Experimental

Data Collection

Yellow crystals were mounted and sealed in a glass capillary under nitrogen. The X-ray intensity data were measured at room temperature on a Bruker SMART TM CCD-based X-ray diffractometer system with graphite-monochromated Mo Kα radiation (λ = 0.71073 Å) using ω-scan technique to maximum 2θ value of 48.36°. Crystallographic data: $[C_{40}H_{50}O_3InLiBr_2]_2$: monoclinic, space group C2/c, a = 23.188(3) Å, b =14.567 (2) Å, c = 24.921 (4) Å, α = 90° , β = 103.296(3)° , γ = 90° , V = 8192(2) Å^3, Z = 4, F.W. = 1720.76, μ = 2.561 mm^{-1}, d = 1.395 g/cm^3, F(000) = 3488.

Data Reduction [1]

Of the 6046 independent reflections in total 11561 reflections collected, 3945 were observed ($I > 2\ \sigma(I)$). The linear absorption coefficient for Mo Kα radiation is 2.561 mm^{-1}. The data were corrected for Lorentz and polarization effects. A routine absorption correction was applied by using SADABS and any decay correction was made with the SAINT program.

Structure Solution and Refinement [2]

The structure was solved by direct methods using the SHELXTL 5.1 Software Package. The non-hydrogen atoms except for carbon atoms C19, C20, C26, C27 and one oxygen atom O3 and all carbon atoms of three diethyl ether molecules were refined anisotropically while hydrogen atoms were placed in ideal positions with their coordinates and thermal parameters riding on the attached carbon atoms. The carbon atoms C35, C36, C35', C36' of one diethyl ether were found disordered with site occupancy of 0.55 for C35, C36, and site occupancy of 0.45 for C35', C36'. No disorder for some phenyl rings was found, although some carbon atoms of them have a little high thermal parameters. This compound is monoclinic, space group C2/c (No.15). The final residual values based on 349 variable parameters and 3945 observed reflections ($I > 2\ \sigma(I)$) are $R1$ = 0.0574, $wR2$ = 0.1357 and those for all 6046 unique reflections are $R1$ = 0.0983, $wR2$ = 0.1568. The goodness-of-fit indicator for all data is 1.046. The largest peak on the final difference map is 0.896 e/Å^3.

Summary

The compound crystallizes in the monoclinic space group C2/c (No.15). The asymmetric unit for the compound contains one half $(Ph_4C_4In)_2Li_2Br_4$ molecule and three diethyl ether molecules. The other half molecule of $(Ph_4C_4In)_2Li_2Br_4$ is generated by a two-fold axis perpendicular to the line between In(1) and In(1a). The two of diethyl ether molecules are coordinated to lithium atoms and another is a discrete solvent molecule. Structure solution, refinement and the calculation of derived results were performed using the SHELXTL 5.1[3] package of computer programs. Neutral atom scattering factors were those of Cromer and Waber,[4] and the real and imaginary anomalous dispersion corrections were those of Cromer.[5]

References

1. Data Reduction:

Intensity

$I = [S - B/R] \cdot V$

Standard Deviation in Intensity

$s(I) = [S + B/R^2]^{1/2} \cdot V$

Structure Factor

$F = (I/Lp)^{1/2}$

Standard Deviation in Structure Factor

$s(F) = s(I)/(2 \cdot F \cdot Lp)$

Where: S = total scan count
B = sum of background counts
R = ratio of background counting time to scan counting time
V = scan rate
Lp = Lorentz-polarization correction

2. Least-Squares Refinement:

Weighting Scheme

$weight = 1/[\sigma^2(F_o) + (0.087*P)^2 + 0.15*P]$ where $P = [Max(F_o^2, 0) + 2*F_c^2]/3$;

R-factors:

$R = \Sigma \| F_o| - |F_c| | / \Sigma |F_o|$;

Weighted R-factor on F^2:

$wR2 = [\Sigma[w (F_o^2 - F_c^2)^2] / \Sigma[w(F_o^2)^2]]^{1/2}$; $w = 1/[\sigma^2(F_o^2) + (0.095*P)^2]$;

Goodness of Fit Indicator:

goodness-of-fit = $[\Sigma[w (F_o^2 - F_c^2)^2] / (N_{observns} - N_{params})$

3. Sheldrick, G.M. *SHELXTL 5.1, Crystallographic Computing System*; Siemens Analytical X-Ray Instruments, Madison, WI, 1997.

4. Cromer, D.T. and Waber, J.T. International Tables for X-ray Crystallography, Vol. IV, Table 2.2B, The Kynoch Press, Birmingham England, 1974.

