

**Crystallographic Data Tables of Chapter 2**

	(1)	(3)	(4)	(5)	(6) H <sub>2</sub> O	[7*]
Empirical formula	C <sub>9</sub> H <sub>6</sub> N <sub>7</sub> Cl	C <sub>13</sub> H <sub>16</sub> N <sub>8</sub>	C <sub>11</sub> H <sub>12</sub> N <sub>8</sub>	C <sub>12</sub> H <sub>13</sub> N <sub>7</sub> S	C <sub>15</sub> H <sub>13</sub> N <sub>7</sub> O <sub>2</sub>	C <sub>30</sub> H <sub>26</sub> N <sub>9</sub> O <sub>8</sub> Cl <sub>2</sub> P
Formula weight	247.66	284.34	256.29	287.35	323.32	742.47
Temperature/K	120.15	120.15	120.00(13)	100.00(10)	120.00(13)	120.15
Crystal system	monoclinic	monoclinic	monoclinic	monoclinic	triclinic	triclinic
Space group	P2 <sub>1</sub> /c	P2 <sub>1</sub> /n	P2 <sub>1</sub> /c	P2 <sub>1</sub> /n	P-1	P-1
a/Å	6.64461(18)	8.61844(16)	6.5520(2)	4.3800(3)	4.1472(6)	10.5271(4)
b/Å	14.8768(3)	9.2332(2)	16.4896(5)	31.301(3)	13.3919(9)	18.4165(7)
c/Å	10.7740(3)	17.9584(3)	11.3238(3)	9.9554(6)	14.9609(13)	19.0679(8)
α/°	90	90	90	90	114.286(8)	63.193(4)
β/°	106.770(3)	92.7312(18)	99.818(3)	91.249(6)	93.470(10)	77.378(3)
γ/°	90	90	90	90	90.376(8)	87.953(3)
Volume/Å <sup>3</sup>	1019.72(4)	1427.44(5)	1205.50(6)	1364.56(19)	755.54(14)	3211.3(2)
Z	4	4	4	4	2	4
ρ <sub>calc</sub> /mg/mm <sup>3</sup>	1.613	1.323	1.412	1.399	1.421	1.536
μ/mm <sup>-1</sup>	3.242	0.718	0.790	2.131	0.841	2.873
2θ range for data collection	10.44 to 147.44°	9.862 to 147.708°	9.57 to 133.2°	9.324 to 147.368°	7.246 to 147.84°	8.63 to 148.154°
Index ranges	-5 ≤ h ≤ 8, -18 ≤ k ≤ 15, -13 ≤ l ≤ 11	-10 ≤ h ≤ 10, -11 ≤ k ≤ 6, -21 ≤ l ≤ 21	-3 ≤ h ≤ 7, -17 ≤ k ≤ 19, -13 ≤ l ≤ 12	-5 ≤ h ≤ 5, -38 ≤ k ≤ 26, -12 ≤ l ≤ 11	-5 ≤ h ≤ 3, -16 ≤ k ≤ 16, -18 ≤ l ≤ 15	-12 ≤ h ≤ 13, -22 ≤ k ≤ 17, -23 ≤ l ≤ 18
Reflections collected	3786	5268	4416	4924	5027	28051
Independent reflections	1944 [R <sub>int</sub> = 0.0336, R <sub>sigma</sub> = 0.0398]	2788 [R <sub>int</sub> = 0.0285, R <sub>sigma</sub> = 0.0346]	2101 [R <sub>int</sub> = 0.0170, R <sub>sigma</sub> = 0.0218]	2642 [R <sub>int</sub> = 0.0352, R <sub>sigma</sub> = 0.0436]	2799 [R <sub>int</sub> = 0.0419, R <sub>sigma</sub> = 0.0559]	12154 [R <sub>int</sub> = 0.0369, R <sub>sigma</sub> = 0.0385]
Data/restraints/parameters	1944/6/172	2788/6/192	2101/0/208	2642/0/183	2799/0/224	12154/0/901
Goodness-of-fit on F <sup>2</sup>	1.061	1.033	1.209	1.018	1.753	1.14
Final R indexes [I ≥ 2σ(I)]	R <sub>1</sub> = 0.0415, wR <sub>2</sub> = 0.1110	R <sub>1</sub> = 0.0570, wR <sub>2</sub> = 0.1638	R <sub>1</sub> = 0.0429, wR <sub>2</sub> = 0.1198	R <sub>1</sub> = 0.0616, wR <sub>2</sub> = 0.1460	R <sub>1</sub> = 0.1041, wR <sub>2</sub> = 0.2805	R <sub>1</sub> = 0.0596, wR <sub>2</sub> = 0.1671
Final R indexes [all data]	R <sub>1</sub> = 0.0448, wR <sub>2</sub> = 0.1145	R <sub>1</sub> = 0.0634, wR <sub>2</sub> = 0.1736	R <sub>1</sub> = 0.0462, wR <sub>2</sub> = 0.1216	R <sub>1</sub> = 0.0687, wR <sub>2</sub> = 0.1496	R <sub>1</sub> = 0.1317, wR <sub>2</sub> = 0.2993	R <sub>1</sub> = 0.0664, wR <sub>2</sub> = 0.1720
Largest diff. peak/hole / e Å <sup>-3</sup>	0.33/-0.28	0.49/-0.45	0.23/-0.23	0.47/-0.46	1.16/-0.49	0.74/-0.56

	<b>(8)</b>	<b>(9) CH<sub>2</sub>Cl<sub>2</sub></b>	<b>(10) CH<sub>3</sub>NO<sub>2</sub></b>	<b>(11)H BF<sub>4</sub></b>	<b>[2]Ba</b>	<b>[2]Bβ</b>
Empirical formula	C <sub>18</sub> H <sub>21</sub> N <sub>9</sub>	C <sub>25</sub> H <sub>29</sub> N <sub>9</sub> Cl <sub>2</sub>	C <sub>22</sub> H <sub>24</sub> N <sub>10</sub> O <sub>8</sub>	C <sub>25.5</sub> H <sub>20.5</sub> N <sub>10.5</sub> O <sub>3</sub> BF <sub>4</sub>	C <sub>24</sub> H <sub>18</sub> N <sub>18</sub> FeB <sub>2</sub> F <sub>8</sub>	C <sub>24</sub> H <sub>18</sub> N <sub>18</sub> F <sub>8</sub> FeB <sub>2</sub>
Formula weight	363.44	526.47	556.51	608.83	788.03	788.03
Temperature/K	120.15	120.15	120.15	120.1(4)	100.15	120.15
Crystal system	monoclinic	triclinic	monoclinic	triclinic	cubic	tetragonal
Space group	P2 <sub>1</sub> /n	P-1	P2 <sub>1</sub> /n	P-1	Ia-3d	I4 <sub>1</sub> /acd
a/Å	14.7507(2)	9.8021(5)	8.1805(2)	13.8786(3)	18.4913(9)	18.3777(11)
b/Å	7.46790(10)	10.0189(5)	13.4270(3)	15.6730(4)	18.4913(9)	18.3777(11)
c/Å	15.9180(2)	13.2198(5)	23.6246(4)	24.5043(6)	18.4913(9)	18.0915(19)
α/°	90	100.616(4)	90	90.979(2)	90	90
β/°	90.8590(10)	98.278(4)	97.876(2)	98.120(2)	90	90
γ/°	90	97.875(4)	90	102.827(2)	90	90
Volume/Å <sup>3</sup>	1753.28(4)	1244.77(10)	2570.42(10)	5138.6(2)	6322.7(9)	6110.2(8)
Z	4	2	4	8	8	8
ρ <sub>calc</sub> /mg/mm <sup>3</sup>	1.377	1.405	1.438	1.574	1.656	1.713
μ/mm <sup>-1</sup>	0.728	2.620	0.958	1.1	0.533	4.89
2θ range for data collection	8.112 to 147.68°	6.908 to 147.598°	7.556 to 147.618°	6.606 to 147.726°	5.23 to 49.962°	13.62 to 147.28°
Index ranges	-18 ≤ h ≤ 15, -6 ≤ k ≤ 9, -18 ≤ l ≤ 19	-12 ≤ h ≤ 9, -10 ≤ k ≤ 11, -16 ≤ l ≤ 14	-9 ≤ h ≤ 8, -16 ≤ k ≤ 16, -20 ≤ l ≤ 29	-16 ≤ h ≤ 14, -19 ≤ k ≤ 19, -27 ≤ l ≤ 30	-22 ≤ h ≤ 15, -22 ≤ k ≤ 21, -18 ≤ l ≤ 21	-20 ≤ h ≤ 22, -19 ≤ k ≤ 22, -21 ≤ l ≤ 14
Reflections collected	6244	9548	11253	48526	5825	7642
Independent reflections	3419 [R <sub>int</sub> = 0.0239, R <sub>sigma</sub> = 0.0255]	4691 [R <sub>int</sub> = 0.0423, R <sub>sigma</sub> = 0.0458]	5035 [R <sub>int</sub> = 0.0345, R <sub>sigma</sub> = 0.0439]	19518 [R <sub>int</sub> = 0.0204, R <sub>sigma</sub> = 0.0227]	479 [R <sub>int</sub> = 0.0723, R <sub>sigma</sub> = 0.0544]	1508 [R <sub>int</sub> = 0.0262, R <sub>sigma</sub> = 0.0162]
Data/restraints/parameters	3419/0/307	4691/18/362	5035/0/433	19518/40/1619	479/15/71	1508/10/139
Goodness-of-fit on F <sup>2</sup>	1.047	1.031	1.02	1.096	1.052	1.073
Final R indexes [I ≥ 2σ (I)]	R <sub>1</sub> = 0.0425, wR <sub>2</sub> = 0.1113	R <sub>1</sub> = 0.0657, wR <sub>2</sub> = 0.1649	R <sub>1</sub> = 0.0426, wR <sub>2</sub> = 0.1082	R <sub>1</sub> = 0.0742, wR <sub>2</sub> = 0.2110	R <sub>1</sub> = 0.0670, wR <sub>2</sub> = 0.2199	R <sub>1</sub> = 0.0677, wR <sub>2</sub> = 0.1885
Final R indexes [all data]	R <sub>1</sub> = 0.0453, wR <sub>2</sub> = 0.1156	R <sub>1</sub> = 0.0726, wR <sub>2</sub> = 0.1716	R <sub>1</sub> = 0.0541, wR <sub>2</sub> = 0.1159	R <sub>1</sub> = 0.0897, wR <sub>2</sub> = 0.2254	R <sub>1</sub> = 0.0758, wR <sub>2</sub> = 0.2305	R <sub>1</sub> = 0.0773, wR <sub>2</sub> = 0.1970
Largest diff. peak/hole / e Å <sup>-3</sup>	0.26/-0.24	1.39/-0.94	0.34/-0.27	1.37/-0.85	0.23/-0.21	0.27/-0.43

	[2]C	[3]Ba	[3]Bβ	[3]C	[3]Fe <sup>III</sup> C	[4]B
Empirical formula	C <sub>24</sub> H <sub>18</sub> N <sub>18</sub> O <sub>8</sub> Cl <sub>2</sub> Fe	C <sub>26.5</sub> H <sub>33</sub> B <sub>2</sub> N <sub>16</sub> F <sub>8</sub> FeC 1	C <sub>26.57</sub> H <sub>33.13</sub> N <sub>16</sub> Cl <sub>1.13</sub> FeF <sub>8</sub> B <sub>2</sub>	C <sub>29</sub> H <sub>41</sub> N <sub>19</sub> O <sub>14</sub> Cl <sub>2</sub> Fe	C <sub>26</sub> H <sub>32</sub> N <sub>16</sub> O <sub>10</sub> FeCl <sub>2.5</sub>	C <sub>23</sub> H <sub>25.5</sub> N <sub>16.5</sub> FeB <sub>2</sub> F <sub>8</sub>
Formula weight	1082.49	840.61	846.27	1006.56	873.15	762.57
Temperature/K	173.15	120.15	120.15	119.99(13)	120.15	119.97(12)
Crystal system	cubic	triclinic	triclinic	monoclinic	orthorhombic	tetragonal
Space group	Ia-3d	P-1	P-1	P2 <sub>1</sub> /c	Pccn	I4 <sub>1</sub> /a
a/Å	18.6873(7)	10.0683(2)	15.2623(4)	24.3026(5)	13.5412(3)	16.69418(16)
b/Å	18.6873(7)	17.5266(4)	15.8321(4)	8.05029(13)	37.7323(8)	16.69418(16)
c/Å	18.6873(7)	20.7431(4)	26.6969(6)	25.5700(6)	14.4418(3)	23.9859(4)
α/°	90	93.730(2)	73.692(2)	90	90	90
β/°	90	97.211(2)	76.320(2)	117.725(3)	90	90
γ/°	90	91.065(2)	66.569(2)	90	90	90
Volume/Å <sup>3</sup>	6525.9(4)	3622.42(13)	5623.4(3)	4428.27(19)	7378.9(3)	6684.77(16)
Z	7	4	6	4	8	8
ρ <sub>calc</sub> /mg/mm <sup>3</sup>	1.928	1.541	1.499	1.51	1.572	1.515
μ/mm <sup>-1</sup>	6.265	4.801	4.728	4.576	5.612	4.429
2θ range for data collection	11.6 to 147.66°	6.414 to 147.756°	6.222 to 147.578°	6.93 to 147.526°	6.936 to 147.802°	6.45 to 147.858°
Index ranges	-15 ≤ h ≤ 11, -8 ≤ k ≤ 21, -23 ≤ l ≤ 15	-12 ≤ h ≤ 11, -20 ≤ k ≤ 21, -17 ≤ l ≤ 25	-13 ≤ h ≤ 18, -17 ≤ k ≤ 19, -32 ≤ l ≤ 33	-30 ≤ h ≤ 28, -6 ≤ k ≤ 9, -27 ≤ l ≤ 31	-16 ≤ h ≤ 16, -46 ≤ k ≤ 45, -17 ≤ l ≤ 11	-19 ≤ h ≤ 19, -12 ≤ k ≤ 20, -28 ≤ l ≤ 19
Reflections collected	2322	31645	50241	17259	20957	8157
Independent reflections	549 [R <sub>int</sub> = 0.0424, R <sub>sigma</sub> = 0.0253]	13735 [R <sub>int</sub> = 0.0392, R <sub>sigma</sub> = 0.0407]	21225 [R <sub>int</sub> = 0.0319, R <sub>sigma</sub> = 0.0363]	8715 [R <sub>int</sub> = 0.0287, R <sub>sigma</sub> = 0.0381]	7345 [R <sub>int</sub> = 0.0516, R <sub>sigma</sub> = 0.0528]	3279 [R <sub>int</sub> = 0.0331, R <sub>sigma</sub> = 0.0314]
Data/restraints/parameters	549/0/51	13735/0/990	21225/212/1466	8715/20/589	7345/4/508	3279/3/235
Goodness-of-fit on F <sup>2</sup>	1.534	1.033	1.054	1.025	1.1	1.037
Final R indexes [I ≥ 2σ (I)]	R <sub>1</sub> = 0.1585, wR <sub>2</sub> = 0.3700	R <sub>1</sub> = 0.0424, wR <sub>2</sub> = 0.1097	R <sub>1</sub> = 0.0872, wR <sub>2</sub> = 0.2430	R <sub>1</sub> = 0.0446, wR <sub>2</sub> = 0.1102	R <sub>1</sub> = 0.0663, wR <sub>2</sub> = 0.1537	R <sub>1</sub> = 0.0643, wR <sub>2</sub> = 0.1859
Final R indexes [all data]	R <sub>1</sub> = 0.2249, wR <sub>2</sub> = 0.4397	R <sub>1</sub> = 0.0459, wR <sub>2</sub> = 0.1131	R <sub>1</sub> = 0.1030, wR <sub>2</sub> = 0.2606	R <sub>1</sub> = 0.0498, wR <sub>2</sub> = 0.1138	R <sub>1</sub> = 0.0788, wR <sub>2</sub> = 0.1606	R <sub>1</sub> = 0.0686, wR <sub>2</sub> = 0.1920
Largest diff. peak/hole / e Å <sup>-3</sup>	0.62/-0.81	0.54/-0.79	0.91/-0.85	0.85/-0.50	0.88/-0.71	1.41/-0.53

