Pyrazine-bridged polymetallic Copper Iridium clusters

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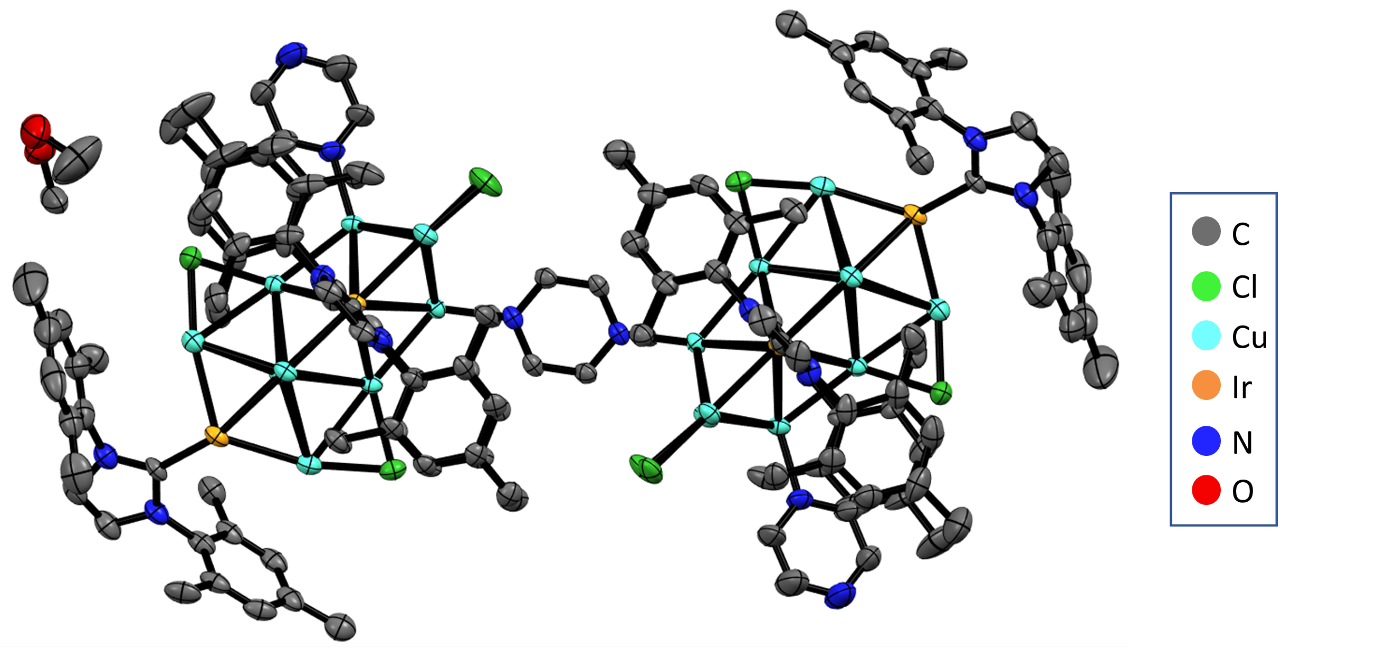
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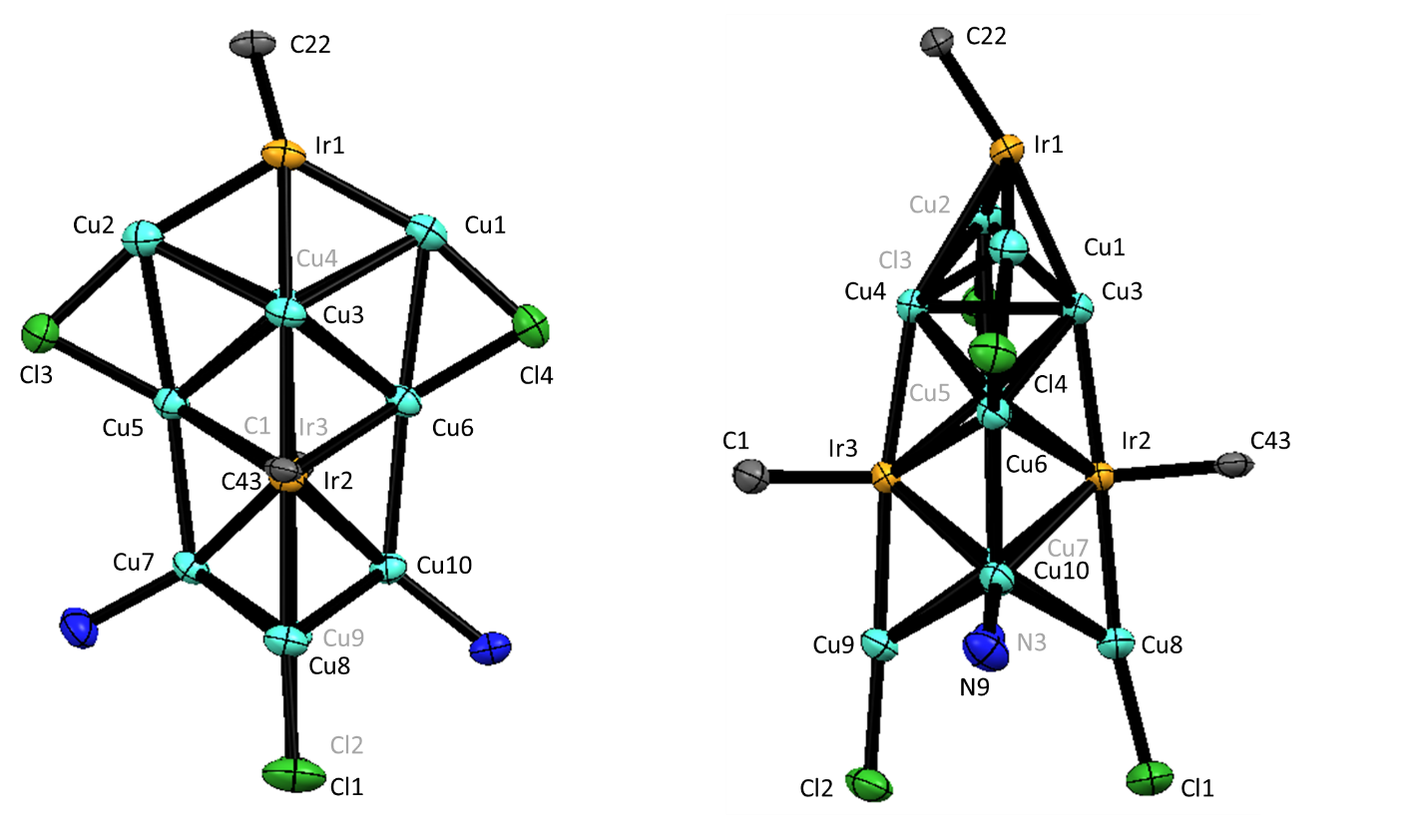
1. A single crystal X-ray structure for a unique heterometallic Cu20Ir6 cluster bridged by a pyrazine ligand is presented. Such clusters are rare with the polymetallic cores displaying unusual geometry, and such clusters may have future uses in materials science, catalysis, molecular storage, and catalysis.
2. Single crystals of a unique heterometallic Cu-Ir cluster have been prepared and the structure revealed using X-ray crystallography. The cluster is polymetallic with two Cu10Ir3cores bridged by a pyrazine ligand. Each polymetallic centre within the cluster contains three stabilising *N*-heterocyclic carbenes, four Cl ligands, and a non-bridging pyrazine. Notably, each Cu-Ir core is arranged in an unusual shape containing 13 vertices, 22 faces, and 32 sides. The atoms within each tridecametallic core are arranged in four planes, with 2, 4, 4, 3 metals in each plane. Ir atoms are present in alternate planes with an Ir site featuring in the peripheral bimetallic plane, and two Ir sites featuring on opposite sides of the non-adjacent tetrametallic plane. These clusters could have implications in materials chemistry, as metal organic frameworks often feature reactive sites bridged by linking pyrazine units. Furthermore, these clusters could exhibit interesting reactivity as short intermetallic distances show a high level of metalophilic interactions and such clusters could be suitable for electron transfer reactions and molecular capture and storage.
3. clusters; polymetallic; heterometallic; Cu; Ir; pyrazine
4. Chemical context

Polynuclear metallic clusters, particularly those featuring organic ligands, are highly important as they can appear as intermediates or decomposition products in many transition-metal catalysed reactions. Metallic clusters can also exhibit properties between monometallic transition metal complexes and higher order aggregates and nanoparticles. (Tang & Zhao, 2020) Therefore, their synthesis, preparation, and analysis is highly important to advance current understanding on how such species can play a role in catalysis. Metal clusters based on Cu are particularly exciting as a wide range of CuxXyLz clusters have been reported, where X is typically a halide or hydride, and, L is a thioester, phosphine, or *N*-heterocycle. (Harvey & Knorr, 2016; Dhayal *et al.*, 2016; Graham *et al.*, 2000; Liu & Astruc, 2018; Troyano *et al.*, 2021) There are also examples of heterometallic clusters containing Cu sites mixed with a range of other transition metals such as Re, Fe, Ir, Os, Co, Mo, W, Ag, and Au. (Sculfort & Braunstein, 2011; Croizat et al., 2016; Hau et al., 2016; Yip et al., 2007; Gao et al., 2024; Zhang et al., 2023) These mixed metal clusters provide a unique example to explore metalophilic interactions(Sculfort & Braunstein, 2011) and often have novel spectroscopic properties (Yip *et al.*, 2007; Zhang, Zhang *et al.*, 2023) or catalytic activity, (Gao *et al.*, 2024; Zhang, Zhang *et al.*, 2023) particularly as Cu complexes find many uses in carbon-carbon and carbon-heteroatom bond formation. To this end, we were able to prepare a single crystal of a novel heterometallic cluster containing two Cu10Ir3units bridged by a pyrazine ligand which was examined using X-ray diffraction studies (Figure 1).



1. X-ray crystal structure of [(Cu10Ir3Cl4(IMes)3(pyrazine))2(pyrazine)], with thermal ellipsoids given at 50% probability. Note that hydrogen atoms are omitted for clarity.
2. Structural commentary

The title compound [(Cu10Ir3Cl4(IMes)3(pyrazine))2(pyrazine)] (where IMes is 1,3-bis(2,4,6-trimethyl-phenyl)imidazol-2-ylidene) contains two tridecametallic Cu10Ir3units, each stabilised by four Cl ligands, three *N*-heterocyclic carbene (IMes) ligands, and a pyrazine ligand, with a bridging pyrazine linking two of these {Cu10Ir3Cl4(IMes)3(pyrazine)} units. Each Cu10Ir3core is arranged in a geometry containing 13 vertices, 22 faces, and 32 sides with the atoms arranged in four planes with 2, 4, 4 and 3 metals in each plane (Figure 2). The majority of the core consists of Cu, with two existing as naked atoms with only interactions to adjacent Cu and Ir. Of the remaining eight Cu sites, four are bonded to Cl ligands that bridge two Cu atoms across different atomic planes within the metallic core. Two of the three Cu atoms in a peripheral plane are bonded to terminal Cl, with the third ligated to a terminal pyrazine. Interestingly, a bridging pyrazine is bonded to a Cu in a tetrametallic plane and provides a link to another {Cu10Ir3Cl4(NHC)3(pyrazine)} unit, with the whole molecule having a centre of inversion in the middle of the bridging pyrazine ring. Within each core, Ir atoms are located in alternate planes with an Ir site featuring in the peripheral bimetallic plane, and two Ir sites featuring on opposite sides of the non-adjacent tetrametallic plane. This arrangement is likely a consequence of the bulky carbene ligand attached to Ir. The bond distances between the atoms within the tridecametallic core, and the ligands directly bound to them, are shown in Table 1. All 18 Cu-Cu distances range from 2.4916 to 3.0417 Å. All but three of these distances are shorter than the sum of the Cu van der Waals radii (2.80 Å) and most are close to the sum of the Cu atomic radii (2.556 Å) which suggest strong metalophilic interactions within the cluster. (Sculfort & Braunstein, 2011) There appears no significant differences between Cu-Cu and Ir-Cu bond lengths in the structure (2.66 ± 0.13, n = 18 and 2.62 ± 0.07, n = 16).

