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# *N*-(4-Methylpyridin-2-yl)-5*H*-dibenzo-[*d*,*f*][1,3]diazepine-6-carboxamide toluene hemisolvate

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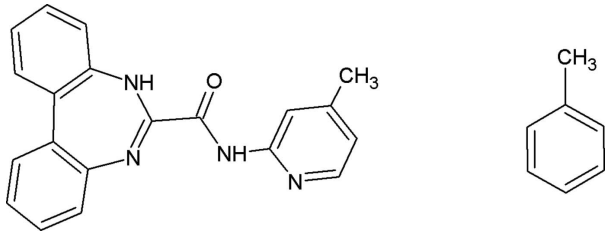
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.002 Å; disorder in solvent or counterion; $R$ factor = 0.052; $wR$ factor = 0.148; data-to-parameter ratio = 30.0.

The title compound, $C_{20}H_{16}N_4O\cdot0.5C_7H_8$, crystallizes with half a toluene molecule (disordered about a centre of inversion); the seven-membered diazepine ring adopts a twist-boat conformation. The structure features two intramolecular (N—H···O and N—H···N) and one intermolecular hydrogen bond. Additional intermolecular $\pi$–$\pi$ interactions (3.556–3.651 Å) give rise to a three-dimensional network in the crystal structure.

## Related literature

For literature on the medicinal and psychotherapeutic uses of similar compounds, see Evans *et al.* (1988); Sternbach (1978). For comparative bond distances, see Allen *et al.* (1987).




## Experimental

### *Crystal data*

$C_{20}H_{16}N_4O\cdot0.5C_7H_8$
$M_r$ = 374.44
Triclinic, $P\overline{1}$
$a$ = 8.9323 (2) Å
$b$ = 9.6863 (2) Å
$c$ = 12.1478 (3) Å
$\alpha$ = 77.050 (1)°
$\beta$ = 87.529 (1)°
$\gamma$ = 70.191 (1)°
$V$ = 963.08 (4) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 293 (2) K
0.29 × 0.21 × 0.15 mm

### *Data collection*

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*DENZO* and *SCALEPACK*; Otwinowski & Minor, 1997)
$T_{min}$ = 0.977, $T_{max}$ = 0.988
17061 measured reflections
8631 independent reflections
5192 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.0595

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.148
$S$ = 1.02
8631 reflections
288 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.18 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N3—H3···O1 | 0.88 (1) | 2.22 (1) | 2.677 (1) | 112 (1) |
| N3—H3···O1$^{i}$ | 0.88 (1) | 2.31 (1) | 3.108 (1) | 151 (1) |
| N2—H2···N4 | 0.87 (1) | 2.19 (1) | 2.644 (1) | 112 (1) |

Symmetry code: (i) $-x, -y+1, -z+1$.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

The authors thank the Joint X-ray Laboratory, Faculty of Chemistry, and SLAFiBS, Jagiellonian University, for making available the Nonius KappaCCD diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2316).

## References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
Evans, B. E. *et al.* (1988). *J. Med. Chem.* **31**, 2235–2246.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Nonius (2000). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Sternbach, L. H. (1978). *Prog. Drug Res.* **22**, 229–266.

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# supplementary materials

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# *N*-(4-Methylpyridin-2-yl)-5*H*-dibenzo[*d*,*f*][1,3]diazepine-6-carboxamide toluene hemisolvate