	[4]Co	[5]B	[6]B	[8]B $\alpha$	[8]B $\beta$	[9]B
Empirical formula	C <sub>24</sub> H <sub>27</sub> B <sub>2</sub> CoF <sub>8</sub> N <sub>17</sub>	C <sub>24</sub> H <sub>26</sub> B <sub>2</sub> N <sub>14</sub> F <sub>8</sub> FeS <sub>2</sub>	C <sub>30</sub> H <sub>22</sub> B <sub>2</sub> F <sub>8</sub> FeN <sub>14</sub> O <sub>2</sub>	C <sub>38</sub> H <sub>46</sub> Cl <sub>4</sub> FeB <sub>2</sub> F <sub>8</sub> N <sub>18</sub>	C <sub>37.23</sub> H <sub>44.7</sub> B <sub>2</sub> Cl <sub>0.51</sub> F <sub>8</sub> eN <sub>18</sub> O <sub>0.24</sub>	C <sub>51.55</sub> H <sub>59.55</sub> N <sub>19.55</sub> FeF <sub>8</sub> Cl <sub>0.9</sub> B <sub>2</sub>
Formula weight	786.17	804.18	840.08	1126.2	995.59	1214.41
Temperature/K	120.15	120.1(2)	290.54(10)	120.15	120.1(3)	120.15
Crystal system	tetragonal	monoclinic	tetragonal	monoclinic	triclinic	triclinic
Space group	I4 <sub>1</sub> /a	P2 <sub>1</sub> /c	I4 <sub>1</sub> /a	C2/c	P-1	P-1
a/Å	16.8248(3)	10.6294(4)	19.690(3)	27.3406(5)	16.1734(6)	11.2397(4)
b/Å	16.8248(3)	18.5191(7)	19.690(3)	9.9219(2)	16.5387(5)	11.3391(4)
c/Å	23.8974(6)	16.9812(5)	17.537(6)	18.1090(4)	30.6371(11)	22.2242(7)
$\alpha$ /°	90	90	90	90	100.281(3)	78.760(3)
$\beta$ /°	90	91.214(3)	90	91.578(2)	91.354(3)	83.433(3)
$\gamma$ /°	90	90	90	90	106.688(3)	84.586(3)
Volume/Å <sup>3</sup>	6764.7(3)	3341.9(2)	6799(3)	4910.59(19)	7699.4(5)	2752.30(16)
Z	8	4	8	4	6	2
$\rho_{\text{calc}}$ /mg/mm <sup>3</sup>	1.544	1.598	1.641	1.523	1.288	1.465
$\mu$ /mm <sup>-1</sup>	4.799	5.578	4.448	5.18	3.256	3.324
2 $\theta$ range for data collection	10.494 to 147.324°	7.064 to 147.572°	6.75 to 148.094°	9.484 to 147.716°	5.722 to 124.994°	8.152 to 147.852°
Index ranges	-19 ≤ h ≤ 19, -20 ≤ k ≤ 17, -29 ≤ l ≤ 22	-13 ≤ h ≤ 12, -22 ≤ k ≤ 18, -20 ≤ l ≤ 18	-24 ≤ h ≤ 23, -22 ≤ k ≤ 16, -20 ≤ l ≤ 21	-33 ≤ h ≤ 34, -7 ≤ k ≤ 11, -18 ≤ l ≤ 21	-15 ≤ h ≤ 18, -18 ≤ k ≤ 18, -35 ≤ l ≤ 33	-13 ≤ h ≤ 12, -11 ≤ k ≤ 14, -25 ≤ l ≤ 27
Reflections collected	8664	13488	11718	7961	48224	23761
Independent reflections	3328 [R <sub>int</sub> = 0.0262, R <sub>sigma</sub> = 0.0281]	6567 [R <sub>int</sub> = 0.0383, R <sub>sigma</sub> = 0.0438]	3385 [R <sub>int</sub> = 0.1552, R <sub>sigma</sub> = 0.1714]	4725 [R <sub>int</sub> = 0.0421, R <sub>sigma</sub> = 0.0585]	23213 [R <sub>int</sub> = 0.0456, R <sub>sigma</sub> = 0.0604]	10379 [R <sub>int</sub> = 0.0354, R <sub>sigma</sub> = 0.0412]
Data/restraints/parameters	3328/8/256	6567/20/485	3385/30/271	4725/0/327	23213/1339/1827	10379/23/753
Goodness-of-fit on F <sup>2</sup>	1.045	1.093	1.014	1.018	1.771	1.042
Final R indexes [I ≥ 2 $\sigma$ (I)]	R <sub>1</sub> = 0.0552, wR <sub>2</sub> = 0.1514	R <sub>1</sub> = 0.0818, wR <sub>2</sub> = 0.2260	R <sub>1</sub> = 0.1071, wR <sub>2</sub> = 0.2459	R <sub>1</sub> = 0.0551, wR <sub>2</sub> = 0.1381	R <sub>1</sub> = 0.1683, wR <sub>2</sub> = 0.4429	R <sub>1</sub> = 0.0659, wR <sub>2</sub> = 0.1804
Final R indexes [all data]	R <sub>1</sub> = 0.0581, wR <sub>2</sub> = 0.1537	R <sub>1</sub> = 0.0883, wR <sub>2</sub> = 0.2308	R <sub>1</sub> = 0.2491, wR <sub>2</sub> = 0.3354	R <sub>1</sub> = 0.0669, wR <sub>2</sub> = 0.1493	R <sub>1</sub> = 0.2053, wR <sub>2</sub> = 0.4740	R <sub>1</sub> = 0.0746, wR <sub>2</sub> = 0.1921
Largest diff. peak/hole / e Å <sup>-3</sup>	0.62/-0.46	2.53/-0.69	0.46/-0.47	0.77/-0.63	2.87/-0.92	1.73/-0.70

<b>[12]B</b>	
Empirical formula	C <sub>22</sub> H <sub>22</sub> N <sub>14</sub> FeB <sub>2</sub> F <sub>8</sub> O <sub>2</sub>
Formula weight	744
Temperature/K	120.0(2)
Crystal system	tetragonal
Space group	I4 <sub>1</sub> /a
a/Å	18.4986(3)
b/Å	18.4986(3)
c/Å	17.6215(3)
$\alpha$ /°	90
$\beta$ /°	90
$\gamma$ /°	90
Volume/Å <sup>3</sup>	6030.0(2)
Z	8
$\rho_{\text{calc}}$ /mm <sup>3</sup>	1.639
$\mu$ /mm <sup>-1</sup>	4.921
2 $\theta$ range for data collection	6.928 to 147.582°
Index ranges	-17 ≤ h ≤ 17, -22 ≤ k ≤ 21, 12, -15 ≤ l ≤ 21
Reflections collected	6770
Independent reflections	2980 [R <sub>int</sub> = 0.0362, R <sub>sigma</sub> = 0.0390]
Data/restraints/parameters	2980/20/244
Goodness-of-fit on F <sup>2</sup>	1.037
Final R indexes [I ≥ 2σ (I)]	R <sub>1</sub> = 0.0425, wR <sub>2</sub> = 0.1094
Final R indexes [all data]	R <sub>1</sub> = 0.0501, wR <sub>2</sub> = 0.1151
Largest diff. peak/hole / e Å <sup>-3</sup>	0.41/-0.40

**Crystallographic Data Tables of Chapter 3**

	(15)	(16)	[2]Cu $\alpha$	[2]Cu $\beta$	[2]CoB	[2]NiB
Empirical formula	C <sub>8</sub> H <sub>5</sub> F <sub>2</sub> N <sub>3</sub>	C <sub>14</sub> H <sub>11</sub> N <sub>7</sub>	C <sub>54.25</sub> H <sub>55.75</sub> N <sub>42.25</sub> O <sub>13</sub> . 5Cu <sub>2</sub> B <sub>3</sub> F <sub>12</sub>	C <sub>18</sub> Cu <sub>2.5</sub> F <sub>6</sub> N <sub>16</sub> H <sub>0.13</sub>	C <sub>24</sub> H <sub>12</sub> B <sub>2</sub> CoF <sub>8</sub> N <sub>18</sub>	C <sub>24</sub> H <sub>18</sub> B <sub>2</sub> F <sub>8</sub> N <sub>18</sub> Ni
Formula weight	181.15	277.3	1903.17	713.53	779.2	790.89
Temperature/K	125.00(10)	120.15	120.15	120.1(3)	173.15	120.15
Crystal system	monoclinic	orthorhombic	triclinic	cubic	cubic	cubic
Space group	Pc	Pna2 <sub>1</sub>	P-1	Ia-3d	Ia-3d	Ia-3d
a/Å	3.7144(1)	17.6194(3)	12.3863(2)	19.3722(16)	18.4968(7)	18.4872(14)
b/Å	22.5373(4)	4.80380(10)	16.8435(4)	19.3722(16)	18.4968(7)	18.4872(14)
c/Å	27.5151(4)	30.6786(5)	21.0449(5)	19.3722(16)	18.4968(7)	18.4872(14)
$\alpha$ /°	90	90	107.687(2)	90	90	90
$\beta$ /°	95.599(1)	90	91.6590(17)	90	90	90
$\gamma$ /°	90	90	106.9559(18)	90	90	90
Volume/Å <sup>3</sup>	2292.37(8)	2596.64(8)	3967.74(16)	7270.0(18)	6328.4(7)	6318.5(14)
Z	12	8	2	7.99968	7.99968	8
$\rho_{\text{calc}}$ /mg/mm <sup>3</sup>	1.575	1.419	1.593	1.304	1.636	1.663
$\mu$ /mm <sup>-1</sup>	1.171	0.763	1.682	2.318	4.754	1.787
2 $\theta$ range for data collection	7.56 to 147.18°	10.04 to 147.76°	7.524 to 148°	17.126 to 108.136°	13.54 to 147.28°	13.548 to 147.484°
Index ranges	-2 ≤ h ≤ 4, -27 ≤ k ≤ 25, -33 ≤ l ≤ 32	-21 ≤ h ≤ 20, -5 ≤ k ≤ 4, -38 ≤ l ≤ 37	-15 ≤ h ≤ 14, -20 ≤ k ≤ 20, -25 ≤ l ≤ 25	-12 ≤ h ≤ 13, -16 ≤ k ≤ 18, -20 ≤ l ≤ 15	-14 ≤ h ≤ 5, -21 ≤ k ≤ 8, -22 ≤ l ≤ 5	-22 ≤ h ≤ 8, -15 ≤ k ≤ 22, -6 ≤ l ≤ 13
Reflections collected	8315	16693	33988	1932	2333	2176
Independent reflections	4502 [R <sub>int</sub> = 0.0293, R <sub>sigma</sub> = 0.0409]	2639 [R <sub>int</sub> = 0.0371, R <sub>sigma</sub> = 0.0222]	15025 [R <sub>int</sub> = 0.0315, R <sub>sigma</sub> = 0.0337]	372 [R <sub>int</sub> = 0.0224, R <sub>sigma</sub> = 0.0135]	522 [R <sub>int</sub> = 0.0262, R <sub>sigma</sub> = 0.0180]	521 [R <sub>int</sub> = 0.0195, R <sub>sigma</sub> = 0.0146]
Data/restraints/parameters	4502/2/703	2639/1/445	15025/93/1131	372/10/86	522/13/71	521/15/71
Goodness-of-fit on F <sup>2</sup>	1.021	1.033	1.033	2.909	1.124	1.09
Final R indexes [I ≥ 2 $\sigma$ (I)]	R <sub>1</sub> = 0.0356, wR <sub>2</sub> = 0.0918	R <sub>1</sub> = 0.0280, wR <sub>2</sub> = 0.0716	R <sub>1</sub> = 0.0742, wR <sub>2</sub> = 0.2102	R <sub>1</sub> = 0.2312, wR <sub>2</sub> = 0.5548	R <sub>1</sub> = 0.0779, wR <sub>2</sub> = 0.2263	R <sub>1</sub> = 0.0781, wR <sub>2</sub> = 0.2314
Final R indexes [all data]	R <sub>1</sub> = 0.0372, wR <sub>2</sub> = 0.0940	R <sub>1</sub> = 0.0291, wR <sub>2</sub> = 0.0727	R <sub>1</sub> = 0.0805, wR <sub>2</sub> = 0.2184	R <sub>1</sub> = 0.2576, wR <sub>2</sub> = 0.5915	R <sub>1</sub> = 0.0979, wR <sub>2</sub> = 0.2492	R <sub>1</sub> = 0.0926, wR <sub>2</sub> = 0.2621
Largest diff. peak/hole / e Å <sup>-3</sup>	0.21/-0.29	0.13/-0.20	1.67/-0.92	1.29/-0.90	0.21/-0.29	0.24/-0.13

	[2]MnCa	[2]MnCb	[2]AgBa	[2]AgBb	[2]AgC
Empirical formula	C <sub>24</sub> H <sub>18</sub> Cl <sub>2</sub> MnN <sub>18</sub> O <sub>8</sub>	C <sub>91.8</sub> H <sub>86.4</sub> Cl <sub>8</sub> Mn <sub>4</sub> N <sub>70.8</sub> O <sub>55.6</sub>	C <sub>13</sub> H <sub>12</sub> N <sub>10</sub> O <sub>2</sub> AgF <sub>4</sub> B	C <sub>12</sub> H <sub>9</sub> AgBF <sub>4</sub> N <sub>9</sub>	C <sub>12</sub> H <sub>9</sub> AgClN <sub>9</sub> O <sub>4</sub>
Formula weight	793.59	3590.61	535.01	473.96	486.6
Temperature/K	120.15	120.15	120.15	120.15	120.15
Crystal system	cubic	monoclinic	monoclinic	orthorhombic	orthorhombic
Space group	Ia-3d	P2/c	P2 <sub>1</sub>	Pca2 <sub>1</sub>	Pca2 <sub>1</sub>
a/Å	18.7291(13)	12.5714(3)	10.8501(6)	17.9041(8)	18.3941(9)
b/Å	18.7291(13)	18.1601(4)	12.4336(6)	10.1194(3)	10.1003(3)
c/Å	18.7291(13)	32.1404(12)	14.5101(9)	18.2383(6)	18.0884(7)
α/°	90	90	90	90	90
β/°	90	92.044(3)	108.445(7)	90	90
γ/°	90	90	90	90	90
Volume/Å <sup>3</sup>	6569.8(14)	7332.9(4)	1856.94(19)	3304.4(2)	3360.6(2)
Z	7.99968	2	4	8	8
ρ <sub>calc</sub> /mg/mm <sup>3</sup>	1.605	1.626	1.914	1.905	1.924
μ/mm <sup>-1</sup>	4.234	5.067	9.424	10.38	11.498
2θ range for data collection	17.72 to 148.228°	5.6 to 148.44°	8.59 to 147.69°	8.74 to 147.92°	8.76 to 147.14°
Index ranges	-23 ≤ h ≤ 1, -21 ≤ k ≤ 9, -13 ≤ l ≤ 15	-10 ≤ h ≤ 14, -22 ≤ k ≤ 18, -35 ≤ l ≤ 39	-8 ≤ h ≤ 13, -13 ≤ k ≤ 15, -17 ≤ l ≤ 14	-19 ≤ h ≤ 21, -8 ≤ k ≤ 12, -22 ≤ l ≤ 21	-22 ≤ h ≤ 18, -7 ≤ k ≤ 12, -22 ≤ l ≤ 15
Reflections collected	2202	32578	8889	13057	8711
Independent reflections	543 [R <sub>int</sub> = 0.0336, R <sub>sigma</sub> = 0.0230]	14082 [R <sub>int</sub> = 0.0563, R <sub>sigma</sub> = 0.0665]	5181 [R <sub>int</sub> = 0.0524, R <sub>sigma</sub> = 0.0658]	6138 [R <sub>int</sub> = 0.0540, R <sub>sigma</sub> = 0.0642]	4890 [R <sub>int</sub> = 0.0442, R <sub>sigma</sub> = 0.0561]
Data/restraints/parameters	543/15/72	14082/80/1050	5181/18/562	6138/41/479	4890/51/524
Goodness-of-fit on F <sup>2</sup>	1.319	1.037	1.057	1.041	1.06
Final R indexes [I ≥ 2σ (I)]	R <sub>1</sub> = 0.1009, wR <sub>2</sub> = 0.3069	R <sub>1</sub> = 0.0920, wR <sub>2</sub> = 0.2435	R <sub>1</sub> = 0.0864, wR <sub>2</sub> = 0.2219	R <sub>1</sub> = 0.0471, wR <sub>2</sub> = 0.1160	R <sub>1</sub> = 0.0657, wR <sub>2</sub> = 0.1698
Final R indexes [all data]	R <sub>1</sub> = 0.1144, wR <sub>2</sub> = 0.3308	R <sub>1</sub> = 0.1146, wR <sub>2</sub> = 0.2633	R <sub>1</sub> = 0.0879, wR <sub>2</sub> = 0.2241	R <sub>1</sub> = 0.0561, wR <sub>2</sub> = 0.1232	R <sub>1</sub> = 0.0791, wR <sub>2</sub> = 0.1840
Largest diff. peak/hole / e Å <sup>-3</sup>	0.24/-0.35	1.32/-1.05	5.13/-1.65	0.77/-0.77	3.22/-1.22

	[2]AgB MeOH	[13]AgB MeCN	[13]AgB MeNO <sub>2</sub> α	[13]AgB MeNO <sub>2</sub> β	[14]AgB MeCN	[14]AgB MeNO <sub>2</sub>
Empirical formula	C <sub>13</sub> H <sub>13</sub> AgBF <sub>4</sub> N <sub>9</sub> O	C <sub>36</sub> H <sub>34</sub> N <sub>18</sub> B <sub>2</sub> F <sub>8</sub> Ag <sub>2</sub>	C <sub>28.3</sub> H <sub>22.9</sub> N <sub>14.3</sub> O <sub>0.6</sub> Ag <sub>2</sub> F <sub>8</sub> B <sub>2</sub>	C <sub>29</sub> H <sub>25</sub> N <sub>15</sub> O <sub>2</sub> B <sub>2</sub> F <sub>8</sub> Ag <sub>2</sub>	C <sub>30</sub> H <sub>26</sub> Ag <sub>2</sub> B <sub>2</sub> F <sub>8</sub> N <sub>18</sub>	C <sub>52</sub> H <sub>40</sub> Ag <sub>5</sub> B <sub>2</sub> F <sub>8</sub> N <sub>32</sub>
Formula weight	506	1108.17	962.27	1005	1028.05	2208.65
Temperature/K	120.15	120.15	120.15	120.15	279.56(10)	120.15
Crystal system	orthorhombic	monoclinic	monoclinic	triclinic	monoclinic	monoclinic
Space group	Pbca	P2 <sub>1</sub> /c	P2 <sub>1</sub> /c	P-1	P2 <sub>1</sub>	C2/c
a/Å	15.4505(4)	12.0956(3)	25.2990(6)	12.1548(7)	11.3714(3)	29.8766(6)
b/Å	13.9523(4)	6.87477(17)	11.6702(3)	12.6789(8)	6.8555(2)	10.9444(2)
c/Å	16.3341(4)	26.4110(5)	23.1012(5)	13.5860(8)	23.7996(6)	25.5649(8)
α/°	90	90	90	66.821(6)	90	90
β/°	90	93.935(2)	91.705(2)	65.231(5)	90.420(2)	115.208(2)
γ/°	90	90	90	72.775(5)	90	90
Volume/Å <sup>3</sup>	3521.14(16)	2191.01(9)	6817.5(3)	1725.9(2)	1855.29(9)	7563.2(3)
Z	8	2	8	2	2	4
ρ <sub>calc</sub> /mg/mm <sup>3</sup>	1.909	1.68	1.875	1.934	1.84	1.94
μ/mm <sup>-1</sup>	9.831	7.927	10.054	1.235	9.303	1.391
2θ range for data collection	10.12 to 147.42°	6.71 to 147.746°	7.658 to 147.59°	5.536 to 59.298°	7.42 to 147.88°	3.42 to 50.14°
Index ranges	-15 ≤ h ≤ 18, -14 ≤ k ≤ 17, -20 ≤ l ≤ 13	-14 ≤ h ≤ 12, -8 ≤ k ≤ 7, -32 ≤ l ≤ 28	-30 ≤ h ≤ 25, -13 ≤ k ≤ 10, -28 ≤ l ≤ 22	-16 ≤ h ≤ 16, -15 ≤ k ≤ 17, -18 ≤ l ≤ 14	-13 ≤ h ≤ 14, -8 ≤ k ≤ 8, -23 ≤ l ≤ 28	-36 ≤ h ≤ 36, -11 ≤ k ≤ 13, -31 ≤ l ≤ 27
Reflections collected	20146	8334	29229	18262	8140	15662
Independent reflections	3515 [R <sub>int</sub> = 0.0343, R <sub>sigma</sub> = 0.0214]	4291 [R <sub>int</sub> = 0.0314, R <sub>sigma</sub> = 0.0384]	13111 [R <sub>int</sub> = 0.0385, R <sub>sigma</sub> = 0.0444]	8207 [R <sub>int</sub> = 0.0284, R <sub>sigma</sub> = 0.0439]	5174 [R <sub>int</sub> = 0.0267, R <sub>sigma</sub> = 0.0368]	5480 [R <sub>int</sub> = 0.0416, R <sub>sigma</sub> = 0.0533]
Data/restraints/parameters	3515/0/266	4291/36/314	13111/1/975	8207/0/519	5174/21/539	5480/28/458
Goodness-of-fit on F <sup>2</sup>	1.059	1.042	1.032	1.044	1.141	1.607
Final R indexes [I ≥ 2σ (I)]	R <sub>1</sub> = 0.0274, wR <sub>2</sub> = 0.0629	R <sub>1</sub> = 0.0573, wR <sub>2</sub> = 0.1534	R <sub>1</sub> = 0.0540, wR <sub>2</sub> = 0.1408	R <sub>1</sub> = 0.0360, wR <sub>2</sub> = 0.0689	R <sub>1</sub> = 0.0509, wR <sub>2</sub> = 0.1339	R <sub>1</sub> = 0.0870, wR <sub>2</sub> = 0.2568
Final R indexes [all data]	R <sub>1</sub> = 0.0334, wR <sub>2</sub> = 0.0656	R <sub>1</sub> = 0.0644, wR <sub>2</sub> = 0.1615	R <sub>1</sub> = 0.0680, wR <sub>2</sub> = 0.1512	R <sub>1</sub> = 0.0490, wR <sub>2</sub> = 0.0752	R <sub>1</sub> = 0.0519, wR <sub>2</sub> = 0.1345	R <sub>1</sub> = 0.1127, wR <sub>2</sub> = 0.2701
Largest diff. peak/hole / e Å <sup>-3</sup>	0.77/-0.73	1.57/-1.08	1.98/-1.76	0.69/-0.77	1.46/-1.26	2.98/-1.08



