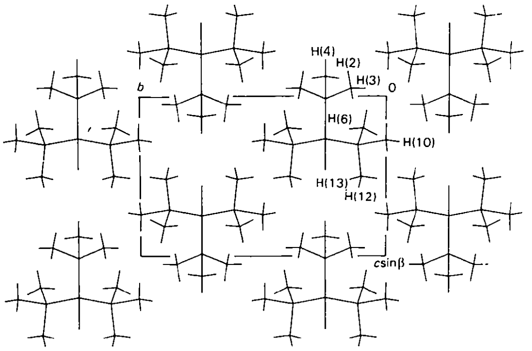

Fig. 2. Packing of adjacent molecules viewed along **a**.

compound. The molecular packing is shown in Fig. 2. The molecules pack in interlocking layers along **b** with the iodine 'head' fitting into the $(PMe_3)_3$ 'tail'. The closest contacts within the layers are H(1)···H(6)-$(1+x,y,z)$ 2·59 and H(2)···H(12)$(x,y,-1+z)$ 2·56 Å. Between layers the closest contacts are H(11)···H(12)-$(-x,-y,1-z)$ 2·45 and H(14)···H(14)$(-1-x,-y,1-z)$ 2·54 Å.

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# Structure of Bis(acetylacetonato)beryllium(II), $Be(C_5H_7O_2)_2$, at 119 K

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**Abstract.** $M_r = 207{\cdot}2$, monoclinic, $P2_1$, $a = 13{\cdot}437$ (1), $b = 11{\cdot}196$ (2), $c = 7{\cdot}656$ (1) Å, $\beta = 100{\cdot}79$ (1)°, $V = 1131{\cdot}4$ (3) Å$^3$, $Z = 4$, $D_x = 1{\cdot}22$ g cm$^{-3}$, Mo $K\alpha$, $\lambda = 0{\cdot}7107$ Å, $\mu = 0{\cdot}94$ cm$^{-1}$, $F(000) = 452$, $T = 119$ (1) K, final $R = 0{\cdot}037$ for 3114 unique observed reflections. The two independent molecules are similar and each Be atom is chelated in a distorted tetrahedron by the two ligands with a Be—O distance of 1·618 (5) Å and an O—Be—O angle of 107·5 (4)°. The chelate rings of one molecule are planar while those of the other molecule are folded about the bites by 13·7 (2) and 8·7 (3)°.

**Introduction.** The structure of the title compound has been determined at room temperature (Stewart & Morosin, 1975). However, examination of this structure shows that equivalent bond distances are distributed over wide ranges, for example, for O—C from 1·24 (1) to 1·29 (1) Å. Stewart & Morosin collected various data sets over several years because of instrumental changes and serious radiation degradation of crystals for different specimens. Their structure analysis was based on 1929 averaged intensities of these data sets, which were corrected for degradation individually. As part of our structural studies on β-diketonato complexes, the refinement of the title complex was undertaken at low temperature in order to obtain more accurate information concerning the molecular geometry and the location of H atoms, and further to compare the molecular structure with that observed by gas electron diffraction (Shibata, Ohta & Iijima, 1980).

**Experimental.** Colorless crystals of title compound obtained by crystallization from benzene. Crystal specimen cooled by blowing cold $N_2$ gas evaporated from liquid $N_2$ and kept at 119 (1) K. Lattice constants by least-squares refinement of setting angles of 50 reflections ($20 < 2\theta < 30°$). Cell-volume contraction on cooling 3·7%. Graphite-monochromated Mo $K\alpha$ radiation, Rigaku four-circle diffractometer AFC-5.

