

Serendipitous preparation of *fac*-(acetonitrile- κN)trichlorido[(1,2,5,6- η)-cycloocta-1,5-diene]-iridium(III)

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A reaction between $[(\text{COD})\text{IrCl}]_2$ (COD is cycloocta-1,5-diene), HCl and indene failed to provide the hoped for chloridoindenyliridium dimer, but instead produced the title compound, $[\text{IrCl}_3(\text{CH}_3\text{CN})(\text{C}_8\text{H}_{12})]$, which is an octahedral complex of iridium(III) with a chelating cycloocta-1,5-diene ligand, three chloride ligands in a *fac* arrangement, and one acetonitrile ligand. Attempts to devise a rational synthesis for the title compound were unsuccessful.

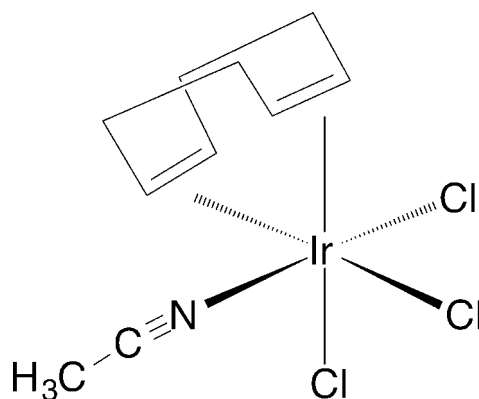
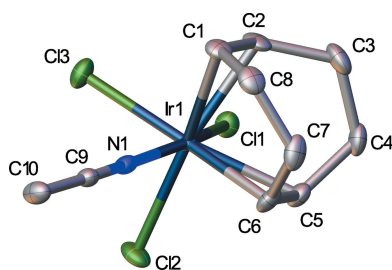
Keywords: crystal structure; iridium; cycloocta-diene; acetonitrile

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1. Chemical context

We have published recently on the synthesis of a series of tetramethylalkylcyclopentadienyliridium complexes by the direct reaction between tetramethylalkylcyclopentadiene and iridium chloride, giving the $[\text{Cp}^*\text{IrCl}_2]_2$ dimer (Morris *et al.*, 2014). From the dimer, a variety of other compounds can be made, such as amino acid complexes, that have shown significant anti-mycobacterial activity (Karpin *et al.*, 2013). Some of the reactions produced low yields of the chlorido-bridged dimer, thus limiting the number of products that could be made and tested.



An alternate route to Cp*-type chlorido iridium dimers was reported using $[(\text{COD})\text{IrCl}]_2$ as the starting material (El Amouri *et al.*, 1994) and, in our hands, this route does have promise for providing higher yields for many of the compounds. However, in the case of indene, there was no indication that an indenyl iridium complex had been prepared. Instead, a yellow–brown intractable solid was formed. Several attempts to dissolve the solid and to separate products through fractional crystallization all failed. During the course of this work-up, one of the solvents used was acetonitrile. At some point, the product mixture was allowed to stand in solution, and after about 24 hours several very nicely shaped

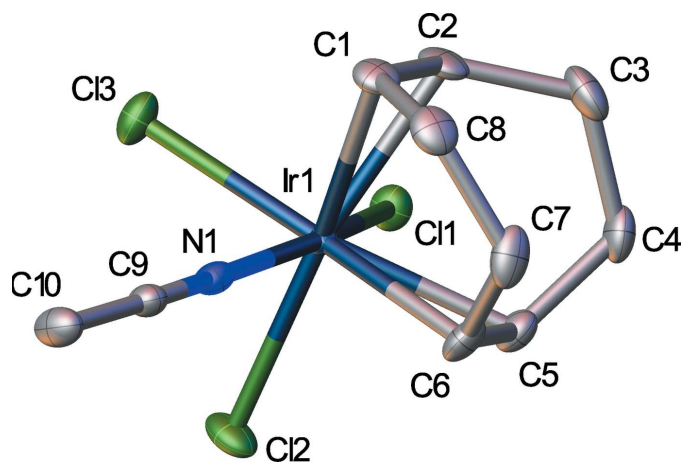


Figure 1
The asymmetric unit of the title compound. Displacement ellipsoids are shown at the 50% probability level.

rectangular prisms had formed in the sample. These crystals were examined by X-ray crystallography and the results of that structure determination are reported here.

