Revelation of coordination patterns in C_5-cyclic lithiumorganics

Supporting Informations

*Reent Michel, Regine Herbst-Irmer, and Dietmar Stalke**

Institut für Anorganische Chemie der Georg-August-Universität Göttingen, Tammannstraße 4,

37077 Göttingen, Germany

E-mail: dstalke@chemie.uni-goettingen.de

Fax: +49-551-39-3459. Tel.: +49-551-39-3000.

Content:

Crystallographic data table	page 2
Checkcif for (η^5-Cp)Li(PMDETA) (**1**)	page 4
Checkcif for (η^5-Cp)Li(TMEDA) (**2**)	page 6
Checkcif for (η^5-Cp)Li(DME) (**3**)	page 8
Checkcif for [(η^5-Cp)$_2$Li][Li(Diglyme)$_2$] (**4**)	page 10
Checkcif for (η^2-Ind)Li(PMDETA) (**5**)	page 12
Checkcif for {(η^5-Ind)Li(DME}(μ-DME) {(η^5-Ind)Li(DME} (**6**)	page 14
Checkcif for [Ind][Li(Diglyme)$_2$] (**7**)	page 16

	1	**2**	**3**	**4**	**5**	**6**	**7**
CCDC deposition number	827709	827710	827711	827712	827713	827714	827715
empirical formula	$C_{14}H_{28}N_3Li$	$C_{11}H_{21}N_2Li$	$C_9H_{15}O_2Li$	$C_{22}H_{38}O_6Li_2$	$C_{18}H_{30}N_3Li$	$C_{30}H_{44}O_6Li_2$	$C_{21}H_{35}O_6Li$
M [g/mol]	245.33	188.24	162.15	412.40	295.39	514.53	390.43
crystal system	triclinic	orthorhombic	orthorhombic	triclinic	monoclinic	monoclinic	monoclinic
space group	$P1;^{-}$	$P2_12_12_1$	$Pna2_1$	$P1;^{-}$	$P2_1/c$	$P2_1/n$	$C2/c$
T [K]	100(2)	100(2)	100(2)	100(2)	100(2)	100(2)	100(2)
a [Å]	8.139(3)	5.831(2)	9.692(2)	8.619(2)	15.837(2)	8.881(2)	36.584(4)
b [Å]	8.843(3)	13.989(2)	13.926(2)	9.165(2)	13.004(2)	13.654(2)	7.991(2)
c [Å]	12.721(4)	29.834(2)	7.449(2)	16.009(3)	17.389(2)	12.272(2)	15.871(2)
α	71.90(2)°	90°	90°	89.43(2)°	90°	90°	90°
β	86.88(2)°	90°	90°	79.81(2)°	90.06(2)°	90.04(2)°	107.67(2)°
γ	65.43(2)°	90°	90°	81.58(2)°	90°	90°	90°
V [$Å^3$]	788.5(5)	2433.6(9)	1005.4(4)	1231.1(5)	3581.2(8)	1488.1(5)	4420.9(13)
Z	2	8	4	2	8	2	8
ρ_{calc} [Mg/m^{-3}]	1.033	1.028	1.071	1.113	1.096	1.148	1.173
μ [mm^{-1}]	0.061	0.060	0.072	0.077	0.064	0.077	0.083
F(000)	272	832	352	448	1296	556	1696