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Crystal dimensions 0·47 × 0·39 × 0·31 mm. Intensities to 70° in $2\theta$; $\theta$–$2\theta$ scans, scan range $(1\cdot2 + 0\cdot5\tan\theta)°$, scan speed 3° min$^{-1}$ in $2\theta$; octants ($h$ 0–21, $k$ 0–18, $l$ −12–12); 5371 reflections measured, 3114 unique with $|F_o| > 3\sigma(F_o)$. Data corrected for Lorentz and polarization effects, not for absorption. Three standard reflections, no significant loss in intensity throughout data collection. Room-temperature positional parameters served as starting set for refinement with *UNICS*III program system (Sakurai & Kobayashi, 1979); atomic scattering factors for Be, C and O from *International Tables for X-ray Crystallography* (1974), for H from Stewart, Davidson & Simpson (1965). All H atoms located on difference-Fourier maps. Positional and anisotropic thermal parameters (isotropic for H atoms) refined by block-diagonal least squares, weighting scheme $w = [\sigma^2(F_o) + (0{\cdot}015F_o)^2]^{-1}$. $\sum w(F_o - F_c)^2$ minimized. Refinement converged to $R = 0{\cdot}037$ and $wR = 0{\cdot}040$; $(\Delta/\sigma)_{\max} = 0{\cdot}1$; highest peak in final difference Fourier map 0·16 e Å$^{-3}$. Final atomic parameters are in Table 1.[*] The numbering scheme is the same as that of Stewart & Morosin (1975).

**Discussion.** The crystals of the title compound contain two independent molecules (I and II), in which the Be atom is chelated *via* O atoms by two acetylacetone ligands. The bond distances and angles for each molecule are given in Fig. 1. The geometry around the Be atom is distorted tetrahedral with O—Be—O angles in the range 106·9 (2)–112·6 (2)°. The average angle of the intrachelate O—Be—O is 107·5 (4)° and that of the interchelate is 110·5 (15)°. The values of non-bonded O···O distances and O—Be—O angles are given in Table 2. The average value of the O···O bite [2·609 (3) Å] is shorter than that of the non-bite [2·657 (18) Å]. The Be—O distances are in the range 1·607 (3)–1·626 (3) Å, average value 1·618 (5) Å, and are in agreement with the values found in the crystal structures of tetrahedral oxalato (Jaber, Faure & Loiseleur, 1978) and malonato (Duc, Faure & Loiseleur, 1978) complexes of Be. The ligands have the following average distances and angles: O—C, 1·282 (5); C—C, 1·391 (5); C—$CH_3$, 1·499 (2) Å; O—C—C, 122·9 (3); O—C—$CH_3$, 120·7 (5)°. For e.s.d.'s of average values throughout this paper, see footnote to Table 2.

The molecular conformation obtained is essentially the same as that found at room temperature (Stewart & Morosin, 1975). However, the accuracy of the structural parameters is improved significantly, the e.s.d.'s of

Table 1. *Positional parameters* (×10$^4$) *and equivalent isotropic temperature factors, $B_{eq}$*(Å$^2$),*for non*-H *atoms*

$$B_{\rm eq} = \tfrac{4}{3}\textstyle\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