2. Structural commentary

While the total number of cycloocta-1,5-diene complexes structurally characterized is quite large, the number that are directly comparable to the title compound is small. The title compound is a pseudo-octahedral complex of iridium with three chloride ligands occupying one face of the octahedron and the alkenes of the COD and the acetonitrile ligand occupying the opposite face (Fig. 1). Considering the varying ligands about the central iridium, there is very little distortion from ideal octahedral angles, with the most significant distortion being the N1–Ir1–Cl2 distorted away from the COD group with an angle of 164.05 (11)°. All other angles, including those involving the alkene centroids, deviate by no more than 5° from the ideal. All three Ir–Cl bond lengths are similar [range 2.3603 (11) to 2.3670 (11) Å], which is in keeping with both types of *trans* ligands, alkene and acetonitrile, being expected to be strong *trans*-influence ligands and would have a similar magnitude of effect on the chloride *trans* to either ligand.

The facial Ir–Cl distances may be contrasted with the average distance of 2.441 (2) Å for *fac*-[(Me₃P)₃IrCl₃] (CCDC: 896073) and related compounds (Merola *et al.*, 2013) that have somewhat longer Ir–Cl distances due to the effect of the *trans* PMe₃ groups.

Choudhury *et al.* (2005) reported on a COD complex of iridium with three chlorides and a SnCl₃ ligand completing the octahedral coordination about the central Ir atom (CCDC: 273475). In that case, though, the compound is a dinuclear one with Ir–Cl–Ir bridges. So, there are long Ir–Cl bonds (those involved in bridging) of 2.544 (4) Å and a shorter terminal Ir–Cl bond of 2.385 (6) Å. C=C bond lengths for the COD

Table 1
Experimental details.

| | |
|--|---|
| Crystal data | |
| Chemical formula | [IrCl ₃ (C ₂ H ₃ N)(C ₈ H ₁₂)] |
| <i>M_r</i> | 447.78 |
| Crystal system, space group | Orthorhombic, <i>P</i> 2 ₁ 2 ₁ 2 ₁ |
| Temperature (K) | 100 |
| <i>a</i> , <i>b</i> , <i>c</i> (Å) | 8.25131 (10), 11.85605 (14), 12.94150 (15) |
| <i>V</i> (Å ³) | 1266.04 (3) |
| <i>Z</i> | 4 |
| Radiation type | Mo <i>K</i> α |
| <i>μ</i> (mm ^{−1}) | 11.15 |
| Crystal size (mm) | 0.22 × 0.15 × 0.11 |
| Data collection | |
| Diffractometer | Agilent Xcalibur Eos Gemini ultra |
| Absorption correction | Analytical (<i>SCALE3 ABSPACK</i> ; Clark & Reid, 1995) |
| <i>T_{min}</i> , <i>T_{max}</i> | 0.204, 0.396 |
| No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections | 27207, 4333, 4173 |
| <i>R_{int}</i> | 0.040 |
| (sin θ/λ) _{max} (Å ^{−1}) | 0.755 |
| Refinement | |
| <i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i> | 0.019, 0.038, 1.08 |
| No. of reflections | 4333 |
| No. of parameters | 137 |
| H-atom treatment | H-atom parameters constrained |
| Δρ _{max} , Δρ _{min} (e Å ^{−3}) | 1.07, −0.74 |
| Absolute structure | Flack <i>x</i> determined using 1715 quotients [(<i>I</i> ⁺) − (<i>I</i> [−])] / [(<i>I</i> ⁺) + (<i>I</i> [−])] (Parsons <i>et al.</i> , 2013) |
| Absolute structure parameter | −0.011 (4) |

Computer programs: *CrysAlis PRO* (Agilent, 2014), *SHELXS97* and *SHELXL97* (Sheldrick, 2008) and *OLEX2* (Dolomanov *et al.*, 2009).

ring are similar to the title compound at 1.38 (1) and 1.41 (2) Å.

