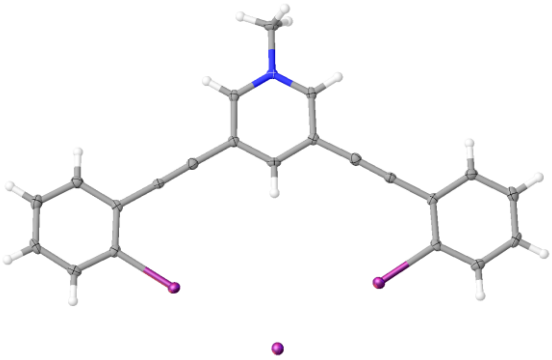
X-Ray Crystallography

UNIVERSITY OF MONTANA



Experimental

Example writeup: Data for **umt_ob175** were collected from at 100 K on a Bruker D8 VENTURE Duo Fixed Chi Three-Circle Diffractometer using MoK_α radiation (λ = 0.71073 Å). All data were integrated with SAINT V8.38A and corrected for absorption using SADABS 2016/2. [1,2] The structure was solved by dual methods with ShelXT and refined by full-matrix least-squares methods against F^2 using ShelXL.[3,4] All non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atoms in the investigated structure were located from difference Fourier maps but finally their positions were placed in geometrically calculated positions and refined using a riding model. Isotropic thermal parameters of the placed hydrogen atoms were fixed to 1.2 times the U value of the atoms they are linked to (1.5 times for methyl groups). Calculations and refinement of structures were carried out using APEX4[5], and Olex2[6] software. This report and the CIF file were generated using FinalCif.[7]

Acknowledgement:

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Crystallographic Data for umt_ob175:

M= 673.04 g/mol, $C_{22}H_{14}I_3N$, orthorhombic, a = 21.0383(12) Å, b = 13.7820(8) Å, c = 21.0383(12) Å, α =90°, β = 90°, γ = 90°, V = 2117.5(2) Å3, Z = 4, T=100K, μ(MoKα) = 4.434 mm^{-1}, ρ_{calc} = 2.111 g/cm3, 44308 reflections measured, 2984 unique, R1 = 0.0265 (I > 2σ(I)), wR2 = 0.0484 (all data).

Table 1. Crystal data and structure refinement for umt_ob175

CCDC number	2075105
Empirical formula	$C_{22}H_{14}I_3N$
Formula weight	673.04
Temperature [K]	100
Crystal system	orthorhombic
Space group (number)	$Pbcn$ (60)
a [Å]	21.0383(12)
b [Å]	13.7820(8)
c [Å]	7.3031(4)
α [°]	90
β [°]	90
γ [°]	90
Volume [Å^3]	2117.5(2)
Z	4
ρ_{calc} [gcm^{-3}]	2.111
μ [mm^{-1}]	4.434
F(000)	1248
Crystal size [mm^3]	0.02×0.02×0.45
Crystal colour	yellow
Crystal shape	rod
Radiation	MoK_α (λ=0.71073 Å)
2θ range [°]	5.91 to 59.21 (0.72 Å)
Index ranges	−29 ≤ h ≤ 28 −19 ≤ k ≤ 19 −10 ≤ l ≤ 9
Reflections collected	44308
Independent reflections	2984 R_{int} = 0.0595 R_{sigma} = 0.0283
Completeness to θ = 25.242°	99.9 %
Data / Restraints / Parameters	2984 / 0 / 121
Goodness-of-fit on F^2	1.058
Final *R* indexes [*I*≥2σ(*I*)]	R_1 = 0.0265 wR_2 = 0.0446
Final *R* indexes [all data]	R_1 = 0.0444 wR_2 = 0.0484
Largest peak/hole [eÅ^{-3}]	0.76/−0.50

Table 2. Atomic coordinates and Ueq [Å^2] for umt_ob175

Atom	*x*	*y*	*z*	U_{eq}
I1	0.35678(2)	0.68214(2)	0.65645(2)	0.01439(5)
I2	0.500000	0.82437(2)	0.750000	0.01423(6)
N1	0.500000	0.2069(2)	0.750000	0.0108(6)
C1	0.44572(13)	0.25409(19)	0.7183(3)	0.0134(5)
H1	0.407820	0.218369	0.696926	0.016
C2	0.44398(13)	0.35564(19)	0.7161(4)	0.0134(6)
C3	0.500000	0.4057(3)	0.750000	0.0138(8)
H3	0.500000	0.474640	0.750003	0.017
C4	0.38556(13)	0.4040(2)	0.6801(4)	0.0152(6)
C5	0.33748(13)	0.44700(19)	0.6468(4)	0.0135(5)
C6	0.27968(13)	0.4963(2)	0.5994(3)	0.0127(5)
C7	0.27642(12)	0.5978(2)	0.5893(3)	0.0132(6)
C8	0.22039(14)	0.6427(2)	0.5335(4)	0.0162(6)
H8	0.218262	0.711493	0.526308	0.019
C9	0.16775(13)	0.5872(2)	0.4886(4)	0.0173(6)
H9	0.130014	0.618075	0.447111	0.021
C10	0.16975(13)	0.4868(2)	0.5037(4)	0.0186(6)
H10	0.133017	0.449317	0.476943	0.022
C11	0.22527(13)	0.4416(2)	0.5576(4)	0.0161(6)
H11	0.226653	0.372868	0.566476	0.019
C12	0.500000	0.0993(3)	0.750000	0.0162(8)
H12A	0.471276	0.075628	0.845703	0.024
H12B	0.543133	0.075628	0.773809	0.024
H12C	0.485591	0.075628	0.630488	0.024