index ranges	$-9 \le h \le 9$	$-7 \le h \le 7$	$-11 \le h \le 11$	$-12 \le h \le 12$	$-19 \le h \le 19$	$-10 \le h \le 10$	$-47 \le h \le 47$
	$-10 \le k \le 10$	$-17 \le k \le 17$	$-16 \le k \le 16$	$-13 \le k \le 13$	$-16 \le k \le 16$	$-16 \le k \le 16$	$-10 \le k \le 10$
	$-15 \le l \le 15$	$-38 \le l \le 38$	$-8 \le l \le 8$	$-22 \le l \le 22$	$-21 \le l \le 21$	$-14 \le l \le 14$	$-20 \le l \le 20$
θ-min, max	1.69°, 25.01°	1.37°, 27.15°	2.56°, 25.03°	1.29°, 30.51°	1.17°, 26.05°	2.23°, 25.71°	2.34°–27.48°
max., min. transm.	1.0000, 0.9288	1.0000, 0.8954	1.0000, 0.7366	1.0000, 0.8246	1.0000, 0.7070	1.0000/0.9327	1.0000, 0.9498
g_1, g_2	0.0442, 0.1658	0.0473, 0.2552	0.0399, 0.1584	0.0432, 0.2575,	0.0453, 0.5008	0.043, 0.4414	0.0115, 5.2042
refl. collected	8541	61652	8724	39303	56824	27593	42651
unique refl.	2767	3122	966	7495	7074	2833	5083
restraints	509	802	162	0	732	0	229
parameters	273	399	146	278	526	176	339
GooF	1.032	1.054	1.077	1.053	1.029	1.036	1.223
$R1$, w$R2$ [$I > 2\sigma(I)$]	0.0380, 0.0934	0.0281, 0.0771	0.0318, 0.0742	0.0353, 0.0924	0.0307, 0.0815	0.0372, 0.0955	0.0483, 0.0976
$R1$, $wR2$ (all data)	0.0496, 0.1001	0.0304, 0.0785	0.0374, 0.0765	0.0403, 0.0963	0.0330, 0.0834	0.0424, 0.0993	0.0492, 0.0979
largest diff. peak, hole (e $Å^{-3}$)	0.147, –0.142	0.200, –0.175	0.116, –0.104	0.378, –0.214	0.199, –0.165	0.222, –0.159	0.180, –0.229
Extinction coeff.	0.017(4)	-	-	-	-	0.0037(11)	0.00197(13)

The absolute structure of **2** and **3** could not be determined by refining the Flack X parameter (Flack, H. D., *Acta Crystallogr., Sect. A.* **1983,** *39*, 876.) due to the absence of a heavy scatterer. Therefore Friedel pairs were merged.

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 1

No syntax errors found. CIF dictionary Interpreting this report

Datablock: 1

```
Bond precision:  C-C = 0.0020 A          Wavelength=0.71073

Cell:           a=8.139(3)        b=8.843(3)       c=12.721(4)
                alpha=71.90(2)    beta=86.88(2)    gamma=65.43(2)
Temperature:    100 K

                Calculated                Reported
Volume          788.5(5)                  788.5(5)
Space group     P -1                      P-1
Hall group      -P 1                      ?
Moiety formula  C14 H28 Li N3             ?
Sum formula     C14 H28 Li N3             C14 H28 Li N3
Mr              245.33                    245.33
Dx,g cm-3       1.033                     1.033
Z               2                         2
Mu (mm-1)       0.061                     0.061
F000            272.0                     272.0
F000'           272.07
h,k,lmax        9,10,15                   9,10,15
Nref            2782                      2767
Tmin,Tmax       0.993,0.994               0.929,1.000
Tmin'           0.991

Correction method= MULTI-SCAN

Data completeness= 0.995          Theta(max)= 25.010

R(reflections)= 0.0380( 2246)     wR2(reflections)= 0.1001( 2767)

S = 1.032                  Npar= 273
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

```
PLAT230_ALERT_2_C Hirshfeld Test Diff for    C1     --  C2      ..       5.18 su
PLAT366_ALERT_2_C Short? C(sp?)-C(sp?) Bond  C1     -   C2     ...       1.39 Ang.
PLAT366_ALERT_2_C Short? C(sp?)-C(sp?) Bond  C2     -   C3     ...       1.39 Ang.
PLAT911_ALERT_3_C Missing # FCF Refl Between THmin & STh/L=  0.595         14
```

```
PLAT922_ALERT_1_C wR2 * 100.0 in the CIF and FCF Differ by .......       0.20
PLAT923_ALERT_1_C S    values in the CIF and FCF Differ by .......       0.02
PLAT927_ALERT_1_C Reported and Calculated  wR2 * 100.0 Differ by .       0.18
```

Alert level G