	<b>[16]AgB</b>	<b>[2]Mn (tFacac)</b>	<b>[14]B</b>	<b>[14]C</b>	<b>[16]B</b>	<b>[16]C</b>
Empirical formula	C <sub>15</sub> H <sub>12.5</sub> N <sub>7.5</sub> AgF <sub>4</sub> B	C <sub>22</sub> H <sub>9</sub> N <sub>9</sub> O <sub>4</sub> F <sub>12</sub> Mn	C <sub>26</sub> H <sub>20</sub> B <sub>2</sub> F <sub>8</sub> FeN <sub>16</sub>	C <sub>26.9</sub> H <sub>23.2</sub> N <sub>16.9</sub> O <sub>9.93</sub> Cl <sub>2</sub> Fe	C <sub>28</sub> H <sub>22</sub> B <sub>2</sub> F <sub>8</sub> FeN <sub>14</sub>	C <sub>28</sub> H <sub>22</sub> Cl <sub>2</sub> FeN <sub>14</sub> O <sub>8</sub>
Formula weight	492.51	746.32	786.05	868.77	784.06	809.34
Temperature/K	120.00(14)	120.15	120.15	120.15	120.15	120.15
Crystal system	triclinic	triclinic	orthorhombic	monoclinic	monoclinic	monoclinic
Space group	P-1	P-1	Fddd	P2 <sub>1</sub> /n	C2/c	C2/c
a/Å	7.7430(4)	10.6998(3)	17.8353(8)	15.2393(2)	22.6253(11)	22.817(2)
b/Å	14.1195(7)	11.7470(4)	25.3177(13)	31.6772(3)	10.9031(6)	10.9391(11)
c/Å	16.2206(8)	12.3563(4)	27.4906(11)	15.9371(2)	14.8499(8)	14.9832(19)
$\alpha$ /°	79.265(4)	85.926(3)	90	90	90	90
$\beta$ /°	81.328(4)	89.053(3)	90	111.9450(10)	121.921(3)	121.911(7)
$\gamma$ /°	83.528(4)	63.320(3)	90	90	90	90
Volume/Å <sup>3</sup>	1715.85(15)	1384.00(8)	12413.3(10)	7136.00(15)	3109.3(3)	3174.6(6)
Z	4	2	16	8	4	4
$\rho_{\text{calc}}$ /mm <sup>3</sup>	1.907	1.791	1.682	1.617	1.675	1.693
$\mu$ /mm <sup>-1</sup>	9.998	5.106	4.793	5.477	4.763	6.031
2 $\theta$ range for data collection	6.4 to 147.98°	7.174 to 134.098°	6.86 to 147.2°	6.598 to 147.592°	9.21 to 147.02°	9.132 to 147.374°
Index ranges	-9 ≤ h ≤ 7, -17 ≤ k ≤ 14, -18 ≤ l ≤ 20	-12 ≤ h ≤ 9, -13 ≤ k ≤ 10, -14 ≤ l ≤ 14	-17 ≤ h ≤ 21, -29 ≤ k ≤ 31, -34 ≤ l ≤ 26	-18 ≤ h ≤ 13, -31 ≤ k ≤ 38, -14 ≤ l ≤ 19	-27 ≤ h ≤ 25, -13 ≤ k ≤ 11, -12 ≤ l ≤ 17	-28 ≤ h ≤ 28, -13 ≤ k ≤ 10, -17 ≤ l ≤ 14
Reflections collected	12912	10655	14884	31568	5937	6406
Independent reflections	6490 [R <sub>int</sub> = 0.0396, R <sub>sigma</sub> = 0.0505]	4760 [R <sub>int</sub> = 0.0161, R <sub>sigma</sub> = 0.0172]	3101 [R <sub>int</sub> = 0.0626, R <sub>sigma</sub> = 0.0447]	13640 [R <sub>int</sub> = 0.0292, R <sub>sigma</sub> = 0.0311]	2953 [R <sub>int</sub> = 0.0163, R <sub>sigma</sub> = 0.0220]	3062 [R <sub>int</sub> = 0.0488, R <sub>sigma</sub> = 0.0595]
Data/restraints/parameters	6490/0/515	4760/0/433	3101/6/293	13640/72/1059	2953/0/240	3062/0/240
Goodness-of-fit on F <sup>2</sup>	1.117	1.025	1.091	1.08	1.088	1.077
Final R indexes [I ≥ 2 $\sigma$ (I)]	R <sub>1</sub> = 0.0676, wR <sub>2</sub> = 0.1849	R <sub>1</sub> = 0.0342, wR <sub>2</sub> = 0.0920	R <sub>1</sub> = 0.0755, wR <sub>2</sub> = 0.2077	R <sub>1</sub> = 0.0673, wR <sub>2</sub> = 0.1831	R <sub>1</sub> = 0.0384, wR <sub>2</sub> = 0.1020	R <sub>1</sub> = 0.0665, wR <sub>2</sub> = 0.1730
Final R indexes [all data]	R <sub>1</sub> = 0.0748, wR <sub>2</sub> = 0.1893	R <sub>1</sub> = 0.0347, wR <sub>2</sub> = 0.0925	R <sub>1</sub> = 0.0851, wR <sub>2</sub> = 0.2157	R <sub>1</sub> = 0.0729, wR <sub>2</sub> = 0.1879	R <sub>1</sub> = 0.0398, wR <sub>2</sub> = 0.1032	R <sub>1</sub> = 0.0903, wR <sub>2</sub> = 0.1945
Largest diff. peak/hole / e Å <sup>-3</sup>	2.72/-1.13	0.80/-0.58	1.29/-0.57	1.26/-0.64	0.90/-0.45	1.42/-0.73

	<b>Fe<sup>III</sup>[16]<sub>8</sub>F<sub>8</sub> α</b>	<b>Fe<sup>III</sup>[16]<sub>8</sub>F<sub>8</sub> β</b>
Empirical formula	C <sub>114.5</sub> H <sub>98</sub> N <sub>56</sub> Fe <sub>3</sub> F <sub>1</sub> 2BO <sub>2.5</sub>	C <sub>110.5</sub> H <sub>81</sub> B <sub>0.25</sub> F <sub>9</sub> Fe <sub>3</sub> N <sub>59</sub>
Formula weight	2704.84	2576.5
Temperature/K	150.15	173.15
Crystal system	hexagonal	tetragonal
Space group	P6 <sub>1</sub> 22	P4 <sub>3</sub> 22
a/Å	17.7834(3)	16.8219(2)
b/Å	17.7834(3)	16.8219(2)
c/Å	72.6622(12)	89.587(2)
α/°	90	90
β/°	90	90
γ/°	120	90
Volume/Å <sup>3</sup>	19900.7(7)	25351.0(7)
Z	6	8
ρ <sub>calc</sub> /mg/mm <sup>3</sup>	1.354	1.35
μ/mm <sup>-1</sup>	3.341	3.429
2θ range for data collection	6.802 to 147.154°	10.56 to 147.94°
Index ranges	-21 ≤ h ≤ 15, -14 ≤ k ≤ 21, -88 ≤ l ≤ 39	-20 ≤ h ≤ 20, -20 ≤ k ≤ 20, -108 ≤ l ≤ 111
Reflections collected	45728	149768
Independent reflections	13216 [R <sub>int</sub> = 0.0439, R <sub>sigma</sub> = 0.0411]	25200 [R <sub>int</sub> = 0.1390, R <sub>sigma</sub> = 0.0805]
Data/restraints/parameters	13216/72/799	25200/22/1639
Goodness-of-fit on F <sup>2</sup>	1.025	2.886
Final R indexes [I ≥ 2σ (I)]	R <sub>1</sub> = 0.0865, wR <sub>2</sub> = 0.2484	R <sub>1</sub> = 0.1767, wR <sub>2</sub> = 0.4243
Final R indexes [all data]	R <sub>1</sub> = 0.1047, wR <sub>2</sub> = 0.2661	R <sub>1</sub> = 0.1828, wR <sub>2</sub> = 0.4280
Largest diff. peak/hole / e Å <sup>-3</sup>	0.62/-0.76	1.38/-1.63

**Crystallographic Data Tables of Chapter 4**

	<b>(17) (CHCl<sub>3</sub>)<sub>2</sub></b>	<b>(21)</b>	<b>(23)</b>	<b>(24)</b>	<b>(25)</b>	<b>(26)<i>a</i></b>
Empirical formula	C <sub>19</sub> H <sub>20</sub> N <sub>7</sub> Cl <sub>7</sub>	C <sub>9</sub> H <sub>7</sub> N <sub>7</sub>	C <sub>4</sub> H <sub>5</sub> Cl <sub>2</sub> N <sub>2</sub> NaO <sub>3</sub>	C <sub>10</sub> H <sub>8</sub> N <sub>6</sub> O	C <sub>11</sub> H <sub>10</sub> N <sub>6</sub>	C <sub>10</sub> H <sub>9</sub> N <sub>7</sub>
Formula weight	594.57	213.2	222.99	228.22	226.25	227.24
Temperature/K	120.15	119.98(14)	120.15	100.15	119.97(15)	120.15
Crystal system	triclinic	monoclinic	monoclinic	monoclinic	monoclinic	orthorhombic
Space group	P-1	P2 <sub>1</sub> /n	C2/c	P2 <sub>1</sub> /c	P2 <sub>1</sub> /c	P2 <sub>1</sub> 2 <sub>1</sub> 2 <sub>1</sub>
a/Å	8.8877(3)	14.3972(8)	31.8022(15)	3.7553(1)	7.3035(16)	3.84290(10)
b/Å	11.8205(5)	4.1060(2)	3.61080(10)	17.3319(4)	10.830(3)	14.8655(6)
c/Å	12.9737(5)	16.4295(11)	15.6872(7)	44.3260(14)	13.455(3)	18.0160(10)
α/°	111.562(4)	90	90	90	90	90
β/°	92.791(3)	103.275(7)	116.599(3)	92.170(3)	91.735(18)	90
γ/°	90.188(3)	90	90	90	90	90
Volume/Å <sup>3</sup>	1265.77(9)	945.28(10)	1610.73(12)	2882.95(14)	1063.7(4)	1029.19(8)
Z	2	4	8	12	4	4
ρ <sub>calc</sub> /mg/mm <sup>3</sup>	1.56	1.498	1.839	1.577	1.413	1.467
μ/mm <sup>-1</sup>	7.365	0.857	7.57	0.106	0.765	0.822
2θ range for data collection	7.338 to 147.608°	7.38 to 147.14°	6.216 to 147.324°	1.78 to 59.04°	10.48 to 147.42°	7.72 to 133.14°
Index ranges	-6 ≤ h ≤ 11, -14 ≤ k ≤ 14, -15 ≤ l ≤ 16	-17 ≤ h ≤ 17, -4 ≤ k ≤ 4, -14 ≤ l ≤ 20	-38 ≤ h ≤ 33, -3 ≤ k ≤ 4, -16 ≤ l ≤ 18	-5 ≤ h ≤ 5, -24 ≤ k ≤ 18, -63 ≤ l ≤ 63	-8 ≤ h ≤ 8, -12 ≤ k ≤ 9, -16 ≤ l ≤ 16	-4 ≤ h ≤ 4, -17 ≤ k ≤ 10, -21 ≤ l ≤ 19
Reflections collected	9655	3204	5509	35410	3921	3698
Independent reflections	4741 [R <sub>int</sub> = 0.0457, R <sub>sigma</sub> = 0.0450]	1814 [R <sub>int</sub> = 0.0299, R <sub>sigma</sub> = 0.0301]	1602 [R <sub>int</sub> = 0.0343, R <sub>sigma</sub> = 0.0307]	8769 [R <sub>int</sub> = 0.1863, R <sub>sigma</sub> = 0.1398]	2057 [R <sub>int</sub> = 0.0224, R <sub>sigma</sub> = 0.0283]	1119 [R <sub>int</sub> = 0.0289, R <sub>sigma</sub> = 0.0276]
Data/restraints/parameters	4741/0/298	1814/0/166	1602/0/121	8769/0/532	2057/0/176	1119/0/181
Goodness-of-fit on F <sup>2</sup>	1.063	1.089	1.07	0.915	1.184	1.156
Final R indexes [I ≥ 2σ(I)]	R <sub>1</sub> = 0.0651, wR <sub>2</sub> = 0.1766	R <sub>1</sub> = 0.0426, wR <sub>2</sub> = 0.1070	R <sub>1</sub> = 0.0333, wR <sub>2</sub> = 0.0867	R <sub>1</sub> = 0.0782, wR <sub>2</sub> = 0.1723	R <sub>1</sub> = 0.0684, wR <sub>2</sub> = 0.2411	R <sub>1</sub> = 0.0336, wR <sub>2</sub> = 0.0849
Final R indexes [all data]	R <sub>1</sub> = 0.0683, wR <sub>2</sub> = 0.1826	R <sub>1</sub> = 0.0512, wR <sub>2</sub> = 0.1174	R <sub>1</sub> = 0.0367, wR <sub>2</sub> = 0.0892	R <sub>1</sub> = 0.1180, wR <sub>2</sub> = 0.1889	R <sub>1</sub> = 0.0744, wR <sub>2</sub> = 0.2471	R <sub>1</sub> = 0.0355, wR <sub>2</sub> = 0.0864
Largest diff. peak/hole / e Å <sup>-3</sup>	1.32/-1.04	0.26/-0.29	0.51/-0.40	0.49/-0.41	0.51/-0.46	0.16/-0.22

	(26) $\beta$	(28)	(29)	[18] $\alpha$	[18] $\beta$	[18] $\beta$ second
Empirical formula	C <sub>10</sub> H <sub>9</sub> N <sub>7</sub>	C <sub>16</sub> H <sub>22</sub> N <sub>6</sub> O <sub>2</sub> S	C <sub>12</sub> H <sub>12</sub> N <sub>6</sub> O	C <sub>68</sub> H <sub>72</sub> N <sub>28</sub> O <sub>4</sub> Cl <sub>8</sub> Fe <sub>4</sub>	C <sub>204</sub> H <sub>204</sub> B <sub>6</sub> Cl <sub>12</sub> F <sub>24</sub> Fe <sub>12</sub> N <sub>84</sub> O <sub>18</sub>	B <sub>4</sub> C <sub>202</sub> Cl <sub>8</sub> F <sub>16</sub> Fe <sub>11</sub> N <sub>84</sub> O <sub>18</sub>
Formula weight	227.24	362.46	256.28	1852.54	5736.95	5136.05
Temperature/K	120.15	125.15	173.15	100.15	173(2)	119.99(13)
Crystal system	monoclinic	monoclinic	triclinic	tetragonal	trigonal	monoclinic
Space group	C2/c	P2 <sub>1</sub> /c	P-1	I4 <sub>1</sub> /a	R-3	P2/n
a/Å	17.0960(11)	20.9115(7)	6.3701(4)	15.8674(2)	31.3735(3)	20.7431(7)
b/Å	10.5273(4)	14.1951(6)	8.0327(6)	15.8674(2)	31.3735(3)	20.3593(7)
c/Å	13.4500(6)	12.6266(4)	12.0083(11)	33.3955(4)	26.6504(7)	33.5696(18)
$\alpha$ /°	90	90	77.757(7)	90	90	90
$\beta$ /°	121.235(4)	104.380(3)	88.305(6)	90	90	91.582(4)
$\gamma$ /°	90	90	75.102(6)	90	120	90
Volume/Å <sup>3</sup>	2069.78(18)	3630.7(2)	580.09(8)	8408.13(18)	22717.5(7)	14171.5(10)
Z	8	8	2	4	3.000006	2
$\rho_{\text{calc}}$ /mg/mm <sup>3</sup>	1.458	1.326	1.467	1.463	1.258	1.204
$\mu$ /mm <sup>-1</sup>	0.1	1.778	0.837	0.994	0.691	5.705
2 $\theta$ range for data collection	6.74 to 59.22°	7.6 to 146.92°	7.54 to 146.36°	2.76 to 72.16°	3.262 to 51.006°	6.084 to 147.7°
Index ranges	-21 ≤ h ≤ 17, -14 ≤ k ≤ 10, -16 ≤ l ≤ 18	-25 ≤ h ≤ 23, -9 ≤ k ≤ 17, -15 ≤ l ≤ 15	-7 ≤ h ≤ 5, -9 ≤ k ≤ 9, -12 ≤ l ≤ 14	-26 ≤ h ≤ 26, -25 ≤ k ≤ 27, -55 ≤ l ≤ 56	-39 ≤ h ≤ 36, -39 ≤ k ≤ 38, -33 ≤ l ≤ 31	-25 ≤ h ≤ 24, -25 ≤ k ≤ 24, -40 ≤ l ≤ 40
Reflections collected	5264	14542	4021	88895	54935	62251
Independent reflections	2471 [R <sub>int</sub> = 0.0188, R <sub>sigma</sub> = 0.0272]	7127 [R <sub>int</sub> = 0.0246, R <sub>sigma</sub> = 0.0298]	2144 [R <sub>int</sub> = 0.0355, R <sub>sigma</sub> = 0.0452]	10473 [R <sub>int</sub> = 0.1534, R <sub>sigma</sub> = 0.0921]	10262 [R <sub>int</sub> = 0.0629, R <sub>sigma</sub> = 0.0508]	27796 [R <sub>int</sub> = 0.0747, R <sub>sigma</sub> = 0.0952]
Data/restraints/parameters	2471/0/181	7127/2/469	2144/0/208	10473/0/253	10262/0/440	27796/0/1542
Goodness-of-fit on F <sup>2</sup>	1.099	1.031	1.037	1.202	1.31	1.253
Final R indexes [I ≥ 2 $\sigma$ (I)]	R <sub>1</sub> = 0.0403, wR <sub>2</sub> = 0.1024	R <sub>1</sub> = 0.0343, wR <sub>2</sub> = 0.0877	R <sub>1</sub> = 0.0512, wR <sub>2</sub> = 0.1359	R <sub>1</sub> = 0.1257, wR <sub>2</sub> = 0.3155	R <sub>1</sub> = 0.1250, wR <sub>2</sub> = 0.3530	R <sub>1</sub> = 0.1369, wR <sub>2</sub> = 0.3591
Final R indexes [all data]	R <sub>1</sub> = 0.0455, wR <sub>2</sub> = 0.1061	R <sub>1</sub> = 0.0405, wR <sub>2</sub> = 0.0918	R <sub>1</sub> = 0.0575, wR <sub>2</sub> = 0.1416	R <sub>1</sub> = 0.2825, wR <sub>2</sub> = 0.3493	R <sub>1</sub> = 0.1619, wR <sub>2</sub> = 0.3747	R <sub>1</sub> = 0.1965, wR <sub>2</sub> = 0.4051
Largest diff. peak/hole / e Å <sup>-3</sup>	0.31/-0.22	0.26/-0.31	0.28/-0.29	1.11/-0.59	1.33/-0.59	2.43/-0.91

