Appendices – X-ray crystallographic data

Crystal data for [(THF)Li(TMP){PhOSi(CH3)2CH2}ZntBu] (**13**)

Empirical formula C26 H48 Li N O2 Si Zn

Formula weight 507.05

Temperature 123(2) K

Wavelength 0.71073 Å

Crystal system Triclinic

Space group P-1

Unit cell dimensions a = 11.2813(5) Å = 79.264(4)°.

b = 11.3700(6) Å = 79.106(4)°.

c = 13.0987(6) Å  = 61.575(5)°.

Volume 1441.91(12) Å3

Z 2

Density (calculated) 1.168 Mg/m3

Absorption coefficient 0.913 mm-1

F(000) 548

Crystal size 0.28 x 0.22 x 0.14 mm3

Theta range for data collection 2.73 to 30.61°.

Index ranges -16<=h<=15, -15<=k<=16, -18<=l<=18

Reflections collected 20418

Independent reflections 7908 [R(int) = 0.0326]

Completeness to theta = 27.00° 98.8 %

Absorption correction Semi-empirical from equivalents

Max. and min. transmission 1.00000 and 0.93421

Refinement method Full-matrix least-squares on F2

Data / restraints / parameters 7908 / 0 / 324

Goodness-of-fit on F2 0.974

Final R indices [I>2sigma(I)] R1 = 0.0319, wR2 = 0.0721

R indices (all data) R1 = 0.0478, wR2 = 0.0752

Largest diff. peak and hole 0.467 and -0.360 e.Å-3

Crystal data for [(TMEDA)Na(TMP){PhOSi(CH3)2CH2}ZntBu] (**14**)

Empirical formula C28 H56 N3 Na O Si Zn

Formula weight 567.21

Temperature 123(2) K

Wavelength 0.71073 Å

Crystal system Orthorhombic

Space group Pna21

Unit cell dimensions a = 22.0991(9) Å = 90°.

b = 10.3628(4) Å = 90°.

c = 14.3036(7) Å  = 90°.

Volume 3275.6(2) Å3

Z 4

Density (calculated) 1.150 Mg/m3

Absorption coefficient 0.822 mm-1

F(000) 1232

Crystal size 0.2 x 0.12 x 0.10 mm3

Theta range for data collection 2.43 to 27.49°.

Index ranges -26<=h<=28, -13<=k<=10, -18<=l<=12

Reflections collected 16094

Independent reflections 6089 [R(int) = 0.0648]

Completeness to theta = 26.00° 99.9 %

Absorption correction None

Refinement method Full-matrix least-squares on F2

Data / restraints / parameters 6089 / 1 / 330

Goodness-of-fit on F2 0.870

Final R indices [I>2sigma(I)] R1 = 0.0471, wR2 = 0.0910

R indices (all data) R1 = 0.0849, wR2 = 0.0974

Absolute structure parameter 0.037(16)

Largest diff. peak and hole 1.226 and -0.458 e.Å-3

Crystal data for [(PhOSiMe3)Li(TMP){PhOSi(CH3)2CH2}ZntBu] (**16**)

Empirical formula C31 H54 Li N O2 Si2 Zn

Formula weight 601.24

Temperature 123(2) K

Wavelength 0.71073 Å

Crystal system Monoclinic

Space group P2(1)/c

Unit cell dimensions a = 12.4930(3) Å = 90°.

b = 12.8910(3) Å = 92.494(2)°.

c = 20.8841(5) Å  = 90°.

Volume 3360.14(14) Å3

Z 4

Density (calculated) 1.189 Mg/m3

Absorption coefficient 0.828 mm-1

F(000) 1296

Crystal size 0.2 x 0.2 x 0.1 mm3

Theta range for data collection 2.49 to 30.55°.

Index ranges -17<=h<=17, -17<=k<=18, -28<=l<=29

Reflections collected 55721

Independent reflections 9627 [R(int) = 0.0426]

Completeness to theta = 27.00° 99.9 %

Absorption correction Semi-empirical from equivalents

Max. and min. transmission 1.00000 and 0.95347

Refinement method Full-matrix least-squares on F2

Data / restraints / parameters 9627 / 0 / 363

Goodness-of-fit on F2 0.997

Final R indices [I>2sigma(I)] R1 = 0.0352, wR2 = 0.0685

R indices (all data) R1 = 0.0635, wR2 = 0.0742

Largest diff. peak and hole 0.489 and -0.299 e.Å-3

Crystal data for [(TMEDA)Na(μ-TMP)Zn{OC(Ph)(tBu)(*η*5-C5H3)Fe(*η*5-C5H5)}] (18)

Empirical formula C36 H56 Fe N3 Na O Zn

Formula weight 691.05

Temperature 123(2) K

Wavelength 0.71073 Å

Crystal system Monoclinic

Space group P 1 21/c 1

Unit cell dimensions a = 16.5928(8) Å = 90°.

b = 11.8192(4) Å = 111.772(5)°.

c = 19.0005(8) Å  = 90°.