```
PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite         24
PLAT003_ALERT_2_G Number of Uiso or Uij Restrained Atom Sites ....         24
PLAT154_ALERT_1_G The su's on the Cell Angles are Equal  (x 10000)       2000 Deg.
PLAT242_ALERT_2_G Check Low       Ueq as Compared to Neighbors for         N2
PLAT301_ALERT_3_G Note: Main Residue  Disorder ...................         67 Perc.
PLAT432_ALERT_2_G Short Inter X...Y Contact  C7'    ..  C13'    ..       3.12 Ang.
PLAT720_ALERT_4_G Number of Unusual/Non-Standard Labels ..........         10
PLAT793_ALERT_4_G The Model has Chirality at N2      (Verify) ....          S
PLAT793_ALERT_4_G The Model has Chirality at N2'     (Verify) ....          R
PLAT811_ALERT_5_G No ADDSYM Analysis: Too Many Excluded Atoms ....          !
PLAT860_ALERT_3_G Note: Number of Least-Squares Restraints .......        509
PLAT909_ALERT_3_G Percentage of Observed Data at Theta(Max) still          59 Perc.
```

```
   0 ALERT level A = Most likely a serious problem - resolve or explain
   0 ALERT level B = A potentially serious problem, consider carefully
   7 ALERT level C = Check. Ensure it is not caused by an omission or oversight
  12 ALERT level G = General information/check it is not something unexpected

   4 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   7 ALERT type 2 Indicator that the structure model may be wrong or deficient
   4 ALERT type 3 Indicator that the structure quality may be low
   3 ALERT type 4 Improvement, methodology, query or suggestion
   1 ALERT type 5 Informative message, check
```

---

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 2

No syntax errors found. CIF dictionary Interpreting this report

Datablock: 2

```
Bond precision:  C-C = 0.0017 A          Wavelength=0.71073

Cell:              a=5.831(2)      b=13.989(2)      c=29.834(2)
                   alpha=90        beta=90          gamma=90
Temperature:       100 K

                Calculated                Reported
Volume          2433.6(9)                 2433.6(9)
Space group     P 21 21 21                P2(1)2(1)2(
Hall group      P 2ac 2ab                 ?
Moiety formula  C11 H21 Li N2             ?
Sum formula     C11 H21 Li N2             C11 H21 Li N2
Mr              188.24                    188.24
Dx,g cm-3       1.028                     1.028
Z               8                         8
Mu (mm-1)       0.060                     0.060
F000            832.0                     832.0
F000'           832.22
h,k,lmax        7,17,38                   7,17,38
Nref            3125[ 5410]               3122
Tmin,Tmax       0.993,0.994               0.895,1.000
Tmin'           0.988

Correction method= MULTI-SCAN

Data completeness= 1.00/0.58      Theta(max)= 27.150

R(reflections)= 0.0281( 2944)     wR2(reflections)= 0.0785( 3122)

S = 1.054                  Npar= 399
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

```
PLAT089_ALERT_3_C Poor Data / Parameter Ratio (Zmax .LT. 18) .....       7.82
PLAT912_ALERT_4_C Missing # of FCF Reflections Above STh/L=  0.600          4
```

