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The Crystal and Molecular Structure of Tris-acetylacetonatoruthenium(III)

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The compound $(C_5H_7O_2)_3Ru$ crystallizes in the monoclinic space group $P2_1/c$, with $a = 13{\cdot}86$ (1), $b = 7{\cdot}53$ (1), $c = 16{\cdot}01$ (4) Å, and $\beta = 99{\cdot}10$ (1)°. The central ruthenium atom is surrounded octahedrally by six oxygen atoms. The average Ru–O distance is 2·00 Å. The crystal is isomorphous with the tris-acetylacetonato complexes of Al, Co, Cr, Mn and Rh.

Introduction

Acetylacetone is the most common of the β-diketones. The presence of at least one hydrogen atom on the carbon between the two carbonyl groups allows the occurrence of a keto–enol tautomerism (Gould, 1959); the hydrogen atom may readily be replaced by a metal to form a complex compound. Complexes with β-diketones have been reported for all of the non-radioactive metallic or metalloidal elements of the periodic table (Fackler, 1966). Since no diffraction studies had been reported on complexes other than the first row, and since linkage isomerism is known to occur in complexes of β-diketones (Thompson, 1971), the crystal structure of tris-acetylacetonatoruthenium(III) was determined.

Experimental

A crystalline sample of the compound was purchased from Research Organic/Inorganic Chemical Corporation. Rotation and Weissenberg photographs obtained from a needle-like crystal showed reciprocal lattice symmetry C_{2h}–$2/m$. The systematic absences of $h0l$ reflections when l is odd and $0k0$ reflections when k is odd lead unambiguously to the space group $P2_1/c$. Unit-cell parameters were averaged from measurements on rotation, Weissenberg and precession photographs. A crystal density of 1·54 g cm^{-3} was determined by flotation in KI solution. This is in agreement with the value of 1·59 g cm^{-3} calculated on the basis of four molecules of $(C_5H_7O_2)_3Ru$ per unit cell. Film data were collected from a crystal whose diameter was about 0·17 mm. The corresponding linear absorption coefficient of 83·3 cm^{-1} for Cu $K\alpha$ radiation gives rise to an optimum crystal size (Stout & Jensen, 1968) of $t_{\text{opt}} = 2/\mu$ or 0·24 mm. Since the actual crystal was 0·17 mm, no correction for absorption was made.

Equi-inclination Weissenberg photographs using a Nonius Integrating Camera and Cu $K\alpha$ radiation ($\lambda = 1{\cdot}5418$ Å) were taken for hkl, $0 \le k \le 6$. The intensities were visually estimated with a calibrated intensity film strip and the data of different levels were correlated to a single scale by comparison of the lengths of exposures. The structure is based on 1317 symmetry independent observed reflections thus obtained.

Determination of the structure

A Patterson function was computed, in the expectation that the Ru position would readily be found by examination of vectors between symmetry related Ru atoms. In searching the Harker plane $(u,\frac{1}{2},w)$ and the Harker line $(0,v,\frac{1}{2})$ two possible Ru positions were found. Since the Ru y-coordinate is close to 0·25, the Patterson vector for the single interaction $2x,2y,2z$ could not be differentiated from the $2x,\frac{1}{2},\frac{1}{2}+2z$ peak in the Harker plane. It was found that possible Ru position A (0·24, 0·25, 0·22) afforded a lower initial R value (0·50) than did Ru position B (0·26, 0·25, 0·03) which gave an R value of 0·53, and so Ru (A) was refined further and the rest of the structure developed by usual heavy atom procedures. The ensuing structure appeared unsatisfactory, however, and failed to refine below an R value of 0·17. Ru(A) was then discarded and six oxygen atoms and 15 carbon atoms were located without difficulty from an electron density map generated by Ru(B). At this point, the R value was 12·5% with all atoms isotropic and the refinement by diagonal approximation.

Refinement of the structure

Because of computer core limitations, the maximum number of parameters which may be refined by full-matrix least-squares refinement on our CDC 3150 computer is 65. Consequently, it was possible to refine a maximum of seven anisotropic atoms in each full-matrix refinement, with the parameters of the remaining atoms constant. Therefore, four passes of three cycles each of full-matrix least-squares refinement were carried out. First the ruthenium atom and six oxygen atoms were refined resulting in an R index equal 10·2%. Second, the first ring (consisting of two oxygen atoms and five carbon atoms) was refined; the R index dropped to 10·0%. Separate refinement of the second and third rings resulted in R values of 9·9 and 9·7%, respectively. Finally, all 22 atoms were refined by diagonal approximation with the final R index equal to 9·6%. No positional parameter shifted by more than 19% of its standard deviation.

Fourier, least-squares and distance calculations were performed using the programs *FORDAP*, *LSLONG* and *DISTAN* given to us by A. Zalkin. The full-matrix least-squares program minimizes the function $\sum w(|F_o| - |F_c|)^2/\sum wF_o^2$; F_o and F_c are the observed and calculated structure factors, respectively and w is the weighting factor. For each reflection w was set to $1{\cdot}0\sigma^2(F_o)$. Atomic scattering factors were obtained from Cromer & Waber (1965). A correction for anomalous dispersion for ruthenium was included in the least-squares calculations (Cromer, 1965). No hydrogen atoms were introduced in the calculations, because few peaks in a difference map could be assigned unambiguously to reasonable hydrogen positions.

The final values of the positional and anisotropic thermal parameters obtained from the least-squares refinement are given in Tables 1 and 2. Observed and calculated structure factors are listed in Table 3.

