HAMOR, T. A., CAUGHEY, W. S. & HOARD, J. L. (1965). *J. Chem. Phys.* **43**, 3100.
HOARD, T. L., HAMOR, M. J., HAMOR, T. A. & CAUGHEY, W. S. (1965). *J. Amer. Chem. Soc.* **87**, 2312.
International Tables for X-ray Crystallography (1962). Vol. III. Birmingham: Kynoch Press.
KOMIYAMA, Y. & LINGAFELTER, E. C. (1964). *Acta Cryst.* **17**, 1145.
LADELL, J. & KATZ, J. L. (1954). *Acta Cryst.* **7**, 460.
MAZZI, F. (1953). *R. C. Soc. Mineral, Ital.* **9**, 148.
PFIEFFER, P., HESSE, T., PFITZINGER, H., SCHOLL, W. & THERIOT, H. (1937). *J. Prakt. Chem.* **149**, 217.
SILVERS, S. J. & TULINSKY, A. (1967). *J. Amer. Chem. Soc.* **89**, (13), 3331.
SRIVASTAVA, R. C. & LINGAFELTER, E. C. (1966). *Acta Cryst.* **20**, 918.
STAKLEBERG, M. VON (1947). *Z. Inorg. Chem.* **253**, 136.
SUGIHARA, A., ASHIDA, T., SASADA, Y. & KAKUDO, M. (1968). *Acta Cryst.* B**24**, 203.
WERNER, P. (1964). *Acta Chem. Scand.* **18**, 1851.
WILSON, A. J. C. (1942). *Nature, Lond.* **150**, 152.

Acta Cryst. (1971). B27, 1649

The Crystal Structure of α-Bromoacetophenone

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The crystal structure of α-bromoacetophenone has been determined using three-dimensional X-ray diffraction data. The compound crystallizes in the orthorthombic system with $a=9{\cdot}74_3$, $b=18{\cdot}93_5$, $c=4{\cdot}22_2$ Å. Space group is $P2_12_12_1$, with four molecules in the unit cell. The structure has been refined by the least-squares method with individual anisotropic temperature factors for each atom to an R value of 0·107. In the crystal, the molecules are loosely separated by van der Waals distances all greater than 3 Å, the molecules themselves being arranged in two distinct layers, making an angle of ~122° with each other.

The spatial geometry of the acetophenone group and Br–C linkages in the group have not been reported before. The present structure analysis was attempted with this objective in view, as part of a larger programme in this department for determining the crystal structures of simple organic molecules.

The compound α-bromoacetophenone ($C_6H_5CO.CH_2.Br$) or phenacyl bromide crystallizes as colourless

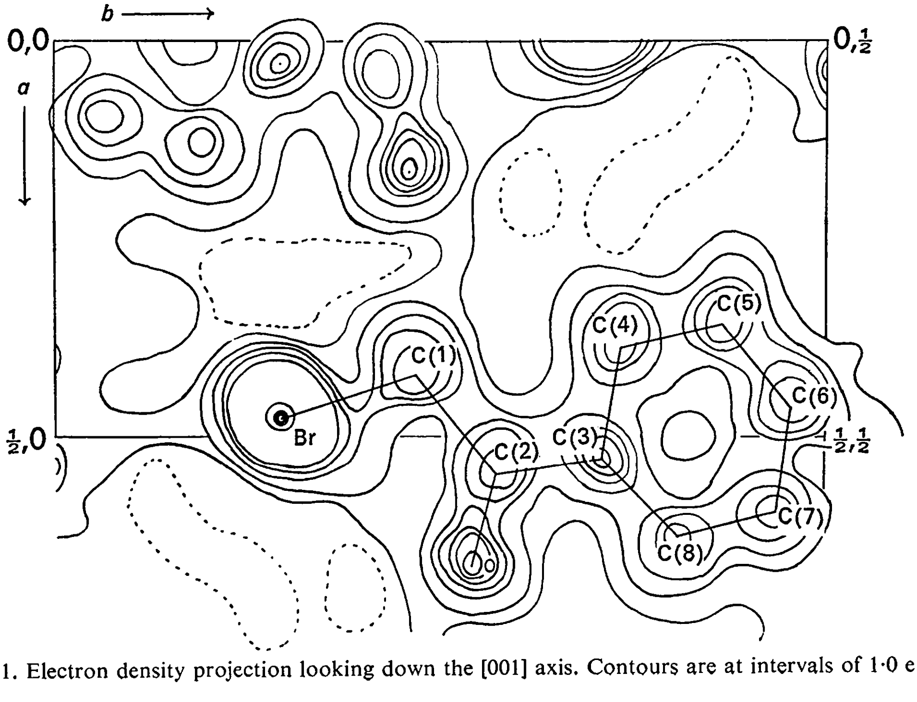

Fig. 1. Electron density projection looking down the [001] axis. Contours are at intervals of 1·0 e.Å².

elongated plates, (010) being the plate face. When exposed to the atmosphere, the crystals decompose leaving an oily brown liquid with a pungent and irritating smell. The long edge of the crystal corresponds to the *c* axis (4·22 Å), the shortest in the crystal.