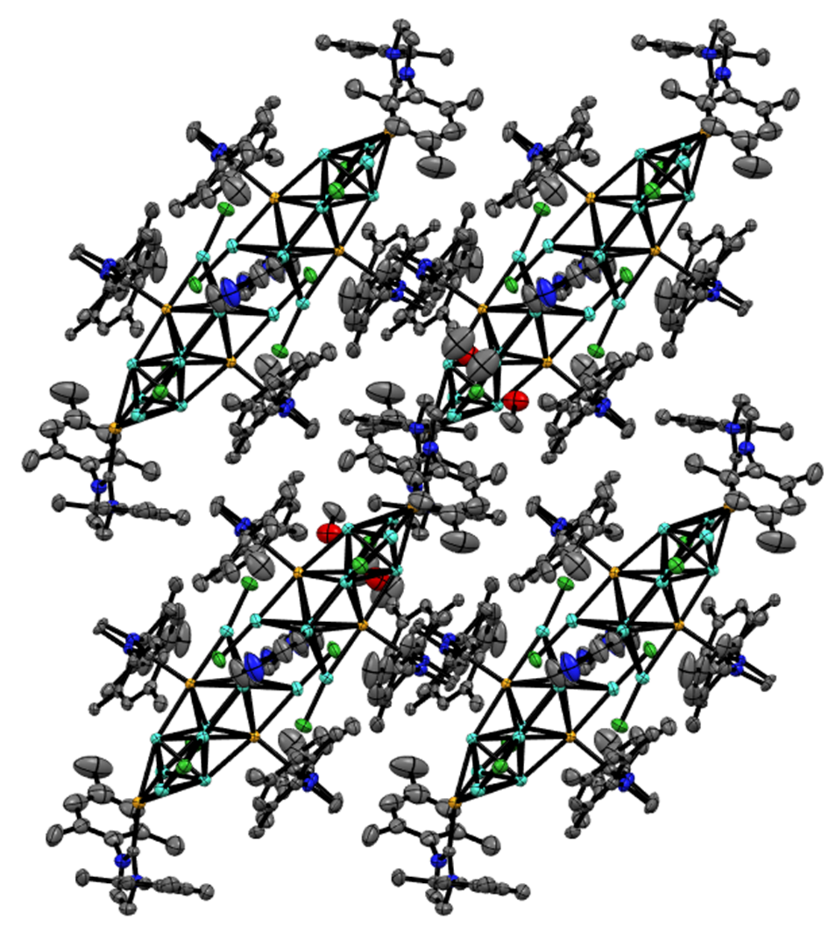


1. Tridecametallic cores of [(Cu10Ir3Cl4(IMes)3(pyrazine))2(pyrazine)], with thermal ellipsoids shown at 50% probability. Note that only the donor atoms of the ligands attached to the polyatomic core are shown. The cores are shown in two different orientations, rotated by 90o around the Ir1, Cu3, Cu4, Ir2, Ir3, Cu8, Cu9, Cu8, Cu9 plane. Note that atom labels marked in grey correspond to atoms hidden from view.
2. Key bond distances within {Cu10Ir3Cl4(IMes)3(pyrazine)} centres

|  |  |  |
| --- | --- | --- |
| Atom | Atom | Length/Å |
| C1 | Ir3 | 2.032(9) |
| C22 | Ir1 | 2.054(8) |
| C43 | Ir2 | 2.022(9) |
| Cl1 | Cu8 | 2.127(3) |
| Cl2 | Cu9 | 2.129(3) |
| Cl3 | Cu2 | 2.183(3) |
| Cl3 | Cu5 | 2.290(3) |
| Cl4 | Cu1 | 2.172(3) |
| Cl4 | Cu6 | 2.287(3) |
| Cu1 | Cu3 | 2.8138(19) |
| Cu1 | Cu4 | 2.716(2) |
| Cu1 | Cu6 | 2.6274(18) |
| Cu1 | Ir1 | 2.5227(15) |
| Cu2 | Cu3 | 2.716(2) |
| Cu2 | Cu4 | 2.8052(19) |
| Cu2 | Cu5 | 2.5877(18) |
| Cu2 | Ir1 | 2.5338(15) |
| Cu3 | Cu4 | 2.4916(18) |
| Cu3 | Cu5 | 2.6364(18) |
| Cu3 | Cu6 | 2.6145(18) |
| Cu3 | Ir1 | 2.6325(13) |
| Cu3 | Ir2 | 2.5665(13) |
| Cu4 | Cu5 | 2.5860(18) |
| Cu4 | Cu6 | 2.6325(18) |
| Cu4 | Ir1 | 2.7478(13) |
| Cu4 | Ir3 | 2.6337(13) |
| Cu5 | Cu7 | 2.5597(18) |
| Cu5 | Ir2 | 2.6856(13) |
| Cu5 | Ir3 | 2.6585(13) |
| Cu6 | Cu10 | 2.5899(17) |
| Cu6 | Ir2 | 2.6763(12) |
| Cu6 | Ir3 | 2.6559(13) |
| Cu7 | Cu8 | 2.5971(19) |
| Cu7 | Cu9 | 2.5615(19) |
| Cu7 | Cu10 | 3.0417(18) |
| Cu7 | Ir2 | 2.6089(13) |
| Cu7 | Ir3 | 2.6462(13) |
| Cu7 | N3 | 1.992(8) |
| Cu8 | Cu10 | 2.6155(18) |
| Cu8 | Ir2 | 2.5304(13) |
| Cu9 | Cu10 | 2.5802(18) |
| Cu9 | Ir3 | 2.5350(13) |
| Cu10 | Ir2 | 2.6378(13) |
| Cu10 | Ir3 | 2.6847(13) |
| Cu10 | N9 | 2.014(7) |
| Cu10 | N9 | 2.014(7) |

1. Supramolecular features

The methanol solvent clearly fills voids left by the packing of [(Cu10Ir3Cl4(IMes)3(pyrazine))2(pyrazine)] as the shortest interactions are between methanol and the three terminal CH3 groups of the IMes ligand. Long range interactions between the molecules of [(Cu10Ir3Cl4(IMes)3(pyrazine))2(pyrazine)] involve the non-bridging pyrazine ligands on adjacent molecules, with the shortest 2.327 Å interaction between the two H65 protons, and a 2.483 Å interaction between the free pyrazine N4 and the H65 proton of a non-bridging pyrazine on a neighbouring molecule. This suggests that the pyrazine ligand is important in both linking the two tridecametallic cores, and also packing the crystals together, which is unsurprising given its role in the formation of higher order polymers and metal organic frameworks. (Silva *et al.*, 2023; Zhang, Kong *et al.*, 2023; Kawamura *et al.*, 2017) Long range interactions between IMes ligands of different molecules are also important with distances of 2.377 Å and 2.383 Å between pairs of *ortho* CH3 and *para* CH3 on the mesityl rings of adjacent molecules (H19B/H41C and H20B and H42B). The crystal packing is shown in Figure 3.



1. Crystal packing of [(Cu10Ir3Cl4(IMes)3(pyrazine))2(pyrazine)] shown along the crystallographic c axis. Thermal ellipsoids are shown at 50% probability and hydrogen atoms are omitted for clarity.
2. Database survey

A search of the crystallographic database did not reveal any comparable tridecametallic polymetallic clusters. A few X-ray structures for pentatomic Cu-Ir clusters have been reported, but these contain cores with a trigonal bipyramidal geometry with either Cu3Ir2Lx (Rhodes *et al.*, 1985) or Ir4CuLx(Adams *et al.*, 2013) arrangements. Reported Cu-Ir distances are between 2.6628 and 2.79 Å, which are generally longer than those in the cluster presented here (2.5304 to 2.7478 Å). The short Cu-Ir distances suggest strong metal-metal interactions, and could indicate Cu=Ir bonds.(Rhodes *et al.*, 1985) There are many more examples of homometallic Cu clusters in the database, an analysis of 35 of these Cu-Cu bond lengths revealed an average intermetallic distance of 2.95 ± 0.25 Å (mean ± 1 standard deviation) which is consistent with the inter Cu distances in the structure reported here (2.66 ± 0.13 Å), albeit slightly longer.(Johnsson *et al.*, 2000; Rao *et al.*, 1983; Baumgartner *et al.*, 1990) Similar to short Cu-Ir distances, this suggests Cu-Cu interactions are also strong and metalophillic.

1. Synthesis and crystallization

The pyrazine-bridged polymetallic Cu Ir cluster was prepared by reaction of [Ir(Cl)(COD)(IMes)] (2.20 mg) (COD is *cis*,*cis*-1,5-cyclooctadiene and IMes is 1,3-bis(2,4,6-trimethyl-phenyl)imidazol-2-ylidene) with pyrazine (2.52 mg) and H2 (3 bar) in methanol-*d*4 (0.6 mL) for 3-4 hours at 298 K in a 5 mm NMR tube with a J. Youngs tap. At this point the pressure was released by opening the lid and Cu(OAc)2 (3.76 mg) in methanol-*d*4 (0.1 mL) was added to the solution. After being left for 1 hour at room temperature the solution was cooled to 278 K in a fridge for several weeks to form single crystals, which were found by X-ray diffraction to be the title compound.

1. Refinement

A suitable crystal was selected and mounted on an Oxford-Diffraction SuperNova dual-source X-ray diffractometer equipped with Cu and molybdenum sources and a HyPix-6000HE detector. Cooling to 110 K was achieved using an Oxford Instruments Cryojet. Using Olex2,12 the structure was solved with the SHELXT13 structure solution program using Intrinsic Phasing and refined with the SHELXL14 refinement package using Least Squares minimization. Details of the structural refinement and key parameters of the unit cell(s) are given in Table 2. The crystal contained disordered solvent methanol. One methanol was modelled in two positions with a common oxygen site in a refined ratio of 0.60:0.40(3). Another methanol was partially occupied with a refined occupancy of 0.59(2). There was additional solvent which was too disordered to model using discrete atoms. Therefore, a solvent mask was used which predicted a void with a volume of 430 cubic angstroms containing 66 electrons per unit cell. This would be equivalent to 3.67 methanol molecules. The crystal contains high residual density due to unresolved effects of the crystal having a minor twin present. Attempts to model this as two non-merohedral components were unsuccessful.