Table 1. *Positional coordinates for non-hydrogen atoms**

	x	y	z
Ru	0·2376 (1)	0·2688 (3)	0·4727 (1)
O(1)	0·111 (1)	0·344 (3)	0·410 (1)
O(2)	0·179 (1)	0·185 (3)	0·575 (1)
O(3)	0·367 (1)	0·186 (3)	0·526 (1)
O(4)	0·301 (2)	0·356 (3)	0·377 (1)
O(5)	0·211 (2)	0·027 (2)	0·425 (1)
O(6)	0·259 (2)	0·512 (3)	0·524 (1)
C(1)	0·031 (2)	0·319 (6)	0·430 (2)
C(12)	0·015 (2)	0·228 (8)	0·509 (2)
C(2)	0·087 (2)	0·184 (5)	0·574 (2)
C(*X*1)	−0·055 (3)	0·375 (7)	0·366 (3)
C(*X*2)	0·066 (3)	0·113 (7)	0·660 (2)
C(3)	0·433 (2)	0·294 (5)	0·565 (2)
C(36)	0·421 (2)	0·472 (5)	0·583 (2)
C(6)	0·339 (2)	0·568 (4)	0·564 (1)
C(*X*3)	0·530 (2)	0·208 (6)	0·597 (2)
C(*X*6)	0·335 (3)	0·763 (5)	0·596 (2)
C(4)	0·307 (2)	0·252 (6)	0·312 (2)
C(45)	0·273 (2)	0·081 (4)	0·299 (1)
C(5)	0·231 (2)	−0·023 (4)	0·357 (2)
C(*X*4)	0·353 (2)	0·349 (4)	0·247 (2)
C(*X*5)	0·205 (2)	−0·221 (5)	0·332 (2)

* Numbers in parentheses in this and subsequent tables are estimated standard deviations of the last digits.

Table 2. *Anisotropic temperature factors*

The temperature factors are of the form $\exp(-\sum_i\sum_j B_{ij}h_ih_jb_ib_j/4)$ where b_i is the length of the ith reciprocal cell dimension. Units of the B_{ij} are Å^2.

	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Ru	2·87	4·54	2·28	0·38	0·46	0·05
O(1)	3·5	6·6	6·0	0·5	−0·1	−0·4
O(2)	4·9	6·7	2·7	−0·7	0·2	−0·3
O(3)	2·6	6·2	2·3	0·5	0·6	0·3
O(4)	6·2	5·4	3·3	0·0	2·3	0·7
O(5)	5·7	3·8	3·4	−1·6	0·4	−0·6
O(6)	6·1	5·5	3·7	0·4	0·5	−0·1
C(1)	3·8	12·1	4·0	0·9	−0·9	−3·6
C(12)	4·2	16·8	8·0	3·5	0·4	−4·8
C(2)	4·5	9·2	6·0	−1·0	1·3	−1·8
C(*X*1)	6·1	16·5	6·4	5·0	−2·6	−3·7
C(*X*2)	8·4	14·0	6·0	−0·6	4·0	0·8
C(3)	3·5	8·2	3·5	1·5	0·9	3·3
C(36)	4·6	6·0	3·2	1·6	1·1	0·2
C(6)	5·4	4·7	2·4	0·1	1·5	−0·9
C(*X*3)	3·8	10·5	7·2	1·6	0·9	3·4
C(*X*6)	9·1	4·9	5·3	−2·4	1·8	−2·2
C(4)	4·0	8·6	2·7	1·8	−0·1	0·2
C(45)	5·8	5·8	0·5	−1·3	0·0	−0·9
C(5)	3·4	5·1	2·9	1·7	−1·6	−0·9
C(*X*4)	9·9	6·7	1·6	−1·5	1·9	1·5
C(*X*5)	5·4	4·4	6·3	−0·6	−0·9	−1·9

Discussion of the structure

The molecular structure of tris-acetylacetonatoruthenium(III) is shown in Fig. 1. In order to facilitate a comparison of structural details with similar complexes, the numbering system used is the same as that reported for $(C_5H_7O_2)_3M$: M = Al and Co (Hon & Pfluger, 1973); M = Cr and Mn (Morosin, 1965),

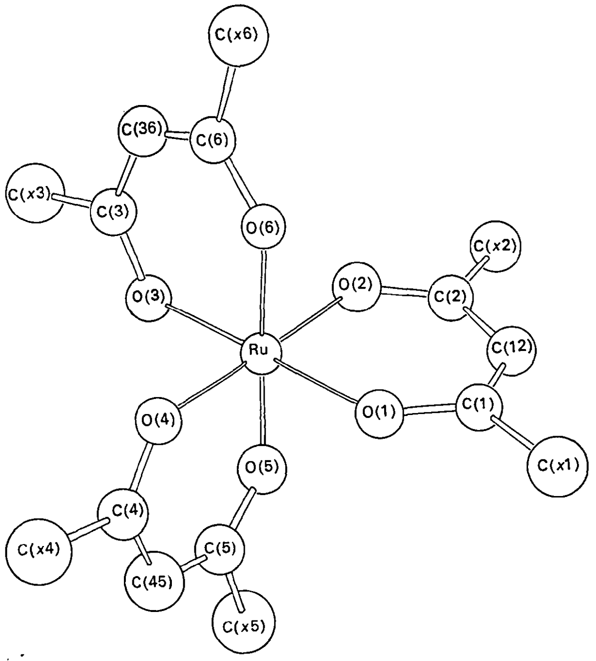

Fig. 1. Structure and labeling scheme for tris-acetylacetonato-ruthenium(III).

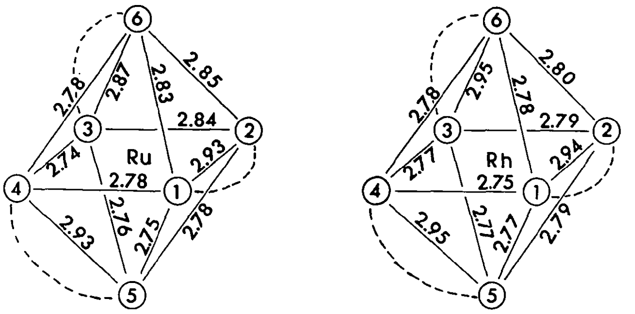

Fig. 2. Comparison of the dimensions of the oxygen octahedra of two tris-acetylacetonatometal(III) complexes: ruthenium and rhodium.

(Morosin & Brathovde, 1964). Along with M=Rh (Morrow & Parker, 1973), the aforementioned complexes and the complex reported here form a six-membered set of isomorphous crystals. The bond lengths and angles of equivalent atoms are listed in Tables 4 and 5 in the same way as the corresponding data are listed for the Al and Co complexes (*vide supra*) to facilitate comparisons. Average ring bond distances and angles are shown in Fig. 2. The average C(1)–C(*X*1) bond distance of 1·53 Å agrees well with the normal carbon–carbon single bond distance. The ring carbon–carbon distance of 1·40 Å displays the expected shortening due to delocalization of the π electrons in the six-membered rings. Equations for the planes defined by three sets of eight atoms each were calculated and the distances from each atom to its plane were calculated; the average deviations of the atoms in planes 12, 36 and 45 are 0·032, 0·038 and 0·021 Å respectively. It has been observed (Hon & Pfluger, 1973) that the 12 ring, which lies nearly perpendicular to the *b* axis, undergoes unusually large thermal motion along the *b* axis. This appears to be the case with tris(AcAc)$_3$Ru(III) as well, and to some extent for all of the complexes listed in Table 6.

