.57 | 3.467 (4) | 156 |
| C22—H22$C$···$Cg$1$^{ii}$ | 0.96 | 2.73 | 3.486 (4) | 136 |

Symmetry codes: (i) $-x, -y, -z+1$; (ii) $-x, -y+1, -z+1$. $Cg$1 is the centroid of the C2–C7 ring.

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

## References

Heinisch, G. & Kopelent, H. (1992). *Prog. Med. Chem.* **29**, 141–183.
Kolar, P. & Tisler, M. (1999). *Adv. Heterocycl. Chem.* **75**, 167–241.
Rigaku/MSC (2005). *CrystalClear*. Rigaku/MSC, The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

# supporting information

## Methyl 3-(4-{6-methyl-4-[3-(trifluoromethyl)phenyl]pyridazin-3-yloxy}phenyl)-propanoate

### Yu-Hong Xiao, You-Quan Zhu, Xiao-Mao Zou, Fang-Zhong Hu and Hua-Zheng Yang

### S1. Comment

Many pyridazine derivatives have been found to exhibit biological activities such as insecticidal, fungicidal, herbicidal, plant-growth regulatory activity, *etc* (Heinisch & Kopelent, 1992). For example, pyridate, credazine and maleic hydrazide (Kolar & Tisler, 1999) have been commercialized as herbicides. In order to discover new biologically active pyridazine compounds, the title compound was synthesized and its structure is reported here.

In the title molecule (Fig. 1), the C2—C7 and C13—C18 benzene rings form dihedral angles of 41.89 (10)° and 67.44 (10)°, respectively, with the central pyridazine ring (C2—C4/C12/N1/N2). The methylpropanoate group is nearly coplanar with the C13—C18 benzene ring (dihedral angle 3.9 (2)°).

The crystal packing is stabilized by C—H···O hydrogen bonds and C—H···$\pi$ interactions (Table 1) involving the C2—C7 ring (centroid *Cg*1).

### S2. Experimental

A mixture of methyl 3-(4-hydroxyphenyl)propanoate (2.3 mmol), 3-chloro-4- (3-(trifluoromethyl)phenyl)-6-methyl-pyridazine (2.3 mmol) and $K_2CO_3$ (5 mmol) was stirred at 313 K for 2 h. The crude product was recrystallized from ethanol and single crystals of the title compound suitable for X-ray analysis were grown from ethyl acetate-petroleum ether (3:1 *v/v*) at room temperature.

### S3. Refinement

The trifluoromethyl group is disordered over two orienatations (C1/F1/F2/F3 and C1/F1′/F2′/F3′) with refined occupancies of 0.636 (12) and 0.364 (12). All C—F distances were restrained to 1.34 (1) Å and the $U^{ij}$ components of disordered F atoms were restrained to be approximately isotropic. All H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and included in the final cycles of refinement using a riding model, with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}$(methyl C).
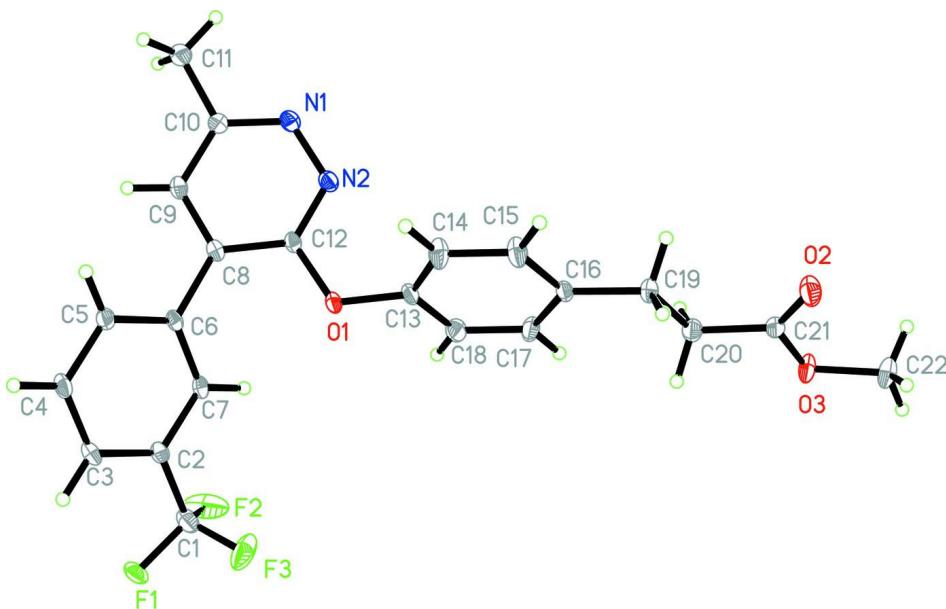
**Figure 1**

The molecular structure of the title compound, showing 30% probability displacement ellipsoids. Only one disorder component is shown.