**M. Hodorowicz, K. Stadnicka, B. Trzewik and B. Zaleska**

## Comment

Benzodiazepines are one of the most important substances in medicinal chemistry, representing the prototypical 'privileged structure' (Evans *et al.* 1988). Compounds based on the diazepine skeleton are psychotherapeutic agents widely used for the treatment of anxiety and neurosis (Sternbach, 1978). The crystal structure analysis of *N*-(4-methylpyridin-2-yl)-5*H*-dibenzo[*d*,*f*][1,3] diazepine-6-carboxamide was performed to find out the influence of methyl substituent on molecular geometry and packing properties. The molecular structure of the title compound is shown in Fig. 1. The symmetrically independent part of the unit cell is composed of title compound molecule and disordered toluene molecule. The core of the diazepine molecule is composed of a seven-membered ring to which a pyridin-2-ylamino carbonyl substituent is attached and two benzene rings are fused. The puckering parameters of the seven-membered ring (atoms in N3, C2,···,C31 sequence): q2 = 0.5752, q3 = 0.0819, QT = 0.5810, φ2 = −6.33, φ3 = 10.46, θ2 = 81.89°, indicate a twisted-boat conformation with a pseudo-mirror plane ($C_S$) through the N3 atom and the centre of C41—C46 bond. From the chemical point of view there is also a pseudo 2-fold axis ($C_2$) bisecting the diazepine ring through C2 and the center of C36–C46 bond. The bond lengths and valence angles are typical and comparable with the values reported in the literature (Allen *et al.* 1987). Two N atoms are involved in hydrogen bonds: two intra- and one intermolecular (Table 1). The intermolecular N3—H3···O1 [−*x*, 1 − *y*, 1 − *z*] interaction is relatively weak due to simultaneous donor participation in intramolecular interaction (bifurcated hydrogen bond). In addition, there are two types of π–π interactions, which assist in the stabilization of the three-dimensional-packing. The benzene ring C31···C36 is strictly parallel to another one in position [1 − *x*, 1 − *y*, 1 − *z*], with interplanar spacing of 3.556 Å, the ring-centroid separation of 3.883 Å and with ring offset of 1.560 Å. The pyridine ring is nearly parallel to another benzene ring C41···C46 of the molecule at [1 − *x*, − *y*, 1 − *z*]. The interplanar spacing is 3.651 Å, the corresponding ring-centroid separation is 3.790 Å, resulting in the offset of 1.017 Å. The π–π interaction are illustrated in Fig. 2. A s it can be seen in Fig. 2, the solvent accessible cavities at 0, 1/2, 1 are filled with disordered toluene molecules which stabilized three-dimensional structure (total potential solvent area of 195.7 $Å^3$ makes 20.3% of unit cell volume).

## Experimental

Recrystallization from toluene afforded crystals suitable for X-ray measurements.

## Refinement

H atoms bonded to N atoms were located in a difference Fourier map and refined with distance restraints of N—H = 0.87 (2) Å, and with $U_{iso}$(H) = 1.2$U_{eq}$(N). All other H atom positions were also observed in difference Fourier map. Nevertheless, in the refinement procedure the hydrogen atoms were positioned geometrically and refined using a riding model (including free rotation about the C—C bond), with C—H = 0.95–0.99 Å (C—H = 0.97 Å for $CH_2$ groups, 0.96 Å for $CH_3$ groups, and 0.93 Å for aromatic CH) and with $U_{iso}$(H) = 1.5$U_{eq}$(C) for methyl groups and $U_{iso}$(H) = 1.2$U_{eq}$(C) for all other H atoms. Refinement was performed with data set combined of two independent measurements and with BASF parameter as the scale factor for second data set (BASF parameter converged to 0.94477)

## Figures

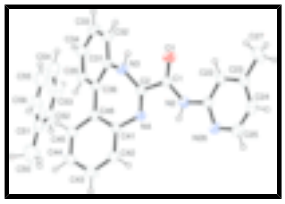

Fig. 1. *ORTEP-3* (Farrugia, 1997) drawing of the title compound with labels. Displacement ellipsoids of non-H atoms drawn at 30% probability level.

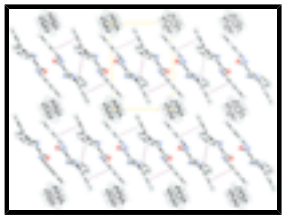

Fig. 2. *ORTEP-3* (Farrugia, 1997) drawing of the crystal packing viewed along [010] with marked π–π interactions (dashed bonds). Hydrogen atoms are omitted.