Alert level G

```
REFLT03_ALERT_4_G Please check that the estimate of the number of Friedel pairs is
            correct. If it is not, please give the correct count in the
            _publ_section_exptl_refinement section of the submitted CIF.
           From the CIF: _diffrn_reflns_theta_max          27.15
           From the CIF: _reflns_number_total              3122
           Count of symmetry unique reflns         3125
           Completeness (_total/calc)            99.90%
           TEST3: Check Friedels for noncentro structure
           Estimate of Friedel pairs measured         0
           Fraction of Friedel pairs measured     0.000
           Are heavy atom types Z>Si present         no
PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite         34
PLAT003_ALERT_2_G Number of Uiso or Uij Restrained Atom Sites ....         32
PLAT153_ALERT_1_G The su's on the Cell Axes   are Equal (x 100000)        200 Ang.
PLAT242_ALERT_2_G Check Low       Ueq as Compared to Neighbors for        N3'
PLAT301_ALERT_3_G Note: Main Residue  Disorder ...................         57 Perc.
PLAT710_ALERT_4_G Delete 1-2-3 or 2-3-4 Linear Torsion Angle ... #        270
            C21 -N4  -LI2 -C14    95.80  0.80   1.555   1.555   1.555   1.555
PLAT710_ALERT_4_G Delete 1-2-3 or 2-3-4 Linear Torsion Angle ... #        271
            C22 -N4  -LI2 -C14   -25.50  0.90   1.555   1.555   1.555   1.555
PLAT710_ALERT_4_G Delete 1-2-3 or 2-3-4 Linear Torsion Angle ... #        272
            C20 -N4  -LI2 -C14  -147.40  0.80   1.555   1.555   1.555   1.555
PLAT710_ALERT_4_G Delete 1-2-3 or 2-3-4 Linear Torsion Angle ... #        379
            C15 -C14 -LI2 -N4    -60.70  0.80   1.555   1.555   1.555   1.555
PLAT710_ALERT_4_G Delete 1-2-3 or 2-3-4 Linear Torsion Angle ... #        380
            C13 -C14 -LI2 -N4     56.50  0.80   1.555   1.555   1.555   1.555
PLAT720_ALERT_4_G Number of Unusual/Non-Standard Labels ..........         10
PLAT811_ALERT_5_G No ADDSYM Analysis: Too Many Excluded Atoms ....          !
PLAT860_ALERT_3_G Note: Number of Least-Squares Restraints .......        802
```

```
   0 ALERT level A = Most likely a serious problem - resolve or explain
   0 ALERT level B = A potentially serious problem, consider carefully
   2 ALERT level C = Check. Ensure it is not caused by an omission or oversight
  14 ALERT level G = General information/check it is not something unexpected

   1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   3 ALERT type 2 Indicator that the structure model may be wrong or deficient
   3 ALERT type 3 Indicator that the structure quality may be low
   8 ALERT type 4 Improvement, methodology, query or suggestion
   1 ALERT type 5 Informative message, check
```

---

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 3

No syntax errors found. CIF dictionary Interpreting this report

Datablock: 3

```
Bond precision:  C-C = 0.0034 A          Wavelength=0.71073

Cell:              a=9.692(2)      b=13.926(2)      c=7.449(2)
                   alpha=90        beta=90          gamma=90
Temperature:       100 K

                Calculated                Reported
Volume          1005.4(4)                 1005.4(4)
Space group     P n a 21                  Pna2(1)
Hall group      P 2c -2n                  ?
Moiety formula  C9 H15 Li O2              ?
Sum formula     C9 H15 Li O2              C9 H15 Li O2
Mr              162.15                    162.15
Dx,g cm-3       1.071                     1.071
Z               4                         4
Mu (mm-1)       0.072                     0.072
F000            352.0                     352.0
F000'           352.16
h,k,lmax        11,16,8                   11,16,8
Nref            966[ 1781]                966
Tmin,Tmax       0.991,0.993               0.737,1.000
Tmin'           0.989

Correction method= MULTI-SCAN

Data completeness= 1.00/0.54      Theta(max)= 25.030

R(reflections)= 0.0318( 872)      wR2(reflections)= 0.0765( 966)

S = 1.077                  Npar= 146
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level B

PLAT213_ALERT_2_B Atom C6' has ADP max/min Ratio ..... 4.60 prola

---

Alert level C