5. Cromer, D.T. International Tables for X-ray Crystallography, Vol. IV, Table 2.3.1, The Kynoch Press, Birmingham England, 1974.

Table 1. Crystallographic data for $(Et_2O)_2Li(Br)_2In\{(C_4Ph_4)\}_2In(Br)_2Li(OEt_2)_2$

Crystal Data	
empirical formula	$[C_{40}H_{50}O_3InLiBr_2]_2$
fw	1720.76
color; habit	yellow, cubic
space group	monoclinic, *C2/c* (#15)
unit cell dimens	
a	23.188(3) Å
b	14.567(2) Å
c	24.921(4) Å
α	90°
β	103.296(3)°
γ	90°
vol (Å^3)	8192(2)
formula units/cell	4
D_{calc} (g/cm^3)	1.395
F(000)	3488
Temp (K)	293(2)
Data Collection and Refinement	
maximum 2θ angle	48.36°
total no. of reflns collected	11561
no. of indep reflns	6046
no. of reflns observed [I > 2σ(*I*)]	3945
no of params refined	349
abs coeff (mm^{-1})	2.561
refinement method	Full-matrix least-squares on F^2
final R indices [I > 2σ(*I*)]	*R1* = 0.0574; *wR2*= 0.1357
R indices (all data)	*R 1*= 0.0983; *wR2*= 0.1568
goodness of fit	*S* = 1.046
largest diff. peak and hole(e$^-$/Å^3)	0.896

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for $(Et_2O)_2Li(Br)_2In\{(C_4Ph_4)\}_2In(Br)_2Li(OEt_2)_2$.

	x	y	z	U(eq)
In(1)	582(1)	8335(1)	2226(1)	46(1)
Li(1)	1969(8)	8527(13)	1775(7)	95(5)
Br(1)	1595(1)	7402(1)	2422(1)	69(1)
Br(2)	916(1)	9205(1)	1409(1)	70(1)
C(1)	714(3)	9231(5)	2958(3)	47(2)
C(2)	783(4)	10237(5)	2902(3)	55(2)
C(3)	1194(5)	10606(6)	2625(4)	79(3)
C(4)	1252(6)	11544(7)	2601(5)	108(4)
C(5)	909(7)	12121(7)	2821(6)	113(4)
C(6)	509(5)	11786(7)	3087(4)	90(3)
C(7)	448(4)	10851(6)	3132(3)	69(2)
C(8)	762(3)	8851(5)	3455(3)	41(2)
C(9)	975(3)	9361(5)	3986(3)	53(2)
C(10)	1502(4)	9840(6)	4094(3)	65(2)
C(11)	1691(5)	10265(7)	4597(4)	87(3)
C(12)	1359(5)	10276(7)	4982(4)	87(3)
C(13)	847(5)	9821(7)	4887(3)	81(3)
C(14)	653(4)	9345(6)	4394(3)	67(2)
C(15)	631(3)	7858(5)	3521(3)	50(2)
C(16)	1082(4)	7348(5)	3945(3)	60(2)
C(17)	1680(4)	7414(7)	3952(4)	76(3)
C(18)	2105(5)	6953(8)	4371(5)	105(4)
C(19)	1904(6)	6462(9)	4746(6)	116(4)
C(20)	1325(5)	6416(8)	4774(5)	112(4)
C(21)	929(5)	6851(7)	4352(4)	93(3)
C(22)	125(3)	7450(5)	3244(3)	47(2)
C(23)	36(4)	6442(5)	3268(3)	58(2)
C(24)	-448(4)	6098(6)	3449(4)	79(3)
C(25)	-500(5)	5107(8)	3489(5)	122(5)
C(26)	-61(6)	4579(10)	3344(5)	115(4)
C(27)	379(5)	4895(8)	3178(4)	101(3)
C(28)	437(5)	5839(6)	3130(4)	78(3)
O(1)	2581(4)	9259(6)	2264(4)	127(3)
O(2)	2282(4)	7988(7)	1208(3)	132(3)
C(29)	2780(9)	10112(12)	2226(8)	181(7)
C(30)	2457(8)	10591(12)	1729(7)	185(7)
C(31)	2957(8)	8813(12)	2783(7)	166(6)
C(32)	2756(8)	9119(12)	3256(7)	189(7)
C(33)	1879(8)	7383(12)	834(8)	179(7)
C(34)	1949(9)	6446(15)	1009(8)	217(9)
C(35)	2977(16)	8340(40)	1224(14)	147(15)
C(35')	2831(17)	7750(40)	1216(14)	123(17)
C(36)	3027(17)	8790(40)	817(17)	183(18)
C(36')	2950(20)	8190(50)	770(20)	160(20)
O(3)	-174(9)	2920(14)	4424(7)	249(7)
C(37)	455(16)	2960(20)	4720(15)	340(17)
C(38)	466(16)	4040(20)	4874(15)	390(20)
C(39)	-337(11)	2124(18)	4262(10)	230(10)

C(40)	-1005(12)	2095(19)	3990(11)	277(12)

*Atoms of C35, C35' C36, C36' are disordered and occupancy for atoms C35 C36 is 0.55, and occupancy for atoms C35', C36' is 0.45, respectively.

