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# (3,5-Di-*tert*-butyl-2-ethoxybenzylidene)-[2-(3,5-di-*tert*-butyl-1*H*-pyrazol-1-yl)-ethyl]amine

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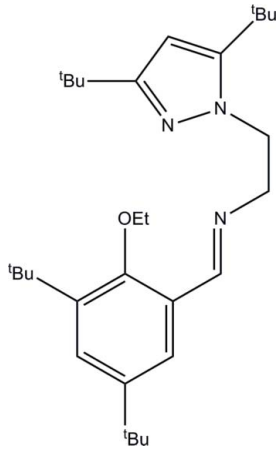
Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.046; $wR$ factor = 0.123; data-to-parameter ratio = 16.2.

The angles within the benzene ring in the title compound, $C_{30}H_{49}N_3O$, ranging from 116.34 (16) to 124.18 (16)°, reflect the presence of electron-donating and electron-withdrawing substituents. The angles at the two electron-donating *tert*-butyl substituents are smaller than 120°, at the electron-withdrawing ethoxy substituent larger than 120°, and at the imine substituent equal to 119.59 (16)°. The latter does not reflect the electron-donating nature of the imine group due to the presence of other substituents.

## Related literature

For information on (pyrazol-1-yl)imine ligands that feature phenol in cobalt and palladium complexes see: Ainooson (2010); Boltina *et al.* (2012). Geometrical parameters were checked with *Mogul* (Bruno *et al.*, 2002). Related compounds were found in the Cambridge Structural Database (Allen, 2002).




## Experimental

### *Crystal data*

$C_{30}H_{49}N_3O$
$M_r$ = 467.72
Triclinic, $P\overline{1}$
$a$ = 10.9220 (3) Å
$b$ = 11.6071 (4) Å
$c$ = 12.0283 (4) Å
$\alpha$ = 78.452 (2)°
$\beta$ = 82.775 (2)°
$\gamma$ = 79.146 (2)°
$V$ = 1461.11 (8) Å$^3$
$Z$ = 2
Cu $K\alpha$ radiation
$\mu$ = 0.49 mm$^{-1}$
$T$ = 100 K
0.12 × 0.10 × 0.09 mm

### *Data collection*

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)
$T_{min}$ = 0.944, $T_{max}$ = 0.958
25868 measured reflections
5197 independent reflections
3560 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.058

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.123
$S$ = 1.00
5197 reflections
320 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.19 e Å$^{-3}$

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL* and *FCF_filter* (Guzei, 2007); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXTL*, *publCIF* (Westrip, 2010) and *modiCIFer* (Guzei, 2007).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2447).

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# supplementary materials

*Acta Cryst.* (2012). E**68**, o2515–o2516 [doi:10.1107/S160053681203231X]

## (3,5-Di-*tert*-butyl-2-ethoxybenzylidene)[2-(3,5-di-*tert*-butyl-1*H*-pyrazol-1-yl)ethyl]amine

**Lara C. Spencer, Ilia A. Guzei, Michael K. Ainooson and James Darkwa**

### Comment

(Pyrazol-1-yl)imine ligands that feature phenol have recently been used as ligands in preparing cobalt (Ainooson, 2010) and palladium (Boltina *et al*., 2012) complexes, but the phenol proton reacts with cobalt and palladium to form an undesirable HCl by-product. To avoid the formation of HCl in such reactions, we have studied compounds where the phenol is replaced with an alkyl or aryl group. One example of such a potential ligand precursor is the ethoxy derivative, **I**, the title compound, reported herein.

The *Mogul* check of **I** confirmed that the geometrical parameters are typical except for the C12—N2—N1 angle and the C12—N2—C7 angle (Bruno *et al.*, 2002). A search of the Cambridge Structural Database (CSD; Allen, 2002) yielded 45 related compounds that have the pyrazole—C—C—N—C—benzene backbone. In these related compounds the angle comparable to the C12—N2—N1 angle in **I** had an average of 118 (6)° and a range of 101.62 to 123.99°. The 115.52 (14)° value for the C12—N2—N1 angle in **I** is within the range and within the standard deviation of the average for the related compounds, and thus should not be considered atypical. For the 45 related compounds the comparable C7—N2—C12 angle had an average of 126 (5)° with a range of values from 103.56 to 133.10°. The value for **I** of 132.37 (15)° is within the range of values for the related compounds.

The angles of the C15···C20 benzene ring range from 116.34 (16) to 124.18 (16)° deviating from the ideal 120° angle due to the presence of electron donating and electron withdrawing substituents. The two *tert*-butyl groups on atoms C17 and C19 are electron donating and the angles at these two carbon atoms in the benzene ring are expectedly smaller than the ideal 120° at 117.23 (17) and 116.34 (16)°, respectively. The ethoxy group at C20 is electron withdrawing and thus the angle at its *ipso* carbon atom is expected to exceed 120°, and indeed the angle measures 121.38 (17)°. The imine group at C15 is expected to be an electron donor with its *ipso* angle spanning less than 120°. This expectation is supported by a CSD search of monosubstituted benzene rings bearing an imine group: in 234 crystals the angle of interest averaged over 304 entries is 118.9 (9)°. In the case of **I**, however, the ring angle at C15 is very close to 120 at 119.59 (16)°. This is likely due to the presence of other substituents.