## *N*-(4-Methylpyridin-2-yl)-5*H*-dibenzo[d,f][1,3]diazepine-6- carboxamide toluene hemisolvate

*Crystal data*

| | |
|---|---|
| $C_{20}H_{16}N_4O·0.5C_7H_8$ | $Z = 2$ |
| $M_r = 374.44$ | $F_{000} = 394$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.291$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation<br>$\lambda = 0.71073$ Å |
| $a = 8.9323$ (2) Å | Cell parameters from 2469 reflections |
| $b = 9.6863$ (2) Å | $\theta = 1.0$–$27.5°$ |
| $c = 12.1478$ (3) Å | $\mu = 0.08$ mm$^{-1}$ |
| $\alpha = 77.050$ (1)° | $T = 293$ (2) K |
| $\beta = 87.529$ (1)° | Prism, orange |
| $\gamma = 70.191$ (1)° | 0.29 × 0.21 × 0.15 mm |
| $V = 963.08$ (4) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Nonius KappaCCD diffractometer | 8631 independent reflections |
| Radiation source: fine-focus sealed tube | 5192 reflections with $I > 2\sigma(I)$ |
| Monochromator: horizontally mounted graphite crystal | $R_{int} = 0.0000$ |
| Detector resolution: 9 pixels mm$^{-1}$ | $\theta_{max} = 27.4°$ |
| $T = 293(2)$ K | $\theta_{min} = 2.4°$ |
| φ and ω scans to fill asymmetric unit | $h = -11 \rightarrow 11$ |
| Absorption correction: multi-scan (DENZO and SCALEPACK; Otwinowski & Minor, 1997) | $k = -12 \rightarrow 12$ |
| $T_{min} = 0.977$, $T_{max} = 0.988$ | $l = 0 \rightarrow 15$ |
| 17061 measured reflections | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | H atoms treated by a mixture of independent and constrained refinement |
| $R[F^2 > 2\sigma(F^2)] = 0.052$ | $w = 1/[\sigma^2(F_o^2) + (0.0656P)^2 + 0.0957P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2) = 0.148$ | $(\Delta/\sigma)_{max} = 0.01$ |
| $S = 1.02$ | $\Delta\rho_{max} = 0.18$ e Å$^{-3}$ |
| 8631 reflections | $\Delta\rho_{min} = -0.18$ e Å$^{-3}$ |
| 288 parameters | Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$ |
| Primary atom site location: structure-invariant direct methods | Extinction coefficient: 0.014 (3) |
| 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 with redundant (*i.e.* not merged/unique) data set: data set combined of two independent measurements; refinement with BASF parameter as the scale factor for second data set. 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 $F^2 > 2$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) |
|---|---|---|---|---|---|
| C1 | 0.17801 (13) | 0.23113 (12) | 0.51837 (9) | 0.0458 (3) | |
| O1 | 0.07918 (10) | 0.32376 (9) | 0.56173 (8) | 0.0611 (2) | |
| C2 | 0.27718 (12) | 0.28173 (12) | 0.42283 (9) | 0.0430 (3) | |
| N3 | 0.23275 (11) | 0.43460 (10) | 0.38922 (9) | 0.0505 (3) | |
| H3 | 0.1546 (16) | 0.4821 (14) | 0.4286 (10) | 0.058 (4)* | |
| C31 | 0.34450 (12) | 0.51109 (11) | 0.36175 (9) | 0.0435 (3) | |
| C32 | 0.32397 (14) | 0.63436 (12) | 0.40814 (10) | 0.0534 (3) | |
| H32 | 0.2384 | 0.6645 | 0.4540 | 0.064* | |
| C33 | 0.42946 (16) | 0.71276 (13) | 0.38688 (11) | 0.0605 (3) | |
| H33 | 0.4140 | 0.7963 | 0.4174 | 0.073* | |
| C34 | 0.55721 (15) | 0.66709 (14) | 0.32055 (12) | 0.0603 (3) | |
| H34 | 0.6297 | 0.7184 | 0.3071 | 0.072* | |
| C35 | 0.57746 (14) | 0.54485 (13) | 0.27409 (10) | 0.0521 (3) | |

