and no holes deeper than -2.51 e.Å^{-3}; these extreme values are found near iodine atoms. The estimated standard deviation in the density amounts to about 0.66 e.Å^{-3} according to the formula of Cruickshank & Rollett (1953). All atomic centers lie on nearly flat regions.

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The Crystal Structure of Rh$_2$Te$_3$

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The compound Rh$_3$Te$_2$ is orthorhombic, space group *Amam*, with four molecules in the cell, $a = 7.694$, $b = 12.446$, $c = 3.697$ Å.
The structure is of a new type. There are two kinds of rhodium and two kinds of tellurium atom in the structure. The number and mean lengths of the bonds are: Rh$_I$–5Te $= 2.69$ Å, Rh$_I$–4Rh $= 2.84$ Å, Rh$_{II}$–5Te $= 2.68$ Å, Rh$_{II}$–5Rh $= 2.94$ Å, Te$_I$–8Rh $= 2.73$ Å, Te$_{II}$–7Rh $= 2.66$ Å.

Introduction

During a recent visit to the Physics Department of the University of California at San Diego the writer examined X-ray diffraction patterns of numerous preparations in the Rh–Te system. It was shown by indexing of the powder pattern that samples obtained by quenching from the melt at the exact composition Rh$_3$Te$_2$ contained a single phase. Small departures from the ideal stoichiometric ratio resulted in two-phase preparations, the second phase being Rh metal on the Rh-rich side and the high-temperature form (NiAs type) of RhTe (Geller, 1954) on the Te-rich side.

Variations in the lattice spacings of Rh$_3$Te$_2$ were not observed. It must be concluded that the homogeneity range is very narrow and that the structure is fully ordered.

Upon annealing for days at 450°C Rh$_3$Te$_2$ is transformed into rhodium metal and a new phase of approximate composition Rh:Te $\simeq 1.1$. However, this new phase is not identical with the low temperature form of RhTe.

The structure

The analysis of the powder patterns of Rh$_3$Te$_2$ gave an orthorhombic base centered translation lattice A with periods.

$$a = 7.694 \pm 0.004, b = 12.446 \pm 0.008, c = 3.697 \pm 0.002 \text{ Å}.$$

The calculated density with four molecules per cell is $\varrho = 10.56$ g.cm^{-3}.

The approximate structure was readily deduced from the powder data with the following results:

Space group: *Amam*
4Me in 4(a): (000) ($\frac{1}{2}$00)
4Me in 4(c_1): $\pm (\frac{1}{4}y_1 0)$ with $y_1 \simeq 0.53$
4Me in 4(c_2): $\pm (\frac{1}{4}y_2 0)$ with $y_2 \simeq 0.745$
8Me in 8(f): $\pm (x_3 y_3 0)$ ($\frac{1}{2} + x_3$, $\bar{y}_3$, 0)
with $x_3 \simeq 0.06$ $y_3 \simeq 0.34$.
However, it was not possible to distinguish between Rh and Te atoms.

Subsequently one single crystal was isolated from one of the quenched samples. The crystal was 2 mm

long ($\|c$) and bounded laterally by (010) and several sets of damaged prism faces (HK0). The needle thickness was 0·169 mm along [100] and 0·060 mm along [010]. As a crude approximation the crystal could be described as an elliptic cylinder with μR ranging from 8·9 to 22·6.

The complete set of intensities HK0 and HK1 were measured with Cu $K\alpha$ radiation and a counter spectrometer. Because of the large μR values and the irregular shape the absorption correction was quite unreliable except at large scattering angles.

Complete order being anticipated the Rh and Te atoms can be distributed over the metal sites in four distinct ways as follows:

Sites	A	B	C	D
4(a)	Rh	Rh	Te	Te
4(c_1)	Rh	Te	Rh	Te
4(c_2)	Rh	Te	Te	Rh
8(f)	Te	Rh	Rh	Rh .

Least-square refinements were made for the four possible structures. Because of the absorption problem experimental $|F|$'s were assigned zero weight for $2\theta < 106°$ and unit weight for $2\theta > 106°$. The Thomas–Fermi–Dirac f curves were corrected for dispersion, and a fixed overall temperature factor, $B = 0·40$ Å², was assumed. The results of the refinements are shown in Table 1. The residual R_1 refers to all reflections, R_2 to those with $2\theta > 106°$.

It is seen that the position parameters are nearly the same for the four possible structures, but that the σ and R values are distinctly smaller for structure C.

The contribution from the HK0 and HK1 reflections to the electron density at the various sites was evaluated with the following results (in relative units):

Fig. 1. Projection of the structure on the (001) plane. Small circles represent rhodium, large circles tellurium atoms. The height above the projection plane is either $z=0$ (open circles) or $z=\frac{1}{2}$ (shaded circles). Bond lengths in Å.

4(a): 4·17,   4(c_1): 3·84,   4(c_2): 4·60,   8(f): 3·55.

Thus, this procedure also decides in favor of structure C.

The structures A, B and D are unreasonable in respect to bond formation between tellurium atoms. In structure D each Te atom would have four Te–Te bonds of length 2·70 Å. There would be three such bonds of length 2·89–3·05 Å in structure A, and an impossibly short Te–Te bond of 2·63 Å would occur in structure B.

Table 2 gives the data for the innermost lines of a diffractometer pattern in which the observed intensities are modified by preferred orientation in the sample. The agreement between observed and calculated structure factors for the reflections with $2\theta > 106°$ is shown in Table 3.