### Experimental

A mixture of 3,5-di-tertiarybutyl-2-ethoxybenzaldehyde (0.40 g, 1.50 mmol), 2-(3,5-di-tertiarybutylpyrazol-1-yl)ethyl-amine hydrochloride (0.39 g, 1.70 mmol) and excess anhydrous magnesium sulfate (0.40 g, 3.30 mmol) in ethanol (20 ml) was refluxed for 4 h. The yellow filtrate obtained after filtration was evaporated to a yellow oil, which was re-dissolved in dichloromethane (20 ml) and layered hexane (10 ml) and kept at 269 K for 3 days, to afford light yellow crystals. Yield: 0.67 g (95%). $^1$H ($CDCl_3$) $\delta$: 1.23 (s, 9H, $^t$Bu); 1.28 (s, 9H, $^t$Bu); 1.35 (s, 9H, $^t$Bu); 1.36 (s, 9H, $^t$Bu); 1.47 (t, 3H, $^3J_{HH}$= 6.9 Hz, $CH_3CH_2O$); 3.74 (q, 2H, $^3J_{HH}$= 6.9 Hz $^2J_{HH}$ = 6.9 Hz, $OCH_2CH_3$); 4.14 (t, 2H, $^3J_{HH}$ = 5.7 Hz, $CH_2$); 4.47 (t, 2H, $^2J_{HH}$ = 6.3 Hz, $CH_2$); 5.71 (s, 1H, pz-H); 7.37 (d, 1H, $^4J_{HH}$= 2.7 Hz, Ar—H); 7.69 (d, 1H, $^4J_{HH}$ = 2.4 Hz, Ar—

H); 8.42 (s, 1H, CH=N); $^{13}C\{^{1}H\}$ ($CDCl_3$) $\delta$: 15.3; 30.5; 30.6; 30.9; 31.2; 31.4; 31.9; 34.6; 35.1; 50.7; 61.7; 72.3; 98.9; 122.5; 126.8; 128.9; 141.9; 145.6; 151.6; 156.8; 159.9; 160.4. IR (Diamond ATR, $cm^{-1}$): 1631$\upsilon$(CH=N), 1320 $\upsilon$(C—O). HRMS (ESI) (m/$z$) [$M^+$+$H^+$]: Anal. Calcd. for $C_{30}H_{50}N_3O$: 468.3954. Found: 468.3976.

### Refinement

All H-atoms were placed in idealized locations and refined as riding with appropriate thermal displacement coefficients: $U_{iso}$(H) = 1.2 times $U_{eq}$(bearing atom) for C($sp^2$)-H and C($sp^3$)-2H hydrogen atoms and $U_{iso}$(H) = 1.5 times $U_{eq}$(bearing atom) for C($sp^3$)-3H hydrogen atoms. Default effective $X$—H distances for $T$ = 100 K were used: C($sp^2$)-H = 0.95, C($sp^3$)-2H = 0.99, C($sp^3$)-3H = 0.98 Å.

### Computing details

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT* (Bruker, 2007); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008) and FCF_filter (Guzei, 2007); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008, *publCIF* (Westrip, 2010) and modiCIFer (Guzei, 2007).

