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# *tert*-Butyl *N*-benzyl-*N*-[4-(4-fluorobenzoylmethyl)-2-pyridyl]carbamate

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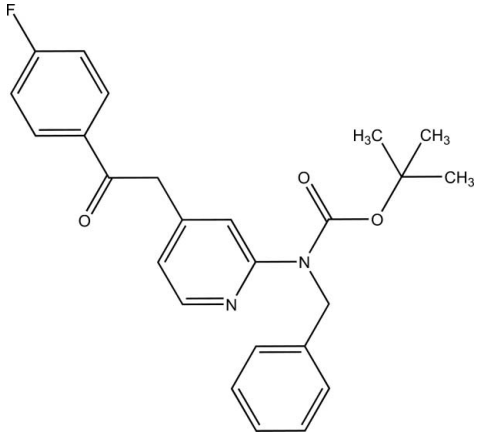
Key indicators: single-crystal X-ray study; $T$ = 193 K; mean $\sigma$(C–C) = 0.009 Å; $R$ factor = 0.110; $wR$ factor = 0.339; data-to-parameter ratio = 14.5.

In the crystal structure of the title compound, $C_{25}H_{25}FN_2O_3$, the pyridine ring makes dihedral angles of 75.1 (3), 39.4 (3) and 74.6 (3)° with the phenyl ring, the carbamate plane and the 4-fluorophenyl ring, respectively. The phenyl ring makes dihedral angles of 77.2 (3) and 23.6 (3)° with the carbamate plane and the 4-fluorophenyl ring, respectively. The 4-fluorophenyl ring is perpendicular to the carbamate plane, the dihedral angle between them being 89.5 (3)°.

## Related literature

For preparation of the title compound, see: Koch *et al.* (2008*a*). For applications of the vicinal 4-fluorophenyl/pyridin-4-yl pharmacophore in p38 MAP kinase inhibitors, see, for example: Koch *et al.* (2008*a*); for thiazolopyridines, see: Miwatashi *et al.* (2005); for pyrazolopyridines, see: Stevens *et al.* (2005). For a related structure, see: Koch, *et al.* (2008*b*).




## Experimental

### Crystal data

$C_{25}H_{25}FN_2O_3$
$M_r$ = 420.47
Monoclinic, $C2/c$
$a$ = 38.054 (7) Å
$b$ = 7.9320 (6) Å
$c$ = 14.589 (3) Å
$\beta$ = 102.142 (8)°
$V$ = 4305.1 (11) Å$^3$
$Z$ = 8
Cu $K\alpha$ radiation
$\mu$ = 0.75 mm$^{-1}$
$T$ = 193 (2) K
0.35 × 0.30 × 0.18 mm

### Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: none
4272 measured reflections
4091 independent reflections
2192 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.076
3 standard reflections
frequency: 60 min
intensity decay: 3%

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.110
$wR(F^2)$ = 0.339
$S$ = 1.10
4091 reflections
283 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.48 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.64 e Å$^{-3}$

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *CORINC* (Dräger & Gattow, 1971); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2118).

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# supplementary materials

*Acta Cryst.* (2008). E**64**, o2221 [ **doi:10.1107/S160053680803448X** ]

# *tert*-Butyl *N*-benzyl-*N*-[4-(4-fluorobenzoylmethyl)-2-pyridyl]carbamate

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## Comment

The title compound, (**I**), was obtained as an intermediate in the synthesis of 2-alkylsulfanyl-5-(2-aminopyridin-4-yl)-4-(4-fluorophenyl)imidazoles as potent p38 MAP kinase inhibitors (Koch *et al.* 2008*a*).

The vicinal 4-fluorophenyl/pyridin-4-yl system is a pharmacophore in different p38 MAP kinase inhibitors, like the imidazolopyridines (Koch *et al.* 2008*a*,*b*) and related pyridine compounds (Miwatashi *et al.* 2005), (Stevens *et al.* 2005).

