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An unusual case of tetartohedral twinning: the low-temperature phase of bis[μ_3-1,3-bis(*tert*-butyl-azanidyl)-1,1,3,3-tetramethyldisiloxane]tetra-lithium(I)

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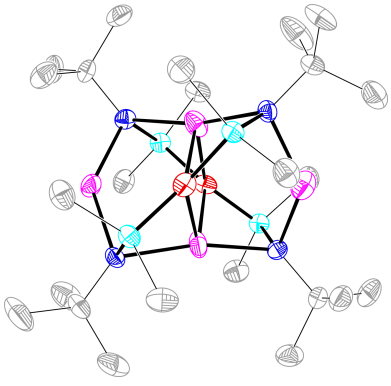
The low-temperature phase of the title compound, $[Li_4(C_{12}H_{30}N_2OSi_2)_2]$, crystallizes in the space group $P2_1$ with the pseudo-tetragonal cell constants a = 10.761 (2), b = 13.030 (3), c = 13.035 (3) Å, β = 90.05 (2)° and Z = 2. The special values of the cell constants (two equal axes, monoclinic $\beta \approx 90°$) each lead to twinning, so that the usual *SHELXL* TWIN command cannot be used; instead an intensity file in HKLF5 format was constructed, involving sets of eight reflections, of equal intensity, with indices connected by the twinning operations and by inversion; thus seven scale factors (defined by the BASF command) were refined. The final $wR2$ value is 0.067, with $R1$ = 0.026. In contrast to the previously known forms, both tetragonal, the Li atoms are not disordered; two of them are two-coordinate (bonded to two N atoms) and two are four-coordinate (bonded to two N and two O atoms). Some personal recollections of twin treatment over the last 40 years are presented.

1. Introduction

George Sheldrick will be remembered in particular for his powerful *SHELX* suite of programs for solving and refining X-ray crystal structures. In this article/memorial, we concentrate on one special aspect, the simplified and user-friendly handling of data from twinned crystals, introduced in *SHELXL-93* (Herbst-Irmer & Sheldrick, 1998). There had already been programs for twin treatment (*e.g. CRYSTALS*; Betteridge *et al.*, 2003; Watkin, 2026), but this version of *SHELXL* demonstrated that this treatment could also be applied to routine structures. Apart from a general method suitable for all kinds of twins, George also designed a more straightforward method, which needs only two extra commands in the instruction file: TWIN to define the twin law and BASF for the fractional contribution of the minor component (which is then refined). This is only possible if all components of the twin matrix are integers and if there is only one twin operator (*i.e.* for twinning by merohedry or pseudomerohedry), although additional twinning by inversion can also be treated in this manner; the default twin law is [$\bar{1}$00/0$\bar{1}$0/00$\bar{1}$], corresponding to inversion twinning alone. In the general approach, the information about the twinning, *e.g.* which reflections of the main domain overlap with which reflections of the minor domain(s), is incorporated in a special-format HKL file, the so-called HKLF5 file. Usually, this type of HKL file is used for non-merohedric twins. The overlap information is acquired during data processing of the diffractometer data (with routines internal to the diffractometer programs taking



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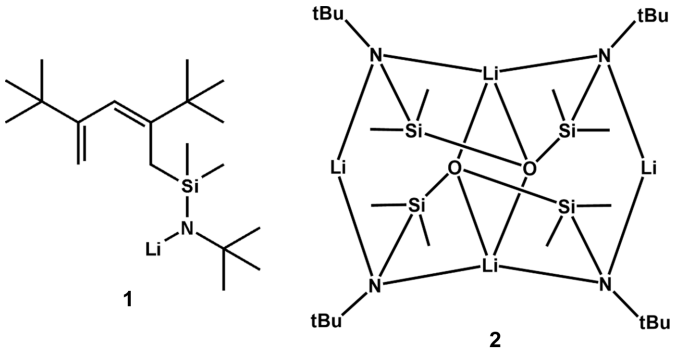
account of the experimental setup, for example, mosaicity, detector distance, goniostat position and rotation increment, without documenting this information for further programs). Additionally, a special program is needed that can manage the scaling and absorption correction for this type of data; George wrote the program *TWINABS* (Sevvana *et al.*, 2019), which performs the same function as *SADABS* (Krause *et al.*, 2015), used for single-crystal data, and is additionally able to merge the data in a valid manner and to detwin the data of such non-merohedric twins for structure solution. (It should be noted that some of George's auxiliary programs are specific to, and only obtainable from, a particular diffractometer firm; other brands have their own programs that perform similar functions.)



Scheme 1

Here we describe the solution and refinement of a structure from a tetartohedrally twinned crystal, where more than one twin law is necessary for an appropriate description of the twinning.

Crystals of a compound believed to be the lithium silylamide **1** (see Scheme 1) were investigated at 100 K [for investigations of other silylamide-based complexes by Walter and co-workers, see *e.g.* a CSD Communication (Jones *et al.*, 2021), reporting a structure determined at roughly the same time as that of the title compound, but deposited several years later, or a more recent publication (Münster *et al.*, 2025)] (see Scheme 1). The diffraction pattern was, apparently successfully, indexed in terms of an effectively tetragonal cell, with $a \approx b \approx 13.032$ (3), $c \approx 10.761$ (2) Å. The cell, if correct, seemed impossibly small for the suggested structure, but the data were collected anyway. Standard solution attempts with *SHELXT* (Sheldrick, 2015*a*) failed in Laue classes 4/*m* and 4/*mmm*. No systematic absences were detected by automatic procedures, but continued efforts to solve the structure in all possible tetragonal space groups (those lacking systematic absences) proved unsuccessful. The intensity distribution indicated probable twinning, with a very low $<|E^2 - 1|>$ value of 0.427 (for details, see the supporting information) (Herbst-Irmer & Sheldrick, 1998; Herbst-Irmer, 2016).

2. Experimental

Data were recorded at 101 K on an Agilent Xcalibur A diffractometer using mirror-focussed Cu $K\alpha$ radiation and the control program *CrysAlis PRO* (Agilent, 2012). Absorption corrections, using the *SCALE3 ABSPACK* scaling algorithm of *CrysAlis PRO*, were based on multiscans. The structure was solved (see below) with *SHELXS* (Sheldrick, 2008) and refined with *SHELXL* (Sheldrick, 2015*b*) within the GUI *ShelXle* (Hübschle *et al.*, 2011). Further details are given in Table S3.

3. Analysis of twinning

Use of the program *XPREP* (Sheldrick, 2015*c*) showed an interesting pattern of $R_{\rm int}$ values for cells of steadily decreasing symmetry. The value of 0.021 for a monoclinic cell with $a = 10.761$ (2), $b = 13.030$ (3), $c = 13.035$ (3) Å and $\beta = 90.05$ (2)° was significantly lower than the values of 0.057–0.090 for other symmetries (tetragonal, orthorhombic, other monoclinic settings) (for details, see the supporting information). It thus seemed worthwhile to investigate this monoclinic setting further. However, the lack of systematic absences seemed to indicate three possible space groups, namely *P*2, *Pm* or *P*2/*m* (the first two extremely rare), yet no structure solution could be found in these space groups, neither with *SHELXT*, *SHELXS* nor *SHELXD* (Sheldrick, 2008). However, *SHELXT* did propose space group $P2_1$, but without finding a sensible structure.