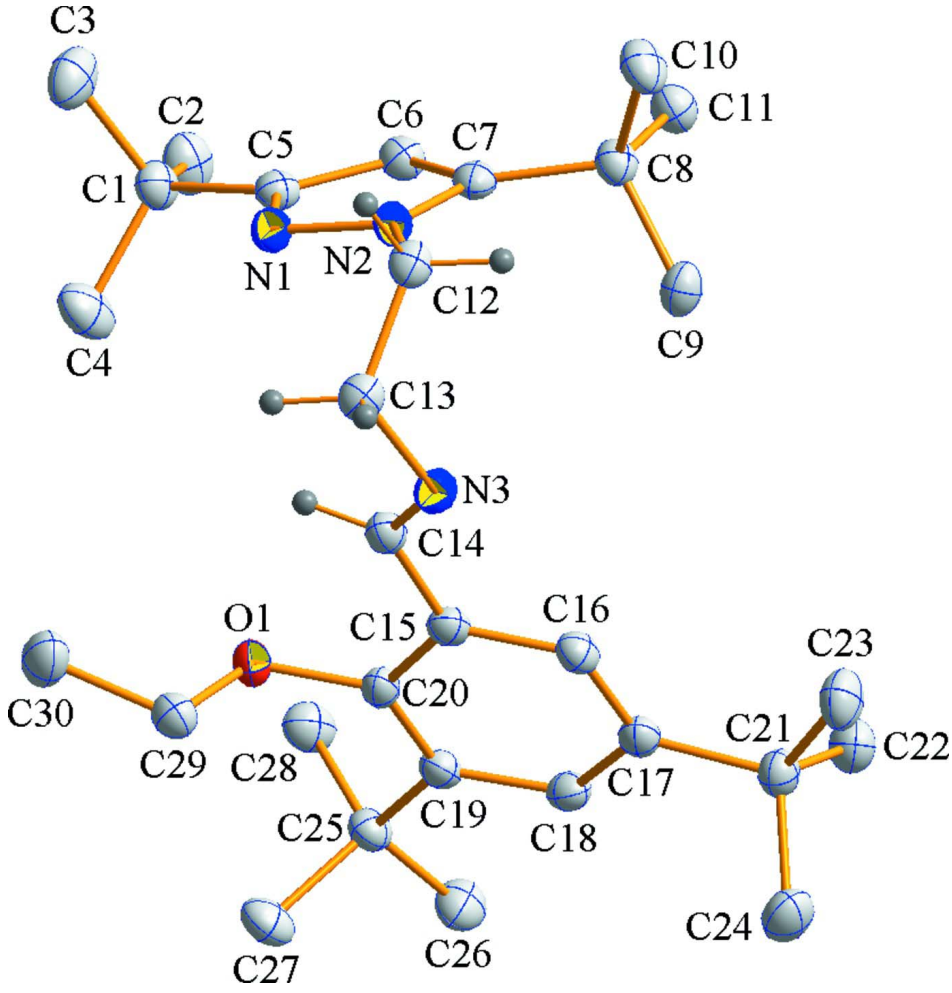


**Figure 1**

Molecular structure of **I** (Brandenburg, 1999). The thermal ellipsoids are shown at 50% probability level. The hydrogen atoms on C12, C13 and C14 are shown to clarify the location of the imine double bond. All other hydrogen atoms are not shown.

**(3,5-Di-*tert*-butyl-2-ethoxybenzylidene)[2-(3,5-di-*tert*- butyl-1*H*-pyrazol-1-yl)ethyl]amine**

*Crystal data*

$C_{30}H_{49}N_3O$
$M_r = 467.72$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 10.9220\ (3)$ Å
$b = 11.6071\ (4)$ Å
$c = 12.0283\ (4)$ Å
$\alpha = 78.452\ (2)°$
$\beta = 82.775\ (2)°$
$\gamma = 79.146\ (2)°$
$V = 1461.11\ (8)$ Å$^3$
$Z = 2$
$F(000) = 516$
$D_x = 1.063$ Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 3414 reflections
$\theta = 3.8–67.9°$
$\mu = 0.49$ mm$^{-1}$
$T = 100$ K
Block, colourless
0.12 × 0.10 × 0.09 mm

*Data collection*

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
0.50° $\omega$ and 0.5 ° $\varphi$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)
$T_{min}$ = 0.944, $T_{max}$ = 0.958
25868 measured reflections
5197 independent reflections
3560 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.058
$\theta_{max}$ = 69.4°, $\theta_{min}$ = 3.8°
$h$ = −13→13
$k$ = −13→13
$l$ = −14→14

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.123
$S$ = 1.00
5197 reflections
320 parameters
0 restraints
0 constraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.068P)^2 + 0.050P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.19 e Å$^{-3}$

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.34819 (11) | 0.66011 (10) | 0.25603 (10) | 0.0235 (3) |
| N1 | 0.28108 (14) | 0.73254 (13) | −0.14877 (12) | 0.0232 (4) |
| N2 | 0.15493 (14) | 0.73534 (13) | −0.14402 (12) | 0.0209 (3) |
| N3 | 0.11031 (14) | 0.56315 (13) | 0.07218 (12) | 0.0234 (4) |
| C1 | 0.42582 (17) | 0.87382 (17) | −0.16003 (15) | 0.0251 (4) |
| C2 | 0.41947 (19) | 1.00119 (18) | −0.14135 (18) | 0.0349 (5) |
| H2A | 0.5045 | 1.0180 | −0.1440 | 0.052* |
| H2B | 0.3749 | 1.0100 | −0.0668 | 0.052* |
| H2C | 0.3749 | 1.0573 | −0.2012 | 0.052* |
| C3 | 0.49426 (19) | 0.8607 (2) | −0.27671 (17) | 0.0383 (5) |
| H3A | 0.4992 | 0.7788 | −0.2886 | 0.057* |
| H3B | 0.5790 | 0.8786 | −0.2803 | 0.057* |
| H3C | 0.4485 | 0.9162 | −0.3362 | 0.057* |
| C4 | 0.4974 (2) | 0.78700 (19) | −0.06747 (19) | 0.0385 (5) |
| H4A | 0.5025 | 0.7051 | −0.0795 | 0.058* |
| H4B | 0.4536 | 0.7952 | 0.0075 | 0.058* |
| H4C | 0.5821 | 0.8051 | −0.0711 | 0.058* |
| C5 | 0.29479 (17) | 0.84524 (16) | −0.15391 (14) | 0.0215 (4) |
| C6 | 0.17783 (17) | 0.91988 (16) | −0.15335 (14) | 0.0231 (4) |
| H6 | 0.1627 | 1.0038 | −0.1566 | 0.028* |
| C7 | 0.08916 (17) | 0.84814 (16) | −0.14709 (14) | 0.0213 (4) |
| C8 | −0.05270 (17) | 0.88116 (16) | −0.14342 (15) | 0.0240 (4) |
| C9 | −0.11551 (18) | 0.82093 (17) | −0.03081 (16) | 0.0282 (4) |
| H9A | −0.0978 | 0.7342 | −0.0259 | 0.042* |
| H9B | −0.2062 | 0.8483 | −0.0280 | 0.042* |
| H9C | −0.0826 | 0.8419 | 0.0333 | 0.042* |