1. Key bond distances within {Cu10Ir3Cl4(IMes)3(pyrazine)} centres

|  |  |
| --- | --- |
| Empirical formula | C70.59H84.35Cl4Cu10Ir3N9O1.59 |
| Formula weight | 2438.08 |
| Temperature/K | 110.5(8) |
| Crystal system | triclinic |
| Space group | P-1 |
| a/Å | 12.5708(3) |
| b/Å | 14.8757(5) |
| c/Å | 23.1383(7) |
| α/° | 84.392(3) |
| β/° | 82.291(2) |
| γ/° | 85.686(2) |
| Volume/Å3 | 4258.8(2) |
| Z | 2 |
| ρcalcg/cm3 | 1.901 |
| μ/mm‑1 | 12.929 |
| F(000) | 2345.0 |
| Crystal size/mm3 | 0.153 × 0.117 × 0.074 |
| Radiation | Cu Kα (λ = 1.54184) |
| 2Θ range for data collection/° | 7.11 to 134.15 |
| Index ranges | -13 ≤ h ≤ 15, -17 ≤ k ≤ 17, -27 ≤ l ≤ 25 |
| Reflections collected | 45533 |
| Independent reflections | 15184 [Rint = 0.0455, Rsigma = 0.0440] |
| Data/restraints/parameters | 15184/0/919 |
| Goodness-of-fit on F2 | 1.084 |
| Final R indexes [I>=2σ (I)] | R1 = 0.0537, wR2 = 0.1306 |
| Final R indexes [all data] | R1 = 0.0599, wR2 = 0.1336 |
| Largest diff. peak/hole / e Å-3 | 3.01/-2.56 |

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Supporting information

1. Fractional Atomic Coordinates (×104) and Equivalent Isotropic Displacement Parameters (Å2×103) for sbd24013. Ueq is defined as 1/3 of the trace of the orthogonalised UIJ tensor.

| **Atom** | ***x*** | ***y*** | ***z*** | **U(eq)** |
| --- | --- | --- | --- | --- |
| C1 | 10318(7) | 3061(6) | 6817(4) | 35(2) |
| C2 | 11554(9) | 4028(7) | 6378(5) | 48(3) |
| C3 | 11621(8) | 3952(8) | 6941(5) | 50(3) |
| C4 | 10741(8) | 3107(7) | 7834(5) | 44(2) |
| C5 | 9968(8) | 3610(8) | 8182(5) | 47(3) |
| C6 | 9884(10) | 3365(10) | 8779(6) | 64(3) |
| C7 | 10488(12) | 2642(10) | 9020(5) | 67(4) |
| C8 | 11257(10) | 2196(10) | 8657(5) | 59(3) |
| C9 | 11393(9) | 2419(8) | 8060(5) | 49(3) |
| C10 | 12261(9) | 1914(8) | 7665(5) | 51(3) |
| C11 | 10327(18) | 2366(15) | 9680(7) | 111(7) |
| C12 | 9306(10) | 4388(9) | 7939(6) | 61(3) |
| C13 | 10378(7) | 3438(6) | 5732(4) | 36(2) |
| C14 | 9542(8) | 4051(6) | 5584(4) | 36(2) |
| C15 | 9159(8) | 3985(7) | 5062(5) | 45(3) |
| C16 | 9574(8) | 3347(7) | 4685(5) | 42(2) |
| C17 | 10410(8) | 2763(6) | 4834(4) | 38(2) |
| C18 | 10844(7) | 2791(6) | 5365(4) | 33(2) |
| C19 | 11739(8) | 2120(7) | 5515(4) | 41(2) |
| C20 | 9080(11) | 3269(9) | 4127(6) | 60(3) |
| C21 | 9017(9) | 4742(6) | 5987(5) | 46(3) |
| C22 | 5071(7) | 5250(6) | 7576(4) | 33(2) |
| C23 | 4786(9) | 6775(7) | 7541(6) | 53(3) |
| C24 | 4520(10) | 6458(8) | 8099(6) | 55(3) |
| C25 | 4475(11) | 4919(8) | 8646(5) | 56(3) |
| C26 | 3482(10) | 4575(8) | 8779(6) | 56(3) |
| C27 | 3347(16) | 3967(10) | 9276(8) | 93(6) |
| C28 | 4164(18) | 3720(11) | 9634(7) | 88(5) |
| C29 | 5119(15) | 4109(10) | 9491(6) | 78(4) |
| C30 | 5324(11) | 4720(9) | 9001(5) | 58(3) |
| C31 | 6401(11) | 5102(10) | 8842(7) | 71(4) |
| C32 | 3970(20) | 3010(14) | 10156(9) | 150(12) |
| C33 | 2617(12) | 4824(11) | 8403(8) | 82(5) |
| C34 | 5373(8) | 6118(6) | 6598(5) | 42(2) |
| C35 | 4557(8) | 6070(7) | 6251(5) | 45(3) |
| C36 | 4837(9) | 6099(7) | 5651(6) | 49(3) |
| C37 | 5893(10) | 6173(7) | 5396(5) | 50(3) |
| C38 | 6675(9) | 6261(6) | 5761(5) | 46(3) |
| C39 | 6451(8) | 6244(6) | 6357(5) | 41(2) |
| C40 | 7302(9) | 6340(8) | 6749(5) | 51(3) |
| C41 | 6179(11) | 6145(8) | 4747(6) | 60(3) |
| C42 | 3415(9) | 5954(8) | 6536(6) | 57(3) |
| C43 | 5943(7) | 14(6) | 7238(4) | 30.5(19) |
| C44 | 4553(7) | -818(6) | 7667(5) | 35(2) |
| C45 | 4444(7) | -675(5) | 7096(4) | 32.0(19) |
| C46 | 5480(7) | 158(6) | 6220(4) | 29.5(18) |
| C47 | 4926(7) | 967(6) | 6037(4) | 29.8(18) |
| C48 | 5177(7) | 1302(6) | 5460(4) | 32.2(19) |
| C49 | 5936(8) | 878(6) | 5071(4) | 36(2) |
| C50 | 6413(7) | 53(7) | 5266(4) | 36(2) |
| C51 | 6184(8) | -330(7) | 5833(4) | 38(2) |
| C52 | 6711(9) | -1237(7) | 6023(5) | 43(2) |
| C53 | 6235(8) | 1308(7) | 4459(4) | 42(2) |
| C54 | 4130(7) | 1455(6) | 6456(4) | 32.7(19) |
| C55 | 5841(8) | -404(7) | 8314(4) | 38(2) |
| C56 | 6601(8) | -1098(8) | 8483(5) | 51(3) |
| C57 | 6933(10) | -1070(11) | 9018(6) | 69(4) |
| C58 | 6599(11) | -406(13) | 9385(6) | 77(5) |
| C59 | 5836(10) | 241(10) | 9211(5) | 62(3) |
| C60 | 5452(8) | 276(8) | 8679(4) | 43(2) |
| C61 | 4611(9) | 975(7) | 8507(5) | 47(3) |
| C62 | 7038(13) | -410(17) | 9968(6) | 110(8) |
| C63 | 6958(9) | -1866(8) | 8097(6) | 62(3) |
| C64 | 9285(10) | 325(10) | 8800(5) | 61(3) |
| C65 | 9789(11) | 104(10) | 9287(5) | 66(4) |
| C66 | 11209(14) | -494(14) | 8739(7) | 94(6) |
| C67 | 10749(11) | -257(11) | 8246(6) | 73(4) |
| C68 | 9250(7) | 683(6) | 5015(4) | 31.0(19) |
| C69 | 9720(7) | 375(6) | 4489(4) | 34(2) |
| Cl1 | 9288(2) | -1896.2(18) | 6846.6(16) | 61.1(8) |
| Cl2 | 11829.6(19) | -126.4(18) | 6658.3(12) | 47.4(6) |
| Cl3 | 7183(2) | 2379.1(17) | 8786.4(10) | 43.5(5) |
| Cl4 | 6845(2) | 2993.8(16) | 5522.8(10) | 40.0(5) |
| Cu1 | 6097.6(12) | 3617.1(9) | 6300.1(6) | 38.5(3) |
| Cu2 | 6152.3(12) | 3136.3(9) | 8196.4(6) | 38.8(3) |
| Cu3 | 6037.1(10) | 2254.2(8) | 7235.0(6) | 30.9(3) |
| Cu4 | 7472.5(10) | 3349.0(8) | 7120.4(6) | 31.4(3) |
| Cu5 | 7654.1(10) | 1942.6(9) | 7859.6(6) | 30.6(3) |
| Cu6 | 7492.1(10) | 2210.4(8) | 6316.1(5) | 29.5(3) |
| Cu7 | 9000.3(10) | 624.3(9) | 7591.1(6) | 31.4(3) |
| Cu8 | 8469.7(11) | -616.9(9) | 6987.3(6) | 36.0(3) |
| Cu9 | 10576.3(10) | 907.7(9) | 6785.1(6) | 35.4(3) |
| Cu10 | 8879.4(10) | 832.3(8) | 6280.2(5) | 29.1(3) |
| Ir1 | 5349.0(3) | 3955.9(3) | 7328.5(2) | 34.02(11) |
| Ir2 | 7223.9(3) | 787.3(2) | 7111.7(2) | 25.03(10) |
| Ir3 | 9133.6(3) | 2179.1(2) | 6932.1(2) | 26.33(10) |
| N1 | 10875(6) | 3350(6) | 7213(4) | 39.3(19) |
| N2 | 10739(6) | 3475(5) | 6283(4) | 35.8(17) |
| N3 | 9707(7) | 97(6) | 8279(4) | 44(2) |
| N4 | 10731(10) | -340(11) | 9270(5) | 84(4) |
| N5 | 4716(7) | 5509(6) | 8115(4) | 45(2) |
| N6 | 5108(7) | 6043(5) | 7225(4) | 42.0(19) |
| N7 | 5481(6) | -404(5) | 7756(3) | 33.9(17) |
| N8 | 5311(5) | -165(5) | 6833(3) | 28.3(15) |
| N9 | 9528(6) | 319(5) | 5529(3) | 30.6(16) |
| O1 | 6658(10) | 2014(8) | 11082(5) | 93(4) |
| C71 | 7890(20) | 3806(18) | 10273(10) | 79(9) |
| O2 | 8081(17) | 3229(12) | 10769(7) | 79(6) |
| C70 | 6370(40) | 1690(30) | 10558(15) | 142(14) |
| C72 | 7110(60) | 2270(50) | 10470(20) | 142(14) |

1. Anisotropic Displacement Parameters (Å2×103) for sbd24013. The Anisotropic displacement factor exponent takes the form: -2π2[h2a\*2U11+2hka\*b\*U12+…].

