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(*RS*)-*trans*-2-Methyl-3-(2,6-dimethyl-4-chlorophenyl)oxaziridine

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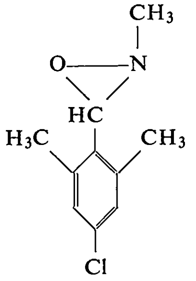
The crystal structure of a compound containing a three-membered CNO ring has been determined from visually estimated X-ray data and fully refined in three dimensions. An unusual feature in the structure is an intermolecular C–H···O bridge.

Introduction

By ultraviolet irradiation of *N*-methylated *anti*-2,6-dimethyl-4-bromobenzaldoxime a compound was formed which Brehm, Jensen & Jerslev (1966), by a two-dimensional X-ray study, proved to be *trans*-2-methyl-3-(2,6-dimethyl-4-bromo)oxaziridine. Furthermore it was shown that the corresponding chlorine compound is isomorphous with the bromine compound. Since apparently no information on the dimensions of an oxaziridine ring was available, a detailed crystal structure determination of the chlorine compound has been carried out. A short account of the results was presented at the IUC congress in Moscow (Jensen & Jerslev, 1966).



Experimental

Racemic *trans*-2-methyl-3-(2,6-dimethyl-4-chlorophenyl)oxaziridine crystallizes from 96% ethanol as flat monoclinic needles elongated in the **b** direction and with only {102} well developed. The melting point is 83–84°C, $\varrho = 1{\cdot}30$. The crystals are volatile, but kept in the dark in a stoppered glass vessel they are stable for at least several months. During the X-ray exposures the crystals were enclosed in Lindemann capillaries. The cell dimensions as determined from zero level Weissenberg photographs are: $a = 11{\cdot}3_1$, $b = 4{\cdot}18_0$, $c = 21{\cdot}2_8$ Å, $\beta = 96{\cdot}4°$. $Z = 4$. The absence of reflexions $0k0$ with k odd and $h0l$ with l odd indicated the space group $P2_1/c$. From a crystal with cross section $0{\cdot}2 \times 0{\cdot}04$ mm, length 0·4 mm, the reflexions $h0l$, $h1l$, $h2l$ and $h3l$ were recorded at room temperature with Cu $K\alpha$ radiation. Weissenberg multiple film technique was used, and the intensities were estimated visually by means of a calibrated scale with about 10% intensity variation between the steps. The intensities of the layers were brought onto a common scale by comparison with equivalent reflexions recorded from a crystal which had been cut to a cross section of $0{\cdot}18 \times 0{\cdot}04$ mm and was placed on the camera with [102] as the axis of rotation; two zones were photographed. The absorption coefficient for Cu $K\alpha$ radiation has a value of $30{\cdot}5$ cm^{-1} and no correction for absorption was applied. For the structure determination 927 independent reflexions were used, representing about 60% of the spots within the sphere of reflexion. Since the intensities fall off rapidly with increasing diffraction angle, this figure represents nearly all reflexions obtainable in practice at room temperature with copper radiation.

Structure determination

All computations were performed on the Saab D21 computer, using the integrated set of programs by Abrahamsson, Aleby, Larsson, Nilsson, Selin & Westerdahl (1965). After the Lorentz and polarization corrections had been performed, an electron density projection $\varrho(xz)$ was calculated, using the x and z parameters known from the isomorphous bromine compound. Furthermore a three-dimensional sharpened Patterson synthesis was calculated, for which a list of peak coordinates and heights was printed out. The slope of the molecule was then estimated from the x and z parameters found and the approximately known bond lengths; inspection of the peaks of the Patterson synthesis gave a check and small corrections to this estimate. The Harker section $^{s}P(x\tfrac{1}{2}z)$ showed two peaks of about the same height, which both could be interpreted as Cl–Cl vectors, thus indicating that the chlorine must be situated at about $y = 0{\cdot}25$. Which of the two peaks was the Harker peak was found by means of the subsequent least-squares refinement. Refinement was attempted first with one, then with the other peak interpreted as the Harker peak, but in the

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first case refinement stopped at an R value of 36%, whereas in the other case the R value for all reflexions dropped to 16·8% during 5 cycles of block-diagonal least-squares treatment.

