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Refinement of the structure of LiV_2O_5.

By Donald N. Anderson and Roger D. Willett, *Department of Chemistry, Washington State University, Pullman, Washington* 99163, *U.S.A.*

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The structure determined by Galy & Hardy [*Acta Cryst.* (1955). **19**, 432] has been refined in the space group *Pnma* to a lower *R* value (11·0%) with only slight changes in the atomic parameters.

The retigraph data obtained by Galy & Hardy (1955) have been used in a least-squares refinement which confirms the general coordination found by their Fourier refinement. Only the lithium position shifted by more than 0·1 Å.

The previous refinement was reported in the non-centric space group $Pna2_1$ but refined in $Pn2_1a$ with coordinates restricted to $y=0{\cdot}0$ to give a centrosymmetric structure. Therefore, Galy & Hardy's coordinates translated to $y=\frac{1}{4}$ were used to initialize the refinement in the centrosymmetric space group *Pnma*. Using full-matrix least squares, the observed *R* (see definition note †, Table 1) decreased from 18% to 11%. The new parameters are given in Table 1. The scattering factors were obtained from *International Tables for X-ray Crystallography* (1969) except for the vanadium scattering factors calculated by Cromer & Waber (1965).

Table 1. *Lattice constants and atomic parameters for* LiV_2O_5*

Space group *Pnma*
$a=9{\cdot}702$ (5), $b=3{\cdot}607$ (3),
$c=10{\cdot}664$ (9) Å; $Z=4$.
All atoms are in special positions at $y=\frac{1}{4}$.

	x	*z*	*B*
Li(1)	0·1778 (52)	0·2263 (46)	2·4 (10)
V(2)	0·3762 (5)	0·4982 (5)	1·8 (2)
V(3)	0·0657 (5)	0·5987 (5)	1·6 (2)
O(4)	0·2406 (19)	0·6341 (17)	1·5 (4)
O(5)	0·4763 (23)	0·7739 (19)	2·2 (11)
O(6)	0·2863 (24)	0·3667 (20)	2·2 (4)
O(7)	0·5725 (23)	0·4470 (21)	2·1 (4)
O(8)	0·4395 (27)	0·0256 (42)	1·8 (13)

* The number in parentheses is the e.s.d. in the last digits of each number.

† $R=\Sigma\|F_o|-|F_c\|/\Sigma|F_o|=11{\cdot}0$.

The vanadium coordination now appears to be more nearly square pyramidal than trigonal bipyramidal. The square pyramid of oxygen atoms about each V(2) atom has four oxygen atoms forming the base of the pyramid which deviate an average of 0·11 Å from the mean plane. The distance of the V(2) atom above this base plane is 0·650 Å. The V(3) coordination pyramid has a base exhibiting greater planarity, (distance from mean plane averages 0·06 Å) and the vanadium sits 0·586 Å above the mean plane. However, the basal vanadium–oxygen distances (1·73 to 1·97 Å) indicate the actual geometry is only approximately square pyramidal.

The bond distances (Table 2 and Fig. 1), coordination volumes, and distances from the mean plane (2·31 and 2·15 Å respectively) for the V(2) and V(3) atoms suggest that the former approximates the +4 oxidation state compared to a +5 state for the V(3) atom.

The Li atom has a crude octahedral coordination with one distance [to O(6)] considerably less than the others.

The vanadium coordination in LiV_2O_5 differs greatly from that found by Caughlan, Smith & Watenpaugh (1966) for $VO(OCH_3)_3$, in that rather than a sixth coordination oxygen at 2·25–2·30 Å the closest additional oxygen is at distance of 3·09 Å and distinctly displaced to one side of a possible sixth coordination site.

This sixth oxygen is also found in Darriet, Muhll & Galy's (1969) study of $Pb_{0\ 193}V_2O_5$, although in that structure one of the vanadium atoms has its sixth oxygen atom at 2·64 Å allowing it a close similarity to the V(2) found in LiV_2O_5.

Reference

Caughlan, C. N., Smith, H. M. & Watenpaugh, K. (1966). *Inorg. Chem.* **5**, 2131.

Table 2. *Selected interatomic distances and angles for* $LiV_2\ O_5$*

V(2)–V(2$^{\mathrm{i}}$)	3·004 (8) Å
V(2)–V(3)	3·197 (8)
V(3)–V(3$^{\mathrm{ii}}$)	3·051 (9)
Li—O(6)	1·830 (53)
Li—O(7)	2·112 (55)
Li—O(4$^{\mathrm{iv,v}}$)	2·201 (31)
Li—O(5$^{\mathrm{iv,v}}$)	2·397 (36)
V(2)–O(6)	1·651 (22)
V(2)–O(4)	1·957 (19)
V(2)–O(7$^{\mathrm{i,iii}}$)	1·960 (9)
V(2)–O(7)	1·981 (24)
V(3)–O(5$^{\mathrm{vi}}$)	1·612 (20)
V(3)–O(4)	1·738 (20)
V(3)–O(8$^{\mathrm{vi}}$)	1·805 (35)
V(3)–O(8$^{\mathrm{iv,v}}$)	1·966 (18)