| | | | | | |
|---|---|---|---|---|---|
| H35 | 0.6645 | 0.5149 | 0.2294 | 0.062* | |
| C36 | 0.47134 (12) | 0.46415 (11) | 0.29185 (9) | 0.0427 (2) | |
| C46 | 0.49668 (12) | 0.33809 (12) | 0.23420 (9) | 0.0430 (2) | |
| C45 | 0.56872 (14) | 0.34457 (14) | 0.13001 (10) | 0.0560 (3) | |
| H45 | 0.5935 | 0.4302 | 0.0970 | 0.067* | |
| C44 | 0.60463 (17) | 0.22908 (16) | 0.07395 (12) | 0.0687 (4) | |
| H44 | 0.6513 | 0.2380 | 0.0040 | 0.082* | |
| C43 | 0.57127 (17) | 0.10084 (17) | 0.12164 (12) | 0.0702 (4) | |
| H43 | 0.5972 | 0.0214 | 0.0852 | 0.084* | |
| C42 | 0.49880 (14) | 0.09106 (14) | 0.22422 (11) | 0.0565 (3) | |
| H42 | 0.4765 | 0.0039 | 0.2565 | 0.068* | |
| C41 | 0.45811 (12) | 0.20788 (12) | 0.28090 (9) | 0.0428 (2) | |
| N4 | 0.38063 (10) | 0.17891 (10) | 0.38303 (8) | 0.0450 (2) | |
| N2 | 0.20866 (12) | 0.08255 (11) | 0.54501 (9) | 0.0513 (3) | |
| H2 | 0.2779 (16) | 0.0327 (15) | 0.5026 (12) | 0.069 (4)* | |
| C21 | 0.12759 (13) | −0.00035 (12) | 0.61882 (9) | 0.0458 (3) | |
| C22 | 0.03000 (14) | 0.05641 (14) | 0.70102 (10) | 0.0531 (3) | |
| H22 | 0.0155 | 0.1528 | 0.7103 | 0.064* | |
| C23 | −0.04599 (15) | −0.03245 (15) | 0.76942 (10) | 0.0577 (3) | |
| C24 | −0.01902 (16) | −0.17409 (15) | 0.75086 (11) | 0.0645 (4) | |
| H24 | −0.0679 | −0.2374 | 0.7946 | 0.077* | |
| C25 | 0.08051 (16) | −0.22065 (14) | 0.66741 (12) | 0.0624 (3) | |
| H25 | 0.0970 | −0.3166 | 0.6565 | 0.075* | |
| N26 | 0.15561 (12) | −0.13681 (10) | 0.60055 (8) | 0.0539 (3) | |
| C27 | −0.1531 (2) | 0.0259 (2) | 0.86000 (13) | 0.0853 (5) | |
| H27A | −0.1950 | −0.0498 | 0.8997 | 0.102* | |
| H27B | −0.2393 | 0.1146 | 0.8260 | 0.102* | |
| H27C | −0.0933 | 0.0502 | 0.9121 | 0.102* | |
| C50 | 0.0601 (10) | 0.3448 (10) | −0.1225 (8) | 0.141 (4) | 0.50 |
| H50A | 0.0559 | 0.4006 | −0.1991 | 0.169* | 0.50 |
| H50B | −0.0135 | 0.2908 | −0.1153 | 0.169* | 0.50 |
| H50C | 0.1659 | 0.2749 | −0.1032 | 0.169* | 0.50 |
| C51 | 0.0177 (5) | 0.4482 (4) | −0.0469 (2) | 0.0800 (9) | 0.50 |
| C52 | 0.1356 (3) | 0.4569 (3) | 0.0196 (3) | 0.0699 (17) | 0.50 |
| H52 | 0.2411 | 0.3975 | 0.0150 | 0.084* | 0.50 |
| C53 | 0.0958 (5) | 0.5544 (4) | 0.0928 (2) | 0.0986 (13) | 0.50 |
| H53 | 0.1747 | 0.5603 | 0.1373 | 0.118* | 0.50 |
| C54 | −0.0619 (6) | 0.6433 (4) | 0.0996 (3) | 0.118 (4) | 0.50 |
| H54 | −0.0886 | 0.7085 | 0.1486 | 0.142* | 0.50 |
| C55 | −0.1799 (3) | 0.6345 (4) | 0.0332 (4) | 0.1057 (14) | 0.50 |
| H55 | −0.2854 | 0.6940 | 0.0377 | 0.127* | 0.50 |
| C56 | −0.1400 (4) | 0.5370 (5) | −0.0401 (3) | 0.099 (3) | 0.50 |
| H56 | −0.2189 | 0.5312 | −0.0845 | 0.119* | 0.50 |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0459 (6) | 0.0426 (6) | 0.0505 (6) | −0.0172 (5) | 0.0101 (5) | −0.0112 (5) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O1 | 0.0620 (5) | 0.0468 (5) | 0.0748 (6) | −0.0191 (4) | 0.0306 (5) | −0.0182 (4) |
| C2 | 0.0421 (5) | 0.0398 (6) | 0.0486 (6) | −0.0160 (4) | 0.0072 (5) | −0.0103 (5) |
| N3 | 0.0454 (5) | 0.0385 (5) | 0.0656 (6) | −0.0132 (4) | 0.0204 (5) | −0.0124 (4) |