Crystal data

The lattice parameters were determined using high $\sin\theta$ reflexions on normal-beam Weissenberg photographs on which pure silver lines were superimposed. All experimental work had to be done with the crystals sealed in thin-walled (diameter 1 mm) Lindemann glass capillary tubes.

The unit-cell parameters were:

$a=9{\cdot}74_3$ Å
$b=18{\cdot}93_5$
$c=4{\cdot}22_2$
ϱ_{obs} (powder) $=1{\cdot}647$ g.cm^{-3}
$\varrho_{calc}=1{\cdot}70_8$ g.cm^{-3}
$Z=4$

The only absences observed were *h*00, 0*k*0, 00*l* when *h*, *k* or *l* were odd. This fixes the space group uniquely as $P2_12_12_1$.

Reflexions for structure analysis were collected by normal-beam and equi-inclination photography and because of the necessity to work with crystals sealed in Lindemann glass capillary tubes, the data had to be necessarily limited. The crystals were small (0·05 × 0·03 × 0·02 cm in size.) Roughly about 60% of the theoretically possible reflexions were observed. The intensities were estimated visually, corrected for the geometrical factors (Lorentz–polarization factor, spot-shape) and put on an absolute scale using statistical methods.

Structure determination and refinement

Although the space group is non-centrosymmetric, the projections can all be made centrosymmetric by shifting the origins on to the respective screw axes. Since the *c* axis was the shortest and the structure contained a heavy bromine atom, the heavy-atom technique was

