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# organic compounds

# N-{(1Z)-1-[(6-Chloropyridin-3-ylmethyl)-(ethyl)amino]-3-(3-chlorophenyl)-2-nitro-5-oxohex-1-enyl}-N-methylacetamide

## Chuan-Wen Sun,* Ying Wu and Jing Wang

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In the title compound, $C_{23}H_{26}Cl_2N_4O_4$, the dihedral angle between the mean planes of the pyridine and 3-chlorophenyl rings is 22.63 (2)°. The nitro group is in a $Z$ conformation.

## Related literature

For general background to neonicotinoid compounds and their application as insecticides, see: Tomizawa & Casida, (2000); Minamida *et al.* (1993); Kashiwada *et al.* (1996). For the synthesis, see: Zhang *et al.* (2010).
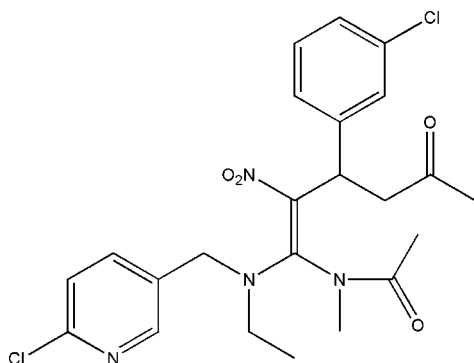
## Experimental

### Crystal data

| | |
|---|---|
| $C_{23}H_{26}Cl_2N_4O_4$ | $\gamma = 107.878\ (3)°$ |
| $M_r = 493.38$ | $V = 1204.1\ (3)$ Å$^3$ |
| Triclinic, $P\bar{1}$ | $Z = 2$ |
| $a = 7.7948\ (13)$ Å | Mo $K\alpha$ radiation |
| $b = 12.649\ (2)$ Å | $\mu = 0.31$ mm$^{-1}$ |
| $c = 13.021\ (2)$ Å | $T = 298$ K |
| $\alpha = 91.364\ (3)°$ | $0.16 \times 0.12 \times 0.10$ mm |
| $\beta = 98.765\ (2)°$ | |

### Data collection

| | |
|---|---|
| Bruker SMART APEX CCD area-detector diffractometer | 7096 measured reflections |
| | 4200 independent reflections |
| Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) | 3870 reflections with $I > 2\sigma(I)$ |
| $T_{min} = 0.943$, $T_{max} = 0.970$ | $R_{int} = 0.020$ |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)] = 0.064$ | 302 parameters |
| $wR(F^2) = 0.148$ | H-atom parameters constrained |
| $S = 1.17$ | $\Delta\rho_{max} = 0.47$ e Å$^{-3}$ |
| 4200 reflections | $\Delta\rho_{min} = -0.38$ e Å$^{-3}$ |

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; 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: *SHELXTL*.

## References

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# supporting information

# *N*-{(1*Z*)-1-[(6-Chloropyridin-3-ylmethyl)(ethyl)amino]-3-(3-chlorophenyl)-2-nitro-5-oxohex-1-enyl}-*N*-methylacetamide

**Chuan-Wen Sun, Ying Wu and Jing Wang**

### S1. Comment

Neonicotinoid insecticides have gained worldwide attention for being the fastest growing class of insecticides in modern crop protection, with wide spread use against sucking and chewing pests. Since imidacloprid (IMI) was first introduced to the market in 1991, many new neonicotinoid insecticides (NNSs) are now being sold. Nitenpyram, a chloronicotinyl derivative marketed in 1995, was characterized with a much lower toxicity against the mammals than imidacloprid (Tomizawa & Casida, 2000; Minamida *et al.*, 1993; Kashiwada *et al.*, 1996). In this paper, the title compound, (I), a new derivative, has been synthesized and characterized by X-ray diffraction.

In the title compound, $C_{23}H_{26}Cl_2N_4O_4$, there is one molecule in the asymmetric unit (Fig. 1). The dihedral angle between the mean planes of the pyridine and 3-chlorophenyl rings is 22.63 (2)°. As compared with the *(E)* configuration of the nitro group in the crystal structure of nitenpyram, the nitro group in the title compound is in the *(Z)* configuration as anticipated.

