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# *tert*-Butyl *N*-{4-methyl-3-[4-(3-pyridyl)-pyrimidin-2-yloxy]phenyl}carbamate

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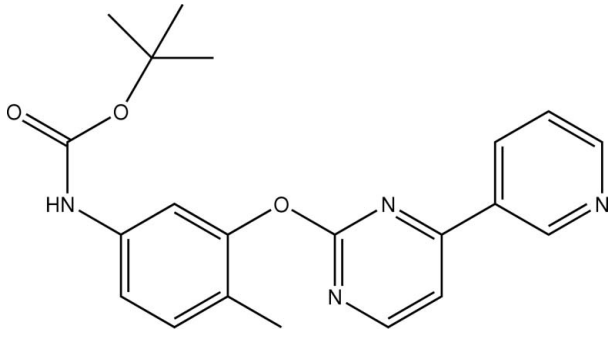
Key indicators: single-crystal X-ray study; $T$ = 294 K; mean $\sigma$(C–C) = 0.005 Å; $R$ factor = 0.058; $wR$ factor = 0.184; data-to-parameter ratio = 14.4.

In the molecule of the title compound, $C_{21}H_{22}N_4O_3$, the pyrimidine ring is oriented at dihedral angles of 0.51 (3) and 50.76 (3)° to the pyridine and benzene rings, respectively. In the crystal structure, intermolecular N—H···N hydrogen bonds link the molecules into centrosymmetric dimers, forming $R_2^2(24)$ ring motifs; the dimers are linked by intermolecular C—H···O hydrogen bonds into a two-dimensional network. $\pi$–$\pi$ contacts between the benzene rings and between the pyrimidine and pyridine rings [centroid–centroid distances = 3.891 (1) and 3.646 (1) Å, respectively] may further stabilize the structure. Two weak C—H···$\pi$ interactions are also present.

## Related literature

For bond-length data, see: Allen *et al.* (1987). For ring-motifs, see: Bernstein *et al.* (1995).



## Experimental

### *Crystal data*

$C_{21}H_{22}N_4O_3$
$M_r$ = 378.43
Triclinic, $P\overline{1}$
$a$ = 9.951 (2) Å
$b$ = 10.733 (2) Å
$c$ = 11.577 (2) Å
$\alpha$ = 114.74 (3)°
$\beta$ = 107.14 (3)°
$\gamma$ = 99.97 (3)°
$V$ = 1008.6 (6) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 294 K
0.30 × 0.20 × 0.10 mm

### *Data collection*

Enraf–Nonius CAD-4 diffractometer
Absorption correction: $\psi$ scan (North *et al.*, 1968)
$T_{min}$ = 0.975, $T_{max}$ = 0.992
3882 measured reflections
3652 independent reflections
2333 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.026
3 standard reflections
frequency: 120 min
intensity decay: 1%

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.184
$S$ = 1.01
3652 reflections
254 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.29 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.27 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1$A$···N4$^{i}$ | 0.86 | 2.10 | 2.944 (4) | 165 |
| C15—H15$A$···O2$^{ii}$ | 0.93 | 2.45 | 3.382 (4) | 177 |
| C18—H18$A$···O2$^{ii}$ | 0.93 | 2.39 | 3.319 (4) | 174 |
| C3—H3$B$···$Cg1^{i}$ | 0.96 | 2.86 | 3.560 (3) | 131 |
| C12—H12$B$···$Cg2^{iii}$ | 0.96 | 2.90 | 3.788 (3) | 154 |

Symmetry codes: (i) $-x, -y+2, -z+1$; (ii) $-x, -y+1, -z+1$; (iii) $-x+1, -y+2, -z+2$. $Cg1$ and $Cg2$ are the centroids of the C6–C11 and N2/N3/C13–C16 rings, respectively.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2725).

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# supplementary materials

*Acta Cryst.* (2009). E**65**, o1849 [ **doi:10.1107/S1600536809025604** ]

# *tert*-Butyl *N*-{4-methyl-3-[4-(3-pyridyl)pyrimidin-2-yloxy]phenyl}carbamate

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## Comment

Some derivatives of phenol are important chemical materials. We report herein the crystal structure of the title compound.

