

Table 6. *Contacts* (Å) less than 3.50 Å

Symmetry code			
i	$\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$	viii	$\frac{1}{2}-x, y-\frac{1}{2}, z-\frac{1}{2}$
iv	$\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$	ix	$\bar{x}, \bar{y}, 1-z$
vi	$x-\frac{1}{2}, \bar{y}-\frac{1}{2}, \frac{1}{2}+z$	x	$\bar{x}, \bar{y}-1, 1-z$
O(1)-O(4 <sup>viii</sup> )	3.370 (4)	O(6)-O(2 <sup>iv</sup> )	3.392 (5)
O(2)-O(5 <sup>i</sup> )	3.301 (4)	O(6)-O(3 <sup>iv</sup> )	2.872 (5)
O(2)-O(4 <sup>viii</sup> )	3.426 (4)	O(6)-O(5 <sup>iv</sup> )	3.010 (4)
O(2)-O(7)	2.795 (3)	O(6)-O(7 <sup>iv</sup> )	3.036 (4)
O(2)-O(9)	3.489 (4)	O(7)-N(1)	3.156 (3)
O(5)-O(7)	2.924 (2)	O(9)-O(4 <sup>vi</sup> )	2.747 (2)
O(5)-O(8)	3.319 (2)	O(9)-O(8 <sup>ix</sup> )	3.274 (4)
		O(9)-O(9 <sup>x</sup> )	3.379 (4)

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## The Crystal and Molecular Structure of Bis-(2,4-pentanedionato)beryllium\*

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The crystal structure of bis-(2,4-pentanedionato)beryllium,  $\text{Be}(\text{CH}_3\text{COCHCOCH}_3)_2$ , has been solved by direct methods. This compound crystallizes in space group  $P2_1$  with cell dimensions  $a_0 = 13.537$  (2),  $b_0 = 11.378$  (2),  $c_0 = 7.762$  (1) Å and  $\beta = 100.76$  (1)° with two molecules in the asymmetric unit ( $Z = 4$ ). Least-squares refinement of parameters using 2076 Mo  $K\alpha$ , counter-collected intensities yielded an  $R$  value of 0.066. The average interatomic separation for Be-O is 1.62 (2) Å and the average O-Be-O chelate ring angle is 107 (1)°. In one molecule the Be atom is located on each of the least-squares planes through the ring atoms of the two different chelate ligands while in the other molecule the Be atom is 0.20 Å removed from either of the two least-squares planes.

### Introduction

Several years ago, discrepancies between the listed bond lengths and angles and those calculated using the published atomic parameters were noted in the crystal structure of bis-(2,4-pentanedionato)beryllium (or beryllium acetylacetonate), hereinafter  $\text{Be}(\text{Acac})_2$ , reported by Amirthaligam, Padmanabhan & Shankar (1960), hereinafter APS. Initial attempts to refine a set of peak-height intensity data using the published parameters were unsuccessful leading us to question the validity of the published structure. Various attempts (Stewart & Morosin, 1972) over several years to obtain the correct phases of a sufficient number of structure factors to lead to the correct structure were unsuccessful until the early coding of program *TANGEN* (X-RAY system: Stewart, Kruger, Ammon, Dickerson & Hall, 1972) was completed. With its use, the structure was then solved and refined.

### Experimental details and results

The crystal specimens were grown from slowly evaporating methanol solutions. Weissenberg and precession photography verified previously assigned space groups as  $P2_1$  or  $P2_1/m$  (Bullen, 1957); the statistical distribution of the normalized structure factors corroborated APS's choice, based on the  $N(z)$  test (Howells, Phillips & Rogers, 1950) of the noncentrosymmetric space group and the final solution of the structure confirmed it. Lattice constants were determined as  $a_0 = 13.537$  (2),  $b_0 = 11.378$  (2),  $c_0 = 7.762$  (1) Å and  $\beta = 100.76$  (1)° by measurements made on a Picker diffractometer (Cu  $K\alpha$ ). There are two independent molecules in the asymmetric unit ( $Z = 4$ ).