| C31 | 0.0451 (6) | 0.0366 (5) | 0.0464 (6) | −0.0134 (4) | 0.0077 (5) | −0.0062 (4) |
| C32 | 0.0592 (7) | 0.0416 (6) | 0.0570 (7) | −0.0142 (5) | 0.0147 (6) | −0.0128 (5) |
| C33 | 0.0734 (8) | 0.0438 (7) | 0.0698 (8) | −0.0238 (6) | 0.0090 (7) | −0.0187 (6) |
| C34 | 0.0628 (7) | 0.0495 (7) | 0.0760 (9) | −0.0293 (6) | 0.0082 (7) | −0.0139 (6) |
| C35 | 0.0500 (6) | 0.0488 (7) | 0.0588 (7) | −0.0208 (5) | 0.0128 (5) | −0.0101 (5) |
| C36 | 0.0435 (5) | 0.0381 (6) | 0.0446 (6) | −0.0137 (4) | 0.0060 (5) | −0.0063 (4) |
| C46 | 0.0387 (5) | 0.0444 (6) | 0.0449 (6) | −0.0130 (4) | 0.0064 (5) | −0.0104 (5) |
| C45 | 0.0584 (7) | 0.0615 (7) | 0.0519 (7) | −0.0253 (6) | 0.0156 (6) | −0.0150 (6) |
| C44 | 0.0777 (9) | 0.0797 (10) | 0.0563 (8) | −0.0304 (7) | 0.0272 (7) | −0.0292 (7) |
| C43 | 0.0783 (9) | 0.0711 (9) | 0.0721 (9) | −0.0259 (7) | 0.0227 (8) | −0.0404 (7) |
| C42 | 0.0594 (7) | 0.0505 (7) | 0.0644 (8) | −0.0199 (6) | 0.0138 (6) | −0.0221 (6) |
| C41 | 0.0389 (5) | 0.0421 (6) | 0.0459 (6) | −0.0114 (4) | 0.0058 (5) | −0.0112 (4) |
| N4 | 0.0454 (5) | 0.0399 (5) | 0.0496 (5) | −0.0148 (4) | 0.0107 (4) | −0.0110 (4) |
| N2 | 0.0558 (6) | 0.0414 (5) | 0.0557 (6) | −0.0171 (4) | 0.0203 (5) | −0.0110 (4) |
| C21 | 0.0452 (6) | 0.0430 (6) | 0.0466 (6) | −0.0160 (5) | 0.0030 (5) | −0.0033 (5) |
| C22 | 0.0604 (7) | 0.0534 (7) | 0.0495 (6) | −0.0248 (6) | 0.0104 (6) | −0.0121 (5) |
| C23 | 0.0575 (7) | 0.0733 (9) | 0.0446 (6) | −0.0305 (6) | 0.0065 (6) | −0.0053 (6) |
| C24 | 0.0656 (8) | 0.0658 (9) | 0.0628 (8) | −0.0354 (7) | 0.0029 (7) | 0.0049 (6) |
| C25 | 0.0651 (8) | 0.0464 (7) | 0.0745 (9) | −0.0238 (6) | 0.0006 (7) | −0.0026 (6) |
| N26 | 0.0550 (6) | 0.0419 (5) | 0.0615 (6) | −0.0163 (4) | 0.0049 (5) | −0.0058 (4) |
| C27 | 0.0911 (11) | 0.1162 (13) | 0.0609 (9) | −0.0534 (10) | 0.0294 (8) | −0.0204 (9) |
| C50 | 0.189 (11) | 0.164 (8) | 0.103 (4) | −0.106 (7) | 0.024 (5) | −0.027 (5) |
| C51 | 0.104 (3) | 0.076 (2) | 0.0657 (19) | −0.055 (2) | 0.010 (2) | 0.0105 (16) |
| C52 | 0.049 (3) | 0.080 (4) | 0.076 (2) | −0.037 (3) | −0.010 (2) | 0.018 (2) |
| C53 | 0.100 (3) | 0.114 (4) | 0.089 (3) | −0.062 (3) | −0.022 (2) | 0.008 (2) |
| C54 | 0.157 (9) | 0.081 (4) | 0.114 (6) | −0.049 (5) | 0.037 (5) | −0.010 (4) |
| C55 | 0.081 (3) | 0.105 (3) | 0.117 (4) | −0.037 (2) | 0.014 (3) | 0.011 (3) |
| C56 | 0.096 (6) | 0.114 (7) | 0.084 (3) | −0.054 (5) | −0.007 (3) | 0.015 (3) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—O1 | 1.223 (1) | N2—H2 | 0.87 (1) |
| C1—N2 | 1.335 (1) | C21—N26 | 1.327 (1) |
| C1—C2 | 1.524 (2) | C21—C22 | 1.380 (2) |
| C2—N4 | 1.278 (1) | C22—C23 | 1.385 (2) |
| C2—N3 | 1.364 (1) | C22—H22 | 0.9300 |
| N3—C31 | 1.421 (1) | C23—C24 | 1.379 (2) |
| N3—H3 | 0.88 (1) | C23—C27 | 1.506 (2) |
| C31—C32 | 1.386 (2) | C24—C25 | 1.370 (2) |
| C31—C36 | 1.397 (1) | C24—H24 | 0.9300 |
| C32—C33 | 1.381 (2) | C25—N26 | 1.340 (2) |
| C32—H32 | 0.9300 | C25—H25 | 0.9300 |
| C33—C34 | 1.373 (2) | C27—H27A | 0.9600 |
| C33—H33 | 0.9300 | C27—H27B | 0.9600 |
| C34—C35 | 1.378 (2) | C27—H27C | 0.9600 |
| C34—H34 | 0.9300 | C50—C51 | 1.455 (8) |