The solution to this problem relies on a correct interpretation of the twinning. First, the $R_{\rm int}$ values in Laue classes 4/*m* and 4/*mmm* are identical: this could be connected with twinning of the type well known for truly tetragonal structures in 4/*m* as twinning by merohedry [*e.g.* Taouss *et al.* (2013), see below]. Secondly, the (near) equality of the *b* and *c* axes allows further twinning by a 180° rotation [also well known in the equivalent twinning by pseudomerohedry of monoclinic structures with $a \approx c$, *e.g.* Armstrong *et al.* (1993)] that exchanges these axes, thus emulating a fourfold axis. If one now supposes that the true space group is the common space group $P2_1$, the systematic absences ($0k0$ absent for k odd) along the b^* axis of one twin component would be masked by the lack of absences along the c^* axis of the second component, so there would appear to be no absences.

The structure was then solved with *SHELXS* and also with *SHELXD* (for details, see the supporting information), showing that it is not the expected compound **1**, but the oxygen-containing compound bis[μ_3-1,3-bis(*tert*-butylazanidyl)-1,1,3,3-tetramethyldisiloxane]tetralithium(I), **2**, which presumably arose by reaction with small amounts of adventitious water. The information about the twinning was not used for the structure solution. To refine the structure, the presence of tetartohedral twinning precludes the use of the simple procedure 'twin matrix plus scale factor'. Here three twin matrices would be needed: [$\bar{1}00/001/010$] (twinning by pseudomerohedry of monoclinic structures with $b \approx c$), [$100/0\bar{1}0/00\bar{1}$] (twinning by pseudomerohedry of a monoclinic structure with $\beta \approx 90°$) and the combination of both: [$\bar{1}00/00\bar{1}/0\bar{1}0$]. Therefore, the HKL file was converted to HKLF5 format (using a locally written program) by generating further reflections $-h, l, k$; $h, -k, -l$ and $-h, -l, -k$ for each parent

Table 1
Comparison between the two refinements with and without twinning by inversion.

Refinement	Without twinning by inversion	With twinning by inversion
Data	7547	7547
Parameters	366	370
$R1$ [$I > 2\sigma(I)$]	0.0264	0.0257
$wR2$ (all data)	0.0689	0.0671
k_1 (h, k, l)	0.3533	0.174
k_2 ($-h$, l, k)	0.2526 (12)	0.118 (17)
k_3 ($-h$, $-l$, $-k$)	0.2481 (12)	0.148 (17)
k_4 (h, $-k$, $-l$)	0.1460 (12)	0.074 (15)
k_5 ($-h$, $-k$, $-l$)		0.18 (2)
k_6 (h, $-l$, $-k$)		0.134 (17)
k_7 (h, l, k)		0.100 (17)
k_8 ($-h$, k, l)		0.072 (15)
Bond precision C—C (Å)	0.0078	0.0075

reflection *hkl*, whereby all reflections of the set of four were assigned equal intensities to that of the parent reflection, but different batch scale factors. This was also achievable with *TWINROTMAT* in *PLATON* (Spek, 2020) (see the supporting information). *TWINABS* would not be an option for this kind of twinning, because there is a nearly exact overlap of the reflections of the different domains and only one orientation matrix was used for the integration. The three fractional contributions of the additional domains refined to 0.2526 (12), 0.2481 (12) and 0.1460 (12), leading to a fractional contribution of the main domain of 0.3533. Since the structure is non-centrosymmetric, the Flack parameter was calculated as 0.49 (3) by the classical fit to all intensities (Flack, 1983) and 0.183 (9) from 3568 selected quotients (Parsons *et al.*, 2013). Accordingly, *SHELXL* (Sheldrick, 2015*b*) produced the warning message '**Possible inversion twin or centrosymmetric space group**' and *checkCIF* (https://checkcif.iucr.org/) showed a similar B-alert. Here this would mean *additional* twinning by inversion, and the HKL file thus needs to be expanded by the addition of the reflections $-h$, $-k$, $-l$; h, $-l$, $-k$; $-h$, k, l and h, l, k. Table 1 compares the two refinements. The results are very similar. But the anomalous dispersion of silicon is limited, even for copper radiation, so that the Friedel differences will be small and the improvement by taking additional twinning by inversion into account is marginal. Therefore, both CIF files are made available (for details see the supporting information).

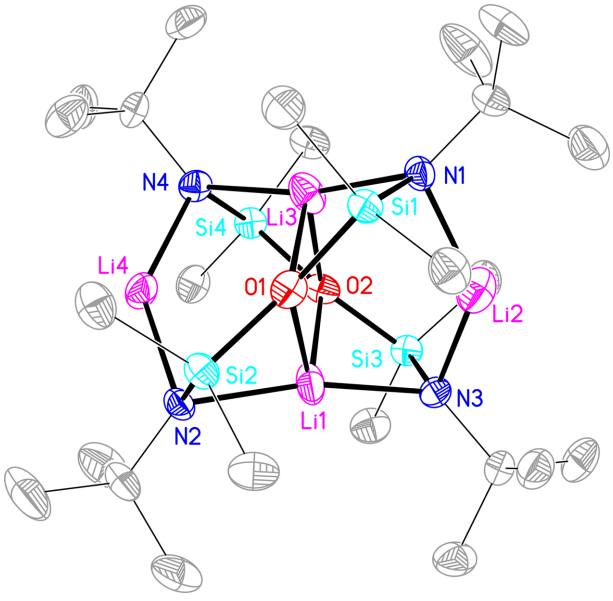


Figure 1
The molecule of compound **2** in the crystal, with ellipsoids shown at the 50% probability level. H atoms have been omitted for clarity.

Table 2
Selected bond lengths (Å) of the three phases of **2**.

	α-phase (293 K)[a]	β-phase (230 K)[b]	γ-phase (101 K)[c]
Si—O	1.662 (2)	1.656 (2)	1.667 (4)–1.684 (4)
Si—N	1.669 (4)	1.664 (4)	1.684 (4)–1.692 (4)
Si—C	1.873 (5)	1.863 (7)	1.860 (5)–1.902 (5)
N—Li **II**	1.917 (3)	1.92 (2)	1.945 (10), 1.955 (9)
			1.981 (10), 1.989 (10)
N—Li **IV**	2.019 (6)	2.01 (2)	1.974 (11), 1.996 (11)
			2.022 (11), 2.027 (12)
O1—Li **IV**	2.37 (2)	2.36 (2)	2.282 (11), 2.286 (10)
O2—Li **IV**			2.345 (11), 2.359 (10)
Li—Li	2.75 (2)	2.92 (5)	2.788 (11)–2.863 (14)

Notes and references: (*a*) Veith *et al.* (2000); (*b*) Veith *et al.* (2009); (*c*) this work, **IV** with coordination number 4 and **II** with coordination number 2.