In the molecule of the title compound (Fig 1), the bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. Rings A (C6-C11), B (N2/N3/C13-C16) and C (N4/C17-C21) are, of course, planar and the dihedral angles between them are A/B = 50.76 (3), A/C = 50.58 (3) and B/C = 0.51 (3) °.

In the crystal structure, intermolecular N-H···N hydrogen bonds (Table 1) link the molecules into centrosymmetric dimers forming $R_2^2(24)$ ring motifs (Bernstein *et al.*, 1995), and then intermolecular C-H···O hydrogen bonds (Table 1) link them into a two dimensional network (Fig. 2), in which they may be effective in the stabilization of the structure. The π–π contacts between the phenyl rings and between the pyrimidine and the pyridine rings, Cg1—Cg1$^{i}$ and Cg2—Cg3$^{ii}$ [symmetry codes: (i) 1 - x, 2 - y, 1 - z, (ii) -x, 1 - y, 1 - z, where Cg1, Cg2 and Cg3 are centroids of the rings A (C6-C11), B (N2/N3/C13-C16) and C (N4/C17-C21), respectively] may further stabilize the structure, with centroid-centroid distances of 3.891 (1) and 3.646 (1) Å, respectively. There also exist two weak C—H···π interactions (Table 1).

## Experimental

To a mixture of 2-(methylsulfonyl)-4-(pyridin-3-yl)pyrimidine (47.1 g, 0.2 mol) in DMF (75 ml) and *tert*-butyl-3-hydroxy-4-methylphenylcarbamate (44.7 g, 0.2 mol) in DMF (150 ml) was added sodium hydride (44.4 g) slowly and was stirred for 18 h at room temperature. After acidified with citric acid the reaction mixture was poured into ice-water (2000 ml). The precipitate was filtered, washed with water and was extracted with dichloromethane.The dichloromethane layer was dried over anhydrous magnesium sulfate and evaporated *in vacuo*. The residue was recrystallized from ethyl ether to give the title compound (yield; 73.4 g). Crystals suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution.

## Refinement

H atoms were positioned geometrically with N-H = 0.86 Å (for NH) and C-H = 0.93 and 0.96 Å for aromatic and methyl H atoms, respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = xU_{eq}(C,N)$, where x = 1.5 for methyl H and x = 1.2 for all other H atoms.

## Figures

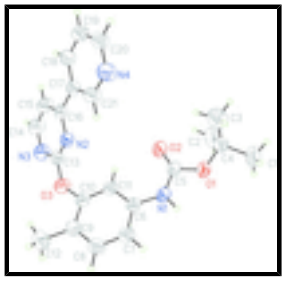

Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

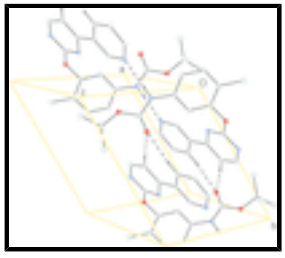

Fig. 2. A partial packing diagram of the title compound. Hydrogen bonds are shown as dashed lines.

