exp_2355+FCl3MB

Table 1 Crystal data and structure refinement for exp_2355+FCl3MB.

Identification code	exp_2355+FCl3MB
Empirical formula	$C_{32}H_{36}N_6OCl_8Fe_2S_2$
Formula weight	964.126
Temperature/K	N/A
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	15.0077(5)
b/Å	17.4746(6)
c/Å	7.9490(4)
α/°	90
β/°	92.572(4)
γ/°	90
Volume/Å^3	2082.55(14)
Z	2
$_{calc}$g/cm^3	1.538
μ/mm^{-1}	11.505
F(000)	985.3
Crystal size/mm^3	N/A × N/A × N/A
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	5.9 to 143.14
Index ranges	$-17 \leq h \leq 18$, $-21 \leq k \leq 21$, $-9 \leq l \leq 9$
Reflections collected	21130
Independent reflections	4037 [R_{int} = 0.0946, R_{sigma} = 0.0707]
Data/restraints/parameters	4037/0/230
Goodness-of-fit on F^2	0.995
Final R indexes [I>=2σ (I)]	R_1 = 0.0555, wR_2 = 0.1332
Final R indexes [all data]	R_1 = 0.1007, wR_2 = 0.1621
Largest diff. peak/hole / e Å^{-3}	0.42/-0.55

Table 2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for exp_2355+FCl3MB. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
Fe	7079.4(5)	4237.5(5)	3863.7(11)	71.9(3)
S5	1765.7(8)	3449.9(7)	5042.7(18)	67.9(4)
Cl1	7053.4(10)	3061.8(8)	4811(2)	89.5(4)
Cl2	8464.4(9)	4530.9(9)	3318(2)	93.0(5)
Cl3	6586.8(11)	5027.8(9)	5744(2)	101.1(5)
Cl4	6253.1(12)	4306.0(10)	1540(2)	107.6(6)
N10	1183(2)	1755(2)	5645(5)	63.1(10)
N3	-1097(3)	3963(3)	7968(5)	74.6(12)
N7	4452(3)	2100(3)	2782(6)	73.9(12)
C14	1958(3)	1885(3)	4943(6)	62.1(12)
C11	649(3)	2318(3)	6119(6)	59.5(11)
C13	2325(3)	2616(3)	4587(6)	58.6(11)
C12	817(3)	3120(3)	5961(6)	59.9(11)
C1	-162(3)	2113(3)	6874(7)	69.3(13)
C7	3646(3)	2040(3)	3465(6)	64.1(12)
C6	3140(3)	2693(3)	3859(6)	65.4(12)
C3	-540(3)	3433(3)	7317(6)	64.4(12)
C4	233(3)	3655(3)	6544(6)	66.1(13)
C2	-741(3)	2637(3)	7443(7)	68.6(13)
C8	3291(4)	1307(3)	3834(7)	76.7(15)
C9	2487(4)	1242(3)	4513(7)	76.5(14)
C72	4834(4)	2840(3)	2429(8)	87.9(17)
C31	-1908(4)	3755(3)	8796(7)	87.1(17)
C71	4999(4)	1422(4)	2444(9)	97.1(19)
C32	-885(4)	4783(3)	7928(9)	101(2)