### S2. Experimental

The title compound was prepared by the literature method (Zhang *et al.*, 2010). It was obtained using volatilization of petroleum ether and ethyl acetate solution at room temperature, giving yellow crystals (yield 78.6%). Anal. calcd. for $C_{23}H_{26}Cl_2N_4O_4$ C 55.99, H 5.31, N 11.36%. found, C 55.97, H 5.32, N 11.38%.

### S3. Refinement

In (I), H atoms bonded to C and N atoms were located at their ideal positions and subsequently treated as riding modes with C–H distances of 0.93Å (aromatic), 0.97Å (methylene), 0.98Å (methine) and 0.96Å (methyl) with $U_{iso}(H) = 1.2U_{eq}$(aromatic, methylene methine C) or $1.5U_{eq}$(methyl C).
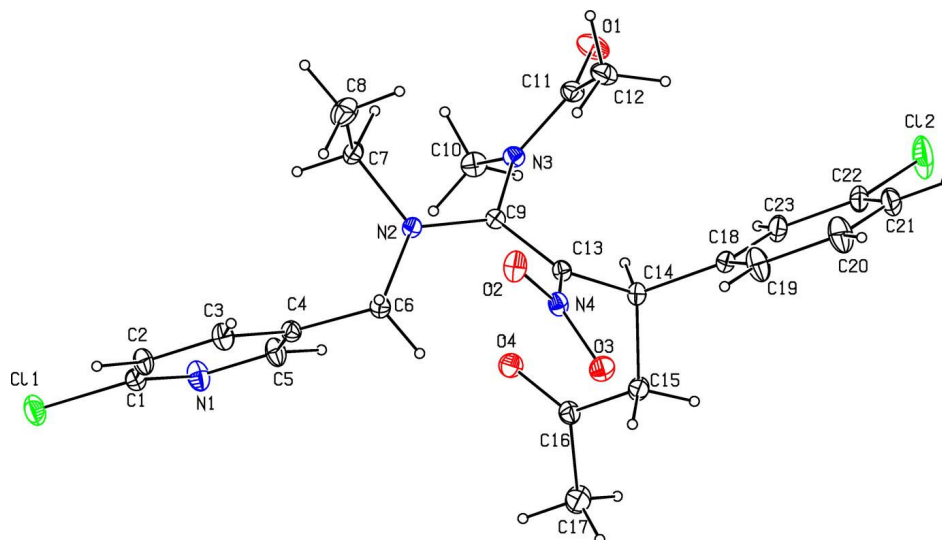
**Figure 1**

The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

## *N*-{(1*Z*)-1-[(6-Chloropyridin-3-ylmethyl)(ethyl)amino]-3-(3- chlorophenyl)-2-nitro-5-oxohex-1-enyl}-*N*-methylacetamide

*Crystal data*

| | |
|---|---|
| $C_{23}H_{26}Cl_2N_4O_4$ | $Z = 2$ |
| $M_r = 493.38$ | $F(000) = 516$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.361$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $a = 7.7948$ (13) Å | Cell parameters from 4525 reflections |
| $b = 12.649$ (2) Å | $\theta = 2.2–28.3°$ |
| $c = 13.021$ (2) Å | $\mu = 0.31$ mm$^{-1}$ |
| $\alpha = 91.364$ (3)° | $T = 298$ K |
| $\beta = 98.765$ (2)° | Block, yellow |
| $\gamma = 107.878$ (3)° | $0.16 \times 0.12 \times 0.10$ mm |
| $V = 1204.1$ (3) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Bruker SMART APEX CCD area-detector diffractometer | 7096 measured reflections |
| | 4200 independent reflections |
| Radiation source: fine-focus sealed tube | 3870 reflections with $I > 2\sigma(I)$ |
| Graphite monochromator | $R_{int} = 0.020$ |
| phi and $\omega$ scans | $\theta_{max} = 25.0°$, $\theta_{min} = 2.2°$ |
| Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) | $h = -7 \rightarrow 9$ |
| | $k = -15 \rightarrow 13$ |
| $T_{min} = 0.943$, $T_{max} = 0.970$ | $l = -15 \rightarrow 15$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | 4200 reflections |
| Least-squares matrix: full | 302 parameters |
| $R[F^2 > 2\sigma(F^2)] = 0.064$ | 0 restraints |
| $wR(F^2) = 0.148$ | Primary atom site location: structure-invariant |
| $S = 1.17$ | direct methods |