4. Discussion

Compound **2** was investigated crystallographically in some detail by Veith and co-workers. At room temperature, **2** crystallizes in the space group $P4_2/nmc$, with $a = b = 13.095$ (8), $c = 11.045$ (6) Å and imposed $\overline{4}m2$ symmetry, whereby the Li atoms occupy alternative positions (Veith *et al.*, 2000), leading to a molecular symmetry of 222. In the temperature region 163–243 K, the space group is $P4_2/n$, with $a = b = 12.999$ (2), $c = 10.866$ (2) Å and imposed $\overline{4}$ symmetry, again with alternative Li-atom positions (Veith *et al.*, 2009). The authors also reported a low-temperature phase (below 163 K), investigated using powder methods, with a monoclinic cell having $a = 12.893$ (6), $b = 12.971$ (6), $c = 10.854$ (7) Å and $\beta = 91.06$ (3)°, and formal symmetry probably 1. It seems tolerably certain that this low-temperature phase is in fact the one reported here for **2**.

The structure of the room-temperature form of compound **2** was also investigated by Ikeda *et al.* (2002), but without mentioning the split Li-atom positions (the article centred on chromium complexes of **2**).

The structure of **2** is shown in Fig. 1. The formal symmetry of the molecule is indeed reduced to 1, as must be the case in space group $P2_1$, which lacks special positions. The Li atoms are ordered, in contrast to the other two phases of **2**, and occupy two types of positions. Li1 and Li3 are coordinated by two O- and two N-atom donors, whereas Li2 and Li4 are only coordinated by two N-atom donors. Table 2 compares selected bond lengths for the three phases. The bond lengths at the Si atoms are slightly longer than expected. The lowering of the

point symmetry is shown in the bond lengths of the now ordered Li atoms. The Li—O bond lengths can be divided into two groups [mean values: Li—O1 2.284 (11) and Li—O2 2.352 (11) Å], as can the N—Li bond lengths [mean values N—Li1/3 1.985 (11) and 2.025 (11), and N—Li2/4 1.950 (10) and 1.985 (10) Å].

The phase transitions of **2** show the typical behaviour on cooling, the symmetry being lowered from point group $\overline{4}2m$ to $\overline{4}$, and finally 1 and from Laue class 4/*mmm* to 4/*m* and then 2/*m*. This very often leads to twinning, as observed here (*e.g.* Attiwell *et al.*, 2026).

5. Some reminiscences about twinning (PGJ)

Having recently noted the 50th anniversary of my first publication, I have clearly long since reached my anecdotage and have been allowed by considerate Editors to indulge in occasional reminiscences about the good/bad old days, *e.g.* the changing fashions in wavelength preferences (Abu-Zaied *et al.*, 2024). It may thus be appropriate here to muse briefly on the subject of twinning.

In retrospect, my experiences of twinning began in the early 1980s with the attempted structure determination of cyclohexanone oxime, which crystallizes in the trigonal space group $P\overline{3}$. A dataset was registered and the structure was solved, but could not be refined satisfactorily; a second attempt with a new crystal also failed. As a comparative beginner, I put this down to bad luck or gremlins and abandoned the structure. Nowadays, with my somewhat reduced level of ignorance, I would of course suspect twinning by merohedry. Indeed, an article has since appeared presenting the structure of cyclohexanone oxime as such a twin (Lutz *et al.*, 2004). It is however worth remembering that the standard methods of treating twinned data, specifically the TWIN command in the *SHELX* refinement program (Sheldrick, 2015*b*), have only been available since the 1990s (Herbst-Irmer & Sheldrick, 1998). It is unfortunately probable that many structures abandoned in the pre-TWIN era might well have been rescuable, if suitable software had been available. It should also not be forgotten that this was the pre-area-detector era, when the diffraction patterns could not be directly visualized on the diffractometer as frames.

Similarly, in the early 1990s, I attempted to determine the structure of (*tert*-butyl)bis(isopropyl)iodophosphane iodide. Although recognising, from the $R_{\rm int}$ values, that the structure must be monoclinic despite the metrically orthorhombic cell, I was unable to refine the monoclinic structure successfully (with typical symptoms of twinning, which I did not recognise at the time: poor R values, large residual electron-density peaks, many badly-fitting reflections with $F_{\rm o} >> F_{\rm c}$) and again abandoned the attempt. While sorting out old structures for deposition in 2013, I rediscovered the dataset and realised that the structure must be twinned by pseudomerohedry (with hindsight, the $R_{\rm int}$ value for orthorhombic symmetry was not impossibly high); the addition of appropriate TWIN (180° rotation about the *a* axis) and BASF commands to the refinement led to an entirely acceptable monoclinic structure, now deposited (Jones & Ruthe, 2013).

It took me several years to acquire enough practical knowledge of twinning phenomena, and the corresponding remedies, to deal with most of the obvious cases that I later encountered. During this time, I was helped considerably by two experts in the field; Regine Herbst-Irmer, co-author of this article, and Ina Dix (with whom we have unfortunately lost contact). Regine has also written many collections of useful tips for recognising the symptoms of twinning and for refining twinned structures (*e.g.* Herbst-Irmer, 2016). My previously hazy understanding of why and how twins form was also considerably clarified by the 'asymmetric brick' analogy in the excellent general article on twinning by Parsons (2003).

Three further examples may be informative, even if they correspond to advice given earlier by Regine (her tips bear repetition!). The first is the structure of a homobenzene derivative (Wullbrandt *et al.*, 2010). This was the reverse of the 'cyclohexanone oxime' experience; I managed to interpret the structure as being twinned by pseudomerohedry, after an attempt by others to determine the structure in the 1980s, which was unsuccessful because they did not have the necessary software (for discovering the true cell and twinning law) at the time. The cell as found by the diffractometer program was *C*-centred monoclinic, but was discarded in favour of the corresponding primitive triclinic cell on the basis of the $R_{\rm int}$ values. Structure solution was successful in $P\overline{1}$, but the refinement led to high R values. I therefore assumed twinning by pseudomerohedry *via* 180° rotation about the 'monoclinic' *b* axis; use of the corresponding twin matrix allowed the refinement to be successfully completed. The general problem of determining the twin matrix is not always trivial; in this case, one needs the matrices for interconversion of the monoclinic and triclinic cells (one is necessarily the inverse of the other). The twin matrix is then the product [matrix triclinic → monoclinic] × [rotation matrix 180° around the monoclinic *b* axis] × [matrix monoclinic → triclinic]. One should also note in passing that the program *XPREP* (also written by George) can be used to determine the matrices and their products, and it also gives the $R_{\rm int}$ values for the different symmetries. Somewhat more recent programs/routines, such as *TwinRotMat* in *PLATON* (Spek, 2020), also help to identify the twin law.

