



Crystal structure of unsymmetrical α -diimine palladium(II) complex cis -[ArN=C(Me)–(Et)C=NAr]PdCl₂ [Ar = 2,6-(iPr)₂C₆H₃]

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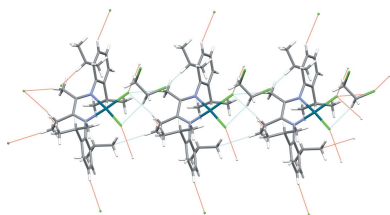
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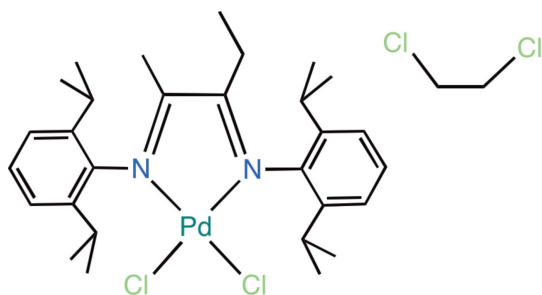
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The unsymmetrical α -diimine ligand N -{2-[2,6-bis(propan-2-yl)phenylimino]pentan-3-ylidene}-2,6-bis(propan-2-yl)aniline, [ArN=C(Me)–(Et)C=NAr] [Ar = 2,6-(iPr)₂C₆H₃], (I), and the corresponding palladium complex, cis -(N -{2-[2,6-bis(propan-2-yl)phenylimino]pentan-3-ylidene}-2,6-bis(propan-2-yl)aniline)-dichloridopalladium(II) 1,2-dichloroethane monosolvate, [PdCl₂(C₂₉H₄₂N₂)]·C₂H₄Cl₂ or cis [PdCl₂{I}], (II), have been synthesized and characterized. The crystal and molecular structure of the palladium(II) complex have been established by single-crystal X-ray diffraction. The compound crystallized along with a 1,2-dichloroethane solvent of crystallization. The coordination plane of the Pd^{II} atom shows a slight tetrahedral distortion from square-planar, as indicated by the dihedral angle between the PdCl₂ and PdN₂ planes of 4.19 (8)°. The chelate ring is folded along the N···N vector by 7.1 (1)°.

1. Chemical context

α -Diimines (or) 1,4-diaza-1,3-butadienes (DAD) are one of the most versatile classes of chelating nitrogen-donor ligands, and are well known to stabilize several transition metal complexes at various oxidation levels (Bart *et al.*, 2005; Greene *et al.*, 2014). Nickel and palladium complexes of α -diimines are reported to be effective catalysts for various olefin polymerization and co-polymerization reactions (Ittel *et al.*, 2000). Furthermore, the polymer properties, topology and stability of these catalysts can be tuned by altering the steric and electronic properties of the α -diimine ligands (Gates *et al.*, 2000). These observations have motivated the synthesis of several nickel and palladium complexes with α -diimine ligands containing various substituents at the imine nitrogen atom (Nakamura *et al.*, 2009). α -Diimine ligands may be conveniently prepared by condensation reactions between alkyl or aryl amine with 1,2-diketones. Most of the reported α -diimine ligands possess molecular C₂ symmetry, while very few unsymmetrical α -diimine ligands, obtained by varying the substituents on the nitrogen atom, have been reported (Jeon & Kim, 2008). We report herein the synthesis and spectroscopic characterization of the unsymmetrical α -diimine ligand [ArN=C(Et)–(Me)C=NAr], (I), [Ar = 2,6-i(Pr)₂C₆H₃] and the corresponding palladium complex cis -[PdCl₂{I}] (II), where the α -diimine ligand backbone contains methyl and ethyl substituents. The crystal structure of compound (II) has been established using single-crystal X-ray diffraction.





2. Structural commentary

The molecular structure of Pd^{II} complex (II), is presented in Fig. 1. Compound (II) crystallized along with a solvent molecule of 1,2-dichloroethane, which is disordered over the two crystallographic positions. The molecular structure of (II) reveals the chelation of the α -diimine ligand to the palladium(II) atom. The Pd1–N1 and Pd1–N2 distances are 2.0280 (19) and 2.0200 (18) Å, respectively, and are in the typical range for palladium α -diimine complexes (Zou & Chen, 2016). The C1–C2 bond length is 1.492 (3) Å, which is slightly shorter than a standard C–C bond length (1.54 Å; Chandrasekaran *et al.*, 2014), and similarly minimal elongation of the C1–N1 and C2–N2 bonds confirms the slight delocalization of the double bonds. As expected, the palladium(II) atom is in a distorted square-planar geometry, with an N2–Pd1–N1 angle of 79.01 (8)°. The coordination plane shows a slight tetrahedral distortion from square-planar, as indicated by the dihedral angle between the Cl1–Pd1–Cl2 and N1–Pd1–N2 planes of 4.19 (8)°. The chelate ring is folded along the N1···N2 vector by 7.1 (1)°. The aryl substituents at N1 and N2 are nearly perpendicular to the metal–ligand plane, subtending dihedral angles of 81.82 (2)° (C6–C11 aryl ring) and 86.74 (2)° (C18–C23 aryl ring). The aryl substituents in square-planar α -diimine complexes are anticipated to lie perpendicular to the metal–ligand plane due to steric repulsion.

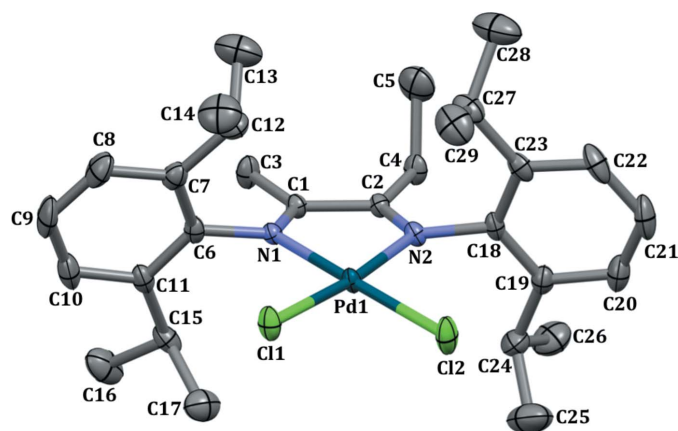


Figure 1
Perspective view of palladium complex (II) with displacement ellipsoids drawn at the 50% probability level. All H atoms and solvent molecule have been omitted for clarity.