| | |
|---|---|
| Secondary atom site location: difference Fourier map | $w = 1/[\sigma^2(F_o^2) + (0.0453P)^2 + 0.9109P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| Hydrogen site location: inferred from neighbouring sites | $(\Delta/\sigma)_{max} = 0.011$ $\Delta\rho_{max} = 0.47$ e Å$^{-3}$ |
| H-atom parameters constrained | $\Delta\rho_{min} = -0.38$ e Å$^{-3}$ |

*Special details*

**Experimental**. $^1$H NMR (CDCl$_3$, 400 Hz): 8.47 (d, $J = 1.9$ Hz,1$H$, Py—H), 7.83 (dd, $J = 8.2$, 2.4 Hz, 1$H$, Py—H), 7.42 (d, $J = 8.2$ Hz, 1$H$, Py—H), 7.12 (d, $J = 10.2$ Hz, 1$H$, Ph—H), 7.01–6.91 (m, 3$H$, Ph—H), 4.27 (d, $J = 14.7$ Hz, 1$H$), 4.12 (d, $J = 7.7$ Hz, 1$H$), 4.00 (dd, $J = 10.8$, 3.8 Hz, 1$H$), 3.78 (d, $J = 14.8$ Hz, 1$H$), 3.31 (s, 3$H$, NCH$_3$), 3.09 (dd, $J = 14.4$, 7.2 Hz, 1$H$, NCH$_2$), 2.89 (d, $J = 3.9$Hz, 1$H$, NCH$_2$), 2.26 (s, 1$H$), 2.13 (s, 3$H$), 1.67 (s, 3$H$), 1.17 (t, J = 7.2 Hz, 3$H$, NCH$_2$CH$_3$). IR(KBr, cm$^{-1}$) 2945 (CH$_3$), 1710 (C=O), 1658 (C=C), 1378, 1390(NO$_2$), 1660, 1621, 1538(benzene).

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 | 0.9628 (4) | 0.8187 (2) | 0.8649 (3) | 0.0521 (7) |
| C2 | 0.9304 (4) | 0.8215 (3) | 0.7596 (2) | 0.0559 (8) |
| H2 | 1.0030 | 0.8779 | 0.7259 | 0.067* |
| C3 | 0.7857 (4) | 0.7376 (3) | 0.7052 (2) | 0.0540 (8) |
| H3 | 0.7581 | 0.7367 | 0.6331 | 0.065* |
| C4 | 0.6813 (4) | 0.6546 (2) | 0.7573 (2) | 0.0427 (6) |
| C5 | 0.7293 (4) | 0.6599 (3) | 0.8637 (2) | 0.0596 (8) |
| H5 | 0.6613 | 0.6036 | 0.8995 | 0.071* |
| C6 | 0.5256 (4) | 0.5591 (2) | 0.6992 (2) | 0.0451 (6) |
| H6A | 0.5205 | 0.5665 | 0.6249 | 0.054* |
| H6B | 0.5492 | 0.4896 | 0.7142 | 0.054* |
| C7 | 0.3021 (4) | 0.6592 (2) | 0.7287 (2) | 0.0480 (7) |
| H7A | 0.4057 | 0.7180 | 0.7664 | 0.058* |
| H7B | 0.1998 | 0.6501 | 0.7655 | 0.058* |
| C8 | 0.2535 (6) | 0.6925 (3) | 0.6212 (3) | 0.0785 (11) |
| H8A | 0.3540 | 0.7011 | 0.5843 | 0.118* |
| H8B | 0.2277 | 0.7618 | 0.6258 | 0.118* |
| H8C | 0.1475 | 0.6360 | 0.5846 | 0.118* |
| C9 | 0.2212 (3) | 0.4559 (2) | 0.73211 (18) | 0.0348 (5) |
| C10 | 0.1460 (5) | 0.4994 (3) | 0.9036 (2) | 0.0550 (8) |
| H10A | 0.0962 | 0.5579 | 0.9175 | 0.083* |
| H10B | 0.2771 | 0.5277 | 0.9188 | 0.083* |
| H10C | 0.1029 | 0.4395 | 0.9465 | 0.083* |
| C11 | −0.0970 (4) | 0.4176 (3) | 0.7564 (2) | 0.0519 (7) |
| C12 | −0.1607 (4) | 0.3915 (3) | 0.6418 (3) | 0.0650 (9) |
| H12A | −0.2290 | 0.3138 | 0.6283 | 0.098* |