Table 3. Bond lengths [Å] and angles [deg] for $(Et_2O)_2Li(Br)_2In\{(C_4Ph_4)\}_2In(Br)_2Li(OEt_2)_2$

In(1)-C(1)	2.206(7)
In(1)-C(22a)	2.200(7)
In(1)-Br(1)	2.6589(10)
In(1)-Br(2)	2.6610(10)
In(1)-In(1a)	3.2935(11)
Li(1)-O(2)	1.90(2)
Li(1)-O(1)	1.96(2)
Li(1)-Br(2)	2.598(17)
Li(1)-Br(1)	2.585(18)
C(1)-C(8)	1.338(9)
C(1)-C(2)	1.484(10)
C(2)-C(7)	1.393(11)
C(2)-C(3)	1.407(11)
C(3)-C(4)	1.375(13)
C(4)-C(5)	1.356(17)
C(5)-C(6)	1.348(16)
C(6)-C(7)	1.377(12)
C(8)-C(9)	1.498(10)
C(8)-C(15)	1.494(10)
C(9)-C(10)	1.380(11)
C(9)-C(14)	1.391(11)
C(10)-C(11)	1.377(11)
C(11)-C(12)	1.361(13)
C(12)-C(13)	1.333(13)
C(13)-C(14)	1.392(11)
C(15)-C(22)	1.355(10)
C(15)-C(16)	1.503(10)
C(16)-C(21)	1.358(13)
C(16)-C(17)	1.386(12)
C(17)-C(18)	1.428(13)
C(18)-C(19)	1.343(16)
C(19)-C(20)	1.361(15)
C(20)-C(21)	1.380(14)
C(22)-C(23)	1.485(10)
C(22)-In(1a)	2.200(7)
C(23)-C(28)	1.380(12)
C(23)-C(24)	1.395(12)
C(24)-C(25)	1.454(14)
C(25)-C(26)	1.387(17)
C(26)-C(27)	1.273(15)
C(27)-C(28)	1.389(13)
O(1)-C(29)	1.335(17)
O(1)-C(31)	1.528(18)
O(2)-C(35')	1.31(4)
O(2)-C(33)	1.456(18)
O(2)-C(35)	1.68(5)
C(29)-C(30)	1.47(2)
C(31)-C(32)	1.44(2)
C(33)-C(34)	1.43(2)

C(35)-C(35')	0.92(4)
C(35)-C(36')	1.13(5)
C(35)-C(36)	1.24(5)
C(35')-C(36')	1.36(6)
C(35')-C(36)	1.93(6)
C(36)-C(36')	0.90(6)
O(3)-C(39)	1.26(2)
O(3)-C(37)	1.48(3)
C(37)-C(38)	1.62(4)
C(39)-C(40)	1.54(3)

C(1)-In(1)-C(22a)	137.0(3)
C(1)-In(1)-Br(1)	101.67(18)
C(22a)-In(1)-Br(1)	108.43(18)
C(1)-In(1)-Br(2)	109.99(19)
C(22a)-In(1)-Br(2)	100.58(19)
Br(1)-In(1)-Br(2)	89.08(3)
C(1)-In(1)-In(1a)	68.19(19)
C(22a)-In(1)-In(1a)	68.84(19)
Br(1)-In(1)-In(1a)	134.23(3)
Br(2)-In(1)-In(1a)	136.68(3)
O(2)-Li(1)-O(1)	110.0(10)
O(2)-Li(1)-Br(2)	112.6(8)
O(1)-Li(1)-Br(2)	119.9(9)
O(2)-Li(1)-Br(1)	116.3(9)
O(1)-Li(1)-Br(1)	104.8(8)
Br(2)-Li(1)-Br(1)	92.1(6)
Li(1)-Br(1)-In(1)	88.3(4)
Li(1)-Br(2)-In(1)	88.0(4)
C(8)-C(1)-C(2)	120.5(6)
C(8)-C(1)-In(1)	119.2(5)
C(2)-C(1)-In(1)	120.2(5)
C(7)-C(2)-C(3)	117.5(8)
C(7)-C(2)-C(1)	120.9(7)
C(3)-C(2)-C(1)	121.6(8)
C(4)-C(3)-C(2)	119.0(10)
C(5)-C(4)-C(3)	121.8(12)
C(4)-C(5)-C(6)	120.5(10)
C(5)-C(6)-C(7)	119.6(11)
C(2)-C(7)-C(6)	121.6(10)
C(1)-C(8)-C(9)	123.6(6)
C(1)-C(8)-C(15)	121.7(6)
C(9)-C(8)-C(15)	114.7(6)
C(10)-C(9)-C(14)	117.8(7)
C(10)-C(9)-C(8)	121.3(7)
C(14)-C(9)-C(8)	120.9(7)
C(11)-C(10)-C(9)	119.1(8)
C(10)-C(11)-C(12)	122.3(9)
C(13)-C(12)-C(11)	119.6(9)
C(12)-C(13)-C(14)	119.8(9)
C(9)-C(14)-C(13)	121.2(8)
C(22)-C(15)-C(16)	121.5(7)
C(22)-C(15)-C(8)	122.9(6)
C(16)-C(15)-C(8)	115.5(6)