U_{eq} is defined as 1/3 of the trace of the orthogonalized U_{ij} tensor.

Table 3. Anisotropic displacement parameters (Å^2) for umt_ob175. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\, h^2(a^*)^2U_{11} + k^2(b^*)^2U_{22} + \ldots + 2hka^*b^*U_{12} \,]$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
I1	0.01498(9)	0.01288(9)	0.01531(9)	−0.00170(7)	−0.00029(7)	−0.00083(8)
I2	0.01360(12)	0.01607(12)	0.01302(12)	0.000	0.00141(9)	0.000
N1	0.0119(15)	0.0107(14)	0.0097(15)	0.000	0.0022(13)	0.000
C1	0.0110(12)	0.0176(14)	0.0118(13)	−0.0013(11)	0.0003(11)	−0.0006(11)
C2	0.0136(14)	0.0144(13)	0.0121(13)	−0.0009(11)	0.0000(11)	0.0007(11)
C3	0.019(2)	0.0095(17)	0.0132(18)	0.000	0.0019(17)	0.000
C4	0.0151(14)	0.0150(14)	0.0155(14)	−0.0021(11)	−0.0002(11)	−0.0010(12)
C5	0.0159(13)	0.0109(13)	0.0137(13)	−0.0019(11)	0.0000(11)	−0.0015(11)
C6	0.0127(13)	0.0158(13)	0.0095(12)	0.0021(10)	0.0014(11)	0.0023(11)
C7	0.0121(13)	0.0164(14)	0.0111(12)	−0.0002(11)	0.0019(11)	−0.0028(11)
C8	0.0197(15)	0.0138(13)	0.0152(14)	0.0003(11)	0.0014(12)	0.0036(12)
C9	0.0136(14)	0.0234(16)	0.0151(13)	0.0019(12)	−0.0004(11)	0.0078(12)
C10	0.0134(14)	0.0248(17)	0.0177(14)	−0.0016(12)	0.0009(12)	−0.0036(13)
C11	0.0160(14)	0.0137(13)	0.0187(13)	0.0014(12)	0.0019(12)	0.0005(11)
C12	0.020(2)	0.0104(18)	0.0185(19)	0.000	0.0027(18)	0.000

Table 4. Bond lengths and angles for umt_ob175

Atom–Atom	Length [Å]
I1–C7	2.109(3)
N1–C1$^{\#1}$	1.334(3)
N1–C1	1.334(3)
N1–C12	1.482(5)
C1–C2	1.400(4)
C2–C3	1.388(3)
C2–C4	1.423(4)
C4–C5	1.197(4)
C5–C6	1.435(4)
C6–C7	1.403(4)
C6–C11	1.404(4)
C7–C8	1.392(4)
C8–C9	1.386(4)
C9–C10	1.389(4)
C10–C11	1.381(4)

Atom–Atom–Atom	Angle [°]
C1–N1–C1$^{\#1}$	121.7(3)
C1–N1–C12	119.17(16)
C1$^{\#1}$–N1–C12	119.17(16)
N1–C1–C2	120.8(3)
C1–C2–C4	119.5(3)
C3–C2–C1	118.2(3)
C3–C2–C4	122.2(3)
C2–C3–C2$^{\#1}$	120.4(3)
C5–C4–C2	177.9(3)
C4–C5–C6	177.5(3)
C7–C6–C5	121.7(2)
C7–C6–C11	118.9(2)
C11–C6–C5	119.3(2)
C6–C7–I1	119.88(19)
C8–C7–I1	120.1(2)
C8–C7–C6	120.0(3)
C9–C8–C7	120.0(3)
C8–C9–C10	120.5(3)
C11–C10–C9	119.9(3)
C10–C11–C6	120.6(3)

Symmetry transformations used to generate equivalent atoms:
#1: 1-X, +Y, 1.5-Z;

References

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