|  | | | | | | |
| --- | --- | --- | --- | --- | --- | --- |
| **Atom** | **U11** | **U22** | **U33** | **U23** | **U13** | **U12** |
| C1 | 29(5) | 34(5) | 42(5) | -7(4) | -6(4) | 1(4) |
| C2 | 36(6) | 45(6) | 64(7) | -5(5) | 4(5) | -24(5) |
| C3 | 24(5) | 71(8) | 57(7) | -16(6) | 1(4) | -14(5) |
| C4 | 38(6) | 50(6) | 48(6) | -22(5) | 0(4) | -14(5) |
| C5 | 29(5) | 63(7) | 52(6) | -21(5) | -2(4) | -9(5) |
| C6 | 45(7) | 87(10) | 61(8) | -31(7) | 2(6) | -2(7) |
| C7 | 78(9) | 83(9) | 43(6) | -19(6) | -2(6) | -19(8) |
| C8 | 52(7) | 76(8) | 50(7) | -5(6) | -17(5) | 6(6) |
| C9 | 33(5) | 64(7) | 54(6) | -13(5) | -13(5) | -5(5) |
| C10 | 42(6) | 59(7) | 55(6) | -19(5) | -12(5) | 5(5) |
| C11 | 131(17) | 143(18) | 49(8) | -9(10) | 6(9) | 26(14) |
| C12 | 54(7) | 60(7) | 75(8) | -32(6) | -19(6) | 6(6) |
| C13 | 29(5) | 31(5) | 45(5) | 1(4) | 1(4) | -7(4) |
| C14 | 31(5) | 22(4) | 51(6) | 8(4) | 7(4) | -9(4) |
| C15 | 31(5) | 39(5) | 58(6) | 14(5) | 1(5) | 3(4) |
| C16 | 39(6) | 33(5) | 54(6) | 9(4) | -9(5) | -13(4) |
| C17 | 37(5) | 32(5) | 44(5) | -2(4) | -1(4) | -8(4) |
| C18 | 29(5) | 23(4) | 45(5) | 6(4) | 4(4) | -9(4) |
| C19 | 35(5) | 42(5) | 43(5) | -1(4) | -2(4) | 1(4) |
| C20 | 64(8) | 56(7) | 62(7) | 0(6) | -21(6) | 1(6) |
| C21 | 43(6) | 26(5) | 67(7) | 2(5) | 2(5) | -5(4) |
| C22 | 26(4) | 20(4) | 52(5) | -7(4) | -1(4) | 0(3) |
| C23 | 50(7) | 32(5) | 77(8) | -11(5) | -2(6) | -2(5) |
| C24 | 53(7) | 39(6) | 69(8) | -12(5) | 0(6) | 13(5) |
| C25 | 66(8) | 46(6) | 54(7) | -17(5) | 9(6) | -1(6) |
| C26 | 56(7) | 43(6) | 64(7) | -14(5) | 17(6) | -10(5) |
| C27 | 106(14) | 57(9) | 98(12) | -16(8) | 57(11) | -14(9) |
| C28 | 117(15) | 70(10) | 67(10) | -11(8) | 19(10) | 6(10) |
| C29 | 108(13) | 66(9) | 59(8) | -20(7) | -7(8) | 12(9) |
| C30 | 70(8) | 53(7) | 53(7) | -12(6) | -11(6) | 5(6) |
| C31 | 63(9) | 68(9) | 85(10) | -13(7) | -19(7) | -2(7) |
| C32 | 250(30) | 88(14) | 83(13) | 22(11) | 48(17) | 19(17) |
| C33 | 53(8) | 87(11) | 105(12) | -22(9) | 14(8) | -25(8) |
| C34 | 31(5) | 27(5) | 64(7) | 5(4) | 3(5) | -3(4) |
| C35 | 32(5) | 29(5) | 70(7) | 11(5) | -5(5) | -1(4) |
| C36 | 40(6) | 34(5) | 76(8) | 9(5) | -23(5) | -7(4) |
| C37 | 54(7) | 27(5) | 65(7) | 5(5) | 0(6) | 4(5) |
| C38 | 39(6) | 27(5) | 67(7) | 6(5) | 4(5) | 0(4) |
| C39 | 37(5) | 19(4) | 65(7) | 2(4) | -2(5) | -4(4) |
| C40 | 41(6) | 42(6) | 68(7) | -7(5) | 1(5) | -5(5) |
| C41 | 61(8) | 47(7) | 71(8) | 1(6) | -9(6) | -7(6) |
| C42 | 33(6) | 53(7) | 85(9) | 16(6) | -21(6) | -6(5) |
| C43 | 29(5) | 23(4) | 36(5) | 3(3) | -1(4) | 10(3) |
| C44 | 23(4) | 24(4) | 57(6) | 4(4) | -6(4) | -11(3) |
| C45 | 28(5) | 18(4) | 49(5) | 4(4) | -4(4) | -11(3) |
| C46 | 25(4) | 24(4) | 41(5) | -3(4) | -9(4) | -1(3) |
| C47 | 16(4) | 33(5) | 43(5) | -7(4) | -10(4) | -5(3) |
| C48 | 25(4) | 33(5) | 40(5) | -6(4) | -10(4) | 0(4) |
| C49 | 34(5) | 36(5) | 40(5) | -8(4) | -11(4) | -9(4) |
| C50 | 24(4) | 43(5) | 43(5) | -11(4) | -10(4) | -6(4) |
| C51 | 30(5) | 37(5) | 50(6) | -18(4) | -12(4) | 0(4) |
| C52 | 43(6) | 34(5) | 56(6) | -14(4) | -20(5) | 11(4) |
| C53 | 36(5) | 50(6) | 43(5) | -6(5) | -8(4) | -7(5) |
| C54 | 21(4) | 26(4) | 49(5) | -3(4) | 1(4) | -2(3) |
| C55 | 32(5) | 37(5) | 44(5) | 13(4) | -6(4) | -11(4) |
| C56 | 32(5) | 59(7) | 61(7) | 35(6) | -16(5) | -21(5) |
| C57 | 46(7) | 90(10) | 63(8) | 41(8) | -10(6) | -13(7) |
| C58 | 52(8) | 138(15) | 40(7) | 39(8) | -20(6) | -34(9) |
| C59 | 50(7) | 98(10) | 35(6) | 5(6) | 8(5) | -27(7) |
| C60 | 40(6) | 53(6) | 34(5) | 9(4) | 2(4) | -17(5) |
| C61 | 43(6) | 44(6) | 50(6) | -5(5) | 12(5) | -11(5) |
| C62 | 59(9) | 220(20) | 55(8) | 21(11) | -27(7) | -38(12) |
| C63 | 36(6) | 47(7) | 99(10) | 20(7) | -13(6) | -2(5) |
| C64 | 53(7) | 84(9) | 42(6) | 9(6) | -13(5) | 20(6) |
| C65 | 60(8) | 97(10) | 39(6) | -9(6) | -9(5) | 21(7) |
| C66 | 74(10) | 139(16) | 61(9) | 4(9) | -15(8) | 37(10) |
| C67 | 54(8) | 103(11) | 54(7) | -1(7) | -12(6) | 40(8) |
| C68 | 23(4) | 27(4) | 45(5) | -11(4) | -6(4) | -1(3) |
| C69 | 27(5) | 36(5) | 38(5) | -1(4) | -3(4) | 1(4) |
| Cl1 | 45.7(15) | 33.1(13) | 103(2) | -8.3(14) | -8.4(15) | 9.3(11) |
| Cl2 | 29.9(12) | 51.0(14) | 63.1(15) | -16.5(12) | -12.1(11) | 11.8(10) |
| Cl3 | 44.8(13) | 49.8(13) | 34.0(11) | -3.6(10) | -2.4(10) | 4.3(11) |
| Cl4 | 42.8(13) | 39.3(12) | 38.2(11) | 5.7(9) | -10.8(10) | -6.9(10) |
| Cu1 | 37.4(8) | 31.5(7) | 46.6(8) | 2.3(6) | -9.9(6) | -0.5(6) |
| Cu2 | 36.0(8) | 35.7(7) | 43.5(8) | -3.5(6) | -1.9(6) | 0.1(6) |
| Cu3 | 24.2(6) | 23.9(6) | 43.8(7) | 0.3(5) | -4.1(5) | -2.1(5) |
| Cu4 | 26.6(7) | 25.7(6) | 41.2(7) | -0.6(5) | -2.2(5) | -3.4(5) |
| Cu5 | 27.2(7) | 30.8(6) | 33.1(6) | -1.3(5) | -3.4(5) | -0.8(5) |
| Cu6 | 26.6(6) | 27.2(6) | 34.2(7) | 2.3(5) | -5.0(5) | -2.7(5) |
| Cu7 | 25.2(6) | 32.1(7) | 37.3(7) | 1.6(5) | -9.1(5) | -0.9(5) |
| Cu8 | 30.5(7) | 27.2(6) | 49.6(8) | -1.2(6) | -6.6(6) | 2.4(5) |
| Cu9 | 23.9(7) | 36.4(7) | 45.6(8) | -5.7(6) | -5.5(6) | 3.5(5) |
| Cu10 | 23.8(6) | 28.4(6) | 34.4(7) | -3.5(5) | -0.9(5) | -1.6(5) |
| Ir1 | 28.1(2) | 23.77(19) | 49.9(2) | -1.82(16) | -5.44(17) | -0.47(15) |
| Ir2 | 20.62(18) | 22.20(17) | 31.70(19) | 1.61(13) | -3.76(14) | -2.22(13) |
| Ir3 | 20.07(18) | 26.05(18) | 32.78(19) | -2.04(14) | -2.77(14) | -2.77(14) |
| N1 | 30(4) | 38(4) | 50(5) | -7(4) | 3(4) | -12(3) |
| N2 | 25(4) | 35(4) | 47(5) | -5(3) | 2(3) | -7(3) |
| N3 | 36(5) | 56(5) | 39(5) | 2(4) | -13(4) | 4(4) |
| N4 | 64(7) | 137(12) | 46(6) | -1(7) | -15(5) | 28(8) |
| N5 | 43(5) | 35(4) | 57(5) | -8(4) | -3(4) | -2(4) |
| N6 | 40(5) | 27(4) | 57(5) | -4(4) | -1(4) | -1(3) |
| N7 | 33(4) | 30(4) | 37(4) | 3(3) | -2(3) | -5(3) |
| N8 | 15(3) | 23(3) | 47(4) | 3(3) | -7(3) | -4(3) |
| N9 | 25(4) | 26(4) | 41(4) | -7(3) | 0(3) | -3(3) |
| O1 | 110(9) | 93(8) | 66(6) | -1(6) | 12(6) | 13(7) |
| C71 | 86(18) | 75(16) | 61(14) | 8(12) | 15(13) | 32(14) |
| O2 | 109(16) | 70(11) | 63(10) | -8(8) | -26(10) | -2(10) |
| C70 | 190(40) | 180(40) | 75(17) | 10(20) | -40(20) | -70(30) |
| C72 | 190(40) | 180(40) | 75(17) | 10(20) | -40(20) | -70(30) |