Table 3. *Observed and calculated structure factors*

FCA(0,0,0) = 726

L	FOB	FCA
H,K= 0, 0		
2	190	206
4	44	47
9	23	-0
H,K= 1, 0		
2	94	110
4	37	-28
6	99	90
8	85	83
10	81	78
12	70	74
14	33	31
H,K= 2, 0		
0	150	-231
1	13	0
2	76	-80
4	25	-22
6	33	-36
10	21	19
12	58	55
14	62	66
16	42	44
18	41	49
H,K= 3, 0		
0	42	36
2	160	-176
4	31	-40
6	69	-78
8	75	-82
10	69	-69
12	52	-55
H,K= 4, 0		
0	131	157
2	79	94
4	67	70
7	89	-0
10	40	-41
12	50	-51
14	48	-48
16	37	-35
H,K= 5, 0		
0	46	50
2	130	138
4	131	139
6	106	110
8	65	62
12	44	49
H,K= 6, 0		
0	111	-113
2	95	-106
8	27	26
14	36	41
16	34	42
H,K= 7, 0		
0	77	-80
2	85	-88
4	75	-80
6	65	-69
8	58	-61
H,K= 8, 0		
0	62	60
2	58	52
4	48	49
6	42	25
10	29	-32
12	38	-44
14	44	-52
16	31	-41
H,K= 9, 0		
0	69	72
2	85	81
4	70	76
6	43	42
8	27	27
H,K= 10, 0		
0	46	-48
8	26	29
10	42	44
12	36	37
H,K= 11, 0		
0	49	-39
6	46	-48
8	47	-46
H,K= 1, 0		
-2	76	-66
-4	15	-14
-6	36	-42
-8	98	-95
-10	91	-92
-12	69	-62
H,K= 2, 0		
-2	136	-152
-4	207	-213
-6	136	-136
-8	35	-28
-12	44	37
-14	28	25
-16	39	37
-18	49	51
-20	28	33
H,K= 3, 0		
-2	126	121
-4	87	84
-6	125	128
-8	146	143
-10	85	78
-12	43	42
-16	47	52
H,K= 4, 0		
-2	144	146
-4	109	111
-6	128	130
-8	90	87
-10	32	27
-12	36	-33
-16	37	-43
-18	40	-43
-20	25	-30
H,K= 5, 0		
-2	25	25
-4	23	-23
-6	86	-83
-8	84	-81
-10	98	-100
-12	58	-49
-14	40	-39
-16	39	-45
H,K= 6, 0		
-2	83	-85
-4	106	-108
-6	115	-112
-8	59	-53
-12	34	26
-16	42	43
-18	35	37
-20	18	29
H,K= 7, 0		
-4	22	24
-6	43	39
-8	51	49
-10	81	78
-12	36	33
-14	51	56
-16	43	47
H,K= 8, 0		
-2	117	126
-4	134	148
-6	97	91
-8	82	82
-10	35	32
-16	28	-32
-18	31	-38
H,K= 9, 0		
-2	37	36
-4	27	-25
-6	47	-43
-8	36	-29
-14	39	-47
-16	34	-35
H,K= 10, 0		
-2	85	-89
-4	100	-98
-6	81	-79
-8	78	-74
H,K= 11, 0		
-2	62	-58
-8	38	40
-10	68	69
-12	47	51
-14	39	39
-16	26	28
H,K= 12, 0		
-2	59	63
-4	68	72
-6	51	55
-8	44	44
H,K= 13, 0		
-2	32	28
-10	27	-33
H,K= 14, 0		
-2	37	-36
-4	32	-31
-6	26	-31
-8	35	-40
-10	28	-35
H,K= 0, 1		
2	62	60
3	60	54
4	72	62
5	22	-18
6	71	73
8	74	74
9	23	15
10	93	96
12	77	75
H,K= 1, 1		
1	45	-46
2	122	-141
3	19	-16
4	75	-72
6	26	-25
9	34	32
12	41	38
14	50	48
H,K= 2, 1		
0	30	-29
1	11	10
2	149	-143
3	15	-14
4	55	-61
6	63	-62
7	33	-32
8	60	-66
9	44	-40
10	100	-99
12	79	-84
14	44	-38
H,K= 3, 1		
0	83	89
1	75	75
2	152	157
4	71	74
5	28	-31
6	39	34
10	43	-43
12	67	-66
14	61	-59
16	35	-37
H,K= 4, 1		
1	38	38
2	105	105
3	80	80
4	107	105
5	39	31
6	95	98
8	92	95
10	73	69
12	43	44
H,K= 5, 1		
0	98	-107
1	61	-61
2	151	-146
4	55	-48
8	33	32
10	42	34
12	38	35
14	38	37
16	28	32
H,K= 6, 1		
0	53	-48
1	42	-35
2	106	-102
3	62	-57
4	116	-121
6	76	-76
8	68	-77
10	54	-56
12	40	-41
H,K= 7, 1		
0	85	91
1	40	36
2	80	81
5	29	-26
10	29	-29
12	29	-36
14	40	-42
H,K= 8, 1		
0	31	31
2	76	73
3	37	29
4	96	98
6	70	72
8	52	52
H,K= 9, 1		
0	52	-53
2	40	-38
10	37	36
12	36	41
14	34	39
H,K= 10, 1		
0	53	-50
2	57	-58
4	58	-56
6	48	-48
8	42	-44
10	27	-26
H,K= 11, 1		
0	32	31
10	38	-34
H,K= 12, 1		
2	28	31
4	44	43
6	51	50
8	39	35
H,K= 14, 1		
0	48	-51
2	45	-49
4	35	-39
6	29	-34
H,K= 1, 1		
-2	186	-186
-3	38	23
-4	181	-175
-6	59	-51
-7	37	33
-12	32	29
-14	44	38
-16	37	35
-18	48	50
H,K= 2, 1		
-1	12	-12
-2	18	19
-3	107	-99
-4	131	117
-5	62	-56
-6	155	151
-7	60	-47
-8	134	119
-10	94	83
-12	70	65
-16	32	33
H,K= 3, 1		
-2	26	20
-4	91	93
-5	47	-44
-6	127	110
-7	33	-33
-8	90	88
-10	58	49
-16	37	-39
-18	40	-44
H,K= 4, 1		
-1	29	-24
-2	47	34
-4	52	-45
-5	48	41
-6	147	-141
-7	63	55
-8	127	-120
-10	108	-102
-12	65	-67
-14	30	-38
-16	37	-41
H,K= 5, 1		
-2	73	-77
-4	103	-104
-5	39	26
-6	90	-80
-8	32	-29
-16	35	36
-18	28	32
H,K= 6, 1		
-1	21	-19
-2	64	-58
-3	19	-17
-5	32	-26
-6	68	61
-7	23	-25
-8	75	67
-10	101	94
-12	67	63