| | | | | |
|---|---|---|---|---|
| C10 | −0.10238 (19) | 0.84830 (18) | −0.24532 (16) | 0.0323 (5) |
| H10A | −0.0657 | 0.8911 | −0.3166 | 0.048* |
| H10B | −0.1937 | 0.8707 | −0.2408 | 0.048* |
| H10C | −0.0793 | 0.7622 | −0.2434 | 0.048* |
| C11 | −0.08922 (18) | 1.01676 (16) | −0.15143 (16) | 0.0284 (4) |
| H11A | −0.0619 | 1.0402 | −0.0859 | 0.043* |
| H11B | −0.1803 | 1.0394 | −0.1516 | 0.043* |
| H11C | −0.0487 | 1.0573 | −0.2220 | 0.043* |
| C12 | 0.11597 (17) | 0.62036 (16) | −0.13713 (15) | 0.0236 (4) |
| H12A | 0.1468 | 0.5897 | −0.2084 | 0.028* |
| H12B | 0.0234 | 0.6312 | −0.1297 | 0.028* |
| C13 | 0.16583 (18) | 0.52953 (16) | −0.03632 (15) | 0.0237 (4) |
| H13A | 0.1469 | 0.4504 | −0.0402 | 0.028* |
| H13B | 0.2579 | 0.5231 | −0.0407 | 0.028* |
| C14 | 0.17833 (17) | 0.60915 (15) | 0.12313 (15) | 0.0224 (4) |
| H14 | 0.2602 | 0.6181 | 0.0892 | 0.027* |
| C15 | 0.13623 (17) | 0.64936 (15) | 0.23260 (14) | 0.0213 (4) |
| C16 | 0.00919 (17) | 0.66223 (15) | 0.27274 (15) | 0.0224 (4) |
| H16 | −0.0489 | 0.6422 | 0.2303 | 0.027* |
| C17 | −0.03316 (17) | 0.70369 (15) | 0.37326 (14) | 0.0219 (4) |
| C18 | 0.05555 (17) | 0.73581 (16) | 0.43085 (15) | 0.0225 (4) |
| H18 | 0.0270 | 0.7671 | 0.4985 | 0.027* |
| C19 | 0.18307 (17) | 0.72475 (15) | 0.39523 (14) | 0.0217 (4) |
| C20 | 0.22212 (16) | 0.67702 (15) | 0.29574 (14) | 0.0209 (4) |
| C21 | −0.17007 (17) | 0.71489 (16) | 0.42364 (15) | 0.0251 (4) |
| C22 | −0.22034 (19) | 0.84395 (17) | 0.44064 (17) | 0.0315 (5) |
| H22A | −0.1704 | 0.8656 | 0.4931 | 0.047* |
| H22B | −0.3079 | 0.8503 | 0.4725 | 0.047* |
| H22C | −0.2147 | 0.8982 | 0.3671 | 0.047* |
| C23 | −0.25348 (19) | 0.68147 (19) | 0.34618 (18) | 0.0347 (5) |
| H23A | −0.2492 | 0.7341 | 0.2717 | 0.052* |
| H23B | −0.3401 | 0.6903 | 0.3807 | 0.052* |
| H23C | −0.2245 | 0.5985 | 0.3366 | 0.052* |
| C24 | −0.1774 (2) | 0.63108 (19) | 0.53977 (17) | 0.0358 (5) |
| H24A | −0.1479 | 0.5485 | 0.5294 | 0.054* |
| H24B | −0.2643 | 0.6393 | 0.5737 | 0.054* |
| H24C | −0.1247 | 0.6520 | 0.5903 | 0.054* |
| C25 | 0.27425 (17) | 0.76568 (16) | 0.46157 (15) | 0.0237 (4) |
| C26 | 0.20450 (19) | 0.82364 (19) | 0.56031 (16) | 0.0319 (5) |
| H26A | 0.1424 | 0.8919 | 0.5307 | 0.048* |
| H26B | 0.2643 | 0.8509 | 0.5993 | 0.048* |
| H26C | 0.1622 | 0.7652 | 0.6142 | 0.048* |
| C27 | 0.36982 (19) | 0.65946 (17) | 0.51248 (17) | 0.0314 (5) |
| H27A | 0.3256 | 0.5969 | 0.5571 | 0.047* |
| H27B | 0.4197 | 0.6857 | 0.5620 | 0.047* |
| H27C | 0.4251 | 0.6280 | 0.4508 | 0.047* |
| C28 | 0.34370 (19) | 0.85899 (17) | 0.38318 (16) | 0.0305 (5) |
| H28A | 0.3907 | 0.8244 | 0.3193 | 0.046* |
| H28B | 0.4017 | 0.8834 | 0.4263 | 0.046* |