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

| <i>D</i> –H··· <i>A</i> | <i>D</i> –H | H··· <i>A</i> | <i>D</i> ··· <i>A</i> | <i>D</i> –H··· <i>A</i> |
|-----------------------------|-------------|---------------|-----------------------|-------------------------|
| C3–H3B···Cl1 ⁱ | 0.98 | 2.67 | 3.604 (3) | 160 |
| C4–H4A···Cl1 ⁱ | 0.99 | 2.79 | 3.763 (3) | 166 |
| C15–H15···Cl4 ⁱ | 1.00 | 2.80 | 3.586 (3) | 136 |
| C21–H21···Cl1 ⁱⁱ | 0.95 | 2.74 | 3.633 (3) | 156 |

Symmetry codes: (i) $x + 1, y, z$; (ii) $-x + 1, y - \frac{1}{2}, -z + \frac{3}{2}$.

3. Supramolecular features

In the crystal lattice, the components are linked through weak C–H···Cl hydrogen-bonding interactions between the complex and solvent molecule 1,2-dichloroethane (Table 1, Fig. 2).

4. Database survey

A search of the Cambridge Structure Database (Version 5.38 with updates Nov 2016; Groom *et al.*, 2016) confirmed that the Pd^{II} complex *cis*-[ArN=C(Me)–(Et)C=NAr]PdCl₂ (Ar = 2,6-(iPr)₂C₆H₃) containing unsymmetrical α -diimine ligands has not previously been structurally characterized. However, the crystal structures of several Pd^{II} complexes containing symmetrical α -diimine ligands (IJONIE, Cope-Eatough *et al.*, 2003; FEGVOD, Coventry *et al.*, 2004; EBEXAK, Tempel *et al.*, 2000; APOFOC, Tian *et al.*, 2016; TABSOH, Chang *et al.*, 2016) have been reported. In all of these complexes, the Pd^{II} atom exhibits a slightly distorted square-planar geometry.

5. Synthesis and crystallization

Synthesis of [ArN=C(Me)–(Et)C=NAr] [Ar = 2,6-(iPr)₂C₆H₃] (I). A 100 mL round-bottom flask containing a magnetic bar was charged with 2,3-pentanedione (1 mL, 0.96 g, 9.6 mmol) and 2,6-disopropylaniline (4.0 mL, 3.76 g, 21.2 mmol). Over this, 50 mL of MeOH was added followed by a few drops of formic acid. The reaction mixture was heated to 343 K for 12 h. It was then cooled to room temperature and the solvent removed under reduced pressure. The resulting yellow pasty solid was dissolved in 15 mL of pentane and

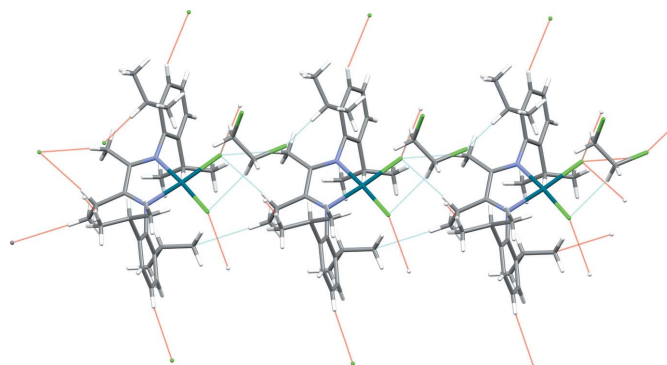


Figure 2
Hydrogen-bonding interactions in the crystal lattice.

Table 2
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | [PdCl ₂ (C ₂₉ H ₄₂ N ₂)]·C ₂ H ₄ Cl ₂ |
| <i>M_r</i> | 694.90 |
| Crystal system, space group | Monoclinic, <i>P</i> ₂ ₁ / <i>c</i> |
| Temperature (K) | 150 |
| <i>a</i> , <i>b</i> , <i>c</i> (Å) | 8.7203 (12), 20.124 (3), 19.526 (3) |
| β (°) | 100.405 (2) |
| <i>V</i> (Å ³) | 3370.2 (8) |
| <i>Z</i> | 4 |
| Radiation type | Mo <i>K</i> α |
| μ (mm ⁻¹) | 0.89 |
| Crystal size (mm) | 0.12 × 0.07 × 0.06 |
| Data collection | |
| Diffractometer | Bruker SMART APEX CCD |
| Absorption correction | Multi-scan (<i>SADABS</i> ; Bruker, 2013) |
| <i>T_{min}</i> , <i>T_{max}</i> | 0.75, 0.95 |
| No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections | 61138, 8905, 7064 |
| <i>R_{int}</i> | 0.066 |
| (<i>sin</i> θ / λ) _{max} (Å ⁻¹) | 0.684 |
| Refinement | |
| <i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i> | 0.036, 0.084, 1.02 |
| No. of reflections | 8905 |
| No. of parameters | 366 |
| No. of restraints | 3 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³) | 1.05, -0.72 |

Computer programs: *APEX2* and *SAINT* (Bruker, 2013), *SHELXT* (Sheldrick, 2015a), *SHELXL2013* (Sheldrick, 2015b), *Mercury* (Macrae *et al.*, 2006) and *SHELXTL* (Sheldrick, 2008).