## *tert*-Butyl *N*-{4-methyl-3-[4-(3-pyridyl)pyrimidin-2- yloxy]phenyl}carbamate

*Crystal data*

| | |
|---|---|
| $C_{21}H_{22}N_4O_3$ | $Z = 2$ |
| $M_r = 378.43$ | $F_{000} = 400$ |
| Triclinic, $P\bar{1}$ | $D_x = 1.246$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $a = 9.951$ (2) Å | Cell parameters from 25 reflections |
| $b = 10.733$ (2) Å | $\theta = 9$–13º |
| $c = 11.577$ (2) Å | $\mu = 0.09$ mm$^{-1}$ |
| $\alpha = 114.74$ (3)º | $T = 294$ K |
| $\beta = 107.14$ (3)º | Block, colorless |
| $\gamma = 99.97$ (3)º | 0.30 × 0.20 × 0.10 mm |
| $V = 1008.6$ (6) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Enraf–Nonius CAD-4 diffractometer | $R_{int} = 0.026$ |
| Radiation source: fine-focus sealed tube | $\theta_{max} = 25.3$º |
| Monochromator: graphite | $\theta_{min} = 2.1$º |
| $T = 294$ K | $h = 0 \rightarrow 11$ |
| $\omega/2\theta$ scans | $k = -12 \rightarrow 12$ |
| Absorption correction: $\psi$ scan (North *et al.*, 1968) | $l = -13 \rightarrow 13$ |
| $T_{min} = 0.975$, $T_{max} = 0.992$ | 3 standard reflections |
| 3882 measured reflections | every 120 min |
| 3652 independent reflections | intensity decay: 1% |

2333 reflections with $I > 2\sigma(I)$

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | H-atom parameters constrained |
| $R[F^2 > 2\sigma(F^2)] = 0.058$ | $w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 0.07P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2) = 0.184$ | $(\Delta/\sigma)_{max} < 0.001$ |
| $S = 1.01$ | $\Delta\rho_{max} = 0.29$ e Å$^{-3}$ |
| 3652 reflections | $\Delta\rho_{min} = -0.27$ e Å$^{-3}$ |
| 254 parameters | Extinction correction: SHELXL97 (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$ |
| Primary atom site location: structure-invariant direct methods | Extinction coefficient: 0.033 (5) |
| Secondary atom site location: difference Fourier map | |

*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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.0153 (2) | 0.8866 (2) | 0.2102 (2) | 0.0600 (6) |
| O2 | 0.0613 (2) | 0.7528 (2) | 0.3167 (2) | 0.0719 (7) |
| O3 | 0.4840 (2) | 0.8449 (2) | 0.7311 (2) | 0.0594 (6) |
| N1 | 0.2087 (2) | 0.9880 (2) | 0.4085 (2) | 0.0498 (6) |
| H1A | 0.2141 | 1.0599 | 0.3929 | 0.060* |
| N2 | 0.2426 (2) | 0.7463 (2) | 0.7031 (2) | 0.0429 (5) |
| N3 | 0.4351 (3) | 0.6437 (3) | 0.7463 (3) | 0.0574 (7) |
| N4 | −0.1874 (3) | 0.7568 (2) | 0.6228 (3) | 0.0567 (7) |
| C1 | −0.1644 (4) | 0.8538 (4) | 0.0096 (4) | 0.0910 (12) |
| H1B | −0.1731 | 0.9458 | 0.0640 | 0.136* |
| H1C | −0.2570 | 0.7920 | −0.0708 | 0.136* |
| H1D | −0.0855 | 0.8694 | −0.0200 | 0.136* |
| C2 | −0.1138 (4) | 0.6358 (4) | 0.0150 (4) | 0.0862 (11) |
| H2B | −0.0922 | 0.5919 | 0.0724 | 0.129* |
| H2C | −0.0339 | 0.6503 | −0.0136 | 0.129* |