| | | | | |
|---|---|---|---|---|
| H28C | 0.2830 | 0.9287 | 0.3539 | 0.046* |
| C29 | 0.40841 (17) | 0.53699 (16) | 0.26496 (16) | 0.0256 (4) |
| H29A | 0.3571 | 0.4911 | 0.2341 | 0.031* |
| H29B | 0.4186 | 0.5006 | 0.3457 | 0.031* |
| C30 | 0.53345 (19) | 0.53526 (18) | 0.19799 (18) | 0.0356 (5) |
| H30A | 0.5220 | 0.5677 | 0.1175 | 0.053* |
| H30B | 0.5784 | 0.4530 | 0.2055 | 0.053* |
| H30C | 0.5820 | 0.5840 | 0.2270 | 0.053* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0171 (7) | 0.0243 (7) | 0.0286 (7) | −0.0024 (6) | −0.0019 (5) | −0.0048 (5) |
| N1 | 0.0190 (8) | 0.0283 (9) | 0.0239 (8) | −0.0067 (7) | −0.0030 (6) | −0.0052 (6) |
| N2 | 0.0178 (8) | 0.0230 (8) | 0.0237 (8) | −0.0061 (7) | −0.0033 (6) | −0.0049 (6) |
| N3 | 0.0226 (9) | 0.0247 (8) | 0.0239 (8) | −0.0065 (7) | −0.0013 (6) | −0.0050 (6) |
| C1 | 0.0209 (11) | 0.0286 (10) | 0.0284 (10) | −0.0074 (9) | −0.0053 (8) | −0.0059 (8) |
| C2 | 0.0281 (12) | 0.0329 (11) | 0.0478 (13) | −0.0120 (10) | −0.0061 (9) | −0.0089 (9) |
| C3 | 0.0272 (12) | 0.0542 (14) | 0.0397 (12) | −0.0167 (11) | 0.0002 (9) | −0.0158 (10) |
| C4 | 0.0327 (13) | 0.0399 (12) | 0.0457 (13) | −0.0115 (11) | −0.0180 (10) | −0.0003 (10) |
| C5 | 0.0230 (11) | 0.0235 (9) | 0.0196 (9) | −0.0067 (9) | −0.0042 (7) | −0.0036 (7) |
| C6 | 0.0243 (11) | 0.0229 (9) | 0.0235 (10) | −0.0040 (9) | −0.0041 (7) | −0.0064 (7) |
| C7 | 0.0225 (10) | 0.0256 (9) | 0.0166 (9) | −0.0042 (9) | −0.0030 (7) | −0.0048 (7) |
| C8 | 0.0211 (10) | 0.0274 (10) | 0.0254 (10) | −0.0043 (9) | −0.0047 (8) | −0.0074 (8) |
| C9 | 0.0223 (11) | 0.0288 (10) | 0.0341 (11) | −0.0068 (9) | 0.0022 (8) | −0.0081 (8) |
| C10 | 0.0255 (11) | 0.0399 (12) | 0.0344 (11) | −0.0020 (10) | −0.0108 (9) | −0.0121 (9) |
| C11 | 0.0254 (11) | 0.0284 (10) | 0.0309 (10) | −0.0029 (9) | −0.0051 (8) | −0.0045 (8) |
| C12 | 0.0224 (10) | 0.0267 (10) | 0.0249 (9) | −0.0072 (9) | −0.0026 (7) | −0.0092 (8) |
| C13 | 0.0247 (11) | 0.0240 (9) | 0.0248 (10) | −0.0074 (9) | −0.0015 (8) | −0.0072 (7) |
| C14 | 0.0200 (10) | 0.0236 (9) | 0.0231 (9) | −0.0043 (8) | −0.0034 (7) | −0.0019 (7) |
| C15 | 0.0229 (10) | 0.0201 (9) | 0.0203 (9) | −0.0046 (8) | −0.0031 (7) | −0.0009 (7) |
| C16 | 0.0213 (10) | 0.0234 (9) | 0.0233 (9) | −0.0047 (8) | −0.0059 (7) | −0.0032 (7) |
| C17 | 0.0208 (10) | 0.0204 (9) | 0.0229 (9) | −0.0034 (8) | −0.0020 (7) | −0.0005 (7) |
| C18 | 0.0255 (11) | 0.0231 (9) | 0.0180 (9) | −0.0029 (8) | −0.0011 (7) | −0.0030 (7) |
| C19 | 0.0219 (10) | 0.0213 (9) | 0.0209 (9) | −0.0030 (8) | −0.0052 (7) | −0.0001 (7) |
| C20 | 0.0170 (10) | 0.0210 (9) | 0.0230 (9) | −0.0033 (8) | −0.0014 (7) | 0.0000 (7) |
| C21 | 0.0203 (10) | 0.0274 (10) | 0.0279 (10) | −0.0056 (9) | −0.0012 (8) | −0.0048 (8) |
| C22 | 0.0263 (11) | 0.0330 (11) | 0.0345 (11) | −0.0031 (9) | −0.0006 (8) | −0.0080 (9) |
| C23 | 0.0217 (11) | 0.0430 (12) | 0.0424 (12) | −0.0084 (10) | −0.0014 (9) | −0.0127 (10) |
| C24 | 0.0320 (12) | 0.0361 (11) | 0.0368 (12) | −0.0103 (10) | 0.0040 (9) | −0.0008 (9) |
| C25 | 0.0225 (11) | 0.0268 (10) | 0.0225 (9) | −0.0040 (9) | −0.0065 (7) | −0.0037 (7) |
| C26 | 0.0294 (12) | 0.0404 (12) | 0.0300 (11) | −0.0088 (10) | −0.0072 (8) | −0.0104 (9) |
| C27 | 0.0307 (12) | 0.0326 (11) | 0.0325 (11) | −0.0057 (10) | −0.0142 (9) | −0.0020 (8) |
| C28 | 0.0311 (12) | 0.0323 (11) | 0.0315 (11) | −0.0115 (10) | −0.0079 (9) | −0.0048 (8) |
| C29 | 0.0249 (11) | 0.0248 (10) | 0.0269 (10) | −0.0020 (9) | −0.0043 (8) | −0.0050 (8) |
| C30 | 0.0296 (12) | 0.0330 (11) | 0.0402 (12) | −0.0002 (10) | 0.0028 (9) | −0.0052 (9) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C20 | 1.390 (2) | C14—C15 | 1.475 (2) |
| O1—C29 | 1.445 (2) | C14—H14 | 0.9500 |
| N1—C5 | 1.333 (2) | C15—C20 | 1.396 (2) |
| N1—N2 | 1.3663 (19) | C15—C16 | 1.401 (2) |
| N2—C7 | 1.365 (2) | C16—C17 | 1.384 (2) |
| N2—C12 | 1.459 (2) | C16—H16 | 0.9500 |
| N3—C14 | 1.268 (2) | C17—C18 | 1.402 (2) |
| N3—C13 | 1.463 (2) | C17—C21 | 1.533 (2) |
| C1—C5 | 1.519 (2) | C18—C19 | 1.394 (2) |
| C1—C3 | 1.527 (3) | C18—H18 | 0.9500 |
| C1—C2 | 1.527 (3) | C19—C20 | 1.403 (2) |
| C1—C4 | 1.530 (3) | C19—C25 | 1.544 (2) |
| C2—H2A | 0.9800 | C21—C23 | 1.530 (3) |
| C2—H2B | 0.9800 | C21—C24 | 1.537 (3) |
| C2—H2C | 0.9800 | C21—C22 | 1.540 (3) |
| C3—H3A | 0.9800 | C22—H22A | 0.9800 |
| C3—H3B | 0.9800 | C22—H22B | 0.9800 |
| C3—H3C | 0.9800 | C22—H22C | 0.9800 |
| C4—H4A | 0.9800 | C23—H23A | 0.9800 |
| C4—H4B | 0.9800 | C23—H23B | 0.9800 |
| C4—H4C | 0.9800 | C23—H23C | 0.9800 |
| C5—C6 | 1.402 (3) | C24—H24A | 0.9800 |
| C6—C7 | 1.377 (2) | C24—H24B | 0.9800 |
| C6—H6 | 0.9500 | C24—H24C | 0.9800 |
| C7—C8 | 1.522 (3) | C25—C26 | 1.531 (3) |
| C8—C11 | 1.536 (3) | C25—C28 | 1.536 (3) |
| C8—C9 | 1.537 (3) | C25—C27 | 1.536 (3) |
| C8—C10 | 1.541 (2) | C26—H26A | 0.9800 |
| C9—H9A | 0.9800 | C26—H26B | 0.9800 |
| C9—H9B | 0.9800 | C26—H26C | 0.9800 |
| C9—H9C | 0.9800 | C27—H27A | 0.9800 |
| C10—H10A | 0.9800 | C27—H27B | 0.9800 |
| C10—H10B | 0.9800 | C27—H27C | 0.9800 |
| C10—H10C | 0.9800 | C28—H28A | 0.9800 |
| C11—H11A | 0.9800 | C28—H28B | 0.9800 |
| C11—H11B | 0.9800 | C28—H28C | 0.9800 |
| C11—H11C | 0.9800 | C29—C30 | 1.495 (3) |
| C12—C13 | 1.522 (2) | C29—H29A | 0.9900 |
| C12—H12A | 0.9900 | C29—H29B | 0.9900 |
| C12—H12B | 0.9900 | C30—H30A | 0.9800 |
| C13—H13A | 0.9900 | C30—H30B | 0.9800 |
| C13—H13B | 0.9900 | C30—H30C | 0.9800 |
| | | | |
| C20—O1—C29 | 115.07 (13) | C20—C15—C16 | 119.59 (16) |
| C5—N1—N2 | 105.26 (14) | C20—C15—C14 | 120.33 (16) |
| C7—N2—N1 | 112.10 (14) | C16—C15—C14 | 120.07 (16) |
| C7—N2—C12 | 132.37 (15) | C17—C16—C15 | 121.13 (16) |
| N1—N2—C12 | 115.52 (14) | C17—C16—H16 | 119.4 |