-14	55	62
-16	39	47
H,K= 7, 1		
-2	85	85
-4	107	107
-6	73	69
-8	47	40
-10	29	26
-16	37	-38
-18	39	-32
H,K= 8, 1		
-5	46	-37
-8	41	-35
-10	45	-42
-12	40	-40
-14	57	-58
-16	41	-42
H,K= 9, 1		
-2	91	-96
-4	123	-121
-6	94	-91
-8	66	-61
H,K= 10, 1		
-2	46	-38
-8	36	34
-10	36	35
-12	39	43
-14	44	44
-16	26	31
H,K= 11, 1		
-2	51	64
-4	75	77
-6	64	67
-8	44	44
H,K= 12, 1		
-10	39	-41
-12	45	-49
-14	42	-44
-16	22	-28
H,K= 13, 1		
-2	30	-33
-4	38	-37
-6	43	-46
-8	37	-43
-10	27	-34
H,K= 0, 2		
1	83	87
2	135	-137
3	98	96
4	92	-80
5	38	34
6	54	-46
9	46	44
12	25	25
14	51	52
16	40	37
18	30	31
H,K= 1, 2		
0	20	-18
1	17	15
2	65	-56
3	48	42
4	61	-58
6	95	-94
7	33	-32
8	105	-102
9	32	-33
10	95	-86
12	80	-77
14	42	-41
H,K= 2, 2		
0	81	74
2	72	73
3	45	-46
4	70	73
5	47	-46
6	50	44
9	35	-37
10	36	-36
12	45	-42
14	58	-58
16	35	-37
H,K= 3, 2		
1	44	44
2	46	40
4	72	71
5	27	31
6	111	109
7	46	43
8	108	102
9	25	22
10	89	79
12	66	58
H,K= 4, 2		
0	70	-69
2	79	-88
4	74	-67
6	43	-34
7	21	26
10	42	38
12	56	49
14	55	50
16	29	27
H,K= 5, 2		
0	38	-31
1	61	-54
2	86	-85
3	76	-72
4	136	-135
6	113	-113
7	35	31
8	82	-77
10	57	-56
12	42	-43
H,K= 6, 2		
0	46	43
1	40	37
2	70	78
4	50	54
5	23	-24
8	28	-26
10	40	-35
12	38	-37
14	38	-34
H,K= 7, 2		
0	48	48
2	58	57
4	105	96
6	80	70
8	59	57
10	51	48
12	25	25
H,K= 8, 2		
0	50	-51
2	60	-50
4	43	-37
5	41	36
10	32	29
12	39	40
H,K= 9, 2		
0	54	-48
2	62	-50
4	92	-88
6	63	-64
8	40	-37
10	34	-31
H,K= 10, 2		
0	36	35
1	31	-29
2	27	26
6	30	-32
8	25	-22
10	25	-26
12	33	-34
H,K= 11, 2		
0	31	29
2	33	31
4	64	59
6	56	57
8	35	29
10	29	27
H,K= 12, 2		
6	29	27
H,K= 13, 2		
0	42	-40
2	43	-44
4	54	-48
6	36	-36
H,K= 1, 2		
-1	49	-43
-3	55	-47
-4	33	35
-5	20	16
-6	108	103
-7	41	-34
-8	137	123
-10	98	86
-12	81	68
-14	37	30
H,K= 2, 2		
-2	66	55
-3	21	-13
-4	74	72
-6	87	79
-8	53	48
-9	40	32
-14	44	-41
-16	51	-48
-18	41	-39
H,K= 3, 2		
-1	14	9
-4	63	-61
-5	19	20
-6	129	-112
-7	60	54
-8	143	-130
-9	31	-29
-10	119	-107
-12	87	-77
-14	40	-41
H,K= 4, 2		
-1	32	26
-2	93	-82
-3	24	-13
-4	83	-78
-5	44	39
-6	70	-61
-8	44	-35
-9	46	-39
-10	32	-28
-16	33	33
-18	30	33
H,K= 5, 2		
-1	33	-30
-5	20	-20
-6	47	36
-7	26	-23
-8	82	71
-10	102	95
-12	89	84
-14	67	66
-16	33	31
H,K= 6, 2		
-1	29	25
-2	63	60
-4	77	79
-5	43	-40
-6	69	64
-8	51	44
-9	31	25
-16	28	-26
-18	23	-25
H,K= 7, 2		
-1	48	43
-2	28	24
-3	27	23
-4	23	-23
-5	22	19
-6	45	-37
-8	40	-42
-10	58	-54
-12	61	-62
-14	63	-67
-16	44	-44
H,K= 8, 2		
-2	78	-75
-4	86	-78
-6	65	-66
-8	61	-59
-10	31	-30
H,K= 9, 2		
-8	30	27
-10	35	33
-12	38	42
-14	44	46
-16	31	35
H,K= 10, 2		
-1	31	-30
-2	63	64
-4	64	67
-6	57	57
-8	48	45
H,K= 11, 2		
-10	35	-35
-12	44	-45
-14	41	-45
-16	29	-32
H,K= 12, 2		
-2	44	-41
-4	46	-47
-6	45	-49
-8	37	-38
H,K= 13, 2		
-12	24	31
-14	26	31
H,K= 0, 3		
1	20	-22
2	9	-5
3	18	11
4	44	-38
5	24	18
6	62	-59
8	83	-78
10	69	-65
12	59	-55
14	34	-36
H,K= 1, 3		
0	80	95
1	91	-86
2	123	117
3	103	-97
4	89	86
5	55	-51
6	44	41
7	19	-17
9	44	-39
10	32	-32
11	25	-21
12	28	-29
14	31	-29
16	23	-28
H,K= 2, 3		
0	12	11
1	40	36
2	39	34
3	34	-28
4	63	62
6	60	54
7	20	22
8	93	90
9	33	32
10	75	72
12	47	44
14	23	26
H,K= 3, 3		
0	82	-84
1	21	21
2	99	-94
3	54	53
4	55	-49
5	36	33
6	25	-22
7	34	33
9	37	37
10	39	35
11	25	19
12	24	24
14	32	37
16	34	40
H,K= 4, 3		
0	23	-19
1	61	-57
2	45	-43
3	20	-22
4	94	-81
6	77	-73
8	87	-83
10	55	-52
12	22	-24
H,K= 5, 3		
0	78	76
2	75	73
3	31	-30
4	31	33
5	18	-17
6	26	17
7	24	-24
10	41	-42
12	32	-32
14	27	-31
16	31	-31
H,K= 6, 3		
0	51	48
1	36	31
2	70	69
4	74	72
5	29	-24
6	50	57
8	46	47
10	45	41
H,K= 7, 3		
0	55	-60
2	50	-44
3	35	26
4	23	-21
10	36	36
12	29	32
14	27	26
H,K= 8, 3		
0	37	-31
2	54	-52
4	66	-68
6	51	-49
8	43	-41
10	27	-26
H,K= 9, 3		
0	52	52
1	26	-25