In the crystal structure of the title compound **I**, Fig. 1, the pyridine ring makes dihedral angles of 75.1 (3)°, 39.4 (3)° and 74.6 (3)° to the phenyl ring (C1–C6), the carbamate plane and the 4-fluorophenyl ring (C18–C23), respectively. The phenyl ring (C1–C6) makes dihedral angles of 77.2 (3)° and 23.6 (3)° to the carbamate plane and the 4-fluorophenyl ring (C18–C23), respectively. The 4-fluorophenyl ring (C18–C23) is perpendicular to the carbamate acid plane, the dihedral angle between them is 89.5 (3)°.

The 4-fluorophenyl group is rotating away from the pyridine ring system compared to the recently published crystal structure of methyl 4-(5-(4-fluorophenyl)-4-(pyridin-4-yl)-1*H*-imidazol-2-ylthio)butanoate (Koch *et al.* 2008*b*).

## Experimental

*tert*-Butyl *N*-benzyl-*N*-(4-methylpyridin-2-yl)carbamate (6.27 g, 21.0 mmol) and ethyl 4-fluorobenzoate (3.89 g, 23.1 mmol) were dissolved in dry THF (60 ml) under argon atmosphere. The solution was cooled to 273 K and NaHMDS (21.0 ml, 42.0 mmol, 2 *M* in THF) was added dropwise. The mixture was allowed to stir at this temperature for 1 h and additional 2.5 h at room temperature. The reaction was quenched with saturated $NH_4Cl$ solution, EtOAc was added and the mixture was extracted twice with water. The organic layer was dried (sodium sulfate) and concentrated *in vacuo*. The crude product was purified by flash chromatography (silica gel, petroleum ether/ethylacetate 5–1 to 3–1) to yield 5.40 g (61%) of **I** as colourless crystals (Koch *et al.* 2008*a*).

## Refinement

Hydrogen atoms attached to carbons were placed at calculated positions with C—H = 0.95 Å (aromatic) or 0.98–0.99 Å ($sp^3$ C-atom). All H atoms were refined in the riding-model approximation with isotropic displacement parameters (set at 1.2–1.5 times of the $U_{eq}$ of the parent atom).

## Figures

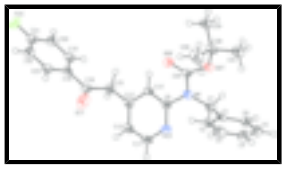


Fig. 1. View of compound **I**. Displacement ellipsoids are drawn at the 50% probability level. H atoms are depicted as circles of arbitrary size.

## *tert*-Butyl *N*-benzyl-*N*-[4-(4-fluorobenzoylmethyl)-2-pyridyl]carbamate

*Crystal data*

| | |
|---|---|
| $C_{25}H_{25}FN_2O_3$ | $F_{000}$ = 1776 |
| $M_r$ = 420.47 | $D_x$ = 1.297 Mg m$^{-3}$ |
| Monoclinic, *C*2/*c* | Cu *K*α radiation<br>λ = 1.54178 Å |
| Hall symbol: -C 2yc | Cell parameters from 25 reflections |
| *a* = 38.054 (7) Å | θ = 15–28º |
| *b* = 7.9320 (6) Å | μ = 0.75 mm$^{-1}$ |
| *c* = 14.589 (3) Å | *T* = 193 (2) K |
| β = 102.142 (8)º | Plate, colourless |
| *V* = 4305.1 (11) Å$^3$ | 0.35 × 0.30 × 0.18 mm |
| *Z* = 8 | |