**Methyl 3-(4-{6-methyl-4-[3-(trifluoromethyl)phenyl]pyridazin-3- yloxy}phenyl)propanoate**

*Crystal data*
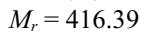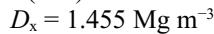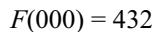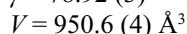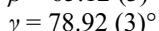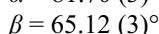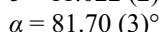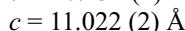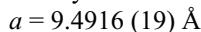
| | |
|---|---|
| $C_{22}H_{19}F_3N_2O_3$ | $Z = 2$ |
| $M_r = 416.39$ | $F(000) = 432$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.455$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $a = 9.4916$ (19) Å | Cell parameters from 2351 reflections |
| $b = 10.232$ (2) Å | $\theta = 2.0–27.9°$ |
| $c = 11.022$ (2) Å | $\mu = 0.12$ mm$^{-1}$ |
| $\alpha = 81.70$ (3)° | $T = 113$ K |
| $\beta = 65.12$ (3)° | Plate, colourless |
| $\gamma = 78.92$ (3)° | $0.20 \times 0.16 \times 0.06$ mm |
| $V = 950.6$ (4) Å$^3$ | |
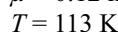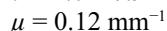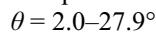
*Data collection*

| | |
|---|---|
| Rigaku Saturn CCD area-detector diffractometer | 5529 measured reflections |
| | 3319 independent reflections |
| Radiation source: rotating anode | 1862 reflections with $I > 2\sigma(I)$ |
| Confocal monochromator | $R_{int} = 0.054$ |
| Detector resolution: 7.31 pixels mm$^{-1}$ | $\theta_{max} = 25.0°$, $\theta_{min} = 2.0°$ |
| $\omega$ and $\varphi$ scans | $h = -10\rightarrow11$ |
| Absorption correction: multi-scan | $k = -12\rightarrow11$ |
| (*CrystalClear*; Rigaku/MSC, 2005) | $l = -13\rightarrow11$ |
| $T_{min} = 0.977$, $T_{max} = 0.993$ | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier |
| Least-squares matrix: full | map |
| $R[F^2 > 2\sigma(F^2)] = 0.055$ | Hydrogen site location: inferred from |
| $wR(F^2) = 0.147$ | neighbouring sites |
| $S = 1.06$ | H-atom parameters constrained |
| 3319 reflections | $w = 1/[\sigma^2(F_o^2) + (0.0675P)^2]$ |
| 301 parameters | where $P = (F_o^2 + 2F_c^2)/3$ |
| 72 restraints | $(\Delta/\sigma)_{max} = 0.003$ |
| Primary atom site location: structure-invariant | $\Delta\rho_{max} = 0.40$ e Å$^{-3}$ |
| direct methods | $\Delta\rho_{min} = -0.39$ e Å$^{-3}$ |