The second example is one of twinning by merohedry (Taouss *et al.*, 2013). The structure of the simple molecule imidazolin-2-one (2-imidazolidinone) had to our great surprise never been determined. We recorded a dataset that was assigned a tetragonal cell, space group $I4_1/amd$, by the diffractometer software (despite a rather high $R_{\rm int}$ value of 0.17). The structure was solved, but failed to refine satisfactorily; in particular, the five-membered ring, which should be non-planar, appeared to lie in a crystallographic mirror plane with high U value components perpendicular to this plane. We therefore reduced the symmetry to $I4_1/a$, leading to a more reasonable ring geometry, but the R values were still quite high. Particularly in view of the low $<|E^2 - 1|>$ value of 0.71, the obvious next step was to test for twinning by merohedry;

this indeed made the structure completely acceptable, whereby the well-known tetragonal twin law [010/100/00$\bar{1}$] led to a BASF value of 0.208 (2). In general, it is worthwhile to test any structure in Laue group 4/*m* for such twinning (similar considerations apply to the other high-symmetry crystal systems with more than one Laue group, namely cubic and hexagonal/trigonal). At the time, we cast doubt on an earlier structure of the compound in *Fdd*2 by Zhang *et al.* (2009), which had an *R* value of 0.14 but no deposited coordinates. A room-temperature structure in *Fddd*, clearly a new polymorph, was however later reported by Bellas & Matzger (2022).

The third example is the hydrocarbon $C_{20}H_{22}$ (Yildizhan *et al.*, 2015), which crystallized in an apparently orthorhombic cell. The suggested space group was $Pmc2_1$, but the structure was not solvable. R_{int} values were also not helpful, being 0.22 for orthorhombic and 0.20 for all three possible monoclinic cells with $\beta = 90^\circ$. The monoclinic cell that retained the same axis order however provided a recognisable solution; the initially poor refinement was improved to an entirely acceptable quality by assuming twinning by pseudomerohedry (rotation of 180° around the *a* axis), with a batch scale factor of *ca* 50%. It is perhaps surprising that such 50:50 twins can still lead to a structure solution. This leads to the one novel tip from this section: $Pmc2_1$ (and $Pmn2_1$) are rare space groups; if the structure cannot be solved or refined properly, it may be monoclinic $P2_1/c$ or $P2_1/n$ with $\beta \approx 90^\circ$ (by no means a rare combination) and twinned by pseudomerohedry.

Thanks to the commands TWIN and BASF, 'normal' twinning no longer holds many terrors, even for the non-expert. However, the structure of **2**, as presented here, is an example of a probable twin that I was unable to interpret. In such cases one must approach an expert! For a further example of a complex twinning phenomenon that was successfully unravelled, see the recent structure of a new form of white phosphorus (Herbst-Irmer *et al.*, 2023).

All of the above examples involve twinning by (pseudo) merohedry. The other major twinning type is non-merohedric twinning, where some, but not all, reflections of the twin components overlap. Older diffractometer types and their programs lacked the necessary software to analyse such twins adequately (auto-indexing routines found either no cell, or one with one or more impossibly long axes), and the only chance in general was to find a non-twinned crystal. The development of area detectors and suitable software has enabled such twins to be routinely identified, and new refinement techniques (in particular, the HKLF5 method developed by George) have allowed the twinned datasets to be refined. However, the increased efficiency in recording data to ever higher diffraction angles (powerful sources, sensitive detectors and, of course, low-temperature data collection) has blurred the different treatment of twinning by pseudomerohedry and non-merohedric twinning. For example, for a monoclinic compound with $\beta = 90.5^\circ$, twinned by pseudomerohedry, the two twin components might yield effectively overlapped data to $2\theta_{max}$ 50° (Mo $K\alpha$ radiation), thus enabling normal refinement with an appropriate TWIN matrix (even if this forces the refined β angle to a value nearer 90° than the true value). For a more modern dataset, recorded to $2\theta_{max}$ 90° (often feasible these days), the reflections of the two twin components may however diverge appreciably at high angle, necessitating an HKLF5-type treatment if the integration box cannot be enlarged to encompass both parts of the split reflections.

6. Conclusion

Nowadays, the identification and refinement of twinned structures is often routine. Many programs, such as *PLATON* (Spek, 2020) or *OLEX2* (Bourhis *et al.*, 2015), offer options to test for twinning, and even suggest a twin law. Data integration programs provide routines for determining multiple orientation matrices, which can be used for integration and to generate an HKL file containing information about the overlap. Modern refinement programs are capable of interpreting these files. We owe thanks for this development to George Sheldrick, who laid the groundwork with his HKLF5 format and, with TWIN/BASF, found a simple way of defining integer twin matrices. This article also relates how the handling of twinned structures – not only by experts, but by practising crystallographers – gradually developed.

Nevertheless, there are always interesting examples – such as structure **2** here – where the automatic routines are insufficient, where even the powerful structure-solving program *SHELXT* fails, where determining the space group is not trivial and where creating the HKLF5 file is not a routine task. It remains to be seen how the handling of twinned structures will be impacted by the use of AI.

7. Related literature

The following references are cited in the supporting information: Albrecht *et al.* (2020); Lutz *et al.* (2015); Petricek *et al.* (2014).

Acknowledgements

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Conflict of interest

The authors declare no conflict of interest.

Data availability

More details of the structure determination are provided in the supporting information. The structure has been deposited in the Cambridge Structural Database and is accessible at no charge through the joint Cambridge Crystallographic Data Centre and Fachinformationszentrum Karlsruhe Access Structures Service.

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An unusual case of tetartohedral twinning: the low-temperature phase of bis-[μ_3-1,3-bis(*tert*-butylazanidyl)-1,1,3,3-tetramethyldisiloxane]tetralithium(I)

Regine Herbst-Irmer, Peter G. Jones and Matthias Freytag

Computing details

Bis[μ_3-1,3-bis(*tert*-butylazanidyl)-1,1,3,3-tetramethyldisiloxane]tetralithium(I)

Crystal data

$[Li_4(C_{12}H_{30}N_2OSi_2)_2]$
M_r = 576.88
Tetragonal, $P2_1$
a = 10.761 (2) Å
c = 13.035 (3) Å
V = 1827.7 (7) Å^3
Z = 2
$F(000)$ = 632
D_x = 1.048 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 26532 reflections
θ = 3.4–75.7°
μ = 1.69 mm^{-1}
T = 101 K
Prism, colourless
0.25 × 0.12 × 0.10 mm