	[18] $\gamma$	[18]OMe HBF <sub>4</sub>	[20] $\alpha$	[20] $\beta$	[21]B	[21]C
Empirical formula	C <sub>213</sub> H <sub>210</sub> BCl <sub>7.5</sub> F <sub>4</sub> Fe <sub>1</sub> 1.5N <sub>87</sub> O <sub>25</sub>	C <sub>18</sub> H <sub>22</sub> BF <sub>4</sub> N <sub>7</sub> O	C <sub>30</sub> H <sub>39</sub> N <sub>15</sub> Fe <sub>3</sub> Cl <sub>8</sub> O <sub>6</sub>	C <sub>20</sub> H <sub>26</sub> Cl <sub>5</sub> Fe <sub>2</sub> N <sub>10</sub> O <sub>4</sub>	C <sub>85.3</sub> H <sub>75.7</sub> B <sub>8.5</sub> F <sub>33.5</sub> Fe <sub>4</sub> N <sub>62.9</sub>	C <sub>48</sub> H <sub>54</sub> N <sub>38</sub> O <sub>10</sub> Fe <sub>3</sub> B <sub>6</sub> F <sub>24</sub>
Formula weight	5383.68	439.24	1156.91	759.45	2933.77	2011.7
Temperature/K	100(2)	290.72(10)	120.01(18)	120.02(19)	120.15	120.15
Crystal system	cubic	monoclinic	triclinic	monoclinic	triclinic	monoclinic
Space group	P2 <sub>1</sub> 3	P2 <sub>1</sub> /c	P1	P2 <sub>1</sub> /c	P-1	P2/c
a/Å	35.75390(10)	9.6550(12)	12.6310(6)	10.92289(14)	16.1862(6)	14.8615(5)
b/Å	35.75390(10)	17.5961(12)	12.8201(5)	15.6120(2)	17.2352(5)	21.7038(6)
c/Å	35.75390(10)	12.0628(6)	17.8520(8)	18.7573(2)	23.7427(7)	12.9700(4)
$\alpha$ /°	90	90	88.192(4)	90	97.839(2)	90
$\beta$ /°	90	91.546(7)	79.889(4)	101.7651(11)	91.234(3)	112.969(4)
$\gamma$ /°	90	90	61.112(5)	90	105.400(3)	90
Volume/Å <sup>3</sup>	45705.7(4)	2048.6(3)	2487.0(2)	3131.45(7)	6314.5(4)	3851.8(2)
Z	4	4	2	4	2	2
$\rho_{\text{calc}}$ /mg/mm <sup>3</sup>	0.782	1.424	1.545	1.6108	1.543	1.735
$\mu$ /mm <sup>-1</sup>	0.409	1.011	11.344	11.746	4.676	5.72
2 $\theta$ range for data collection	1.562 to 72.33°	8.9 to 147.2°	7.892 to 147.604°	7.44 to 147.48°	6.62 to 147.84°	6.46 to 147.492°
Index ranges	-42 ≤ h ≤ 60, -55 ≤ k ≤ 58, -48 ≤ l ≤ 35	-11 ≤ h ≤ 9, -10 ≤ k ≤ 21, -13 ≤ l ≤ 14	-15 ≤ h ≤ 14, -13 ≤ k ≤ 15, -21 ≤ l ≤ 22	-12 ≤ h ≤ 9, -19 ≤ k ≤ 13, -23 ≤ l ≤ 20	-19 ≤ h ≤ 19, -20 ≤ k ≤ 18, -26 ≤ l ≤ 29	-17 ≤ h ≤ 18, -25 ≤ k ≤ 26, -13 ≤ l ≤ 16
Reflections collected	477922	4372	20844	13185	54575	15271
Independent reflections	74650 [R <sub>int</sub> = 0.1645, R <sub>sigma</sub> = 0.1089]	2691 [R <sub>int</sub> = 0.0235, R <sub>sigma</sub> = 0.0342]	11955 [R <sub>int</sub> = 0.0483, R <sub>sigma</sub> = 0.0605]	6008 [R <sub>int</sub> = 0.0244, R <sub>sigma</sub> = 0.0307]	23884 [R <sub>int</sub> = 0.0327, R <sub>sigma</sub> = 0.0370]	7546 [R <sub>int</sub> = 0.0218, R <sub>sigma</sub> = 0.0290]
Data/restraints/parameters	74650/0/1079	2691/0/289	11955/3/1270	6008/0/378	23884/72/1755	7546/74/597
Goodness-of-fit on F <sup>2</sup>	0.943	1.107	1.022	1.052	1.022	1.089
Final R indexes [I ≥ 2 $\sigma$ (I)]	R <sub>1</sub> = 0.1397, wR <sub>2</sub> = 0.3170	R <sub>1</sub> = 0.0527, wR <sub>2</sub> = 0.1470	R <sub>1</sub> = 0.0483, wR <sub>2</sub> = 0.1192	R <sub>1</sub> = 0.0295, wR <sub>2</sub> = 0.0693	R <sub>1</sub> = 0.0777, wR <sub>2</sub> = 0.2221	R <sub>1</sub> = 0.0794, wR <sub>2</sub> = 0.2252
Final R indexes [all data]	R <sub>1</sub> = 0.2818, wR <sub>2</sub> = 0.3916	R <sub>1</sub> = 0.0722, wR <sub>2</sub> = 0.1631	R <sub>1</sub> = 0.0558, wR <sub>2</sub> = 0.1258	R <sub>1</sub> = 0.0352, wR <sub>2</sub> = 0.0727	R <sub>1</sub> = 0.0893, wR <sub>2</sub> = 0.2353	R <sub>1</sub> = 0.1092, wR <sub>2</sub> = 0.2538
Largest diff. peak/hole / e Å <sup>-3</sup>	3.82/-0.75	0.22/-0.31	0.78/-0.72	0.50/-0.42	1.21/-0.71	1.31/-1.03

	[21]Ca	[21]Ag	[22]B	[22]B	[22]B	[22]B
Empirical formula	C <sub>27</sub> H <sub>21</sub> N <sub>21</sub> O <sub>8</sub> Cl <sub>2</sub> C	C <sub>9</sub> H <sub>7</sub> N <sub>7</sub> AgF <sub>4</sub> B	C <sub>132</sub> H <sub>114</sub> B <sub>7</sub> N <sub>78</sub> O <sub>18</sub> F <sub>2</sub> 8Fe <sub>9</sub>	C <sub>132</sub> H <sub>114</sub> B <sub>7</sub> N <sub>78</sub> O <sub>18</sub> F <sub>28</sub> F e <sub>9</sub>	C <sub>132</sub> H <sub>114</sub> B <sub>7</sub> N <sub>78</sub> O <sub>18</sub> F <sub>28</sub> F e <sub>9</sub>	C <sub>132</sub> H <sub>114</sub> B <sub>7</sub> N <sub>78</sub> O <sub>18</sub> F <sub>28</sub> Fe 9
Formula weight	878.63	407.9	4191.33	4191.33	4191.33	4191.33
Temperature/K	119.99(18)	119.97(13)	100.15	150.15	200.15	250.15
Crystal system	trigonal	monoclinic	monoclinic	monoclinic	monoclinic	monoclinic
Space group	P-3c1	P2 <sub>1</sub> /m	C2/c	C2/c	C2/c	C2/c
a/Å	9.7608(4)	5.6623(1)	28.138(12)	28.1814(3)	28.3179(3)	28.4475(3)
b/Å	9.7608(4)	14.2076(3)	20.942(11)	20.9721(2)	21.0820(2)	21.1969(2)
c/Å	20.2060(10)	7.7351(2)	29.140(17)	29.1619(4)	29.2509(3)	29.3353(4)
α/°	90	90	90	90	90	90
β/°	90	91.837(2)	93.17(4)	93.064(1)	92.810(1)	92.571(1)
γ/°	120	90	90	90	90	90
Volume/Å <sup>3</sup>	1667.18(16)	621.95(2)	17145(15)	17210.7(3)	17441.7(3)	17671.3(3)
Z	2	2	4	4	4	4
ρ <sub>calc</sub> /mm <sup>3</sup>	1.75	2.178	1.624	1.618	1.596	1.575
μ/mm <sup>-1</sup>	3.873	13.589	0.789	0.786	0.776	0.766
2θ range for data collection	8.752 to 133.116°	13.04 to 147.6°	2.36 to 55°	2.34 to 50.14°	2.34 to 50.18°	2.66 to 50.14°
Index ranges	-7 ≤ h ≤ 11, -10 ≤ k ≤ 11, -24 ≤ l ≤ 21	-6 ≤ h ≤ 7, -15 ≤ k ≤ 17, -8 ≤ l ≤ 9	-37 ≤ h ≤ 37, -28 ≤ k ≤ 28, -39 ≤ l ≤ 39	-26 ≤ h ≤ 33, -22 ≤ k ≤ 22, -28 ≤ l ≤ 31	-26 ≤ h ≤ 33, -22 ≤ k ≤ 23, -28 ≤ l ≤ 31	-33 ≤ h ≤ 26, -22 ≤ k ≤ 23, -31 ≤ l ≤ 28
Reflections collected	3682	2286	125210	35961	36429	36842
Independent reflections	974 [R <sub>int</sub> = 0.0220, R <sub>sigma</sub> = 0.0154]	1240 [R <sub>int</sub> = 0.0298, R <sub>sigma</sub> = 0.0339]	21605 [R <sub>int</sub> = 0.0617, R <sub>sigma</sub> = 0.0404]	12633 [R <sub>int</sub> = 0.0371, R <sub>sigma</sub> = 0.0493]	12794 [R <sub>int</sub> = 0.0321, R <sub>sigma</sub> = 0.0440]	12946 [R <sub>int</sub> = 0.0337, R <sub>sigma</sub> = 0.0487]
Data/restraints/parameters	974/0/91	1240/0/109	21605/117/1254	12633/108/1237	12794/192/1271	12946/194/1271
Goodness-of-fit on F <sup>2</sup>	1.05	1.063	1.089	1.1	1.102	1.07
Final R indexes [I ≥ 2σ (I)]	R <sub>1</sub> = 0.0421, wR <sub>2</sub> = 0.1172	R <sub>1</sub> = 0.0379, wR <sub>2</sub> = 0.1031	R <sub>1</sub> = 0.0805, wR <sub>2</sub> = 0.2590	R <sub>1</sub> = 0.0785, wR <sub>2</sub> = 0.2483	R <sub>1</sub> = 0.0759, wR <sub>2</sub> = 0.2430	R <sub>1</sub> = 0.0799, wR <sub>2</sub> = 0.2479
Final R indexes [all data]	R <sub>1</sub> = 0.0451, wR <sub>2</sub> = 0.1210	R <sub>1</sub> = 0.0391, wR <sub>2</sub> = 0.1061	R <sub>1</sub> = 0.0963, wR <sub>2</sub> = 0.2741	R <sub>1</sub> = 0.0896, wR <sub>2</sub> = 0.2595	R <sub>1</sub> = 0.0873, wR <sub>2</sub> = 0.2554	R <sub>1</sub> = 0.0947, wR <sub>2</sub> = 0.2638
Largest diff. peak/hole / e Å <sup>-3</sup>	0.61/-0.61	1.52/-1.68	1.49/-0.84	1.16/-0.73	0.93/-0.61	0.77/-0.53

	[22]Bβ	[24]B	[24]C	[25]B	[25]NCS	[25]NCSe
Empirical formula	C <sub>122</sub> H <sub>70</sub> B <sub>8</sub> F <sub>36</sub> Fe <sub>10</sub> N <sub>68</sub> O <sub>10</sub>	C <sub>12</sub> H <sub>17</sub> N <sub>7</sub> O <sub>4</sub> FeF <sub>8</sub> B <sub>2</sub>	C <sub>14</sub> H <sub>18</sub> N <sub>8</sub> O <sub>11</sub> Cl <sub>2</sub> Fe	C <sub>14</sub> H <sub>20</sub> N <sub>6</sub> O <sub>3</sub> FeF <sub>8</sub> B <sub>2</sub>	C <sub>13</sub> H <sub>10</sub> N <sub>8</sub> FeS <sub>2</sub>	C <sub>24</sub> H <sub>20</sub> N <sub>14</sub> Se <sub>2</sub> Fe
Formula weight	3977.44	552.8	601.11	549.83	398.26	718.31
Temperature/K	119.99(13)	125.15	120.15	120.01(16)	120.0(3)	120.15
Crystal system	monoclinic	triclinic	triclinic	monoclinic	tetragonal	triclinic
Space group	C2/m	P-1	P-1	P2 <sub>1</sub> /n	I4 <sub>1</sub> /a	P-1
a/Å	27.1275(8)	10.0049(9)	10.4570(3)	11.1084(2)	16.1912(6)	8.1773(4)
b/Å	18.4128(5)	10.1934(11)	15.2881(4)	9.56750(10)	16.1912(6)	12.1684(6)
c/Å	18.5841(4)	11.1633(11)	15.3002(4)	21.0007(4)	25.146(3)	14.1965(8)
α/°	90	109.358(9)	86.767(2)	90	90	77.437(4)
β/°	106.889(3)	94.272(7)	77.965(3)	102.628(2)	90	83.062(4)
γ/°	90	91.322(8)	76.781(2)	90	90	89.966(4)
Volume/Å <sup>3</sup>	8882.2(4)	1069.75(18)	2328.76(11)	2177.96(6)	6592.1(8)	1368.22(12)
Z	2	2	4	4	16	2
ρ <sub>calc</sub> /mm <sup>3</sup>	1.487	1.716	1.715	1.677	1.605	1.744
μ/mm <sup>-1</sup>	7.297	6.653	7.983	6.481	9.83	7.834
2θ range for data collection	7.036 to 147.402	8.42 to 146.9°	8.34 to 147.46°	8.36 to 147.24°	6.5 to 146.78°	6.42 to 147.44°
Index ranges	-33 ≤ h ≤ 29, -13 ≤ k ≤ 21, -22 ≤ l ≤ 17	-12 ≤ h ≤ 10, -12 ≤ k ≤ 12, -13 ≤ l ≤ 11	-12 ≤ h ≤ 12, -19 ≤ k ≤ 18, -19 ≤ l ≤ 18	-13 ≤ h ≤ 12, -11 ≤ k ≤ 8, -25 ≤ l ≤ 25	-19 ≤ h ≤ 19, -14 ≤ k ≤ 19, -23 ≤ l ≤ 29	-10 ≤ h ≤ 8, -12 ≤ k ≤ 15, -17 ≤ l ≤ 17
Reflections collected	16548	8475	15126	8404	7784	10627
Independent reflections	8532 [R <sub>int</sub> = 0.0264, R <sub>sigma</sub> = 0.0364]	4018 [R <sub>int</sub> = 0.0328, R <sub>sigma</sub> = 0.0423]	15126 [R <sub>int</sub> = 0.0000, R <sub>sigma</sub> = 0.0232]	4261 [R <sub>int</sub> = 0.0210, R <sub>sigma</sub> = 0.0284]	3232 [R <sub>int</sub> = 0.0219, R <sub>sigma</sub> = 0.0263]	5148 [R <sub>int</sub> = 0.0343, R <sub>sigma</sub> = 0.0427]
Data/restraints/parameters	8532/0/631	4018/26/325	15126/32/699	4261/24/343	3232/0/218	5148/0/372
Goodness-of-fit on F <sup>2</sup>	1.249	1.029	1.087	1.048	1.076	1.031
Final R indexes [I ≥ 2σ (I)]	R <sub>1</sub> = 0.0993, wR <sub>2</sub> = 0.2807	R <sub>1</sub> = 0.0533, wR <sub>2</sub> = 0.1369	R <sub>1</sub> = 0.0575, wR <sub>2</sub> = 0.1615	R <sub>1</sub> = 0.0372, wR <sub>2</sub> = 0.0937	R <sub>1</sub> = 0.0558, wR <sub>2</sub> = 0.1506	R <sub>1</sub> = 0.0420, wR <sub>2</sub> = 0.1117
Final R indexes [all data]	R <sub>1</sub> = 0.1168, wR <sub>2</sub> = 0.3024	R <sub>1</sub> = 0.0587, wR <sub>2</sub> = 0.1421	R <sub>1</sub> = 0.0677, wR <sub>2</sub> = 0.1691	R <sub>1</sub> = 0.0420, wR <sub>2</sub> = 0.0969	R <sub>1</sub> = 0.0633, wR <sub>2</sub> = 0.1565	R <sub>1</sub> = 0.0473, wR <sub>2</sub> = 0.1171
Largest diff. peak/hole / e Å <sup>-3</sup>	1.85/-0.82	0.99/-0.61	1.25/-0.82	0.47/-0.34	0.64/-0.40	1.25/-0.68