Volume 3460.5(3) Å3

Z 4

Density (calculated) 1.326 Mg/m3

Absorption coefficient 1.157 mm-1

F(000) 1472

Crystal size 0.1 x 0.1 x 0.1 mm3

Theta range for data collection 2.64 to 28.00°.

Index ranges -21<=h<=21, -15<=k<=15, -23<=l<=25

Reflections collected 20669

Independent reflections 8165 [R(int) = 0.0648]

Completeness to theta = 28.00° 97.9 %

Absorption correction Semi-empirical from equivalents

Max. and min. transmission 1.00000 and 0.89645

Refinement method Full-matrix least-squares on F2

Data / restraints / parameters 8165 / 14 / 395

Goodness-of-fit on F2 0.787

Final R indices [I>2sigma(I)] R1 = 0.0464, wR2 = 0.0690

R indices (all data) R1 = 0.1137, wR2 = 0.0771

Largest diff. peak and hole 0.818 and -0.479 e.Å-3

Crystal data for [(TMEDA)Na(μ-TMP)Zn{OC(Ph)(tBu)(*η*5-C5H3)Fe(*η*5-C5H5)}.C7H8] (18B)

Empirical formula C43 H64 Fe N3 Na O Zn

Formula weight 783.18

Temperature 123(2) K

Wavelength 0.71073 Å

Crystal system Triclinic

Space group P -1

Unit cell dimensions a = 11.0692(8) Å = 102.227(5)°.

b = 14.4319(10) Å = 111.723(5)°.

c = 14.6345(7) Å  = 100.148(6)°.

Volume 2037.4(2) Å3

Z 2

Density (calculated) 1.277 Mg/m3

Absorption coefficient 0.991 mm-1

F(000) 836

Crystal size 0.1 x 0.1 x 0.1 mm3

Theta range for data collection 2.87 to 27.00°.

Index ranges -14<=h<=13, -18<=k<=18, -18<=l<=18

Reflections collected 20067

Independent reflections 8608 [R(int) = 0.0562]

Completeness to theta = 26.00° 98.8 %

Absorption correction Semi-empirical from equivalents

Max. and min. transmission 1.00000 and 0.48981

Refinement method Full-matrix least-squares on F2

Data / restraints / parameters 8608 / 14 / 486

Goodness-of-fit on F2 1.027

Final R indices [I>2sigma(I)] R1 = 0.0430, wR2 = 0.0948

R indices (all data) R1 = 0.0641, wR2 = 0.1091

Largest diff. peak and hole 0.748 and -0.814 e.Å-3

[PhC(OH)(*t*Bu)(*η*5-C5H3I)Fe(*η*5-C5H5)] (19) and [PhC(OH)(*t*Bu)(*η*5-C5H3I)Fe(*η*5-C5H4I)] (20)

Empirical formula C21 H22.95 Fe I1.05 O

Formula weight 480.44

Temperature 123(2) K

Wavelength 0.71073 Å

Crystal system qmonoclinic’

Space group ‘P21/c’

Unit cell dimensions a = 9.6150(3) Å = 90°.

b = 11.7549(4) Å = 105.283(3)°.

c = 17.2226(5) Å  = 90°.

Volume 1877.72(10) Å3

Z 4

Density (calculated) 1.677 Mg/m3

Absorption coefficient 2.452 mm-1

F(000) 944

Crystal size 0.1 x 0.1 x 0.1 mm3

Theta range for data collection 2.83 to 30.15°.

Index ranges -13<=h<=12, -16<=k<=16, -23<=l<=24

Reflections collected 27212

Independent reflections 5212 [R(int) = 0.0547]

Completeness to theta = 27.00° 99.9 %

Absorption correction Semi-empirical from equivalents

Max. and min. transmission 1.00000 and 0.84395

Refinement method Full-matrix least-squares on F2

Data / restraints / parameters 5212 / 3 / 206

Goodness-of-fit on F2 1.128

Final R indices [I>2sigma(I)] R1 = 0.0505, wR2 = 0.1106

R indices (all data) R1 = 0.0653, wR2 = 0.1161

Largest diff. peak and hole 1.336 and -0.621 e.Å-3

[4-*t*Bu-C6H4C(=O)(*η*5-C5H4)Fe(*η*5-C5H5)] (21)

Empirical formula C21 H22 Fe O

Formula weight 346.24

Temperature 123(2) K

Wavelength 0.71073 Å

Crystal system Monoclinic

Space group P 1 21/n 1

Unit cell dimensions a = 10.9164(6) Å = 90°.

b = 25.1685(13) Å = 91.060(5)°.

c = 18.3340(11) Å  = 90°.