stored at 248 K for 3 d, forming a yellow precipitate, which was isolated by filtration and then dried under vacuum, to afford the product as a yellow solid. Yield: 90% (3.63 g). ¹H NMR (CDCl₃): 1.08 (*t*, *J* = 7.8 Hz, 3H, CH₂CH₃), 1.15 (*d*, *J* = 6.8 Hz, 6H, iPr-CH₃), 1.20 (*d*, *J* = 6.8 Hz, 6H, iPr-CH₃), 1.39 (*d*, *J* = 6.7 Hz, 6H, iPr-CH₃), 1.46 (*d*, *J* = 6.7 Hz, 6H, iPr-CH₃), 2.05 (*s*, 3H, CH₃), 2.43 (*q*, *J* = 7.6 Hz, 2H, CH₂CH₃), 2.93 (*sep*, *J* = 6.7 Hz, 2H, iPr-CH), 3.05 (*sep*, *J* = 6.8 Hz, 2H, iPr-CH), 7.08–7.26 (*m*, 6H, Ar-H). IR (cm⁻¹): 2957 (*m*), 2926 (*w*), 2868 (*w*), 1631 (*m*), 1458 (*w*), 1433 (*w*), 1362 (*m*), 1323 (*w*), 1254 (*w*), 1183 (*m*), 1123 (*m*), 1056 (*w*), 934 (*w*), 792 (*m*), 761 (*s*), 688 (*w*). Analysis calculated for C₂₉H₄₂N₂: C, 83.20; H, 10.11; N, 6.69. Found: C, 83.35; H, 10.07; N, 6.72.

Growing X-ray quality crystals of the ligand by slow evaporation from various solvents such as hexane, diethyl ether, dichloromethane and toluene was unsuccessful.

Synthesis of *cis*[PdCl₂(I**)] (**II**).** A dichloromethane (10 mL) solution of [Pd(COD)Cl₂] (0.10 g, 0.35 mmol) was added dropwise to 5 mL dichloromethane solution of (**I**) (0.15 g, 0.35 mmol) at room temperature. The reaction mixture was stirred for 4 h to give a clear yellow solution. The solvent was removed under reduced pressure, and the resulting yellow solid was washed with 3 × 5 mL of pentane and dried *in vacuo*, affording a yellow powder as the product. Yield: 85% (0.17 g). ¹H NMR (CDCl₃): 1.08 (*t*, *J* = 7.8 Hz, 3H, CH₂CH₃), 1.20 (*d*, *J* = 6.7 Hz, 12H, iPr-CH₃), 1.52 (*d*, *J* = 6.7 Hz, 12H, iPr-CH₃), 2.05 (*s*, 3H, CH₃), 2.43 (*q*, *J* = 7.8 Hz, CH₂CH₃), 2.75 (*sep*, *J* = 6.8 Hz, 2H, iPr-CH), 2.93 (*sep*, *J* = 6.6 Hz, 2H, iPr-CH), 7.08–

7.24 (*m*, 6H, Ar-H). IR (cm⁻¹): 3031 (*w*), 2989 (*w*), 1523 (*m*), 1478 (*m*), 1448 (*w*), 1419 (*m*), 1341 (*s*), 1310 (*w*), 1247 (*w*), 1177 (*w*), 1088 (*m*), 994 (*s*), 906 (*m*), 865 (*s*), 823 (*m*), 791 (*s*), 767 (*m*). Analysis calculated for C₂₉H₄₂N₂PdCl₂: C, 58.44; H, 7.10; N, 4.70. Found: C, 58.86; H, 7.02; N, 4.94.

X-ray quality crystals of compound (**II**) were obtained by vapor diffusion of pentane over 1,2-dichloroethane solution.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. H-atoms attached to carbon were placed in calculated positions (C–H = 0.95–1.00 Å). All were included as riding contributions with isotropic displacement parameters 1.2–1.5 times those of the parent atoms. The 1,2-dichloroethane solvent molecule is disordered over two resolved sites in an 0.8596 (15):0.1404 (15) ratio. The minor component was refined with restraints that its geometry approximate that of the major component.

Acknowledgements

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supporting information

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Crystal structure of unsymmetrical α -diimine palladium(II) complex
***cis*-[ArN=C(Me)–(Et)C=NAr]PdCl₂ [Ar = 2,6-(iPr)₂C₆H₃]**

Shravan Kumar Ellandula, Cosmos Opoku Amoako, Joel T. Mague and Perumalreddy Chandrasekaran

Computing details

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT* (Bruker, 2013); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2015b); molecular graphics: *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

cis-(*N*-[2-[2,6-bis(propan-2-yl)phenylimino]pentan-3-ylidene]-2,6-bis(propan-2-yl)aniline)dichloridopalladium(II) 1,2-dichloroethane monosolvate

Crystal data

[PdCl₂(C₂₉H₄₂N₂)]·C₂H₄Cl₂
M_r = 694.90
 Monoclinic, *P2₁/c*
a = 8.7203 (12) Å
b = 20.124 (3) Å
c = 19.526 (3) Å
 β = 100.405 (2)°
V = 3370.2 (8) Å³
Z = 4

F(000) = 1440
D_x = 1.370 Mg m⁻³
 Mo *K* α radiation, λ = 0.71073 Å
 Cell parameters from 9868 reflections
 θ = 2.3–29.0°
 μ = 0.89 mm⁻¹
T = 150 K
 Block, orange
 0.12 × 0.07 × 0.06 mm

Data collection

Bruker SMART APEX CCD
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 Detector resolution: 8.3660 pixels mm⁻¹
 φ and ω scans
 Absorption correction: multi-scan
 (*SADABS*; Bruker, 2013)
T_{min} = 0.75, *T_{max}* = 0.95

61138 measured reflections
 8905 independent reflections
 7064 reflections with *I* > 2 σ (*I*)
R_{int} = 0.066
 θ_{\max} = 29.1°, θ_{\min} = 2.0°
h = -11→11
k = -27→27
l = -26→26

Refinement

Refinement on *F*²
 Least-squares matrix: full
R[*F*² > 2 σ (*F*²)] = 0.036
wR(*F*²) = 0.084
S = 1.02
 8905 reflections

366 parameters
 3 restraints
 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

$$w = 1/[\sigma^2(F_o^2) + (0.0262P)^2 + 4.3833P]$$

where $P = (F_o^2 + 2F_c^2)/3$

$$(\Delta/\sigma)_{\max} = 0.001$$

$$\Delta\rho_{\max} = 1.05 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.72 \text{ e } \text{\AA}^{-3}$$

Special details

Experimental. The diffraction data were obtained from 3 sets of 400 frames, each of width 0.5° in ω , collected at $\varphi = 0.00, 90.00$ and 180.00° and 2 sets of 800 frames, each of width 0.45° in φ , collected at $\omega = -30.00$ and 210.00° . The scan time was 15 sec/frame.