1. Bond Lengths

| **Atom** | **Atom** | **Length/Å** |  | **Atom** | **Atom** | **Length/Å** |
| --- | --- | --- | --- | --- | --- | --- |
| C1 | Ir3 | 2.032(9) |  | C50 | C51 | 1.382(14) |
| C1 | N1 | 1.345(13) |  | C51 | C52 | 1.509(13) |
| C1 | N2 | 1.381(12) |  | C55 | C56 | 1.416(15) |
| C2 | C3 | 1.311(16) |  | C55 | C60 | 1.399(16) |
| C2 | N2 | 1.414(13) |  | C55 | N7 | 1.424(13) |
| C3 | N1 | 1.391(13) |  | C56 | C57 | 1.363(19) |
| C4 | C5 | 1.396(15) |  | C56 | C63 | 1.523(19) |
| C4 | C9 | 1.371(17) |  | C57 | C58 | 1.37(2) |
| C4 | N1 | 1.439(14) |  | C58 | C59 | 1.38(2) |
| C5 | C6 | 1.386(18) |  | C58 | C62 | 1.523(18) |
| C5 | C12 | 1.484(17) |  | C59 | C60 | 1.377(16) |
| C6 | C7 | 1.39(2) |  | C60 | C61 | 1.491(16) |
| C7 | C8 | 1.369(19) |  | C64 | C65 | 1.366(16) |
| C7 | C11 | 1.531(19) |  | C64 | N3 | 1.316(15) |
| C8 | C9 | 1.379(17) |  | C65 | N4 | 1.309(17) |
| C9 | C10 | 1.529(15) |  | C66 | C67 | 1.350(19) |
| C13 | C14 | 1.395(14) |  | C66 | N4 | 1.328(19) |
| C13 | C18 | 1.389(14) |  | C67 | N3 | 1.370(15) |
| C13 | N2 | 1.418(13) |  | C68 | C69 | 1.384(13) |
| C14 | C15 | 1.374(16) |  | C68 | N9 | 1.337(12) |
| C14 | C21 | 1.509(14) |  | C69 | N91 | 1.345(12) |
| C15 | C16 | 1.377(16) |  | Cl1 | Cu8 | 2.127(3) |
| C16 | C17 | 1.371(14) |  | Cl2 | Cu9 | 2.129(3) |
| C16 | C20 | 1.524(16) |  | Cl3 | Cu2 | 2.183(3) |
| C17 | C18 | 1.414(14) |  | Cl3 | Cu5 | 2.290(3) |
| C18 | C19 | 1.500(13) |  | Cl4 | Cu1 | 2.172(3) |
| C22 | Ir1 | 2.054(8) |  | Cl4 | Cu6 | 2.287(3) |
| C22 | N5 | 1.350(13) |  | Cu1 | Cu3 | 2.8138(19) |
| C22 | N6 | 1.366(12) |  | Cu1 | Cu4 | 2.716(2) |
| C23 | C24 | 1.338(18) |  | Cu1 | Cu6 | 2.6274(18) |
| C23 | N6 | 1.377(14) |  | Cu1 | Ir1 | 2.5227(15) |
| C24 | N5 | 1.413(14) |  | Cu2 | Cu3 | 2.716(2) |
| C25 | C26 | 1.370(18) |  | Cu2 | Cu4 | 2.8052(19) |
| C25 | C30 | 1.429(19) |  | Cu2 | Cu5 | 2.5877(18) |
| C25 | N5 | 1.449(15) |  | Cu2 | Ir1 | 2.5338(15) |
| C26 | C27 | 1.39(2) |  | Cu3 | Cu4 | 2.4916(18) |
| C26 | C33 | 1.49(2) |  | Cu3 | Cu5 | 2.6364(18) |
| C27 | C28 | 1.41(3) |  | Cu3 | Cu6 | 2.6145(18) |
| C28 | C29 | 1.36(3) |  | Cu3 | Ir1 | 2.6325(13) |
| C28 | C32 | 1.53(2) |  | Cu3 | Ir2 | 2.5665(13) |
| C29 | C30 | 1.39(2) |  | Cu4 | Cu5 | 2.5860(18) |
| C30 | C31 | 1.49(2) |  | Cu4 | Cu6 | 2.6325(18) |
| C34 | C35 | 1.393(16) |  | Cu4 | Ir1 | 2.7478(13) |
| C34 | C39 | 1.413(14) |  | Cu4 | Ir3 | 2.6337(13) |
| C34 | N6 | 1.440(14) |  | Cu5 | Cu7 | 2.5597(18) |
| C35 | C36 | 1.384(17) |  | Cu5 | Ir2 | 2.6856(13) |
| C35 | C42 | 1.513(15) |  | Cu5 | Ir3 | 2.6585(13) |
| C36 | C37 | 1.387(16) |  | Cu6 | Cu10 | 2.5899(17) |
| C37 | C38 | 1.400(17) |  | Cu6 | Ir2 | 2.6763(12) |
| C37 | C41 | 1.499(18) |  | Cu6 | Ir3 | 2.6559(13) |
| C38 | C39 | 1.368(16) |  | Cu7 | Cu8 | 2.5971(19) |
| C39 | C40 | 1.517(16) |  | Cu7 | Cu9 | 2.5615(19) |
| C43 | Ir2 | 2.022(9) |  | Cu7 | Cu10 | 3.0417(18) |
| C43 | N7 | 1.372(11) |  | Cu7 | Ir2 | 2.6089(13) |
| C43 | N8 | 1.363(12) |  | Cu7 | Ir3 | 2.6462(13) |
| C44 | C45 | 1.340(14) |  | Cu7 | N3 | 1.992(8) |
| C44 | N7 | 1.408(12) |  | Cu8 | Cu10 | 2.6155(18) |
| C45 | N8 | 1.412(11) |  | Cu8 | Ir2 | 2.5304(13) |
| C46 | C47 | 1.401(12) |  | Cu9 | Cu10 | 2.5802(18) |
| C46 | C51 | 1.389(13) |  | Cu9 | Ir3 | 2.5350(13) |
| C46 | N8 | 1.446(12) |  | Cu10 | Ir2 | 2.6378(13) |
| C47 | C48 | 1.382(13) |  | Cu10 | Ir3 | 2.6847(13) |
| C47 | C54 | 1.495(12) |  | Cu10 | N9 | 2.014(7) |
| C48 | C49 | 1.382(13) |  | O1 | C70 | 1.45(3) |
| C49 | C50 | 1.387(14) |  | O1 | C72 | 1.48(5) |
| C49 | C53 | 1.506(14) |  | C71 | O2 | 1.40(3) |