*Special details*

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

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

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| F1 | 1.0340 (5) | 0.3434 (7) | 0.0132 (7) | 0.0359 (15) | 0.636 (12) |
| F2 | 0.8747 (11) | 0.3445 (7) | −0.0736 (7) | 0.072 (2) | 0.636 (12) |
| F3 | 0.7991 (6) | 0.4176 (6) | 0.1223 (8) | 0.079 (3) | 0.636 (12) |
| F1′ | 1.0297 (11) | 0.3418 (14) | −0.0418 (14) | 0.071 (4) | 0.364 (12) |
| F2′ | 0.7900 (12) | 0.3936 (8) | −0.0042 (15) | 0.055 (3) | 0.364 (12) |
| F3′ | 0.8553 (19) | 0.4059 (12) | 0.1421 (11) | 0.076 (4) | 0.364 (12) |
| O1 | 0.39298 (18) | 0.1498 (2) | 0.2418 (2) | 0.0286 (6) | |
| O2 | −0.5057 (2) | 0.5985 (2) | 0.6250 (2) | 0.0409 (7) | |
| O3 | −0.4466 (2) | 0.7531 (2) | 0.4543 (2) | 0.0330 (6) | |
| N1 | 0.3114 (2) | −0.1545 (3) | 0.1899 (3) | 0.0246 (7) | |
| N2 | 0.2934 (2) | −0.0267 (3) | 0.2179 (3) | 0.0251 (7) | |
| C1 | 0.8892 (3) | 0.3235 (3) | 0.0444 (3) | 0.0327 (9) | |
| C2 | 0.8618 (3) | 0.1868 (3) | 0.1025 (3) | 0.0228 (8) | |
| C3 | 0.9650 (3) | 0.1072 (3) | 0.1502 (3) | 0.0258 (8) | |
| H3 | 1.0518 | 0.1399 | 0.1464 | 0.031* | |
| C4 | 0.9393 (3) | −0.0202 (3) | 0.2033 (3) | 0.0266 (8) | |
| H4 | 1.0083 | −0.0738 | 0.2361 | 0.032* | |
| C5 | 0.8108 (3) | −0.0688 (3) | 0.2079 (3) | 0.0241 (8) | |
| H5 | 0.7947 | −0.1555 | 0.2430 | 0.029* | |
| C6 | 0.7056 (2) | 0.0103 (3) | 0.1608 (3) | 0.0194 (7) | |
| C7 | 0.7313 (3) | 0.1389 (3) | 0.1080 (3) | 0.0205 (7) | |
| H7 | 0.6616 | 0.1931 | 0.0763 | 0.025* | |
| C8 | 0.5697 (3) | −0.0462 (3) | 0.1662 (3) | 0.0191 (7) | |
| C9 | 0.5840 (3) | −0.1748 (3) | 0.1379 (3) | 0.0227 (7) | |
| H9 | 0.6818 | −0.2278 | 0.1092 | 0.027* | |