Table 3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for exp_2355+FCl3MB. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Fe	69.6(5)	59.9(5)	86.9(6)	-5.2(4)	9.7(4)	-3.1(4)
S5	72.2(7)	48.1(6)	83.5(9)	-6.7(5)	6.9(6)	0.7(6)
Cl1	96.2(9)	64.6(8)	108.1(12)	3.0(7)	7.3(8)	6.7(8)
Cl2	76.7(8)	85.3(10)	118.8(13)	-15.4(7)	23.8(8)	-21.6(9)
Cl3	106.5(10)	77.4(9)	122.2(13)	1.4(8)	36.7(9)	-14.3(9)
Cl4	119.4(12)	86.2(11)	114.6(13)	-24.2(9)	-26.3(10)	14.2(9)
N10	67(2)	52(2)	71(3)	-6.8(19)	7(2)	-0.2(19)
N3	79(3)	73(3)	73(3)	-4(2)	11(2)	-7(2)
N7	69(2)	81(3)	72(3)	-1(2)	6(2)	1(2)
C14	68(3)	53(3)	65(3)	-6(2)	0(2)	1(2)
C11	62(3)	57(3)	59(3)	-5(2)	-4(2)	4(2)
C13	64(3)	51(3)	60(3)	-7(2)	-4(2)	2(2)
C12	65(3)	53(3)	61(3)	-4(2)	-4(2)	0(2)
C1	71(3)	61(3)	76(3)	-11(2)	-4(3)	9(3)
C7	62(3)	67(3)	63(3)	-3(2)	0(2)	0(2)
C6	68(3)	61(3)	67(3)	-11(2)	-1(2)	0(2)
C3	70(3)	64(3)	58(3)	0(2)	0(2)	-3(2)
C4	74(3)	50(3)	74(3)	-6(2)	-4(3)	-3(2)
C2	65(3)	70(3)	71(3)	-9(3)	3(2)	3(3)
C8	83(3)	58(3)	90(4)	7(3)	13(3)	5(3)
C9	88(4)	53(3)	88(4)	-10(3)	7(3)	0(3)
C72	79(3)	95(4)	90(4)	-24(3)	8(3)	5(3)
C31	86(4)	93(4)	83(4)	16(3)	13(3)	-7(3)
C71	78(3)	100(5)	114(5)	16(3)	18(3)	5(4)
C32	111(5)	68(4)	126(6)	5(3)	16(4)	-19(4)

Table 4 Bond Lengths for exp_2355+FCl3MB.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Fe	Cl1	2.1889(16)	N7	C71	1.473(7)
Fe	Cl2	2.2029(15)	C14	C13	1.425(6)
Fe	Cl3	2.1880(17)	C14	C9	1.426(7)
Fe	Cl4	2.1812(19)	C11	C12	1.430(6)
S5	C13	1.729(5)	C11	C1	1.426(6)
S5	C12	1.728(5)	C13	C6	1.382(6)
N10	C14	1.331(6)	C12	C4	1.376(6)
N10	C11	1.334(6)	C1	C2	1.355(7)
N3	C3	1.365(6)	C7	C6	1.413(7)
N3	C31	1.456(7)	C7	C8	1.423(7)
N3	C32	1.467(7)	C3	C4	1.393(7)
N7	C7	1.353(6)	C3	C2	1.427(7)
N7	C72	1.447(7)	C8	C9	1.349(7)

Table 5 Bond Angles for exp_2355+FCl3MB.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
Cl2	Fe	Cl1	108.53(7)	C1	C11	C12	116.2(4)
Cl3	Fe	Cl1	110.22(7)	C14	C13	S5	121.2(4)
Cl3	Fe	Cl2	109.78(7)	C6	C13	S5	116.9(4)
Cl4	Fe	Cl1	108.94(7)	C6	C13	C14	121.9(4)
Cl4	Fe	Cl2	109.03(8)	C11	C12	S5	121.1(4)
Cl4	Fe	Cl3	110.31(8)	C4	C12	S5	117.6(4)
C12	S5	C13	103.0(2)	C4	C12	C11	121.2(4)
C11	N10	C14	122.8(4)	C2	C1	C11	122.8(5)
C31	N3	C3	122.7(5)	C6	C7	N7	121.8(5)
C32	N3	C3	121.1(4)	C8	C7	N7	120.1(5)
C32	N3	C31	116.1(5)	C8	C7	C6	118.1(4)
C72	N7	C7	121.1(5)	C7	C6	C13	120.6(4)
C71	N7	C7	121.8(5)	C4	C3	N3	121.0(5)
C71	N7	C72	117.0(4)	C2	C3	N3	120.0(5)
C13	C14	N10	126.0(4)	C2	C3	C4	119.0(5)
C9	C14	N10	118.3(4)	C3	C4	C12	121.0(4)
C9	C14	C13	115.7(4)	C3	C2	C1	119.7(5)
C12	C11	N10	125.9(4)	C9	C8	C7	120.6(5)
C1	C11	N10	117.9(4)	C8	C9	C14	123.2(5)