2	39	34
7	24	-23
9	23	-24
10	35	-37
12	33	-34
H,K= 10, 3		
0	46	44
2	47	43
4	48	49
6	41	42
8	31	29
H,K= 11, 3		
0	32	-35
1	30	27
8	21	23
10	25	27
H,K= 12, 3		
0	34	-33
2	36	-35
4	36	-34
6	36	-30
H,K= 1, 3		
-1	35	33
-2	129	123
-3	82	76
-4	112	105
-5	81	74
-6	75	69
-7	37	30
-8	21	23
-9	28	26
-12	32	-30
-14	34	-35
-16	27	-34
-18	21	-28
H,K= 2, 3		
-2	15	-13
-3	43	39
-4	64	-59
-6	78	-70
-8	66	-58
-9	22	-25
-10	67	-64
-11	25	-24
-12	57	-58
-14	39	-39
H,K= 3, 3		
-2	78	-80
-4	80	-79
-6	75	-66
-7	30	-27
-8	56	-52
-9	46	-41
-10	22	-24
-11	28	-29
-14	34	37
-16	30	34
-18	23	30
H,K= 4, 3		
-1	32	-31
-3	32	-28
-4	52	49
-5	25	-22
-6	68	65
-8	66	61
-10	67	63
-12	54	52
-14	38	42
-16	24	24
H,K= 5, 3		
-2	87	88
-4	93	88
-6	64	66
-7	43	42
-8	52	48
-9	40	37
-10	25	23
-11	25	24
-16	23	-24
-18	23	-29
H,K= 6, 3		
-1	42	43
-2	10	17
-3	33	34
-4	23	-25
-5	40	42
-6	36	-36
-8	50	-45
-10	50	-49
-12	50	-54
-14	41	-43
-16	24	-28
H,K= 7, 3		
-2	72	-74
-4	66	-60
-6	55	-57
-8	58	-55
-9	27	-26
-10	36	-31
-15	27	26
-18	19	28
H,K= 8, 3		
-8	41	42
-10	47	46
-12	38	44
-14	26	28
H,K= 9, 3		
-1	33	-28
-2	68	67
-4	54	56
-6	57	58
-7	40	36
-8	49	46
-9	43	40
-10	27	31
H,K= 10, 3		
-8	27	-28
-10	34	-37
-12	36	-41
-14	31	-33
-16	18	-21
H,K= 11, 3		
-1	30	29
-2	50	-52
-4	43	-42
-6	45	-43
-8	31	-32
H,K= 12, 3		
-12	27	35
-14	26	34
H,K= 13, 3		
-2	29	35
-4	30	33
-6	39	42
-8	27	33
H,K= 0, 4		
1	36	-41
2	102	101
3	50	-48
4	64	64
5	52	-48
6	49	45
7	32	-27
11	24	-27
13	26	-26
14	26	-25
16	29	-32
H,K= 1, 4		
0	8	-10
1	47	46
2	29	27
4	53	52
6	79	70
8	80	73
10	34	28
12	23	24
14	20	22
16	28	13
H,K= 2, 4		
0	97	-101
1	22	20
2	59	-56
3	23	21
4	45	-37
5	39	37
6	43	-40
7	47	44
9	30	25
10	23	21
11	22	22
14	28	28
16	31	33
H,K= 3, 4		
0	31	-31
1	47	-40
2	53	-47
4	62	-57
5	24	-24
6	64	-56
8	60	-54
12	23	-21
14	21	-20
15	18	19
H,K= 4, 4		
0	100	95
2	57	51
3	25	-23
4	35	30
5	31	-30
6	28	24
7	35	-32
10	30	-24
12	25	-23
14	34	-33
16	24	-29
H,K= 5, 4		
0	60	55
1	53	44
2	66	65
3	30	23
4	44	37
6	37	31
8	46	45
10	31	29
H,K= 6, 4		
0	69	-64
2	33	-29
3	21	20
4	19	-20
5	23	25
7	22	20
10	34	33
12	19	21
14	20	25
H,K= 7, 4		
0	38	-34
2	46	-38
4	42	-36
6	40	-36
8	46	-39
10	37	-28
14	14	-7
H,K= 8, 4		
0	50	43
2	22	20
3	22	-21
5	21	-20
10	29	-27
12	27	-25
H,K= 9, 4		
0	44	42
2	44	39
4	41	38
6	36	32
8	32	28
H,K= 10, 4		
0	31	-30
2	24	-23
10	20	23
H,K= 11, 4		
0	42	-35
2	33	-31
3	21	-16
4	35	-31
6	24	-22
H,K= 1, 4		
-1	55	56
-2	29	-25
-3	48	45
-5	41	37
-6	56	-50
-8	68	-60
-10	49	-42
-12	36	-32
-14	23	-28
H,K= 2, 4		
-1	19	-12
-2	105	-103
-4	72	-66
-6	54	-47
-7	24	-21
-16	26	31
-18	22	29
H,K= 3, 4		
-1	54	-53
-2	30	26
-3	62	-62
-4	55	49
-5	50	-47
-6	62	52
-8	46	37
-10	47	46
-12	36	37
-14	28	33
H,K= 4, 4		
-2	134	99
-4	88	82
-6	45	41
-7	25	24
-8	27	26
H,K= 5, 4		
-1	50	48
-3	50	48
-4	32	-31
-5	51	53
-6	34	-26
-7	29	28
-8	45	-39
-10	51	-45
-12	40	-37
-14	25	-28
H,K= 6, 4		
-2	92	-94
-4	95	-74
-6	45	-41
-7	24	-20
-8	40	-39
H,K= 7, 4		
-1	26	-21
-3	40	-35
-5	44	-41
-8	38	33
-10	41	39
-12	34	40
H,K= 8, 4		
-2	72	67
-4	63	56
-5	28	26
-6	57	58
-7	32	31
-8	54	49
-9	28	30
H,K= 9, 4		
-10	34	-36
-12	28	-29
H,K= 10, 4		
-1	25	29
-2	45	-43
-4	36	-33
-6	46	-42
-8	45	-44
-9	28	-27
-10	24	-26
H,K= 11, 4		
-10	29	29
H,K= 12, 4		
-2	39	38
-4	29	38
-6	32	33
-8	32	33
H,K= 0, 5		
1	20	21
2	18	17
3	37	35
4	41	42
5	37	34
6	68	65
7	13	14
8	49	40
10	33	29
12	33	33
13	17	-19
14	20	21
15	13	-12
H,K= 1, 5		
0	26	-32
1	6	4
2	48	-48
3	23	19
4	51	-48
5	41	39
6	31	-27
7	37	32
9	25	24
11	24	23
14	20	19
16	18	20
H,K= 2, 5		
0	14	-14
1	29	-27
2	37	-36
3	30	-29
4	59	-57
5	31	-30
6	61	-54
7	26	-24
8	46	-42
9	16	-16
10	24	-23
12	27	-26
13	19	20
14	12	-12
15	15	17
H,K= 3, 5		
0	52	55
2	49	47
3	26	-21
4	35	28