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| C14—N3—C13 | 116.03 (15) | C15—C16—H16 | 119.4 |
| C5—C1—C3 | 109.60 (15) | C16—C17—C18 | 117.23 (17) |
| C5—C1—C2 | 110.26 (16) | C16—C17—C21 | 123.37 (16) |
| C3—C1—C2 | 109.32 (16) | C18—C17—C21 | 119.40 (16) |
| C5—C1—C4 | 109.16 (15) | C19—C18—C17 | 124.18 (16) |
| C3—C1—C4 | 109.32 (17) | C19—C18—H18 | 117.9 |
| C2—C1—C4 | 109.16 (16) | C17—C18—H18 | 117.9 |
| C1—C2—H2A | 109.5 | C18—C19—C20 | 116.34 (16) |
| C1—C2—H2B | 109.5 | C18—C19—C25 | 121.02 (16) |
| H2A—C2—H2B | 109.5 | C20—C19—C25 | 122.64 (16) |
| C1—C2—H2C | 109.5 | O1—C20—C15 | 118.59 (16) |
| H2A—C2—H2C | 109.5 | O1—C20—C19 | 120.00 (15) |
| H2B—C2—H2C | 109.5 | C15—C20—C19 | 121.38 (17) |
| C1—C3—H3A | 109.5 | C23—C21—C17 | 111.74 (15) |
| C1—C3—H3B | 109.5 | C23—C21—C24 | 108.76 (16) |
| H3A—C3—H3B | 109.5 | C17—C21—C24 | 108.86 (15) |
| C1—C3—H3C | 109.5 | C23—C21—C22 | 108.38 (16) |
| H3A—C3—H3C | 109.5 | C17—C21—C22 | 110.09 (15) |
| H3B—C3—H3C | 109.5 | C24—C21—C22 | 108.96 (16) |
| C1—C4—H4A | 109.5 | C21—C22—H22A | 109.5 |
| C1—C4—H4B | 109.5 | C21—C22—H22B | 109.5 |
| H4A—C4—H4B | 109.5 | H22A—C22—H22B | 109.5 |
| C1—C4—H4C | 109.5 | C21—C22—H22C | 109.5 |
| H4A—C4—H4C | 109.5 | H22A—C22—H22C | 109.5 |
| H4B—C4—H4C | 109.5 | H22B—C22—H22C | 109.5 |
| N1—C5—C6 | 110.44 (15) | C21—C23—H23A | 109.5 |
| N1—C5—C1 | 118.91 (16) | C21—C23—H23B | 109.5 |
| C6—C5—C1 | 130.64 (16) | H23A—C23—H23B | 109.5 |
| C7—C6—C5 | 106.78 (16) | C21—C23—H23C | 109.5 |
| C7—C6—H6 | 126.6 | H23A—C23—H23C | 109.5 |
| C5—C6—H6 | 126.6 | H23B—C23—H23C | 109.5 |
| N2—C7—C6 | 105.42 (15) | C21—C24—H24A | 109.5 |
| N2—C7—C8 | 124.98 (15) | C21—C24—H24B | 109.5 |
| C6—C7—C8 | 129.60 (16) | H24A—C24—H24B | 109.5 |
| C7—C8—C11 | 108.78 (15) | C21—C24—H24C | 109.5 |
| C7—C8—C9 | 111.03 (15) | H24A—C24—H24C | 109.5 |
| C11—C8—C9 | 107.95 (15) | H24B—C24—H24C | 109.5 |
| C7—C8—C10 | 111.28 (15) | C26—C25—C28 | 107.19 (16) |
| C11—C8—C10 | 107.43 (15) | C26—C25—C27 | 107.37 (15) |
| C9—C8—C10 | 110.24 (15) | C28—C25—C27 | 109.42 (16) |
| C8—C9—H9A | 109.5 | C26—C25—C19 | 111.32 (15) |
| C8—C9—H9B | 109.5 | C28—C25—C19 | 110.49 (14) |
| H9A—C9—H9B | 109.5 | C27—C25—C19 | 110.93 (15) |
| C8—C9—H9C | 109.5 | C25—C26—H26A | 109.5 |
| H9A—C9—H9C | 109.5 | C25—C26—H26B | 109.5 |
| H9B—C9—H9C | 109.5 | H26A—C26—H26B | 109.5 |
| C8—C10—H10A | 109.5 | C25—C26—H26C | 109.5 |
| C8—C10—H10B | 109.5 | H26A—C26—H26C | 109.5 |
| H10A—C10—H10B | 109.5 | H26B—C26—H26C | 109.5 |