Over the period in which this material was studied, various data sets were collected from different specimens because of both instrumental changes as well as radiation degradation of the crystals. The successful solution of the structure employed a Mo  $K\alpha$  intensity data set,  $I_b$ , obtained by taking averages on fixed-count, balanced-filter, hand-set (Morosin, 1965) data sets which individually had been approximately corrected

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for degradation. Degradation was followed by observing three to five different  $hkl$  intensities periodically and using a straight line through such points for each specific crystal specimen. The behavior from specimen to specimen (10–20% degradation) as well as for different  $hkl$  intensities (0–40% degradation) was not identical. In order to achieve the best estimate for the intensities, the quadrant of reciprocal space was scanned from different directions. Subsequently the  $\theta$ – $2\theta$  scan technique on an automated unit was used to measure another set of Mo  $K\alpha$  intensity data; shells of reciprocal space with increasing  $2\theta$  angle were measured with a

scan speed of  $\frac{1}{2}$  deg  $\text{min}^{-1}$ . Average intensity decrease for the reflections selected as standards during data collection was about 5% for this specimen. The data set ( $I_f$ ) used for final refinement of our structure consisted of scaling as a function of  $2\theta$ ,  $k(2\theta)$ , the stronger intensities of the previous balance filter data set ( $I_b$ ) with the corresponding radiation degradation corrected scanned values ( $I_s$ ) in the form  $I_f = 0.7I_s + 0.3k(2\theta)I_b$  when both  $I_s$  and  $I_b$  were observed and  $I_f = k(2\theta)I_b$  when  $I_s$  were unobserved. Over 100 intensities were 'flagged' for very poor agreement and values from the several collections were compared in order to estimate