Base of V(2) pyramid (4 angles along rim):	
O(4)——V(2)–O(7$^{\mathrm{i,iii}}$)	87·1 (7)°
O(7$^{\mathrm{i,iii}}$)–V(2)–O(7)	80·7 (7)
To apex of V(2) pyramid:	
O(6)——V(2)–O(4)	105·9 (10)
O(6)——V(2)–O(7$^{\mathrm{i,iii}}$)	112·8 (6)
O(6)——V(2)–O(7)	105·9 (11)
Base of V(3) pyramid (4 angles along rim):	
O(4)——V(3)–O(8$^{\mathrm{iv,v}}$)	96·4 (8)
O(8$^{\mathrm{vi}}$) —V(3)–O(8$^{\mathrm{iv,v}}$)	72·0 (9)
To apex of V(3) pyramid:	
O(5$^{\mathrm{vi}}$)—V(3)–O(4)	110·0 (11)
O(5$^{\mathrm{vi}}$)—V(3)–O(8$^{\mathrm{vi}}$)	104·7 (15)
O(5$^{\mathrm{vi}}$)—V(3)–O(8$^{\mathrm{iv,v}}$)	108·7 (12)

* Superscripts refer to the following translations (multiple superscripts indicate additional distances or angles with the same magnitude):

(i) $1-x,\ 1-y,\ 1-z$
(ii) $-x,\ 1-y,\ 1-z$
(iii) $1-x,\ -y,\ 1-z$
(iv) $\frac{1}{2}-x,\ \frac{1}{2}+y,\ \frac{1}{2}+z$
(v) $\frac{1}{2}-x,\ y-\frac{1}{2},\ \frac{1}{2}+z$
(vi) $x-\frac{1}{2},\ \frac{1}{2}-y,\ \frac{1}{2}-z$.

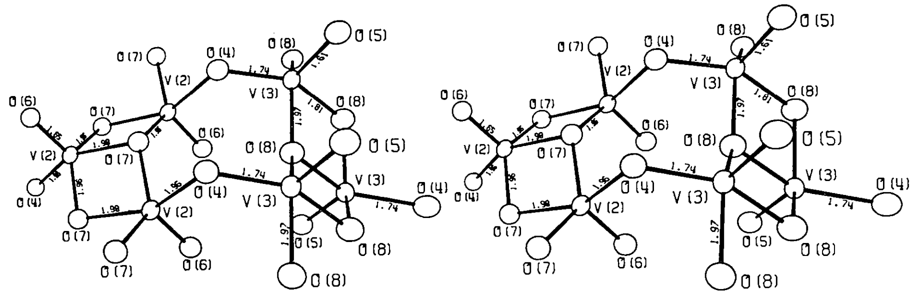

Fig. 1. A portion of the structure of LiV$_2$O , showing the coordination about the vanadium atoms.

Cromer, D. T. & Waber, J. T. (1965). *Acta Cryst.* **18**, 104.
Darriet, J., Muhll, R. & Galy, J. (1969). *Bull. Soc. franç. Minér. Crist.* **92**, 17.
Galy, J. & Hardy, A. (1955). *Acta Cryst.* **19**, 432.
International Tables for X-ray Crystallography (1969). Vol. I. Birmingham: Kynoch Press.

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The crystal structure of $(-)_{546}$-*cis*-β-dinitro-(L-3,8-dimethyltriethylenetetramine)cobalt(III) perchlorate. A correction.

By M. Ito, F. Marumo and Y. Saito, *The Institute for Solid State Physics, The University of Tokyo, Roppongi, Tokyo* 106, *Japan*

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Corrected values for crystal data are given.

In a recent article (Ito, Marumo & Saito, 1970) incorrect values were given for M, D_x, $F(000)$ and μ in the crystal data of $(-)_{546}$-*cis*-β-[Co(NO$_2$)$_2$(L-3,8-dimetrien)]ClO$_4$. The correct values are as follows: $M=424$, $D_x=1{\cdot}76$ g.cm^{-3}, $F(000)=880$, $\mu=69$ cm^{-1}.

Reference

Ito, M., Marumo, F. & Saito, Y. (1970). *Acta Cryst.* B**26** 1408.

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Raumgruppenbestimmung durch Datengruppen-Einzelstatistik.

Von M. Dräger und G. Gattow, *Institut für Anorganische Chemie und Kernchemie, Johannes Gutenberg-Universität, D-*65 *Mainz, Deutschland* (*BRD*)

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A new space group test in connexion with the 'average-multiple method' of Rogers [*Acta Cryst.* (1950), **3**, 455] is demonstrated.

Wilson (1949, 1950) und Rogers (1950) zeigten, dass die Intensitäten der reziproken Gitterpunkte eines Kristalls ausser der Laue-Symmetrie und den durch Translationselemente bedingten systematischen Auslöschungen noch zwei weitere charakteristische Eigenschaften besitzen, die nach einer statistischen Auswertung zur Raumgruppenbestimmung herangezogen werden können. Mittels der 'distribution method' wird geprüft, ob die Gesamtzelle oder eine spezielle Zone ein Symmetriezentrum enthält oder nicht, und die 'average-multiple method' stellt fest, ob die mittlere Intensität in Zonen, die parallel möglichen Symmetrieelementen liegen, ein ganzzahliges Vielfaches der mittleren Intensität der übrigen Zonen beträgt. Zur Durchführung der 'distribution method' sind verschiedene Tests entwickelt worden (vgl. Rogers, 1965), während die 'average multiple method' bislang kaum praktische Anwendung erfahren hat. Dieses ist verwunderlich, da 'distribution'-Tests häufig wegen erheblicher Abweichungen von den idealen Werten nicht mehr eindeutig interpretierbar sind, während die 'average multiple method' infolge der Grösse des zu beobachtenden Effekts noch eine klare Entscheidung zulässt. Ein Grund für die Vernachlässigung der letzteren Methode ist sicherlich das Fehlen eines systematischen und schnellen Rechenverfahrens zur Durchführung eines 'average multiple'-Tests. Wir entwickelten deshalb ein Verfahren, das im Rahmen der allgemeinen