| | | | | |
|---|---|---|---|---|
| H2D | −0.2057 | 0.5727 | −0.0659 | 0.129* |
| C3 | −0.2421 (4) | 0.7685 (5) | 0.1575 (4) | 0.0929 (12) |
| H3B | −0.2485 | 0.8630 | 0.2080 | 0.139* |
| H3C | −0.2119 | 0.7312 | 0.2196 | 0.139* |
| H3D | −0.3381 | 0.7032 | 0.0834 | 0.139* |
| C4 | −0.1288 (3) | 0.7813 (3) | 0.0971 (3) | 0.0550 (8) |
| C5 | 0.0919 (3) | 0.8649 (3) | 0.3128 (3) | 0.0490 (7) |
| C6 | 0.3229 (3) | 1.0147 (3) | 0.5304 (3) | 0.0417 (6) |
| C7 | 0.4275 (3) | 1.1551 (3) | 0.6155 (3) | 0.0515 (7) |
| H7A | 0.4202 | 1.2258 | 0.5898 | 0.062* |
| C8 | 0.5415 (3) | 1.1899 (3) | 0.7372 (3) | 0.0573 (8) |
| H8A | 0.6102 | 1.2844 | 0.7926 | 0.069* |
| C9 | 0.5577 (3) | 1.0880 (3) | 0.7807 (3) | 0.0505 (7) |
| C10 | 0.4535 (3) | 0.9502 (3) | 0.6937 (3) | 0.0449 (6) |
| C11 | 0.3367 (3) | 0.9096 (3) | 0.5703 (3) | 0.0459 (7) |
| H11A | 0.2689 | 0.8147 | 0.5150 | 0.055* |
| C12 | 0.6828 (4) | 1.1258 (4) | 0.9146 (3) | 0.0766 (10) |
| H12A | 0.6742 | 1.0413 | 0.9256 | 0.115* |
| H12B | 0.6764 | 1.2028 | 0.9920 | 0.115* |
| H12C | 0.7773 | 1.1573 | 0.9114 | 0.115* |
| C13 | 0.3789 (3) | 0.7420 (3) | 0.7256 (3) | 0.0446 (6) |
| C14 | 0.3369 (3) | 0.5396 (3) | 0.7410 (3) | 0.0610 (8) |
| H14A | 0.3692 | 0.4692 | 0.7562 | 0.073* |
| C15 | 0.1907 (3) | 0.5281 (3) | 0.7147 (3) | 0.0569 (8) |
| H15A | 0.1244 | 0.4510 | 0.7090 | 0.068* |
| C16 | 0.1453 (3) | 0.6373 (3) | 0.6967 (2) | 0.0404 (6) |
| C17 | −0.0094 (3) | 0.6389 (3) | 0.6684 (3) | 0.0404 (6) |
| C18 | −0.1188 (3) | 0.5330 (3) | 0.6602 (3) | 0.0507 (7) |
| H18A | −0.0970 | 0.4574 | 0.6725 | 0.061* |
| C19 | −0.2597 (3) | 0.5405 (3) | 0.6339 (3) | 0.0571 (8) |
| H19A | −0.3347 | 0.4697 | 0.6277 | 0.068* |
| C20 | −0.2896 (3) | 0.6538 (3) | 0.6167 (3) | 0.0525 (7) |
| H20A | −0.3854 | 0.6584 | 0.6000 | 0.063* |
| C21 | −0.0505 (3) | 0.7479 (3) | 0.6477 (3) | 0.0519 (7) |
| H21A | 0.0217 | 0.8189 | 0.6514 | 0.062* |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0613 (12) | 0.0482 (11) | 0.0602 (12) | 0.0112 (10) | 0.0039 (10) | 0.0353 (10) |
| O2 | 0.0703 (14) | 0.0469 (12) | 0.0834 (15) | 0.0087 (10) | 0.0013 (12) | 0.0443 (11) |
| O3 | 0.0419 (11) | 0.0666 (13) | 0.0880 (15) | 0.0232 (10) | 0.0201 (10) | 0.0567 (12) |
| N1 | 0.0556 (14) | 0.0365 (12) | 0.0589 (14) | 0.0166 (11) | 0.0147 (12) | 0.0307 (11) |
| N2 | 0.0440 (13) | 0.0406 (12) | 0.0466 (13) | 0.0166 (10) | 0.0146 (10) | 0.0258 (10) |
| N3 | 0.0573 (15) | 0.0581 (15) | 0.0733 (17) | 0.0319 (13) | 0.0248 (13) | 0.0435 (14) |
| N4 | 0.0489 (14) | 0.0506 (14) | 0.0806 (18) | 0.0231 (12) | 0.0241 (13) | 0.0406 (14) |
| C1 | 0.089 (3) | 0.086 (3) | 0.079 (2) | 0.018 (2) | 0.000 (2) | 0.053 (2) |
| C2 | 0.101 (3) | 0.064 (2) | 0.071 (2) | 0.030 (2) | 0.023 (2) | 0.0216 (19) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C3 | 0.068 (2) | 0.119 (3) | 0.106 (3) | 0.039 (2) | 0.037 (2) | 0.066 (3) |