| | | | |
|---|---|---|---|
| C35—C36 | 1.402 (2) | C50—H50A | 0.9600 |
| C35—H35 | 0.9300 | C50—H50B | 0.9600 |
| C36—C46 | 1.488 (2) | C50—H50C | 0.9600 |
| C46—C45 | 1.393 (2) | C51—C52 | 1.3900 |
| C46—C41 | 1.410 (2) | C51—C56 | 1.3900 |
| C45—C44 | 1.377 (2) | C52—C53 | 1.3900 |
| C45—H45 | 0.9300 | C52—H52 | 0.9300 |
| C44—C43 | 1.372 (2) | C53—C54 | 1.3900 |
| C44—H44 | 0.9300 | C53—H53 | 0.9300 |
| C43—C42 | 1.378 (2) | C54—C55 | 1.3900 |
| C43—H43 | 0.9300 | C54—H54 | 0.9300 |
| C42—C41 | 1.391 (1) | C55—C56 | 1.3900 |
| C42—H42 | 0.9300 | C55—H55 | 0.9300 |
| C41—N4 | 1.414 (1) | C56—H56 | 0.9300 |
| N2—C21 | 1.410 (1) | | |
| O1—C1—N2 | 126.0 (1) | C2—N4—C41 | 123.5 (1) |
| O1—C1—C2 | 120.4 (1) | C1—N2—C21 | 128.8 (1) |
| N2—C1—C2 | 113.7 (1) | C1—N2—H2 | 114.6 (9) |
| N4—C2—N3 | 130.1 (1) | C21—N2—H2 | 115.8 (9) |
| N4—C2—C1 | 117.1 (1) | N26—C21—C22 | 124.3 (1) |
| N3—C2—C1 | 112.7 (1) | N26—C21—N2 | 112.9 (1) |
| C2—N3—C31 | 122.7 (1) | C22—C21—N2 | 122.8 (1) |
| C2—N3—H3 | 113.0 (8) | C21—C22—C23 | 118.9 (1) |
| C31—N3—H3 | 111.5 (8) | C21—C22—H22 | 120.5 |
| C32—C31—C36 | 120.8 (1) | C23—C22—H22 | 120.5 |
| C32—C31—N3 | 117.3 (1) | C24—C23—C22 | 117.4 (1) |
| C36—C31—N3 | 121.9 (1) | C24—C23—C27 | 122.3 (1) |
| C33—C32—C31 | 120.6 (1) | C22—C23—C27 | 120.3 (1) |
| C33—C32—H32 | 119.7 | C25—C24—C23 | 119.4 (1) |
| C31—C32—H32 | 119.7 | C25—C24—H24 | 120.3 |
| C34—C33—C32 | 119.8 (1) | C23—C24—H24 | 120.3 |
| C34—C33—H33 | 120.1 | N26—C25—C24 | 124.2 (1) |
| C32—C33—H33 | 120.1 | N26—C25—H25 | 117.9 |
| C33—C34—C35 | 119.6 (1) | C24—C25—H25 | 117.9 |
| C33—C34—H34 | 120.2 | C21—N26—C25 | 115.7 (1) |
| C35—C34—H34 | 120.2 | C23—C27—H27A | 109.5 |
| C34—C35—C36 | 122.3 (1) | C23—C27—H27B | 109.5 |
| C34—C35—H35 | 118.9 | H27A—C27—H27B | 109.5 |
| C36—C35—H35 | 118.9 | C23—C27—H27C | 109.5 |
| C31—C36—C35 | 116.8 (1) | H27A—C27—H27C | 109.5 |
| C31—C36—C46 | 124.0 (1) | H27B—C27—H27C | 109.5 |
| C35—C36—C46 | 119.1 (1) | C52—C51—C56 | 120.0 |
| C45—C46—C41 | 117.3 (1) | C52—C51—C50 | 119.7 (5) |
| C45—C46—C36 | 118.8 (1) | C56—C51—C50 | 120.3 (5) |
| C41—C46—C36 | 123.9 (1) | C51—C52—C53 | 120.0 |
| C44—C45—C46 | 122.5 (1) | C51—C52—H52 | 120.0 |
| C44—C45—H45 | 118.7 | C53—C52—H52 | 120.0 |
| C46—C45—H45 | 118.7 | C52—C53—C54 | 120.0 |
| C43—C44—C45 | 119.8 (1) | C52—C53—H53 | 120.0 |