| | | | |
|---|---|---|---|
| H12B | −0.0569 | 0.4085 | 0.6066 | 0.098* |
| H12C | −0.2373 | 0.4353 | 0.6169 | 0.098* |
| C13 | 0.2214 (3) | 0.3532 (2) | 0.69263 (19) | 0.0368 (6) |
| C14 | 0.1557 (4) | 0.2493 (2) | 0.7497 (2) | 0.0421 (6) |
| H14 | 0.1153 | 0.2752 | 0.8105 | 0.051* |
| C15 | 0.3084 (4) | 0.2029 (2) | 0.7966 (2) | 0.0542 (7) |
| H15A | 0.2532 | 0.1285 | 0.8171 | 0.065* |
| H15B | 0.3801 | 0.1969 | 0.7433 | 0.065* |
| C16 | 0.4344 (4) | 0.2730 (2) | 0.8897 (2) | 0.0500 (7) |
| C17 | 0.5633 (6) | 0.2214 (3) | 0.9493 (3) | 0.0795 (11) |
| H17A | 0.6664 | 0.2789 | 0.9873 | 0.119* |
| H17B | 0.6046 | 0.1792 | 0.9019 | 0.119* |
| H17C | 0.5019 | 0.1731 | 0.9972 | 0.119* |
| C18 | −0.0129 (4) | 0.1572 (2) | 0.6946 (2) | 0.0454 (6) |
| C19 | −0.0443 (5) | 0.1205 (3) | 0.5905 (3) | 0.0766 (11) |
| H19 | 0.0405 | 0.1535 | 0.5483 | 0.092* |
| C20 | −0.2016 (6) | 0.0346 (4) | 0.5483 (3) | 0.0891 (13) |
| H20 | −0.2201 | 0.0106 | 0.4782 | 0.107* |
| C21 | −0.3293 (5) | −0.0151 (3) | 0.6078 (3) | 0.0746 (11) |
| H21 | −0.4345 | −0.0724 | 0.5793 | 0.090* |
| C22 | −0.2985 (5) | 0.0214 (3) | 0.7100 (3) | 0.0661 (9) |
| C23 | −0.1431 (4) | 0.1059 (2) | 0.7539 (3) | 0.0581 (8) |
| H23 | −0.1258 | 0.1286 | 0.8242 | 0.070* |
| Cl1 | 1.14377 (13) | 0.92531 (8) | 0.93748 (8) | 0.0793 (3) |
| Cl2 | −0.45945 (18) | −0.04123 (10) | 0.78826 (12) | 0.1249 (6) |
| N1 | 0.8686 (4) | 0.7416 (2) | 0.9194 (2) | 0.0676 (8) |
| N2 | 0.3480 (3) | 0.55437 (17) | 0.72679 (16) | 0.0377 (5) |
| N3 | 0.0879 (3) | 0.45855 (18) | 0.79346 (17) | 0.0416 (5) |
| N4 | 0.2894 (3) | 0.34678 (19) | 0.59894 (18) | 0.0459 (6) |
| O1 | −0.2039 (3) | 0.4098 (2) | 0.8168 (2) | 0.0819 (8) |
| O2 | 0.2890 (3) | 0.41880 (18) | 0.53559 (15) | 0.0616 (6) |
| O3 | 0.3403 (3) | 0.26542 (19) | 0.57805 (18) | 0.0650 (6) |
| O4 | 0.4321 (3) | 0.36466 (18) | 0.91429 (17) | 0.0595 (6) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0429 (16) | 0.0456 (16) | 0.0630 (19) | 0.0094 (13) | 0.0035 (14) | 0.0074 (14) |
| C2 | 0.0512 (17) | 0.0492 (17) | 0.0596 (19) | 0.0001 (14) | 0.0176 (15) | 0.0138 (14) |
| C3 | 0.0560 (18) | 0.0566 (18) | 0.0424 (16) | 0.0040 (14) | 0.0158 (14) | 0.0045 (13) |
| C4 | 0.0409 (14) | 0.0416 (14) | 0.0454 (15) | 0.0103 (12) | 0.0126 (12) | 0.0029 (12) |
| C5 | 0.0566 (19) | 0.0544 (18) | 0.0537 (19) | −0.0035 (15) | 0.0088 (15) | 0.0142 (14) |
| C6 | 0.0449 (15) | 0.0438 (15) | 0.0470 (16) | 0.0104 (12) | 0.0166 (12) | −0.0009 (12) |
| C7 | 0.0543 (17) | 0.0365 (14) | 0.0537 (17) | 0.0151 (13) | 0.0096 (13) | 0.0014 (12) |
| C8 | 0.104 (3) | 0.062 (2) | 0.072 (2) | 0.034 (2) | 0.004 (2) | 0.0170 (18) |
| C9 | 0.0377 (13) | 0.0398 (13) | 0.0273 (12) | 0.0119 (11) | 0.0069 (10) | 0.0048 (10) |
| C10 | 0.0659 (19) | 0.0668 (19) | 0.0377 (15) | 0.0243 (16) | 0.0184 (14) | −0.0012 (13) |
| C11 | 0.0449 (16) | 0.0550 (17) | 0.0609 (19) | 0.0194 (14) | 0.0162 (14) | 0.0080 (14) |