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 $F^2 > 2\text{sigma}(F^2)$ 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. H-atoms attached to carbon were placed in calculated positions (C—H = 0.95 - 1.00 Å). All were included as riding contributions with isotropic displacement parameters 1.2 - 1.5 times those of the attached atoms. The dichloroethane solvent molecule is disordered over two resolved sites in an 86:14 ratio. The minor component was refined with restraints that its geometry approximate that of the major component.

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

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|-----|-------------|--------------|--------------|----------------------------------|-----------|
| Pd1 | 0.58521 (2) | 0.59694 (2) | 0.75141 (2) | 0.01564 (5) | |
| Cl1 | 0.37729 (6) | 0.66529 (3) | 0.75421 (4) | 0.02564 (14) | |
| Cl2 | 0.41985 (7) | 0.51846 (3) | 0.69488 (4) | 0.03261 (16) | |
| N1 | 0.7481 (2) | 0.66321 (9) | 0.79623 (10) | 0.0149 (4) | |
| N2 | 0.7819 (2) | 0.54249 (9) | 0.75778 (10) | 0.0161 (4) | |
| C1 | 0.8916 (3) | 0.64435 (11) | 0.79990 (12) | 0.0169 (4) | |
| C2 | 0.9110 (3) | 0.57412 (11) | 0.77849 (12) | 0.0164 (4) | |
| C3 | 1.0301 (3) | 0.68623 (12) | 0.82792 (15) | 0.0255 (5) | |
| H3A | 0.9973 | 0.7326 | 0.8309 | 0.038* | |
| H3B | 1.1064 | 0.6832 | 0.7969 | 0.038* | |
| H3C | 1.0774 | 0.6704 | 0.8744 | 0.038* | |
| C4 | 1.0697 (3) | 0.54369 (12) | 0.78581 (13) | 0.0219 (5) | |
| H4A | 1.1408 | 0.5757 | 0.7690 | 0.026* | |
| H4B | 1.0635 | 0.5036 | 0.7560 | 0.026* | |
| C5 | 1.1381 (4) | 0.52428 (17) | 0.86098 (18) | 0.0447 (8) | |
| H5A | 1.1389 | 0.5632 | 0.8912 | 0.067* | |
| H5B | 1.2449 | 0.5081 | 0.8634 | 0.067* | |
| H5C | 1.0742 | 0.4892 | 0.8764 | 0.067* | |
| C6 | 0.7112 (2) | 0.72682 (11) | 0.82331 (13) | 0.0185 (5) | |
| C7 | 0.6592 (3) | 0.72574 (13) | 0.88714 (14) | 0.0244 (5) | |
| C8 | 0.6266 (3) | 0.78723 (15) | 0.91504 (16) | 0.0360 (7) | |
| H8 | 0.5945 | 0.7887 | 0.9590 | 0.043* | |
| C9 | 0.6403 (3) | 0.84530 (15) | 0.87975 (18) | 0.0406 (8) | |
| H9 | 0.6177 | 0.8865 | 0.8995 | 0.049* | |

| | | | | |
|------|------------|--------------|--------------|-------------|
| C10 | 0.6868 (3) | 0.84447 (13) | 0.81571 (17) | 0.0341 (7) |
| H10 | 0.6935 | 0.8851 | 0.7917 | 0.041* |
| C11 | 0.7240 (3) | 0.78526 (12) | 0.78559 (14) | 0.0228 (5) |
| C12 | 0.6414 (3) | 0.66100 (14) | 0.92528 (14) | 0.0305 (6) |
| H12 | 0.6311 | 0.6245 | 0.8900 | 0.037* |
| C13 | 0.7856 (4) | 0.6460 (2) | 0.97959 (19) | 0.0546 (9) |
| H13A | 0.8762 | 0.6414 | 0.9567 | 0.082* |
| H13B | 0.7698 | 0.6046 | 1.0037 | 0.082* |
| H13C | 0.8037 | 0.6825 | 1.0134 | 0.082* |
| C14 | 0.4941 (4) | 0.6598 (2) | 0.95766 (19) | 0.0508 (9) |
| H14A | 0.5043 | 0.6923 | 0.9956 | 0.076* |
| H14B | 0.4802 | 0.6153 | 0.9760 | 0.076* |
| H14C | 0.4034 | 0.6711 | 0.9221 | 0.076* |
| C15 | 0.7721 (3) | 0.78374 (13) | 0.71450 (14) | 0.0273 (6) |
| H15 | 0.8556 | 0.7494 | 0.7165 | 0.033* |
| C16 | 0.8396 (4) | 0.84954 (16) | 0.69451 (19) | 0.0442 (8) |
| H16A | 0.7579 | 0.8836 | 0.6881 | 0.066* |
| H16B | 0.8794 | 0.8439 | 0.6510 | 0.066* |
| H16C | 0.9249 | 0.8634 | 0.7316 | 0.066* |
| C17 | 0.6363 (4) | 0.76249 (16) | 0.65765 (16) | 0.0384 (7) |
| H17A | 0.5981 | 0.7189 | 0.6693 | 0.058* |
| H17B | 0.6720 | 0.7598 | 0.6129 | 0.058* |
| H17C | 0.5520 | 0.7952 | 0.6544 | 0.058* |
| C18 | 0.7773 (3) | 0.47125 (11) | 0.74689 (13) | 0.0189 (5) |
| C19 | 0.7886 (3) | 0.44508 (12) | 0.68145 (14) | 0.0237 (5) |
| C20 | 0.7675 (3) | 0.37677 (13) | 0.67250 (16) | 0.0322 (6) |
| H20 | 0.7749 | 0.3574 | 0.6289 | 0.039* |
| C21 | 0.7363 (3) | 0.33683 (14) | 0.72531 (18) | 0.0382 (7) |
| H21 | 0.7187 | 0.2906 | 0.7175 | 0.046* |
| C22 | 0.7304 (3) | 0.36388 (13) | 0.78997 (17) | 0.0338 (7) |
| H22 | 0.7125 | 0.3354 | 0.8266 | 0.041* |
| C23 | 0.7499 (3) | 0.43164 (12) | 0.80269 (14) | 0.0233 (5) |
| C24 | 0.8220 (3) | 0.48775 (14) | 0.62187 (14) | 0.0321 (6) |
| H24 | 0.8367 | 0.5346 | 0.6389 | 0.038* |
| C25 | 0.6858 (4) | 0.4866 (2) | 0.56041 (18) | 0.0575 (10) |
| H25A | 0.6683 | 0.4410 | 0.5433 | 0.086* |
| H25B | 0.7103 | 0.5150 | 0.5230 | 0.086* |
| H25C | 0.5916 | 0.5032 | 0.5755 | 0.086* |
| C26 | 0.9727 (4) | 0.46537 (18) | 0.59885 (17) | 0.0437 (8) |
| H26A | 1.0602 | 0.4699 | 0.6379 | 0.066* |
| H26B | 0.9916 | 0.4931 | 0.5599 | 0.066* |
| H26C | 0.9627 | 0.4188 | 0.5840 | 0.066* |
| C27 | 0.7369 (3) | 0.46004 (14) | 0.87351 (15) | 0.0303 (6) |
| H27 | 0.7809 | 0.5061 | 0.8761 | 0.036* |
| C28 | 0.8298 (5) | 0.4201 (2) | 0.93406 (19) | 0.0575 (10) |
| H28A | 0.7837 | 0.3757 | 0.9352 | 0.086* |
| H28B | 0.8267 | 0.4430 | 0.9780 | 0.086* |
| H28C | 0.9382 | 0.4159 | 0.9276 | 0.086* |