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|---|---|---|---|
| C8—C10—H10C | 109.5 | C25—C27—H27A | 109.5 |
| H10A—C10—H10C | 109.5 | C25—C27—H27B | 109.5 |
| H10B—C10—H10C | 109.5 | H27A—C27—H27B | 109.5 |
| C8—C11—H11A | 109.5 | C25—C27—H27C | 109.5 |
| C8—C11—H11B | 109.5 | H27A—C27—H27C | 109.5 |
| H11A—C11—H11B | 109.5 | H27B—C27—H27C | 109.5 |
| C8—C11—H11C | 109.5 | C25—C28—H28A | 109.5 |
| H11A—C11—H11C | 109.5 | C25—C28—H28B | 109.5 |
| H11B—C11—H11C | 109.5 | H28A—C28—H28B | 109.5 |
| N2—C12—C13 | 111.74 (14) | C25—C28—H28C | 109.5 |
| N2—C12—H12A | 109.3 | H28A—C28—H28C | 109.5 |
| C13—C12—H12A | 109.3 | H28B—C28—H28C | 109.5 |
| N2—C12—H12B | 109.3 | O1—C29—C30 | 107.58 (15) |
| C13—C12—H12B | 109.3 | O1—C29—H29A | 110.2 |
| H12A—C12—H12B | 107.9 | C30—C29—H29A | 110.2 |
| N3—C13—C12 | 111.54 (15) | O1—C29—H29B | 110.2 |
| N3—C13—H13A | 109.3 | C30—C29—H29B | 110.2 |
| C12—C13—H13A | 109.3 | H29A—C29—H29B | 108.5 |
| N3—C13—H13B | 109.3 | C29—C30—H30A | 109.5 |
| C12—C13—H13B | 109.3 | C29—C30—H30B | 109.5 |
| H13A—C13—H13B | 108.0 | H30A—C30—H30B | 109.5 |
| N3—C14—C15 | 123.02 (17) | C29—C30—H30C | 109.5 |
| N3—C14—H14 | 118.5 | H30A—C30—H30C | 109.5 |
| C15—C14—H14 | 118.5 | H30B—C30—H30C | 109.5 |