| C4 | 0.0508 (17) | 0.0516 (17) | 0.0580 (18) | 0.0161 (14) | 0.0135 (15) | 0.0299 (15) |
| C5 | 0.0509 (16) | 0.0428 (15) | 0.0578 (17) | 0.0181 (13) | 0.0175 (14) | 0.0313 (14) |
| C6 | 0.0423 (14) | 0.0407 (14) | 0.0512 (16) | 0.0192 (12) | 0.0210 (13) | 0.0275 (13) |
| C7 | 0.0546 (17) | 0.0417 (15) | 0.0616 (18) | 0.0178 (13) | 0.0215 (15) | 0.0296 (14) |
| C8 | 0.0544 (17) | 0.0427 (16) | 0.0601 (18) | 0.0088 (13) | 0.0149 (15) | 0.0219 (14) |
| C9 | 0.0457 (15) | 0.0533 (17) | 0.0540 (17) | 0.0176 (14) | 0.0182 (13) | 0.0293 (14) |
| C10 | 0.0398 (14) | 0.0521 (16) | 0.0591 (17) | 0.0218 (13) | 0.0230 (13) | 0.0375 (14) |
| C11 | 0.0455 (15) | 0.0396 (14) | 0.0556 (17) | 0.0150 (12) | 0.0194 (13) | 0.0271 (13) |
| C12 | 0.066 (2) | 0.079 (2) | 0.065 (2) | 0.0115 (18) | 0.0066 (17) | 0.0370 (19) |
| C13 | 0.0442 (15) | 0.0469 (15) | 0.0458 (15) | 0.0180 (12) | 0.0138 (12) | 0.0278 (13) |
| C14 | 0.064 (2) | 0.0559 (18) | 0.086 (2) | 0.0355 (16) | 0.0306 (17) | 0.0495 (18) |
| C15 | 0.0608 (19) | 0.0468 (16) | 0.078 (2) | 0.0229 (14) | 0.0281 (16) | 0.0424 (16) |
| C16 | 0.0502 (15) | 0.0341 (13) | 0.0380 (14) | 0.0169 (12) | 0.0157 (12) | 0.0193 (11) |
| C17 | 0.0445 (15) | 0.0343 (13) | 0.0423 (14) | 0.0150 (11) | 0.0142 (12) | 0.0210 (11) |
| C18 | 0.0527 (17) | 0.0448 (15) | 0.0610 (18) | 0.0179 (13) | 0.0187 (14) | 0.0343 (14) |
| C19 | 0.0512 (17) | 0.0516 (17) | 0.072 (2) | 0.0109 (14) | 0.0213 (15) | 0.0392 (16) |
| C20 | 0.0439 (15) | 0.0542 (17) | 0.0623 (18) | 0.0186 (13) | 0.0200 (14) | 0.0321 (15) |
| C21 | 0.0466 (16) | 0.0412 (15) | 0.073 (2) | 0.0157 (12) | 0.0196 (14) | 0.0363 (15) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| O1—C4 | 1.463 (3) | C6—C7 | 1.391 (4) |
| O1—C5 | 1.344 (3) | C6—C11 | 1.399 (3) |
| O2—C5 | 1.211 (3) | C7—C8 | 1.373 (4) |
| O3—C10 | 1.418 (3) | C7—H7A | 0.9300 |
| O3—C13 | 1.349 (3) | C8—C9 | 1.396 (4) |
| N1—C5 | 1.345 (3) | C8—H8A | 0.9300 |
| N1—C6 | 1.403 (3) | C9—C10 | 1.372 (4) |
| N1—H1A | 0.8600 | C9—C12 | 1.509 (4) |
| N3—C13 | 1.349 (3) | C10—C11 | 1.380 (4) |
| N3—C14 | 1.318 (4) | C11—H11A | 0.9300 |
| N4—C20 | 1.327 (3) | C12—H12A | 0.9600 |
| N4—C21 | 1.336 (3) | C12—H12B | 0.9600 |
| C1—C4 | 1.518 (4) | C12—H12C | 0.9600 |
| C1—H1B | 0.9600 | C14—C15 | 1.367 (4) |
| C1—H1C | 0.9600 | C14—H14A | 0.9300 |
| C1—H1D | 0.9600 | C15—C16 | 1.397 (3) |
| N2—C13 | 1.317 (3) | C15—H15A | 0.9300 |
| N2—C16 | 1.344 (3) | C16—C17 | 1.482 (4) |
| C2—C4 | 1.512 (4) | C17—C18 | 1.380 (4) |
| C2—H2B | 0.9600 | C17—C21 | 1.391 (3) |
| C2—H2C | 0.9600 | C18—C19 | 1.368 (4) |
| C2—H2D | 0.9600 | C18—H18A | 0.9300 |
| C3—C4 | 1.507 (5) | C19—C20 | 1.379 (4) |
| C3—H3B | 0.9600 | C19—H19A | 0.9300 |
| C3—H3C | 0.9600 | C20—H20A | 0.9300 |
| C3—H3D | 0.9600 | C21—H21A | 0.9300 |
| C5—O1—C4 | 122.7 (2) | C9—C8—H8A | 118.9 |