| | | | | | |
|------|---------------|--------------|--------------|-------------|-------------|
| C29 | 0.5665 (4) | 0.46518 (17) | 0.88211 (18) | 0.0448 (8) | |
| H29A | 0.5082 | 0.4916 | 0.8439 | 0.067* | |
| H29B | 0.5608 | 0.4867 | 0.9266 | 0.067* | |
| H29C | 0.5212 | 0.4206 | 0.8813 | 0.067* | |
| C30 | 0.2422 (5) | 0.6816 (2) | 0.5728 (2) | 0.0542 (11) | 0.8596 (15) |
| H30A | 0.3380 | 0.6585 | 0.5652 | 0.065* | 0.8596 (15) |
| H30B | 0.2716 | 0.7124 | 0.6126 | 0.065* | 0.8596 (15) |
| C31 | 0.1302 (4) | 0.63193 (19) | 0.5906 (2) | 0.0400 (8) | 0.8596 (15) |
| H31A | 0.1851 | 0.6022 | 0.6274 | 0.048* | 0.8596 (15) |
| H31B | 0.0916 | 0.6044 | 0.5490 | 0.048* | 0.8596 (15) |
| Cl3 | 0.1657 (2) | 0.72810 (7) | 0.49755 (7) | 0.0786 (4) | 0.8596 (15) |
| Cl4 | -0.03082 (14) | 0.66888 (7) | 0.61983 (7) | 0.0674 (4) | 0.8596 (15) |
| C30A | 0.221 (3) | 0.6435 (9) | 0.5775 (14) | 0.0542 (11) | 0.1404 (15) |
| H30C | 0.1800 | 0.6054 | 0.5478 | 0.065* | 0.1404 (15) |
| H30D | 0.3091 | 0.6269 | 0.6127 | 0.065* | 0.1404 (15) |
| C31A | 0.0988 (15) | 0.6665 (12) | 0.6140 (9) | 0.0400 (8) | 0.1404 (15) |
| H31C | 0.1251 | 0.7116 | 0.6330 | 0.048* | 0.1404 (15) |
| H31D | 0.0927 | 0.6364 | 0.6535 | 0.048* | 0.1404 (15) |
| Cl3A | 0.2944 (13) | 0.7025 (4) | 0.5251 (4) | 0.0786 (4) | 0.1404 (15) |
| Cl4A | -0.0833 (8) | 0.6687 (4) | 0.5576 (4) | 0.0674 (4) | 0.1404 (15) |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|--------------|--------------|--------------|--------------|
| Pd1 | 0.01196 (8) | 0.01091 (8) | 0.02380 (10) | -0.00011 (6) | 0.00254 (6) | -0.00023 (7) |
| Cl1 | 0.0149 (2) | 0.0192 (3) | 0.0426 (4) | 0.0026 (2) | 0.0049 (2) | -0.0047 (3) |
| Cl2 | 0.0189 (3) | 0.0178 (3) | 0.0571 (5) | -0.0020 (2) | -0.0040 (3) | -0.0095 (3) |
| N1 | 0.0152 (8) | 0.0123 (9) | 0.0173 (9) | 0.0000 (7) | 0.0029 (7) | 0.0009 (7) |
| N2 | 0.0143 (8) | 0.0135 (9) | 0.0212 (10) | 0.0008 (7) | 0.0046 (8) | 0.0024 (8) |
| C1 | 0.0158 (10) | 0.0155 (11) | 0.0197 (11) | -0.0001 (8) | 0.0038 (9) | 0.0003 (9) |
| C2 | 0.0173 (10) | 0.0144 (11) | 0.0183 (11) | 0.0005 (8) | 0.0051 (9) | 0.0013 (9) |
| C3 | 0.0163 (11) | 0.0200 (12) | 0.0392 (15) | -0.0007 (9) | 0.0023 (11) | -0.0067 (11) |
| C4 | 0.0132 (10) | 0.0201 (12) | 0.0326 (14) | 0.0021 (9) | 0.0050 (10) | -0.0045 (10) |
| C5 | 0.0336 (16) | 0.0442 (19) | 0.053 (2) | 0.0115 (14) | 0.0008 (15) | 0.0008 (16) |
| C6 | 0.0129 (10) | 0.0150 (11) | 0.0268 (13) | 0.0009 (8) | 0.0017 (9) | -0.0033 (9) |
| C7 | 0.0209 (11) | 0.0260 (13) | 0.0254 (13) | 0.0028 (10) | 0.0015 (10) | -0.0062 (10) |
| C8 | 0.0333 (14) | 0.0381 (17) | 0.0370 (16) | 0.0069 (12) | 0.0075 (13) | -0.0174 (13) |
| C9 | 0.0378 (16) | 0.0240 (15) | 0.059 (2) | 0.0068 (12) | 0.0051 (15) | -0.0209 (14) |
| C10 | 0.0300 (14) | 0.0151 (13) | 0.0554 (19) | 0.0022 (10) | 0.0026 (13) | -0.0025 (12) |
| C11 | 0.0172 (11) | 0.0149 (11) | 0.0347 (14) | 0.0003 (9) | 0.0003 (10) | 0.0010 (10) |
| C12 | 0.0357 (14) | 0.0342 (15) | 0.0235 (13) | 0.0018 (12) | 0.0101 (12) | -0.0009 (11) |
| C13 | 0.049 (2) | 0.068 (3) | 0.045 (2) | 0.0088 (18) | 0.0030 (16) | 0.0187 (18) |
| C14 | 0.0477 (19) | 0.064 (2) | 0.047 (2) | -0.0008 (17) | 0.0233 (17) | 0.0024 (17) |
| C15 | 0.0246 (12) | 0.0206 (13) | 0.0367 (15) | 0.0009 (10) | 0.0061 (11) | 0.0094 (11) |
| C16 | 0.0363 (16) | 0.0362 (18) | 0.057 (2) | -0.0141 (13) | -0.0009 (15) | 0.0167 (15) |
| C17 | 0.0381 (16) | 0.0409 (18) | 0.0355 (16) | -0.0128 (13) | 0.0043 (13) | 0.0061 (14) |
| C18 | 0.0136 (10) | 0.0115 (10) | 0.0310 (13) | 0.0004 (8) | 0.0026 (9) | -0.0005 (9) |
| C19 | 0.0219 (11) | 0.0187 (12) | 0.0298 (14) | 0.0000 (9) | 0.0026 (10) | -0.0052 (10) |