Data collection

Agilent Xcalibur Atlas Nova diffractometer
Radiation source: Nova (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.3543 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Agilent, 2012)
T_{min} = 0.480, T_{max} = 1.000
38449 measured reflections
7547 independent reflections
7547 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 76.0°, θ_{min} = 3.4°
h = −13→13
k = −16→16
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.067
S = 1.05
7547 reflections
370 parameters
19 restraints
Primary atom site location: dual space
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0464P)^2 + 0.316P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refined as a 8-component twin

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Si1	0.50994 (10)	0.28261 (8)	0.76708 (9)	0.0208 (2)
Si2	0.54480 (11)	0.52312 (8)	0.76772 (9)	0.0223 (2)
Si3	0.94493 (11)	0.34687 (8)	0.60009 (9)	0.0223 (2)
Si4	0.98702 (10)	0.36822 (8)	0.84113 (9)	0.0222 (2)
O1	0.5777 (3)	0.3983 (3)	0.7615 (2)	0.0237 (6)
O2	0.9168 (3)	0.3661 (2)	0.7257 (3)	0.0248 (6)
N1	0.6363 (3)	0.2090 (3)	0.7470 (3)	0.0226 (7)
N2	0.6903 (4)	0.5711 (3)	0.7565 (3)	0.0230 (8)
N3	0.7996 (3)	0.3620 (3)	0.5538 (3)	0.0223 (7)
N4	0.8616 (4)	0.3852 (3)	0.9164 (3)	0.0235 (7)
C1	0.6438 (4)	0.0968 (3)	0.7474 (4)	0.0246 (9)
C2	0.6491 (7)	0.0580 (4)	0.6349 (5)	0.0477 (14)
H2A	0.632433	−0.015895	0.633257	0.072*
H2B	0.731809	0.071429	0.606443	0.072*
H2C	0.586423	0.094060	0.593862	0.072*
C3	0.5317 (5)	0.0453 (3)	0.7956 (5)	0.0387 (12)
H3A	0.545007	−0.029108	0.797718	0.058*
H3B	0.457472	0.060436	0.754773	0.058*
H3C	0.520328	0.071192	0.865565	0.058*
C4	0.7599 (6)	0.0633 (4)	0.8034 (7)	0.0543 (19)
H4A	0.766586	−0.011720	0.800969	0.081*
H4B	0.755565	0.085814	0.875019	0.081*
H4C	0.832902	0.093894	0.770521	0.081*
C5	0.3823 (4)	0.2767 (4)	0.6662 (4)	0.0316 (9)
H5A	0.346897	0.207407	0.664517	0.047*
H5B	0.417195	0.293367	0.598829	0.047*
H5C	0.317000	0.326235	0.683324	0.047*
C6	0.4331 (5)	0.2742 (4)	0.8963 (4)	0.0334 (10)
H6A	0.394614	0.206484	0.904221	0.050*
H6B	0.369261	0.327482	0.901835	0.050*
H6C	0.495516	0.284138	0.950215	0.050*
C7	0.7242 (5)	0.6781 (3)	0.7402 (4)	0.0302 (10)
C8	0.7062 (6)	0.7077 (5)	0.6271 (5)	0.0456 (14)
H8A	0.738125	0.777167	0.615698	0.068*
H8B	0.617569	0.705474	0.609950	0.068*
H8C	0.751495	0.659179	0.583447	0.068*
C9	0.8626 (6)	0.6922 (4)	0.7660 (6)	0.0481 (15)
H9A	0.888005	0.762505	0.749385	0.072*
H9B	0.912217	0.643632	0.725877	0.072*
H9C	0.875854	0.679548	0.839353	0.072*
C10	0.6486 (7)	0.7524 (4)	0.8066 (6)	0.0583 (19)
H10A	0.675594	0.822986	0.793119	0.087*
H10B	0.661731	0.736239	0.879265	0.087*

H10C	0.560149	0.745621	0.790016	0.087*
C11	0.4331 (5)	0.5547 (4)	0.6627 (4)	0.0354 (11)
H11A	0.414503	0.628276	0.664062	0.053*
H11B	0.356171	0.515618	0.672509	0.053*
H11C	0.469941	0.536641	0.596368	0.053*
C12	0.4620 (5)	0.5478 (4)	0.8920 (4)	0.0325 (10)
H12A	0.443975	0.621281	0.898216	0.049*
H12B	0.514820	0.526129	0.949311	0.049*
H12C	0.384069	0.508973	0.893292	0.049*
C13	0.7642 (4)	0.3730 (3)	0.4445 (3)	0.0261 (9)
C14	0.6239 (5)	0.3493 (4)	0.4343 (4)	0.0344 (11)
H14A	0.598131	0.357910	0.362705	0.052*
H14B	0.576486	0.396414	0.477895	0.052*
H14C	0.608046	0.278422	0.455945	0.052*
C15	0.8332 (5)	0.2991 (5)	0.3744 (4)	0.0403 (12)
H15A	0.802606	0.306848	0.304001	0.060*
H15B	0.819032	0.228490	0.397575	0.060*
H15C	0.922346	0.314268	0.376520	0.060*
C16	0.7836 (6)	0.4827 (4)	0.4057 (5)	0.0387 (12)
H16A	0.774205	0.484185	0.330938	0.058*
H16B	0.867262	0.505971	0.424263	0.058*
H16C	0.721817	0.528080	0.437094	0.058*
C17	1.0174 (5)	0.2169 (4)	0.5871 (4)	0.0352 (11)
H17A	1.023893	0.198994	0.514251	0.053*
H17B	0.965707	0.165988	0.622270	0.053*
H17C	1.100492	0.217693	0.617866	0.053*
C18	1.0655 (4)	0.4424 (4)	0.5579 (4)	0.0358 (10)
H18A	1.077815	0.436725	0.483634	0.054*
H18B	1.144053	0.428245	0.593276	0.054*
H18C	1.037455	0.511886	0.574808	0.054*
C19	0.8561 (5)	0.3721 (4)	1.0284 (3)	0.0254 (9)
C20	0.8382 (5)	0.2585 (4)	1.0554 (4)	0.0384 (12)
H20A	0.838429	0.250374	1.130182	0.058*
H20B	0.905987	0.218141	1.025711	0.058*
H20C	0.758623	0.234527	1.027746	0.058*
C21	0.7427 (5)	0.4321 (5)	1.0692 (4)	0.0375 (11)
H21A	0.738356	0.425048	1.143964	0.056*
H21B	0.666605	0.404640	1.038327	0.056*
H21C	0.751194	0.504834	1.051239	0.056*
C22	0.9698 (5)	0.4118 (5)	1.0834 (4)	0.0375 (10)
H22A	0.955988	0.409068	1.157658	0.056*
H22B	0.985503	0.482956	1.062685	0.056*
H22C	1.041709	0.369283	1.065614	0.056*
C23	1.0749 (4)	0.2430 (4)	0.8592 (4)	0.0322 (10)
H23A	1.115884	0.243077	0.926388	0.048*
H23B	1.137466	0.235917	0.805142	0.048*
H23C	1.016517	0.185336	0.855519	0.048*
C24	1.1064 (5)	0.4715 (4)	0.8394 (4)	0.0331 (11)