*Data collection*

| | |
|---|---|
| Enraf–Nonius CAD-4 diffractometer | $\theta_{max}$ = 70.1º |
| Monochromator: graphite | $\theta_{min}$ = 2.4º |
| *T* = 193(2) K | *h* = −46→45 |
| ω/2θ scans | *k* = −9→0 |
| Absorption correction: none | *l* = 0→17 |
| 4272 measured reflections | 3 standard reflections |
| 4091 independent reflections | every 60 min |
| 2192 reflections with $I > 2\sigma(I)$ | intensity decay: 3% |
| $R_{int}$ = 0.076 | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)]$ = 0.110 | H-atom parameters constrained |
| $wR(F^2)$ = 0.339 | $w = 1/[\sigma^2(F_o^2) + (0.182P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| *S* = 1.10 | $(\Delta/\sigma)_{max} < 0.001$ |
| 4091 reflections | $\Delta\rho_{max}$ = 0.48 e Å$^{-3}$ |

| | |
|---|---|
| 283 parameters | $\Delta\rho_{min} = -0.64$ e Å$^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Extinction correction: none |

*Special details*

**Experimental**. No absorption correction was applied because of irregular crystal shape and low crystal quality. The crystal diffracted only very weak (less the 55% observed reflections).

**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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.30976 (15) | 0.4803 (7) | 0.3672 (4) | 0.0242 (12) |
| C2 | 0.33725 (17) | 0.5800 (8) | 0.4143 (4) | 0.0324 (14) |
| H2 | 0.3612 | 0.5383 | 0.4269 | 0.039* |
| C3 | 0.33038 (19) | 0.7421 (8) | 0.4438 (5) | 0.0406 (16) |
| H3 | 0.3495 | 0.8094 | 0.4773 | 0.049* |
| C4 | 0.2957 (2) | 0.8035 (9) | 0.4240 (5) | 0.0422 (17) |
| H4 | 0.2909 | 0.9137 | 0.4437 | 0.051* |
| C5 | 0.26812 (18) | 0.7057 (9) | 0.3759 (4) | 0.0386 (16) |
| H5 | 0.2443 | 0.7486 | 0.3619 | 0.046* |
| C6 | 0.27507 (16) | 0.5439 (8) | 0.3476 (4) | 0.0297 (13) |
| H6 | 0.2559 | 0.4764 | 0.3146 | 0.036* |
| C7 | 0.31548 (15) | 0.3018 (8) | 0.3346 (4) | 0.0265 (13) |
| H7A | 0.2945 | 0.2325 | 0.3406 | 0.032* |
| H7B | 0.3163 | 0.3058 | 0.2672 | 0.032* |
| N8 | 0.34806 (12) | 0.2177 (6) | 0.3852 (3) | 0.0273 (11) |
| C9 | 0.37651 (15) | 0.1822 (7) | 0.3379 (4) | 0.0241 (12) |
| N10 | 0.38074 (14) | 0.3047 (6) | 0.2779 (3) | 0.0326 (12) |
| C11 | 0.40604 (19) | 0.2738 (9) | 0.2270 (5) | 0.0440 (18) |
| H11 | 0.4097 | 0.3582 | 0.1838 | 0.053* |
| C12 | 0.42649 (17) | 0.1328 (9) | 0.2326 (4) | 0.0389 (16) |
| H12 | 0.4433 | 0.1178 | 0.1934 | 0.047* |
| C13 | 0.42200 (15) | 0.0098 (8) | 0.2982 (4) | 0.0268 (13) |
| C14 | 0.39684 (15) | 0.0377 (7) | 0.3519 (4) | 0.0240 (12) |
| H14 | 0.3936 | −0.0418 | 0.3981 | 0.029* |
| C15 | 0.44316 (16) | −0.1512 (8) | 0.3080 (4) | 0.0297 (13) |
| H15A | 0.4419 | −0.1996 | 0.2448 | 0.036* |
| H15B | 0.4319 | −0.2328 | 0.3443 | 0.036* |