| | | | | | | |
|------|-------------|-------------|-------------|--------------|--------------|--------------|
| C20 | 0.0293 (13) | 0.0214 (13) | 0.0458 (18) | -0.0015 (11) | 0.0062 (13) | -0.0126 (12) |
| C21 | 0.0302 (14) | 0.0146 (13) | 0.071 (2) | -0.0030 (11) | 0.0135 (15) | -0.0070 (13) |
| C22 | 0.0282 (13) | 0.0176 (13) | 0.059 (2) | 0.0010 (10) | 0.0180 (13) | 0.0119 (13) |
| C23 | 0.0165 (11) | 0.0199 (12) | 0.0344 (14) | 0.0043 (9) | 0.0069 (10) | 0.0068 (10) |
| C24 | 0.0440 (16) | 0.0282 (15) | 0.0230 (13) | -0.0005 (12) | 0.0034 (12) | -0.0029 (11) |
| C25 | 0.059 (2) | 0.074 (3) | 0.0342 (18) | 0.009 (2) | -0.0053 (17) | 0.0033 (18) |
| C26 | 0.0471 (18) | 0.055 (2) | 0.0325 (16) | -0.0054 (16) | 0.0156 (15) | -0.0039 (15) |
| C27 | 0.0309 (14) | 0.0307 (15) | 0.0315 (15) | 0.0014 (11) | 0.0116 (12) | 0.0090 (12) |
| C28 | 0.057 (2) | 0.076 (3) | 0.0402 (19) | 0.017 (2) | 0.0103 (17) | 0.0219 (19) |
| C29 | 0.0393 (17) | 0.047 (2) | 0.054 (2) | 0.0057 (14) | 0.0258 (16) | 0.0071 (16) |
| C30 | 0.048 (2) | 0.069 (3) | 0.045 (2) | -0.008 (2) | 0.0088 (19) | -0.006 (2) |
| C31 | 0.048 (2) | 0.034 (2) | 0.036 (2) | 0.0034 (17) | 0.0031 (17) | -0.0031 (16) |
| Cl3 | 0.1267 (13) | 0.0573 (8) | 0.0562 (7) | -0.0177 (8) | 0.0283 (8) | 0.0119 (6) |
| Cl4 | 0.0621 (7) | 0.0740 (8) | 0.0735 (8) | 0.0170 (6) | 0.0320 (6) | 0.0125 (7) |
| C30A | 0.048 (2) | 0.069 (3) | 0.045 (2) | -0.008 (2) | 0.0088 (19) | -0.006 (2) |
| C31A | 0.048 (2) | 0.034 (2) | 0.036 (2) | 0.0034 (17) | 0.0031 (17) | -0.0031 (16) |
| Cl3A | 0.1267 (13) | 0.0573 (8) | 0.0562 (7) | -0.0177 (8) | 0.0283 (8) | 0.0119 (6) |
| Cl4A | 0.0621 (7) | 0.0740 (8) | 0.0735 (8) | 0.0170 (6) | 0.0320 (6) | 0.0125 (7) |