5	22	-28
6	14	9
7	31	-26
9	19	-18
11	14	-15
12	15	-12
14	20	-23
H,K= 4, 5		
0	37	33
1	53	48
2	46	43
3	50	48
4	41	38
5	33	31
6	35	30
8	30	34
10	26	25
11	17	-18
12	24	25
13	18	-20
14	10	9
15	11	-12
H,K= 5, 5		
0	57	-58
2	40	-36
4	12	-11
5	12	9
7	21	18
9	23	21
10	21	18
11	19	19
12	13	15
14	20	24
H,K= 6, 5		
0	28	-27
1	47	-42
2	34	-36
3	26	-19
4	28	-24
6	37	-28
8	38	-38
10	29	-30
12	14	-15
13	11	15
H,K= 7, 5		
0	39	36
2	19	17
3	14	-13
5	20	-22
7	28	-27
9	23	-21
10	17	-17
11	13	-16
12	17	-19
H,K= 8, 5		
0	14	11
1	28	25
2	34	30
4	38	35
6	35	30
8	32	31
10	23	23
H,K= 9, 5		
0	18	-19
1	14	15
2	14	-16
3	25	21
5	24	19
7	20	21
9	18	13
10	16	17
12	12	18
H,K= 10, 5		
0	24	-23
1	25	-22
2	35	-34
3	16	-14
4	34	-35
6	23	-22
8	19	-19
10	13	-14
H,K= 11, 5		
0	18	17
1	16	-17
3	20	-20
H,K= 12, 5		
0	23	25
1	21	16
2	25	28
4	25	23
6	12	12
H,K= 1, 5		
-2	44	-44
-3	13	-11
-4	56	-53
-5	34	-29
-6	39	-32
-7	27	-23
-9	21	-17
-11	30	-28
-12	18	17
-13	18	-17
-14	21	20
-16	17	22
H,K= 2, 5		
-1	28	-27
-2	16	14
-3	48	-40
-4	40	35
-5	49	-43
-6	64	59
-7	16	-13
-8	31	21
-10	36	34
-12	41	37
-13	23	22
-14	28	27
-15	15	16
-17	11	15
H,K= 3, 5		
-2	51	48
-4	57	54
-5	16	12
-6	27	18
-7	15	13
-11	30	28
-13	18	18
-14	19	-19
-16	17	-17
H,K= 4, 5		
-1	34	31
-2	18	13
-3	41	36
-4	29	-26
-5	[illegible]	[illegible]
-6	57	[illegible]
-8	36	-31
-9	17	17
-10	45	-34
-12	36	-32
-14	34	-24
-15	15	-15
-16	13	-15
-17	11	-16
H,K= 5, 5		
-2	50	-58
-4	63	-59
-5	16	-14
-6	19	-17
-7	31	-18
-8	17	-17
-9	20	-19
-11	29	-26
-13	17	-16
-16	17	13
H,K= 6, 5		
-1	47	-43
-2	31	-28
-3	49	-43
-5	37	-36
-6	34	27
-8	31	29
-10	38	34
-12	37	36
-14	25	22
-16	16	17
H,K= 7, 5		
-2	50	48
-4	55	53
-6	35	31
-8	23	19
-11	26	23
-13	18	23
H,K= 8, 5		
-1	22	20
-2	17	14
-3	31	25
-5	30	25
-8	24	-26
-10	34	-36
-12	32	-32
-14	17	-21
-16	11	-18
H,K= 9, 5		
-2	33	-29
-4	38	-35
-6	38	-37
-7	17	-17
-8	30	-28
-9	20	-17
-11	18	-18
H,K= 10, 5		
-1	18	-18
-2	18	-16
-3	19	-18
-10	25	24
-12	19	20
-14	16	22
H,K= 11, 5		
-2	22	19
-4	18	19
-6	23	27
-8	25	26
H,K= 12, 5		
-1	17	15
-2	16	15
-3	16	14
-5	16	18
H,K= 13, 5		
-4	21	-23
-6	26	-29
-8	20	-23
H,K= 14, 5		
-3	14	-16
-5	18	-20
-7	14	-16
H,K= 0, 6		
1	4	3
2	15	-21
3	11	11
4	20	-28
5	15	21
6	18	-26
7	25	38
8	21	31
11	13	23
13	9	14
14	8	17
H,K= 1, 6		
1	12	-19
2	6	-9
3	12	-17
4	17	-24
5	14	-20
6	34	-47
8	28	-39
10	18	-26
12	15	-25
H,K= 2, 6		
0	21	32
2	17	23
3	12	-13
4	14	19
5	20	-20
6	10	14
7	27	-42
8	18	-28
H,K= 3, 6		
1	14	18
2	17	17
3	15	22
4	21	29
5	18	27
6	29	43
7	11	15
8	26	37
10	11	18
12	8	17
H,K= 4, 6		
0	22	-33
1	15	19
2	10	-13
3	11	14
5	11	22
7	16	25
9	12	22
H,K= 5, 6		
1	20	-27
2	15	-26
3	13	-19
4	18	-26
6	19	-30
8	19	-27
10	10	-17
H,K= 6, 6		
0	13	18
8	16	-10
10	12	-16
11	9	-13
H,K= 7, 6		
2	17	23
4	16	22
6	16	26
8	14	23
10	10	16
H,K= 8, 6		
3	10	18
5	16	25
7	14	23
9	11	17
H,K= 9, 6		
0	10	-16
1	9	-14
2	15	-20
3	9	-11
5	17	-6
7	13	6
9	9	9
10	7	-13
H,K= 10, 6		
2	13	15
4	12	1
6	12	-9
8	9	-12
H,K= 11, 6		
0	12	19
1	8	14
2	13	21
4	12	23
H,K= 1, 6		
-1	15	-24
-3	16	-21
-4	9	9
-5	19	-28
-6	27	32
-8	23	26
-10	21	32
-12	13	28
H,K= 2, 6		
-2	22	30
-3	9	12
-4	25	32
-6	11	14
-7	16	20
-9	19	25
-11	15	26
-13	12	20
H,K= 3, 6		
-1	20	31
-3	17	23
-5	14	20
-6	13	-21
-8	12	-16
-10	21	-31
-12	19	-30
-14	11	-20
H,K= 4, 6		
-2	26	-37
-4	31	-42
-6	13	-17
-7	12	-16
-9	15	-23
-11	14	-22
H,K= 5, 6		
-1	26	-41
-3	19	-25
-6	13	16
-10	17	26
-12	17	27
H,K= 6, 6		
-2	15	24
-4	28	36
-6	15	23
-8	14	21
-9	14	24
-11	15	26
-13	11	21
H,K= 7, 6		
-1	20	28
-3	19	28
-7	12	16
-10	14	-25
-12	13	-23
H,K= 8, 6		
-4	13	-20
-8	14	-21
-9	12	-17
-11	11	-20
H,K= 9, 6		
-1	13	-20
-2	12	-16
-3	12	-16
-10	14	25
-12	11	21
H,K= 10, 6		
-8	11	16
-9	11	13
-11	10	17
H,K= 11, 6		
-1	11	17