The atomic parameters obtained were used for a three-dimensional difference Fourier synthesis, in which the peak scanning program revealed 11 of the 12 hydrogen atoms in the molecule. There were some spurious peaks with heights of the same order of magnitude, but inspection of the maps clearly revealed the positions of all 12 hydrogen atoms, though not with any great precision. Two cycles of full-matrix least-squares treatment of the Cl, O, N and C parameters including the hydrogen atoms fixed at the positions found above with a common arbitrarily chosen B value equal to 4 Å^2 reduced the R value to 13·7%.

An attempt to refine the hydrogen positional parameters by a least-squares treatment was made. With all parameters of the Cl, O, N and C atoms fixed and again with a common hydrogen thermal parameter

Table 1. *Positional* (xyz) *and vibrational* (U_{ij}) *parameters of the atoms*

Anisotropic vibrations allowed for with
$\exp\,[-2\pi^2(h^2a^{*2}U_{11}+k^2b^{*2}U_{22}+l^2c^{*2}U_{33}+2klb^*c^*U_{23}+2lhc^*a^*U_{31}+2hka^*b^*U_{12})]$

	x	y	z	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Cl	0·2889	0·2747	0·4300	0·1036	0·0917	0·0637	−0·0108	0·0151	−0·0098
C(1)	0·2844	0·3951	0·5086	0·0939	0·0404	0·0495	−0·0000	0·0097	−0·0267
C(2)	0·1867	0·5511	0·5258	0·0605	0·0393	0·0648	−0·0041	0·0099	−0·0042
C(3)	0·1813	0·6291	0·5884	0·0549	0·0230	0·0793	0·0027	0·0133	−0·0102
C(4)	0·2769	0·5428	0·6340	0·0576	0·0106	0·0634	−0·0051	0·0115	−0·0057
C(5)	0·3755	0·3985	0·6135	0·0627	0·0222	0·0800	0·0013	0·0104	−0·0084
C(6)	0·3815	0·3128	0·5516	0·0673	0·0421	0·0871	−0·0009	0·0180	−0·0144
C(7)	0·2712	0·6344	0·7003	0·0551	0·0247	0·0713	0·0028	0·0098	−0·0035
C(8)	0·0738	0·8005	0·6068	0·0652	0·0435	0·0897	−0·0031	0·0079	0·0108
C(9)	0·4839	0·3208	0·6624	0·0544	0·0650	0·0884	0·0138	0·0001	0·0080
C(10)	0·1480	0·7018	0·7844	0·0777	0·0379	0·0795	0·0064	0·0204	−0·0058
O	0·3026	0·3946	0·7477	0·0681	0·0405	0·0800	0·0063	0·0030	0·0117
N	0·1761	0·5077	0·7325	0·0577	0·0286	0·0690	0·0012	0·0149	−0·0036
H(101)	0·2090	0·8472	0·8085	$B=4$ Å^2 (arbitrarily chosen)					
H(102)	0·0613	0·8600	0·7689						
H(103)	0·1254	0·6044	0·8090						
H(91)	0·4534	0·1855	0·6850						
H(92)	0·5321	0·1437	0·6475						
H(93)	0·5147	0·4941	0·6838						
H(81)	0·0224	0·6241	0·6292						
H(82)	0·0231	0·8725	0·5726						
H(83)	0·0865	1·0014	0·6362						
H(7)	0·2905	0·8618	0·7200						
H(6)	0·4610	0·2023	0·5317						
H(2)	0·1251	0·6713	0·4929						

Table 2. *Distances and angles involving hydrogen atoms*

Standard deviations: σ(C–H)$\simeq$0·10 Å σ(H–H)$\simeq$0·14 Å $\sigma(\angle$CCH)$\simeq$6° $\sigma(\angle$HCH)$\simeq$9°