C(21)-C(16)-C(17)	117.5(8)
C(21)-C(16)-C(15)	122.0(8)
C(17)-C(16)-C(15)	120.4(8)
C(16)-C(17)-C(18)	119.7(10)
C(19)-C(18)-C(17)	117.9(11)
C(18)-C(19)-C(20)	124.5(13)
C(19)-C(20)-C(21)	115.5(12)
C(16)-C(21)-C(20)	124.7(11)
C(15)-C(22)-C(23)	121.7(6)
C(15)-C(22)-In(1a)	117.8(5)
C(23)-C(22)-In(1a)	120.5(5)
C(28)-C(23)-C(24)	119.4(8)
C(28)-C(23)-C(22)	120.7(8)
C(24)-C(23)-C(22)	119.8(8)
C(23)-C(24)-C(25)	117.6(10)
C(26)-C(25)-C(24)	117.1(12)
C(27)-C(26)-C(25)	125.1(14)
C(26)-C(27)-C(28)	119.1(13)
C(23)-C(28)-C(27)	121.6(10)
C(29)-O(1)-C(31)	108.5(13)
C(29)-O(1)-Li(1)	132.4(12)
C(31)-O(1)-Li(1)	119.0(10)
C(35')-O(2)-C(33)	109(2)
C(35')-O(2)-C(35)	33.0(17)
C(33)-O(2)-C(35)	132.1(18)
C(35')-O(2)-Li(1)	129.1(19)
C(33)-O(2)-Li(1)	115.3(11)
C(35)-O(2)-Li(1)	112.6(17)
O(1)-C(29)-C(30)	112.6(16)
C(32)-C(31)-O(1)	109.4(15)
O(2)-C(33)-C(34)	111.7(16)
C(35')-C(35)-C(36')	82(4)
C(35')-C(35)-C(36)	126(5)
C(36')-C(35)-C(36)	44(3)
C(35')-C(35)-O(2)	51(3)
C(36')-C(35)-O(2)	94(4)
C(36)-C(35)-O(2)	114(3)
C(35)-C(35')-O(2)	96(4)
C(35)-C(35')-C(36')	56(3)
O(2)-C(35')-C(36')	104(3)
C(35)-C(35')-C(36)	31(3)
O(2)-C(35')-C(36)	97(3)
C(36')-C(35')-C(36)	25(3)
C(36')-C(36)-C(35)	61(4)
C(36')-C(36)-C(35')	39(4)
C(35)-C(36)-C(35')	23(2)
C(36)-C(36')-C(35)	74(5)
C(36)-C(36')-C(35')	116(6)
C(35)-C(36')-C(35')	42(3)
C(39)-O(3)-C(37)	113(2)
O(3)-C(37)-C(38)	97(3)
O(3)-C(39)-C(40)	111(2)

*Atoms of C35, C35' C36, C36' are disordered and occupancy for atoms C35, C36 is 0.55, and occupancy for atoms C35', C36' is 0.45, respectively. Symmetry transformations used to generate equivalent atoms:
A: -x, y, -z+1/2

Table 4. Anisotropic displacement parameters ($Å^2 \times 10^3$) for $(Et_2O)_2Li(Br)_2In\{(C_4Ph_4)\}_2In(Br)_2Li(OEt_2)_2$

