

Fig. 2. Stereo drawing of copper polyhedra around Hf(2) ( $z=1$ ) and Hf(1) ( $z \approx \frac{2}{3}, \frac{4}{3}$ ).

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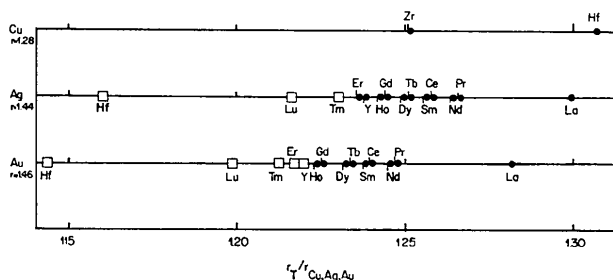


Fig. 3. Radius-ratio values for compounds with  $GdAg_{3.6}$  (circles) and  $MoNi_4$  or  $ZrAu_4$  structure types (squares). Metallic radii for coordination number 12 according to the periodic table of elements of Sargent–Welch.

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## 1,2,3,4,4a,5,11a-Heptahydroacetoxy-11βH-dibenz[b,e]azepine-6-one

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**Abstract.**  $C_{16}H_{19}NO_3$ , monoclinic,  $P2_1/c$ ,  $a=9.462$  (4),  $b=16.082$  (6),  $c=10.280$  (6) Å,  $\beta=110.9$  (1)°,  $Z=4$ ,  $D_{calc}=1.24$  g cm<sup>-3</sup>. The structure was solved by the symbolic addition procedure and refined by full-matrix least-squares calculations to a final  $R$  value of 4.8%. The molecule has a *cis* junction between the seven-membered ring and the saturated six-membered ring and the peptide group which is part of the seven-membered ring is planar.

**Experimental.** One method of preparing benzazepinones is by carrying out a Norrish type II photoreaction (Wagner, 1971) on *N*-alkylated phthalimides (Kana-

oka, Migita, Koyama, Sata, Nakai & Mizoguchi, 1973). The benzazepinone to be discussed here was obtained as one of the products in such a reaction (Kanaoka, Koyama, Flippen, Karle & Witkop, 1974). Crystals used in the X-ray analysis were provided by Dr B. Witkop of the National Institutes of Health. An automatic computer-controlled diffractometer was used to collect 2346 independent reflections from a colorless crystal ( $\sim 0.40 \times 0.45 \times 0.16$  mm) with  $Cu K\alpha$  radiation ( $\lambda=1.54178$  Å, Ni filter). Data were collected by the  $\theta$ - $2\theta$  scanning technique (max  $\sin \theta/\lambda=0.521$ ) over a scan width of  $1.75^\circ$  and at a scanning speed of  $2^\circ$  min<sup>-1</sup>.

Table 1. *Fractional coordinates and thermal parameters with standard deviations*