| | | | |
|---|---|---|---|
| C13—O3—C10 | 123.90 (19) | C10—C9—C8 | 116.0 (3) |
| C5—N1—C6 | 128.6 (2) | C10—C9—C12 | 121.7 (3) |
| C5—N1—H1A | 115.7 | C8—C9—C12 | 122.3 (3) |
| C6—N1—H1A | 115.7 | C9—C10—C11 | 124.2 (2) |
| C13—N2—C16 | 116.2 (2) | C9—C10—O3 | 114.6 (2) |
| C14—N3—C13 | 113.6 (2) | C11—C10—O3 | 120.9 (2) |
| C4—C1—H1B | 109.5 | C10—C11—C6 | 118.4 (2) |
| C4—C1—H1C | 109.5 | C10—C11—H11A | 120.8 |
| H1B—C1—H1C | 109.5 | C6—C11—H11A | 120.8 |
| C4—C1—H1D | 109.5 | C9—C12—H12A | 109.5 |
| H1B—C1—H1D | 109.5 | C9—C12—H12B | 109.5 |
| H1C—C1—H1D | 109.5 | H12A—C12—H12B | 109.5 |
| C4—C2—H2B | 109.5 | C9—C12—H12C | 109.5 |
| C4—C2—H2C | 109.5 | H12A—C12—H12C | 109.5 |
| H2B—C2—H2C | 109.5 | H12B—C12—H12C | 109.5 |
| C4—C2—H2D | 109.5 | N2—C13—O3 | 120.7 (2) |
| H2B—C2—H2D | 109.5 | N2—C13—N3 | 128.4 (3) |
| H2C—C2—H2D | 109.5 | O3—C13—N3 | 110.9 (2) |
| C4—C3—H3B | 109.5 | N3—C14—C15 | 124.3 (3) |
| C4—C3—H3C | 109.5 | N3—C14—H14A | 117.8 |
| H3B—C3—H3C | 109.5 | C15—C14—H14A | 117.8 |
| C4—C3—H3D | 109.5 | C14—C15—C16 | 117.1 (3) |
| H3B—C3—H3D | 109.5 | C14—C15—H15A | 121.4 |
| H3C—C3—H3D | 109.5 | C16—C15—H15A | 121.4 |
| C20—N4—C21 | 117.2 (2) | N2—C16—C15 | 120.4 (2) |
| O1—C4—C3 | 109.0 (3) | N2—C16—C17 | 116.8 (2) |
| O1—C4—C2 | 110.8 (2) | C15—C16—C17 | 122.8 (2) |
| C3—C4—C2 | 112.3 (3) | C18—C17—C21 | 117.2 (2) |
| O1—C4—C1 | 102.0 (2) | C18—C17—C16 | 122.0 (2) |
| C3—C4—C1 | 111.1 (3) | C21—C17—C16 | 120.8 (2) |
| C2—C4—C1 | 111.1 (3) | C19—C18—C17 | 119.3 (2) |
| O2—C5—O1 | 125.6 (3) | C19—C18—H18A | 120.3 |
| O2—C5—N1 | 126.3 (3) | C17—C18—H18A | 120.3 |
| O1—C5—N1 | 108.1 (2) | C18—C19—C20 | 119.4 (3) |
| C7—C6—C11 | 118.9 (2) | C18—C19—H19A | 120.3 |
| C7—C6—N1 | 117.0 (2) | C20—C19—H19A | 120.3 |
| C11—C6—N1 | 124.0 (2) | N4—C20—C19 | 122.8 (3) |
| C8—C7—C6 | 120.3 (3) | N4—C20—H20A | 118.6 |
| C8—C7—H7A | 119.8 | C19—C20—H20A | 118.6 |
| C6—C7—H7A | 119.8 | N4—C21—C17 | 124.0 (2) |
| C7—C8—C9 | 122.2 (3) | N4—C21—H21A | 118.0 |
| C7—C8—H8A | 118.9 | C17—C21—H21A | 118.0 |
| C5—O1—C4—C3 | 64.0 (3) | C16—N2—C13—O3 | 178.7 (2) |
| C5—O1—C4—C2 | −60.1 (4) | C16—N2—C13—N3 | −1.6 (4) |
| C5—O1—C4—C1 | −178.5 (3) | C10—O3—C13—N2 | −9.7 (4) |
| C4—O1—C5—O2 | 8.5 (5) | C10—O3—C13—N3 | 170.6 (2) |
| C4—O1—C5—N1 | −172.4 (2) | C14—N3—C13—N2 | 0.8 (4) |
| C6—N1—C5—O2 | 2.1 (5) | C14—N3—C13—O3 | −179.5 (2) |
| C6—N1—C5—O1 | −177.0 (2) | C13—N3—C14—C15 | 1.1 (5) |