Table 1. *Observed and calculated structure-factor magnitudes*

h	k	l	/F_o/	/F_c/
0	2	0	44.7	52.5
0	4	0	60.3	69.3
0	6	0	42.1	46.5
0	8	0	54.5	54.9
0	10	0	76.2	73.1
0	14	0	36.9	31.3
0	16	0	23.6	16.7
0	18	0	15.0	10.5
0	20	0	5.4	3.9
1	1	0	7.5	5.4
1	2	0	38.9	43.6
1	3	0	25.0	24.6
1	4	0	74.1	77.1
1	5	0	16.4	15.0
1	6	0	67.5	74.8
1	7	0	4.0	1.1
1	8	0	52.5	53.2
1	9	0	19.3	18.2
1	10	0	10.3	8.6
1	11	0	13.2	11.1
1	12	0	39.2	37.9
1	14	0	9.0	8.0
1	15	0	6.3	8.8
1	16	0	7.7	6.2
1	18	0	7.9	8.2
2	0	0	170.6	171.4
2	1	0	22.8	18.5
2	2	0	32.3	32.4
2	3	0	25.0	30.7
2	4	0	71.0	71.1
2	5	0	32.3	37.0
2	6	0	81.1	77.7
2	8	0	32.8	34.1
2	9	0	22.2	18.9
2	10	0	50.2	52.1
2	11	0	17.5	16.7
2	13	0	13.2	14.4
2	14	0	27.1	23.8
2	16	0	22.8	19.2
2	17	0	9.7	7.3
2	18	0	7.9	9.4
2	20	0	6.4	6.8
3	1	0	34.9	34.2
3	2	0	100.9	96.4
3	3	0	45.0	43.9
3	4	0	27.4	28.9
3	6	0	43.2	41.4
3	8	0	29.7	31.8
3	11	0	21.6	22.8
3	12	0	35.7	34.0
3	13	0	10.3	9.0
3	14	0	6.9	8.3
3	16	0	6.3	6.8
3	17	0	4.0	5.6
3	18	0	4.0	2.1
4	0	0	29.7	32.5
4	1	0	38.1	36.9
4	2	0	17.3	18.7
4	3	0	4.0	5.0
4	4	0	51.6	49.6
4	5	0	30.5	31.6
4	6	0	65.8	66.5
4	7	0	5.7	3.3
4	8	0	2.5	5.5
4	9	0	32.3	31.1
4	10	0	30.0	31.4
4	11	0	7.7	7.6
4	12	0	4.0	2.7
4	13	0	4.0	3.8
4	14	0	22.0	18.1
4	15	0	14.4	10.3
4	16	0	14.4	13.1
4	20	0	7.9	8.5
5	1	0	32.0	31.1
5	2	0	43.6	46.4
5	3	0	44.0	43.7
5	5	0	17.0	15.2
5	6	0	26.9	25.8
5	7	0	27.6	25.0
5	8	0	27.6	28.5
5	11	0	23.1	22.6
5	12	0	8.1	5.7
5	13	0	11.8	10.2
5	14	0	13.3	8.3
5	15	0	12.7	13.4
5	17	0	8.9	5.8
6	0	0	36.3	34.7
6	1	0	26.2	25.0
6	2	0	19.3	18.6
6	3	0	14.1	14.6
6	4	0	24.2	23.7
6	5	0	24.5	23.5
6	6	0	22.5	20.4
6	9	0	22.5	20.5
6	10	0	21.3	20.6
6	11	0	4.0	5.4
6	14	0	13.2	13.0
6	15	0	6.3	8.4
7	1	0	14.1	11.0
7	3	0	34.9	33.2
7	4	0	9.0	5.8
7	7	0	23.1	21.4
7	8	0	18.7	18.7
7	11	0	19.0	17.1
8	1	0	10.0	10.7
8	2	0	9.5	7.5
8	3	0	26.8	21.3
8	4	0	15.2	13.4
8	5	0	21.9	19.1
8	6	0	6.3	5.0
9	3	0	19.3	15.2
9	9	0	2.2	1.5
0	1	3	7.8	6.1
0	2	3	27.2	24.3
0	4	3	13.0	13.8
0	5	3	6.6	3.2
0	6	3	16.5	19.1
0	8	3	9.5	7.4
0	10	3	6.0	5.4
0	11	3	4.0	4.0
0	12	3	9.2	9.3
0	16	3	6.0	3.3
0	17	3	3.7	1.2
0	18	3	4.9	2.7
0	0	4	6.3	6.3
0	1	4	5.2	3.8
0	3	4	7.8	8.2
0	7	4	8.1	7.3
0	9	4	4.9	6.0
0	11	4	4.0	3.1
0	13	4	4.6	3.1
0	1	1	33.3	41.9
0	2	1	71.7	65.1
0	3	1	5.1	7.0
0	4	1	30.7	31.6
0	5	1	58.8	60.0
0	6	1	15.9	14.0
0	7	1	19.9	22.7
0	8	1	35.5	39.5
0	9	1	31.4	36.0
0	10	1	5.9	6.0
0	11	1	29.4	27.4
0	12	1	10.0	8.5
0	13	1	9.6	8.6
0	15	1	18.8	16.7
0	16	1	10.2	8.7
0	19	1	8.5	2.3
1	0	1	35.3	34.3
1	1	1	88.8	86.8
1	2	1	93.6	82.7
1	3	1	115.8	116.7
1	4	1	19.3	18.7
	5	1	20.1	19.2
1	6	1	10.5	11.1
1	7	1	66.6	69.8
1	8	1	24.3	23.5
1	9	1	20.7	23.8
1	10	1	13.1	17.9
1	11	1	28.1	33.1
1	12	1	5.1	9.9
1	13	1	23.5	25.8
1	14	1	11.1	11.1
1	17	1	17.9	16.3
1	18	1	5.9	4.0
2	0	1	35.6	34.0
2	1	1	56.3	54.8
2	2	1	34.5	31.9
2	3	1	40.7	36.7
2	4	1	19.4	18.3
2	5	1	61.7	59.4
2	6	1	12.2	11.3
2	7	1	15.1	15.3
2	8	1	21.3	24.0
2	9	1	26.4	36.0
2	10	1	12.5	14.1
2	11	1	22.5	27.6
2	12	1	7.4	7.0
2	13	1	13.4	13.6
2	15	1	18.8	13.9
2	16	1	6.1	5.5
3	0	1	28.6	28.0
3	1	1	35.8	37.0
3	2	1	24.6	23.3
3	3	1	72.7	70.9
3	4	1	46.5	46.3
3	5	1	10.1	9.0
3	6	1	23.7	25.3
3	7	1	38.4	33.9
3	8	1	15.0	18.2
3	9	1	20.5	26.2
3	10	1	8.5	11.5
3	11	1	18.7	22.5
3	12	1	13.3	16.3
3	13	1	22.7	23.8
3	14	1	13.2	13.3
3	17	1	12.7	12.2
4	0	1	42.1	47.2