1. Bond Angles

| **Atom** | **Atom** | **Atom** | **Angle/˚** |  | **Atom** | **Atom** | **Atom** | **Angle/˚** |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| N1 | C1 | Ir3 | 129.6(7) |  | Cl3 | Cu5 | Cu4 | 108.49(8) |
| N1 | C1 | N2 | 105.5(8) |  | Cl3 | Cu5 | Cu7 | 124.87(9) |
| N2 | C1 | Ir3 | 124.9(7) |  | Cl3 | Cu5 | Ir2 | 143.50(9) |
| C3 | C2 | N2 | 107.6(9) |  | Cl3 | Cu5 | Ir3 | 141.38(9) |
| C2 | C3 | N1 | 108.1(10) |  | Cu2 | Cu5 | Cu3 | 62.64(5) |
| C5 | C4 | N1 | 117.9(10) |  | Cu2 | Cu5 | Ir2 | 117.95(6) |
| C9 | C4 | C5 | 123.0(11) |  | Cu2 | Cu5 | Ir3 | 125.23(6) |
| C9 | C4 | N1 | 119.1(9) |  | Cu3 | Cu5 | Ir2 | 57.65(4) |
| C4 | C5 | C12 | 122.8(11) |  | Cu3 | Cu5 | Ir3 | 93.50(5) |
| C6 | C5 | C4 | 115.9(11) |  | Cu4 | Cu5 | Cu2 | 65.67(5) |
| C6 | C5 | C12 | 121.3(11) |  | Cu4 | Cu5 | Cu3 | 56.98(5) |
| C7 | C6 | C5 | 122.7(11) |  | Cu4 | Cu5 | Ir2 | 93.78(5) |
| C6 | C7 | C11 | 120.9(13) |  | Cu4 | Cu5 | Ir3 | 60.27(4) |
| C8 | C7 | C6 | 118.3(12) |  | Cu7 | Cu5 | Cu2 | 173.38(7) |
| C8 | C7 | C11 | 120.7(15) |  | Cu7 | Cu5 | Cu3 | 116.42(6) |
| C7 | C8 | C9 | 121.5(12) |  | Cu7 | Cu5 | Cu4 | 119.86(6) |
| C4 | C9 | C8 | 118.4(11) |  | Cu7 | Cu5 | Ir2 | 59.59(4) |
| C4 | C9 | C10 | 121.2(10) |  | Cu7 | Cu5 | Ir3 | 60.91(4) |
| C8 | C9 | C10 | 120.4(11) |  | Ir3 | Cu5 | Ir2 | 74.79(3) |
| C14 | C13 | N2 | 117.8(9) |  | Cl4 | Cu6 | Cu1 | 51.90(7) |
| C18 | C13 | C14 | 122.4(10) |  | Cl4 | Cu6 | Cu3 | 110.10(8) |
| C18 | C13 | N2 | 119.7(9) |  | Cl4 | Cu6 | Cu4 | 107.57(8) |
| C13 | C14 | C21 | 122.6(10) |  | Cl4 | Cu6 | Cu10 | 125.49(9) |
| C15 | C14 | C13 | 117.7(9) |  | Cl4 | Cu6 | Ir2 | 144.79(8) |
| C15 | C14 | C21 | 119.6(9) |  | Cl4 | Cu6 | Ir3 | 140.09(8) |
| C14 | C15 | C16 | 122.5(9) |  | Cu1 | Cu6 | Cu4 | 62.18(5) |
| C15 | C16 | C20 | 120.5(10) |  | Cu1 | Cu6 | Ir2 | 122.02(6) |
| C17 | C16 | C15 | 118.7(10) |  | Cu1 | Cu6 | Ir3 | 120.01(6) |
| C17 | C16 | C20 | 120.8(10) |  | Cu3 | Cu6 | Cu1 | 64.93(5) |
| C16 | C17 | C18 | 121.9(9) |  | Cu3 | Cu6 | Cu4 | 56.70(5) |
| C13 | C18 | C17 | 116.7(9) |  | Cu3 | Cu6 | Ir2 | 58.02(4) |
| C13 | C18 | C19 | 123.4(9) |  | Cu3 | Cu6 | Ir3 | 94.07(5) |
| C17 | C18 | C19 | 119.8(9) |  | Cu4 | Cu6 | Ir2 | 92.94(5) |
| N5 | C22 | Ir1 | 127.8(7) |  | Cu4 | Cu6 | Ir3 | 59.74(4) |
| N5 | C22 | N6 | 104.1(8) |  | Cu10 | Cu6 | Cu1 | 177.38(7) |
| N6 | C22 | Ir1 | 127.8(7) |  | Cu10 | Cu6 | Cu3 | 117.42(6) |
| C24 | C23 | N6 | 107.5(10) |  | Cu10 | Cu6 | Cu4 | 119.86(6) |
| C23 | C24 | N5 | 105.9(10) |  | Cu10 | Cu6 | Ir2 | 60.09(4) |
| C26 | C25 | C30 | 123.6(12) |  | Cu10 | Cu6 | Ir3 | 61.55(4) |
| C26 | C25 | N5 | 120.1(12) |  | Ir3 | Cu6 | Ir2 | 74.99(3) |
| C30 | C25 | N5 | 116.3(11) |  | Cu5 | Cu7 | Cu8 | 120.17(6) |
| C25 | C26 | C27 | 116.1(15) |  | Cu5 | Cu7 | Cu9 | 119.11(6) |
| C25 | C26 | C33 | 121.4(12) |  | Cu5 | Cu7 | Cu10 | 97.15(5) |
| C27 | C26 | C33 | 122.5(14) |  | Cu5 | Cu7 | Ir2 | 62.60(4) |
| C26 | C27 | C28 | 123.0(16) |  | Cu5 | Cu7 | Ir3 | 61.39(4) |
| C27 | C28 | C32 | 120(2) |  | Cu8 | Cu7 | Cu10 | 54.58(5) |
| C29 | C28 | C27 | 118.2(16) |  | Cu8 | Cu7 | Ir2 | 58.16(4) |
| C29 | C28 | C32 | 122(2) |  | Cu8 | Cu7 | Ir3 | 109.79(6) |
| C28 | C29 | C30 | 122.4(17) |  | Cu9 | Cu7 | Cu8 | 87.55(6) |
| C25 | C30 | C31 | 122.4(12) |  | Cu9 | Cu7 | Cu10 | 54.01(5) |
| C29 | C30 | C25 | 116.5(14) |  | Cu9 | Cu7 | Ir2 | 108.44(6) |
| C29 | C30 | C31 | 121.0(14) |  | Cu9 | Cu7 | Ir3 | 58.23(4) |
| C35 | C34 | C39 | 122.5(10) |  | Ir2 | Cu7 | Cu10 | 55.01(3) |
| C35 | C34 | N6 | 118.9(9) |  | Ir2 | Cu7 | Ir3 | 76.28(3) |
| C39 | C34 | N6 | 118.7(10) |  | Ir3 | Cu7 | Cu10 | 55.81(3) |
| C34 | C35 | C42 | 119.9(11) |  | N3 | Cu7 | Cu5 | 111.3(3) |
| C36 | C35 | C34 | 118.0(10) |  | N3 | Cu7 | Cu8 | 112.0(3) |
| C36 | C35 | C42 | 122.1(11) |  | N3 | Cu7 | Cu9 | 103.8(3) |
| C35 | C36 | C37 | 121.6(11) |  | N3 | Cu7 | Cu10 | 150.8(3) |
| C36 | C37 | C38 | 118.1(11) |  | N3 | Cu7 | Ir2 | 145.4(3) |
| C36 | C37 | C41 | 120.2(12) |  | N3 | Cu7 | Ir3 | 133.3(3) |
| C38 | C37 | C41 | 121.6(11) |  | Cl1 | Cu8 | Cu7 | 127.85(11) |
| C39 | C38 | C37 | 123.1(10) |  | Cl1 | Cu8 | Cu10 | 122.14(11) |
| C34 | C39 | C40 | 120.7(10) |  | Cl1 | Cu8 | Ir2 | 170.41(11) |
| C38 | C39 | C34 | 116.5(10) |  | Cu7 | Cu8 | Cu10 | 71.40(5) |
| C38 | C39 | C40 | 122.8(10) |  | Ir2 | Cu8 | Cu7 | 61.15(4) |
| N7 | C43 | Ir2 | 127.5(7) |  | Ir2 | Cu8 | Cu10 | 61.65(4) |
| N8 | C43 | Ir2 | 127.6(6) |  | Cl2 | Cu9 | Cu7 | 118.91(10) |
| N8 | C43 | N7 | 104.8(8) |  | Cl2 | Cu9 | Cu10 | 118.20(10) |
| C45 | C44 | N7 | 107.8(8) |  | Cl2 | Cu9 | Ir3 | 177.99(10) |
| C44 | C45 | N8 | 106.4(8) |  | Cu7 | Cu9 | Cu10 | 72.54(5) |
| C47 | C46 | N8 | 118.2(8) |  | Ir3 | Cu9 | Cu7 | 62.56(4) |
| C51 | C46 | C47 | 122.2(9) |  | Ir3 | Cu9 | Cu10 | 63.31(4) |
| C51 | C46 | N8 | 119.6(8) |  | Cu6 | Cu10 | Cu7 | 94.99(5) |
| C46 | C47 | C54 | 121.2(8) |  | Cu6 | Cu10 | Cu8 | 118.77(6) |
| C48 | C47 | C46 | 116.9(8) |  | Cu6 | Cu10 | Ir2 | 61.58(4) |
| C48 | C47 | C54 | 121.9(8) |  | Cu6 | Cu10 | Ir3 | 60.43(4) |
| C49 | C48 | C47 | 122.9(9) |  | Cu8 | Cu10 | Cu7 | 54.02(5) |
| C48 | C49 | C50 | 117.7(9) |  | Cu8 | Cu10 | Ir2 | 57.59(4) |
| C48 | C49 | C53 | 120.7(9) |  | Cu8 | Cu10 | Ir3 | 108.06(5) |
| C50 | C49 | C53 | 121.7(9) |  | Cu9 | Cu10 | Cu6 | 117.58(6) |
| C51 | C50 | C49 | 122.4(9) |  | Cu9 | Cu10 | Cu7 | 53.45(5) |
| C46 | C51 | C52 | 121.9(9) |  | Cu9 | Cu10 | Cu8 | 86.77(6) |
| C50 | C51 | C46 | 117.6(9) |  | Cu9 | Cu10 | Ir2 | 107.00(5) |
| C50 | C51 | C52 | 120.5(9) |  | Cu9 | Cu10 | Ir3 | 57.52(4) |
| C56 | C55 | N7 | 118.5(10) |  | Ir2 | Cu10 | Cu7 | 54.13(3) |
| C60 | C55 | C56 | 121.5(10) |  | Ir2 | Cu10 | Ir3 | 75.14(3) |
| C60 | C55 | N7 | 120.0(9) |  | Ir3 | Cu10 | Cu7 | 54.62(3) |
| C55 | C56 | C63 | 120.5(10) |  | N9 | Cu10 | Cu6 | 123.1(2) |
| C57 | C56 | C55 | 116.8(13) |  | N9 | Cu10 | Cu7 | 141.8(2) |
| C57 | C56 | C63 | 122.6(12) |  | N9 | Cu10 | Cu8 | 102.9(2) |
| C56 | C57 | C58 | 124.1(13) |  | N9 | Cu10 | Cu9 | 100.7(2) |
| C57 | C58 | C59 | 117.2(12) |  | N9 | Cu10 | Ir2 | 144.2(2) |
| C57 | C58 | C62 | 120.7(16) |  | N9 | Cu10 | Ir3 | 140.1(2) |
| C59 | C58 | C62 | 122.0(18) |  | C22 | Ir1 | Cu1 | 122.8(3) |
| C60 | C59 | C58 | 123.2(14) |  | C22 | Ir1 | Cu2 | 101.9(3) |
| C55 | C60 | C61 | 120.7(9) |  | C22 | Ir1 | Cu3 | 164.6(3) |
| C59 | C60 | C55 | 117.2(11) |  | C22 | Ir1 | Cu4 | 115.7(3) |
| C59 | C60 | C61 | 122.0(11) |  | Cu1 | Ir1 | Cu2 | 120.28(5) |
| N3 | C64 | C65 | 122.3(11) |  | Cu1 | Ir1 | Cu3 | 66.12(4) |
| N4 | C65 | C64 | 122.8(12) |  | Cu1 | Ir1 | Cu4 | 61.87(4) |
| N4 | C66 | C67 | 123.4(14) |  | Cu2 | Ir1 | Cu3 | 63.40(4) |
| C66 | C67 | N3 | 120.5(13) |  | Cu2 | Ir1 | Cu4 | 64.01(4) |
| N9 | C68 | C69 | 121.9(8) |  | Cu3 | Ir1 | Cu4 | 55.12(4) |
| N91 | C69 | C68 | 121.5(9) |  | C43 | Ir2 | Cu3 | 92.8(2) |
| Cu2 | Cl3 | Cu5 | 70.64(8) |  | C43 | Ir2 | Cu5 | 124.8(3) |
| Cu1 | Cl4 | Cu6 | 72.15(8) |  | C43 | Ir2 | Cu6 | 125.2(2) |
| Cl4 | Cu1 | Cu3 | 106.82(8) |  | C43 | Ir2 | Cu7 | 130.7(2) |
| Cl4 | Cu1 | Cu4 | 108.26(9) |  | C43 | Ir2 | Cu8 | 89.9(2) |
| Cl4 | Cu1 | Cu6 | 55.94(7) |  | C43 | Ir2 | Cu10 | 130.5(3) |
| Cl4 | Cu1 | Ir1 | 165.57(9) |  | Cu3 | Ir2 | Cu5 | 60.21(4) |
| Cu4 | Cu1 | Cu3 | 53.52(5) |  | Cu3 | Ir2 | Cu6 | 59.78(4) |
| Cu6 | Cu1 | Cu3 | 57.31(5) |  | Cu3 | Ir2 | Cu7 | 117.17(4) |
| Cu6 | Cu1 | Cu4 | 59.00(5) |  | Cu3 | Ir2 | Cu10 | 117.42(4) |
| Ir1 | Cu1 | Cu3 | 58.81(4) |  | Cu6 | Ir2 | Cu5 | 83.94(4) |
| Ir1 | Cu1 | Cu4 | 63.14(4) |  | Cu7 | Ir2 | Cu5 | 57.80(4) |
| Ir1 | Cu1 | Cu6 | 110.59(6) |  | Cu7 | Ir2 | Cu6 | 103.93(4) |
| Cl3 | Cu2 | Cu3 | 111.84(9) |  | Cu7 | Ir2 | Cu10 | 70.86(4) |
| Cl3 | Cu2 | Cu4 | 104.44(9) |  | Cu8 | Ir2 | Cu3 | 177.35(4) |
| Cl3 | Cu2 | Cu5 | 56.62(7) |  | Cu8 | Ir2 | Cu5 | 117.92(4) |
| Cl3 | Cu2 | Ir1 | 166.10(9) |  | Cu8 | Ir2 | Cu6 | 118.69(4) |
| Cu3 | Cu2 | Cu4 | 53.62(5) |  | Cu8 | Ir2 | Cu7 | 60.68(4) |
| Cu5 | Cu2 | Cu3 | 59.56(5) |  | Cu8 | Ir2 | Cu10 | 60.76(4) |
| Cu5 | Cu2 | Cu4 | 57.14(5) |  | Cu10 | Ir2 | Cu5 | 104.60(4) |
| Ir1 | Cu2 | Cu3 | 60.07(4) |  | Cu10 | Ir2 | Cu6 | 58.33(4) |
| Ir1 | Cu2 | Cu4 | 61.70(4) |  | C1 | Ir3 | Cu4 | 98.4(3) |
| Ir1 | Cu2 | Cu5 | 110.96(6) |  | C1 | Ir3 | Cu5 | 126.9(3) |
| Cu2 | Cu3 | Cu1 | 104.92(6) |  | C1 | Ir3 | Cu6 | 126.7(3) |
| Cu4 | Cu3 | Cu1 | 61.23(5) |  | C1 | Ir3 | Cu7 | 129.2(3) |
| Cu4 | Cu3 | Cu2 | 65.02(5) |  | C1 | Ir3 | Cu9 | 88.4(3) |
| Cu4 | Cu3 | Cu5 | 60.49(5) |  | C1 | Ir3 | Cu10 | 128.4(3) |
| Cu4 | Cu3 | Cu6 | 62.01(5) |  | Cu4 | Ir3 | Cu5 | 58.50(4) |
| Cu4 | Cu3 | Ir1 | 64.79(4) |  | Cu4 | Ir3 | Cu6 | 59.69(4) |
| Cu4 | Cu3 | Ir2 | 99.09(5) |  | Cu4 | Ir3 | Cu7 | 115.00(4) |
| Cu5 | Cu3 | Cu1 | 120.76(6) |  | Cu4 | Ir3 | Cu10 | 116.38(4) |
| Cu5 | Cu3 | Cu2 | 57.80(5) |  | Cu5 | Ir3 | Cu10 | 104.05(4) |
| Cu6 | Cu3 | Cu1 | 57.76(5) |  | Cu6 | Ir3 | Cu5 | 84.87(4) |
| Cu6 | Cu3 | Cu2 | 125.79(6) |  | Cu6 | Ir3 | Cu10 | 58.02(4) |
| Cu6 | Cu3 | Cu5 | 86.14(5) |  | Cu7 | Ir3 | Cu5 | 57.70(4) |
| Cu6 | Cu3 | Ir1 | 107.61(5) |  | Cu7 | Ir3 | Cu6 | 103.47(4) |
| Ir1 | Cu3 | Cu1 | 55.07(4) |  | Cu7 | Ir3 | Cu10 | 69.58(4) |
| Ir1 | Cu3 | Cu2 | 56.53(4) |  | Cu9 | Ir3 | Cu4 | 173.20(4) |
| Ir1 | Cu3 | Cu5 | 106.43(5) |  | Cu9 | Ir3 | Cu5 | 116.43(4) |
| Ir2 | Cu3 | Cu1 | 119.07(6) |  | Cu9 | Ir3 | Cu6 | 116.80(4) |
| Ir2 | Cu3 | Cu2 | 117.60(6) |  | Cu9 | Ir3 | Cu7 | 59.21(4) |
| Ir2 | Cu3 | Cu5 | 62.14(4) |  | Cu9 | Ir3 | Cu10 | 59.17(4) |
| Ir2 | Cu3 | Cu6 | 62.20(4) |  | C1 | N1 | C3 | 110.4(9) |
| Ir2 | Cu3 | Ir1 | 163.87(6) |  | C1 | N1 | C4 | 127.0(8) |
| Cu1 | Cu4 | Cu2 | 105.15(6) |  | C3 | N1 | C4 | 122.6(9) |
| Cu1 | Cu4 | Ir1 | 54.99(4) |  | C1 | N2 | C2 | 108.4(8) |
| Cu3 | Cu4 | Cu1 | 65.24(5) |  | C1 | N2 | C13 | 127.5(8) |
| Cu3 | Cu4 | Cu2 | 61.36(5) |  | C2 | N2 | C13 | 123.8(8) |
| Cu3 | Cu4 | Cu5 | 62.53(5) |  | C64 | N3 | C67 | 114.9(10) |
| Cu3 | Cu4 | Cu6 | 61.29(5) |  | C64 | N3 | Cu7 | 117.9(7) |
| Cu3 | Cu4 | Ir1 | 60.09(4) |  | C67 | N3 | Cu7 | 124.4(8) |
| Cu3 | Cu4 | Ir3 | 97.57(5) |  | C65 | N4 | C66 | 115.2(11) |
| Cu5 | Cu4 | Cu1 | 126.68(6) |  | C22 | N5 | C24 | 111.0(9) |
| Cu5 | Cu4 | Cu2 | 57.19(5) |  | C22 | N5 | C25 | 126.6(8) |
| Cu5 | Cu4 | Cu6 | 86.80(5) |  | C24 | N5 | C25 | 122.2(9) |
| Cu5 | Cu4 | Ir1 | 104.55(5) |  | C22 | N6 | C23 | 111.3(9) |
| Cu5 | Cu4 | Ir3 | 61.23(4) |  | C22 | N6 | C34 | 125.3(8) |
| Cu6 | Cu4 | Cu1 | 58.82(5) |  | C23 | N6 | C34 | 123.2(9) |
| Cu6 | Cu4 | Cu2 | 121.51(6) |  | C43 | N7 | C44 | 110.1(8) |
| Cu6 | Cu4 | Ir1 | 103.79(5) |  | C43 | N7 | C55 | 126.4(8) |
| Cu6 | Cu4 | Ir3 | 60.57(4) |  | C44 | N7 | C55 | 123.5(8) |
| Ir1 | Cu4 | Cu2 | 54.28(4) |  | C43 | N8 | C45 | 111.0(8) |
| Ir3 | Cu4 | Cu1 | 117.58(6) |  | C43 | N8 | C46 | 124.5(7) |
| Ir3 | Cu4 | Cu2 | 117.81(5) |  | C45 | N8 | C46 | 124.4(7) |
| Ir3 | Cu4 | Ir1 | 157.65(5) |  | C68 | N9 | C691 | 116.6(8) |
| Cl3 | Cu5 | Cu2 | 52.74(7) |  | C68 | N9 | Cu10 | 120.5(6) |
| Cl3 | Cu5 | Cu3 | 111.15(8) |  | C69 | N9 | Cu10 | 122.8(6) |