Volume 5036.4(5) Å3

Z 12

Density (calculated) 1.370 Mg/m3

Absorption coefficient 0.900 mm-1

F(000) 2184

Crystal size 0.3 x 0.05 x 0.05 mm3

Theta range for data collection 2.99 to 27.00°.

Index ranges -13<=h<=13, -32<=k<=32, -23<=l<=23

Reflections collected 45919

Independent reflections 10967 [R(int) = 0.1130]

Completeness to theta = 27.00° 99.9 %

Absorption correction Semi-empirical from equivalents

Max. and min. transmission 1.00000 and 0.83390

Refinement method Full-matrix least-squares on F2

Data / restraints / parameters 10967 / 0 / 631

Goodness-of-fit on F2 0.990

Final R indices [I>2sigma(I)] R1 = 0.0580, wR2 = 0.1008

R indices (all data) R1 = 0.1196, wR2 = 0.1252

Largest diff. peak and hole 0.502 and -0.618 e.Å-3

Crystal data for [(THF)2Li4(*o*-C6H4OMe)4] (25)

Empirical formula C36 H44 Li4 O6

Formula weight 600.47

Temperature 150(2) K

Radiation, wavelength MoK, 0.71073 Å

Crystal system monoclinic

Space group P21/n

Unit cell parameters a = 13.069(4) Å  = 90°

b = 13.676(5) Å  = 94.77(4)°

c = 19.856(12) Å  = 90°

Cell volume 3536(3) Å3

Z 4

Calculated density 1.128 g/cm3

Absorption coefficient  0.073 mm−1

F(000) 1280

Crystal colour and size colourless, 0.15 × 0.10 × 0.10 mm3

Reflections for cell refinement 102 ( range 2.5 to 27.5°)

 range for data collection 4.2 to 20.5°

Index ranges h −12 to 12, k −13 to 13, l −19 to 19

Completeness to  = 20.5° 98.8 %

Reflections collected 16554

Independent reflections 3508 (Rint = 0.0972)

Reflections with F2>2 2234

Absorption correction semi-empirical from equivalents

Min. and max. transmission 0.9891 and 0.9927

Refinement method Full-matrix least-squares on F2

Data / restraints / parameters 3508 / 309 / 452

Final R indices [F2>2] R1 = 0.0900, wR2 = 0.1850

R indices (all data) R1 = 0.1497, wR2 = 0.2133

Goodness-of-fit on F2 1.146

Largest and mean shift/su 0.003 and 0.000

Largest diff. peak and hole 0.22 and −0.23 e Å−3

Crystal data for [(THF)2Li2(*o*-C6H4OMe)4Zn] (**27**)

Empirical formula (total) C36 H44 Li2 O6 Zn

Formula weight 651.96

Temperature 150(2) K

Radiation, wavelength MoK, 0.71073 Å

Crystal system monoclinic

Space group P21/c

Unit cell parameters a = 12.030(5) Å  = 90°

b = 17.467(5) Å  = 107.689(11)°

c = 17.5333(14) Å  = 90°

Cell volume 3510.2(17) Å3

Z 4

Calculated density 1.234 g/cm3

Absorption coefficient  0.741 mm−1

F(000) 1376

Crystal colour and size colourless, 0.58 × 0.28 × 0.12 mm3

Reflections for cell refinement 8031 ( range 2.5 to 27.5°)

 range for data collection 5.2 to 27.5°

Index ranges h −15 to 15, k −21 to 22, l −22 to 22

Completeness to  = 27.5° 95.9 %

Reflections collected 26446

Independent reflections 7763 (Rint = 0.0574)

Reflections with F2>2 4803

Absorption correction semi-empirical from equivalents

Min. and max. transmission 0.6732 and 0.9164

Refinement method Full-matrix least-squares on F2

Data / restraints / parameters 7763 / 284 / 472

Final R indices [F2>2] R1 = 0.0482, wR2 = 0.0831

R indices (all data) R1 = 0.1091, wR2 = 0.1010

Goodness-of-fit on F2 1.030

Largest and mean shift/su 0.001 and 0.000

Largest diff. peak and hole 0.64 and −0.35 e Å−3

Crystal data for [(TMEDA)Li(*o*-C6H4OMe)ZnMe2] (**28**)