	[26]C	[26]NCS	[26]NCSe	[27]C	[27]NCS	[27]NCS MeOH
Empirical formula	C <sub>11</sub> H <sub>10</sub> NOFeCl	C <sub>12</sub> H <sub>9</sub> N <sub>9</sub> FeS <sub>2</sub>	C <sub>12</sub> H <sub>9</sub> N <sub>9</sub> FeSe <sub>2</sub>	C <sub>10</sub> H <sub>12</sub> N <sub>6</sub> O <sub>10</sub> Cl <sub>2</sub> Fe	C <sub>22</sub> H <sub>16</sub> N <sub>14</sub> FeS <sub>2</sub>	C <sub>23</sub> H <sub>20</sub> N <sub>14</sub> OS <sub>2</sub> Fe
Formula weight	263.5	399.25	493.05	503.01	596.44	628.49
Temperature/K	125.15	120.15	110.0(2)	125.15	120.15	120.15
Crystal system	tetragonal	tetragonal	tetragonal	monoclinic	triclinic	monoclinic
Space group	P4 <sub>2</sub> /ncm	I4 <sub>1</sub> /a	I4 <sub>1</sub> /a	C2/c	P-1	P2 <sub>1</sub> /n
a/Å	26.6795(4)	16.0404(2)	16.4591(3)	20.888(2)	8.0972(4)	13.1916(3)
b/Å	26.6795(4)	16.0404(2)	16.4591(3)	9.323(2)	11.5663(6)	14.3276(3)
c/Å	21.9670(8)	25.1086(3)	25.0804(6)	19.916(2)	14.0739(6)	15.8098(4)
α/°	90	90	90	90	101.383(4)	90
β/°	90	90	90	113.519(13)	92.986(4)	110.841(2)
γ/°	90	90	90	90	91.097(4)	90
Volume/Å <sup>3</sup>	15636.0(7)	6460.30(14)	6794.3(2)	3556.3(10)	1289.81(11)	2792.61(12)
Z	67	16	16	8	2	4
ρ <sub>calc</sub> /mg/mm <sup>3</sup>	1.875	1.642	1.928	1.879	1.5357	1.4947
μ/mm <sup>-1</sup>	15.303	1.207	12.161	10.228	6.567	6.123
2θ range for data collection	8.05 to 147.038°	2.92 to 49.82°	6.42 to 147.28°	9.24 to 146.48°	7.8 to 147.64°	7.52 to 146.84°
Index ranges	-29 ≤ h ≤ 23, -33 ≤ k ≤ 25, -18 ≤ l ≤ 26	-19 ≤ h ≤ 19, -16 ≤ k ≤ 18, -28 ≤ l ≤ 25	-20 ≤ h ≤ 17, -19 ≤ k ≤ 17, -20 ≤ l ≤ 30	-25 ≤ h ≤ 25, -7 ≤ k ≤ 11, -20 ≤ l ≤ 24	-10 ≤ h ≤ 8, -14 ≤ k ≤ 14, -16 ≤ l ≤ 17	-16 ≤ h ≤ 15, -11 ≤ k ≤ 17, -17 ≤ l ≤ 19
Reflections collected	32529	13505	7393	6362	9319	11284
Independent reflections	7983 [R <sub>int</sub> = 0.0655, R <sub>sigma</sub> = 0.0539]	2629 [R <sub>int</sub> = 0.0681, R <sub>sigma</sub> = 0.0628]	3326 [R <sub>int</sub> = 0.0311, R <sub>sigma</sub> = 0.0423]	3454 [R <sub>int</sub> = 0.0387, R <sub>sigma</sub> = 0.0535]	4858 [R <sub>int</sub> = 0.0260, R <sub>sigma</sub> = 0.0354]	5480 [R <sub>int</sub> = 0.0269, R <sub>sigma</sub> = 0.0346]
Data/restraints/parameters	7983/26/351	2629/2/223	3326/2/223	3454/44/269	4858/0/352	5480/1/374
Goodness-of-fit on F <sup>2</sup>	1.598	0.904	1.012	1.045	1.017	1.027
Final R indexes [I ≥ 2σ (I)]	R <sub>1</sub> = 0.1192, wR <sub>2</sub> = 0.2903	R <sub>1</sub> = 0.0558, wR <sub>2</sub> = 0.1532	R <sub>1</sub> = 0.0462, wR <sub>2</sub> = 0.1146	R <sub>1</sub> = 0.0889, wR <sub>2</sub> = 0.2285	R <sub>1</sub> = 0.0294, wR <sub>2</sub> = 0.0730	R <sub>1</sub> = 0.0374, wR <sub>2</sub> = 0.0958
Final R indexes [all data]	R <sub>1</sub> = 0.1573, wR <sub>2</sub> = 0.3070	R <sub>1</sub> = 0.1067, wR <sub>2</sub> = 0.1753	R <sub>1</sub> = 0.0671, wR <sub>2</sub> = 0.1276	R <sub>1</sub> = 0.1139, wR <sub>2</sub> = 0.2533	R <sub>1</sub> = 0.0323, wR <sub>2</sub> = 0.0753	R <sub>1</sub> = 0.0448, wR <sub>2</sub> = 0.1008
Largest diff. peak/hole / e Å <sup>-3</sup>	2.53/-0.64	0.33/-0.41	0.84/-0.86	0.68/-0.94	0.29/-0.34	0.77/-0.57



	<b>[27]NCSe MeOH</b>	<b>[27]NCSe EtOH</b>	<b>[27]NCSe Me<sub>2</sub>CO</b>	<b>[28]B</b>
Empirical formula	C <sub>23.5</sub> H <sub>19</sub> N <sub>14</sub> O <sub>0.5</sub> Se <sub>2</sub> F <sub>e</sub>	C <sub>23</sub> H <sub>19</sub> N <sub>14</sub> O <sub>0.5</sub> Se <sub>2</sub> Fe	C <sub>23</sub> H <sub>20</sub> N <sub>14</sub> OSe <sub>2</sub> Fe	C <sub>159</sub> H <sub>176.5</sub> N <sub>74.5</sub> O <sub>20.5</sub> Fe <sub>9</sub> F <sub>28</sub> B <sub>7</sub>
Formula weight	719.27	713.29	722.27	4569.57
Temperature/K	120.15	120.15	120.0(3)	125.15
Crystal system	monoclinic	monoclinic	monoclinic	monoclinic
Space group	P2 <sub>1</sub> /n	P2 <sub>1</sub> /n	P2 <sub>1</sub> /n	P2 <sub>1</sub> /n
a/Å	12.7269(5)	12.6992(7)	12.9026(2)	17.2790(4)
b/Å	14.6440(6)	14.4699(6)	14.5475(2)	24.2218(5)
c/Å	16.1908(7)	16.2226(9)	16.1511(2)	25.7224(4)
α/°	90	90	90	90
β/°	109.447(4)	108.645(6)	109.427(2)	99.479(2)
γ/°	90	90	90	90
Volume/Å <sup>3</sup>	2845.4(2)	2824.6(3)	2858.97(8)	10618.6(4)
Z	4	4	4	2
ρ <sub>calc</sub> /mg/mm <sup>3</sup>	1.6789	1.677	1.6779	1.429
μ/mm <sup>-1</sup>	7.548	7.598	7.527	5.646
2θ range for data collection	7.7 to 147.08°	7.74 to 146.84°	7.64 to 147.42°	7.64 to 147.66°
Index ranges	-14 ≤ h ≤ 15, -10 ≤ k ≤ 17, -18 ≤ l ≤ 19	-15 ≤ h ≤ 13, -17 ≤ k ≤ 15, -19 ≤ l ≤ 19	-16 ≤ h ≤ 8, -17 ≤ k ≤ 15, -16 ≤ l ≤ 19	-10 ≤ h ≤ 20, -29 ≤ k ≤ 29, -30 ≤ l ≤ 31
Reflections collected	10897	11028	14817	47892
Independent reflections	5537 [R <sub>int</sub> = 0.0321, R <sub>sigma</sub> = 0.0432]	5540 [R <sub>int</sub> = 0.0261, R <sub>sigma</sub> = 0.0367]	5531 [R <sub>int</sub> = 0.0253, R <sub>sigma</sub> = 0.0281]	20478 [R <sub>int</sub> = 0.0406, R <sub>sigma</sub> = 0.0491]
Data/restraints/parameters	5537/18/393	5540/6/388	5531/1/383	20478/69/1417
Goodness-of-fit on F <sup>2</sup>	1.028	1.03	1.053	1.024
Final R indexes [I ≥ 2σ (I)]	R <sub>1</sub> = 0.0485, wR <sub>2</sub> = 0.1222	R <sub>1</sub> = 0.0322, wR <sub>2</sub> = 0.0727	R <sub>1</sub> = 0.0300, wR <sub>2</sub> = 0.0759	R <sub>1</sub> = 0.0665, wR <sub>2</sub> = 0.1862
Final R indexes [all data]	R <sub>1</sub> = 0.0676, wR <sub>2</sub> = 0.1368	R <sub>1</sub> = 0.0410, wR <sub>2</sub> = 0.0773	R <sub>1</sub> = 0.0341, wR <sub>2</sub> = 0.0790	R <sub>1</sub> = 0.0759, wR <sub>2</sub> = 0.1957
Largest diff. peak/hole / e Å <sup>-3</sup>	0.75/-0.89	0.36/-0.38	0.64/-0.45	1.57/-1.00

**Crystallographic Data Tables of Chapter 5**

	(31)	(34)	(35)	[30]B	[30]C	[30]NCS
Empirical formula	C <sub>9</sub> H <sub>7</sub> N <sub>7</sub>	C <sub>11</sub> H <sub>11</sub> N <sub>7</sub>	C <sub>11</sub> H <sub>11</sub> N <sub>7</sub>	C <sub>18</sub> H <sub>33.08</sub> N <sub>14</sub> O <sub>9.54</sub> Fe <sub>4</sub> F <sub>26</sub> B <sub>6</sub>	C <sub>40</sub> H <sub>38</sub> N <sub>30</sub> Cl <sub>2</sub> FeO <sub>10</sub>	C <sub>29</sub> H <sub>25</sub> N <sub>23</sub> O <sub>2</sub> FeS <sub>2</sub>
Formula weight	213.22	241.27	241.27	1380.56	1225.75	847.69
Temperature/K	140.00(10)	120.15	120.1(4)	120.15	120.3(6)	120.15
Crystal system	orthorhombic	monoclinic	monoclinic	monoclinic	triclinic	monoclinic
Space group	Pnma	P2 <sub>1</sub> /n	P2 <sub>1</sub> /c	P2 <sub>1</sub> /n	P-1	Pc
a/Å	11.5544(8)	8.4785(6)	14.0928(9)	12.2711(9)	11.1446(5)	11.3844(2)
b/Å	21.2523(13)	23.080(3)	17.7029(12)	15.4774(7)	12.4613(7)	12.6103(2)
c/Å	3.7041(2)	11.6930(19)	9.0562(5)	13.5226(10)	20.5932(9)	13.5813(2)
α/°	90	90	90	90	91.427(4)	90
β/°	90	93.907(9)	92.061(5)	116.083(9)	91.081(4)	105.154(2)
γ/°	90	90	90	90	111.370(5)	90
Volume/Å <sup>3</sup>	909.57(10)	2282.8(5)	2257.9(2)	2306.7(3)	2661.3(2)	1881.94(5)
Z	4	8	8	2	2	2
ρ <sub>calc</sub> /mg/mm <sup>3</sup>	1.557	1.404	1.419	1.988	1.53	1.496
μ/mm <sup>-1</sup>	0.89	0.773	0.782	11.459	3.933	4.798
2θ range for data collection	8.32 to 147.12°	7.66 to 147.88°	6.28 to 147.54°	8.12 to 147.32°	7.62 to 147.5°	7 to 147.56°
Index ranges	-12 ≤ h ≤ 13, -25 ≤ k ≤ 21, -4 ≤ l ≤ 3	-7 ≤ h ≤ 10, -28 ≤ k ≤ 26, -14 ≤ l ≤ 13	-17 ≤ h ≤ 12, -21 ≤ k ≤ 21, -10 ≤ l ≤ 10	-14 ≤ h ≤ 14, -14 ≤ k ≤ 18, -16 ≤ l ≤ 16	-13 ≤ h ≤ 13, -15 ≤ k ≤ 15, -22 ≤ l ≤ 25	-9 ≤ h ≤ 13, -15 ≤ k ≤ 15, -16 ≤ l ≤ 11
Reflections collected	1870	9211	9803	9221	20693	7271
Independent reflections	915 [R <sub>int</sub> = 0.0166, R <sub>sigma</sub> = 0.0214]	4484 [R <sub>int</sub> = 0.0498, R <sub>sigma</sub> = 0.0532]	4312 [R <sub>int</sub> = 0.0225, R <sub>sigma</sub> = 0.0272]	4515 [R <sub>int</sub> = 0.0361, R <sub>sigma</sub> = 0.0471]	10057 [R <sub>int</sub> = 0.0413, R <sub>sigma</sub> = 0.0487]	4466 [R <sub>int</sub> = 0.0203, R <sub>sigma</sub> = 0.0335]
Data/restraints/parameters	915/1/87	4484/3/391	4312/0/329	4515/70/365	10057/44/803	4466/6/526
Goodness-of-fit on F <sup>2</sup>	1.094	1.039	1.112	1.155	1.129	1.043
Final R indexes [I ≥ 2σ (I)]	R <sub>1</sub> = 0.0371, wR <sub>2</sub> = 0.1003	R <sub>1</sub> = 0.0599, wR <sub>2</sub> = 0.1470	R <sub>1</sub> = 0.0472, wR <sub>2</sub> = 0.1198	R <sub>1</sub> = 0.0965, wR <sub>2</sub> = 0.2072	R <sub>1</sub> = 0.0691, wR <sub>2</sub> = 0.1819	R <sub>1</sub> = 0.0316, wR <sub>2</sub> = 0.0748
Final R indexes [all data]	R <sub>1</sub> = 0.0418, wR <sub>2</sub> = 0.1049	R <sub>1</sub> = 0.0870, wR <sub>2</sub> = 0.1670	R <sub>1</sub> = 0.0529, wR <sub>2</sub> = 0.1237	R <sub>1</sub> = 0.1034, wR <sub>2</sub> = 0.2108	R <sub>1</sub> = 0.0782, wR <sub>2</sub> = 0.1877	R <sub>1</sub> = 0.0334, wR <sub>2</sub> = 0.0760
Largest diff. peak/hole / e Å <sup>-3</sup>	0.20/-0.21	0.36/-0.28	0.25/-0.22	1.05/-0.79	0.89/-0.55	0.42/-0.41

	<b>[30]Ag</b>	<b>[30]CuB</b>	<b>[31]C</b>	<b>[32]CoB</b>	<b>[34]B<math>\alpha</math></b>	<b>[34]B<math>\beta</math></b>
Empirical formula	C <sub>36</sub> H <sub>32</sub> N <sub>28</sub> O <sub>18</sub> Ag <sub>4</sub> Cl <sub>4</sub>	C <sub>22</sub> H <sub>24</sub> N <sub>16</sub> O <sub>2</sub> CuF <sub>8</sub> B <sub>2</sub>	C <sub>20</sub> H <sub>20</sub> N <sub>16</sub> O <sub>12</sub> Cl <sub>2</sub> Fe	C <sub>18</sub> N <sub>14</sub> H <sub>16</sub> CoB <sub>2</sub> F <sub>8</sub> O	C <sub>22</sub> H <sub>22</sub> N <sub>14</sub> FeF <sub>8</sub> B <sub>2</sub>	C <sub>26.6</sub> H <sub>28.9</sub> N <sub>16.3</sub> FeB <sub>2</sub> F <sub>8</sub>
Formula weight	1718.18	781.73	803.27	677	712.01	806.43
Temperature/K	120.5(10)	120.15	120.15	120.15	120.15	120.15
Crystal system	triclinic	monoclinic	triclinic	tetragonal	monoclinic	triclinic
Space group	P-1	C2/c	P-1	I4 <sub>1</sub> /a	P2 <sub>1</sub> /c	P-1
a/Å	10.4679(4)	16.9707(2)	9.8851(3)	10.7533(11)	16.4366(10)	12.2681(5)
b/Å	10.6006(5)	14.9399(2)	12.3311(4)	10.7533(11)	10.4987(7)	12.3019(6)
c/Å	12.4432(5)	12.3493(3)	13.6561(5)	21.909(2)	17.0097(10)	13.8884(4)
$\alpha$ /°	91.466(3)	90	98.813(3)	90	90	70.194(3)
$\beta$ /°	97.177(3)	93.459(2)	95.902(3)	90	94.046(5)	73.214(3)
$\gamma$ /°	103.811(4)	90	107.468(3)	90	90	64.870(4)
Volume/Å <sup>3</sup>	1328.14(10)	3125.34(9)	1549.41(9)	2533.4(4)	2927.9(3)	1759.37(12)
Z	1	4	2	4	4	2
$\rho_{\text{calc}}$ /mg/mm <sup>3</sup>	2.148	1.661	1.722	1.775	1.615	1.522
$\mu$ /mm <sup>-1</sup>	14.406	1.908	6.287	0.783	4.984	4.24
2 $\theta$ range for data collection	7.18 to 147.6°	7.9 to 147.6°	7.66 to 147.24°	6.52 to 58.82°	9.9 to 147.18°	8.08 to 147.54°
Index ranges	-12 ≤ h ≤ 12, -12 ≤ k ≤ 13, -15 ≤ l ≤ 14	-20 ≤ h ≤ 21, -17 ≤ k ≤ 12, -15 ≤ l ≤ 14	-12 ≤ h ≤ 11, -14 ≤ k ≤ 15, -15 ≤ l ≤ 16	-7 ≤ h ≤ 14, -14 ≤ k ≤ 10, -20 ≤ l ≤ 30	-20 ≤ h ≤ 20, -12 ≤ k ≤ 10, -20 ≤ l ≤ 21	-15 ≤ h ≤ 12, -15 ≤ k ≤ 12, -15 ≤ l ≤ 17
Reflections collected	10297	5800	12374	3605	12042	13268
Independent reflections	5009 [R <sub>int</sub> = 0.0389, R <sub>sigma</sub> = 0.0464]	3062 [R <sub>int</sub> = 0.0151, R <sub>sigma</sub> = 0.0202]	5820 [R <sub>int</sub> = 0.0243, R <sub>sigma</sub> = 0.0298]	1493 [R <sub>int</sub> = 0.0363, R <sub>sigma</sub> = 0.0553]	5522 [R <sub>int</sub> = 0.0445, R <sub>sigma</sub> = 0.0539]	6639 [R <sub>int</sub> = 0.0288, R <sub>sigma</sub> = 0.0377]
Data/restraints/parameters	5009/0/414	3062/5/242	5820/0/462	1493/20/122	5522/20/444	6639/34/504
Goodness-of-fit on F <sup>2</sup>	1.057	1.055	1.03	1.147	1.026	1.048
Final R indexes [I ≥ 2 $\sigma$ (I)]	R <sub>1</sub> = 0.0435, wR <sub>2</sub> = 0.1172	R <sub>1</sub> = 0.0280, wR <sub>2</sub> = 0.0717	R <sub>1</sub> = 0.0426, wR <sub>2</sub> = 0.1088	R <sub>1</sub> = 0.0837, wR <sub>2</sub> = 0.2102	R <sub>1</sub> = 0.0767, wR <sub>2</sub> = 0.2064	R <sub>1</sub> = 0.0551, wR <sub>2</sub> = 0.1480
Final R indexes [all data]	R <sub>1</sub> = 0.0471, wR <sub>2</sub> = 0.1217	R <sub>1</sub> = 0.0313, wR <sub>2</sub> = 0.0739	R <sub>1</sub> = 0.0460, wR <sub>2</sub> = 0.1122	R <sub>1</sub> = 0.1030, wR <sub>2</sub> = 0.2220	R <sub>1</sub> = 0.0962, wR <sub>2</sub> = 0.2263	R <sub>1</sub> = 0.0596, wR <sub>2</sub> = 0.1520
Largest diff. peak/hole / e Å <sup>-3</sup>	2.12/-1.16	0.32/-0.41	1.07/-0.72	1.14/-0.61	1.81/-0.36	0.85/-0.70