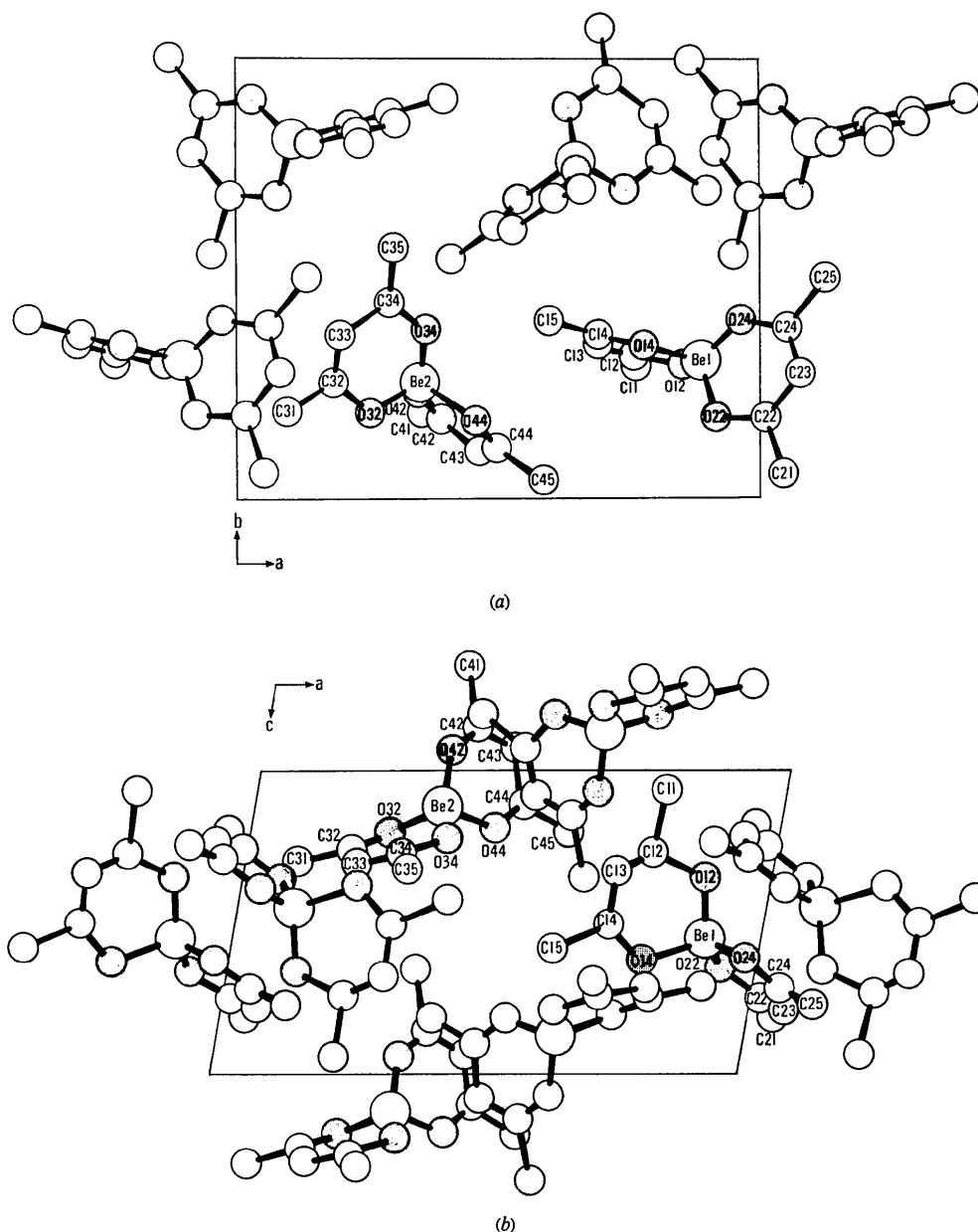


Fig. 1. (a) Projection along the  $c$  axis of the crystal structure of  $\text{Be}(\text{Acac})_2$ . The labeling scheme used in the text is shown. (b) Projection along the  $b$  axis.

the more probable value. This procedure yielded 2076  $hkl$  intensities of which 147 were considered as unobserved in subsequent calculations.

Various structure-solving techniques were investigated during a 'many-year', part-time effort. These included Fourier methods (Buerger, 1959), molecular packing (Williams, 1965), inequalities (Harker & Kasper, 1948; Killean, 1966) and sign relations (Sayre, 1952) on the  $hkl$  projection and other trial and error attempts. Using the newly implemented codes *APHASE* and *TANGEN* of X-RAY72, the structure was successfully solved. The details of these codes are given by Stewart *et al.* (1972). In brief, *APHASE* provides a method of carrying out the search for a productive set of starting phases similar to the symbolic addition procedures of Karle & Karle (1966) while *TANGEN* is used to carry out refinement and extension of the starting set by application of the tangent formula of Karle & Hauptman (1958). In this determination more effort than usual was required. *APHASE* pointed to 716, 904, and 10,0,7 as good origin-definition reflections. The first choices of an enantiomorph-setting reflection were poor and led to failures. However, when the 435 reflection was set to  $\pi/2$  and the various possibilities for the 12,1,3( $\pi$ ), 11,2,1( $3\pi/2$ ), 348( $7\pi/4$ ), 906( $\pi$ ), 615(0), 16,1,2(0) given by the *APHASE* program were utilized in *TANGEN*, a set of phases which revealed the complete structure was produced. The initial structure gave a conventional  $R$  value of 0.35.

The scattering factors used for C and O were taken from Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955), for Be from *International Tables for X-ray Crystallography* (1962) and for H from Stewart, Davidson & Simpson (1965). A Fourier synthesis using phases from the least-squares refinement of the nonhydrogen atoms yielded positions for hydrogen (Table 1). The hydrogen atoms were assigned the isotropic thermal parameters of the carbon atoms to which they were attached. In the full-matrix least-squares refinement, the function  $w(F_o - F_c)^2$  was minimized with  $w = n/\sigma^2$ . Corrections for extinction were not included. Final parameters obtained using anisotropic thermal parameters for the nonhydrogen atoms are listed in Table 2.\* The final residual index,  $R = \sum ||F_o| - |F_c|| / \sum |F_o|$ , is 0.066; the corresponding value for the weakest  $\frac{1}{3}$  of the observed reflections is 0.12. The labeling scheme and packing arrangement are shown in Fig. 1. The interatomic separations and angles are shown in Fig. 2. Values for the ligand separations and angles as well as for the anisotropic thermal parameters with larger values on methyl carbons compared with ring carbon or oxygen atoms are typical of metal-Acac compounds (Morosin, 1965, 1967; Morosin & Montgomery, 1969; Anderson, Neu-