| | | | |
|---|---|---|---|
| C5—N1—C6—C7 | −177.0 (3) | N3—C14—C15—C16 | −2.0 (5) |
| C5—N1—C6—C11 | 2.9 (4) | C13—N2—C16—C15 | 0.5 (4) |
| C11—C6—C7—C8 | −0.6 (4) | C13—N2—C16—C17 | −178.7 (2) |
| N1—C6—C7—C8 | 179.3 (3) | C14—C15—C16—N2 | 1.1 (4) |
| C6—C7—C8—C9 | 0.0 (5) | C14—C15—C16—C17 | −179.7 (3) |
| C7—C8—C9—C10 | 0.6 (4) | N2—C16—C17—C18 | −179.7 (2) |
| C7—C8—C9—C12 | −179.9 (3) | C15—C16—C17—C18 | 1.1 (4) |
| C8—C9—C10—C11 | −0.7 (4) | N2—C16—C17—C21 | 0.9 (4) |
| C12—C9—C10—C11 | 179.9 (3) | C15—C16—C17—C21 | −178.3 (3) |
| C8—C9—C10—O3 | 172.1 (2) | C21—C17—C18—C19 | −0.6 (4) |
| C12—C9—C10—O3 | −7.3 (4) | C16—C17—C18—C19 | 180.0 (2) |
| C13—O3—C10—C9 | 138.8 (3) | C17—C18—C19—C20 | −0.3 (4) |
| C13—O3—C10—C11 | −48.2 (4) | C21—N4—C20—C19 | −0.4 (4) |
| C9—C10—C11—C6 | 0.1 (4) | C18—C19—C20—N4 | 0.8 (5) |
| O3—C10—C11—C6 | −172.3 (2) | C20—N4—C21—C17 | −0.6 (4) |
| C7—C6—C11—C10 | 0.6 (4) | C18—C17—C21—N4 | 1.1 (4) |
| N1—C6—C11—C10 | −179.3 (2) | C16—C17—C21—N4 | −179.5 (3) |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1A···N4$^{i}$ | 0.86 | 2.10 | 2.944 (4) | 165 |
| C15—H15A···O2$^{ii}$ | 0.93 | 2.45 | 3.382 (4) | 177 |
| C18—H18A···O2$^{ii}$ | 0.93 | 2.39 | 3.319 (4) | 174 |
| C3—H3B···Cg1$^{i}$ | 0.96 | 2.86 | 3.560 (3) | 131 |
| C12—H12B···Cg2$^{iii}$ | 0.96 | 2.90 | 3.788 (3) | 154 |