3. Supramolecular features

Although there appear to be some close C–H⋯Cl intermolecular interactions, there are no important supramolecular features to speak of in this structure.

4. Database survey

A substructure search of the CCDC (Groom & Allen, 2014) for the 1,5-COD–Ir fragment resulted in over 850 hits. This is not a surprising result since [CODIrCl]₂ is a convenient, high-yield organometallic starting material made in one step from IrCl₃·H₂O and cycloocta-1,5-diene (Crabtree & Morris, 1977). From [CODIrCl]₂, a wide variety of ligand addition, chloride replacement or bridge-splitting reactions can be carried out, leading to a wide variety of compounds containing the COD chelate. Using *Mercury* (Macrae *et al.*, 2008), an analysis of the COD–Ir search of the database for structures with an octahedral coordination around the metal showed that the C=C bonds of the COD ligands ranged from 1.184 to 1.508 Å with a mean of 1.394 Å. For the title compound, the values of 1.392 (7) and 1.389 (6) Å are pretty much right at the mean for COD C=C bonds.

An analysis of the CCDC database (Groom & Allen, 2014) for octahedral iridium complexes with acetonitrile ligands uncovered 99 hits with Ir–N distances measuring from a minimum of 1.897 Å to a maximum of 2.246 Å with a mean of 2.068 Å. For the title compound, the Ir–N distance of 2.023 (4) Å places it just below the mean.

5. Synthesis and crystallization

The title complex was formed as a few isolated crystals from an attempted reaction between [(COD)IrCl]₂ and indene with HCl in an attempt to synthesize the [indenylIrCl₂]₂ dimer, which would have been a useful starting material for our studies. Unfortunately, this did not provide the desired product. The reaction produced some very intractable solids. After multiple attempts to dissolve the solid in many different solvents, including acetonitrile, some well-shaped prisms formed on the side of the flask and these crystals were used in this investigation and were shown to be that of the title complex. Attempts to make this material in a rational fashion were not successful.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. H atoms were positioned geometrically and refined as riding with C–H = 0.96–0.98 Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$.

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Serendipitous preparation of *fac*-(acetonitrile- κ N)trichlorido[(1,2,5,6- η)-cycloocta-1,5-diene]iridium(III)

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Computing details

Data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO* (Agilent, 2014); data reduction: *CrysAlis PRO* (Agilent, 2014); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: OLEX2 (Dolomanov *et al.*, 2009); software used to prepare material for publication: OLEX2 (Dolomanov *et al.*, 2009).

fac-(Acetonitrile- κ N)trichlorido[(1,2,5,6- η)-cycloocta-1,5-diene]iridium(III)

Crystal data

[IrCl₃(C₂H₃N)(C₈H₁₂)]
 $M_r = 447.78$
 Orthorhombic, $P2_12_12_1$
 $a = 8.25131$ (10) Å
 $b = 11.85605$ (14) Å
 $c = 12.94150$ (15) Å
 $V = 1266.04$ (3) Å³
 $Z = 4$
 $F(000) = 840$

$D_x = 2.349$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 12030 reflections
 $\theta = 4.0$ – 32.2°
 $\mu = 11.15$ mm⁻¹
 $T = 100$ K
 Prism, clear light orange
 0.22 × 0.15 × 0.11 mm

Data collection

Agilent Xcalibur Eos Gemini ultra
 diffractometer
 Radiation source: Enhance (Mo) X-ray Source,
 Agilent Gemini System
 Graphite monochromator
 Detector resolution: 16.0122 pixels mm⁻¹
 ω scans
 Absorption correction: analytical
 (SCALE3 ABSPACK; Clark & Reid, 1995)

$T_{\min} = 0.204$, $T_{\max} = 0.396$
 27207 measured reflections
 4333 independent reflections
 4173 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.040$
 $\theta_{\max} = 32.4^\circ$, $\theta_{\min} = 3.4^\circ$
 $h = -11 \rightarrow 12$
 $k = -17 \rightarrow 17$
 $l = -19 \rightarrow 19$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.019$
 $wR(F^2) = 0.038$
 $S = 1.08$
 4333 reflections
 137 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0124P)^2 + 1.2315P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.002$
 $\Delta\rho_{\max} = 1.07$ e Å⁻³
 $\Delta\rho_{\min} = -0.74$ e Å⁻³