Table 1. *Hydrogen atomic coordinates*

	$x$	$y$	$z$	$U$ ( $\times 10^{-2} \text{ \AA}^2$ )
H(11)	0.707	0.304	0.043	9.3
H(12)	0.812	0.279	0.043	9.3
H(13)	0.780	0.393	0.043	9.3
H(14)	0.646	0.350	0.283	6.4
H(15)	0.597	0.340	0.650	8.2
H(16)	0.622	0.457	0.650	8.2
H(17)	0.562	0.412	0.523	8.2
H(21)	0.073	0.029	0.718	9.2
H(22)	0.012	0.036	0.848	9.2
H(23)	0.105	0.064	0.870	9.2
H(24)	0.146	0.268	0.850	5.9
H(25)	0.176	0.493	0.690	7.7
H(26)	0.171	0.482	0.869	7.7
H(27)	0.098	0.572	0.739	7.7
H(31)	0.110	0.125	0.308	8.4
H(32)	0.056	0.232	0.217	8.4
H(33)	0.064	0.239	0.392	8.4
H(34)	0.154	0.418	0.348	6.0
H(35)	0.353	0.589	0.367	8.8
H(36)	0.276	0.589	0.392	8.8
H(37)	0.292	0.615	0.261	8.8
H(41)	0.415	0.178	-0.413	7.0
H(42)	0.315	0.171	-0.392	7.0
H(43)	0.358	0.279	-0.407	7.0
H(44)	0.492	0.077	-0.148	5.8
H(45)	0.623	0.088	0.261	6.6
H(46)	0.573	0.000	0.283	6.6
H(47)	0.653	0.011	0.130	6.6

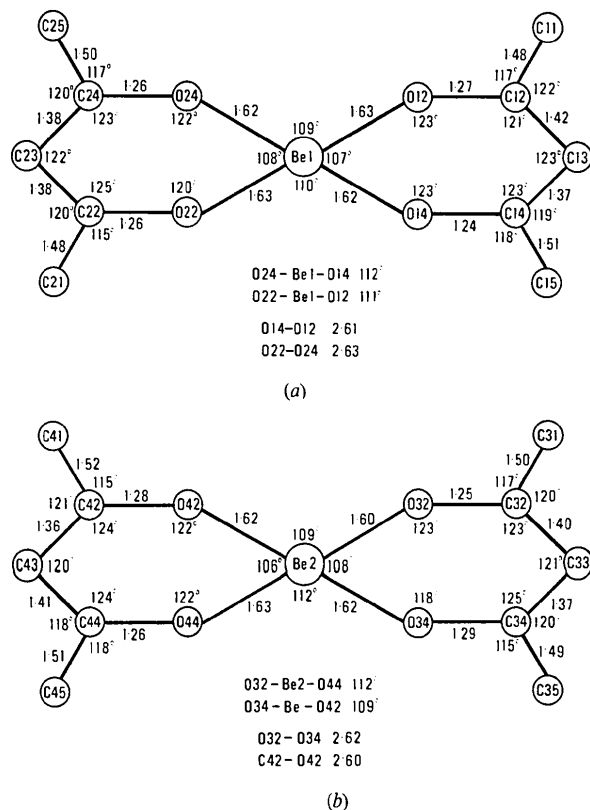


Fig. 2. Interatomic separations and angles for the two molecules in  $\text{Be}(\text{Acac})_2$ . Standard deviations in oxygen-carbon and oxygen-oxygen separations are 0.01 Å while those for the other separations are 0.02 Å; those for angles are 1°.

\* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30829 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

man & Melson, 1973; Dymock & Palenik, 1974). Furthermore, the average Be–O separation of 1.62 (1) Å (APS's values near 1.70 Å) is in agreement with sums of radii and with other tetrahedral Be–O separations of 1.610 (4) Å (Dance & Freeman, 1969) and 1.618 (4) Å (Sikka & Chidambaram, 1969) obtained in BeSO<sub>4</sub>·2H<sub>2</sub>O and 1.653 (1) Å obtained in beryl (Morosin, 1972). The most interesting aspect of this structure concerns the location of the beryllium atoms relative to the least-squares planes through the ring atoms of the chelate ligands. This result is summarized in Table 3 and is discussed below.

### Discussion

Since APS used inequality methods for their structure determination, it is not surprising that portions of molecules in their crystal structure are oriented similarly to that which we obtained. For example, in the *ab* projection our Be(1) molecule differs with respect to one of APS's molecules (denoted as Be' by APS) only in an alternate choice of origin (+½ along *x* allowed *y* shift) while no symmetry relationship is apparent for the other molecule; in the *ac* projection the relative orientations of our C(11)–C(15) and their

Table 2. *Final atomic coordinates and thermal factors*

$U_{ij}$  of the form  $\exp(-2\pi^2 \sum U_{ij} h_i h_j a_i^2)$  in units  $\times 10^{-2}$  Å<sup>2</sup>. For a connected molecule C(21)–C(25) require  $x+1.0$ .