Empirical formula C15 H29 Li N2 O Zn

Formula weight 325.71

Temperature 150(2) K

Radiation, wavelength MoK, 0.71073 Å

Crystal system orthorhombic

Space group P212121

Unit cell parameters a = 8.3053(3) Å  = 90°

b = 13.0406(3) Å  = 90°

c = 16.4544(5) Å  = 90°

Cell volume 1782.11(9) Å3

Z 4

Calculated density 1.214 g/cm3

Absorption coefficient  1.375 mm−1

F(000) 696

Crystal colour and size colourless, 0.18 × 0.14 × 0.04 mm3

Reflections for cell refinement 3057 ( range 2.9 to 29.5°)

 range for data collection 2.9 to 25.0°

Index ranges h −9 to 9, k −15 to 12, l −19 to 18

Completeness to  = 25.0° 99.6 %

Reflections collected 5932

Independent reflections 3047 (Rint = 0.0284)

Reflections with F2>2 2474

Absorption correction semi-empirical from equivalents

Min. and max. transmission 0.7900 and 0.9471

Refinement method Full-matrix least-squares on F2

Data / restraints / parameters 3047 / 0 / 189

Final R indices [F2>2] R1 = 0.0288, wR2 = 0.0406

R indices (all data) R1 = 0.0411, wR2 = 0.0425

Goodness-of-fit on F2 0.887

Absolute structure parameter 0.024(12)

Largest diff. peak and hole 0.31 and −0.32 e Å−3

Crystal data for [(PMDETA)Li(*o*-C6H4OMe)ZntBu2] (30)

Empirical formula C24 H48 Li N3 O Zn

Formula weight 466.96

Temperature 150(2) K

Radiation, wavelength MoK, 0.71073 Å

Crystal system, space group orthorhombic, P212121

Unit cell parameters a = 12.0463(3) Å  = 90°

b = 14.9178(5) Å  = 90°

c = 15.5281(4) Å  = 90°

Cell volume 2790.47(14) Å3

Z 4

Calculated density 1.112 g/cm3

Absorption coefficient  0.897 mm−1

F(000) 1016

Crystal colour and size colourless, 0.43 × 0.39 × 0.26 mm3

Reflections for cell refinement 6916 ( range 3.0 to 29.4°)

 range for data collection 6.8 to 29.4°

Index ranges h −15 to 16, k −20 to 16, l −18 to 20

Completeness to  = 25.0° 97.3 %

Reflections collected 12878

Independent reflections 6292 (Rint = 0.0270)

Reflections with F2>2 4397

Absorption correction semi-empirical from equivalents

Min. and max. transmission 0.6989 and 0.8002

Refinement method Full-matrix least-squares on F2

Data / restraints / parameters 6292 / 1123 / 456

Final R indices [F2>2] R1 = 0.0375, wR2 = 0.0748

R indices (all data) R1 = 0.0619, wR2 = 0.0782

Goodness-of-fit on F2 0.933

Absolute structure parameter 0.013(13)

Largest diff. peak and hole 0.49 and −0.39 e Å−3

Crystal data for [{Mg(THF)6}2+{Zn2Cl6}2-] (**33**)

Empirical formula C24 H48 Cl6 Mg O6 Zn2

Formula weight 800.37

Temperature 123(2) K

Wavelength 0.71073 Å

Crystal system Monoclinic

Space group P21/n

Unit cell dimensions a = 10.2436(2) Å = 90°.

b = 13.7110(3) Å = 97.188(2)°.

c = 12.1826(2) Å  = 90°.

Volume 1697.60(6) Å3

Z 2

Density (calculated) 1.566 Mg/m3

Absorption coefficient 1.938 mm-1

F(000) 828

Crystal size 0.1 x 0.08 x 0.03 mm3

Theta range for data collection 2.45 to 25.98°.

Index ranges -12<=h<=12, -16<=k<=16, -14<=l<=15

Reflections collected 14625

Independent reflections 3329 [R(int) = 0.0319]

Completeness to theta = 25.98° 100.0 %

Absorption correction Semi-empirical from equivalents

Max. and min. transmission 1.00000 and 0.94284

Refinement method Full-matrix least-squares on F2

Data / restraints / parameters 3329 / 0 / 256

Goodness-of-fit on F2 0.941

Final R indices [I>2sigma(I)] R1 = 0.0279, wR2 = 0.0546

R indices (all data) R1 = 0.0516, wR2 = 0.0590

Largest diff. peak and hole 0.434 and -0.318 e.Å-3

Crystal data for [(THF)4Mg(μ-Cl)2Zn(tBu)(Cl)] (**34**)

Empirical formula C20 H41 Cl3 Mg O4 Zn

Formula weight 541.56

Temperature 123(2) K

Wavelength 0.71073 Å

Crystal system Monoclinic

Space group P21/c

Unit cell dimensions a = 12.1266(3) Å = 90°.

b = 10.5782(2) Å = 100.848(2)°.

c = 20.7697(5) Å  = 90°.