| | | | |
|---|---|---|---|
| C10 | 0.4511 (3) | −0.2271 (3) | 0.1519 (3) | 0.0216 (7) |
| C11 | 0.4609 (3) | −0.3680 (3) | 0.1257 (3) | 0.0319 (9) |
| H11A | 0.3575 | −0.3868 | 0.1466 | 0.048* |
| H11B | 0.5055 | −0.4269 | 0.1806 | 0.048* |
| H11C | 0.5259 | −0.3811 | 0.0329 | 0.048* |
| C12 | 0.4159 (3) | 0.0228 (3) | 0.2087 (3) | 0.0202 (7) |
| C13 | 0.2387 (3) | 0.2146 (3) | 0.3058 (3) | 0.0260 (8) |
| C14 | 0.1408 (3) | 0.1712 (3) | 0.4317 (3) | 0.0353 (10) |
| H14 | 0.1721 | 0.0931 | 0.4743 | 0.042* |
| C15 | −0.0044 (3) | 0.2448 (3) | 0.4941 (3) | 0.0340 (9) |
| H15 | −0.0710 | 0.2150 | 0.5792 | 0.041* |
| C16 | −0.0543 (3) | 0.3619 (3) | 0.4340 (3) | 0.0215 (7) |
| C17 | 0.0473 (3) | 0.4015 (3) | 0.3070 (3) | 0.0311 (9) |
| H17 | 0.0168 | 0.4794 | 0.2636 | 0.037* |
| C18 | 0.1928 (3) | 0.3285 (3) | 0.2428 (3) | 0.0324 (9) |
| H18 | 0.2593 | 0.3568 | 0.1570 | 0.039* |
| C19 | −0.2110 (3) | 0.4412 (3) | 0.5096 (3) | 0.0250 (8) |
| H19A | −0.2905 | 0.3833 | 0.5370 | 0.030* |
| H19B | −0.2102 | 0.4673 | 0.5905 | 0.030* |
| C20 | −0.2594 (3) | 0.5645 (3) | 0.4362 (3) | 0.0287 (8) |
| H20A | −0.1815 | 0.6240 | 0.4094 | 0.034* |
| H20B | −0.2615 | 0.5396 | 0.3554 | 0.034* |
| C21 | −0.4164 (3) | 0.6373 (3) | 0.5179 (3) | 0.0246 (8) |
| C22 | −0.5929 (3) | 0.8355 (3) | 0.5257 (4) | 0.0392 (9) |
| H22A | −0.6786 | 0.7956 | 0.5298 | 0.059* |
| H22B | −0.5942 | 0.9226 | 0.4798 | 0.059* |
| H22C | −0.6034 | 0.8431 | 0.6150 | 0.059* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| F1 | 0.0233 (17) | 0.036 (2) | 0.049 (3) | −0.0158 (15) | −0.0157 (19) | 0.011 (3) |
| F2 | 0.123 (5) | 0.063 (4) | 0.071 (4) | −0.058 (3) | −0.075 (4) | 0.042 (3) |
| F3 | 0.041 (3) | 0.028 (3) | 0.102 (5) | 0.003 (2) | 0.027 (3) | 0.006 (3) |
| F1′ | 0.052 (4) | 0.035 (4) | 0.069 (7) | −0.004 (3) | 0.025 (4) | 0.012 (5) |
| F2′ | 0.057 (4) | 0.035 (4) | 0.093 (7) | −0.009 (3) | −0.056 (4) | 0.019 (4) |
| F3′ | 0.129 (8) | 0.040 (5) | 0.051 (5) | −0.008 (6) | −0.026 (6) | −0.014 (4) |
| O1 | 0.0136 (9) | 0.0279 (13) | 0.0422 (15) | 0.0010 (8) | −0.0076 (9) | −0.0135 (11) |
| O2 | 0.0284 (11) | 0.0378 (15) | 0.0377 (16) | 0.0032 (10) | −0.0012 (10) | 0.0053 (12) |
| O3 | 0.0283 (10) | 0.0297 (13) | 0.0279 (14) | 0.0109 (9) | −0.0045 (9) | −0.0039 (11) |
| N1 | 0.0212 (11) | 0.0294 (15) | 0.0253 (16) | −0.0046 (11) | −0.0108 (10) | −0.0031 (12) |
| N2 | 0.0198 (11) | 0.0284 (16) | 0.0286 (16) | −0.0037 (10) | −0.0104 (11) | −0.0046 (12) |
| C1 | 0.0232 (15) | 0.038 (2) | 0.037 (2) | −0.0089 (15) | −0.0131 (15) | 0.0076 (19) |
| C2 | 0.0160 (12) | 0.0280 (18) | 0.0207 (18) | −0.0029 (11) | −0.0039 (11) | −0.0019 (14) |
| C3 | 0.0154 (12) | 0.0360 (19) | 0.0261 (19) | −0.0052 (12) | −0.0083 (12) | −0.0008 (15) |
| C4 | 0.0179 (13) | 0.0323 (19) | 0.030 (2) | −0.0012 (12) | −0.0133 (12) | 0.0042 (15) |
| C5 | 0.0179 (12) | 0.0241 (17) | 0.0269 (19) | −0.0018 (11) | −0.0070 (12) | 0.0003 (14) |
| C6 | 0.0124 (12) | 0.0271 (17) | 0.0154 (16) | −0.0008 (11) | −0.0022 (11) | −0.0053 (13) |