Absolute structure: Flack x determined using
1715 quotients $[(F^+)-(F^-)]/[(F^+)+(F^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: -0.011 (4)

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|--------------|-------------|----------------------------------|
| Ir1 | 0.70199 (2) | 0.47821 (2) | 0.68347 (2) | 0.01178 (4) |
| C11 | 0.68059 (17) | 0.64680 (9) | 0.78048 (9) | 0.0238 (2) |
| C12 | 0.85370 (14) | 0.58778 (9) | 0.56587 (9) | 0.0202 (2) |
| C13 | 0.47576 (13) | 0.52459 (13) | 0.57940 (9) | 0.0275 (2) |
| N1 | 0.5308 (5) | 0.4220 (3) | 0.7828 (3) | 0.0153 (7) |
| C1 | 0.7775 (6) | 0.3484 (4) | 0.5677 (3) | 0.0210 (9) |
| H1 | 0.7227 | 0.3540 | 0.5008 | 0.025* |
| C2 | 0.6888 (7) | 0.2940 (4) | 0.6442 (3) | 0.0210 (9) |
| H2 | 0.5813 | 0.2686 | 0.6219 | 0.025* |
| C3 | 0.7669 (7) | 0.2197 (4) | 0.7247 (4) | 0.0268 (12) |
| H3A | 0.8639 | 0.1863 | 0.6953 | 0.032* |
| H3B | 0.6930 | 0.1587 | 0.7413 | 0.032* |
| C4 | 0.8132 (6) | 0.2806 (4) | 0.8253 (4) | 0.0245 (9) |
| H4A | 0.7241 | 0.2736 | 0.8737 | 0.029* |
| H4B | 0.9063 | 0.2430 | 0.8553 | 0.029* |
| C5 | 0.8528 (5) | 0.4040 (4) | 0.8121 (4) | 0.0202 (9) |
| H5 | 0.8518 | 0.4477 | 0.8764 | 0.024* |
| C6 | 0.9554 (6) | 0.4463 (4) | 0.7360 (4) | 0.0229 (10) |
| H6 | 1.0132 | 0.5148 | 0.7568 | 0.028* |
| C7 | 1.0494 (6) | 0.3734 (5) | 0.6606 (4) | 0.0289 (12) |
| H7A | 1.0816 | 0.3048 | 0.6959 | 0.035* |
| H7B | 1.1476 | 0.4132 | 0.6414 | 0.035* |
| C8 | 0.9592 (6) | 0.3407 (4) | 0.5616 (4) | 0.0254 (11) |
| H8A | 0.9963 | 0.3892 | 0.5061 | 0.030* |
| H8B | 0.9885 | 0.2640 | 0.5437 | 0.030* |
| C9 | 0.4307 (5) | 0.3980 (4) | 0.8376 (3) | 0.0174 (9) |
| C10 | 0.3017 (7) | 0.3700 (4) | 0.9097 (3) | 0.0246 (9) |
| H10A | 0.2025 | 0.4047 | 0.8874 | 0.037* |
| H10B | 0.3294 | 0.3972 | 0.9773 | 0.037* |
| H10C | 0.2879 | 0.2896 | 0.9120 | 0.037* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|-------------|-------------|-------------|
| Ir1 | 0.01257 (6) | 0.01427 (6) | 0.00849 (6) | 0.00042 (6) | 0.00024 (6) | 0.00067 (6) |