Table 4. *Interatomic distances* (Å)

Ru—O(1)	1·97	(2)
Ru—O(2)	2·04	(2)
Ru—O(3)	1·96	(2)
Ru—O(4)	2·01	(2)
Ru—O(5)	1·99	(2)
Ru—O(6)	2·01	(2)
Average	2·00	(2)
O(1)—C(1)	1·21	(4)
O(2)—C(2)	1·29	(4)
O(3)—C(3)	1·31	(3)
O(4)—C(4)	1·32	(4)
O(5)—C(5)	1·23	(3)
O(6)—C(6)	1·26	(4)
Average	1·27	(4)
C(1)—C(12)	1·49	(6)
C(12)-C(2)	1·37	(5)
C(4)—C(45)	1·38	(5)
C(45)-C(5)	1·42	(4)
C(6)—C(36)	1·34	(5)
C(36)-C(3)	1·39	(5)
Average	1·40	(5)
C(1)—C(*X*1)	1·51	(5)
C(2)—C(*X*2)	1·55	(5)
C(3)—C(*X*3)	1·50	(5)
C(4)—C(*X*4)	1·51	(4)
C(5)—C(*X*5)	1·57	(5)
C(6)—C(*X*6)	1·56	(5)
Average	1·53	(5)

Table 5. *Interatomic angles* (°)

O(1)-Ru—O(2)	94·0	(0·9)	O(1)—C(1)—C(12)	125	(3)
O(3)-Ru—O(6)	92·4	(0·9)	O(2)—C(2)—C(12)	127	(3)
O(4)-Ru—O(5)	94·7	(0·8)	O(3)—C(3)—C(36)	127	(3)
Average	93·7	(0·9)	O(4)—C(4)—C(45)	128	(3)
			O(5)—C(5)—C(45)	127	(3)
O(1)-Ru—O(3)	174·8	(0·8)	O(6)—C(6)—C(36)	126	(3)
O(2)-Ru—O(4)	176·8	(0·8)	Average	127	(3)
O(6)-Ru—O(5)	177·3	(0·9)			
Average	176·2	(0·8)	C(1)—C(12)-C(2)	125	(3)
			C(3)—C(36)-C(6)	127	(2)
O(1)-Ru—O(4)	88·6	(0·8)	C(4)—C(45)-C(5)	125	(2)
O(1)-Ru—O(5)	88·2	(0·9)	Average	126	(2)
O(1)-Ru—O(6)	90·8	(1·0)			
O(2)-Ru—O(3)	90·1	(0·7)	O(1)—C(1)—C(*X*1)	115	(3)
O(2)-Ru—O(5)	87·4	(0·8)	O(2)—C(2)—C(*X*2)	109	(2)
O(2)-Ru—O(6)	90·3	(0·9)	O(3)—C(3)—C(*X*3)	114	(3)
O(3)-Ru—O(4)	87·5	(0·8)	O(4)—C(4)—C(*X*4)	111	(3)
O(3)-Ru—O(5)	88·8	(0·8)	O(5)—C(5)—C(*X*5)	116	(2)
O(4)-Ru—O(6)	87·7	(0·9)	O(6)—C(6)—C(*X*6)	114	(2)
Average	88·8	(0·8)	Average	113	(2)
Ru—O(1)-C(1)	126	(2)	C(12)-C(1)—C(*X*1)	120	(3)
Ru—O(2)-C(2)	122	(2)	C(12)-C(2)—C(*X*2)	124	(3)
Ru—O(3)-C(3)	122	(2)	C(36)-C(3)—C(*X*3)	119	(2)
Ru—O(4)-C(4)	120	(2)	C(36)-C(6)—C(*X*6)	120	(2)
Ru—O(5)-C(5)	125	(2)	C(45)-C(4)—C(*X*4)	121	(2)
Ru—O(6)-C(6)	125	(2)	C(45)-C(5)—C(*X*5)	117	(2)
Average	123	(2)	Average	120	(2)

In addition to unit-cell parameters, average M–O, O–O 'bite' and O–O 'non-bite' distances are listed in Table 6 for the isomorphous series. 'Bite' *vs*. 'non-bite' oxygen–oxygen distances for the rhodium and ruthenium complexes are compared in Fig. 2. The individual distances for the rhodium complex were calculated from cell and positional parameters furnished by Morrow & Parker (1973). Similar figures for the other four members of the isomorphous series, aluminum, cobalt, chromium and manganese, as well as for vanadium and iron, have been published by Hon & Pfluger (1973).