	[34]B $\gamma$	[34]C	[35] $\alpha$	[35] $\beta$	[35] $\beta$	[35]B $\gamma$
Empirical formula	C <sub>24</sub> H <sub>27</sub> B <sub>2</sub> F <sub>8</sub> FeN <sub>15</sub> O	C <sub>27</sub> H <sub>29.5</sub> N <sub>16.5</sub> O <sub>8</sub> Cl <sub>2</sub> F <sub>e</sub>	C <sub>22</sub> H <sub>24</sub> N <sub>14</sub> O <sub>9</sub> FeCl <sub>2</sub>	C <sub>25</sub> H <sub>31</sub> N <sub>17</sub> O <sub>14</sub> FeCl <sub>2</sub>	C <sub>25</sub> H <sub>31</sub> Cl <sub>2</sub> FeN <sub>17</sub> O <sub>14</sub>	C <sub>22.93</sub> H <sub>24.8</sub> N <sub>14.93</sub> O <sub>9.87</sub> Cl <sub>2</sub> Fe
Formula weight	771.08	839.92	755.3	920.42	920.42	794.26
Temperature/K	279.56(10)	120.15	120.15	120.00(12)	180.15	120.15
Crystal system	monoclinic	orthorhombic	monoclinic	monoclinic	monoclinic	triclinic
Space group	C2/c	Pnma	C2/c	P2	P2/c	P-1
a/Å	19.2376(11)	17.3703(7)	19.6568(9)	14.8518(6)	12.8402(5)	8.6920(2)
b/Å	9.9883(6)	40.6804(12)	9.3406(6)	10.7707(6)	10.8304(3)	18.5936(5)
c/Å	16.5179(8)	10.0680(2)	16.761(2)	23.9902(11)	15.7159(9)	30.5707(9)
$\alpha$ /°	90	90	90	90	90	78.446(2)
$\beta$ /°	92.661(5)	90	105.477(9)	103.125(5)	118.306(3)	89.894(2)
$\gamma$ /°	90	90	90	90	90	82.716(2)
Volume/Å <sup>3</sup>	3170.5(3)	7114.4(4)	2965.8(5)	3737.3(3)	1924.19(14)	4800.1(2)
Z	4	8	4	4	2	6
$\rho_{\text{calc}}$ /mg/mm <sup>3</sup>	1.615	1.568	1.692	1.636	1.589	1.649
$\mu$ /mm <sup>-1</sup>	4.686	5.422	6.424	5.346	5.191	6.017
2 $\theta$ range for data collection	9.2 to 147.38°	8.7 to 147.5°	9.34 to 147°	6.12 to 147.68°	7.82 to 147.28°	6.86 to 147.82°
Index ranges	-20 ≤ h ≤ 23, -11 ≤ k ≤ 8, -20 ≤ l ≤ 17	-14 ≤ h ≤ 16, -34 ≤ k ≤ 45, -12 ≤ l ≤ 12	-23 ≤ h ≤ 19, -11 ≤ k ≤ 9, -20 ≤ l ≤ 17	-18 ≤ h ≤ 15, -11 ≤ k ≤ 13, -27 ≤ l ≤ 29	-15 ≤ h ≤ 15, -12 ≤ k ≤ 13, -18 ≤ l ≤ 13	-9 ≤ h ≤ 10, -23 ≤ k ≤ 22, -38 ≤ l ≤ 38
Reflections collected	6380	11841	5902	16167	7999	41856
Independent reflections	3099 [R <sub>int</sub> = 0.0354, R <sub>sigma</sub> = 0.0368]	5288 [R <sub>int</sub> = 0.1135, R <sub>sigma</sub> = 0.1034]	2840 [R <sub>int</sub> = 0.0284, R <sub>sigma</sub> = 0.0335]	10788 [R <sub>int</sub> = 0.0304, R <sub>sigma</sub> = 0.0506]	3723 [R <sub>int</sub> = 0.0242, R <sub>sigma</sub> = 0.0285]	18110 [R <sub>int</sub> = 0.0372, R <sub>sigma</sub> = 0.0451]
Data/restraints/parameters	3099/0/248	5288/12/505	2840/0/220	10788/79/1069	3723/0/266	18110/54/1400
Goodness-of-fit on F <sup>2</sup>	1.035	1.068	1.029	1.042	1.055	1.02
Final R indexes [I ≥ 2 $\sigma$ (I)]	R <sub>1</sub> = 0.0488, wR <sub>2</sub> = 0.1305	R <sub>1</sub> = 0.0990, wR <sub>2</sub> = 0.2638	R <sub>1</sub> = 0.0454, wR <sub>2</sub> = 0.1234	R <sub>1</sub> = 0.0655, wR <sub>2</sub> = 0.1677	R <sub>1</sub> = 0.0607, wR <sub>2</sub> = 0.1599	R <sub>1</sub> = 0.0466, wR <sub>2</sub> = 0.1175
Final R indexes [all data]	R <sub>1</sub> = 0.0503, wR <sub>2</sub> = 0.1323	R <sub>1</sub> = 0.1074, wR <sub>2</sub> = 0.2821	R <sub>1</sub> = 0.0500, wR <sub>2</sub> = 0.1292	R <sub>1</sub> = 0.0771, wR <sub>2</sub> = 0.1807	R <sub>1</sub> = 0.0658, wR <sub>2</sub> = 0.1655	R <sub>1</sub> = 0.0570, wR <sub>2</sub> = 0.1259
Largest diff. peak/hole / e Å <sup>-3</sup>	0.81/-0.61	1.48/-1.12	0.68/-0.68	0.61/-1.45	0.89/-0.79	0.75/-0.59

**Crystallographic Data Tables of Chapter 6**

	(40)	(42)	(44)	[40]B-MeNO <sub>2</sub>	[40]B-MeNO <sub>2</sub>	[40]C-MeNO <sub>2</sub>
Empirical formula	C <sub>13</sub> H <sub>11</sub> N <sub>5</sub> OS	C <sub>19</sub> H <sub>17</sub> N <sub>5</sub> O <sub>2</sub> S	C <sub>13</sub> H <sub>12.5</sub> N <sub>6</sub> O <sub>1.25</sub>	C <sub>26</sub> H <sub>22</sub> BF <sub>4</sub> FeN <sub>10</sub> O <sub>2</sub> S <sub>2</sub>	C <sub>26</sub> H <sub>22.5</sub> B <sub>1.75</sub> F <sub>8</sub> FeN <sub>10</sub> O <sub>2</sub> S <sub>2</sub>	C <sub>26.5</sub> H <sub>23</sub> Cl <sub>1.75</sub> FeN <sub>10.5</sub> O <sub>11</sub> .5S <sub>2</sub>
Formula weight	285.33	379.43	272.79	713.33	797.93	854.56
Temperature/K	120.02(10)	120.15	120.00(14)	120.15	270	129.97(10)
Crystal system	orthorhombic	monoclinic	monoclinic	orthorhombic	orthorhombic	orthorhombic
Space group	P2 <sub>1</sub> 2 <sub>1</sub> 2 <sub>1</sub>	Cc	C2/c	Pbca	Pbca	Pbca
a/Å	7.86450(10)	25.2282(14)	25.8969(4)	10.9298(6)	41.809(3)	41.0204(9)
b/Å	10.4287(2)	4.5977(3)	5.15810(10)	15.5712(10)	11.0246(10)	10.8756(3)
c/Å	15.6259(2)	15.1996(10)	39.7013(7)	41.034(2)	15.9146(13)	15.8319(4)
α/°	90	90	90	90	90	90
β/°	90	92.079(4)	100.871(2)	90	90	90
γ/°	90	90	90	90	90	90
Volume/Å <sup>3</sup>	1281.58(3)	1761.87(19)	5208.08(16)	6983.6(7)	7335.6(10)	7063.0(3)
Z	4	4	16	8	8	8
ρ <sub>calc</sub> /mg/mm <sup>3</sup>	1.479	1.43	1.392	1.3568	1.445	1.607
μ/mm <sup>-1</sup>	2.283	1.853	0.797	5.116	5.092	6.408
2θ range for data collection	10.198 to 146.286°	7.012 to 148.358°	6.952 to 147.556°	8.62 to 148.2°	8.46 to 146.886°	8.622 to 146.692°
Index ranges	-9 ≤ h ≤ 9, -12 ≤ k ≤ 11, -19 ≤ l ≤ 17	-30 ≤ h ≤ 29, -3 ≤ k ≤ 5, -18 ≤ l ≤ 17	-30 ≤ h ≤ 31, -6 ≤ k ≤ 5, -45 ≤ l ≤ 48	-13 ≤ h ≤ 13, -19 ≤ k ≤ 18, -46 ≤ l ≤ 50	-48 ≤ h ≤ 51, -11 ≤ k ≤ 13, -19 ≤ l ≤ 14	-50 ≤ h ≤ 35, -8 ≤ k ≤ 13, -18 ≤ l ≤ 19
Reflections collected	3388	6311	10272	19190	19335	14863
Independent reflections	2293 [R <sub>int</sub> = 0.0182, R <sub>sigma</sub> = 0.0285]	2999 [R <sub>int</sub> = 0.0261, R <sub>sigma</sub> = 0.0282]	5143 [R <sub>int</sub> = 0.0174, R <sub>sigma</sub> = 0.0193]	6908 [R <sub>int</sub> = 0.0588, R <sub>sigma</sub> = 0.0648]	7209 [R <sub>int</sub> = 0.0649, R <sub>sigma</sub> = 0.0657]	6795 [R <sub>int</sub> = 0.0579, R <sub>sigma</sub> = 0.0619]
Data/restraints/parameters	2293/0/214	2999/2/246	5143/2/500	6908/0/417	7209/0/542	6795/0/529
Goodness-of-fit on F <sup>2</sup>	1.045	1.114	1.088	1.032	1.076	2.697
Final R indexes [I ≥ 2σ (I)]	R <sub>1</sub> = 0.0247, wR <sub>2</sub> = 0.0651	R <sub>1</sub> = 0.0358, wR <sub>2</sub> = 0.1043	R <sub>1</sub> = 0.0361, wR <sub>2</sub> = 0.0883	R <sub>1</sub> = 0.1056, wR <sub>2</sub> = 0.2419	R <sub>1</sub> = 0.1259, wR <sub>2</sub> = 0.3563	R <sub>1</sub> = 0.1470, wR <sub>2</sub> = 0.3841
Final R indexes [all data]	R <sub>1</sub> = 0.0250, wR <sub>2</sub> = 0.0657	R <sub>1</sub> = 0.0360, wR <sub>2</sub> = 0.1049	R <sub>1</sub> = 0.0407, wR <sub>2</sub> = 0.0911	R <sub>1</sub> = 0.1211, wR <sub>2</sub> = 0.2494	R <sub>1</sub> = 0.1634, wR <sub>2</sub> = 0.3916	R <sub>1</sub> = 0.1504, wR <sub>2</sub> = 0.3865
Largest diff. peak/hole / e Å <sup>-3</sup>	0.25/-0.17	0.34/-0.55	0.18/-0.23	1.77/-1.56	1.07/-1.19	2.95/-1.10

	<b>[40]C-MeNO<sub>2</sub></b>	<b>[40]C-Me<sub>2</sub>CO</b>	<b>[42]C</b>	<b>[42]B</b>	<b>[44]B-Me<sub>2</sub>CO <math>\alpha</math></b>	<b>[44]B-Me<sub>2</sub>CO <math>\alpha</math></b>
Empirical formula	C <sub>26</sub> H <sub>22</sub> Cl <sub>1.75</sub> FeN <sub>10</sub> O <sub>9.5</sub> S <sub>2</sub>	C <sub>32</sub> H <sub>34</sub> N <sub>10</sub> O <sub>12</sub> FeS <sub>2</sub> C I <sub>2</sub>	C <sub>38</sub> H <sub>34</sub> N <sub>10</sub> O <sub>12</sub> FeS <sub>2</sub> C I <sub>2</sub>	C <sub>38</sub> H <sub>34</sub> B <sub>2</sub> F <sub>8</sub> FeN <sub>10</sub> O <sub>4</sub> S 2	C <sub>29</sub> H <sub>30</sub> N <sub>12</sub> O <sub>3</sub> FeB <sub>2</sub> F <sub>8</sub>	C <sub>29</sub> H <sub>30</sub> B <sub>2</sub> F <sub>8</sub> FeN <sub>12</sub> O <sub>3</sub>
Formula weight	808.54	941.56	1013.62	988.34	824.12	824.12
Temperature/K	280.00(10)	120.15	121(1)	173.15	130.15	240.15
Crystal system	orthorhombic	triclinic	monoclinic	monoclinic	monoclinic	monoclinic
Space group	Pbca	P-1	C2/c	C2/c	P2 <sub>1</sub>	P2 <sub>1</sub> /c
a/Å	16.0980(5)	13.9305(6)	16.1718(4)	16.0676(7)	41.2123(19)	8.6982(2)
b/Å	42.064(2)	14.5291(5)	7.9654(3)	7.9446(3)	20.7682(6)	20.8981(4)
c/Å	11.0528(4)	22.3075(10)	32.0532(10)	32.1708(11)	51.7215(12)	21.6926(5)
$\alpha$ /°	90	82.424(3)	90	90	90	90
$\beta$ /°	90	76.960(4)	98.560(3)	99.157(3)	104.778(3)	110.687(2)
$\gamma$ /°	90	65.937(4)	90	90	90	90
Volume/Å <sup>3</sup>	7484.3(6)	4012.3(3)	4082.9(2)	4054.3(3)	42804(3)	3688.95(14)
Z	8	4	4	4	48	4
$\rho_{\text{calc}}$ /mm <sup>3</sup>	1.435	1.559	1.649	1.619	1.535	1.484
$\mu$ /mm <sup>-1</sup>	5.968	5.852	5.803	4.774	4.226	4.086
2 $\theta$ range for data collection	8.408 to 146.978°	7.46 to 148°	11.066 to 147.77°	11.144 to 147.544°	6.08 to 148.72°	6.08 to 147.88°
Index ranges	-13 ≤ h ≤ 19, -35 ≤ k ≤ 35, -9 ≤ l ≤ 13	-16 ≤ h ≤ 17, -18 ≤ k ≤ 12, -26 ≤ l ≤ 27	-19 ≤ h ≤ 17, -9 ≤ k ≤ 7, -39 ≤ l ≤ 39	-19 ≤ h ≤ 19, -9 ≤ k ≤ 9, -36 ≤ l ≤ 39	-51 ≤ h ≤ 38, -25 ≤ k ≤ 23, -62 ≤ l ≤ 63	-8 ≤ h ≤ 10, -25 ≤ k ≤ 17, -26 ≤ l ≤ 25
Reflections collected	10286	35551	7839	4529	183463	14746
Independent reflections	4484 [R <sub>int</sub> = 0.0477, R <sub>sigma</sub> = 0.0503]	15166 [R <sub>int</sub> = 0.0441, R <sub>sigma</sub> = 0.0479]	4017 [R <sub>int</sub> = 0.0331, R <sub>sigma</sub> = 0.0408]	4529 [R <sub>int</sub> = ?, R <sub>sigma</sub> = 0.0271]	134249 [R <sub>int</sub> = 0.0372, R <sub>sigma</sub> = 0.0539]	7227 [R <sub>int</sub> = 0.0205, R <sub>sigma</sub> = 0.0256]
Data/restraints/parameters	4484/0/495	15166/52/1076	4017/0/296	4529/0/297	134249/491/8304	7227/84/504
Goodness-of-fit on F <sup>2</sup>	2.056	1.034	1.209	1.189	1.048	1.082
Final R indexes [I ≥ 2 $\sigma$ (I)]	R <sub>1</sub> = 0.1189, wR <sub>2</sub> = 0.3179	R <sub>1</sub> = 0.0706, wR <sub>2</sub> = 0.1866	R <sub>1</sub> = 0.0616, wR <sub>2</sub> = 0.1616	R <sub>1</sub> = 0.0723, wR <sub>2</sub> = 0.1768	R <sub>1</sub> = 0.0841, wR <sub>2</sub> = 0.2265	R <sub>1</sub> = 0.0769, wR <sub>2</sub> = 0.2142
Final R indexes [all data]	R <sub>1</sub> = 0.1332, wR <sub>2</sub> = 0.3277	R <sub>1</sub> = 0.0782, wR <sub>2</sub> = 0.1949	R <sub>1</sub> = 0.0653, wR <sub>2</sub> = 0.1630	R <sub>1</sub> = 0.0770, wR <sub>2</sub> = 0.1790	R <sub>1</sub> = 0.1256, wR <sub>2</sub> = 0.2691	R <sub>1</sub> = 0.0891, wR <sub>2</sub> = 0.2271
Largest diff. peak/hole / e Å <sup>-3</sup>	0.72/-0.42	1.38/-0.70	1.27/-0.63	0.84/-0.53	1.28/-0.92	0.76/-0.69