```
PLAT089_ALERT_3_C Poor Data / Parameter Ratio (Zmax .LT. 18) .....       6.62
PLAT230_ALERT_2_C Hirshfeld Test Diff for    C4     --  C5      ..       5.42 su
PLAT250_ALERT_2_C Large U3/U1 Ratio for Average U(i,j) Tensor ....       2.12
PLAT366_ALERT_2_C Short? C(sp?)-C(sp?) Bond  C1     -   C2     ...       1.38 Ang.
PLAT366_ALERT_2_C Short? C(sp?)-C(sp?) Bond  C2     -   C3     ...       1.38 Ang.
PLAT366_ALERT_2_C Short? C(sp?)-C(sp?) Bond  C3     -   C4     ...       1.37 Ang.
PLAT366_ALERT_2_C Short? C(sp?)-C(sp?) Bond  C4     -   C5     ...       1.39 Ang.
```

Alert level G

```
REFLT03_ALERT_4_G Please check that the estimate of the number of Friedel pairs is
            correct. If it is not, please give the correct count in the
            _publ_section_exptl_refinement section of the submitted CIF.
           From the CIF: _diffrn_reflns_theta_max          25.03
           From the CIF: _reflns_number_total               966
           Count of symmetry unique reflns          966
           Completeness (_total/calc)            100.00%
           TEST3: Check Friedels for noncentro structure
           Estimate of Friedel pairs measured         0
           Fraction of Friedel pairs measured     0.000
           Are heavy atom types Z>Si present         no
PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite         11
PLAT003_ALERT_2_G Number of Uiso or Uij Restrained Atom Sites ....         10
PLAT153_ALERT_1_G The su's on the Cell Axes   are Equal (x 100000)        200 Ang.
PLAT301_ALERT_3_G Note: Main Residue  Disorder ...................         33 Perc.
PLAT720_ALERT_4_G Number of Unusual/Non-Standard Labels ..........         10
PLAT779_ALERT_4_G Suspect or Irrelevant (Bond) Angle in CIF .... #         85
              O2   -C8   -O2'     1.555   1.555   1.555             20.90 Deg.
PLAT779_ALERT_4_G Suspect or Irrelevant (Bond) Angle in CIF .... #         90
              C7'  -C8   -C7      1.555   1.555   1.555             27.10 Deg.
PLAT779_ALERT_4_G Suspect or Irrelevant (Bond) Angle in CIF .... #        104
              H8A  -C8   -H8'1    1.555   1.555   1.555             41.70 Deg.
PLAT779_ALERT_4_G Suspect or Irrelevant (Bond) Angle in CIF .... #        111
              H8B  -C8   -H8'2    1.555   1.555   1.555             41.40 Deg.
PLAT779_ALERT_4_G Suspect or Irrelevant (Bond) Angle in CIF .... #        113
              O2   -C9   -O2'     1.555   1.555   1.555             20.90 Deg.
PLAT779_ALERT_4_G Suspect or Irrelevant (Bond) Angle in CIF .... #        125
              H9A  -C9   -H9'1    1.555   1.555   1.555             23.60 Deg.
PLAT779_ALERT_4_G Suspect or Irrelevant (Bond) Angle in CIF .... #        131
              H9B  -C9   -H9'2    1.555   1.555   1.555             39.00 Deg.
PLAT779_ALERT_4_G Suspect or Irrelevant (Bond) Angle in CIF .... #        138
              H9C  -C9   -H9'3    1.555   1.555   1.555             38.90 Deg.
PLAT811_ALERT_5_G No ADDSYM Analysis: Too Many Excluded Atoms ....          !
PLAT860_ALERT_3_G Note: Number of Least-Squares Restraints .......        162
PLAT909_ALERT_3_G Percentage of Observed Data at Theta(Max) still          73 Perc.
```

```
   0 ALERT level A = Most likely a serious problem - resolve or explain
   1 ALERT level B = A potentially serious problem, consider carefully
   7 ALERT level C = Check. Ensure it is not caused by an omission or oversight
  17 ALERT level G = General information/check it is not something unexpected

   1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   9 ALERT type 2 Indicator that the structure model may be wrong or deficient
   4 ALERT type 3 Indicator that the structure quality may be low
  10 ALERT type 4 Improvement, methodology, query or suggestion
   1 ALERT type 5 Informative message, check
```

Comment to the ALERT level b:

The occupancy of atom C6' is under 10 %.

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 4

No syntax errors found. CIF dictionary Interpreting this report

Datablock: 4