| | | | | |
|---|---|---|---|---|
| C16 | 0.48232 (17) | −0.1287 (8) | 0.3558 (5) | 0.0341 (15) |
| O17 | 0.49559 (13) | 0.0077 (6) | 0.3700 (5) | 0.0666 (18) |
| C18 | 0.50420 (15) | −0.2837 (8) | 0.3851 (4) | 0.0268 (13) |
| C19 | 0.49111 (16) | −0.4454 (8) | 0.3620 (4) | 0.0279 (13) |
| H19 | 0.4676 | −0.4597 | 0.3249 | 0.034* |
| C20 | 0.51188 (18) | −0.5852 (8) | 0.3923 (4) | 0.0337 (15) |
| H20 | 0.5030 | −0.6957 | 0.3766 | 0.040* |
| C21 | 0.54570 (18) | −0.5608 (8) | 0.4458 (4) | 0.0332 (14) |
| C22 | 0.55982 (19) | −0.4035 (9) | 0.4691 (5) | 0.0413 (17) |
| H22 | 0.5835 | −0.3907 | 0.5054 | 0.050* |
| C23 | 0.53868 (16) | −0.2635 (8) | 0.4385 (4) | 0.0325 (14) |
| H23 | 0.5479 | −0.1535 | 0.4541 | 0.039* |
| F24 | 0.56589 (11) | −0.6962 (5) | 0.4773 (3) | 0.0487 (11) |
| C25 | 0.35189 (15) | 0.1753 (7) | 0.4796 (3) | 0.0213 (12) |
| O26 | 0.37827 (11) | 0.1119 (5) | 0.5264 (3) | 0.0299 (10) |
| O27 | 0.32169 (10) | 0.2188 (5) | 0.5079 (2) | 0.0260 (9) |
| C28 | 0.32262 (16) | 0.2339 (8) | 0.6097 (3) | 0.0307 (14) |
| C29 | 0.32429 (19) | 0.0591 (10) | 0.6534 (4) | 0.0440 (18) |
| H29A | 0.3033 | −0.0063 | 0.6227 | 0.066* |
| H29B | 0.3245 | 0.0693 | 0.7204 | 0.066* |
| H29C | 0.3462 | 0.0018 | 0.6451 | 0.066* |
| C30 | 0.3536 (2) | 0.3456 (11) | 0.6559 (5) | 0.053 (2) |
| H30A | 0.3763 | 0.2953 | 0.6482 | 0.080* |
| H30B | 0.3537 | 0.3566 | 0.7228 | 0.080* |
| H30C | 0.3509 | 0.4573 | 0.6265 | 0.080* |
| C31 | 0.28696 (18) | 0.3212 (10) | 0.6091 (4) | 0.0443 (18) |
| H31A | 0.2861 | 0.4284 | 0.5752 | 0.066* |
| H31B | 0.2847 | 0.3428 | 0.6737 | 0.066* |
| H31C | 0.2672 | 0.2488 | 0.5779 | 0.066* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.032 (3) | 0.026 (3) | 0.018 (3) | −0.006 (2) | 0.012 (2) | 0.000 (2) |
| C2 | 0.034 (3) | 0.027 (3) | 0.039 (3) | −0.003 (3) | 0.014 (3) | 0.006 (3) |
| C3 | 0.052 (4) | 0.029 (4) | 0.042 (4) | −0.015 (3) | 0.013 (3) | −0.005 (3) |
| C4 | 0.061 (5) | 0.028 (4) | 0.042 (4) | 0.010 (3) | 0.021 (3) | −0.005 (3) |
| C5 | 0.039 (4) | 0.045 (4) | 0.034 (3) | 0.010 (3) | 0.014 (3) | 0.005 (3) |
| C6 | 0.035 (3) | 0.033 (3) | 0.023 (3) | 0.004 (3) | 0.009 (2) | 0.002 (3) |