H24A	1.151088	0.472065	0.904870	0.050*
H24B	1.065735	0.537974	0.828867	0.050*
H24C	1.165132	0.458799	0.783379	0.050*
Li1	0.7490 (11)	0.4549 (7)	0.6659 (7)	0.0329 (15)
Li2	0.6980 (9)	0.2521 (8)	0.6098 (7)	0.0361 (19)
Li3	0.7524 (10)	0.3048 (7)	0.8183 (8)	0.0321 (16)
Li4	0.7831 (8)	0.5139 (7)	0.8748 (6)	0.0312 (19)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Si1	0.0174 (5)	0.0212 (4)	0.0240 (5)	−0.0013 (4)	0.0018 (4)	0.0010 (4)
Si2	0.0182 (6)	0.0207 (5)	0.0279 (5)	0.0024 (4)	0.0014 (4)	−0.0007 (4)
Si3	0.0182 (5)	0.0244 (5)	0.0242 (5)	0.0016 (4)	0.0032 (5)	0.0008 (4)
Si4	0.0150 (5)	0.0261 (5)	0.0256 (5)	−0.0015 (5)	−0.0004 (4)	0.0014 (4)
O1	0.0158 (14)	0.0300 (13)	0.0253 (14)	−0.0009 (12)	−0.0022 (11)	−0.0011 (13)
O2	0.0201 (14)	0.0230 (13)	0.0313 (13)	0.0026 (12)	0.0040 (13)	0.0049 (12)
N1	0.0245 (18)	0.0198 (17)	0.023 (2)	−0.0018 (14)	0.0051 (15)	−0.0033 (14)
N2	0.0267 (19)	0.0162 (16)	0.026 (2)	0.0027 (13)	0.0042 (15)	−0.0007 (14)
N3	0.0227 (17)	0.0244 (18)	0.0197 (16)	−0.0008 (15)	0.0040 (13)	0.0008 (13)
N4	0.0205 (17)	0.0266 (19)	0.0235 (17)	0.0002 (14)	−0.0044 (14)	0.0017 (14)
C1	0.022 (2)	0.0208 (18)	0.031 (3)	−0.0008 (16)	0.0027 (18)	0.0036 (16)
C2	0.067 (4)	0.027 (2)	0.049 (3)	−0.002 (3)	0.010 (3)	−0.005 (2)
C3	0.036 (3)	0.019 (2)	0.061 (3)	−0.0008 (19)	0.008 (2)	0.0068 (18)
C4	0.037 (3)	0.026 (2)	0.099 (6)	0.003 (2)	−0.023 (3)	0.002 (3)
C5	0.026 (2)	0.030 (2)	0.039 (3)	−0.0018 (19)	−0.0058 (19)	−0.003 (2)
C6	0.033 (2)	0.032 (2)	0.035 (2)	0.000 (2)	0.007 (2)	−0.0006 (19)
C7	0.032 (2)	0.017 (2)	0.041 (3)	0.0001 (18)	0.005 (2)	−0.0022 (19)
C8	0.052 (3)	0.027 (2)	0.058 (4)	−0.007 (2)	−0.001 (3)	0.013 (2)
C9	0.039 (3)	0.032 (3)	0.073 (4)	−0.014 (2)	−0.008 (3)	0.004 (3)
C10	0.074 (4)	0.021 (2)	0.079 (5)	−0.001 (3)	0.033 (4)	−0.003 (3)
C11	0.033 (2)	0.035 (2)	0.038 (3)	0.004 (2)	−0.003 (2)	0.010 (2)
C12	0.026 (2)	0.029 (2)	0.042 (3)	0.0010 (18)	0.005 (2)	−0.0051 (18)
C13	0.030 (2)	0.027 (2)	0.021 (2)	−0.0008 (19)	−0.0014 (17)	−0.0029 (17)
C14	0.038 (3)	0.038 (3)	0.027 (2)	−0.007 (2)	−0.0044 (18)	−0.001 (2)
C15	0.043 (3)	0.055 (3)	0.022 (2)	0.005 (3)	0.008 (2)	−0.007 (2)
C16	0.042 (3)	0.036 (2)	0.038 (3)	−0.013 (2)	−0.007 (2)	0.011 (2)
C17	0.031 (3)	0.037 (2)	0.038 (3)	0.007 (2)	0.002 (2)	−0.0065 (19)
C18	0.029 (2)	0.041 (3)	0.038 (3)	−0.006 (2)	0.004 (2)	0.006 (2)
C19	0.026 (2)	0.031 (2)	0.0184 (18)	0.0018 (19)	0.0015 (17)	−0.0048 (17)
C20	0.047 (3)	0.044 (3)	0.024 (2)	−0.006 (2)	0.006 (2)	0.005 (2)
C21	0.031 (2)	0.053 (3)	0.029 (2)	0.009 (2)	0.006 (2)	0.002 (2)
C22	0.038 (3)	0.044 (3)	0.031 (2)	0.003 (2)	−0.011 (2)	−0.011 (2)
C23	0.024 (2)	0.030 (2)	0.043 (3)	0.0053 (18)	0.002 (2)	0.007 (2)
C24	0.027 (2)	0.040 (3)	0.032 (2)	−0.003 (2)	−0.005 (2)	−0.004 (2)
Li1	0.041 (4)	0.025 (4)	0.033 (5)	0.001 (4)	0.000 (4)	−0.011 (3)
Li2	0.028 (4)	0.048 (5)	0.033 (4)	−0.008 (4)	0.000 (4)	−0.005 (4)
Li3	0.027 (3)	0.025 (4)	0.044 (5)	−0.003 (3)	−0.010 (4)	−0.004 (3)

Li4	0.040 (5)	0.032 (4)	0.022 (3)	0.011 (4)	−0.010 (3)	−0.003 (3)

Geometric parameters (Å, º)