Geometric parameters (Å, °)

| | | | |
|---------|-------------|----------|-----------|
| Pd1—N2 | 2.0200 (18) | C17—H17A | 0.9800 |
| Pd1—N1 | 2.0280 (19) | C17—H17B | 0.9800 |
| Pd1—Cl2 | 2.2840 (7) | C17—H17C | 0.9800 |
| Pd1—Cl1 | 2.2843 (6) | C18—C19 | 1.402 (3) |
| N1—C1 | 1.297 (3) | C18—C23 | 1.405 (3) |
| N1—C6 | 1.443 (3) | C19—C20 | 1.394 (4) |
| N2—C2 | 1.293 (3) | C19—C24 | 1.516 (4) |
| N2—C18 | 1.449 (3) | C20—C21 | 1.373 (4) |
| C1—C2 | 1.492 (3) | C20—H20 | 0.9500 |
| C1—C3 | 1.493 (3) | C21—C22 | 1.384 (4) |
| C2—C4 | 1.497 (3) | C21—H21 | 0.9500 |
| C3—H3A | 0.9800 | C22—C23 | 1.391 (4) |
| C3—H3B | 0.9800 | C22—H22 | 0.9500 |
| C3—H3C | 0.9800 | C23—C27 | 1.519 (4) |
| C4—C5 | 1.532 (4) | C24—C25 | 1.528 (4) |
| C4—H4A | 0.9900 | C24—C26 | 1.531 (4) |
| C4—H4B | 0.9900 | C24—H24 | 1.0000 |
| C5—H5A | 0.9800 | C25—H25A | 0.9800 |
| C5—H5B | 0.9800 | C25—H25B | 0.9800 |
| C5—H5C | 0.9800 | C25—H25C | 0.9800 |
| C6—C7 | 1.401 (3) | C26—H26A | 0.9800 |
| C6—C11 | 1.403 (3) | C26—H26B | 0.9800 |
| C7—C8 | 1.402 (4) | C26—H26C | 0.9800 |
| C7—C12 | 1.522 (4) | C27—C29 | 1.529 (4) |
| C8—C9 | 1.373 (5) | C27—C28 | 1.535 (4) |
| C8—H8 | 0.9500 | C27—H27 | 1.0000 |
| C9—C10 | 1.383 (4) | C28—H28A | 0.9800 |

| | | | |
|-------------|-------------|---------------|-----------|
| C9—H9 | 0.9500 | C28—H28B | 0.9800 |
| C10—C11 | 1.393 (4) | C28—H28C | 0.9800 |
| C10—H10 | 0.9500 | C29—H29A | 0.9800 |
| C11—C15 | 1.521 (4) | C29—H29B | 0.9800 |
| C12—C13 | 1.521 (4) | C29—H29C | 0.9800 |
| C12—C14 | 1.531 (4) | C30—C31 | 1.482 (6) |
| C12—H12 | 1.0000 | C30—C13 | 1.768 (5) |
| C13—H13A | 0.9800 | C30—H30A | 0.9900 |
| C13—H13B | 0.9800 | C30—H30B | 0.9900 |
| C13—H13C | 0.9800 | C31—C14 | 1.772 (4) |
| C14—H14A | 0.9800 | C31—H31A | 0.9900 |
| C14—H14B | 0.9800 | C31—H31B | 0.9900 |
| C14—H14C | 0.9800 | C30A—C31A | 1.460 (8) |
| C15—C16 | 1.528 (4) | C30A—C13A | 1.761 (6) |
| C15—C17 | 1.530 (4) | C30A—H30C | 0.9900 |
| C15—H15 | 1.0000 | C30A—H30D | 0.9900 |
| C16—H16A | 0.9800 | C31A—C14A | 1.762 (5) |
| C16—H16B | 0.9800 | C31A—H31C | 0.9900 |
| C16—H16C | 0.9800 | C31A—H31D | 0.9900 |
| | | | |
| N2—Pd1—N1 | 79.01 (8) | C15—C17—H17A | 109.5 |
| N2—Pd1—Cl2 | 96.29 (6) | C15—C17—H17B | 109.5 |
| N1—Pd1—Cl2 | 174.30 (5) | H17A—C17—H17B | 109.5 |
| N2—Pd1—Cl1 | 173.66 (6) | C15—C17—H17C | 109.5 |
| N1—Pd1—Cl1 | 95.21 (5) | H17A—C17—H17C | 109.5 |
| Cl2—Pd1—Cl1 | 89.62 (2) | H17B—C17—H17C | 109.5 |
| C1—N1—C6 | 121.05 (19) | C19—C18—C23 | 122.9 (2) |
| C1—N1—Pd1 | 115.15 (15) | C19—C18—N2 | 120.0 (2) |
| C6—N1—Pd1 | 123.80 (14) | C23—C18—N2 | 116.9 (2) |
| C2—N2—C18 | 122.18 (19) | C20—C19—C18 | 117.1 (2) |
| C2—N2—Pd1 | 115.72 (15) | C20—C19—C24 | 120.1 (2) |
| C18—N2—Pd1 | 121.80 (14) | C18—C19—C24 | 122.8 (2) |
| N1—C1—C2 | 114.7 (2) | C21—C20—C19 | 121.5 (3) |
| N1—C1—C3 | 124.3 (2) | C21—C20—H20 | 119.2 |
| C2—C1—C3 | 120.83 (19) | C19—C20—H20 | 119.2 |
| N2—C2—C1 | 114.69 (19) | C20—C21—C22 | 120.0 (3) |
| N2—C2—C4 | 124.5 (2) | C20—C21—H21 | 120.0 |
| C1—C2—C4 | 120.7 (2) | C22—C21—H21 | 120.0 |
| C1—C3—H3A | 109.5 | C21—C22—C23 | 121.7 (3) |
| C1—C3—H3B | 109.5 | C21—C22—H22 | 119.2 |
| H3A—C3—H3B | 109.5 | C23—C22—H22 | 119.2 |
| C1—C3—H3C | 109.5 | C22—C23—C18 | 116.8 (3) |
| H3A—C3—H3C | 109.5 | C22—C23—C27 | 120.3 (2) |
| H3B—C3—H3C | 109.5 | C18—C23—C27 | 122.9 (2) |
| C2—C4—C5 | 112.9 (2) | C19—C24—C25 | 111.4 (3) |
| C2—C4—H4A | 109.0 | C19—C24—C26 | 110.6 (2) |
| C5—C4—H4A | 109.0 | C25—C24—C26 | 110.6 (3) |
| C2—C4—H4B | 109.0 | C19—C24—H24 | 108.0 |