| C7 | 0.031 (3) | 0.036 (3) | 0.013 (2) | 0.002 (3) | 0.006 (2) | −0.001 (2) |
| N8 | 0.029 (3) | 0.037 (3) | 0.018 (2) | 0.003 (2) | 0.0098 (19) | 0.002 (2) |
| C9 | 0.027 (3) | 0.027 (3) | 0.019 (2) | −0.002 (2) | 0.006 (2) | 0.002 (2) |
| N10 | 0.044 (3) | 0.029 (3) | 0.027 (3) | 0.007 (2) | 0.015 (2) | 0.009 (2) |
| C11 | 0.054 (4) | 0.046 (4) | 0.040 (4) | 0.013 (3) | 0.031 (3) | 0.021 (3) |
| C12 | 0.042 (4) | 0.049 (4) | 0.035 (3) | 0.012 (3) | 0.028 (3) | 0.011 (3) |
| C13 | 0.029 (3) | 0.029 (3) | 0.022 (3) | −0.001 (3) | 0.005 (2) | −0.003 (2) |
| C14 | 0.032 (3) | 0.025 (3) | 0.015 (2) | −0.003 (2) | 0.006 (2) | 0.001 (2) |
| C15 | 0.037 (3) | 0.030 (3) | 0.026 (3) | 0.003 (3) | 0.015 (2) | 0.000 (3) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C16 | 0.036 (3) | 0.028 (3) | 0.040 (3) | 0.000 (3) | 0.011 (3) | 0.012 (3) |
| O17 | 0.042 (3) | 0.028 (3) | 0.118 (5) | −0.006 (2) | −0.011 (3) | 0.009 (3) |
| C18 | 0.027 (3) | 0.030 (3) | 0.027 (3) | −0.001 (2) | 0.013 (2) | 0.003 (3) |
| C19 | 0.031 (3) | 0.032 (3) | 0.023 (3) | 0.000 (3) | 0.011 (2) | −0.004 (3) |
| C20 | 0.051 (4) | 0.022 (3) | 0.033 (3) | 0.002 (3) | 0.020 (3) | −0.001 (3) |
| C21 | 0.042 (4) | 0.035 (4) | 0.026 (3) | 0.014 (3) | 0.016 (3) | 0.004 (3) |
| C22 | 0.035 (4) | 0.050 (5) | 0.039 (4) | 0.006 (3) | 0.008 (3) | 0.009 (3) |
| C23 | 0.037 (3) | 0.030 (3) | 0.033 (3) | −0.001 (3) | 0.013 (3) | 0.005 (3) |
| F24 | 0.062 (3) | 0.040 (2) | 0.045 (2) | 0.022 (2) | 0.015 (2) | 0.0098 (19) |
| C25 | 0.031 (3) | 0.021 (3) | 0.014 (2) | −0.007 (2) | 0.008 (2) | −0.004 (2) |
| O26 | 0.032 (2) | 0.040 (3) | 0.0165 (19) | 0.0031 (19) | 0.0030 (16) | 0.0027 (18) |
| O27 | 0.033 (2) | 0.032 (2) | 0.0157 (18) | −0.0009 (18) | 0.0114 (16) | −0.0026 (17) |
| C28 | 0.042 (3) | 0.043 (4) | 0.009 (2) | −0.005 (3) | 0.011 (2) | −0.007 (2) |
| C29 | 0.042 (4) | 0.070 (5) | 0.022 (3) | −0.001 (4) | 0.010 (3) | 0.013 (3) |
| C30 | 0.055 (5) | 0.071 (6) | 0.035 (4) | −0.017 (4) | 0.012 (3) | −0.023 (4) |
| C31 | 0.053 (4) | 0.059 (5) | 0.025 (3) | 0.002 (4) | 0.019 (3) | −0.003 (3) |