Volume 2616.68(10) Å3

Z 4

Density (calculated) 1.375 Mg/m3

Absorption coefficient 1.291 mm-1

F(000) 1144

Crystal size 0.20 x 0.12 x 0.03 mm3

Theta range for data collection 2.58 to 28.15°.

Index ranges -15<=h<=14, -13<=k<=11, -27<=l<=24

Reflections collected 10847

Independent reflections 5614 [R(int) = 0.0369]

Completeness to theta = 25.00° 97.8 %

Absorption correction Semi-empirical from equivalents

Max. and min. transmission 1.00000 and 0.86865

Refinement method Full-matrix least-squares on F2

Data / restraints / parameters 5614 / 0 / 283

Goodness-of-fit on F2 0.917

Final R indices [I>2sigma(I)] R1 = 0.0374, wR2 = 0.0636

R indices (all data) R1 = 0.0721, wR2 = 0.0691

Largest diff. peak and hole 0.446 and -0.311 e.Å-3

Crystal data for [{(THF)2Mg(μ-Cl)3ZntBu}2] (**36**)

Empirical formula C31 H58 Cl6 Mg2 O4 Zn2

Formula weight 886.83

Temperature 123(2) K

Wavelength 0.71073 Å

Crystal system monoclinic

Space group C2/m

Unit cell dimensions a = 12.6635(2) Å = 90°.

b = 16.1147(2) Å = 102.450(2)°.

c = 21.4575(4) Å  = 90°.

Volume 4275.83(12) Å3

Z 4

Density (calculated) 1.378 Mg/m3

Absorption coefficient 1.557 mm-1

F(000) 1848

Crystal size 0.15 x 0.10 x 0.05 mm3

Theta range for data collection 2.44 to 30.74°.

Index ranges -17<=h<=17, -21<=k<=23, -30<=l<=30

Reflections collected 24398

Independent reflections 6329 [R(int) = 0.0216]

Completeness to theta = 29.00° 99.5 %

Absorption correction Semi-empirical from equivalents

Max. and min. transmission 1.00000 and 0.86918

Refinement method Full-matrix least-squares on F2

Data / restraints / parameters 6329 / 0 / 254

Goodness-of-fit on F2 1.016

Final R indices [I>2sigma(I)] R1 = 0.0346, wR2 = 0.0754

R indices (all data) R1 = 0.0564, wR2 = 0.0799

Largest diff. peak and hole 0.595 and -0.503 e.Å-3

Crystal data for [{(THF)2Mg(μ-Cl)3ZnnBu}2] (**37**)

Empirical formula C24H50Cl6Mg2O4Zn2

Formula weight 794.70

Temperature 150(2) K

Radiation, wavelength MoK, 0.71073 Å

Crystal system monoclinic

space group P121/c1

Unit cell parameters a = 10.9057(2) Å  = 90°

b = 15.9944(3) Å  = 90.496(2)°

c = 10.2484(2) Å  = 90°

Cell volume 1787.56(6) Å3

Z 2

Calculated density 1.476 g/cm3

Absorption coefficient  1.852 mm−1

F(000) 824

Crystal colour and size colourless, 0.26 × 0.22 × 0.10 mm3

Reflections for cell refinement 5934 ( range 3.0 to 28.5°)

 range for data collection 3.0 to 28.6°

Index ranges h −13 to 13, k −17 to 19, l −8 to 12

Completeness to  = 28.6° 83.1 %

Reflections collected 9826

Independent reflections 3784 (Rint = 0.0221)

Reflections with F2>2 3020

Absorption correction semi-empirical from equivalents

Min. and max. transmission 0.6445 and 0.8364

Refinement method Full-matrix least-squares on F2

Data / restraints / parameters 3784 / 0 / 173

Final R indices [F2>2] R1 = 0.0250, wR2 = 0.0610

R indices (all data) R1 = 0.0353, wR2 = 0.0626

Goodness-of-fit on F2 1.019

Largest and mean shift/su 0.002 and 0.000

Largest diff. peak and hole 0.77 and −0.42 e Å−3

Crystal data for [{(THF)2Mg(μ-Cl)3ZnEt}2] (**38**)