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{11}$	$U_{22}$	$U_{33}$	$U_{12}$	$U_{13}$	$U_{23}$
Be(1)	0.887 (1)	0.318 (1)	0.560 (2)	3.0 (6)	5.1 (8)	6.1 (0.6)	-0.1 (6)	0.5 (6)	0.1 (7)
Be(2)	0.359 (1)	0.261 (1)	0.146 (2)	3.3 (5)	4.0 (7)	5.0 (6)	0.7 (5)	1.3 (5)	0.1 (5)
O(12)	0.8628 (6)	0.308 (1)	0.346 (1)	5.3 (5)	7.0 (6)	5.7 (4)	-0.7 (5)	0.7 (4)	-0.5 (5)
O(14)	0.7817 (5)	0.353 (1)	0.618 (1)	3.2 (3)	6.0 (5)	5.4 (4)	0.9 (3)	0.2 (3)	0.9 (4)
O(22)	0.9290 (5)	0.194†	0.648 (1)	3.3 (4)	4.8 (5)	6.4 (4)	0.0 (4)	-0.3 (3)	-0.0 (4)
O(24)	0.9727 (6)	0.4169 (9)	0.616 (1)	3.8 (4)	4.9 (5)	7.5 (5)	-0.5 (4)	-0.7 (3)	0.8 (4)
O(32)	0.2618 (6)	0.2031 (9)	0.203 (1)	3.8 (4)	5.2 (5)	5.6 (4)	-0.0 (4)	1.5 (3)	-0.1 (4)
O(34)	0.3701 (6)	0.3947 (8)	0.221 (1)	4.1 (4)	3.9 (4)	6.4 (4)	0.4 (3)	2.0 (3)	-0.7 (4)
O(42)	0.3437 (5)	0.2634 (8)	-0.0657 (9)	3.6 (3)	4.8 (4)	4.4 (3)	1.1 (4)	0.5 (3)	0.1 (3)
O(44)	0.4600 (5)	0.1842 (9)	0.2153 (9)	4.1 (4)	4.9 (4)	4.8 (4)	1.7 (4)	0.9 (3)	0.2 (3)
C(11)	0.765 (2)	0.318 (3)	0.063 (3)	11.5 (13)	9.4 (16)	9.4 (10)	-1.5 (14)	0.0 (10)	-2.3 (11)
C(12)	0.776 (1)	0.327 (1)	0.255 (2)	6.8 (7)	3.7 (6)	6.4 (6)	-0.9 (5)	-1.3 (6)	-0.3 (5)
C(13)	0.694 (1)	0.357 (1)	0.335 (2)	4.9 (6)	5.7 (7)	6.8 (6)	0.0 (6)	-2.2 (6)	-0.2 (6)
C(14)	0.7024 (7)	0.370 (1)	0.513 (2)	3.0 (5)	3.4 (5)	7.6 (6)	0.1 (4)	-0.3 (4)	0.8 (5)
C(15)	0.609 (1)	0.400 (2)	0.586 (2)	4.4 (6)	8.5 (10)	10.8 (11)	1.6 (8)	0.7 (0.7)	2.0 (10)
C(21)	0.047 (2)	0.070 (2)	0.812 (3)	8.8 (12)	7.3 (11)	9.5 (10)	2.9 (10)	-0.5 (9)	0.4 (10)
C(22)	0.0158 (8)	0.188 (1)	0.742 (1)	3.5 (5)	5.3 (7)	5.0 (4)	0.8 (5)	0.5 (4)	-0.7 (5)
C(23)	0.0803 (9)	0.282 (1)	0.781 (2)	3.1 (5)	7.1 (8)	6.3 (6)	0.1 (5)	-0.7 (5)	-1.2 (6)
C(24)	0.0551 (8)	0.394 (1)	0.714 (1)	2.9 (4)	6.6 (8)	4.8 (5)	-0.5 (5)	0.2 (4)	-0.4 (5)
C(25)	0.1277 (9)	0.493 (2)	0.759 (2)	5.4 (6)	8.2 (10)	8.2 (8)	-2.5 (8)	-0.0 (6)	-0.5 (8)
C(31)	0.106 (1)	0.199 (2)	0.298 (2)	6.4 (8)	11.1 (15)	10.1 (10)	-1.3 (10)	4.4 (7)	0.6 (11)
C(32)	0.1980 (8)	0.262 (1)	0.263 (1)	3.7 (5)	7.6 (9)	4.1 (5)	0.9 (6)	1.3 (4)	0.5 (5)
C(33)	0.2097 (9)	0.383 (1)	0.298 (1)	3.6 (5)	7.4 (8)	5.6 (6)	2.6 (6)	1.7 (5)	-0.0 (6)
C(34)	0.2941 (9)	0.441 (1)	0.273 (1)	5.5 (6)	5.3 (7)	3.9 (4)	2.8 (6)	0.6 (5)	-0.2 (5)
C(35)	0.306 (2)	0.569 (2)	0.316 (2)	12.2 (15)	5.3 (9)	8.1 (9)	2.5 (10)	2.2 (9)	-0.2 (7)
C(41)	0.363 (1)	0.201 (2)	-0.348 (2)	8.6 (9)	8.7 (11)	4.3 (5)	0.6 (10)	-0.4 (5)	-0.2 (7)
C(42)	0.393 (1)	0.194 (1)	-0.149 (1)	4.5 (5)	4.4 (6)	4.1 (4)	-0.7 (5)	0.8 (4)	-0.6 (5)
C(43)	0.468 (1)	0.122 (1)	-0.071 (1)	4.9 (6)	5.9 (7)	4.6 (5)	1.0 (6)	0.9 (4)	-1.6 (5)
C(44)	0.499 (1)	0.121 (1)	0.113 (1)	2.3 (4)	4.2 (6)	4.9 (5)	0.6 (4)	0.3 (4)	-0.3 (4)
C(45)	0.589 (1)	0.047 (2)	0.191 (2)	3.7 (6)	6.4 (7)	7.6 (7)	2.1 (6)	0.0 (6)	-0.0 (7)

† Parameter fixed for least-squares refinement.