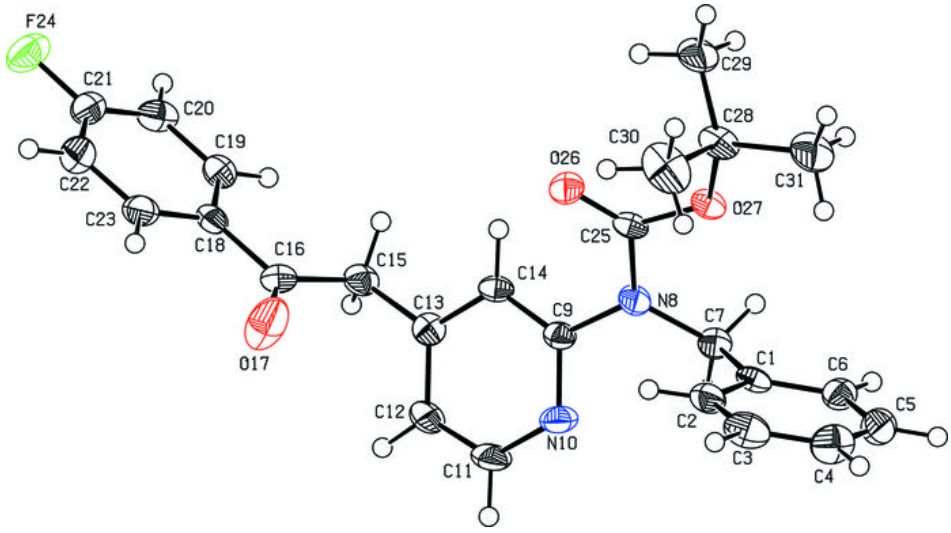
*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—C2 | 1.374 (8) | O27—C28 | 1.484 (6) |
| C1—C6 | 1.385 (8) | C28—C30 | 1.515 (9) |
| C1—C7 | 1.525 (8) | C28—C29 | 1.521 (9) |
| C2—C3 | 1.398 (9) | C28—C31 | 1.522 (9) |
| C3—C4 | 1.378 (10) | C2—H2 | 0.9500 |
| C4—C5 | 1.375 (10) | C3—H3 | 0.9500 |
| C5—C6 | 1.391 (9) | C4—H4 | 0.9500 |
| C7—N8 | 1.464 (7) | C5—H5 | 0.9500 |
| N8—C25 | 1.396 (6) | C6—H6 | 0.9500 |
| N8—C9 | 1.429 (7) | C7—H7A | 0.9900 |
| C9—N10 | 1.340 (7) | C7—H7B | 0.9900 |
| C9—C14 | 1.374 (8) | C11—H11 | 0.9500 |
| N10—C11 | 1.356 (7) | C12—H12 | 0.9500 |
| C11—C12 | 1.355 (9) | C14—H14 | 0.9500 |
| C12—C13 | 1.403 (8) | C15—H15A | 0.9900 |
| C13—C14 | 1.376 (7) | C15—H15B | 0.9900 |
| C13—C15 | 1.500 (8) | C19—H19 | 0.9500 |
| C15—C16 | 1.517 (9) | C20—H20 | 0.9500 |
| C16—O17 | 1.194 (8) | C22—H22 | 0.9500 |
| C16—C18 | 1.496 (8) | C23—H23 | 0.9500 |
| C18—C23 | 1.387 (8) | C29—H29A | 0.9800 |
| C18—C19 | 1.391 (8) | C29—H29B | 0.9800 |
| C19—C20 | 1.380 (8) | C29—H29C | 0.9800 |
| C20—C21 | 1.371 (9) | C30—H30A | 0.9800 |
| C21—F24 | 1.344 (7) | C30—H30B | 0.9800 |
| C21—C22 | 1.373 (10) | C30—H30C | 0.9800 |
| C22—C23 | 1.389 (9) | C31—H31A | 0.9800 |
| C25—O26 | 1.199 (7) | C31—H31B | 0.9800 |
| C25—O27 | 1.345 (6) | C31—H31C | 0.9800 |
| C2—C1—C6 | 119.1 (6) | C4—C3—H3 | 120.00 |