```
Bond precision:  C-C = 0.0013 A          Wavelength=0.71073

Cell:           a=8.619(2)        b=9.165(2)       c=16.009(3)
                alpha=89.43(2)    beta=79.81(2)    gamma=81.58(2)
Temperature:    100 K

                Calculated                Reported
Volume          1231.1(5)                 1231.1(5)
Space group     P -1                      P-1
Hall group      -P 1                      ?
Moiety formula  C12 H28 Li O6, C10 H10 Li ?
Sum formula     C22 H38 Li2 O6            C22 H38 Li2 O6
Mr              412.40                    412.40
Dx,g cm-3       1.112                     1.113
Z               2                         2
Mu (mm-1)       0.077                     0.077
F000            448.0                     448.0
F000'           448.22
h,k,lmax        12,13,22                  12,13,22
Nref            7520                      7495
Tmin,Tmax       0.989,0.994               0.925,1.000
Tmin'           0.988

Correction method= MULTI-SCAN

Data completeness= 0.997          Theta(max)= 30.510

R(reflections)= 0.0353( 6573)     wR2(reflections)= 0.0963( 7495)

S = 1.053                  Npar= 278
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

```
PLAT911_ALERT_3_C Missing # FCF Refl Between THmin & STh/L=  0.600          7
PLAT912_ALERT_4_C Missing # of FCF Reflections Above STh/L=  0.600         21
```

Alert level G

```
PLAT154_ALERT_1_G The su's on the Cell Angles are Equal  (x 10000)       2000 Deg.
PLAT764_ALERT_4_G Overcomplete CIF Bond List Detected (Rep/Expd) .       1.24 Ratio
```

```
   0 ALERT level A = Most likely a serious problem - resolve or explain
   0 ALERT level B = A potentially serious problem, consider carefully
   2 ALERT level C = Check. Ensure it is not caused by an omission or oversight
   2 ALERT level G = General information/check it is not something unexpected

   1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   0 ALERT type 2 Indicator that the structure model may be wrong or deficient
   1 ALERT type 3 Indicator that the structure quality may be low
   2 ALERT type 4 Improvement, methodology, query or suggestion
   0 ALERT type 5 Informative message, check
```

---

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 5

No syntax errors found. CIF dictionary Interpreting this report

Datablock: 5

```
Bond precision:  C-C = 0.0019 A          Wavelength=0.71073

Cell:            a=15.837(2)     b=13.004(2)        c=17.389(2)
                 alpha=90        beta=90.06(2)      gamma=90
Temperature:     100 K

                Calculated                Reported
Volume          3581.2(8)                 3581.2(8)
Space group     P 21/c                    P2(1)/c
Hall group      -P 2ybc                   ?
Moiety formula  C18 H30 Li N3             ?
Sum formula     C18 H30 Li N3             C18 H30 Li N3
Mr              295.39                    295.39
Dx,g cm-3       1.096                     1.096
Z               8                         8
Mu (mm-1)       0.064                     0.064
F000            1296.0                    1296.0
F000'           1296.36
h,k,lmax        19,16,21                  19,16,21
Nref            7077                      7074
Tmin,Tmax       0.987,0.987               0.707,1.000
Tmin'           0.987

Correction method= MULTI-SCAN

Data completeness= 1.000          Theta(max)= 26.050

R(reflections)= 0.0307( 6706)     wR2(reflections)= 0.0834( 7074)

S = 1.029                  Npar= 526
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level B

PLAT413_ALERT_2_B Short Inter XH3 .. XHn H32C .. H12D .. 2.07 Ang.

Alert level C

```
PLAT230_ALERT_2_C Hirshfeld Test Diff for    C4     --  C5      ..       5.90 su
PLAT366_ALERT_2_C Short? C(sp?)-C(sp?) Bond  C1     -   C2     ...       1.38 Ang.
PLAT366_ALERT_2_C Short? C(sp?)-C(sp?) Bond  C19    -   C20    ...       1.39 Ang.
PLAT911_ALERT_3_C Missing # FCF Refl Between THmin & STh/L=  0.600          2
PLAT912_ALERT_4_C Missing # of FCF Reflections Above STh/L=  0.600          3
```

Alert level G