	<b>[44]B-Me<sub>2</sub>CO β</b>	<b>[44]C-Me<sub>2</sub>CO</b>	<b>[44]C-Me<sub>2</sub>CO</b>	<b>[44]B-MeCN α</b>	<b>[44]B-MeCN β</b>	<b>[44]B-MeCN β</b>
Empirical formula	C <sub>30.98</sub> H <sub>33.96</sub> N <sub>12</sub> O <sub>3.6</sub> FeF <sub>8</sub> B <sub>2</sub>	C <sub>29</sub> H <sub>30</sub> Cl <sub>2</sub> FeN <sub>12</sub> O <sub>11</sub>	C <sub>29</sub> H <sub>30</sub> Cl <sub>2</sub> FeN <sub>12</sub> O <sub>11</sub>	C <sub>30</sub> H <sub>30</sub> N <sub>14</sub> O <sub>2</sub> F <sub>8</sub> B <sub>2</sub> Fe	C <sub>28</sub> H <sub>27</sub> B <sub>2</sub> F <sub>8</sub> FeN <sub>13</sub> O <sub>2</sub>	C <sub>28</sub> H <sub>27</sub> N <sub>13</sub> O <sub>2</sub> FeB <sub>2</sub> F <sub>8</sub>
Formula weight	862.45	849.4	849.4	848.15	807.09	807.1
Temperature/K	120.15	120.15	170.15	120.15	125.15	200.15
Crystal system	monoclinic	monoclinic	monoclinic	triclinic	triclinic	triclinic
Space group	P2 <sub>1</sub> /n	P2 <sub>1</sub> /c	P2 <sub>1</sub> /c	P-1	P-1	P-1
a/Å	13.2056(2)	17.1922(6)	8.6833(2)	8.0593(3)	8.2204(5)	8.2250(3)
b/Å	18.0204(3)	20.9911(8)	21.0160(4)	12.7731(7)	12.3298(10)	12.5381(6)
c/Å	16.9075(3)	22.4727(12)	21.6241(5)	18.7851(16)	17.2739(14)	17.5713(11)
α/°	90	90	90	96.071(6)	82.969(7)	83.356(5)
β/°	92.1540(10)	115.394(3)	110.459(2)	102.319(5)	79.317(6)	79.977(5)
γ/°	90	90	90	101.556(4)	85.481(6)	80.825(4)
Volume/Å <sup>3</sup>	4020.64(12)	7326.4(5)	3697.23(14)	1828.5(2)	1704.7(2)	1754.35(15)
Z	4	8	4	2	2	2
ρ <sub>calc</sub> /mg/mm <sup>3</sup>	1.425	1.54	1.526	1.54	1.5722	1.528
μ/mm <sup>-1</sup>	3.787	5.299	5.25	4.136	4.394	4.27
2θ range for data collection	7.18 to 147.54°	6.06 to 147.74°	6.06 to 147.52°	7.16 to 147.68°	7.24 to 147.18°	7.18 to 147.22°
Index ranges	-12 ≤ h ≤ 16, -20 ≤ k ≤ 22, -16 ≤ l ≤ 20	-21 ≤ h ≤ 16, -25 ≤ k ≤ 25, -27 ≤ l ≤ 27	-7 ≤ h ≤ 10, -22 ≤ k ≤ 26, -26 ≤ l ≤ 24	-6 ≤ h ≤ 9, -15 ≤ k ≤ 15, -21 ≤ l ≤ 23	-7 ≤ h ≤ 10, -15 ≤ k ≤ 14, -21 ≤ l ≤ 20	-9 ≤ h ≤ 8, -14 ≤ k ≤ 15, -21 ≤ l ≤ 21
Reflections collected	16328	32771	16029	13810	13040	13864
Independent reflections	7747 [R <sub>int</sub> = 0.0239, R <sub>sigma</sub> = 0.0305]	14397 [R <sub>int</sub> = 0.0325, R <sub>sigma</sub> = 0.0371]	7277 [R <sub>int</sub> = 0.0291, R <sub>sigma</sub> = 0.0342]	6908 [R <sub>int</sub> = 0.0239, R <sub>sigma</sub> = 0.0314]	6359 [R <sub>int</sub> = 0.0773, R <sub>sigma</sub> = 0.1020]	6607 [R <sub>int</sub> = 0.0337, R <sub>sigma</sub> = 0.0416]
Data/restraints/parameters	7747/32/528	14397/32/994	7277/60/500	6908/22/515	6359/0/490	6607/56/519
Goodness-of-fit on F <sup>2</sup>	1.041	1.035	1.045	1.052	1.061	1.089
Final R indexes [I ≥ 2σ (I)]	R <sub>1</sub> = 0.0751, wR <sub>2</sub> = 0.2208	R <sub>1</sub> = 0.0788, wR <sub>2</sub> = 0.2147	R <sub>1</sub> = 0.0765, wR <sub>2</sub> = 0.2044	R <sub>1</sub> = 0.0422, wR <sub>2</sub> = 0.1088	R <sub>1</sub> = 0.1258, wR <sub>2</sub> = 0.3803	R <sub>1</sub> = 0.1002, wR <sub>2</sub> = 0.3135
Final R indexes [all data]	R <sub>1</sub> = 0.0851, wR <sub>2</sub> = 0.2333	R <sub>1</sub> = 0.0876, wR <sub>2</sub> = 0.2230	R <sub>1</sub> = 0.0844, wR <sub>2</sub> = 0.2113	R <sub>1</sub> = 0.0442, wR <sub>2</sub> = 0.1107	R <sub>1</sub> = 0.1501, wR <sub>2</sub> = 0.3984	R <sub>1</sub> = 0.1069, wR <sub>2</sub> = 0.3175
Largest diff. peak/hole / e Å <sup>-3</sup>	1.07/-0.55	1.47/-1.13	0.76/-0.78	0.81/-0.67	3.16/-1.00	1.80/-0.56

	[44]C-MeCN	[44]C-MeCN	[44]B-MeNO <sub>2</sub>	[44]C-MeNO <sub>2</sub>	[44]B-EtCN	[44]B-EtCN
Empirical formula	C <sub>28</sub> H <sub>27</sub> N <sub>13</sub> O <sub>10</sub> FeCl <sub>2</sub>	C <sub>28</sub> H <sub>27</sub> N <sub>13</sub> O <sub>10</sub> FeCl <sub>2</sub>	C <sub>28</sub> H <sub>30</sub> N <sub>14</sub> O <sub>6</sub> FeF <sub>8</sub> B <sub>2</sub>	C <sub>28</sub> H <sub>30</sub> N <sub>14</sub> O <sub>6</sub> FeCl <sub>8</sub>	C <sub>29</sub> H <sub>29</sub> B <sub>2</sub> N <sub>13</sub> O <sub>2</sub> F <sub>8</sub> Fe	C <sub>29</sub> H <sub>29</sub> B <sub>2</sub> N <sub>13</sub> O <sub>2</sub> F <sub>8</sub> Fe
Formula weight	832.38	832.38	888.13	998.11	821.12	821.12
Temperature/K	160.15	180.15	120.15	120.15	160.15	187
Crystal system	triclinic	triclinic	orthorhombic	orthorhombic	triclinic	triclinic
Space group	P-1	P-1	Pbcn	Pbcn	P-1	P-1
a/Å	8.3202(8)	8.2792(5)	20.7377(3)	20.8034(15)	8.2658(5)	8.2464(3)
b/Å	12.3819(8)	12.5887(8)	8.8852(2)	8.8426(4)	12.3645(10)	12.5340(9)
c/Å	17.5192(9)	17.7177(13)	19.6895(3)	20.0469(15)	17.5585(11)	17.7464(9)
α/°	81.925(5)	82.782(6)	90	90	83.555(6)	83.832(5)
β/°	78.760(6)	79.633(6)	90	90	79.079(5)	79.727(4)
γ/°	85.352(6)	80.612(5)	90	90	84.390(6)	82.425(5)
Volume/Å <sup>3</sup>	1750.0(2)	1783.3(2)	3627.96(11)	3687.8(4)	1745.5(2)	1782.48(17)
Z	2	2	4	4	2	2
ρ <sub>calc</sub> /mm <sup>3</sup>	1.58	1.55	1.626	1.798	1.562	1.53
μ/mm <sup>-1</sup>	5.521	5.417	4.283	9.187	4.303	4.213
2θ range for data collection	7.22 to 147.74°	7.16 to 147.66°	8.52 to 147.6°	8.5 to 147.2°	7.218 to 147.43°	7.142 to 147.576°
Index ranges	-7 ≤ h ≤ 9, -15 ≤ k ≤ 15, -20 ≤ l ≤ 21	-8 ≤ h ≤ 9, -15 ≤ k ≤ 15, -21 ≤ l ≤ 21	-17 ≤ h ≤ 25, -9 ≤ k ≤ 7, -24 ≤ l ≤ 24	-24 ≤ h ≤ 25, -10 ≤ k ≤ 10, -23 ≤ l ≤ 24	-9 ≤ h ≤ 9, -15 ≤ k ≤ 15, -21 ≤ l ≤ 21	-9 ≤ h ≤ 9, -15 ≤ k ≤ 15, -21 ≤ l ≤ 22
Reflections collected	13173	13692	8233	9763	10076	11206
Independent reflections	6578 [R <sub>int</sub> = 0.0462, R <sub>sigma</sub> = 0.0501]	6720 [R <sub>int</sub> = 0.0280, R <sub>sigma</sub> = 0.0341]	3449 [R <sub>int</sub> = 0.0224, R <sub>sigma</sub> = 0.0264]	3626 [R <sub>int</sub> = 0.0326, R <sub>sigma</sub> = 0.0347]	10076 [R <sub>int</sub> = ?, R <sub>sigma</sub> = 0.0420]	11206 [R <sub>int</sub> = ?, R <sub>sigma</sub> = 0.0383]
Data/restraints/parameters	6578/26/484	6720/66/520	3449/1/269	3626/0/269	10076/0/500	11206/30/491
Goodness-of-fit on F <sup>2</sup>	1.096	1.084	1.022	1.031	1.059	1.083
Final R indexes [I ≥ 2σ (I)]	R <sub>1</sub> = 0.0875, wR <sub>2</sub> = 0.2520	R <sub>1</sub> = 0.0730, wR <sub>2</sub> = 0.2073	R <sub>1</sub> = 0.0343, wR <sub>2</sub> = 0.0839	R <sub>1</sub> = 0.0383, wR <sub>2</sub> = 0.0972	R <sub>1</sub> = 0.0631, wR <sub>2</sub> = 0.1595	R <sub>1</sub> = 0.0614, wR <sub>2</sub> = 0.1619
Final R indexes [all data]	R <sub>1</sub> = 0.0963, wR <sub>2</sub> = 0.2599	R <sub>1</sub> = 0.0785, wR <sub>2</sub> = 0.2107	R <sub>1</sub> = 0.0408, wR <sub>2</sub> = 0.0876	R <sub>1</sub> = 0.0465, wR <sub>2</sub> = 0.1036	R <sub>1</sub> = 0.0836, wR <sub>2</sub> = 0.1746	R <sub>1</sub> = 0.0783, wR <sub>2</sub> = 0.1724
Largest diff. peak/hole / e Å <sup>-3</sup>	0.97/-0.81	0.88/-0.55	0.35/-0.31	0.34/-0.44	0.57/-0.49	0.60/-0.48



	[44]B-EtCN	[44]C-EtCN	[44]C-EtCN	[44]C-EtCN	[44]B+C-EtCN	[44]B+C-EtCN
Empirical formula	C <sub>29</sub> H <sub>29</sub> B <sub>2</sub> N <sub>13</sub> O <sub>2</sub> F <sub>8</sub> Fe	C <sub>29</sub> H <sub>29</sub> N <sub>13</sub> O <sub>10</sub> Cl <sub>2</sub> Fe	C <sub>29</sub> H <sub>29</sub> N <sub>13</sub> O <sub>10</sub> Cl <sub>2</sub> Fe	C <sub>29</sub> H <sub>29</sub> N <sub>13</sub> O <sub>10</sub> Cl <sub>2</sub> Fe	C <sub>29</sub> H <sub>29</sub> N <sub>13</sub> O <sub>6.4</sub> Cl <sub>1.1</sub> FeB <sub>0.9</sub> F <sub>3.6</sub>	C <sub>29</sub> H <sub>29</sub> N <sub>13</sub> O <sub>6.4</sub> Cl <sub>1.1</sub> FeB <sub>0.9</sub> F <sub>3.6</sub>
Formula weight	821.12	846.4	846.4	846.4	835.03	835.02
Temperature/K	200	120.15	170.15	240.15	125.15	180.15
Crystal system	triclinic	triclinic	triclinic	triclinic	triclinic	triclinic
Space group	P-1	P-1	P-1	P-1	P-1	P-1
a/Å	8.2454(3)	8.3410(3)	8.3390(2)	8.3297(4)	8.3093(4)	8.3169(4)
b/Å	12.6105(6)	12.3849(4)	20.3671(5)	12.6635(8)	12.3574(6)	20.4484(7)
c/Å	17.8210(9)	17.6860(5)	22.8144(7)	18.0861(10)	17.5746(6)	22.6673(8)
α/°	84.191(4)	82.187(3)	108.029(3)	83.662(5)	82.690(3)	107.978(3)
β/°	80.215(4)	78.664(3)	98.689(2)	80.003(4)	78.693(4)	99.002(3)
γ/°	81.291(4)	85.140(3)	95.458(2)	80.933(5)	85.091(4)	95.195(3)
Volume/Å <sup>3</sup>	1799.57(14)	1771.57(10)	3600.56(18)	1848.56(18)	1751.76(13)	3581.8(3)
Z	2	2	4	2	2	4
ρ <sub>calc</sub> /mg/mm <sup>3</sup>	1.515	1.587	1.561	1.521	1.583	1.5483
μ/mm <sup>-1</sup>	4.173	5.464	5.376	5.236	4.968	4.859
2θ range for data collection	7.112 to 147.77°	7.218 to 147.172°	7.152 to 147.742°	7.094 to 147.486°	7.22 to 147.32°	7.16 to 147.06°
Index ranges	-9 ≤ h ≤ 9, -15 ≤ k ≤ 15, -22 ≤ l ≤ 22	-10 ≤ h ≤ 8, -15 ≤ k ≤ 15, -17 ≤ l ≤ 22	-10 ≤ h ≤ 10, -25 ≤ k ≤ 25, -22 ≤ l ≤ 28	-9 ≤ h ≤ 9, -15 ≤ k ≤ 15, -22 ≤ l ≤ 22	-10 ≤ h ≤ 9, -14 ≤ k ≤ 11, -21 ≤ l ≤ 21	-6 ≤ h ≤ 9, -24 ≤ k ≤ 25, -28 ≤ l ≤ 25
Reflections collected	11551	13532	19056	11656	13546	28990
Independent reflections	11551 [R <sub>int</sub> = ?, R <sub>sigma</sub> = 0.0348]	6672 [R <sub>int</sub> = 0.0310, R <sub>sigma</sub> = 0.0368]	19056 [R <sub>int</sub> = ?, R <sub>sigma</sub> = 0.0423]	11656 [R <sub>int</sub> = ?, R <sub>sigma</sub> = 0.0356]	6565 [R <sub>int</sub> = 0.0449, R <sub>sigma</sub> = 0.0579]	13444 [R <sub>int</sub> = 0.0363, R <sub>sigma</sub> = 0.0458]
Data/restraints/parameters	11551/30/491	6672/0/498	19056/10/993	11656/60/506	6565/48/490	13444/90/972
Goodness-of-fit on F <sup>2</sup>	1.064	1.088	1.039	1.086	1.029	1.306
Final R indexes [I ≥ 2σ (I)]	R <sub>1</sub> = 0.0573, wR <sub>2</sub> = 0.1547	R <sub>1</sub> = 0.0737, wR <sub>2</sub> = 0.2422	R <sub>1</sub> = 0.0749, wR <sub>2</sub> = 0.1990	R <sub>1</sub> = 0.0753, wR <sub>2</sub> = 0.2022	R <sub>1</sub> = 0.0497, wR <sub>2</sub> = 0.1171	R <sub>1</sub> = 0.0847, wR <sub>2</sub> = 0.2241
Final R indexes [all data]	R <sub>1</sub> = 0.0700, wR <sub>2</sub> = 0.1620	R <sub>1</sub> = 0.0770, wR <sub>2</sub> = 0.2443	R <sub>1</sub> = 0.0960, wR <sub>2</sub> = 0.2156	R <sub>1</sub> = 0.0913, wR <sub>2</sub> = 0.2146	R <sub>1</sub> = 0.0627, wR <sub>2</sub> = 0.1250	R <sub>1</sub> = 0.1044, wR <sub>2</sub> = 0.2404
Largest diff. peak/hole / e Å <sup>-3</sup>	0.72/-0.55	1.19/-0.57	0.75/-0.58	0.49/-0.90	0.47/-0.83	2.83/-3.35