| C12 | 0.0472 (18) | 0.080 (2) | 0.066 (2) | 0.0234 (17) | −0.0005 (15) | −0.0007 (18) |
|---|---|---|---|---|---|---|
| C13 | 0.0363 (13) | 0.0391 (14) | 0.0337 (13) | 0.0075 (11) | 0.0109 (10) | 0.0009 (10) |
| C14 | 0.0458 (15) | 0.0363 (14) | 0.0398 (14) | 0.0068 (11) | 0.0067 (12) | 0.0007 (11) |
| C15 | 0.0574 (18) | 0.0411 (15) | 0.0588 (18) | 0.0132 (13) | −0.0013 (15) | 0.0030 (13) |
| C16 | 0.0471 (16) | 0.0479 (17) | 0.0512 (17) | 0.0095 (13) | 0.0079 (13) | 0.0063 (13) |
| C17 | 0.084 (3) | 0.064 (2) | 0.079 (3) | 0.0224 (19) | −0.022 (2) | 0.0009 (18) |
| C18 | 0.0453 (15) | 0.0348 (14) | 0.0536 (17) | 0.0101 (12) | 0.0059 (13) | 0.0043 (12) |
| C19 | 0.072 (2) | 0.080 (2) | 0.052 (2) | −0.0111 (19) | 0.0038 (17) | 0.0028 (17) |
| C20 | 0.086 (3) | 0.089 (3) | 0.057 (2) | −0.010 (2) | −0.013 (2) | −0.005 (2) |
| C21 | 0.055 (2) | 0.053 (2) | 0.094 (3) | −0.0023 (16) | −0.0104 (19) | 0.0010 (19) |
| C22 | 0.0543 (19) | 0.0384 (16) | 0.098 (3) | 0.0009 (14) | 0.0198 (18) | −0.0028 (17) |
| C23 | 0.0622 (19) | 0.0373 (15) | 0.069 (2) | 0.0038 (14) | 0.0196 (16) | −0.0040 (14) |
| Cl1 | 0.0610 (5) | 0.0625 (5) | 0.0888 (7) | −0.0048 (4) | −0.0140 (5) | 0.0050 (5) |
| Cl2 | 0.1075 (9) | 0.0734 (7) | 0.1642 (13) | −0.0375 (6) | 0.0766 (9) | −0.0304 (7) |
| N1 | 0.0650 (18) | 0.0645 (17) | 0.0553 (16) | −0.0018 (14) | 0.0001 (13) | 0.0126 (13) |
| N2 | 0.0418 (12) | 0.0355 (11) | 0.0369 (11) | 0.0115 (9) | 0.0115 (9) | 0.0027 (9) |
| N3 | 0.0413 (12) | 0.0477 (13) | 0.0389 (12) | 0.0151 (10) | 0.0144 (10) | 0.0016 (10) |