4	1	1	38.3	41.1
4	2	1	4.2	5.8
4	3	1	31.3	31.7
4	4	1	28.7	31.0
4	5	1	39.1	41.6
4	6	1	19.1	18.8
4	7	1	13.2	16.2
4	8	1	14.3	19.1
4	9	1	17.4	23.6
4	10	1	12.9	14.7
4	11	1	11.0	14.3
4	14	1	12.2	11.0
4	15	1	10.9	7.5
5	0	1	17.4	18.2
5	1	1	27.1	27.0
5	2	1	22.7	25.0
5	3	1	37.1	37.2
5	4	1	16.8	17.7
5	5	1	10.6	9.3
5	6	1	20.2	22.0
5	7	1	25.3	27.6
5	8	1	12.8	14.7
5	9	1	17.1	19.6
5	11	1	11.5	15.0
5	12	1	10.3	10.5
5	13	1	16.2	17.6
6	0	1	31.9	31.7
6	1	1	17.9	18.4
6	2	1	7.0	10.3
6	3	1	7.1	11.1
6	4	1	22.0	21.2
6	5	1	13.9	16.1
6	6	1	20.9	21.8
6	7	1	7.7	10.8
6	8	1	14.9	16.0
6	9	1	5.8	6.8
6	10	1	14.7	18.7
6	11	1	5.1	10.3
6	12	1	5.0	6.0
6	14	1	14.2	13.0
7	0	1	5.5	6.5
7	1	1	12.4	12.4
7	2	1	23.6	22.0
7	3	1	19.4	19.4
7	4	1	5.9	7.3
7	5	1	15.2	15.8
7	6	1	14.3	14.3
7	7	1	14.6	13.7
7	8	1	8.5	10.3
7	9	1	6.1	7.9
8	0	1	18.1	15.5
8	1	1	12.1	10.9
8	3	1	6.1	7.4
8	4	1	10.7	12.3
0	0	2	22.5	17.5
0	1	2	23.3	19.0
0	2	2	7.8	6.6
0	3	2	31.1	33.4
0	4	2	86.8	76.3
0	5	2	4.1	10.1
0	6	2	47.5	47.0
0	7	2	27.8	26.3
0	8	2	12.3	10.5
0	9	2	7.4	6.8
0	10	2	19.7	18.3
0	11	2	13.5	12.5
0	12	2	5.7	3.3
0	13	2	13.5	12.4
0	14	2	12.7	12.3
0	15	2	5.7	5.1
0	16	2	10.2	[illegible]
0	17	2	10.2	5.9
1	0	2	7.2	6.1
1	1	2	38.7	31.6
1	2	2	43.0	32.6
1	3	2	24.6	24.3
1	4	2	17.4	19.2
1	5	2	32.1	36.2
1	6	2	2.2	7.1
1	7	2	2.2	6.5
1	8	2	22.2	25.1
1	9	2	20.9	22.3
1	10	2	3.7	4.5
1	11	2	10.8	10.8
1	12	2	13.1	11.3
1	13	2	8.5	9.1
1	14	2	8.1	8.2
1	15	2	6.4	8.1
1	16	2	6.5	4.9
1	17	2	4.5	0.8
1	18	2	8.4	2.3
1	19	2	8.7	4.4
2	0	2	44.4	40.7
2	1	2	30.2	30.2
2	2	2	17.4	18.4
2	3	2	37.4	36.6
2	4	2	44.9	43.3
2	5	2	11.3	9.9
2	6	2	23.4	27.6
2	7	2	20.8	24.2
2	8	2	7.6	8.3
2	9	2	9.9	10.8
2	10	2	18.1	23.7
2	11	2	12.7	13.5
2	12	2	8.3	6.5
2	13	2	11.1	10.9
2	14	2	12.1	14.8
2	15	2	3.8	5.6
2	16	2	5.9	4.3
2	17	2	9.8	6.4
2	18	2	2.6	2.4
2	19	2	2.5	2.9
2	20	2	7.2	6.0
3	0	2	14.3	13.0
3	1	2	15.8	13.2
3	2	2	27.5	25.6
3	3	2	13.6	11.8
3	4	2	22.9	25.0
3	5	2	23.3	25.6
3	6	2	16.7	19.3
3	7	2	12.1	15.3
3	8	2	17.0	22.0
3	9	2	10.2	13.0
3	10	2	5.7	7.7
3	11	2	8.2	11.2
3	12	2	4.9	6.4
3	13	2	5.7	4.5
3	14	2	10.4	10.2
3	15	2	7.0	6.0
3	17	2	3.7	3.6
3	18	2	7.2	4.5
3	19	2	5.9	2.9
4	0	2	36.9	38.3
4	1	2	22.6	24.4
4	2	2	21.3	23.5
4	3	2	25.8	27.8
4	4	2	13.2	12.1
4	5	2	13.2	13.3
4	6	2	17.8	16.7
4	7	2	13.9	17.0
4	8	2	11.3	11.6
4	9	2	3.3	4.5
4	10	2	16.7	22.0
4	11	2	10.5	10.7
4	12	2	6.3	8.3
4	13	2	9.8	9.7
4	14	2	8.4	10.0
4	15	2	5.3	3.0
4	16	2	8.2	4.5
4	17	2	8.1	6.1
5	0	2	17.2	15.5
5	1	2	8.3	8.7
5	2	2	17.5	19.0
5	3	2	12.0	12.6
5	4	2	14.6	14.5
5	5	2	11.2	13.1
5	6	2	5.6	8.9
5	7	2	14.5	16.8
5	8	2	4.1	5.9
5	9	2	4.2	7.8
5	10	2	4.4	6.5
5	11	2	6.3	9.9
5	12	2	4.5	6.1
5	13	2	4.6	3.6
5	14	2	10.6	8.1
5	15	2	2.6	2.7
6	0	2	16.3	15.6
6	1	2	11.9	12.8
6	2	2	14.0	13.2
6	3	2	9.9	10.3
6	4	2	9.5	10.1
6	5	2	9.6	9.7
6	6	2	17.4	19.4
6	8	2	11.4	11.3
6	10	2	10.5	10.7
6	11	2	2.6	5.2
6	12	2	3.8	4.7
6	13	2	3.8	4.9
6	14	2	2.6	3.7
6	16	2	3.4	6.2
6	17	2	7.7	4.1
7	0	2	18.1	15.4
7	1	2	8.7	8.6
7	2	2	7.1	6.1
7	3	2	6.1	7.3
7	4	2	9.5	10.4
7	7	2	10.5	11.7
7	9	2	6.5	6.6
7	10	2	5.3	6.2
7	11	22	4.5	6.1
7	12	2	4.5	4.7
7	13	2	3.6	2.8
7	14	2	7.9	5.6
8	0	2	9.9	12.1
8	1	2	9.9	11.0
8	2	2	7.5	4.1
8	4	2	4.6	4.8
8	5	2	3.7	5.1
8	6	2	9.2	8.3
8	8	2	4.5	6.1
8	9	2	4.5	6.0
8	10	2	2.5	3.0
9	0	2	12.4	10.3
9	1	2	6.4	4.9
10	0	2	9.4	6.3