Structure C with the parameter values from Table 1 gives the following bond lengths (to $\pm 0·01$ Å):

Rh_I in 4(c_1), Rh_{II} in 8f), Te_I in 4(a), Te_{II} in 4(c_2)

Rh_I	–4Te_I	2·70 Å	Te_I –4Rh_I	2·70 Å
	–1Te_{II}	2·64	–4Rh_{II}	2·76
	–2Rh_{II}	2·82		
	–2Rh_{II}	2·86		

Table 2. *Diffractometer data*

HKL	sin² θ × 10⁴		Intensity	
	Calc.	Obs.	Calc.	Obs.
020	153		0·1	nil
120	253		1·0	trace
200	401	400	1·4	5
011	472	473	10·1	8
220	554	555	5·8	19
111	572	572	9·8	25
040	612	613	10·8	21
140	712	714	9·8	14
031	778	778	34·8	38
211	873 }	877	19·5 }	31
131	878 }		4·3 }	
240	1013	1015	10·0	22
320	1055	1061	16·9	23
231	1179	1181	70·4	71
311	1374 }	1373	42·6 }	54
060	1377 }		6·4 }	
051	1391		0·1	
160	1477 }	1492	3·9 }	38
151	1491 }		33·0 }	
340	1514		1·3	
400	1604	1604	11·5	5
331	1680		0·5	nil
002	1734	1736	25·7	45
420	1757		1·3	nil
260	1778 }	1778	19·5 }	77
251	1792 }		5·9 }	

Table 1. *Coordinates* (× 10⁴) *and R values*

Structure possibility

	A		B		C		D	
	Value	σ	Value	σ	Value	σ	Value	σ
Y_1	5335	14	5332	9	5337	8	5332	10
Y_2	7458	14	7448	9	7460	6	7457	11
X_3	0606	14	0608	12	0608	9	0620	14
Y_3	3395	8	3400	7	3398	5	3396	8
R_1	0·22		0·19		0·14		0·19	
R_2	0·18		0·13		0·10		0·15	

Rh_{II}–$2Te_I$	2·76 Å	Te_{II}–$1Rh_I$	2·64 Å
–$1Te_{II}$	2·61	–$2Rh_{II}$	2·61
–$2Te_{II}$	2·63	–$4Rh_{II}$	2·63

–$1Rh_I$	2·82
–$1Rh_I$	2·86
–$1Rh_{II}$	2·91
–$2Rh_{II}$	3·05

Table 3. Structure factors

HK0	F_c	$\lvert F_o \rvert$	HK1	F_c	$\lvert F_o \rvert$
800	45	41	4,11,1	72	79
3,12,0	−21	30	811	40	40
820	58	58	0,13,1	0	3
840	137	120	1,13,1	36	41
4,12,0	49	60	691	−4	7
780	51	51	831	−48	46
6,10,0	121	123	771	−15	13
0,14,0	−26	32	2,13,1	−20	23
1,14,0	5	8	5,11,1	46	46
860	−49	51	851	36	33
2,14,0	101	111	3,13,1	−73	80
5,12,0	−33	33	791	−71	67
920	−30	31	4,13,1	48	48
3,14,0	70	77	911	55	52
940	−5	6	6,11,1	8	6
7,10,0	17	17	871	79	71
880	74	74	931	−15	14
4,14,0	−26	30	0,15,1	120	97
6,12,0	−8	11	1,15,1	−44	41
960	−27	30			
0,16,0	−15	15			

The shortest Te–Te distance in the structure is 3·70 Å.

In the high temperature (CdI_2) and low temperature (pyrite) forms of $RhTe_2$ Geller (1954) reported Rh–6Te = 2·65 and 2·64 Å. Geller gives Rh–6Te = 2·70 Å and Rh–2Rh = 2·83 Å for the high-temperature form (NiAs) of RhTe.

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The Crystal Structure of Benzotrifuroxan (Hexanitrosobenzene)*

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The crystal structure of benzotrifuroxan (hexanitrosobenzene) has been refined by full-matrix least-squares computations on all positional and thermal parameters to a final weighted R index of 0·040 for 606 reflections of observable intensity. The unit cell is orthorhombic ($a = 6·923$, $b = 19·516$, $c = 6·518$ Å), space group $Pna2_1$, with four molecules per unit cell.

The molecule is essentially planar, and the six nitroso substituents have formed three furoxan rings.

Introduction

There has been considerable interest in the configuration of benzofuroxans. Kaufman & Pickard (1959) cover the classical aspects of this interest in their review article. More recently Harris, Katritzky, Øksne, Bailey & Patterson (1963) have proven the benzofuroxan configuration of this type of compound in solution. Previous X-ray work on this problem consists in two preliminary and somewhat contradictory studies by Hulme (1962) and Gol'der, Todres-Selektor & Bognadov (1961), and work by Britton & Noland (1962) on the structure of 5-chlorobenzofurazan-1-oxide. Britton & Noland found that this compound has the furoxan structure, but their work leaves unsettled some of the important details of the structure.

In this paper we report the results of a study whose purpose was to elucidate the structure of benzotrifuroxan in the solid state. In particular, we were concerned with the relative importance of the nitroso and furoxan configurations in this symmetric compound, and with the pertinent bond angles and interatomic distances.

Benzotrifuroxan is, incidentally, a powerful and relatively sensitive explosive.

Experimental

A sample of benzotrifuroxan was prepared in this Laboratory following the method of Turek (1931). A single crystal suitable for collection of the X-ray intensity data was grown on its mounting fiber by recrystallization from acetone. The longest internal dimension of this crystal was 0·340 mm. The crystal was aligned and preliminary unit-cell dimensions and space group ex-

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