| | $x$ | $y$ | $z$ | $B_{\rm eq}$ |
|---|---|---|---|---|
| O(12) | 8593 (1) | 3058 (1) | 3436 (2) | 2·2 |
| O(14) | 7795 (1) | 3560 (1) | 6196 (2) | 1·8 |
| O(22) | 9265 (1) | 1940 | 6505 (2) | 2·0 |
| O(24) | 9703 (1) | 4189 (1) | 6150 (2) | 2·1 |
| O(32) | 2607 (1) | 2025 (1) | 2096 (2) | 1·8 |
| O(34) | 3725 (1) | 3942 (1) | 2276 (2) | 1·8 |
| O(42) | 3415 (1) | 2620 (1) | −642 (2) | 1·7 |
| O(44) | 4594 (1) | 1794 (1) | 2198 (2) | 1·7 |
| C(11) | 7612 (2) | 3142 (3) | 528 (3) | 3·4 |
| C(12) | 7715 (1) | 3263 (2) | 2508 (2) | 2·0 |
| C(13) | 6892 (1) | 3578 (2) | 3264 (3) | 2·2 |
| C(14) | 6967 (1) | 3712 (2) | 5090 (2) | 1·7 |
| C(15) | 6060 (1) | 4037 (2) | 5870 (3) | 2·6 |
| C(21) | 10452 (2) | 640 (2) | 8214 (3) | 2·8 |
| C(22) | 10150 (1) | 1856 (2) | 7481 (2) | 1·9 |
| C(23) | 10809 (1) | 2817 (2) | 7844 (3) | 2·2 |
| C(24) | 10557 (1) | 3950 (2) | 7159 (2) | 1·9 |
| C(25) | 11279 (2) | 4973 (2) | 7572 (3) | 2·8 |
| C(31) | 1007 (2) | 2069 (3) | 2964 (3) | 3·1 |
| C(32) | 1951 (1) | 2673 (2) | 2654 (2) | 1·9 |
| C(33) | 2093 (1) | 3896 (2) | 2980 (2) | 2·1 |
| C(34) | 2983 (1) | 4470 (2) | 2809 (2) | 1·8 |
| C(35) | 3130 (2) | 5773 (2) | 3241 (3) | 2·7 |
| C(41) | 3580 (2) | 1971 (2) | −3516 (3) | 2·6 |
| C(42) | 3901 (1) | 1937 (2) | −1531 (2) | 1·7 |
| C(43) | 4689 (1) | 1196 (2) | −720 (2) | 1·9 |
| C(44) | 5013 (1) | 1176 (2) | 1117 (2) | 1·6 |
| C(45) | 5905 (1) | 427 (2) | 1917 (3) | 2·2 |
| Be(1) | 8841 (2) | 3186 (2) | 5586 (3) | 1·8 |
| Be(2) | 3605 (2) | 2594 (2) | 1517 (3) | 1·6 |

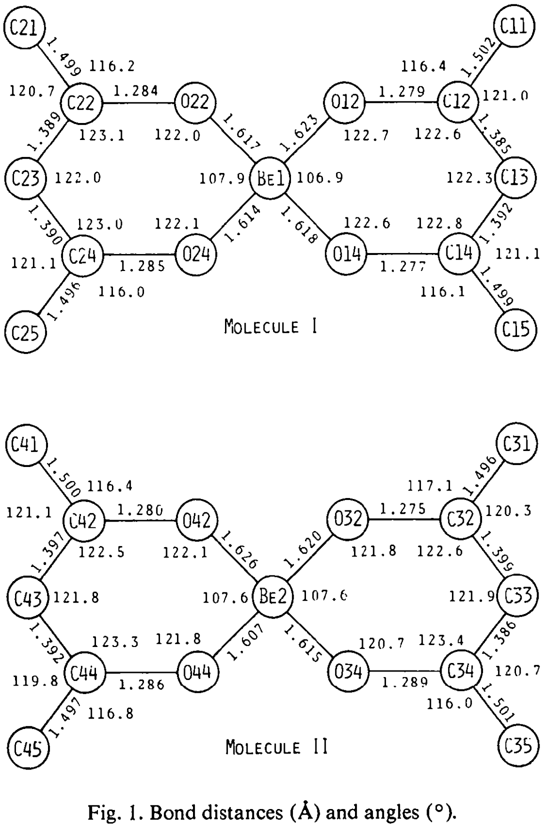

Fig. 1. Bond distances (Å) and angles (°).

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[*] Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication no. SUP 42221 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Interatomic distances and angles*

(*a*) The O···O contacts (Å)

| | | | |
|---|---|---|---|
| O(12) O(14) | 2·604 (2) | O(12) O(22) | 2·666 (2) |
| O(22) O(24) | 2·612 (2) | O(12) O(24) | 2·644 (2) |
| O(32) O(34) | 2·610 (2) | O(14) O(22) | 2·660 (2) |
| O(42) O(44) | 2·608 (2) | O(14) O(24) | 2·666 (2) |
| Average | 2·609 (3)* | O(32) O(42) | 2·621 (2) |
| | | O(32) O(44) | 2·669 (2) |
| | | O(34) O(42) | 2·648 (2) |
| | | O(34) O(44) | 2·680 (2) |
| | | Average | 2·657 (18) |