applied with calculations of the [001] Patterson projection and Fourier refinement, the signs being based on the bromine contributions alone. After some initial difficulties, the bromine positions were fixed at $x=0{\cdot}215$, $y=0{\cdot}150$, $z=0{\cdot}065$ and normal Fourier methods of reiterative refinements were tried. The R values at this stage, were $R(hk0)=0{\cdot}165$, $R(0kl)=0{\cdot}21$. As the overlap was serious in the [100] projection, the structure was further refined by using the full-matrix least-squares program *ORFLS* of Busing, Martin & Levy (1962). Unit weights for all observed reflexions and individual anisotropic temperature factors for each atom were used. The overall R value was 0·107 for observed reflexions only. The contributions of the hydrogen atoms were not included. Fig. 1 shows the electron density projected down the [001] axis. Table 1 gives the observed and calculated structure factors for the structure.

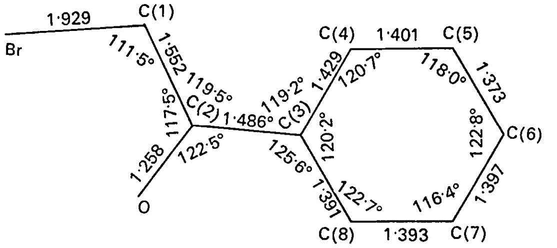

Fig. 2. Numbering scheme of the atoms, and bond lengths and bond-angles in the molecule.

Atomic coordinates and thermal parameters

The final atomic coordinates, referred to the standard origin (*International Tables for X-ray Crystallography*, 1952) with the exclusion of the hydrogen atoms which have not been located, are given in Table 2 (together with their e.s.d.'s, given in parentheses and referring to the last two decimal places). Thermal parameters are listed in Table 3.

Table 2. *Final atomic coordinates* (× 10^4)

	x/a	y/b	z/c
O	6500 (31)	2676 (16)	2335 (77)
C(1)	4117 (42)	2342 (20)	2487 (112)
C(2)	5321 (46)	2849 (24)	3279 (120)
C(3)	5036 (38)	3542 (16)	4794 (79)
C(4)	3692 (38)	3683 (23)	5995 (100)
C(5)	3400 (42)	4323 (17)	7519 (120)
C(6)	4472 (44)	4776 (22)	8098 (116)
C(7)	5807 (56)	4683 (21)	6962 (131)
C(8)	6102 (32)	4032 (24)	5600 (145)
Br	4766 (5)	1477 (3)	0593 (14)

Interatomic distances, bond lengths and angles

The numbering scheme of the atoms in the molecule is shown in Fig. 2. Tables 4 & 5 give bond lengths and bond angles respectively (with their e.s.d.'s) in the molecule. The significant intermolecular contacts between the atoms are listed in Table 6.