| N4 | 0.0456 (13) | 0.0450 (13) | 0.0420 (13) | 0.0053 (10) | 0.0122 (10) | −0.0092 (11) |
| O1 | 0.0523 (14) | 0.118 (2) | 0.0845 (18) | 0.0287 (14) | 0.0336 (13) | 0.0132 (16) |
| O2 | 0.0865 (16) | 0.0549 (13) | 0.0370 (11) | 0.0071 (11) | 0.0221 (11) | 0.0050 (10) |
| O3 | 0.0679 (14) | 0.0622 (14) | 0.0711 (15) | 0.0237 (11) | 0.0270 (12) | −0.0149 (11) |
| O4 | 0.0589 (13) | 0.0600 (14) | 0.0574 (13) | 0.0196 (11) | 0.0041 (10) | −0.0119 (10) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—N1 | 1.321 (4) | C11—C12 | 1.494 (4) |
| C1—C2 | 1.359 (4) | C12—H12A | 0.9600 |
| C1—Cl1 | 1.751 (3) | C12—H12B | 0.9600 |
| C2—C3 | 1.373 (4) | C12—H12C | 0.9600 |
| C2—H2 | 0.9300 | C13—N4 | 1.412 (3) |
| C3—C4 | 1.379 (4) | C13—C14 | 1.513 (4) |
| C3—H3 | 0.9300 | C14—C18 | 1.527 (4) |
| C4—C5 | 1.374 (4) | C14—C15 | 1.536 (4) |
| C4—C6 | 1.510 (4) | C14—H14 | 0.9800 |
| C5—N1 | 1.344 (4) | C15—C16 | 1.508 (4) |
| C5—H5 | 0.9300 | C15—H15A | 0.9700 |
| C6—N2 | 1.466 (3) | C15—H15B | 0.9700 |
| C6—H6A | 0.9700 | C16—O4 | 1.202 (3) |
| C6—H6B | 0.9700 | C16—C17 | 1.492 (5) |
| C7—N2 | 1.476 (3) | C17—H17A | 0.9600 |
| C7—C8 | 1.498 (4) | C17—H17B | 0.9600 |
| C7—H7A | 0.9700 | C17—H17C | 0.9600 |
| C7—H7B | 0.9700 | C18—C23 | 1.380 (4) |
| C8—H8A | 0.9600 | C18—C19 | 1.383 (4) |
| C8—H8B | 0.9600 | C19—C20 | 1.391 (5) |
| C8—H8C | 0.9600 | C19—H19 | 0.9300 |
| C9—N2 | 1.343 (3) | C20—C21 | 1.364 (6) |
| C9—C13 | 1.387 (3) | C20—H20 | 0.9300 |