(*b*) Angles (°)

| | | | |
|---|---|---|---|
| O(12) Be(1) O(22) | 110·7 (2) | O(12) Be(1) O(24) | 109·5 (2) |
| O(14) Be(1) O(22) | 110·6 (2) | O(14) Be(1) O(24) | 111·2 (2) |
| O(32) Be(2) O(42) | 107·8 (1) | O(32) Be(2) O(44) | 111·7 (2) |
| O(34) Be(2) O(42) | 109·6 (2) | O(34) Be(2) O(44) | 112·6 (2) |
| | Average | 110·5 (15) | |

(*c*) The average values of bond distances (Å) and angles (°) involving H with their ranges

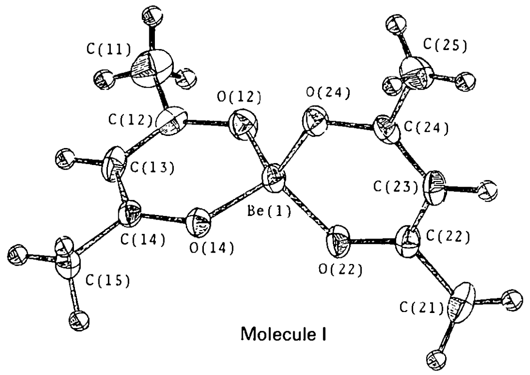
| | | |
|---|---|---|
| C—H | 0·96 (5) | 0·82–1·06 (4) |
| CCH (methyl) | 111 (2) | 106–117 (3) |
| CCH (ring) | 119 (2) | 115–123 (2) |
| HCH | 108 (7) | 95–125 (4) |

* The e.s.d.'s of the average values have been calculated by $[\sum_i(x_i-\bar{x})^2/(n-1)]^{1/2}$.

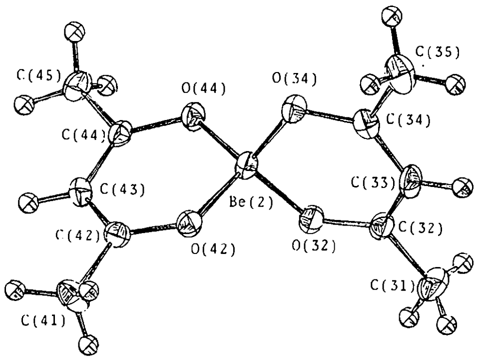
the bond distances and angles not containing H being now 0·003 Å and 0·2°, respectively, the corresponding values at room temperature being 0·01 Å and 1°. The average values of C—H, C—C—H and H—C—H are given in Table 2 with their ranges. Each of the C—H bonds in the eight methyl groups staggers the adjacent O—C bond, as shown in the *ORTEP* (Johnson, 1965) drawing, Fig. 2. The OCCCO skeletons of all four ligands are planar [maximum displacement of an atom from the least-squares plane is 0·014 (2) Å], but the Be-atom displacements from the planes are 0·230 (2) and 0·147 (2) Å for molecule (II) at low temperature while the corresponding values at room temperature were equal, 0·20 Å. The displacements at low temperature are equivalent to folding of the ligands about the bite by 13·7 and 8·7°. Deviations of metal atoms from the acetylacetone planes have been found in many crystals (Morosin, 1967; Ito, Toriumi, Ueno & Saito, 1980). However, such folding has not been observed in molecule (I) at low temperature or at room temperature.

The average distances and angles are in excellent agreement with those for the molecule in the gas phase. This shows that the structures of complexes that are monomeric in the solid phases will be the same as those for free molecules in gas phases (Shibata, Ohta & Iijima, 1980). However, the folding observed in molecule (II) in the solid phase was not detected in the gaseous free molecule and therefore seems to be due to the crystal-packing forces.

All of the intermolecular distances are shorter at low temperature than at room temperature. For example, the distances O(32)···C(41)($x, y, 1+z$) and O(14)···C(11)($x, y, 1+z$) contracted by 0·13 and 0·12 Å, respectively, on cooling. The shortest intermolecular contact between H atoms observed in the present work is 2·52 Å, which is slightly longer than the sum of the van der Waals radii, 2·4 Å.



Molecule I



Molecule II

Fig. 2. *ORTEP* (Johnson, 1965) drawings of molecules (I) and (II).

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