1. Torsion Angles

| **A** | **B** | **C** | **D** | **Angle/˚** |  | **A** | **B** | **C** | **D** | **Angle/˚** |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| C2 | C3 | N1 | C1 | -1.4(13) |  | C49 | C50 | C51 | C52 | 179.1(9) |
| C2 | C3 | N1 | C4 | 179.6(10) |  | C51 | C46 | C47 | C48 | -5.3(13) |
| C3 | C2 | N2 | C1 | 0.0(12) |  | C51 | C46 | C47 | C54 | 176.8(8) |
| C3 | C2 | N2 | C13 | 174.9(9) |  | C51 | C46 | N8 | C43 | 85.6(11) |
| C4 | C5 | C6 | C7 | -3.0(18) |  | C51 | C46 | N8 | C45 | -97.1(10) |
| C5 | C4 | C9 | C8 | 2.0(17) |  | C53 | C49 | C50 | C51 | 177.0(9) |
| C5 | C4 | C9 | C10 | -177.3(10) |  | C54 | C47 | C48 | C49 | 177.8(8) |
| C5 | C4 | N1 | C1 | -86.3(13) |  | C55 | C56 | C57 | C58 | -1.8(17) |
| C5 | C4 | N1 | C3 | 92.5(12) |  | C56 | C55 | C60 | C59 | -0.3(14) |
| C5 | C6 | C7 | C8 | 5(2) |  | C56 | C55 | C60 | C61 | -176.9(9) |
| C5 | C6 | C7 | C11 | -176.8(15) |  | C56 | C55 | N7 | C43 | -89.4(11) |
| C6 | C7 | C8 | C9 | -4(2) |  | C56 | C55 | N7 | C44 | 93.1(11) |
| C7 | C8 | C9 | C4 | 0.4(19) |  | C56 | C57 | C58 | C59 | 4(2) |
| C7 | C8 | C9 | C10 | 179.7(12) |  | C56 | C57 | C58 | C62 | -178.1(12) |
| C9 | C4 | C5 | C6 | -0.7(16) |  | C57 | C58 | C59 | C60 | -3.9(19) |
| C9 | C4 | C5 | C12 | 176.5(11) |  | C58 | C59 | C60 | C55 | 2.3(17) |
| C9 | C4 | N1 | C1 | 95.7(13) |  | C58 | C59 | C60 | C61 | 178.9(11) |
| C9 | C4 | N1 | C3 | -85.5(13) |  | C60 | C55 | C56 | C57 | 0.0(14) |
| C11 | C7 | C8 | C9 | 178.2(15) |  | C60 | C55 | C56 | C63 | 176.0(9) |
| C12 | C5 | C6 | C7 | 179.8(12) |  | C60 | C55 | N7 | C43 | 90.0(12) |
| C13 | C14 | C15 | C16 | -0.3(14) |  | C60 | C55 | N7 | C44 | -87.5(11) |
| C14 | C13 | C18 | C17 | -1.7(13) |  | C62 | C58 | C59 | C60 | 177.8(12) |
| C14 | C13 | C18 | C19 | -179.5(8) |  | C63 | C56 | C57 | C58 | -177.7(12) |
| C14 | C13 | N2 | C1 | 85.1(12) |  | C64 | C65 | N4 | C66 | 5(3) |
| C14 | C13 | N2 | C2 | -88.8(11) |  | C65 | C64 | N3 | C67 | -9(2) |
| C14 | C15 | C16 | C17 | -0.9(15) |  | C65 | C64 | N3 | Cu7 | -170.7(12) |
| C14 | C15 | C16 | C20 | 176.6(10) |  | C66 | C67 | N3 | C64 | 11(2) |
| C15 | C16 | C17 | C18 | 0.9(14) |  | C66 | C67 | N3 | Cu7 | 171.4(14) |
| C16 | C17 | C18 | C13 | 0.4(13) |  | C67 | C66 | N4 | C65 | -3(3) |
| C16 | C17 | C18 | C19 | 178.3(9) |  | C69 | C68 | N9 | C691 | -1.0(14) |
| C18 | C13 | C14 | C15 | 1.7(13) |  | C69 | C68 | N9 | Cu10 | 176.7(7) |
| C18 | C13 | C14 | C21 | 178.7(8) |  | Ir1 | C22 | N5 | C24 | -173.9(8) |
| C18 | C13 | N2 | C1 | -93.6(11) |  | Ir1 | C22 | N5 | C25 | 1.6(16) |
| C18 | C13 | N2 | C2 | 92.5(11) |  | Ir1 | C22 | N6 | C23 | 174.8(8) |
| C20 | C16 | C17 | C18 | -176.6(9) |  | Ir1 | C22 | N6 | C34 | -1.1(14) |
| C21 | C14 | C15 | C16 | -177.4(9) |  | Ir2 | C43 | N7 | C44 | 175.2(6) |
| C23 | C24 | N5 | C22 | -1.3(14) |  | Ir2 | C43 | N7 | C55 | -2.5(13) |
| C23 | C24 | N5 | C25 | -177.1(11) |  | Ir2 | C43 | N8 | C45 | -175.4(6) |
| C24 | C23 | N6 | C22 | -1.1(13) |  | Ir2 | C43 | N8 | C46 | 2.2(12) |
| C24 | C23 | N6 | C34 | 174.9(10) |  | Ir3 | C1 | N1 | C3 | 179.5(7) |
| C25 | C26 | C27 | C28 | 1(2) |  | Ir3 | C1 | N1 | C4 | -1.6(15) |
| C26 | C25 | C30 | C29 | 3.0(18) |  | Ir3 | C1 | N2 | C2 | -179.1(7) |
| C26 | C25 | C30 | C31 | 179.6(12) |  | Ir3 | C1 | N2 | C13 | 6.2(13) |
| C26 | C25 | N5 | C22 | -85.4(14) |  | N1 | C1 | N2 | C2 | -0.8(10) |
| C26 | C25 | N5 | C24 | 89.7(14) |  | N1 | C1 | N2 | C13 | -175.5(9) |
| C26 | C27 | C28 | C29 | 1(2) |  | N1 | C4 | C5 | C6 | -178.6(10) |
| C26 | C27 | C28 | C32 | -177.0(14) |  | N1 | C4 | C5 | C12 | -1.4(15) |
| C27 | C28 | C29 | C30 | -2(2) |  | N1 | C4 | C9 | C8 | 179.8(10) |
| C28 | C29 | C30 | C25 | 0(2) |  | N1 | C4 | C9 | C10 | 0.5(15) |
| C28 | C29 | C30 | C31 | -177.1(14) |  | N2 | C1 | N1 | C3 | 1.4(11) |
| C30 | C25 | C26 | C27 | -3.3(18) |  | N2 | C1 | N1 | C4 | -179.7(9) |
| C30 | C25 | C26 | C33 | 178.2(12) |  | N2 | C2 | C3 | N1 | 0.9(13) |
| C30 | C25 | N5 | C22 | 93.7(13) |  | N2 | C13 | C14 | C15 | -177.0(8) |
| C30 | C25 | N5 | C24 | -91.2(13) |  | N2 | C13 | C14 | C21 | 0.0(13) |
| C32 | C28 | C29 | C30 | 176.6(15) |  | N2 | C13 | C18 | C17 | 176.9(8) |
| C33 | C26 | C27 | C28 | 179.6(14) |  | N2 | C13 | C18 | C19 | -0.9(13) |
| C34 | C35 | C36 | C37 | 0.1(15) |  | N3 | C64 | C65 | N4 | 1(3) |
| C35 | C34 | C39 | C38 | -3.9(14) |  | N4 | C66 | C67 | N3 | -5(3) |
| C35 | C34 | C39 | C40 | 176.9(9) |  | N5 | C22 | N6 | C23 | 0.3(11) |
| C35 | C34 | N6 | C22 | 86.5(12) |  | N5 | C22 | N6 | C34 | -175.6(9) |
| C35 | C34 | N6 | C23 | -88.9(13) |  | N5 | C25 | C26 | C27 | 175.8(11) |
| C35 | C36 | C37 | C38 | -3.0(15) |  | N5 | C25 | C26 | C33 | -2.7(17) |
| C35 | C36 | C37 | C41 | 176.2(10) |  | N5 | C25 | C30 | C29 | -176.1(10) |
| C36 | C37 | C38 | C39 | 2.5(15) |  | N5 | C25 | C30 | C31 | 0.5(17) |
| C37 | C38 | C39 | C34 | 0.8(14) |  | N6 | C22 | N5 | C24 | 0.6(11) |
| C37 | C38 | C39 | C40 | -180.0(9) |  | N6 | C22 | N5 | C25 | 176.2(10) |
| C39 | C34 | C35 | C36 | 3.4(15) |  | N6 | C23 | C24 | N5 | 1.4(14) |
| C39 | C34 | C35 | C42 | -178.8(9) |  | N6 | C34 | C35 | C36 | -176.9(9) |
| C39 | C34 | N6 | C22 | -93.8(12) |  | N6 | C34 | C35 | C42 | 0.9(14) |
| C39 | C34 | N6 | C23 | 90.8(12) |  | N6 | C34 | C39 | C38 | 176.5(8) |
| C41 | C37 | C38 | C39 | -176.6(10) |  | N6 | C34 | C39 | C40 | -2.8(13) |
| C42 | C35 | C36 | C37 | -177.6(10) |  | N7 | C43 | N8 | C45 | 1.2(9) |
| C44 | C45 | N8 | C43 | -0.6(10) |  | N7 | C43 | N8 | C46 | 178.8(7) |
| C44 | C45 | N8 | C46 | -178.2(8) |  | N7 | C44 | C45 | N8 | -0.3(10) |
| C45 | C44 | N7 | C43 | 1.1(10) |  | N7 | C55 | C56 | C57 | 179.4(9) |
| C45 | C44 | N7 | C55 | 178.9(9) |  | N7 | C55 | C56 | C63 | -4.6(13) |
| C46 | C47 | C48 | C49 | -0.1(13) |  | N7 | C55 | C60 | C59 | -179.7(9) |
| C47 | C46 | C51 | C50 | 6.6(13) |  | N7 | C55 | C60 | C61 | 3.7(14) |
| C47 | C46 | C51 | C52 | -175.1(8) |  | N8 | C43 | N7 | C44 | -1.4(9) |
| C47 | C46 | N8 | C43 | -93.9(10) |  | N8 | C43 | N7 | C55 | -179.2(8) |
| C47 | C46 | N8 | C45 | 83.4(10) |  | N8 | C46 | C47 | C48 | 174.2(7) |
| C47 | C48 | C49 | C50 | 3.8(14) |  | N8 | C46 | C47 | C54 | -3.7(12) |
| C47 | C48 | C49 | C53 | -175.6(9) |  | N8 | C46 | C51 | C50 | -172.9(8) |
| C48 | C49 | C50 | C51 | -2.4(14) |  | N8 | C46 | C51 | C52 | 5.4(13) |
| C49 | C50 | C51 | C46 | -2.6(14) |  | N9 | C68 | C69 | N9 | 1.0(15) |