| | | | |
|---|---|---|---|
| C43—C44—H44 | 120.1 | C54—C53—H53 | 120.0 |
| C45—C44—H44 | 120.1 | C55—C54—C53 | 120.0 |
| C44—C43—C42 | 119.2 (1) | C55—C54—H54 | 120.0 |
| C44—C43—H43 | 120.4 | C53—C54—H54 | 120.0 |
| C42—C43—H43 | 120.4 | C56—C55—C54 | 120.0 |
| C43—C42—C41 | 121.9 (1) | C56—C55—H55 | 120.0 |
| C43—C42—H42 | 119.1 | C54—C55—H55 | 120.0 |
| C41—C42—H42 | 119.1 | C55—C56—C51 | 120.0 |
| C42—C41—C46 | 119.2 (1) | C55—C56—H56 | 120.0 |
| C42—C41—N4 | 113.7 (1) | C51—C56—H56 | 120.0 |
| C46—C41—N4 | 127.1 (1) | | |
| O1—C1—C2—N4 | 178.5 (1) | C45—C46—C41—C42 | 2.6 (2) |
| N2—C1—C2—N4 | −2.8 (2) | C36—C46—C41—C42 | −174.7 (1) |
| O1—C1—C2—N3 | −4.9 (2) | C45—C46—C41—N4 | −177.9 (1) |
| N2—C1—C2—N3 | 173.7 (1) | C36—C46—C41—N4 | 4.9 (2) |
| N4—C2—N3—C31 | −44.4 (2) | N3—C2—N4—C41 | −9.1 (2) |
| C1—C2—N3—C31 | 139.6 (1) | C1—C2—N4—C41 | 166.7 (1) |
| C2—N3—C31—C32 | −132.8 (1) | C42—C41—N4—C2 | −149.2 (1) |
| C2—N3—C31—C36 | 46.6 (2) | C46—C41—N4—C2 | 31.2 (2) |
| C36—C31—C32—C33 | −0.5 (2) | O1—C1—N2—C21 | 6.7 (2) |
| N3—C31—C32—C33 | 178.9 (1) | C2—C1—N2—C21 | −171.9 (1) |
| C31—C32—C33—C34 | −0.9 (2) | C1—N2—C21—N26 | 160.4 (1) |
| C32—C33—C34—C35 | 1.1 (2) | C1—N2—C21—C22 | −19.7 (2) |
| C33—C34—C35—C36 | 0.1 (2) | N26—C21—C22—C23 | −0.5 (2) |
| C32—C31—C36—C35 | 1.7 (2) | N2—C21—C22—C23 | 179.6 (1) |
| N3—C31—C36—C35 | −177.7 (1) | C21—C22—C23—C24 | 0.0 (2) |
| C32—C31—C36—C46 | −176.7 (1) | C21—C22—C23—C27 | 179.9 (1) |
| N3—C31—C36—C46 | 3.9 (2) | C22—C23—C24—C25 | 0.3 (2) |
| C34—C35—C36—C31 | −1.5 (2) | C27—C23—C24—C25 | −179.7 (1) |
| C34—C35—C36—C46 | 177.0 (1) | C23—C24—C25—N26 | −0.1 (2) |
| C31—C36—C46—C45 | 148.6 (1) | C22—C21—N26—C25 | 0.7 (2) |
| C35—C36—C46—C45 | −29.8 (2) | N2—C21—N26—C25 | −179.4 (1) |
| C31—C36—C46—C41 | −34.2 (2) | C24—C25—N26—C21 | −0.4 (2) |
| C35—C36—C46—C41 | 147.5 (1) | C56—C51—C52—C53 | 0.0 |
| C41—C46—C45—C44 | −1.1 (2) | C50—C51—C52—C53 | −179.4 (5) |
| C36—C46—C45—C44 | 176.3 (1) | C51—C52—C53—C54 | 0.0 |
| C46—C45—C44—C43 | −0.9 (2) | C52—C53—C54—C55 | 0.0 |
| C45—C44—C43—C42 | 1.4 (2) | C53—C54—C55—C56 | 0.0 |
| C44—C43—C42—C41 | 0.1 (2) | C54—C55—C56—C51 | 0.0 |
| C43—C42—C41—C46 | −2.2 (2) | C52—C51—C56—C55 | 0.0 |
| C43—C42—C41—N4 | 178.2 (1) | C50—C51—C56—C55 | 179.4 (5) |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N3—H3···O1 | 0.88 (1) | 2.22 (1) | 2.677 (1) | 112 (1) |
| N3—H3···O1$^{i}$ | 0.88 (1) | 2.31 (1) | 3.108 (1) | 151 (1) |
| N2—H2···N4 | 0.87 (1) | 2.19 (1) | 2.644 (1) | 112 (1) |