Si1—O1	1.676 (4)	N2—C7	1.457 (5)
Si1—N1	1.685 (4)	N2—Li4	1.981 (9)
Si1—C6	1.880 (5)	N2—Li1	2.022 (11)
Si1—C5	1.902 (5)	N3—C13	1.481 (6)
Si1—Li3	2.709 (10)	N3—Li2	1.945 (10)
Si1—Li2	2.909 (9)	N3—Li1	1.974 (11)
Si2—O1	1.667 (4)	N4—C19	1.470 (5)
Si2—N2	1.692 (4)	N4—Li4	1.955 (9)
Si2—C11	1.867 (5)	N4—Li3	2.027 (12)
Si2—C12	1.877 (5)	C1—C4	1.512 (7)
Si2—Li1	2.718 (11)	C1—C3	1.517 (6)
Si2—Li4	2.921 (8)	C1—C2	1.552 (7)
Si3—O2	1.684 (4)	C1—Li2	2.766 (11)
Si3—N3	1.687 (4)	C2—Li2	2.603 (12)
Si3—C17	1.872 (5)	C7—C10	1.534 (7)
Si3—C18	1.880 (5)	C7—C8	1.536 (8)
Si3—Li1	2.677 (11)	C7—C9	1.538 (8)
Si3—Li2	2.933 (9)	C13—C15	1.521 (7)
Si4—O2	1.683 (4)	C13—C16	1.530 (7)
Si4—N4	1.684 (4)	C13—C14	1.548 (7)
Si4—C24	1.860 (5)	C13—Li2	2.764 (11)
Si4—C23	1.901 (5)	C14—Li2	2.733 (11)
Si4—Li3	2.672 (10)	C19—C22	1.509 (7)
Si4—Li4	2.935 (8)	C19—C20	1.533 (7)
O1—Li1	2.345 (11)	C19—C21	1.545 (6)
O1—Li3	2.359 (10)	C21—Li4	2.784 (10)
O2—Li1	2.282 (11)	Li1—Li3	2.788 (11)
O2—Li3	2.286 (10)	Li1—Li2	2.796 (14)
N1—C1	1.463 (6)	Li1—Li4	2.853 (12)
N1—Li2	1.989 (10)	Li2—Li3	2.863 (14)
N1—Li3	1.996 (11)	Li3—Li4	2.842 (13)
O1—Si1—N1	98.86 (18)	N2—Li1—O1	71.3 (4)
O1—Si1—C6	106.4 (2)	O2—Li1—O1	106.3 (3)
N1—Si1—C6	117.5 (2)	N3—Li1—Si3	39.0 (2)
O1—Si1—C5	108.7 (2)	N2—Li1—Si3	145.8 (5)
N1—Si1—C5	116.9 (2)	O2—Li1—Si3	38.7 (2)
C6—Si1—C5	107.4 (2)	O1—Li1—Si3	128.7 (4)
O1—Si1—Li3	59.7 (2)	N3—Li1—Si2	141.8 (5)
N1—Si1—Li3	47.3 (3)	N2—Li1—Si2	38.4 (2)
C6—Si1—Li3	102.1 (3)	O2—Li1—Si2	129.7 (4)
C5—Si1—Li3	150.4 (3)	O1—Li1—Si2	37.55 (19)
O1—Si1—Li2	77.9 (2)	Si3—Li1—Si2	165.0 (3)
N1—Si1—Li2	41.5 (3)	N3—Li1—Li3	95.4 (5)

C6—Si1—Li2	158.4 (3)	N2—Li1—Li3	96.5 (5)
C5—Si1—Li2	90.6 (3)	O2—Li1—Li3	52.5 (3)
Li3—Si1—Li2	61.2 (3)	O1—Li1—Li3	53.9 (3)
O1—Si2—N2	99.19 (18)	Si3—Li1—Li3	81.4 (3)
O1—Si2—C11	108.4 (2)	Si2—Li1—Li3	83.8 (3)
N2—Si2—C11	116.8 (2)	N3—Li1—Li2	44.1 (3)
O1—Si2—C12	108.07 (19)	N2—Li1—Li2	143.1 (5)
N2—Si2—C12	116.8 (2)	O2—Li1—Li2	76.4 (3)
C11—Si2—C12	106.8 (2)	O1—Li1—Li2	71.8 (3)
O1—Si2—Li1	59.0 (2)	Si3—Li1—Li2	64.8 (3)
N2—Si2—Li1	48.0 (3)	Si2—Li1—Li2	106.2 (4)
C11—Si2—Li1	103.6 (3)	Li3—Li1—Li2	61.7 (4)
C12—Si2—Li1	149.5 (3)	N3—Li1—Li4	146.8 (5)
O1—Si2—Li4	78.3 (2)	N2—Li1—Li4	44.0 (3)
N2—Si2—Li4	40.9 (2)	O2—Li1—Li4	73.1 (3)
C11—Si2—Li4	157.5 (2)	O1—Li1—Li4	71.2 (3)
C12—Si2—Li4	90.7 (2)	Si3—Li1—Li4	110.3 (4)
Li1—Si2—Li4	60.7 (3)	Si2—Li1—Li4	63.2 (3)
O2—Si3—N3	99.40 (17)	Li3—Li1—Li4	60.5 (4)
O2—Si3—C17	107.3 (2)	Li2—Li1—Li4	122.0 (4)
N3—Si3—C17	117.3 (2)	N3—Li2—N1	137.2 (5)
O2—Si3—C18	108.1 (2)	N3—Li2—C2	151.2 (5)
N3—Si3—C18	117.2 (2)	N1—Li2—C2	63.0 (3)
C17—Si3—C18	106.5 (3)	N3—Li2—C14	60.5 (3)
O2—Si3—Li1	57.9 (2)	N1—Li2—C14	141.8 (5)
N3—Si3—Li1	47.4 (3)	C2—Li2—C14	119.7 (4)
C17—Si3—Li1	146.4 (3)	N3—Li2—C13	30.9 (2)
C18—Si3—Li1	106.8 (3)	N1—Li2—C13	161.6 (5)
O2—Si3—Li2	81.8 (2)	C2—Li2—C13	134.7 (4)
N3—Si3—Li2	39.2 (2)	C14—Li2—C13	32.70 (19)
C17—Si3—Li2	90.0 (3)	N3—Li2—C1	154.3 (5)
C18—Si3—Li2	156.4 (3)	N1—Li2—C1	30.7 (2)
Li1—Si3—Li2	59.6 (3)	C2—Li2—C1	33.4 (2)
O2—Si4—N4	99.43 (17)	C14—Li2—C1	145.1 (4)
O2—Si4—C24	108.1 (2)	C13—Li2—C1	167.7 (4)
N4—Si4—C24	117.8 (2)	N3—Li2—Li1	44.9 (3)
O2—Si4—C23	108.6 (2)	N1—Li2—Li1	95.6 (4)
N4—Si4—C23	116.0 (2)	C2—Li2—Li1	157.6 (4)
C24—Si4—C23	106.2 (2)	C14—Li2—Li1	80.7 (4)
O2—Si4—Li3	58.1 (2)	C13—Li2—Li1	67.3 (3)
N4—Si4—Li3	49.3 (3)	C1—Li2—Li1	124.3 (4)
C24—Si4—Li3	151.0 (2)	N3—Li2—Li3	93.7 (4)
C23—Si4—Li3	102.6 (3)	N1—Li2—Li3	44.2 (3)
O2—Si4—Li4	79.02 (19)	C2—Li2—Li3	98.9 (4)
N4—Si4—Li4	39.5 (2)	C14—Li2—Li3	138.0 (4)
C24—Si4—Li4	92.9 (3)	C13—Li2—Li3	123.4 (4)
C23—Si4—Li4	155.3 (2)	C1—Li2—Li3	66.6 (3)
Li3—Si4—Li4	60.7 (3)	Li1—Li2—Li3	59.0 (3)