| | | | | | | |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C11 | 0.0356 (7) | 0.0166 (5) | 0.0193 (5) | -0.0010 (5) | 0.0073 (5) | -0.0029 (4) |
| C12 | 0.0229 (5) | 0.0206 (5) | 0.0170 (5) | -0.0021 (4) | 0.0046 (4) | 0.0034 (4) |
| C13 | 0.0177 (5) | 0.0456 (7) | 0.0191 (5) | 0.0062 (5) | -0.0037 (4) | 0.0099 (6) |
| N1 | 0.0216 (19) | 0.0134 (17) | 0.0110 (16) | -0.0012 (14) | -0.0036 (14) | 0.0013 (13) |
| C1 | 0.027 (3) | 0.022 (2) | 0.014 (2) | -0.002 (2) | 0.0019 (19) | -0.0038 (16) |
| C2 | 0.031 (2) | 0.0162 (19) | 0.0162 (19) | -0.006 (2) | 0.002 (2) | -0.0054 (15) |
| C3 | 0.041 (3) | 0.016 (2) | 0.024 (2) | 0.007 (2) | 0.012 (2) | 0.0051 (18) |
| C4 | 0.026 (2) | 0.027 (2) | 0.020 (2) | 0.0060 (19) | -0.001 (2) | 0.0097 (19) |
| C5 | 0.0196 (19) | 0.028 (2) | 0.0133 (19) | 0.0018 (16) | -0.0060 (19) | 0.006 (2) |
| C6 | 0.015 (2) | 0.036 (3) | 0.018 (2) | 0.0004 (18) | -0.0079 (17) | 0.0053 (19) |
| C7 | 0.016 (2) | 0.040 (3) | 0.030 (3) | 0.010 (2) | 0.0045 (19) | 0.009 (2) |
| C8 | 0.030 (3) | 0.024 (2) | 0.022 (2) | 0.008 (2) | 0.011 (2) | -0.002 (2) |
| C9 | 0.0157 (19) | 0.021 (2) | 0.015 (2) | -0.0023 (16) | -0.0014 (15) | -0.0035 (16) |
| C10 | 0.020 (2) | 0.037 (3) | 0.017 (2) | -0.007 (2) | 0.003 (2) | -0.0031 (18) |

Geometric parameters (Å, °)

| | | | |
|-------------|-------------|------------|-----------|
| Ir1—C11 | 2.3670 (11) | C4—H4A | 0.9700 |
| Ir1—C12 | 2.3603 (11) | C4—H4B | 0.9700 |
| Ir1—C13 | 2.3666 (10) | C4—C5 | 1.509 (6) |
| Ir1—N1 | 2.023 (4) | C5—H5 | 0.9800 |
| Ir1—C1 | 2.236 (4) | C5—C6 | 1.392 (7) |
| Ir1—C2 | 2.245 (4) | C6—H6 | 0.9800 |
| Ir1—C5 | 2.257 (5) | C6—C7 | 1.517 (7) |
| Ir1—C6 | 2.231 (4) | C7—H7A | 0.9700 |
| N1—C9 | 1.125 (6) | C7—H7B | 0.9700 |
| C1—H1 | 0.9800 | C7—C8 | 1.531 (8) |
| C1—C2 | 1.389 (6) | C8—H8A | 0.9700 |
| C1—C8 | 1.504 (7) | C8—H8B | 0.9700 |
| C2—H2 | 0.9800 | C9—C10 | 1.454 (6) |
| C2—C3 | 1.510 (7) | C10—H10A | 0.9600 |
| C3—H3A | 0.9700 | C10—H10B | 0.9600 |
| C3—H3B | 0.9700 | C10—H10C | 0.9600 |
| C3—C4 | 1.537 (7) | | |
| C12—Ir1—C11 | 85.23 (4) | C2—C3—H3B | 108.6 |
| C12—Ir1—C13 | 85.61 (4) | C2—C3—C4 | 114.6 (4) |
| C13—Ir1—C11 | 92.68 (5) | H3A—C3—H3B | 107.6 |
| N1—Ir1—C11 | 83.63 (11) | C4—C3—H3A | 108.6 |
| N1—Ir1—C12 | 164.05 (11) | C4—C3—H3B | 108.6 |
| N1—Ir1—C13 | 83.55 (11) | C3—C4—H4A | 108.6 |
| N1—Ir1—C1 | 113.18 (16) | C3—C4—H4B | 108.6 |
| N1—Ir1—C2 | 77.86 (16) | H4A—C4—H4B | 107.6 |
| N1—Ir1—C5 | 77.73 (16) | C5—C4—C3 | 114.5 (4) |
| N1—Ir1—C6 | 113.88 (16) | C5—C4—H4A | 108.6 |
| C1—Ir1—C11 | 163.18 (12) | C5—C4—H4B | 108.6 |
| C1—Ir1—C12 | 78.41 (12) | Ir1—C5—H5 | 114.5 |
| C1—Ir1—C13 | 89.92 (14) | C4—C5—Ir1 | 110.0 (3) |