The thermal parameters are of the form  $T = \exp [-\frac{1}{4}(B_{11}h^2a^{*2} + B_{22}k^2b^{*2} + B_{33}l^2c^{*2} + 2B_{12}hka^*b^* + 2B_{13}hla^*c^* + 2B_{23}klb^*c^*)]$ . Standard deviations are based solely on least-squares parameters.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> <sub>11</sub>	<i>B</i> <sub>22</sub>	<i>B</i> <sub>33</sub>	<i>B</i> <sub>12</sub>	<i>B</i> <sub>13</sub>	<i>B</i> <sub>23</sub>
N	0.47853 (15)	0.52722 (9)	0.17234 (14)	3.77 (6)	3.46 (6)	3.28 (6)	-0.34 (5)	1.62 (5)	-0.44 (5)
O(1)	0.64701 (15)	0.57085 (8)	0.07607 (13)	5.62 (7)	4.24 (6)	4.41 (6)	-1.05 (5)	3.12 (6)	-0.92 (5)
O(2)	0.33848 (16)	0.74251 (8)	0.33015 (15)	6.82 (8)	3.74 (6)	5.19 (7)	1.16 (6)	3.40 (6)	-0.27 (5)
O(3)	0.30847 (22)	0.81805 (11)	0.14051 (20)	9.94 (12)	5.96 (9)	6.73 (10)	3.07 (8)	4.07 (9)	1.91 (8)
C(1)	0.59595 (20)	0.57399 (11)	0.17185 (18)	4.09 (8)	3.23 (7)	3.75 (8)	0.26 (6)	1.75 (7)	-0.11 (6)
C(2)	0.66696 (20)	0.63093 (11)	0.29285 (18)	4.22 (8)	3.33 (7)	3.57 (8)	-0.27 (6)	1.49 (6)	-0.07 (6)
C(3)	0.57880 (21)	0.68011 (11)	0.34752 (18)	5.05 (9)	3.09 (7)	3.39 (7)	-0.35 (7)	1.74 (7)	-0.08 (6)
C(4)	0.40795 (21)	0.67587 (11)	0.27981 (19)	4.91 (9)	3.25 (7)	3.79 (8)	0.57 (7)	2.22 (7)	-0.21 (6)
C(5)	0.33613 (21)	0.59479 (11)	0.30605 (18)	4.41 (9)	3.72 (8)	3.77 (8)	0.01 (6)	1.99 (7)	0.01 (6)
C(6)	0.43261 (19)	0.51995 (11)	0.29500 (17)	4.16 (8)	3.39 (7)	3.26 (7)	-0.11 (6)	1.70 (6)	0.03 (6)
C(7)	0.35105 (22)	0.43683 (12)	0.29091 (20)	5.02 (9)	3.66 (8)	4.76 (9)	-0.42 (7)	2.37 (8)	0.04 (7)
C(8)	0.18947 (23)	0.43507 (13)	0.18772 (22)	4.93 (10)	5.10 (10)	4.96 (10)	-1.13 (8)	2.12 (8)	-0.58 (8)
C(9)	0.09838 (23)	0.50637 (14)	0.21524 (24)	4.29 (9)	5.61 (11)	6.19 (12)	-0.41 (8)	2.32 (8)	0.03 (9)
C(10)	0.17199 (23)	0.58925 (13)	0.20549 (24)	4.18 (9)	5.01 (10)	6.35 (12)	0.36 (7)	2.26 (8)	0.71 (9)
C(11)	0.82427 (23)	0.63634 (13)	0.34755 (23)	4.51 (9)	4.86 (10)	5.23 (10)	-0.53 (8)	1.62 (8)	-0.26 (8)
C(12)	0.89479 (26)	0.68960 (16)	0.45800 (25)	5.08 (11)	6.45 (13)	5.80 (12)	-1.44 (10)	0.95 (9)	-0.57 (10)
C(13)	0.80827 (30)	0.73765 (15)	0.51251 (25)	7.04 (14)	5.52 (12)	4.82 (11)	-2.04 (10)	1.14 (10)	-1.08 (9)
C(14)	0.65082 (27)	0.73356 (13)	0.45728 (22)	7.07 (13)	3.79 (9)	4.34 (9)	-0.89 (8)	2.32 (9)	-0.75 (7)
C(15)	0.29184 (22)	0.80951 (12)	0.24873 (25)	3.94 (8)	3.65 (9)	5.95 (11)	0.36 (7)	1.59 (8)	-0.50 (8)
C(16)	0.21849 (28)	0.87055 (15)	0.31309 (30)	6.85 (13)	5.13 (12)	8.50 (16)	1.56 (10)	2.82 (12)	-1.35 (11)

The structure was solved by routine application of the symbolic addition procedure for centrosymmetric crystals (Karle & Karle, 1966) with the help of programs written by R. D. Gilardi and S. A. Brenner of this laboratory. Full-matrix least-squares methods (Busing *et al.*, 1971) were used to refine the structure with the atomic scattering factors listed in the *International Tables for X-ray Crystallography* (1962). Data for which  $|F_o| < 3.0\sigma|F_o|$  (259 reflections) were given zero weight and omitted from the refinement. The remaining 2177 reflections were weighted according to the procedure outlined by Gilardi (1973) and the function minimized was  $\sum w(|F_o| - |F_c|)^2$  where  $w =$

Table 1 (cont.)

	<i>x</i>	<i>y</i>	<i>z</i>
H(N)	0.425	0.493	0.088
H(C4)	0.384	0.679	0.177
H(C5)	0.345	0.596	0.412
H(C6)	0.536	0.521	0.380
H(C7)	0.415	0.395	0.279
H(C7)	0.347	0.428	0.386
H(C8)	0.184	0.438	0.087
H(C8)	0.139	0.375	0.194
H(C9)	0.099	0.499	0.317
H(C9)	-0.018	0.508	0.146
H(C10)	0.164	0.597	0.100
H(C10)	0.114	0.639	0.228
H(C11)	0.885	0.598	0.300
H(C12)	0.015	0.692	0.495
H(C13)	0.865	0.775	0.592

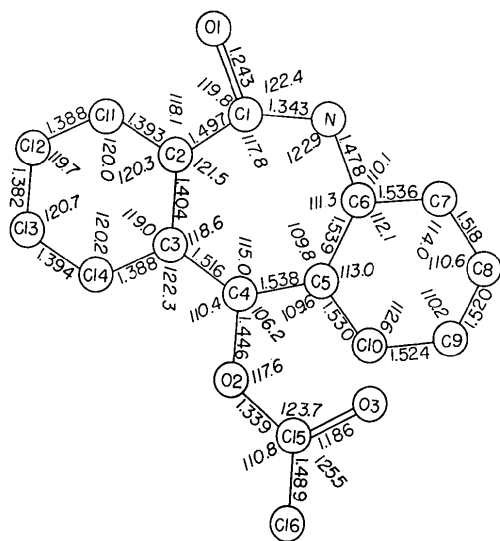


Fig. 1. Bond distances and angles. The numbering scheme is arbitrary and not related to the IUPAC numbering used in the title. Standard deviations are on the order of 0.003 Å for bond lengths and 0.2° for angles.