	U11	U22	U33	U23	U13	U12
In(1)	51(1)	41(1)	47(1)	-4(1)	15(1)	-2(1)
Li(1)	88(11)	109(14)	91(11)	11(10)	29(9)	16(10)
Br(1)	60(1)	75(1)	72(1)	-4(1)	13(1)	15(1)
Br(2)	72(1)	84(1)	61(1)	12(1)	25(1)	-4(1)
C(1)	56(5)	41(4)	48(4)	-7(3)	18(3)	5(3)
C(2)	80(6)	46(5)	40(4)	-4(4)	15(4)	-14(4)
C(3)	108(7)	64(6)	72(6)	-12(5)	36(5)	-27(5)
C(4)	168(12)	58(7)	106(8)	-7(6)	52(8)	-49(7)
C(5)	166(13)	40(6)	124(10)	-5(7)	17(9)	-21(8)
C(6)	123(9)	44(6)	98(8)	-12(5)	13(7)	0(6)
C(7)	96(7)	51(6)	60(5)	-4(4)	17(4)	-1(5)
C(8)	48(4)	39(4)	40(4)	-6(3)	14(3)	0(3)
C(9)	64(5)	48(4)	47(4)	4(4)	15(4)	4(4)
C(10)	74(6)	75(6)	49(5)	-16(4)	19(4)	-24(5)
C(11)	99(7)	97(7)	63(6)	-12(5)	17(5)	-45(6)
C(12)	109(8)	96(7)	56(5)	-24(5)	15(5)	-18(7)
C(13)	105(7)	97(7)	45(5)	-17(5)	22(5)	-23(6)
C(14)	69(5)	84(6)	52(5)	0(4)	23(4)	-13(5)
C(15)	55(5)	54(5)	39(4)	7(3)	11(3)	9(4)
C(16)	78(6)	53(5)	44(4)	2(4)	-1(4)	4(4)
C(17)	65(6)	88(7)	68(5)	-20(5)	4(4)	8(5)
C(18)	65(6)	139(10)	101(8)	-26(8)	-4(6)	19(7)
C(21)	83(7)	91(7)	83(7)	20(6)	-25(5)	-6(6)
C(22)	55(4)	40(4)	44(4)	10(3)	11(3)	-1(4)
C(23)	64(5)	38(5)	64(5)	5(4)	-2(4)	-5(4)
C(24)	74(6)	69(6)	84(6)	21(5)	1(5)	-16(5)
C(25)	107(9)	109(10)	137(10)	42(8)	-2(7)	-54(8)
C(28)	109(8)	43(5)	81(6)	2(4)	18(5)	10(5)
O(1)	121(6)	96(6)	175(8)	-18(5)	55(6)	-40(5)
O(2)	112(6)	188(9)	108(6)	-21(6)	47(5)	35(6)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for $(Et_2O)_2Li(Br)_2In\{(C_4Ph_4)\}_2In(Br)_2Li(OEt_2)_2$.

	x	y	z	U(eq)
H(3A)	1424	10222	2460	95
H(4A)	1534	11789	2430	129
H(5A)	950	12752	2788	135
H(6A)	277	12185	3239	109
H(7A)	175	10624	3320	83
H(10A)	1728	9875	3830	78
H(11A)	2058	10555	4677	104
H(12A)	1489	10600	5309	105
H(13A)	619	9821	5150	97
H(14A)	303	9011	4336	80
H(17A)	1804	7757	3684	91
H(18A)	2508	6992	4384	126
H(19A)	2178	6132	5005	139
H(20A)	1204	6112	5058	134
H(21A)	527	6799	4346	111
H(24A)	-728	6489	3541	94
H(25A)	-814	4838	3606	147
H(26A)	-91	3945	3370	139
H(27A)	658	4500	3090	121
H(28A)	755	6070	3002	94
H(29A)	3197	10087	2222	217
H(29B)	2742	10458	2549	217
H(30A)	2624	11191	1714	278
H(30B)	2047	10647	1740	278
H(30C)	2488	10248	1408	278
H(31A)	2924	8150	2752	199
H(31B)	3370	8978	2822	199
H(32A)	2984	8823	3580	284
H(32B)	2345	8966	3211	284
H(32C)	2805	9772	3293	284
H(33A)	1952	7435	467	215
H(33B)	1474	7574	815	215
H(34A)	1679	6067	752	325
H(34B)	1867	6389	1368	325
H(34C)	2348	6253	1024	325
H(37A)	721	2818	4483	409
H(37B)	541	2576	5045	409
H(38A)	856	4211	5076	587
H(38B)	185	4161	5095	587
H(38C)	363	4400	4541	587
H(39A)	-253	1706	4573	276
H(39B)	-115	1921	3998	276
H(40A)	-1117	1478	3875	415
H(40B)	-1087	2494	3675	415
H(40C)	-1226	2293	4251	415