	<b>[44]B+C-EtCN</b>	<b>[44]B-MeOH</b>	<b>[44]C-EtCN+H<sub>2</sub>O</b>	<b>[44]C-EtOH</b>	<b>[44]C-MeOH+DCE</b>
Empirical formula	C <sub>29</sub> H <sub>29</sub> N <sub>13</sub> O <sub>6.4</sub> Cl <sub>1.1</sub> Fe B <sub>0.9</sub> F <sub>3.6</sub>	C <sub>27</sub> H <sub>28</sub> N <sub>12</sub> O <sub>3</sub> ClFeF <sub>8</sub> B <sub>2</sub>	C <sub>27.5</sub> H <sub>28.5</sub> Cl <sub>2</sub> FeN <sub>12.5</sub> O <sub>11</sub>	C <sub>27</sub> H <sub>27</sub> N <sub>12</sub> O <sub>10.5</sub> FeCl 2	C <sub>27.9</sub> H <sub>30</sub> Cl <sub>2.8</sub> FeN <sub>12</sub> O <sub>11.1</sub>
Formula weight	835.03	798.08	836.88	814.35	866.15
Temperature/K	250.15	120.15	120.15	120.00(12)	120.15
Crystal system	triclinic	monoclinic	monoclinic	triclinic	monoclinic
Space group	P-1	P2 <sub>1</sub> /n	C2/c	P-1	C2/c
a/Å	8.2960(2)	8.2085(2)	42.8996(13)	8.2050(3)	43.0473(15)
b/Å	12.6295(5)	38.9652(11)	8.1211(2)	10.9417(5)	8.1566(3)
c/Å	18.0010(7)	10.8625(3)	20.9286(4)	20.0059(7)	21.1773(6)
α/°	84.076(3)	90	90	79.136(3)	90
β/°	80.160(3)	105.691(3)	106.489(2)	78.929(3)	107.679(3)
γ/°	81.045(3)	90	90	75.532(4)	90
Volume/Å <sup>3</sup>	1829.95(11)	3344.85(16)	6991.5(3)	1687.99(12)	7084.6(4)
Z	2	4	8	2	8
ρ <sub>calc</sub> /mg/mm <sup>3</sup>	1.515	1.5847	1.59	1.602	1.624
μ/mm <sup>-1</sup>	4.756	4.484	5.547	5.71	6.036
2θ range for data collection	7.1 to 147.4°	9.08 to 147.36°	8.598 to 147.372°	8.438 to 147.53°	8.624 to 147.502°
Index ranges	-6 ≤ h ≤ 9, -15 ≤ k ≤ 15, -22 ≤ l ≤ 21	-9 ≤ h ≤ 10, -47 ≤ k ≤ 40, -13 ≤ l ≤ 12	-44 ≤ h ≤ 51, -8 ≤ k ≤ 9, -23 ≤ l ≤ 26	-8 ≤ h ≤ 10, -9 ≤ k ≤ 13, -24 ≤ l ≤ 24	-47 ≤ h ≤ 52, -9 ≤ k ≤ 7, -26 ≤ l ≤ 25
Reflections collected	14861	12629	14488	13392	13905
Independent reflections	6892 [R <sub>int</sub> = 0.0294, R <sub>sigma</sub> = 0.0373]	6289 [R <sub>int</sub> = 0.0319, R <sub>sigma</sub> = 0.0401]	6794 [R <sub>int</sub> = 0.0213, R <sub>sigma</sub> = 0.0274]	6407 [R <sub>int</sub> = 0.0361, R <sub>sigma</sub> = 0.0458]	6944 [R <sub>int</sub> = 0.0169, R <sub>sigma</sub> = 0.0211]
Data/restraints/parameters	6892/65/502	6289/2/484	6794/10/496	6407/20/496	6944/7/508
Goodness-of-fit on F <sup>2</sup>	1.079	1.136	1.028	1.025	0.682
Final R indexes [I ≥ 2σ (I)]	R <sub>1</sub> = 0.0681, wR <sub>2</sub> = 0.1904	R <sub>1</sub> = 0.0614, wR <sub>2</sub> = 0.1400	R <sub>1</sub> = 0.0347, wR <sub>2</sub> = 0.0892	R <sub>1</sub> = 0.0472, wR <sub>2</sub> = 0.1185	R <sub>1</sub> = 0.0532, wR <sub>2</sub> = 0.1700
Final R indexes [all data]	R <sub>1</sub> = 0.0762, wR <sub>2</sub> = 0.1982	R <sub>1</sub> = 0.0645, wR <sub>2</sub> = 0.1418	R <sub>1</sub> = 0.0398, wR <sub>2</sub> = 0.0928	R <sub>1</sub> = 0.0566, wR <sub>2</sub> = 0.1268	R <sub>1</sub> = 0.0551, wR <sub>2</sub> = 0.1740
Largest diff. peak/hole / e Å <sup>-3</sup>	0.67/-1.17	0.66/-0.50	0.48/-0.41	0.72/-0.46	1.20/-1.10

**Crystallographic Data Tables of Chapter 7**

	(49)	(50)	(55)	(60)	(62)	(69) DMSO
Empirical formula	C <sub>12</sub> H <sub>11</sub> N <sub>5</sub> O	C <sub>20</sub> H <sub>23</sub> N <sub>5</sub> O <sub>2</sub> S <sub>2</sub>	C <sub>14</sub> H <sub>15</sub> N <sub>7</sub> O	C <sub>22</sub> H <sub>26</sub> N <sub>6</sub> O <sub>3</sub> S <sub>2</sub>	C <sub>22</sub> H <sub>25</sub> N <sub>5</sub> O <sub>4</sub> S <sub>2</sub>	C <sub>14</sub> H <sub>14</sub> Br <sub>2</sub> N <sub>5</sub> O <sub>3</sub> S
Formula weight	241.26	429.55	297.33	486.61	487.59	174.2
Temperature/K	120.01(10)	120.00(16)	119.99(19)	120.15	150.15	119.95(14)
Crystal system	monoclinic	monoclinic	triclinic	orthorhombic	monoclinic	monoclinic
Space group	P2 <sub>1</sub> /c	C2	P-1	P2 <sub>1</sub> 2 <sub>1</sub> 2 <sub>1</sub>	P2 <sub>1</sub>	P2 <sub>1</sub> /c
a/Å	9.9484(2)	38.6148(7)	7.1406(5)	5.08130(10)	7.1017(1)	11.4282(6)
b/Å	15.9446(8)	5.29280(10)	11.9787(7)	12.2490(4)	17.0738(1)	7.9523(4)
c/Å	7.0988(4)	20.9507(3)	16.6616(9)	36.8728(9)	18.7947(2)	39.3724(15)
α/°	90	90	96.541(5)	90	90	90
β/°	91.643(3)	105.394(2)	99.130(5)	90	91.382(1)	91.722(4)
γ/°	90	90	93.031(6)	90	90	90
Volume/Å <sup>3</sup>	1125.57(9)	4128.29(12)	1394.27(15)	2294.99(10)	2278.25(4)	3576.6(3)
Z	4	8	4	4	4	8
ρ <sub>calc</sub> /mg/mm <sup>3</sup>	1.424	1.382	1.416	1.408	1.422	1.294
μ/mm <sup>-1</sup>	0.801	2.563	0.799	2.42	0.274	0.798
2θ range for data collection	10.48 to 147.54°	7.26 to 147.04°	7.44 to 147.64°	7.6 to 146.66°	2.1 to 52.1°	7.74 to 146.762°
Index ranges	-12 ≤ h ≤ 11, -19 ≤ k ≤ 18, -8 ≤ l ≤ 5	-40 ≤ h ≤ 48, -5 ≤ k ≤ 6, -24 ≤ l ≤ 26	-8 ≤ h ≤ 6, -14 ≤ k ≤ 14, -20 ≤ l ≤ 20	-4 ≤ h ≤ 6, -15 ≤ k ≤ 14, -44 ≤ l ≤ 45	-9 ≤ h ≤ 8, -21 ≤ k ≤ 21, -23 ≤ l ≤ 23	-10 ≤ h ≤ 14, -9 ≤ k ≤ 9, -48 ≤ l ≤ 48
Reflections collected	4282	8327	10490	5883	24658	13380
Independent reflections	2182 [R <sub>int</sub> = 0.0142, R <sub>sigma</sub> = 0.0171]	5960 [R <sub>int</sub> = 0.0229, R <sub>sigma</sub> = 0.0366]	5228 [R <sub>int</sub> = 0.0217, R <sub>sigma</sub> = 0.0290]	3894 [R <sub>int</sub> = 0.0313, R <sub>sigma</sub> = 0.0446]	9365 [R <sub>int</sub> = 0.0678, R <sub>sigma</sub> = 0.0859]	6994 [R <sub>int</sub> = 0.0525, R <sub>sigma</sub> = 0.0599]
Data/restraints/parameters	2182/0/196	5960/13/526	5228/0/487	3894/0/298	9365/98/617	6994/0/459
Goodness-of-fit on F <sup>2</sup>	1.058	1.018	1.018	1.072	1.082	3.493
Final R indexes [I ≥ 2σ (I)]	R <sub>1</sub> = 0.0361, wR <sub>2</sub> = 0.0981	R <sub>1</sub> = 0.0419, wR <sub>2</sub> = 0.1067	R <sub>1</sub> = 0.0340, wR <sub>2</sub> = 0.0845	R <sub>1</sub> = 0.0371, wR <sub>2</sub> = 0.0862	R <sub>1</sub> = 0.0792, wR <sub>2</sub> = 0.2243	R <sub>1</sub> = 0.1828, wR <sub>2</sub> = 0.4649
Final R indexes [all data]	R <sub>1</sub> = 0.0387, wR <sub>2</sub> = 0.1004	R <sub>1</sub> = 0.0452, wR <sub>2</sub> = 0.1097	R <sub>1</sub> = 0.0437, wR <sub>2</sub> = 0.0908	R <sub>1</sub> = 0.0417, wR <sub>2</sub> = 0.0892	R <sub>1</sub> = 0.0933, wR <sub>2</sub> = 0.2341	R <sub>1</sub> = 0.1861, wR <sub>2</sub> = 0.4658
Largest diff. peak/hole / e Å <sup>-3</sup>	0.27/-0.26	1.19/-0.65	0.26/-0.21	0.35/-0.28	0.72/-0.50	3.57/-2.51

	(70)	[62]C	[67]C $\alpha$	[67]C $\alpha$	[67]C $\alpha$	[67]C $\beta$
Empirical formula	C <sub>18</sub> H <sub>20</sub> N <sub>6</sub> O <sub>3</sub>	C <sub>40</sub> H <sub>40.5</sub> Cl <sub>2</sub> FeN <sub>11</sub> O <sub>13</sub>	C <sub>30</sub> H <sub>27</sub> Cl <sub>2</sub> FeN <sub>13</sub> O <sub>14</sub>	C <sub>30</sub> H <sub>27</sub> Cl <sub>2</sub> FeN <sub>13</sub> O <sub>14</sub>	C <sub>30</sub> H <sub>27</sub> Cl <sub>2</sub> FeN <sub>13</sub> O <sub>14</sub>	C <sub>30</sub> H <sub>27.5</sub> Cl <sub>2</sub> FeN <sub>13</sub> O <sub>14</sub>
Formula weight	368.4	1010.08	920.39	920.39	917.37	920.9
Temperature/K	150.01(10)	119.99(18)	124.97(11)	280.00(10)	340.05(11)	120
Crystal system	orthorhombic	monoclinic	monoclinic	monoclinic	monoclinic	triclinic
Space group	Pbca	P2 <sub>1</sub> /c	P2 <sub>1</sub> /n	P2 <sub>1</sub> /n	P2 <sub>1</sub> /n	P-1
a/Å	12.2009(2)	20.163(8)	13.71894(13)	13.7263(2)	13.8747(4)	8.2783(3)
b/Å	10.0221(2)	15.719(2)	16.13558(15)	16.3821(3)	16.2714(5)	13.1029(5)
c/Å	30.9963(5)	20.296(4)	16.61356(15)	16.8383(3)	17.2015(5)	18.9841(8)
$\alpha$ /°	90	90	90	90	90	92.414(3)
$\beta$ /°	90	118.46(3)	93.5129(8)	93.7794(13)	94.581(3)	100.938(3)
$\gamma$ /°	90	90	90	90	90	93.223(3)
Volume/Å <sup>3</sup>	3790.19(12)	5655(3)	3670.72(6)	3778.15(10)	3871.0(2)	2015.68(14)
Z	8	4	4	4	4	2
$\rho_{\text{calc}}$ /mg/mm <sup>3</sup>	1.291	1.186	1.665	1.618	1.574	1.517
$\mu$ /mm <sup>-1</sup>	0.756	3.534	5.416	5.262	5.135	4.931
2 $\theta$ range for data collection	9.22 to 146.9°	7.496 to 147.268	7.644 to 146.996	7.536 to 147.182	7.49 to 147.396	6.766 to 146.974
Index ranges	-14 ≤ h ≤ 15, -12 ≤ k ≤ 9, -26 ≤ l ≤ 38	-24 ≤ h ≤ 21, -18 ≤ k ≤ 13, -24 ≤ l ≤ 24	-15 ≤ h ≤ 12, -19 ≤ k ≤ 18, -19 ≤ l ≤ 20	-15 ≤ h ≤ 13, -20 ≤ k ≤ 18, -18 ≤ l ≤ 20	-13 ≤ h ≤ 16, -20 ≤ k ≤ 18, -21 ≤ l ≤ 19	-9 ≤ h ≤ 10, -13 ≤ k ≤ 16, -19 ≤ l ≤ 23
Reflections collected	9262	16207	17103	17410	16656	15782
Independent reflections	3726 [R <sub>int</sub> = 0.0209, R <sub>sigma</sub> = 0.0214]	9322 [R <sub>int</sub> = 0.0548, R <sub>sigma</sub> = 0.0991]	7058 [R <sub>int</sub> = 0.0190, R <sub>sigma</sub> = 0.0211]	7279 [R <sub>int</sub> = 0.0286, R <sub>sigma</sub> = 0.0305]	7406 [R <sub>int</sub> = 0.0439, R <sub>sigma</sub> = 0.0571]	7516 [R <sub>int</sub> = 0.0298, R <sub>sigma</sub> = 0.0359]
Data/restraints/parameters	3726/0/304	9322/3/624	7058/0/544	7279/0/551	7406/0/575	7516/0/871
Goodness-of-fit on F <sup>2</sup>	1.055	1.358	1.063	1.053	1.035	1.376
Final R indexes [I ≥ 2 $\sigma$ (I)]	R <sub>1</sub> = 0.0397, wR <sub>2</sub> = 0.1055	R <sub>1</sub> = 0.1599, wR <sub>2</sub> = 0.3583	R <sub>1</sub> = 0.0329, wR <sub>2</sub> = 0.0919	R <sub>1</sub> = 0.0806, wR <sub>2</sub> = 0.2374	R <sub>1</sub> = 0.0778, wR <sub>2</sub> = 0.2228	R <sub>1</sub> = 0.1096, wR <sub>2</sub> = 0.3095
Final R indexes [all data]	R <sub>1</sub> = 0.0464, wR <sub>2</sub> = 0.1112	R <sub>1</sub> = 0.3098, wR <sub>2</sub> = 0.4180	R <sub>1</sub> = 0.0339, wR <sub>2</sub> = 0.0927	R <sub>1</sub> = 0.0880, wR <sub>2</sub> = 0.2487	R <sub>1</sub> = 0.1059, wR <sub>2</sub> = 0.2628	R <sub>1</sub> = 0.1235, wR <sub>2</sub> = 0.3283
Largest diff. peak/hole / e Å <sup>-3</sup>	0.24/-0.18	0.60/-0.39	0.93/-0.53	1.53/-1.36	1.08/-0.46	2.04/-0.71

	[68]C	[68]C	[59]C
Empirical formula	C <sub>34</sub> H <sub>34</sub> Cl <sub>2</sub> FeN <sub>14</sub> O <sub>14</sub>	C <sub>34</sub> H <sub>27.67</sub> Cl <sub>2</sub> FeN <sub>14</sub> O <sub>14</sub>	C <sub>29</sub> H <sub>32</sub> Cl <sub>2</sub> FeN <sub>12</sub> O <sub>13.5</sub>
Formula weight	989.5	983.12	892.42
Temperature/K	119.99(17)	290	120.15
Crystal system	triclinic	triclinic	monoclinic
Space group	P-1	P-1	P2 <sub>1</sub> /n
a/Å	13.6232(3)	13.6682(3)	15.9169(3)
b/Å	17.8150(3)	17.9378(5)	14.3134(3)
c/Å	26.3257(4)	26.8890(8)	16.0417(3)
α/°	78.8771(15)	78.756(3)	90
β/°	83.7423(15)	84.199(2)	95.616(2)
γ/°	78.8440(16)	79.817(2)	90
Volume/Å <sup>3</sup>	6133.6(2)	6349.0(3)	3637.16(12)
Z	6	6	4
ρ <sub>calc</sub> /mg/mm <sup>3</sup>	1.607	1.543	1.63
μ/mm <sup>-1</sup>	4.914	4.747	5.421
2θ range for data collection	6.864 to 147.112	6.72 to 147.738	7.46 to 147.38°
Index ranges	-15 ≤ h ≤ 16, -22 ≤ k ≤ 21, -32 ≤ l ≤ 22	-11 ≤ h ≤ 15, -22 ≤ k ≤ 22, -31 ≤ l ≤ 33	-17 ≤ h ≤ 19, -17 ≤ k ≤ 15, -17 ≤ l ≤ 19
Reflections collected	52042	54085	16386
Independent reflections	23091 [R <sub>int</sub> = 0.0259, R <sub>sigma</sub> = 0.0328]	23886 [R <sub>int</sub> = 0.0525, R <sub>sigma</sub> = 0.0657]	7088 [R <sub>int</sub> = 0.0318, R <sub>sigma</sub> = 0.0378]
Data/restraints/parameters	23091/40/1763	23886/69/1731	7088/0/532
Goodness-of-fit on F <sup>2</sup>	1.039	1.019	1.035
Final R indexes [I ≥ 2σ (I)]	R <sub>1</sub> = 0.0617, wR <sub>2</sub> = 0.1624	R <sub>1</sub> = 0.0935, wR <sub>2</sub> = 0.2622	R <sub>1</sub> = 0.0509, wR <sub>2</sub> = 0.1378
Final R indexes [all data]	R <sub>1</sub> = 0.0696, wR <sub>2</sub> = 0.1690	R <sub>1</sub> = 0.1406, wR <sub>2</sub> = 0.3159	R <sub>1</sub> = 0.0589, wR <sub>2</sub> = 0.1450
Largest diff. peak/hole / e Å <sup>-3</sup>	1.74/-1.02	0.75/-0.69	0.93/-0.51