| C7 | 0.0146 (12) | 0.0280 (18) | 0.0161 (17) | 0.0013 (11) | −0.0050 (11) | −0.0031 (13) |
|---|---|---|---|---|---|---|
| C8 | 0.0159 (12) | 0.0283 (18) | 0.0124 (17) | −0.0022 (12) | −0.0054 (11) | −0.0016 (14) |
| C9 | 0.0180 (12) | 0.0278 (18) | 0.0191 (18) | 0.0007 (12) | −0.0059 (12) | −0.0024 (14) |
| C10 | 0.0226 (13) | 0.0246 (17) | 0.0169 (17) | −0.0042 (12) | −0.0069 (12) | −0.0013 (13) |
| C11 | 0.0278 (14) | 0.0296 (19) | 0.035 (2) | −0.0048 (13) | −0.0091 (14) | −0.0047 (16) |
| C12 | 0.0156 (12) | 0.0246 (17) | 0.0194 (18) | 0.0002 (12) | −0.0059 (11) | −0.0059 (14) |
| C13 | 0.0140 (12) | 0.0303 (18) | 0.034 (2) | −0.0019 (12) | −0.0080 (12) | −0.0097 (15) |
| C14 | 0.0269 (15) | 0.040 (2) | 0.033 (2) | 0.0083 (14) | −0.0122 (14) | 0.0010 (17) |
| C15 | 0.0232 (14) | 0.041 (2) | 0.026 (2) | 0.0040 (13) | −0.0049 (13) | 0.0035 (16) |
| C16 | 0.0151 (12) | 0.0253 (17) | 0.0235 (18) | −0.0003 (11) | −0.0064 (12) | −0.0079 (14) |
| C17 | 0.0251 (14) | 0.0235 (18) | 0.031 (2) | 0.0039 (13) | −0.0021 (13) | 0.0000 (15) |
| C18 | 0.0244 (14) | 0.0294 (19) | 0.031 (2) | −0.0042 (13) | 0.0008 (13) | 0.0002 (16) |
| C19 | 0.0210 (13) | 0.0270 (18) | 0.0226 (18) | 0.0019 (12) | −0.0057 (12) | −0.0051 (14) |
| C20 | 0.0255 (14) | 0.0269 (18) | 0.029 (2) | −0.0005 (12) | −0.0073 (13) | −0.0027 (15) |
| C21 | 0.0216 (13) | 0.0245 (18) | 0.027 (2) | −0.0018 (12) | −0.0091 (13) | −0.0046 (15) |
| C22 | 0.0351 (16) | 0.035 (2) | 0.039 (2) | 0.0139 (14) | −0.0126 (15) | −0.0098 (17) |

## Geometric parameters (Å, º)

| | | | |
|---|---|---|---|
| F1—C1 | 1.319 (5) | C8—C12 | 1.405 (3) |
| F2—C1 | 1.347 (5) | C9—C10 | 1.405 (4) |
| F3—C1 | 1.304 (7) | C9—H9 | 0.93 |
| F1′—C1 | 1.302 (8) | C10—C11 | 1.489 (4) |
| F2′—C1 | 1.320 (6) | C11—H11A | 0.96 |
| F3′—C1 | 1.357 (9) | C11—H11B | 0.96 |
| O1—C12 | 1.353 (4) | C11—H11C | 0.96 |
| O1—C13 | 1.405 (3) | C13—C18 | 1.363 (4) |
| O2—C21 | 1.192 (3) | C13—C14 | 1.372 (4) |
| O3—C21 | 1.333 (3) | C14—C15 | 1.375 (4) |
| O3—C22 | 1.442 (3) | C14—H14 | 0.93 |
| N1—C10 | 1.316 (3) | C15—C16 | 1.384 (4) |
| N1—N2 | 1.348 (4) | C15—H15 | 0.93 |
| N2—C12 | 1.317 (3) | C16—C17 | 1.380 (4) |
| C1—C2 | 1.475 (4) | C16—C19 | 1.502 (4) |
| C2—C3 | 1.379 (3) | C17—C18 | 1.379 (4) |
| C2—C7 | 1.393 (4) | C17—H17 | 0.93 |
| C3—C4 | 1.372 (4) | C18—H18 | 0.93 |
| C3—H3 | 0.93 | C19—C20 | 1.501 (4) |
| C4—C5 | 1.382 (4) | C19—H19A | 0.97 |
| C4—H4 | 0.93 | C19—H19B | 0.97 |
| C5—C6 | 1.388 (3) | C20—C21 | 1.491 (4) |
| C5—H5 | 0.93 | C20—H20A | 0.97 |
| C6—C7 | 1.382 (4) | C20—H20B | 0.97 |
| C6—C8 | 1.488 (4) | C22—H22A | 0.96 |
| C7—H7 | 0.93 | C22—H22B | 0.96 |
| C8—C9 | 1.364 (4) | C22—H22C | 0.96 |
| | | | |
| C12—O1—C13 | 119.0 (2) | C10—C11—H11A | 109.5 |