1. Hydrogen Atom Coordinates (Å×104) and Isotropic Displacement Parameters (Å2×103)

| **Atom** | ***x*** | ***y*** | ***z*** | **U(eq)** |
| --- | --- | --- | --- | --- |
| H2 | 11976.62 | 4389.19 | 6086.3 | 58 |
| H3 | 12095.64 | 4255.77 | 7131.07 | 60 |
| H6 | 9393.32 | 3708.57 | 9034.01 | 77 |
| H8 | 11706.36 | 1722.91 | 8819.57 | 71 |
| H10A | 12552.07 | 1379.7 | 7888.69 | 76 |
| H10B | 12840.78 | 2314.26 | 7517.48 | 76 |
| H10C | 11947.77 | 1723.79 | 7333.99 | 76 |
| H11A | 10526.59 | 2856.2 | 9889.77 | 167 |
| H11B | 10781.2 | 1817.18 | 9765.91 | 167 |
| H11C | 9570.85 | 2247.77 | 9805.75 | 167 |
| H12A | 8611.73 | 4183.32 | 7873.73 | 91 |
| H12B | 9682.02 | 4638.13 | 7565.83 | 91 |
| H12C | 9187.37 | 4854.68 | 8215.09 | 91 |
| H15 | 8587.38 | 4396.25 | 4955.76 | 54 |
| H17 | 10705.98 | 2327.05 | 4573.94 | 45 |
| H19A | 11933.99 | 2222.73 | 5898.41 | 61 |
| H19B | 12364.96 | 2192.8 | 5217.18 | 61 |
| H19C | 11500.74 | 1504.53 | 5528.69 | 61 |
| H20A | 9154.4 | 3832.96 | 3872.46 | 90 |
| H20B | 8316.07 | 3156.97 | 4227 | 90 |
| H20C | 9452.17 | 2765.54 | 3922.19 | 90 |
| H21A | 9454.74 | 4775.58 | 6303.05 | 70 |
| H21B | 8297.69 | 4563.11 | 6153.4 | 70 |
| H21C | 8959.05 | 5335.35 | 5764.65 | 70 |
| H23 | 4757.73 | 7392.95 | 7390.84 | 63 |
| H24 | 4254.22 | 6801.57 | 8419.55 | 66 |
| H27 | 2672.93 | 3705.51 | 9378.88 | 111 |
| H29 | 5668.22 | 3958.11 | 9735.7 | 94 |
| H31A | 6719.98 | 5161.38 | 9198.95 | 107 |
| H31B | 6319.53 | 5697.82 | 8625.76 | 107 |
| H31C | 6871.15 | 4695.35 | 8596.02 | 107 |
| H32A | 4599.82 | 2933.09 | 10365.95 | 226 |
| H32B | 3839.77 | 2432.51 | 10014.8 | 226 |
| H32C | 3337.9 | 3210.82 | 10420.01 | 226 |
| H33A | 2898.67 | 4741.98 | 7994.65 | 123 |
| H33B | 2365.42 | 5458.32 | 8438.45 | 123 |
| H33C | 2016.97 | 4436.17 | 8528.73 | 123 |
| H36 | 4291.76 | 6068.13 | 5407.31 | 59 |
| H38 | 7394.9 | 6336.67 | 5586.59 | 56 |
| H40A | 7376.06 | 5784.55 | 7009.68 | 76 |
| H40B | 7089.24 | 6853.06 | 6984.12 | 76 |
| H40C | 7991.55 | 6444.72 | 6508 | 76 |
| H41A | 5579.18 | 6420.68 | 4547.6 | 90 |
| H41B | 6322.83 | 5514.31 | 4655.23 | 90 |
| H41C | 6821.75 | 6480.37 | 4615.01 | 90 |
| H42A | 3401.75 | 5428.98 | 6827.4 | 85 |
| H42B | 2962.46 | 5858.1 | 6236.45 | 85 |
| H42C | 3140.01 | 6498.19 | 6727.88 | 85 |
| H44 | 4087.41 | -1140.83 | 7958.79 | 42 |
| H45 | 3892.01 | -877.26 | 6906.86 | 38 |
| H48 | 4811.7 | 1849.33 | 5325.57 | 39 |
| H50 | 6916.16 | -259.35 | 4999.7 | 43 |
| H52A | 6511.64 | -1376.81 | 6444.96 | 64 |
| H52B | 7494.24 | -1216.11 | 5937.71 | 64 |
| H52C | 6469.65 | -1706.13 | 5810.79 | 64 |
| H53A | 6665.02 | 1826.02 | 4473.6 | 64 |
| H53B | 5579.15 | 1513.84 | 4286.01 | 64 |
| H53C | 6653.87 | 863.5 | 4220.88 | 64 |
| H54A | 4003.05 | 2082.39 | 6294.48 | 39 |
| H54B | 4412.72 | 1449.47 | 6830.67 | 39 |
| H54C | 3451.5 | 1154.06 | 6516.16 | 39 |
| H57 | 7426.74 | -1539.61 | 9144.12 | 82 |
| H59 | 5560.86 | 683.66 | 9470.83 | 74 |
| H61A | 3902.86 | 723.85 | 8593.79 | 70 |
| H61B | 4756.01 | 1155.96 | 8086.19 | 70 |
| H61C | 4622.46 | 1503.89 | 8726.49 | 70 |
| H62A | 7537.87 | -941.16 | 10019.53 | 165 |
| H62B | 6441.39 | -433.13 | 10287.52 | 165 |
| H62C | 7416.09 | 141.28 | 9971.53 | 165 |
| H63A | 7469.11 | -1648 | 7764.1 | 93 |
| H63B | 6330.48 | -2076.39 | 7953.26 | 93 |
| H63C | 7305.53 | -2366.25 | 8325.44 | 93 |
| H64 | 8606.23 | 655.69 | 8838.87 | 73 |
| H65 | 9441.11 | 278.37 | 9652.77 | 80 |
| H66 | 11910.3 | -785.5 | 8702.61 | 112 |
| H67 | 11150.69 | -337.49 | 7875.02 | 87 |
| H68 | 8713.17 | 1168.4 | 5010.75 | 37 |
| H69 | 9508.12 | 659.52 | 4133.53 | 41 |
| H1A | 6315.5 | 1746.57 | 11378.41 | 140 |
| H1B | 7135.11 | 1729.16 | 11261.41 | 140 |
| H71A | 8417.24 | 3650.36 | 9940 | 119 |
| H71B | 7957.94 | 4434.41 | 10349.88 | 119 |
| H71C | 7163.31 | 3738.15 | 10181.57 | 119 |
| H2A | 7776.51 | 2745.61 | 10766.62 | 119 |
| H70A | 6877.74 | 1892.91 | 10220.99 | 213 |
| H70B | 5639.92 | 1937.02 | 10497.69 | 213 |
| H70C | 6384.08 | 1029.8 | 10601.48 | 213 |
| H72A | 6610.64 | 2131.92 | 10203.6 | 213 |
| H72B | 7800.38 | 1923.71 | 10377.13 | 213 |
| H72C | 7226.83 | 2917.15 | 10417.43 | 213 |

1. Atomic Occupancy

| **Atom** | ***Occupancy*** |  | **Atom** | ***Occupancy*** |  | **Atom** | ***Occupancy*** |
| --- | --- | --- | --- | --- | --- | --- | --- |
| H1A | 0.60(3) |  | H1B | 0.40(3) |  | C71 | 0.59(2) |
| H71A | 0.59(2) |  | H71B | 0.59(2) |  | H71C | 0.59(2) |
| O2 | 0.59(2) |  | H2A | 0.59(2) |  | C70 | 0.60(3) |
| H70A | 0.60(3) |  | H70B | 0.60(3) |  | H70C | 0.60(3) |
| C72 | 0.40(3) |  | H72A | 0.40(3) |  | H72B | 0.40(3) |
| H72C | 0.40(3) |  |  |  |  |  |  |

1. Solvent mask information

| **Number** | **X** | **Y** | **Z** | **Volume** | **Electron count** | **Content** |
| --- | --- | --- | --- | --- | --- | --- |
| 1 | 0.000 | 0.500 | 0.000 | 430.0 | 65.7 | ? |