Chemical formula (total) C20H42Cl6Mg2O4Zn2

Formula weight 738.60

Temperature 200(2) K

Radiation, wavelength MoK, 0.71073 Å

Crystal system triclinic

Space group P‾1

Unit cell parameters a = 8.8011(8) Å  = 104.299(7)°

b = 10.3150(8) Å  = 107.476(8)°

c = 10.4190(9) Å  = 107.631(7)°

Cell volume 798.21(12) Å3

Z 1

Calculated density 1.537 g/cm3

Absorption coefficient  2.068 mm−1

F(000) 380

Crystal colour and size colourless, 0.34 × 0.18 × 0.08 mm3

Reflections for cell refinement 3176 ( range 3.1 to 28.4°)

 range for data collection 3.3 to 28.4°

Index ranges h −8 to 11, k −13 to 12, l −13 to 12

Completeness to  = 25.0° 98.2 %

Reflections collected 5097

Independent reflections 3172 (Rint = 0.0171)

Reflections with F2>2 2444

Absorption correction semi-empirical from equivalents

Min. and max. transmission 0.5399 and 0.8520

Refinement method Full-matrix least-squares on F2

Data / restraints / parameters 3172 / 0 / 155

Final R indices [F2>2] R1 = 0.0279, wR2 = 0.0710

R indices (all data) R1 = 0.0390, wR2 = 0.0732

Goodness-of-fit on F2 0.987

Largest and mean shift/su 0.002 and 0.000

Largest diff. peak and hole 0.49 and −0.31 e Å−3

Crystal data for [{(THF)2Mg(μ-Cl)3Zn(*o*-C6H4OMe)}2] (**39**)

Empirical formula C30 H46 Cl6 Mg2 O6 Zn2

Formula weight 894.73

Temperature 123(2) K

Wavelength 1.54180 Å

Crystal system Triclinic

Space group P-1

Unit cell dimensions a = 8.2506(18) Å = 111.74(2)°.

b = 10.743(2) Å = 98.88(2)°.

c = 11.785(3) Å  = 95.063(18)°.

Volume 946.5(4) Å3

Z 1

Density (calculated) 1.570 Mg/m3

Absorption coefficient 6.106 mm-1

F(000) 460

Crystal size 0.2 x 0.1 x 0.05 mm3

Theta range for data collection 4.12 to 65.00°.

Index ranges -9<=h<=9, -8<=k<=12, -13<=l<=10

Reflections collected 5266

Independent reflections 3042 [R(int) = 0.0577]

Completeness to theta = 60.00° 98.1 %

Absorption correction Semi-empirical from equivalents

Max. and min. transmission 1.00000 and 0.48181

Refinement method Full-matrix least-squares on F2

Data / restraints / parameters 3042 / 0 / 209

Goodness-of-fit on F2 1.037

Final R indices [I>2sigma(I)] R1 = 0.0695, wR2 = 0.1898

R indices (all data) R1 = 0.1072, wR2 = 0.2121

Largest diff. peak and hole 1.155 and -0.632 e.Å-3

Crystal data for [{Mg2Cl3(THF)6}+{ZntBu3}-] (**40**)

Empirical formula C40 H83 Cl3 Mg2 O7 Zn

Formula weight 896.40

Temperature 123(2) K

Wavelength 1.54185 Å

Crystal system orthorhombic

Space group P212121

Unit cell dimensions a = 13.5244(2) Å = 90°.

b = 18.7865(3) Å = 90°.

c = 19.3262(3) Å  = 90°.

Volume 4910.33(13) Å3

Z 4

Density (calculated) 1.213 Mg/m3

Absorption coefficient 2.770 mm-1

F(000) 1936

Crystal size 0.1 x 0.1 x 0.04 mm3

Theta range for data collection 5.63 to 61.67°.

Index ranges -14<=h<=15, -21<=k<=16, -14<=l<=22

Reflections collected 22471

Independent reflections 6591 [R(int) = 0.0416]

Completeness to theta = 60.00° 98.8 %

Absorption correction Semi-empirical from equivalents

Max. and min. transmission 1.00000 and 0.67607

Refinement method Full-matrix least-squares on F2

Data / restraints / parameters 6591 / 0 / 486

Goodness-of-fit on F2 0.877

Final R indices [I>2sigma(I)] R1 = 0.0382, wR2 = 0.0719

R indices (all data) R1 = 0.0640, wR2 = 0.0751

Absolute structure parameter 0.279(15)

Largest diff. peak and hole 0.579 and -0.224 e.Å-3

Crystal data for [{Mg3Br3(OEt)2(THF)6}+{Zn2Et5}-] (**42**)

Empirical formula C42 H91 Br3 Mg3 O9 Zn2

Formula weight 1183.55

Temperature 123(2) K

Wavelength 0.71069 Å

Crystal system monoclinic

Space group P 21/n

Unit cell dimensions a = 11.9374(2) Å = 90°.

b = 19.2841(3) Å = 101.776(2)°.

c = 24.3668(4) Å  = 90°.