| | | | |
|---|---|---|---|
| C21—O3—C22 | 116.2 (2) | C10—C11—H11B | 109.5 |
| C10—N1—N2 | 119.7 (2) | H11A—C11—H11B | 109.5 |
| C12—N2—N1 | 119.3 (2) | C10—C11—H11C | 109.5 |
| F1′—C1—F3 | 119.9 (8) | H11A—C11—H11C | 109.5 |
| F3—C1—F1 | 105.4 (5) | H11B—C11—H11C | 109.5 |
| F1′—C1—F2′ | 106.9 (7) | N2—C12—O1 | 117.9 (2) |
| F3—C1—F2′ | 69.2 (6) | N2—C12—C8 | 124.8 (3) |
| F1—C1—F2′ | 126.6 (5) | O1—C12—C8 | 117.2 (2) |
| F1′—C1—F2 | 76.7 (7) | C18—C13—C14 | 120.7 (3) |
| F3—C1—F2 | 109.6 (4) | C18—C13—O1 | 117.6 (2) |
| F1—C1—F2 | 103.9 (4) | C14—C13—O1 | 121.6 (2) |
| F1′—C1—F3′ | 104.6 (9) | C13—C14—C15 | 119.1 (3) |
| F1—C1—F3′ | 82.7 (7) | C13—C14—H14 | 120.5 |
| F2′—C1—F3′ | 95.6 (7) | C15—C14—H14 | 120.5 |
| F2—C1—F3′ | 132.0 (6) | C14—C15—C16 | 122.0 (3) |
| F1′—C1—C2 | 118.4 (6) | C14—C15—H15 | 119.0 |
| F3—C1—C2 | 114.6 (4) | C16—C15—H15 | 119.0 |
| F1—C1—C2 | 112.6 (4) | C17—C16—C15 | 117.1 (3) |
| F2′—C1—C2 | 117.7 (4) | C17—C16—C19 | 123.3 (2) |
| F2—C1—C2 | 110.1 (4) | C15—C16—C19 | 119.5 (2) |
| F3′—C1—C2 | 110.7 (6) | C18—C17—C16 | 121.6 (3) |
| C3—C2—C7 | 120.5 (3) | C18—C17—H17 | 119.2 |
| C3—C2—C1 | 120.2 (2) | C16—C17—H17 | 119.2 |
| C7—C2—C1 | 119.4 (2) | C13—C18—C17 | 119.6 (3) |
| C4—C3—C2 | 119.8 (2) | C13—C18—H18 | 120.2 |
| C4—C3—H3 | 120.1 | C17—C18—H18 | 120.2 |
| C2—C3—H3 | 120.1 | C20—C19—C16 | 116.4 (2) |
| C3—C4—C5 | 120.0 (2) | C20—C19—H19A | 108.2 |
| C3—C4—H4 | 120.0 | C16—C19—H19A | 108.2 |
| C5—C4—H4 | 120.0 | C20—C19—H19B | 108.2 |
| C4—C5—C6 | 120.8 (3) | C16—C19—H19B | 108.2 |
| C4—C5—H5 | 119.6 | H19A—C19—H19B | 107.3 |
| C6—C5—H5 | 119.6 | C21—C20—C19 | 113.1 (2) |
| C7—C6—C5 | 119.1 (2) | C21—C20—H20A | 109.0 |
| C7—C6—C8 | 121.7 (2) | C19—C20—H20A | 109.0 |
| C5—C6—C8 | 119.2 (2) | C21—C20—H20B | 109.0 |
| C6—C7—C2 | 119.8 (2) | C19—C20—H20B | 109.0 |
| C6—C7—H7 | 120.1 | H20A—C20—H20B | 107.8 |
| C2—C7—H7 | 120.1 | O2—C21—O3 | 123.5 (3) |
| C9—C8—C12 | 114.3 (2) | O2—C21—C20 | 125.7 (2) |
| C9—C8—C6 | 121.7 (2) | O3—C21—C20 | 110.9 (2) |
| C12—C8—C6 | 123.9 (3) | O3—C22—H22A | 109.5 |
| C8—C9—C10 | 120.0 (2) | O3—C22—H22B | 109.5 |
| C8—C9—H9 | 120.0 | H22A—C22—H22B | 109.5 |
| C10—C9—H9 | 120.0 | O3—C22—H22C | 109.5 |
| N1—C10—C9 | 121.7 (3) | H22A—C22—H22C | 109.5 |
| N1—C10—C11 | 116.6 (2) | H22B—C22—H22C | 109.5 |
| C9—C10—C11 | 121.7 (2) | | |