C(10)–H(101)	1·01 Å	C(8)–H(81)	1·08 Å	C(9)–H(91)	0·84 Å
H(102)	1·20	H(82)	0·92	H(92)	0·99
H(103)	0·73	H(83)	1·05	H(93)	0·91
H(101)–H(102)	1·79	H(81)–H(82)	1·59	H(91)–H(92)	1·27
H(101)–H(103)	1·39	H(81)–H(83)	1·74	H(91)–H(93)	1·47
H(102)–H(103)	1·50	H(82)–H(83)	1·56	H(92)–H(93)	1·68
H(101)–C(10)–N	122°	H(81)–C(8)–C(3)	106°	H(91)–C(9)–C(5)	100°
H(102)–C(10)–N	110	H(82)–C(8)–C(3)	114	H(92)–C(9)–C(5)	111
H(103)–C(10)–N	111	H(83)–C(8)–C(3)	119	H(93)–C(9)–C(5)	114
H(101)–C(10)–H(102)	107	H(81)–C(8)–H(82)	105	H(91)–C(9)–H(92)	87
H(101)–C(10)–H(103)	104	H(81)–C(8)–H(83)	109	H(91)–C(9)–H(93)	114
H(102)–C(10)–H(103)	99	H(82)–C(8)–H(83)	104	H(92)–C(9)–H(93)	124
C(7)–H(7)	1·05 Å	C(2)–H(2)	1·06 Å	C(6)–H(6)	1·14 Å
H(7)–C(7)–C(4)	126°	H(2)–C(2)–C(1)	123°	H(6)–C(6)–C(1)	117°
H(7)–C(7)–N	106	H(2)–C(2)–C(3)	115	H(6)–C(6)–C(5)	126
H(7)–C(7)–O	109				

$B=4$ Å^2, the hydrogen positions were refined by least-squares treatment. After each cycle the interatomic distances and valency angles connected with hydrogen were printed out. It was obvious that the interatomic distances were not refined, but the directions from carbon to the attached hydrogen atoms improved appreciably, especially during the first cycle. A final cycle with the hydrogen atoms fixed at the positions now obtained gave the thermal and positional parameters for the Cl, O, N and C atoms shown in Table 1 together with the hydrogen positions. The final R value for all observed reflexions including hydrogen atoms is 13·3%. Observed and calculated structure factors are listed in Table 3.