Volume 5491.23(15) Å3

Z 4

Density (calculated) 1.432 Mg/m3

Absorption coefficient 3.136 mm-1

F(000) 2464

Crystal size 0.2 x 0.1 x 0.06 mm3

Theta range for data collection 2.76 to 26.00°.

Index ranges -11<=h<=14, -22<=k<=23, -28<=l<=30

Reflections collected 38318

Independent reflections 10771 [R(int) = 0.0388]

Completeness to theta = 26.00° 99.9 %

Refinement method Full-matrix least-squares on F2

Data / restraints / parameters 10771 / 34 / 590

Goodness-of-fit on F2 1.031

Final R indices [I>2sigma(I)] R1 = 0.0484, wR2 = 0.1052

R indices (all data) R1 = 0.0846, wR2 = 0.1147

Largest diff. peak and hole 0.823 and -0.506 e.Å-3

Crystal data for [{(THF)6Mg2Cl3}+{Mg2(OC(Et)Ph2)2Cl3(THF)}‒] (**44**)

Empirical formula C66 H102 Cl6 Mg4 O11

Formula weight 1381.42

Temperature 150(2) K

Radiation, wavelength MoK, 0.71073 Å

Crystal system triclinic

Space group P‾1

Unit cell parameters a = 12.2922(4) Å  = 76.886(4)°

b = 17.6084(9) Å  = 73.607(4)°

c = 18.4551(9) Å  = 73.777(4)°

Cell volume 3631.7(3) Å3

Z 2

Calculated density 1.263 g/cm3

Absorption coefficient  0.325 mm−1

F(000) 1472

Crystal colour and size colourless, 0.28 × 0.26 × 0.08 mm3

Reflections for cell refinement 7299 ( range 3.2 to 28.5°)

 range for data collection 3.2 to 28.5°

Index ranges h −15 to 12, k −19 to 22, l −22 to 22

Completeness to  = 25.0° 98.3 %

Reflections collected 24263

Independent reflections 14588 (Rint = 0.0394)

Reflections with F2>2 7182

Absorption correction semi-empirical from equivalents

Min. and max. transmission 0.9144 and 0.9744

Refinement method Full-matrix least-squares on F2

Data / restraints / parameters 14588 / 1430 / 832

Final R indices [F2>2] R1 = 0.0455, wR2 = 0.0896

R indices (all data) R1 = 0.1096, wR2 = 0.0995

Goodness-of-fit on F2 0.804

Largest and mean shift/su 0.018 and 0.001

Largest diff. peak and hole 0.50 and −0.37 e Å−3

Crystal data for [{(THF)5Mg3Cl4{OC(H)Ph(CF3)}2] (**50**)

Chemical formula (total) C36 H52 Cl4 F6 Mg3 O7

Formula weight 925.51

Temperature 150(2) K

Radiation, wavelength MoK, 0.71073 Å

Crystal system monoclinic

Space group P121/n1

Unit cell parameters a = 12.7595(4) Å  = 90°

b = 26.1757(8) Å  = 98.802(3)°

c = 13.4412(4) Å  = 90°

Cell volume 4436.3(2) Å3

Z 4

Calculated density 1.386 g/cm3

Absorption coefficient  0.378 mm−1

F(000) 1928

Crystal colour and size colourless, 0.32 × 0.24 × 0.03 mm3

Reflections for cell refinement 5116 ( range 2.8 to 28.4°)

 range for data collection 2.8 to 28.5°

Index ranges h −12 to 15, k −32 to 34, l −17 to 14

Completeness to  = 25.0° 99.0 %

Reflections collected 17079

Independent reflections 9102 (Rint = 0.0365)

Reflections with F2>2 4894

Absorption correction semi-empirical from equivalents

Min. and max. transmission 0.8887 and 0.9888

Refinement method Full-matrix least-squares on F2

Data / restraints / parameters 9102 / 593 / 600

Final R indices [F2>2] R1 = 0.0394, wR2 = 0.0721

R indices (all data) R1 = 0.0908, wR2 = 0.0787

Goodness-of-fit on F2 0.801

Largest and mean shift/su 0.001 and 0.000

Largest diff. peak and hole 0.43 and −0.32 e Å−3

Crystal data for [{Mg2Cl2(THF)6}+{Zn(*p-*Tol)3}-] (**52**)

Empirical formula C45 H69 Cl3 Mg2 O6 Zn

Formula weight 926.34

Temperature 123(2) K

Wavelength 0.71073 Å

Crystal system MONOCLINIC

Space group P21/c

Unit cell dimensions a = 14.6632(14) Å = 90°.

b = 21.3537(19) Å = 107.164(10)°.

c = 15.8903(16) Å  = 90°.