Table 6 Torsion Angles for exp_2355+FCl3MB.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
S5	C13	C14	N10	-0.6(5)	N3	C3	C2	C1	-176.5(5)
S5	C13	C14	C9	-179.8(4)	N7	C7	C6	C13	-178.7(5)
S5	C13	C6	C7	179.3(4)	N7	C7	C8	C9	-179.9(5)
S5	C12	C11	N10	1.8(5)	C14	C13	C6	C7	-1.0(6)
S5	C12	C11	C1	-179.1(4)	C14	C9	C8	C7	-1.8(7)
S5	C12	C4	C3	-179.2(4)	C11	C12	C4	C3	0.4(5)
N10	C14	C13	C6	179.7(5)	C11	C1	C2	C3	-0.9(6)
N10	C14	C9	C8	-178.3(5)	C13	C6	C7	C8	0.1(6)
N10	C11	C12	C4	-177.8(5)	C12	C4	C3	C2	-2.4(6)
N10	C11	C1	C2	178.2(4)	C1	C2	C3	C4	2.7(6)
N3	C3	C4	C12	176.8(5)					

Table 7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for exp_2355+FCl3MB.

Atom	*x*	*y*	*z*	U(eq)
H1	-299(3)	1596(3)	6981(7)	83.2(16)
H6	3356(3)	3178(3)	3627(6)	78.5(15)
H4	357(3)	4173(3)	6419(6)	79.4(15)
H2	-1267(3)	2480(3)	7913(7)	82.4(16)
H8	3617(4)	870(3)	3607(7)	92.0(17)
H9	2267(4)	754(3)	4709(7)	91.8(17)
H72a	4432(13)	3122(10)	1690(40)	132(3)
H72b	4930(30)	3119(10)	3462(9)	132(3)
H72c	5392(14)	2772(3)	1900(50)	132(3)
H31a	-2328(11)	3540(20)	7986(13)	131(3)
H31b	-1768(6)	3387(19)	9670(40)	131(3)
H31c	-2162(16)	4203(5)	9280(50)	131(3)
H71a	5080(30)	1127(14)	3458(16)	146(3)
H71b	4701(15)	1115(14)	1590(40)	146(3)
H71c	5568(12)	1583(4)	2060(60)	146(3)
H32a	-400(20)	4888(6)	8710(40)	152(3)
H32b	-720(30)	4923(6)	6815(16)	152(3)
H32c	-1397(11)	5073(4)	8230(60)	152(3)

Experimental

Single crystals of $C_{32}H_{36}N_6OCl_8Fe_2S_2$ **[exp_2355+FCl3MB]** were **[]**. A suitable crystal was selected and **[]** on a **XtaLAB Synergy, Single source at home/near, HyPix3000** diffractometer. The crystal was kept at N/A K during data collection. Using Olex2 [1], the structure was solved with the olex2.solve [2] structure solution program using Charge Flipping and refined with the olex2.refine [3] refinement package using Gauss-Newton minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2. Bourhis, L.J., Dolomanov, O.V., Gildea, R.J., Howard, J.A.K., Puschmann, H. (2015). Acta Cryst. A71, 59-75.
3. Bourhis, L.J., Dolomanov, O.V., Gildea, R.J., Howard, J.A.K., Puschmann, H. (2015). Acta Cryst. A71, 59-75.

Crystal structure determination of [exp_2355+FCl3MB]

Crystal Data for $C_{32}H_{36}N_6OCl_8Fe_2S_2$ (M =964.126 g/mol): monoclinic, space group $P2_1/c$ (no. 14), a = 15.0077(5) Å, b = 17.4746(6) Å, c = 7.9490(4) Å, β = 92.572(4)°, V = 2082.55(14) Å^3, Z = 2, T = N/A K, μ(Cu Kα) = 11.505 mm^{-1}, *Dcalc* = 1.538 g/cm^3, 21130 reflections measured (5.9° ≤ 2Θ ≤ 143.14°), 4037 unique (R_{int} = 0.0946, R_{sigma} = 0.0707) which were used in all calculations. The final R_1 was 0.0555 (I>=2u(I)) and wR_2 was 0.1621 (all data).

Refinement model description

Number of restraints - 0, number of constraints - 28.

Details:

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1. Fixed Uiso
 At 1.2 times of:
  All C(H) groups
 At 1.5 times of:
  All C(H,H,H) groups
2.a Aromatic/amide H refined with riding coordinates:
 C1(H1), C6(H6), C4(H4), C2(H2), C8(H8), C9(H9)
2.b Idealised Me refined as rotating group:
 C72(H72a,H72b,H72c), C31(H31a,H31b,H31c), C71(H71a,H71b,H71c), C32(H32a,H32b,
 H32c)
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