Table 3. *Least-squares planes through ligand rings and atom deviations ( $\times 10^4$  Å)*

Plane	I	II	III	IV			
O(12)	-6	O(22)	-49	O(32)	-173	O(42)	-47
O(14)	-20	O(24)	33	O(34)	149	O(44)	26
C(12)	30	C(22)	67	C(32)	205	C(42)	71
C(13)	49	C(23)	-30	C(33)	-37	C(43)	-40
C(14)	46	C(24)	-22	C(34)	-144	C(44)	-9
Mean	38		48		171		49
Be(1)	63	Be(1)	-108	Be(2)	-2005	Be(2)	-2009
C(11)	338	C(21)	453	C(31)	822	C(41)	757
C(15)	-343	C(25)	-83	C(35)	51	C(45)	1078

Angles between planes

I, II 89.1°      III, IV 87.8°

C(1)–C(5) (not primed) chelate ring are similar, though the remainder of their molecule requires use of  $\beta^*$  for  $\beta$  and again the other molecule shows no symmetry-related correspondence. Considering the difficulties which preceded the final structure determination with our three-dimensional data, it is understandable why the projection-derived structure was incorrect.

An interesting feature of the structure concerns the slight difference in the geometry of the two types of molecules. The two molecules show great similarity in most details. The Be atoms are tetrahedrally bonded to the two acetylacetonate ligands. Such acetylacetonate ligands may be considered to be planar groups which, including the Be atom *via* two of its bonds, form a closed conjugated ring. When the planes of these rings are calculated, an interesting feature of the geometry of the molecules may be observed, *i.e.*, Be(2) is situated off each of the least-squares planes through its two different chelate rings by about 0.20 Å, an amount typical of some metal–Acac compounds (Morosin, 1967), whereas Be(1) lies on its planes. Table 3 shows the results of a least-squares plane calculation on the two molecules. It is our conjecture that this difference may result from the packing of the molecules. Table 4 lists the intermolecular contact separations which are less than 3.65 Å. One notes that the shorter nearest neighbor interactions involving molecule Be(2) are primarily directed to one side of the chelate ring; in fact, interactions 1, 2, and 3 (Table 4) are primarily into the same vicinity of the same side of the face of the ligand plane, *i.e.*, to C(31), C(32), and O(32) of the C(31)–C(35) chelate ring, whereas 4, 7, and 10 are into the edge of this particular ligand plane. Interaction 8, involving the other side of the molecule, *i.e.*, C(41)–C(45) ring, is into the face of this particular ligand and, with

the symmetry-related, redundant interaction, essentially ‘holds’ the molecule in place while 9 is only into the edge of this ligand plane. (Interactions 5 and 6 involve only molecule Be(1) and act on opposite faces of the C(11)–C(15) ligand; interactions into the face of the other ligand of this molecule are all longer than 3.68 Å, *i.e.*, the shorter interactions 2, 7, and 9 are ‘edge’ interactions as far as molecule Be(1) is concerned.) From such near neighbor interactions it appears that the torque applied to molecule Be(2) evidently twists it, resulting in the metal atoms being ‘popped out’ of the chelate ring.

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Table 4. Intermolecular contact separations\*

A–B	Distance (Å)	Symmetry operation on B	
		x	y
1. C(32)–C(41)	3.474	x	y
2. O(24)–C(31)	3.488	$1-x$	$\frac{1}{2}+y$
3. O(32)–C(41)	3.500	x	y
4. C(35)–O(14)	3.514	$1-x$	$\frac{1}{2}+y$
5. O(14)–C(11)	3.520	x	y
6. O(12)–C(21)	3.526	$2-x$	$\frac{1}{2}+y$
7. O(35)–O(22)	3.539	$1-x$	$\frac{1}{2}+y$
8. O(42)–C(45)	3.543	$1-x$	$\frac{1}{2}+y$
9. O(42)–C(23)	3.550	$-1+x$	y
10. O(12)–C(31)	3.600	$-1+x$	y

\* Redundant values, such as C(41)–C(32) for 1 above involving a different symmetry operation ( $x, y, -1+z$ ), are not listed.