Volume 4753.9(8) Å3

Z 4

Density (calculated) 1.294 Mg/m3

Absorption coefficient 0.756 mm-1

F(000) 1968

Crystal size 0.25 x 0.12 x 0.04 mm3

Theta range for data collection 2.40 to 23.50°.

Index ranges -16<=h<=16, -23<=k<=23, -17<=l<=17

Reflections collected 26225

Independent reflections 6899 [R(int) = 0.1164]

Completeness to theta = 23.50° 98.2 %

Absorption correction Semi-empirical from equivalents

Max. and min. transmission 1.00000 and 0.59124

Refinement method Full-matrix least-squares on F2

Data / restraints / parameters 6899 / 0 / 509

Goodness-of-fit on F2 0.706

Final R indices [I>2sigma(I)] R1 = 0.0498, wR2 = 0.0682

R indices (all data) R1 = 0.1480, wR2 = 0.0785

Largest diff. peak and hole 0.595 and -0.393 e.Å-3

Crystal data for [{Mg(THF)6}2+}{Zn(*o*-C6H4OMe)3}2-] (**64**)

Chemical formula (total) C70H98MgO13Zn2

Formula weight 1302.53

Temperature 150(2) K

Radiation, wavelength MoK, 0.71073 Å

Crystal system tetragonal

Space group P41212

Unit cell parameters a = 12.1808(3) Å  = 90°

b = 12.1808(3) Å  = 90°

c = 44.474(2) Å  = 90°

Cell volume 6598.7(4) Å3

Z 4

Calculated density 1.311 g/cm3

Absorption coefficient  0.799 mm−1

F(000) 2776

Crystal colour and size colourless, 0.46 × 0.42 × 0.32 mm3

Reflections for cell refinement 3175 ( range 3.2 to 28.5°)

 range for data collection 3.2 to 24.0°

Index ranges h −11 to 12, k −10 to 13, l −36 to 50

Completeness to  = 24.0° 99.3 %

Reflections collected 11689

Independent reflections 5159 (Rint = 0.0512)

Reflections with F2>2 3086

Absorption correction semi-empirical from equivalents

Min. and max. transmission 0.7102 and 0.7842

Refinement method Full-matrix least-squares on F2

Data / restraints / parameters 5159 / 858 / 438

Final R indices [F2>2] R1 = 0.0840, wR2 = 0.2298

R indices (all data) R1 = 0.1213, wR2 = 0.2449

Goodness-of-fit on F2 1.008

Absolute structure parameter 0.04(4)

Largest diff. peak and hole 0.70 and −0.42 e Å−3

Crystal data for [(THF)4MgCl{NC-C6H4}ZnI(C6H4CN)(THF)] (**68**)

Empirical formula C34 H48 Cl I Mg N2 O5 Zn

Formula weight 816.77

Temperature 123(2) K

Wavelength 0.71069 Å

Crystal system Monoclinic

Space group P21/c

Unit cell dimensions a = 16.1212(8) Å = 90°.

b = 9.5045(6) Å = 98.132(5)°.

c = 24.1563(14) Å  = 90°.

Volume 3664.1(4) Å3

Z 4

Density (calculated) 1.481 Mg/m3

Absorption coefficient 1.644 mm-1

F(000) 1672

Crystal size .2 x .18 x .12 mm3

Theta range for data collection 2.74 to 27.00°.

Index ranges -19<=h<=20, -12<=k<=12, -30<=l<=29

Reflections collected 17865

Independent reflections 7990 [R(int) = 0.0444]

Completeness to theta = 27.00° 99.9 %

Absorption correction Semi-empirical from equivalents

Max. and min. transmission 1.00000 and 0.83493

Refinement method Full-matrix least-squares on F2

Data / restraints / parameters 7990 / 0 / 406

Goodness-of-fit on F2 0.815

Final R indices [I>2sigma(I)] R1 = 0.0384, wR2 = 0.0540

R indices (all data) R1 = 0.0838, wR2 = 0.0589

Largest diff. peak and hole 0.889 and -0.726 e.Å-3