Si2—O1—Si1	141.5 (2)	N3—Li2—Si1	123.8 (5)
Si2—O1—Li1	83.5 (3)	N1—Li2—Si1	34.1 (2)
Si1—O1—Li1	130.4 (3)	C2—Li2—Si1	84.5 (3)
Si2—O1—Li3	131.2 (3)	C14—Li2—Si1	108.9 (4)
Si1—O1—Li3	82.5 (3)	C13—Li2—Si1	130.7 (4)
Li1—O1—Li3	72.7 (4)	C1—Li2—Si1	59.7 (2)
Si4—O2—Si3	142.3 (2)	Li1—Li2—Si1	79.8 (3)
Si4—O2—Li1	130.6 (3)	Li3—Li2—Si1	56.0 (3)
Si3—O2—Li1	83.4 (3)	N3—Li2—Si3	33.27 (19)
Si4—O2—Li3	83.2 (3)	N1—Li2—Si3	117.4 (4)
Si3—O2—Li3	127.0 (3)	C2—Li2—Si3	126.7 (4)
Li1—O2—Li3	75.2 (4)	C14—Li2—Si3	91.9 (3)
C1—N1—Si1	127.8 (3)	C13—Li2—Si3	59.5 (2)
C1—N1—Li2	105.5 (4)	C1—Li2—Si3	121.8 (4)
Si1—N1—Li2	104.4 (4)	Li1—Li2—Si3	55.6 (3)
C1—N1—Li3	126.1 (4)	Li3—Li2—Si3	75.8 (3)
Si1—N1—Li3	94.4 (3)	Si1—Li2—Si3	127.1 (3)
Li2—N1—Li3	91.9 (4)	N1—Li3—N4	168.3 (5)
C7—N2—Si2	126.7 (3)	N1—Li3—O2	117.2 (5)
C7—N2—Li4	110.3 (4)	N4—Li3—O2	72.8 (3)
Si2—N2—Li4	105.1 (3)	N1—Li3—O1	71.2 (3)
C7—N2—Li1	123.6 (4)	N4—Li3—O1	113.1 (5)
Si2—N2—Li1	93.6 (4)	O2—Li3—O1	105.7 (3)
Li4—N2—Li1	90.9 (4)	N1—Li3—Si4	146.7 (6)
C13—N3—Si3	126.3 (3)	N4—Li3—Si4	39.0 (2)
C13—N3—Li2	106.8 (4)	O2—Li3—Si4	38.70 (19)
Si3—N3—Li2	107.5 (4)	O1—Li3—Si4	128.9 (3)
C13—N3—Li1	125.6 (4)	N1—Li3—Si1	38.3 (2)
Si3—N3—Li1	93.6 (4)	N4—Li3—Si1	140.2 (5)
Li2—N3—Li1	91.0 (4)	O2—Li3—Si1	130.8 (4)
C19—N4—Si4	126.6 (3)	O1—Li3—Si1	37.83 (18)
C19—N4—Li4	110.9 (4)	Si4—Li3—Si1	166.0 (3)
Si4—N4—Li4	107.3 (3)	N1—Li3—Li1	95.7 (4)
C19—N4—Li3	122.9 (4)	N4—Li3—Li1	95.4 (4)
Si4—N4—Li3	91.6 (4)	O2—Li3—Li1	52.3 (3)
Li4—N4—Li3	91.1 (4)	O1—Li3—Li1	53.4 (3)
N1—C1—C4	109.6 (4)	Si4—Li3—Li1	82.8 (3)
N1—C1—C3	113.6 (4)	Si1—Li3—Li1	83.5 (3)
C4—C1—C3	109.2 (4)	N1—Li3—Li4	142.5 (5)
N1—C1—C2	108.9 (4)	N4—Li3—Li4	43.5 (3)
C4—C1—C2	109.3 (5)	O2—Li3—Li4	73.3 (3)
C3—C1—C2	106.1 (4)	O1—Li3—Li4	71.2 (3)
N1—C1—Li2	43.9 (3)	Si4—Li3—Li4	64.2 (3)
C4—C1—Li2	110.5 (4)	Si1—Li3—Li4	106.1 (4)
C3—C1—Li2	139.5 (4)	Li1—Li3—Li4	60.9 (3)
C2—C1—Li2	67.5 (3)	N1—Li3—Li2	44.0 (3)
C1—C2—Li2	79.0 (3)	N4—Li3—Li2	147.2 (5)
N2—C7—C10	112.9 (4)	O2—Li3—Li2	75.0 (4)

N2—C7—C8	110.4 (4)	O1—Li3—Li2	70.3 (3)
C10—C7—C8	108.5 (5)	Si4—Li3—Li2	111.9 (4)
N2—C7—C9	109.0 (4)	Si1—Li3—Li2	62.9 (3)
C10—C7—C9	108.3 (5)	Li1—Li3—Li2	59.3 (4)
C8—C7—C9	107.5 (5)	Li4—Li3—Li2	120.0 (3)
N3—C13—C15	113.0 (4)	N4—Li4—N2	139.1 (5)
N3—C13—C16	111.9 (4)	N4—Li4—C21	59.1 (3)
C15—C13—C16	109.1 (4)	N2—Li4—C21	140.6 (4)
N3—C13—C14	108.2 (4)	N4—Li4—Li3	45.5 (3)
C15—C13—C14	107.4 (4)	N2—Li4—Li3	95.8 (4)
C16—C13—C14	107.0 (4)	C21—Li4—Li3	81.4 (3)
N3—C13—Li2	42.3 (3)	N4—Li4—Li1	95.1 (4)
C15—C13—Li2	103.5 (4)	N2—Li4—Li1	45.1 (3)
C16—C13—Li2	145.6 (4)	C21—Li4—Li1	138.2 (4)
C14—C13—Li2	72.6 (3)	Li3—Li4—Li1	58.6 (3)
C13—C14—Li2	74.7 (3)	N4—Li4—Si2	123.1 (4)
N4—C19—C22	113.5 (4)	N2—Li4—Si2	34.02 (18)
N4—C19—C20	110.2 (4)	C21—Li4—Si2	108.2 (3)
C22—C19—C20	108.9 (4)	Li3—Li4—Si2	79.3 (3)
N4—C19—C21	108.4 (4)	Li1—Li4—Si2	56.1 (3)
C22—C19—C21	107.6 (4)	N4—Li4—Si4	33.22 (19)
C20—C19—C21	108.0 (4)	N2—Li4—Si4	120.2 (4)
C19—C21—Li4	75.9 (3)	C21—Li4—Si4	90.3 (3)
N3—Li1—N2	167.9 (5)	Li3—Li4—Si4	55.1 (3)
N3—Li1—O2	73.9 (4)	Li1—Li4—Si4	77.2 (3)
N2—Li1—O2	115.3 (5)	Si2—Li4—Si4	127.7 (3)
N3—Li1—O1	114.7 (5)		