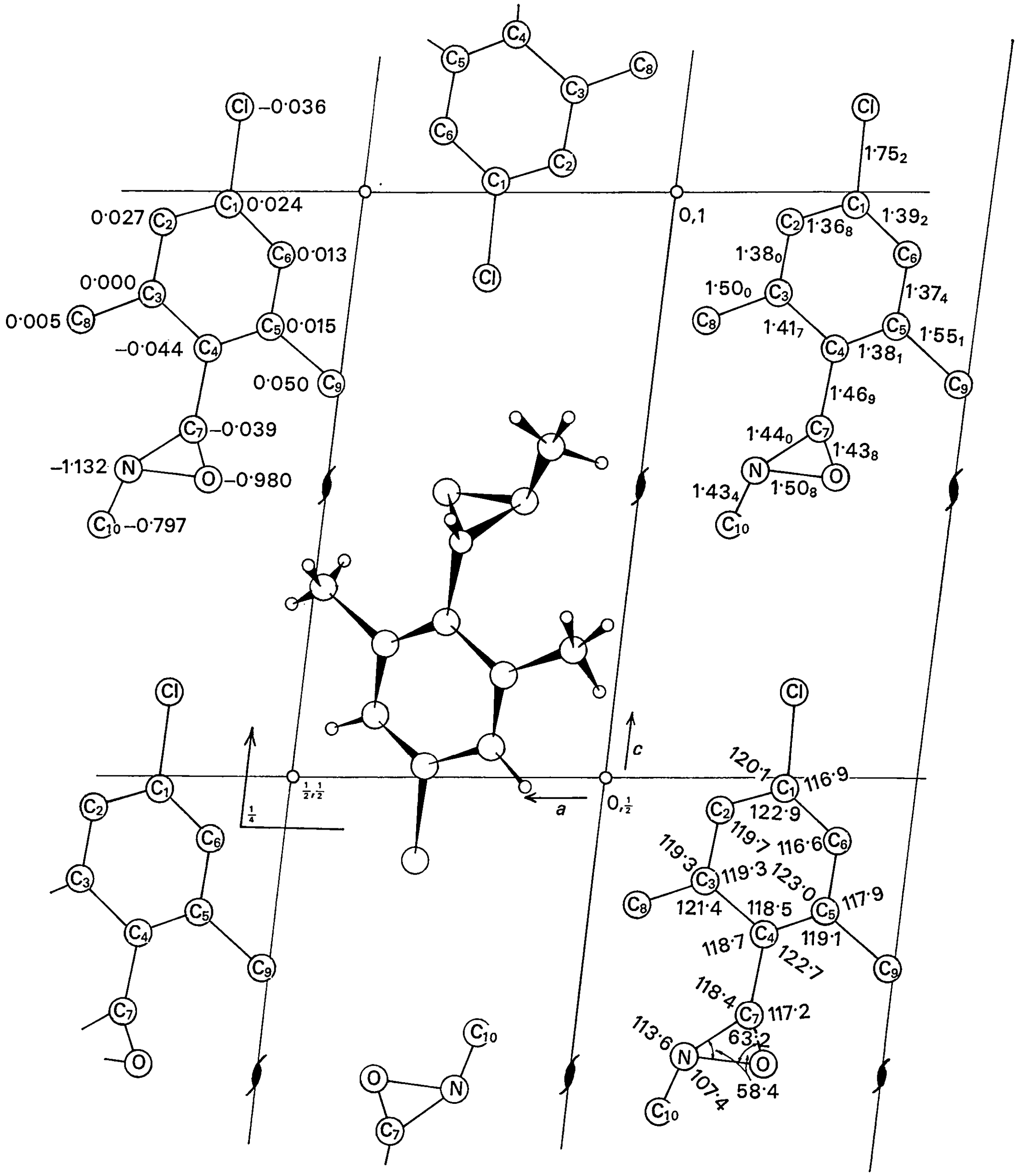

Fig. 1. The structure viewed along the b axis. Upper right molecule: Bond lengths (Å). Standard deviations: σ(C–Cl)$\simeq$0·010 Å, σ(C–C)$\simeq$0·014 Å. σ(C–N$\simeq$0·012 Å, σ(C–O) $\simeq$0·011 Å, σ(N–O)$\simeq$0·010 Å. Lower right molecule: Valency angles (°). Standard deviations: $\sigma(\angle\text{CCC})\simeq\sigma(\angle\text{CCCl})\simeq$0·9°. $\sigma(\angle\text{CCN})\simeq\sigma(\angle\text{CCO})\simeq\sigma(\angle\text{CNC})\simeq\sigma(\angle\text{CNO})\simeq\sigma(\angle\text{CON})\simeq$0·5°. Upper left molecule: Distances (Å) from the mean plane through the six benzene carbon atoms and the chlorine atom. Central molecule: Hydrogen positions are indicated.

Description and discussion of the structure

Fig. 1 shows the structure viewed along the *b* axis, and for the Cl, O, N and C atoms molecular distances and valency angles as well as deviations from the mean plane through the benzene carbon atoms and the chlorine atom are given. Valency angles and interatomic distances connected with the hydrogen atoms are given in Table 2.

The interatomic distances in the benzene ring do not deviate significantly from a common value. The carbon atoms C(1) to C(9) as well as the chlorine atoms are found slightly displaced from the mean plane, but these deviations as well as the deviations of the valency angles in the benzene ring from 120° are probably not structurally important, though a few of them on statistical grounds appear significant. The plane of the oxaziridine ring forms an angle of 56·7° with the mean plane through the benzene ring; the nitrogen atom is placed a little farther away than the oxygen atom from that plane, with which the line N–O form an angle of 5·8°.

In the oxaziridine ring the same value, 1·44 Å is found for both the C–O and the C–N bond, whereas the O–N bond is significantly longer, 1·51 Å. The C–C bond connecting the benzene ring and the oxaziridine ring has the length of a C(sp^2)–C(sp^2) bond. The C–N bond connecting the oxaziridine ring with the methyl group is rather short, 1·43 Å.

Table 3. *Observed and calculated structure factors* (×100)