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

**Fig. 1**

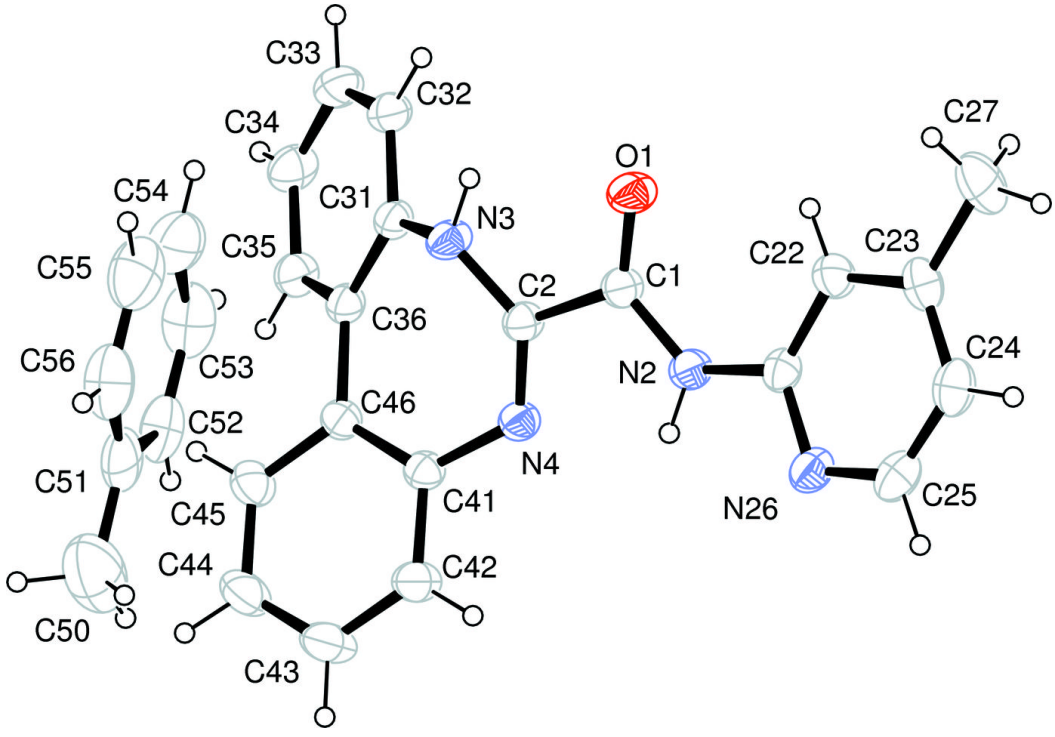
C33
C32
C34
C31
N3
O1
C27
C54
C35
C2
C1
C22
C23
C55
C36
C24
C56
C53
N2
C46
C52
N4
C51
C45
C41
N26
C25
C44
C42
C50
C43

Fig. 2