weight. Hydrogen atoms were located in a difference map, assigned isotropic thermal parameters equal to the final isotropic value of the atom to which they were bonded, and included in the refinement as constant parameters. The final *R* value for the data used in the refinement was 0.048 ( $R_w = 0.044$ ) and for the full set of data the final *R* value was  $R = 0.063$  ( $R_w = 0.062$ ). In Table 1 are listed the final refined coordinates and thermal parameters for the non-hydrogen atoms and the coordinates for the hydrogen atoms as obtained from a difference map.\*

**Discussion.** Fig. 1 illustrates the configuration of the molecule. The aromatic ring plus atoms C(1) and C(4) of the seven-membered ring are coplanar. The saturated

\* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30725 (11 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1 NZ, England.

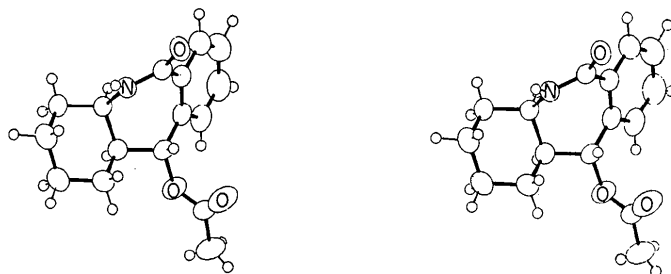


Fig. 2. Configuration of the molecule. The non-hydrogen atoms are shown at their final refined coordinates with anisotropic thermal parameters. The hydrogens are displayed at their difference-map coordinates with arbitrary isotropic thermal factors.

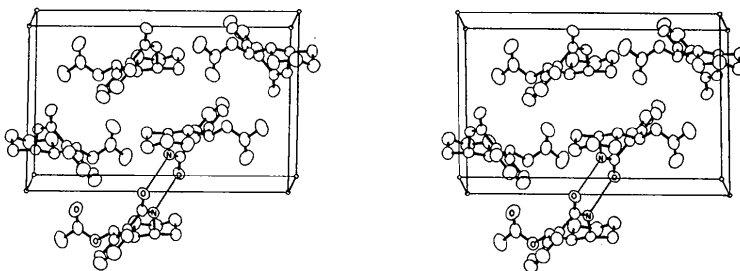


Fig. 3. Molecular packing. The N-H...O hydrogen bond is illustrated across the center of symmetry at  $\frac{1}{2}, \frac{1}{2}, 0$ . The view is seen looking down  $a$  with  $b \rightarrow$  and  $c \uparrow$ .

six-membered ring has a normal chair conformation [average absolute value for the ring torsion angles is  $52.6(2)^\circ$ ]. The seven-membered ring is in a boat conformation. (Torsion angles for this ring are listed in Table 2.) The hydrogens on C(5) and C(6) are on the same side of the fused ring system defining a *cis* junction between the seven-membered ring and the saturated ring. The acetate moiety is planar and extended away from the seven-membered ring. The peptide group [C(2), C(1), O(1), N, and C(6)] is planar. Bond distances and angles for this molecule are illustrated in Fig. 2. The C-C and C-N single bonds which are contiguous to either the aromatic ring or a carbonyl group are shortened because of the conjugative effects of these moieties. The average C-H bond length is  $1.02 \text{ \AA}$  and the N-H distance is  $1.00 \text{ \AA}$ . The two carbonyl bonds [C(1)-O(1) at  $1.243(2) \text{ \AA}$  and C(15)-O(3) at  $1.186(2) \text{ \AA}$ ] have significantly different lengths. This is due to the participation of O(1) as the acceptor in a strong N-H...O intermolecular hydrogen bond ( $H \cdots O = 1.88$ ,  $N \cdots O = 2.87 \text{ \AA}$ ,  $N-H \cdots O = 171.4^\circ$ ) which links the molecules into pairs across a center of symmetry (see Fig. 3). Both stereo drawings were done with *ORTEP* (Johnson, 1965). The hydrogen bond is the only intermolecular approach less than conventionally quoted van der Waals' separations.

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Table 2. *Torsion angles* ( $^\circ$ )

The numbers in parentheses are the estimated standard deviations in the last significant figure.

N—C(1)—C(2)—C(3)	-44.3 (2)	} Seven-membered ring
C(1)—C(2)—C(3)—C(4)	-1.0 (2)	
C(2)—C(3)—C(4)—C(5)	71.9 (2)	
C(3)—C(4)—C(5)—C(6)	-41.6 (2)	
C(4)—C(5)—C(6)—N	-44.7 (2)	
C(5)—C(6)—N—C(1)	81.0 (2)	
C(6)—N—C(1)—C(2)	-9.7 (2)	
C(7)—C(6)—C(5)—C(4)	-168.5 (1)	
N—C(6)—C(5)—C(10)	78.1 (1)	

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