h	k	l	Fobs	Fcalc
0	0	2	777	364
0	0	4	2164	1976
0	0	6	7579	-6658
0	0	8	420	324
0	0	10	767	-575
0	0	12	1332	1429
0	0	14	2051	1553
0	0	16	1682	1381
0	0	20	613	-533
0	0	22	806	-820
1	0	-26	502	593
1	0	-22	811	964
1	0	-20	998	-1011
1	0	-18	951	-816
1	0	-16	2449	-1920
1	0	-14	922	853
1	0	-12	3171	2073
1	0	-10	10961	8305
1	0	-8	3210	2972
1	0	-6	3855	-3622
1	0	-4	1235	-1155
1	0	-2	3195	3541
1	0	0	2540	-2415
1	0	2	5306	-5841
1	0	4	3947	3901
1	0	6	460	-341
1	0	8	741	660
1	0	10	4311	-3380
1	0	12	4598	-3376
1	0	14	1096	-939
1	0	18	391	358
1	0	20	710	650
1	0	22	309	-275
1	0	24	424	-568
1	0	26	441	-608
2	0	-26	296	-335
2	0	-24	324	-343
2	0	-22	564	407
2	0	-18	1738	1798
2	0	-16	1793	-1215
2	0	-14	996	-833
2	0	-12	1902	-1788
2	0	-10	3029	-1678
2	0	-8	1050	-1045
2	0	-6	4564	4801
2	0	-4	1443	-1294
2	0	-2	884	-904
2	0	0	10021	-12867
2	0	2	2293	-1695
2	0	4	9812	-9724
2	0	6	6604	5639
2	0	8	1625	1303
2	0	10	1988	1730
2	0	12	630	-554
2	0	14	3953	-3294
2	0	16	2162	-1882
2	0	18	515	390
2	0	20	817	740
2	0	22	485	489
2	0	24	297	434
3	0	-26	328	-403
3	0	-24	436	-544
3	0	-22	698	-786
3	0	-20	914	979
3	0	-18	418	507
3	0	-16	2103	1997
3	0	-12	945	-797
3	0	-10	4824	-4541
3	0	-8	863	-930
3	0	-6	4454	-4655
3	0	-4	5040	5077
3	0	-2	5916	7158
3	0	0	1229	1247
3	0	4	8208	-7249
3	0	6	2394	-2350
3	0	8	863	760
3	0	10	1886	1704
3	0	12	1309	1184
3	0	14	1430	1248
3	0	16	3905	2901
3	0	18	421	-425
3	0	20	456	-367
3	0	22	387	-285
3	0	24	275	426
4	0	-26	342	404
4	0	-24	496	663
4	0	-18	1926	-1880
4	0	-16	700	758
4	0	-14	297	228
4	0	-12	4055	3564
4	0	-10	1708	1742
4	0	-8	830	-930
4	0	-6	1516	-1511
4	0	-4	4894	-5399
4	0	-2	2029	-1846
4	0	0	2611	2698
4	0	2	1434	1504
4	0	4	3783	3783
4	0	6	4794	4109
4	0	8	1551	-1335
4	0	10	2914	-2595
4	0	12	294	-14
4	0	14	2011	2127
4	0	16	851	867
4	0	20	341	-190
4	0	22	554	-529
4	0	24	183	-340
5	0	-22	649	687
5	0	-18	395	-428
5	0	-16	1100	-1137
5	0	-14	2184	-2333
5	0	-12	910	-915
5	0	-10	1666	1624
5	0	-8	802	875
5	0	-4	3386	-3168
5	0	-2	1777	1505
5	0	0	4253	-4376
5	0	2	819	814
5	0	4	3577	3156
5	0	6	635	794
5	0	10	316	125
5	0	12	1697	-2016
5	0	14	851	-806
5	0	16	1673	-1538
5	0	18	579	498
5	0	20	396	-178
5	0	22	559	404
5	0	24	232	-266
6	0	-24	250	-490
6	0	-20	282	158
6	0	-18	612	537
6	0	-16	1119	-1032
6	0	-14	475	-359
6	0	-10	2385	-2048
6	0	-8	1258	-1279
6	0	-6	613	640
6	0	-4	6097	6450
6	0	-2	1127	1181
6	0	0	1670	1716
6	0	2	3543	-3788
6	0	4	1689	-2154
6	0	6	1578	-1582
6	0	8	1533	1575
6	0	12	1532	1463
6	0	16	488	-476
6	0	18	498	-373
6	0	20	483	-470
7	0	-20	798	-814
7	0	-18	385	-335
7	0	-16	907	957
7	0	-14	3814	3856
7	0	-12	586	620
7	0	-10	304	-51
7	0	-8	676	-783
7	0	-6	483	-834
7	0	-4	1702	1704
7	0	-2	575	-662
7	0	0	2694	2485
7	0	4	1505	1744
7	0	6	1491	-1522
7	0	8	521	-514
7	0	10	324	-382
7	0	12	445	319
7	0	14	331	153
7	0	16	735	882
7	0	18	525	-457
8	0	-22	329	420
8	0	-18	368	418
8	0	-12	589	-602
8	0	-10	1318	1558
8	0	-8	503	-416
8	0	-6	1323	1471
8	0	-4	2107	-2315
8	0	-2	512	-420
8	0	0	1110	-1222
8	0	4	309	-204
8	0	6	1409	1610