| | | | |
|---------------|-----------|----------------|------------|
| C5—C4—H4B | 109.0 | C25—C24—H24 | 108.0 |
| H4A—C4—H4B | 107.8 | C26—C24—H24 | 108.0 |
| C4—C5—H5A | 109.5 | C24—C25—H25A | 109.5 |
| C4—C5—H5B | 109.5 | C24—C25—H25B | 109.5 |
| H5A—C5—H5B | 109.5 | H25A—C25—H25B | 109.5 |
| C4—C5—H5C | 109.5 | C24—C25—H25C | 109.5 |
| H5A—C5—H5C | 109.5 | H25A—C25—H25C | 109.5 |
| H5B—C5—H5C | 109.5 | H25B—C25—H25C | 109.5 |
| C7—C6—C11 | 123.3 (2) | C24—C26—H26A | 109.5 |
| C7—C6—N1 | 116.2 (2) | C24—C26—H26B | 109.5 |
| C11—C6—N1 | 120.5 (2) | H26A—C26—H26B | 109.5 |
| C6—C7—C8 | 116.9 (3) | C24—C26—H26C | 109.5 |
| C6—C7—C12 | 121.8 (2) | H26A—C26—H26C | 109.5 |
| C8—C7—C12 | 121.3 (2) | H26B—C26—H26C | 109.5 |
| C9—C8—C7 | 120.9 (3) | C23—C27—C29 | 111.0 (2) |
| C9—C8—H8 | 119.5 | C23—C27—C28 | 112.8 (3) |
| C7—C8—H8 | 119.5 | C29—C27—C28 | 109.8 (2) |
| C8—C9—C10 | 120.7 (3) | C23—C27—H27 | 107.6 |
| C8—C9—H9 | 119.7 | C29—C27—H27 | 107.6 |
| C10—C9—H9 | 119.7 | C28—C27—H27 | 107.6 |
| C9—C10—C11 | 121.4 (3) | C27—C28—H28A | 109.5 |
| C9—C10—H10 | 119.3 | C27—C28—H28B | 109.5 |
| C11—C10—H10 | 119.3 | H28A—C28—H28B | 109.5 |
| C10—C11—C6 | 116.7 (2) | C27—C28—H28C | 109.5 |
| C10—C11—C15 | 121.7 (2) | H28A—C28—H28C | 109.5 |
| C6—C11—C15 | 121.6 (2) | H28B—C28—H28C | 109.5 |
| C13—C12—C7 | 111.5 (3) | C27—C29—H29A | 109.5 |
| C13—C12—C14 | 111.0 (3) | C27—C29—H29B | 109.5 |
| C7—C12—C14 | 112.5 (2) | H29A—C29—H29B | 109.5 |
| C13—C12—H12 | 107.2 | C27—C29—H29C | 109.5 |
| C7—C12—H12 | 107.2 | H29A—C29—H29C | 109.5 |
| C14—C12—H12 | 107.2 | H29B—C29—H29C | 109.5 |
| C12—C13—H13A | 109.5 | C31—C30—C13 | 112.7 (3) |
| C12—C13—H13B | 109.5 | C31—C30—H30A | 109.1 |
| H13A—C13—H13B | 109.5 | C13—C30—H30A | 109.1 |
| C12—C13—H13C | 109.5 | C31—C30—H30B | 109.1 |
| H13A—C13—H13C | 109.5 | C13—C30—H30B | 109.1 |
| H13B—C13—H13C | 109.5 | H30A—C30—H30B | 107.8 |
| C12—C14—H14A | 109.5 | C30—C31—C14 | 112.7 (3) |
| C12—C14—H14B | 109.5 | C30—C31—H31A | 109.0 |
| H14A—C14—H14B | 109.5 | C14—C31—H31A | 109.0 |
| C12—C14—H14C | 109.5 | C30—C31—H31B | 109.0 |
| H14A—C14—H14C | 109.5 | C14—C31—H31B | 109.0 |
| H14B—C14—H14C | 109.5 | H31A—C31—H31B | 107.8 |
| C11—C15—C16 | 113.5 (2) | C31A—C30A—C13A | 116.3 (16) |
| C11—C15—C17 | 111.3 (2) | C31A—C30A—H30C | 108.2 |
| C16—C15—C17 | 109.9 (2) | C13A—C30A—H30C | 108.2 |
| C11—C15—H15 | 107.3 | C31A—C30A—H30D | 108.2 |

| | | | |
|---------------|-------|----------------|------------|
| C16—C15—H15 | 107.3 | C13A—C30A—H30D | 108.2 |
| C17—C15—H15 | 107.3 | H30C—C30A—H30D | 107.4 |
| C15—C16—H16A | 109.5 | C30A—C31A—C14A | 111.0 (17) |
| C15—C16—H16B | 109.5 | C30A—C31A—H31C | 109.4 |
| H16A—C16—H16B | 109.5 | C14A—C31A—H31C | 109.4 |
| C15—C16—H16C | 109.5 | C30A—C31A—H31D | 109.4 |
| H16A—C16—H16C | 109.5 | C14A—C31A—H31D | 109.4 |
| H16B—C16—H16C | 109.5 | H31C—C31A—H31D | 108.0 |

Hydrogen-bond geometry (\AA , $^\circ$)

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|------------------------------------|-------|-------------|-------------|---------------|
| C3—H3B \cdots C11 ⁱ | 0.98 | 2.67 | 3.604 (3) | 160 |
| C4—H4A \cdots C11 ⁱ | 0.99 | 2.79 | 3.763 (3) | 166 |
| C15—H15 \cdots C14 ⁱ | 1.00 | 2.80 | 3.586 (3) | 136 |
| C21—H21 \cdots C11 ⁱⁱ | 0.95 | 2.74 | 3.633 (3) | 156 |

Symmetry codes: (i) $x+1, y, z$; (ii) $-x+1, y-1/2, -z+3/2$.