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

**Fig. 1**

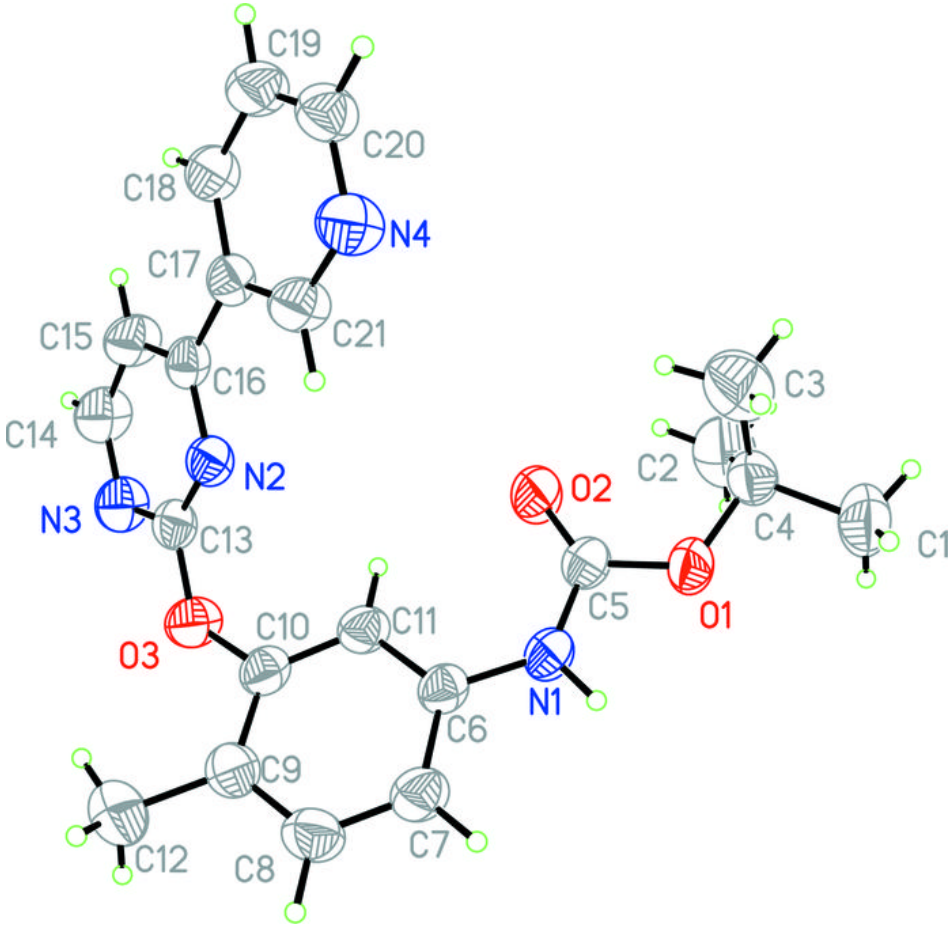
C19
C20
C18
N4
C17
C21
C15
C16
C14
N2
N3
C13
O3
C10
C11
O2
C5
O1
C3
C2
C4
C1
N1
C6
C9
C12
C8
C7

**Fig. 2**