8	0	8	853	-772
8	0	10	773	-858
8	0	12	1048	-1097
8	0	14	377	302
8	0	16	292	-55
8	0	18	336	367
8	0	20	301	407
9	0	-20	371	440
9	0	-14	1193	-1379
9	0	-12	494	-487
9	0	-10	1121	-1067
9	0	-8	672	631
9	0	-6	614	771
9	0	-4	429	275
9	0	2	433	-394
9	0	6	696	808
9	0	8	447	-299
9	0	10	675	725
9	0	12	400	293
9	0	18	310	-63
10	0	-16	577	615
10	0	-10	1553	-1661
10	0	-6	447	-519
10	0	-4	493	496
10	0	-2	1092	-1001
10	0	0	1133	1272
10	0	2	1763	1855
10	0	6	331	-420
10	0	12	652	766
11	0	-18	384	-477
11	0	-10	757	769
11	0	-8	521	620
11	0	-6	327	-316
11	0	-4	550	-574
11	0	-2	607	514
11	0	0	1256	1379
11	0	12	282	-214
12	0	-10	485	731
12	0	-6	485	504
12	0	-4	305	-150
12	0	0	407	-432
12	0	2	498	-526
12	0	6	271	-143
13	0	-12	325	-302
13	0	-10	407	-386
13	0	-4	234	95
13	0	-2	235	178
13	0	0	199	-51
13	0	6	480	-521
0	1	1	6201	8185
0	1	2	3933	3449
0	1	3	649	-689
0	1	4	7960	6172
0	1	5	1409	1417
0	1	6	362	-390
0	1	8	3427	-3180
0	1	9	2315	1903
0	1	10	3559	-2992
0	1	11	679	-486
0	1	12	665	-745
0	1	13	840	735
0	1	14	370	-441
0	1	16	1179	1498
0	1	18	529	942
0	1	22	294	-196
0	1	24	277	-590
0	1	26	174	-407
1	1	-22	444	451
1	1	-16	728	-1084
1	1	-14	1034	-1349
1	1	-13	1042	1248
1	1	-12	2566	-3257
1	1	-11	935	651
1	1	-10	2548	2748
1	1	-9	2757	3303
1	1	-7	1930	-2142
1	1	-6	4266	5323
1	1	-5	2930	3882
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1	1	-3	419	395
1	1	-2	7772	-8930
1	1	-1	4854	6091
1	1	0	2904	-3033
1	1	1	6436	-9085
1	1	2	1227	-1140
1	1	3	3366	3038
1	1	4	336	304
1	1	5	1681	-1877
1	1	6	1517	1436
1	1	7	1089	1057
1	1	8	2718	2575
1	1	9	2564	-2639
1	1	10	3334	3091
1	1	11	1395	-1235
1	1	12	1450	-1224
1	1	14	710	-543
1	1	16	2527	-2618
1	1	17	569	-831
1	1	18	506	-620
1	1	22	322	771
2	1	-24	398	-609
2	1	-23	276	82
2	1	-22	443	-660
2	1	-21	409	-677
2	1	-19	533	702
2	1	-18	859	1111
2	1	-16	589	1453
2	1	-15	529	575
2	1	-12	792	-1401
2	1	-10	1637	-2851
2	1	-9	1539	-2639
2	1	-8	456	-628
2	1	-7	689	1180
2	1	-6	630	-1319
2	1	-5	1272	1169
2	1	-4	2776	2325
2	1	-3	2307	-1737
2	1	-2	6948	7154
2	1	-1	4831	-5408
2	1	0	7708	10484
2	1	1	4975	-4311
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2	1	5	2097	-2318
2	1	6	5438	-5435
2	1	7	682	-781
2	1	8	639	-397
2	1	9	969	-917
2	1	10	2683	2718
2	1	11	576	302
2	1	12	2012	1997
2	1	13	683	-683
2	1	14	461	478
2	1	15	1452	-1477
2	1	16	681	-600
2	1	19	421	479
3	1	-22	292	-135
3	1	-20	895	-1080
3	1	-19	424	772
3	1	-15	1105	1161
3	1	-14	1240	1501
3	1	-13	1187	-1041
3	1	-12	3852	3480
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The hydrogen positions are shown in one of the molecules in Fig. 1. Though the standard deviations of the hydrogen parameters are very large, a fairly good impression of the molecular conformation is obtained. It is apparent that the methyl groups at C(8) and C(9) have different orientations with respect to the benzene ring. The positions adopted may be determined by the spatial requirement of the oxaziridine ring, but intermolecular forces may just as well exert the major influence.