Attempts have been made to correlate the O–O 'bite' distance with the M–O distance (Hon & Pfluger, 1973) and with the metal ion radius to charge ratio (Lingafelter & Braun, 1966), but with limited success. For comparison with the data of Lingafelter & Braun (1966) a value of 0·665 Å was used for the Rh(III) radius (Prewitt, Shannon, Rogers & Sleight, 1969) and a value of 0·69 Å was estimated for Ru(III). These values, along with the data listed in Table 6, do not add convincing support to either correlation, even in this isomorphous series. To further complicate attempts at such correlations, it should be noted that tris-(AcAc)$_3$Fe(III) (Iball & Morgan, 1967) and α-tris(AcAc)$_3$V(III) (Morosin & Montgomery, 1969) crystallize in the orthorhombic space groups *Pbca* and *Pcab*, respectively, while β-tris(AcAc)$_3$V(III) (Morosin & Montgomery, 1969) crystallizes in the monoclinic space group $P2_1/n$.

In addition, the O–O 'bite' distances are less than the O–O 'non-bite' distances for these complexes, while the opposite is true for the six isomorphous complexes

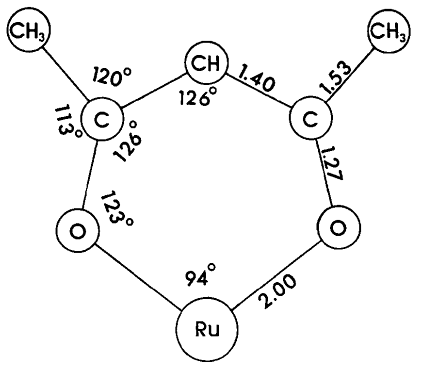

Fig. 3. Mean values of bond lengths and angles for the three rings in tris-acetylacetonatoruthenium(III).

Table 6. *Tris*(*acetylacetonato*)-M(III) *complexes forming an isomorphous series in space group* $P2_1/c$

					Distances (Å)			
					M–O	O–O	O–O	
Metal	a_0	b_0	c_0	β		Bite	Other	Reference
Al	14·069	7·568	16·377	99·00	1·892	2·726	2·666	Pfluger (1973)
Co	13·951	7·470	16·222	99·48	1·898	2·850	2·629	Pfluger (1973)
Cr	14·031	7·551	16·379	99·06	1·952	2·786	2·751	Morosin (1965)
Mn	13·875	7·467	16·20	98·42	1·901	2·851	2·634	Morosin (1964)
Rh	13·925	7·483	16·392	98·63	1·992	2·946	2·790	Morrow (1973)
Ru	13·86	7·53	16·01	99·10	1·997	2·910	2·790	This work

listed in Table 6. The structural details thus depend on complex factors, probably including the role played by unpaired *d* electrons in the central metal ion.

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The Crystal Structure of *trans*-9-Isopropyl-10-methyl-9,10-dihydroanthracene*

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The crystal structure of *trans*-9-isopropyl-10-methyl-9,10-dihydroanthracene, $C_{18}H_{20}$, has been determined by the application of known molecular geometry, symmetry, Patterson map and structure factor calculations. Three-dimensional data were collected on a Datex-automated General Electric diffractometer with Ni-filtered Cu $K\alpha$ radiation to a maximum value of 2θ of 120° ($d_{min}=0{\cdot}9$Å). The crystals are tetragonal, space group $I4_1md$, with $a=b=23{\cdot}1811$ (5) and $c=5{\cdot}2156$ (7) Å, and with eight molecules per unit cell. Each molecule lies on a mirror plane resulting in one half molecule per asymmetric unit. The data were collected as if the crystal were monoclinic; and the resulting four sets of equivalent data have been compared by the use of normal-probability plots. The coordinates of all atoms in the molecule, the isotropic temperature factors for the hydrogen atoms, and the anisotropic temperature factors for the carbon atoms were refined by the method of least squares. The final R index is 0·028. The structure is compared with that previously determined for *cis*-9-methyl-10-ethyl-9,10-dihydroanthracene [Bordner, Stanford & Zieger (1973), *Acta Cryst.* B**29**, 313–318].

Introduction

This compound, 9-isopropyl-10-methyl-9,10-dihydroanthracene, $C_{18}H_{20}$, was prepared by Zieger & Gelbaum (1972) during a study of the alkylation of 10-alkyl-9,10-dihydroanthracenyl-lithiums with alkyl iodides. The structural assignments for *cis* and *trans* stereoisomers were determined by means of n.m.r. nuclear Overhauser enhancement experiments. This structure determination was undertaken to confirm this assignment.

Experimental

The crystal used in this investigation was cut, from a longer crystalline needle of the presumed *trans*-isomer supplied by Zieger & Gelbaum, to a size of $0{\cdot}2\times0{\cdot}3\times0{\cdot}5$ mm and was sealed in a thin-walled capillary to prevent slow decomposition in air. Precession and Weissenberg photographs of the crystal showed tetragonal symmetry and absence of hkl reflections with $h+k+l$ odd and hhl reflections with $2h+l\neq4n$, indicating that the space group was $I4_1md$ or $I\bar{4}2d$. The unit-cell parameters were determined from the 2θ values for 24 reflections measured on a Datex-automated General Electric diffractometer with Ni-filtered Cu $K\alpha$ radiation [λ(Cu $K\alpha$) = 1·5418 Å]. The resulting values for the unit-cell dimensions are: $a=b=23{\cdot}1811$ (5), $c=5{\cdot}2167$ (7) Å. The calculated density of the crystal, assuming eight molecules per unit cell, is 1·12 g cm^{-3}.

Intensity data were collected by the θ–2θ scan method on the diffractometer with Ni-filtered Cu $K\alpha$ radiation. The scan speed was one degree in 2θ per min and background counts of 30 s were collected at

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