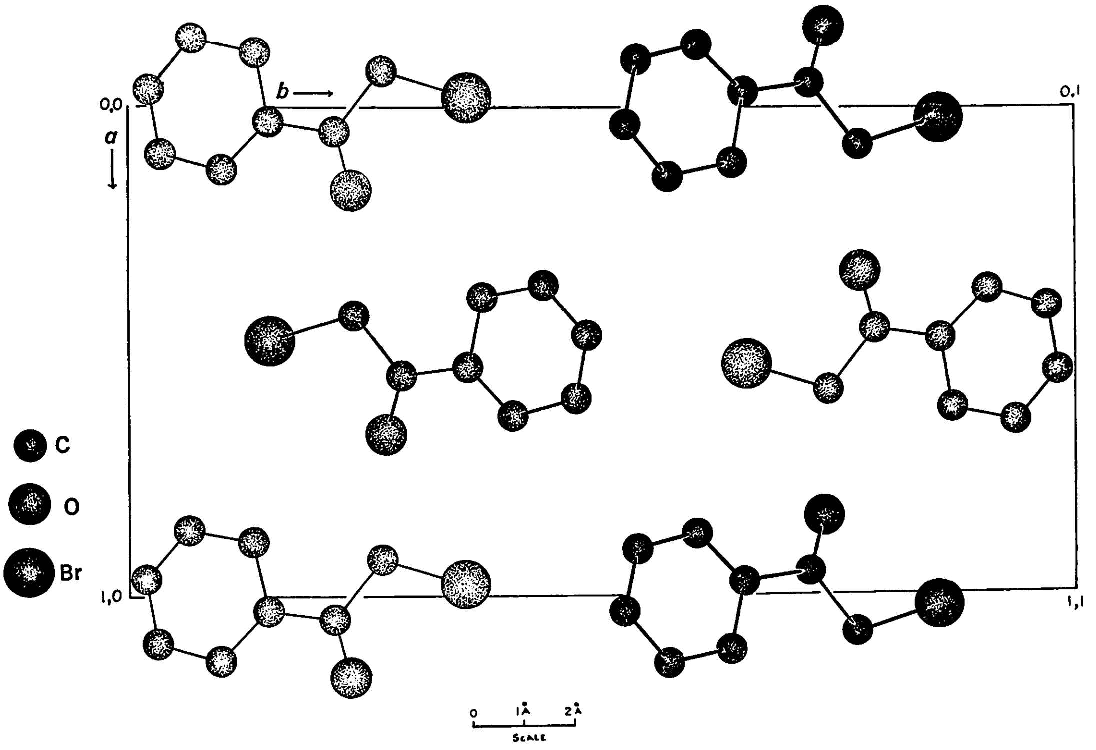

Fig. 3. Crystal structure looking down the [001] axis.

Table 3. *Thermal parameters* ($\times 10^4$) *in* Å^2 *for non-hydrogen atoms*

Estimated standard deviations are in parentheses. Expression used of the type:

$$\exp\left[-(b_{11}h^2+b_{22}k^2+b_{33}l^2+b_{23}kl+b_{31}hl+b_{12}hk)\right]$$

	b_{11}	b_{22}	b_{33}	b_{12}	b_{13}	b_{23}
O	164 (38)	66 (13)	811 (636)	23 (22)	−3 (100)	−22 (50)
C(1)	202 (62)	41 (14)	1132 (680)	−40 (26)	−86 (154)	117 (66)
C(2)	142 (58)	72 (21)	942 (684)	1 (34)	−325 (166)	134 (81)
C(3)	229 (67)	30 (9)	140 (435)	63 (26)	−181 (132)	24 (43)
C(4)	160 (58)	60 (18)	315 (765)	−8 (28)	−38 (121)	36 (68)
C(5)	172 (60)	19 (10)	1457 (673)	−26 (22)	69 (155)	−14 (62)
C(6)	141 (57)	47 (16)	822 (596)	11 (28)	23 (138)	59 (64)
C(7)	323 (102)	29 (13)	1114 (736)	36 (30)	−184 (203)	35 (70)
C(8)	83 (47)	61 (18)	1688 (779)	−34 (25)	−132 (152)	208 (97)
Br	198 (6)	57 (2)	1347 (516)	24 (3)	−43 (18)	−34 (9)

Table 4. *Bond lengths* (Å)

Estimated standard deviations are given in parentheses and refer to the least significant places of decimal.

Br—C(1)	1·929 (40)	C(4)–C(5)	1·401 (57)
O═C(2)	1·258 (54)	C(5)–C(6)	1·373 (57)
C(1)–C(2)	1·552 (60)	C(6)–C(7)	1·397 (70)
C(2)–C(3)	1·486 (55)	C(7)–C(8)	1·393 (65)
C(3)–C(4)	1·429 (53)	C(8)–C(3)	1·391 (52)

It is seen that the structure is rather loosely bound by van der Waals contacts upward of 3 Å. This explains the large thermal motion and decomposition of the crystal when exposed to radiation.