```
PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite         39
PLAT003_ALERT_2_G Number of Uiso or Uij Restrained Atom Sites ....         26
PLAT153_ALERT_1_G The su's on the Cell Axes   are Equal (x 100000)        200 Ang.
PLAT301_ALERT_3_G Note: Main Residue  Disorder ...................         30 Perc.
PLAT779_ALERT_4_G Suspect or Irrelevant (Bond) Angle in CIF .... #        227
              LI1  -C1   -LI1'    1.555   1.555   1.555             10.10 Deg.
PLAT779_ALERT_4_G Suspect or Irrelevant (Bond) Angle in CIF .... #        261
              LI1  -C9   -LI1'    1.555   1.555   1.555              8.30 Deg.
PLAT793_ALERT_4_G The Model has Chirality at N2      (Verify) ....          S
PLAT793_ALERT_4_G The Model has Chirality at N5      (Verify) ....          S
PLAT811_ALERT_5_G No ADDSYM Analysis: Too Many Excluded Atoms ....          !
PLAT860_ALERT_3_G Note: Number of Least-Squares Restraints .......        732
PLAT870_ALERT_4_G ALERTS Related to Twinning Effects Suppressed ..          !
PLAT931_ALERT_5_G Check Twin Law ( 1 0  0)[       ] Estimated BASF       0.41
```

```
   0 ALERT level A = Most likely a serious problem - resolve or explain
   1 ALERT level B = A potentially serious problem, consider carefully
   5 ALERT level C = Check. Ensure it is not caused by an omission or oversight
  12 ALERT level G = General information/check it is not something unexpected

   1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   6 ALERT type 2 Indicator that the structure model may be wrong or deficient
   3 ALERT type 3 Indicator that the structure quality may be low
   6 ALERT type 4 Improvement, methodology, query or suggestion
   2 ALERT type 5 Informative message, check
```

Comment to the ALERT level b:

The occupancy of atom H12D is under 10 %. H32C is a methyl hydrogen atom. There could well be a second position for the methyl group without short distances to H12D.

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 6

No syntax errors found. CIF dictionary Interpreting this report

Datablock: 6

```
Bond precision:  C-C = 0.0020 A          Wavelength=0.71073

Cell:             a=8.881(2)     b=13.654(2)        c=12.272(2)
                  alpha=90       beta=90.04(2)      gamma=90
Temperature:      100 K

                Calculated                Reported
Volume          1488.1(5)                 1488.1(5)
Space group     P 21/n                    P2(1)/n
Hall group      -P 2yn                    ?
Moiety formula  C30 H44 Li2 O6            ?
Sum formula     C30 H44 Li2 O6            C30 H44 Li2 O6
Mr              514.53                    514.53
Dx,g cm-3       1.148                     1.148
Z               2                         2
Mu (mm-1)       0.077                     0.077
F000            556.0                     556.0
F000'           556.26
h,k,lmax        10,16,14                  10,16,14
Nref            2834                      2833
Tmin,Tmax       0.964,0.970               0.933,1.000
Tmin'           0.962

Correction method= MULTI-SCAN

Data completeness= 1.000          Theta(max)= 25.710

R(reflections)= 0.0372( 2504)     wR2(reflections)= 0.0993( 2833)

S = 1.036                  Npar= 176
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level B

PLAT230_ALERT_2_B Hirshfeld Test Diff for C5 -- C6 .. 14.07 su

---

Alert level C

```
PLAT366_ALERT_2_C Short? C(sp?)-C(sp?) Bond  C1     -   C2     ...       1.39 Ang.
PLAT366_ALERT_2_C Short? C(sp?)-C(sp?) Bond  C1     -   C9     ...       1.38 Ang.
PLAT911_ALERT_3_C Missing # FCF Refl Between THmin & STh/L=  0.600          2
PLAT922_ALERT_1_C wR2 * 100.0 in the CIF and FCF Differ by .......       0.15
PLAT923_ALERT_1_C S    values in the CIF and FCF Differ by .......       0.02
PLAT927_ALERT_1_C Reported and Calculated  wR2 * 100.0 Differ by .       0.14
```