| | | | |
|---|---|---|---|
| C2—C1—C7 | 123.2 (5) | C3—C4—H4 | 120.00 |
| C6—C1—C7 | 117.8 (5) | C5—C4—H4 | 120.00 |
| C1—C2—C3 | 120.7 (6) | C4—C5—H5 | 120.00 |
| C4—C3—C2 | 119.6 (6) | C6—C5—H5 | 120.00 |
| C5—C4—C3 | 120.2 (6) | C1—C6—H6 | 120.00 |
| C4—C5—C6 | 120.0 (6) | C5—C6—H6 | 120.00 |
| C1—C6—C5 | 120.5 (6) | N8—C7—H7A | 108.00 |
| N8—C7—C1 | 115.3 (5) | N8—C7—H7B | 108.00 |
| C25—N8—C9 | 119.8 (5) | C1—C7—H7A | 109.00 |
| C25—N8—C7 | 120.7 (4) | C1—C7—H7B | 108.00 |
| C9—N8—C7 | 119.5 (4) | H7A—C7—H7B | 107.00 |
| N10—C9—C14 | 124.1 (5) | N10—C11—H11 | 117.00 |
| N10—C9—N8 | 112.3 (5) | C12—C11—H11 | 117.00 |
| C14—C9—N8 | 123.5 (5) | C11—C12—H12 | 121.00 |
| C9—N10—C11 | 115.0 (5) | C13—C12—H12 | 121.00 |
| C12—C11—N10 | 125.5 (6) | C9—C14—H14 | 120.00 |
| C11—C12—C13 | 117.7 (5) | C13—C14—H14 | 120.00 |
| C14—C13—C12 | 118.4 (5) | C13—C15—H15A | 109.00 |
| C14—C13—C15 | 120.5 (5) | C13—C15—H15B | 109.00 |
| C12—C13—C15 | 121.1 (5) | C16—C15—H15A | 109.00 |
| C9—C14—C13 | 119.1 (5) | C16—C15—H15B | 109.00 |
| C13—C15—C16 | 113.6 (5) | H15A—C15—H15B | 108.00 |
| O17—C16—C18 | 120.4 (6) | C18—C19—H19 | 120.00 |
| O17—C16—C15 | 121.7 (6) | C20—C19—H19 | 120.00 |
| C18—C16—C15 | 118.0 (5) | C19—C20—H20 | 121.00 |
| C23—C18—C19 | 119.4 (6) | C21—C20—H20 | 121.00 |
| C23—C18—C16 | 118.0 (6) | C21—C22—H22 | 121.00 |
| C19—C18—C16 | 122.6 (5) | C23—C22—H22 | 121.00 |
| C20—C19—C18 | 120.8 (6) | C18—C23—H23 | 120.00 |
| C21—C20—C19 | 118.4 (6) | C22—C23—H23 | 120.00 |
| F24—C21—C20 | 118.9 (6) | C28—C29—H29A | 109.00 |
| F24—C21—C22 | 118.4 (6) | C28—C29—H29B | 109.00 |
| C20—C21—C22 | 122.7 (6) | C28—C29—H29C | 109.00 |
| C21—C22—C23 | 118.5 (6) | H29A—C29—H29B | 110.00 |
| C18—C23—C22 | 120.3 (6) | H29A—C29—H29C | 109.00 |
| O26—C25—O27 | 126.9 (5) | H29B—C29—H29C | 109.00 |
| O26—C25—N8 | 124.3 (5) | C28—C30—H30A | 109.00 |
| O27—C25—N8 | 108.8 (5) | C28—C30—H30B | 110.00 |
| C25—O27—C28 | 119.0 (4) | C28—C30—H30C | 109.00 |
| O27—C28—C30 | 110.2 (5) | H30A—C30—H30B | 109.00 |
| O27—C28—C29 | 109.6 (5) | H30A—C30—H30C | 109.00 |
| C30—C28—C29 | 112.8 (6) | H30B—C30—H30C | 109.00 |
| O27—C28—C31 | 101.4 (5) | C28—C31—H31A | 109.00 |
| C30—C28—C31 | 110.2 (6) | C28—C31—H31B | 109.00 |
| C29—C28—C31 | 112.1 (5) | C28—C31—H31C | 109.00 |
| C1—C2—H2 | 120.00 | H31A—C31—H31B | 109.00 |
| C3—C2—H2 | 120.00 | H31A—C31—H31C | 109.00 |
| C2—C3—H3 | 120.00 | H31B—C31—H31C | 110.00 |
| C6—C1—C2—C3 | 1.3 (8) | C12—C13—C15—C16 | 73.5 (7) |