Planarity of the molecule and its orientation

The equation of the best plane through the six carbon atoms of the benzene ring is

$$X-2{\cdot}27119\,Y+4{\cdot}42173\,Z+1{\cdot}23036=0 \qquad (1)$$

and the deviations of the various atoms of the molecule from this plane are given in Table 7.

Table 5. *Bond angles* (°)

Estimated standard deviations are in parentheses.

Br—C(1)–C(2)	111·5 (2·5)	C(4)–C(3)–C(8)	120·2 (3·2)
C(1)–C(2)=O	117·5 (3·2)	C(3)–C(4)–C(5)	120·7 (3·1)
C(1)–C(2)–C(3)	119·5 (3·0)	C(4)–C(5)–C(6)	118·0 (3·4)
O═C(2)–C(3)	122·5 (3·2)	C(5)–C(6)–C(7)	122·8 (3·6)
C(2)–C(3)–C(4)	119·2 (3·3)	C(6)–C(7)–C(8)	116·4 (3·5)
C(2)–C(3)–C(8)	125·6 (3·7)	C(7)–C(8)–C(3)	122·7 (3·4)

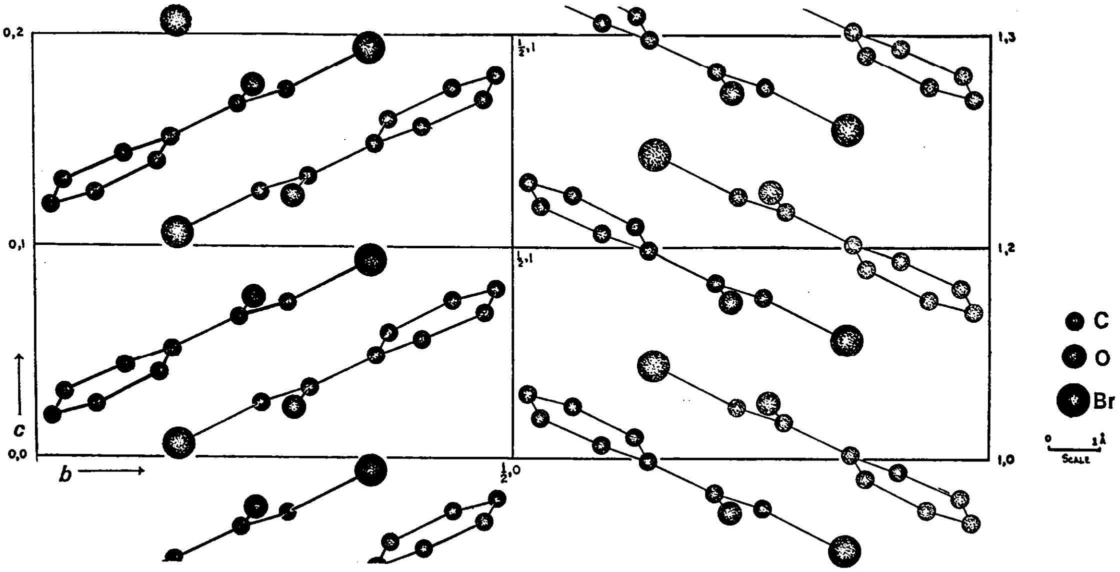

Fig. 4. Crystal structure looking down the [100] axis.

Table 6. *Significant intermolecular contacts between atoms*

Br---C(6) (vi)	3·65 Å	O——C(1) (iii)	3·26 Å
Br——C(8)$^+$ (iv)	4·03	O——Br (iii)	3·77
Br---C(7) (vi)	3·60	O---C(1)$^+$ (iii)	3·35
C(1)–C(8)$^+$ (iv)	4·00	C(5)–C(5) (i)	3·75
Br---O (iv)	3·77	C(5)–C(6)$^-$ (i)	3·77
C(1)–O (iv)	3·26	C(6)–C(5) (i)	3·77
C(1)–O$^+$ (iv)	3·35	C(6)–Br (v)	3·65
O——C(4)$^+$ (iii)	3·41	C(7)–Br (v)	3·60
C(7)–C(7)$^-$ (ii)	4·23	C(8)–C(7)$^-$ (ii)	4·16
C(4)–O$^+$ (iv)	3·41	C(8)–Br$^-$ (iii)	4·03

Key to symmetry related atoms:

(i)	$\frac{1}{2}-x,\ 1+\bar{y},\ \frac{1}{2}+z$	(iv)	$-\frac{1}{2}+x,\ \frac{1}{2}-y,\ \bar{z}$
(ii)	$\frac{3}{2}-x,\ 1+\bar{y},\ \frac{1}{2}+z$	(v)	$1+\bar{x},\ \frac{1}{2}+y,\ \frac{1}{2}-z$
(iii)	$\frac{1}{2}+x,\ \frac{1}{2}-y,\ \bar{z}$	(vi)	$1+\bar{x},\ -\frac{1}{2}+y,\ \frac{1}{2}-z$

+ refers to an atom displaced one unit along the positive *c* direction.