| | | | |
|---|---|---|---|
| C10—N1—N2—C12 | −1.1 (4) | N2—N1—C10—C11 | 178.8 (3) |
| F1′—C1—C2—C3 | 46.6 (11) | C8—C9—C10—N1 | 1.2 (4) |
| F3—C1—C2—C3 | −104.0 (6) | C8—C9—C10—C11 | −178.3 (3) |
| F1—C1—C2—C3 | 16.5 (6) | N1—N2—C12—O1 | −177.8 (2) |
| F2′—C1—C2—C3 | 177.6 (8) | N1—N2—C12—C8 | 2.6 (4) |
| F2—C1—C2—C3 | 131.9 (6) | C13—O1—C12—N2 | 11.0 (4) |
| F3′—C1—C2—C3 | −74.0 (8) | C13—O1—C12—C8 | −169.4 (2) |
| F1′—C1—C2—C7 | −133.2 (10) | C9—C8—C12—N2 | −2.1 (4) |
| F3—C1—C2—C7 | 76.3 (6) | C6—C8—C12—N2 | −178.0 (3) |
| F1—C1—C2—C7 | −163.2 (5) | C9—C8—C12—O1 | 178.3 (3) |
| F2′—C1—C2—C7 | −2.2 (9) | C6—C8—C12—O1 | 2.5 (4) |
| F2—C1—C2—C7 | −47.8 (6) | C12—O1—C13—C18 | −119.9 (3) |
| F3′—C1—C2—C7 | 106.2 (8) | C12—O1—C13—C14 | 64.9 (4) |
| C7—C2—C3—C4 | 0.2 (5) | C18—C13—C14—C15 | −0.4 (5) |
| C1—C2—C3—C4 | −179.6 (3) | O1—C13—C14—C15 | 174.6 (3) |
| C2—C3—C4—C5 | 0.5 (5) | C13—C14—C15—C16 | −0.4 (5) |
| C3—C4—C5—C6 | −0.8 (5) | C14—C15—C16—C17 | 0.9 (5) |
| C4—C5—C6—C7 | 0.5 (5) | C14—C15—C16—C19 | −177.2 (3) |
| C4—C5—C6—C8 | 179.4 (3) | C15—C16—C17—C18 | −0.5 (5) |
| C5—C6—C7—C2 | 0.2 (5) | C19—C16—C17—C18 | 177.4 (3) |
| C8—C6—C7—C2 | −178.7 (3) | C14—C13—C18—C17 | 0.7 (5) |
| C3—C2—C7—C6 | −0.5 (5) | O1—C13—C18—C17 | −174.5 (3) |
| C1—C2—C7—C6 | 179.3 (3) | C16—C17—C18—C13 | −0.3 (5) |
| C7—C6—C8—C9 | 139.4 (3) | C17—C16—C19—C20 | 4.1 (5) |
| C5—C6—C8—C9 | −39.5 (4) | C15—C16—C19—C20 | −178.0 (3) |
| C7—C6—C8—C12 | −45.0 (4) | C16—C19—C20—C21 | 179.8 (3) |
| C5—C6—C8—C12 | 136.1 (3) | C22—O3—C21—O2 | 3.2 (5) |
| C12—C8—C9—C10 | 0.1 (4) | C22—O3—C21—C20 | −177.6 (3) |
| C6—C8—C9—C10 | 176.1 (2) | C19—C20—C21—O2 | −7.7 (5) |
| N2—N1—C10—C9 | −0.8 (4) | C19—C20—C21—O3 | 173.1 (3) |

*Hydrogen-bond geometry (Å, º)*

| D—H···A | D—H | H···A | D···A | D—H···A |
|---|---|---|---|---|
| C11—H11*B*···O2[i] | 0.96 | 2.57 | 3.467 (4) | 156 |
| C22—H22*C*···*Cg*1[ii] | 0.96 | 2.73 | 3.486 (4) | 136 |

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