| | | | |
|---|---|---|---|
| C9—N3 | 1.412 (3) | C21—C22 | 1.359 (5) |
| C10—N3 | 1.467 (3) | C21—H21 | 0.9300 |
| C10—H10A | 0.9600 | C22—C23 | 1.378 (4) |
| C10—H10B | 0.9600 | C22—Cl2 | 1.749 (4) |
| C10—H10C | 0.9600 | C23—H23 | 0.9300 |
| C11—O1 | 1.215 (4) | N4—O2 | 1.244 (3) |
| C11—N3 | 1.377 (4) | N4—O3 | 1.249 (3) |
| | | | |
| N1—C1—C2 | 125.7 (3) | C9—C13—C14 | 121.2 (2) |
| N1—C1—Cl1 | 115.7 (2) | N4—C13—C14 | 119.8 (2) |
| C2—C1—Cl1 | 118.6 (2) | C13—C14—C18 | 117.0 (2) |
| C1—C2—C3 | 117.1 (3) | C13—C14—C15 | 114.0 (2) |
| C1—C2—H2 | 121.4 | C18—C14—C15 | 111.8 (2) |
| C3—C2—H2 | 121.4 | C13—C14—H14 | 104.1 |
| C2—C3—C4 | 120.2 (3) | C18—C14—H14 | 104.1 |
| C2—C3—H3 | 119.9 | C15—C14—H14 | 104.1 |
| C4—C3—H3 | 119.9 | C16—C15—C14 | 113.8 (2) |
| C5—C4—C3 | 117.3 (3) | C16—C15—H15A | 108.8 |
| C5—C4—C6 | 121.4 (2) | C14—C15—H15A | 108.8 |
| C3—C4—C6 | 121.3 (3) | C16—C15—H15B | 108.8 |
| N1—C5—C4 | 124.1 (3) | C14—C15—H15B | 108.8 |
| N1—C5—H5 | 118.0 | H15A—C15—H15B | 107.7 |
| C4—C5—H5 | 118.0 | O4—C16—C17 | 122.0 (3) |
| N2—C6—C4 | 112.9 (2) | O4—C16—C15 | 122.3 (3) |
| N2—C6—H6A | 109.0 | C17—C16—C15 | 115.7 (3) |
| C4—C6—H6A | 109.0 | C16—C17—H17A | 109.5 |
| N2—C6—H6B | 109.0 | C16—C17—H17B | 109.5 |
| C4—C6—H6B | 109.0 | H17A—C17—H17B | 109.5 |
| H6A—C6—H6B | 107.8 | C16—C17—H17C | 109.5 |
| N2—C7—C8 | 112.1 (2) | H17A—C17—H17C | 109.5 |
| N2—C7—H7A | 109.2 | H17B—C17—H17C | 109.5 |
| C8—C7—H7A | 109.2 | C23—C18—C19 | 117.6 (3) |
| N2—C7—H7B | 109.2 | C23—C18—C14 | 117.3 (3) |
| C8—C7—H7B | 109.2 | C19—C18—C14 | 125.2 (3) |
| H7A—C7—H7B | 107.9 | C18—C19—C20 | 120.6 (3) |
| C7—C8—H8A | 109.5 | C18—C19—H19 | 119.7 |
| C7—C8—H8B | 109.5 | C20—C19—H19 | 119.7 |
| H8A—C8—H8B | 109.5 | C21—C20—C19 | 121.2 (4) |
| C7—C8—H8C | 109.5 | C21—C20—H20 | 119.4 |
| H8A—C8—H8C | 109.5 | C19—C20—H20 | 119.4 |
| H8B—C8—H8C | 109.5 | C22—C21—C20 | 118.1 (3) |
| N2—C9—C13 | 126.1 (2) | C22—C21—H21 | 120.9 |
| N2—C9—N3 | 115.3 (2) | C20—C21—H21 | 120.9 |
| C13—C9—N3 | 118.3 (2) | C21—C22—C23 | 121.8 (3) |
| N3—C10—H10A | 109.5 | C21—C22—Cl2 | 119.1 (3) |
| N3—C10—H10B | 109.5 | C23—C22—Cl2 | 119.1 (3) |
| H10A—C10—H10B | 109.5 | C22—C23—C18 | 120.7 (3) |
| N3—C10—H10C | 109.5 | C22—C23—H23 | 119.6 |