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| C5—N1—N2—C7 | 0.53 (18) | C20—C15—C16—C17 | 1.0 (3) |
| C5—N1—N2—C12 | −179.65 (14) | C14—C15—C16—C17 | −177.78 (16) |
| N2—N1—C5—C6 | −0.46 (18) | C15—C16—C17—C18 | 2.0 (2) |
| N2—N1—C5—C1 | 179.95 (14) | C15—C16—C17—C21 | −177.51 (16) |
| C3—C1—C5—N1 | 70.9 (2) | C16—C17—C18—C19 | −2.2 (3) |
| C2—C1—C5—N1 | −168.71 (16) | C21—C17—C18—C19 | 177.33 (16) |
| C4—C1—C5—N1 | −48.8 (2) | C17—C18—C19—C20 | −0.7 (3) |
| C3—C1—C5—C6 | −108.6 (2) | C17—C18—C19—C25 | 178.16 (16) |
| C2—C1—C5—C6 | 11.8 (3) | C29—O1—C20—C15 | −71.21 (19) |
| C4—C1—C5—C6 | 131.7 (2) | C29—O1—C20—C19 | 110.92 (17) |
| N1—C5—C6—C7 | 0.2 (2) | C16—C15—C20—O1 | 178.06 (15) |
| C1—C5—C6—C7 | 179.77 (17) | C14—C15—C20—O1 | −3.2 (2) |
| N1—N2—C7—C6 | −0.38 (19) | C16—C15—C20—C19 | −4.1 (3) |
| C12—N2—C7—C6 | 179.83 (16) | C14—C15—C20—C19 | 174.68 (16) |
| N1—N2—C7—C8 | 179.90 (14) | C18—C19—C20—O1 | −178.33 (15) |
| C12—N2—C7—C8 | 0.1 (3) | C25—C19—C20—O1 | 2.9 (2) |
| C5—C6—C7—N2 | 0.09 (19) | C18—C19—C20—C15 | 3.9 (2) |
| C5—C6—C7—C8 | 179.79 (16) | C25—C19—C20—C15 | −174.95 (16) |
| N2—C7—C8—C11 | −178.72 (16) | C16—C17—C21—C23 | −3.2 (2) |
| C6—C7—C8—C11 | 1.6 (2) | C18—C17—C21—C23 | 177.30 (16) |
| N2—C7—C8—C9 | 62.6 (2) | C16—C17—C21—C24 | 116.99 (19) |
| C6—C7—C8—C9 | −117.0 (2) | C18—C17—C21—C24 | −62.6 (2) |
| N2—C7—C8—C10 | −60.6 (2) | C16—C17—C21—C22 | −123.64 (18) |
| C6—C7—C8—C10 | 119.8 (2) | C18—C17—C21—C22 | 56.8 (2) |

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|---|---|---|---|
| C7—N2—C12—C13 | −124.46 (19) | C18—C19—C25—C26 | −3.6 (2) |
| N1—N2—C12—C13 | 55.76 (19) | C20—C19—C25—C26 | 175.12 (16) |
| C14—N3—C13—C12 | −102.04 (18) | C18—C19—C25—C28 | −122.60 (18) |
| N2—C12—C13—N3 | 66.26 (19) | C20—C19—C25—C28 | 56.1 (2) |
| C13—N3—C14—C15 | 179.25 (16) | C18—C19—C25—C27 | 115.87 (19) |
| N3—C14—C15—C20 | 167.22 (17) | C20—C19—C25—C27 | −65.4 (2) |
| N3—C14—C15—C16 | −14.0 (3) | C20—O1—C29—C30 | 168.72 (15) |