− refers to an atom displaced one unit along the negative *c* direction.

The equation of the plane through the non-ring light atoms C(1), 0, C(2) is

$$X-3\cdot11947\,Y+5\cdot44647\,Z+4\cdot10425=0\,. \tag{2}$$

Planes (1) and (2) make an angle of 3° 30′ with each other. The direction cosines of the normals to plane (1) are $l=0\cdot1972$, $m=-0\cdot4479$ and $n=0\cdot8720$. Those for plane (2) are $l=0\cdot1573$, $m=-0\cdot4908$ and $n=0\cdot8569$.

Table 7. *Deviations of various atoms from plane* (I)

O	0·081 (47) Å	C(4)*	0·035 (63) Å
C(1)	−0·037 (65)	C(5)*	−0·002 (66)
C(2)	0·056 (73)	C(6)*	0·032 (69)
Br	−0·124 (8)	C(7)*	−0·051 (75)
C(3)*	−0·029 (52)	C(8)*	0·057 (79)

* Atoms of the benzene ring.

Description of the structure

Figs. 3 and 4 give views of the crystal structure projected down the [001] and the [100] axes of the unit cell. In the crystal, molecules are arranged in two distinict layers inclined towards each other at an anle of about. 122°, the two layers being separated by $\frac{1}{2}c$ translation. The Br–C distance (1·929 Å) is normal. The angle Br–C(1)–C(2) is 111·5°, *i.e.* close to the,tetrahedravalue. suggesting that the atoms Br, C(2), and two hydrogen atoms conforming to a tetrahedral configuration are attached to the carbon atom C(1).

References

Busing, W. R., Martin, K. O. & Levy, H. A. (1962). *ORFLS. A Fortran Crystallographic Least-Squares Program.* USAEC. Report ORNL-TM-305. Oak Ridge National Laboratory, Oak Ridge, Tennessee. (CDC-3600 version incorporates modifications by W. C. Hamilton, J. A. Ibers, C. K. Johnson & S. Srikanta.)

International Tables for X-ray Crystallography (1952) Vol. I, Space group No. 19. Birmingham: Kynoch Press.

Acta Cryst. (1971). B27, 1653

The Crystal and Molecular Structure of Dichloro(2,7-dimethyl-octa-2,6-diene-1,8-diyl)ruthenium(IV) Dimer

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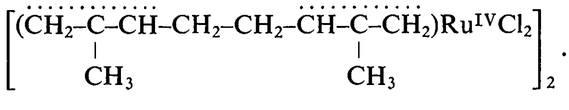
The crystal structure of $[\mathrm{Ru(IV)Cl_2C_{10}H_{16}}]_2$ is reported and discussed. The space group is $P\bar{1}$, with $a=9\cdot49$ (2), $b=8\cdot48$ (2), $c=7\cdot71$ (2) Å, $\alpha=107\cdot8$ (2), $\beta=76\cdot6$ (2), $\gamma=100\cdot1$ (2)° and one molecule per unit cell. The final disagreement index R is 0·082 for the 1008 observed reflexions (0·105 if the 342 non-observable reflexions are taken into account). The coordination around the metal atom may be described in terms of a trigonal bipyramid, two of the equatorial positions being occupied by the terminal allylic group of the organic ligand: this corresponds to a linear tail-to-tail dimer of isoprene.

Introduction

In 1965, Porri & Gallazzi prepared a complex from $\mathrm{RuCl_3}$ and isoprene, having the formula



$$\left[\mathrm{(\overset{\cdots\cdots\cdots}{CH_2{-}\underset{\displaystyle CH_3}{\underset{|}{C}}{-}CH}{-}CH_2{-}CH_2{-}\overset{\cdots\cdots\cdots}{CH{-}\underset{\displaystyle CH_3}{\underset{|}{C}}{-}CH_2})Ru^{IV}Cl_2}\right]_2 . \quad \mathrm{(I)}$$

From chemical, nuclear magnetic resonance and X-ray evidence, the compound was shown to be dimeric, and the organic ligand to be a linear tail-to-tail dimer of isoprene (Porri, Gallazzi, Colombo & Allegra, 1965). As shown in Fig. 1, the metal atom coordinates to the organic ligand *via* σ–π bonding with two terminal allyl groups; to the best of our knowledge this type of co-