The packing of the molecules in the cell does not appear very compact. One contact, however, is of an unusual character. The hydrogen atom attached to C(7) points towards an oxygen atom in the neighbouring unit cell in the **b** direction. It seems justified to describe this contact as an intermolecular C–H···O bridge for the following reasons. The carbon–hydrogen bond in question is very polar since the carbon is attached to three electron withdrawing ligands, *i.e.* oxygen, nitrogen and a substituted phenyl group. The distances involved are C···O 3·34 Å, C–H 1·05 Å, H···O 2·30 Å, and this particular H···O distance is about 0·5 Å shorter than any other intermolecular distance found in this structure between hydrogen and one of the other kinds of atom. Furthermore the angles N–O···C and C–O···C are 100 and 116° respectively, in good accordance with the conception of oxygen as acceptor atom in a hydrogen bridge.

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References

Brehm, L., Jensen, K. G. & Jerslev, B. (1966). *Acta Chem. Scand.* **20**, 915.

Jensen, B. & Jerslev, B. (1966). *Acta Cryst.* **21**, A118.

Abrahamsson, S., Aleby, S., Larsson, K., Nilsson, B., Selin, K. & Westerdahl, A. (1965). *Acta Chem. Scand.* **19**, 758.

Acta Cryst. (1967). **23**, 649

The Crystal Structure Determination of the Complex $(C_6H_5)_3P \rightarrow Au \rightarrow Mn(CO)_4P(OC_6H_5)_3$

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Diamagnetism and the short Au–Mn distance of 2·57 ± 0·01 Å found in the complex $(C_6H_5)_3P \rightarrow Au{-}Mn(CO)_4P(OC_6H_5)_3$ indicate a direct metal to metal bonding between the dissimilar metal atoms. The crystals are triclinic, space group $P\bar{1}$, with two molecules in a cell of dimensions $a = 10{\cdot}76$, $b = 10{\cdot}63$, $c = 16{\cdot}74$ Å, $\alpha = 93{\cdot}6°$, $\beta = 97{\cdot}0°$, $\gamma = 77{\cdot}8°$. The structure consists of discrete molecules. The manganese is octahedrally coordinated. Taking the metal to metal bond direction along the Z axis, the carbonyl groups on the Z plane are in a staggered configuration and are bent towards the metal to metal bond direction. The Mn–P bond of length 2·33 ± 0·01 Å appears to be multiple in nature. The P–Au–Mn bond angle of 165·5 ± 0·3° is significantly non-linear.

Introduction

As a part of a study of metal to metal bonds, the structure determination of the complex triphenylphosphinegold–tetracarbonyltriphenoxyphosphinemanganese, $(C_6H_5)_3P \rightarrow Au{-}Mn(CO)_4P(OC_6H_5)_3$, was undertaken.

Complexes of the type $Ph_3P \rightarrow Au{-}Mn(CO)_4L$ have been prepared, where Ph ≡ phenyl, L ≡ pyridine, $AsPh_3$ or PPh_3. The carbonyl stretching frequencies for all these complexes have been studied. It has been found that the mean carbonyl stretching frequency is practically constant at about 1935 cm^{-1}. For $L \equiv P(OPh)_3$ this increases to 1969 cm^{-1}. This fact has been attributed to the greater π-bonding capacity of the $P(OPh)_3$ group competing with the CO group (Coffey, Lewis & Nyholm, 1964).

Experimental

The crystal belongs to the triclinic system. Cell constants obtained from three zero-layer Weissenberg photographs around each axis with Cu $K\alpha$ radiation are:

$$a = 10{\cdot}76 \pm 0{\cdot}01,\quad b = 10{\cdot}63 \pm 0{\cdot}01,\quad c = 16{\cdot}74 \pm 0{\cdot}014\ \text{Å};$$
$$\alpha = 93{\cdot}6 \pm 0{\cdot}1°,\quad \beta = 97{\cdot}0 \pm 0{\cdot}1°,\quad \gamma = 77{\cdot}8 \pm 0{\cdot}1°.$$

The observed density measured by flotation is $1{\cdot}68_5$ g.cm^{-3}. Thus there are two molecules in the unit cell.

By the equi-inclination technique, nine layers along the a axis and three layers along the b axis were recorded, using Cu $K\alpha$ radiation. The total number of independent reflexions collected from these photographs amounted to 2760. The multiple film technique was