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|---|---|---|---|
| H10A—C10—H10C | 109.5 | C18—C23—H23 | 119.6 |
| H10B—C10—H10C | 109.5 | C1—N1—C5 | 115.6 (3) |
| O1—C11—N3 | 119.2 (3) | C9—N2—C6 | 120.5 (2) |
| O1—C11—C12 | 121.7 (3) | C9—N2—C7 | 121.1 (2) |
| N3—C11—C12 | 119.0 (3) | C6—N2—C7 | 117.4 (2) |
| C11—C12—H12A | 109.5 | C11—N3—C9 | 122.8 (2) |
| C11—C12—H12B | 109.5 | C11—N3—C10 | 117.8 (2) |
| H12A—C12—H12B | 109.5 | C9—N3—C10 | 119.3 (2) |
| C11—C12—H12C | 109.5 | O2—N4—O3 | 120.6 (2) |
| H12A—C12—H12C | 109.5 | O2—N4—C13 | 119.8 (2) |
| H12B—C12—H12C | 109.5 | O3—N4—C13 | 119.5 (2) |
| C9—C13—N4 | 118.9 (2) | | |
| | | | |
| N1—C1—C2—C3 | 0.7 (5) | C20—C21—C22—C23 | −0.3 (6) |
| Cl1—C1—C2—C3 | −179.0 (2) | C20—C21—C22—Cl2 | −179.7 (3) |
| C1—C2—C3—C4 | −0.6 (5) | C21—C22—C23—C18 | 0.5 (5) |
| C2—C3—C4—C5 | −0.4 (5) | Cl2—C22—C23—C18 | 179.9 (3) |
| C2—C3—C4—C6 | −177.9 (3) | C19—C18—C23—C22 | −0.2 (5) |
| C3—C4—C5—N1 | 1.3 (5) | C14—C18—C23—C22 | −179.5 (3) |
| C6—C4—C5—N1 | 178.7 (3) | C2—C1—N1—C5 | 0.1 (5) |
| C5—C4—C6—N2 | 64.1 (4) | Cl1—C1—N1—C5 | 179.8 (3) |
| C3—C4—C6—N2 | −118.5 (3) | C4—C5—N1—C1 | −1.1 (5) |
| N2—C9—C13—N4 | −35.2 (4) | C13—C9—N2—C6 | −14.8 (4) |
| N3—C9—C13—N4 | 152.1 (2) | N3—C9—N2—C6 | 158.1 (2) |
| N2—C9—C13—C14 | 143.0 (3) | C13—C9—N2—C7 | 153.5 (3) |
| N3—C9—C13—C14 | −29.7 (4) | N3—C9—N2—C7 | −33.6 (3) |
| C9—C13—C14—C18 | 116.5 (3) | C4—C6—N2—C9 | −143.8 (2) |
| N4—C13—C14—C18 | −65.4 (3) | C4—C6—N2—C7 | 47.4 (3) |
| C9—C13—C14—C15 | −110.5 (3) | C8—C7—N2—C9 | −94.4 (3) |
| N4—C13—C14—C15 | 67.7 (3) | C8—C7—N2—C6 | 74.3 (3) |
| C13—C14—C15—C16 | 71.6 (3) | O1—C11—N3—C9 | 170.8 (3) |
| C18—C14—C15—C16 | −152.9 (3) | C12—C11—N3—C9 | −13.3 (4) |
| C14—C15—C16—O4 | −10.8 (4) | O1—C11—N3—C10 | −4.9 (4) |
| C14—C15—C16—C17 | 169.4 (3) | C12—C11—N3—C10 | 170.9 (3) |
| C13—C14—C18—C23 | −137.1 (3) | N2—C9—N3—C11 | 126.2 (3) |
| C15—C14—C18—C23 | 88.9 (3) | C13—C9—N3—C11 | −60.3 (3) |
| C13—C14—C18—C19 | 43.7 (4) | N2—C9—N3—C10 | −58.1 (3) |
| C15—C14—C18—C19 | −90.4 (4) | C13—C9—N3—C10 | 115.4 (3) |
| C23—C18—C19—C20 | −0.3 (6) | C9—C13—N4—O2 | −24.5 (4) |
| C14—C18—C19—C20 | 179.0 (3) | C14—C13—N4—O2 | 157.2 (2) |
| C18—C19—C20—C21 | 0.4 (7) | C9—C13—N4—O3 | 159.3 (2) |
| C19—C20—C21—C22 | −0.2 (7) | C14—C13—N4—O3 | −18.9 (4) |