| | | | |
|---|---|---|---|
| C7—C1—C2—C3 | −179.1 (5) | C13—C15—C16—O17 | −11.2 (9) |
| C1—C2—C3—C4 | −1.1 (9) | C13—C15—C16—C18 | 168.9 (5) |
| C2—C3—C4—C5 | 0.2 (10) | O17—C16—C18—C23 | 7.5 (9) |
| C3—C4—C5—C6 | 0.5 (10) | C15—C16—C18—C23 | −172.6 (5) |
| C2—C1—C6—C5 | −0.6 (8) | O17—C16—C18—C19 | −173.4 (7) |
| C7—C1—C6—C5 | 179.8 (5) | C15—C16—C18—C19 | 6.5 (8) |
| C4—C5—C6—C1 | −0.3 (9) | C23—C18—C19—C20 | 0.6 (8) |
| C2—C1—C7—N8 | 22.7 (7) | C16—C18—C19—C20 | −178.4 (5) |
| C6—C1—C7—N8 | −157.7 (5) | C18—C19—C20—C21 | 0.0 (8) |
| C1—C7—N8—C25 | 66.5 (7) | C19—C20—C21—F24 | 178.7 (5) |
| C1—C7—N8—C9 | −112.1 (5) | C19—C20—C21—C22 | −0.9 (9) |
| C25—N8—C9—N10 | −141.6 (5) | F24—C21—C22—C23 | −178.5 (5) |
| C7—N8—C9—N10 | 37.1 (7) | C20—C21—C22—C23 | 1.1 (10) |
| C25—N8—C9—C14 | 39.1 (8) | C19—C18—C23—C22 | −0.5 (9) |
| C7—N8—C9—C14 | −142.3 (6) | C16—C18—C23—C22 | 178.6 (6) |
| C14—C9—N10—C11 | 2.8 (9) | C21—C22—C23—C18 | −0.4 (9) |
| N8—C9—N10—C11 | −176.5 (6) | C9—N8—C25—O26 | 1.4 (9) |
| C9—N10—C11—C12 | 0.0 (11) | C7—N8—C25—O26 | −177.2 (6) |
| N10—C11—C12—C13 | −1.9 (12) | C9—N8—C25—O27 | −179.2 (5) |
| C11—C12—C13—C14 | 1.0 (10) | C7—N8—C25—O27 | 2.2 (7) |
| C11—C12—C13—C15 | 179.1 (6) | O26—C25—O27—C28 | 17.1 (8) |
| N10—C9—C14—C13 | −3.7 (9) | N8—C25—O27—C28 | −162.3 (5) |
| N8—C9—C14—C13 | 175.5 (5) | C25—O27—C28—C30 | 51.0 (7) |
| C12—C13—C14—C9 | 1.6 (8) | C25—O27—C28—C29 | −73.6 (6) |
| C15—C13—C14—C9 | −176.5 (5) | C25—O27—C28—C31 | 167.7 (5) |
| C14—C13—C15—C16 | −108.4 (6) | | |

**Fig. 1**


F24
C21
C20
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C31
O27