Alert level G

```
PLAT128_ALERT_4_G Alternate Setting of Space-group P21/c   .......      P21/n
PLAT153_ALERT_1_G The su's on the Cell Axes   are Equal (x 100000)        200 Ang.
```

```
   0 ALERT level A = Most likely a serious problem - resolve or explain
   1 ALERT level B = A potentially serious problem, consider carefully
   6 ALERT level C = Check. Ensure it is not caused by an omission or oversight
   2 ALERT level G = General information/check it is not something unexpected

   4 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   3 ALERT type 2 Indicator that the structure model may be wrong or deficient
   1 ALERT type 3 Indicator that the structure quality may be low
   1 ALERT type 4 Improvement, methodology, query or suggestion
   0 ALERT type 5 Informative message, check
```

Comment to the ALERT level b:

The whole indenyl anion shows elongated ellipsoids indicating movement in the plane.

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 7

No syntax errors found. CIF dictionary Interpreting this report

Datablock: 7

```
Bond precision:  C-C = 0.0020 A          Wavelength=0.71073

Cell:            a=36.584(4)     b=7.991(2)          c=15.871(2)
                 alpha=90        beta=107.67(2)      gamma=90
Temperature:     100 K

                Calculated                Reported
Volume          4420.9(14)                4420.9(13)
Space group     C 2/c                     C2/c
Hall group      -C 2yc                    ?
Moiety formula  C12 H28 Li O6, C9 H7      ?
Sum formula     C21 H35 Li O6             C21 H35 Li O6
Mr              390.43                    390.43
Dx,g cm-3       1.173                     1.173
Z               8                         8
Mu (mm-1)       0.083                     0.083
F000            1696.0                    1696.0
F000'           1696.88
h,k,lmax        47,10,20                  47,10,20
Nref            5088                      5083
Tmin,Tmax       0.980,0.984               0.950,1.000
Tmin'           0.967

Correction method= MULTI-SCAN

Data completeness= 0.999          Theta(max)= 27.480

R(reflections)= 0.0483( 4939)     wR2(reflections)= 0.0979( 5083)

S = 1.223                  Npar= 339
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

```
PLAT250_ALERT_2_C Large U3/U1 Ratio for Average U(i,j) Tensor ....      2.18
PLAT910_ALERT_3_C Missing # of FCF Reflections Below Th(Min) .....         1
PLAT911_ALERT_3_C Missing # FCF Refl Between THmin & STh/L=  0.600         5
PLAT913_ALERT_3_C Missing # of Very Strong Reflections in FCF ....         1
```

Alert level G

```
PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite         18
PLAT003_ALERT_2_G Number of Uiso or Uij Restrained Atom Sites ....         18
PLAT083_ALERT_2_G SHELXL Second Parameter in WGHT Unusually Large.       5.20
PLAT302_ALERT_4_G Note: Anion/Solvent Disorder ...................        100 Perc.
PLAT811_ALERT_5_G No ADDSYM Analysis: Too Many Excluded Atoms ....          !
PLAT860_ALERT_3_G Note: Number of Least-Squares Restraints .......        229
```

```
   0 ALERT level A = Most likely a serious problem - resolve or explain
   0 ALERT level B = A potentially serious problem, consider carefully
   4 ALERT level C = Check. Ensure it is not caused by an omission or oversight
   6 ALERT level G = General information/check it is not something unexpected

   0 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   4 ALERT type 2 Indicator that the structure model may be wrong or deficient
   4 ALERT type 3 Indicator that the structure quality may be low
   1 ALERT type 4 Improvement, methodology, query or suggestion
   1 ALERT type 5 Informative message, check
```