| | | | |
|--------------|-------------|---------------|-----------|
| C1—Ir1—C2 | 36.12 (16) | C4—C5—H5 | 114.5 |
| C1—Ir1—C5 | 94.14 (18) | C6—C5—Ir1 | 70.9 (3) |
| C2—Ir1—C11 | 159.73 (12) | C6—C5—C4 | 124.1 (5) |
| C2—Ir1—C12 | 114.52 (12) | C6—C5—H5 | 114.5 |
| C2—Ir1—C13 | 93.37 (14) | Ir1—C6—H6 | 113.6 |
| C2—Ir1—C5 | 79.33 (18) | C5—C6—Ir1 | 73.0 (3) |
| C5—Ir1—C11 | 88.79 (13) | C5—C6—H6 | 113.6 |
| C5—Ir1—C12 | 113.44 (12) | C5—C6—C7 | 124.2 (5) |
| C5—Ir1—C13 | 160.95 (12) | C7—C6—Ir1 | 112.4 (3) |
| C6—Ir1—C11 | 92.97 (14) | C7—C6—H6 | 113.6 |
| C6—Ir1—C12 | 78.03 (13) | C6—C7—H7A | 108.4 |
| C6—Ir1—C13 | 162.18 (12) | C6—C7—H7B | 108.4 |
| C6—Ir1—C1 | 79.97 (19) | C6—C7—C8 | 115.7 (4) |
| C6—Ir1—C2 | 87.1 (2) | H7A—C7—H7B | 107.4 |
| C6—Ir1—C5 | 36.15 (17) | C8—C7—H7A | 108.4 |
| C9—N1—Ir1 | 175.1 (4) | C8—C7—H7B | 108.4 |
| Ir1—C1—H1 | 114.7 | C1—C8—C7 | 115.2 (4) |
| C2—C1—Ir1 | 72.3 (3) | C1—C8—H8A | 108.5 |
| C2—C1—H1 | 114.7 | C1—C8—H8B | 108.5 |
| C2—C1—C8 | 122.3 (5) | C7—C8—H8A | 108.5 |
| C8—C1—Ir1 | 110.8 (3) | C7—C8—H8B | 108.5 |
| C8—C1—H1 | 114.7 | H8A—C8—H8B | 107.5 |
| Ir1—C2—H2 | 114.2 | N1—C9—C10 | 178.4 (5) |
| C1—C2—Ir1 | 71.6 (3) | C9—C10—H10A | 109.5 |
| C1—C2—H2 | 114.2 | C9—C10—H10B | 109.5 |
| C1—C2—C3 | 122.5 (5) | C9—C10—H10C | 109.5 |
| C3—C2—Ir1 | 113.0 (3) | H10A—C10—H10B | 109.5 |
| C3—C2—H2 | 114.2 | H10A—C10—H10C | 109.5 |
| C2—C3—H3A | 108.6 | H10B—C10—H10C | 109.5 |
| | | | |
| Ir1—C1—C2—C3 | -106.1 (4) | C3—C4—C5—Ir1 | 32.7 (5) |
| Ir1—C1—C8—C7 | 27.6 (5) | C3—C4—C5—C6 | -47.3 (6) |
| Ir1—C2—C3—C4 | 10.1 (6) | C4—C5—C6—Ir1 | 101.7 (4) |
| Ir1—C5—C6—C7 | -105.8 (4) | C4—C5—C6—C7 | -4.2 (7) |
| Ir1—C6—C7—C8 | 3.1 (6) | C5—C6—C7—C8 | 87.1 (6) |
| C1—C2—C3—C4 | 92.3 (5) | C6—C7—C8—C1 | -20.8 (7) |
| C2—C1—C8—C7 | -54.0 (6) | C8—C1—C2—Ir1 | 103.8 (4) |
| C2—C3—C4—C5 | -29.0 (6) | C8—C1—C2—C3 | -2.3 (7) |