The Structure of 3,4,5-Trichlorotetracyclo[4.4.0.0^{3.9}.0^{4.8}]decan-2-one, a Novel Cage Molecule

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The crystal structure of the novel cage molecule, 3,4,5-trichlorotetracyclo[4.4.0.0^{3·9}.0^{4·8}]decan-2-one, $C_{10}H_9Cl_3O$, has been determined from automatic diffractometer data. The structure was refined by anisotropic least squares to a final R value of 7.3 % for 1168 observed reflections. The space group is $P2_1/c$ with Z=4 and cell dimensions a=7.562, b=13.183, c=10.233 Å, $\beta=94.40^{\circ}$. All hydrogen atoms were found and their positions refined. The almost spherical molecules are arranged in hexagonal close-packing, with the pseudo-hexagonal layers parallel to (001). The thermal parameters were analyzed for rigid body motion and gave nearly isotropic translational and rotational tensors, corresponding to r.m.s. amplitudes of 0.2 Å and 3.4°, respectively.

Introduction

A novel cage system with the chemical composition $C_{10}H_9Cl_3O$ was recently reported by Stedman, Miller & Hoover (1966), who concluded from the method of synthesis and spectral data that the new compound was most likely the 3,4,5-trichlorotetracyclo[4·4·0·0^{3.9·04.8}]-decan-2-one shown in Fig. 1. The conformation of the C-Cl bond on C(5) was not known. The crystal structure was determined in order to verify the configuration of the cage, to clarify the stereochemistry at C(5), and to obtain detailed information about the geometry of the molecule.

Crystal data

The crystals were grown by Mrs L.S. Miller of the Smith Kline and French Laboratories in a form suitable for the structure determination. The space group was determined by means of precession photographs with Mo $K\alpha$ radiation. The lattice constants were measured on a Picker automated single-crystal diffractometer, with Cu $K\alpha$ radiation, and refined by least squares. The density was measured by the flotation method.

3,4,5-Trichlorotetracyclo[$4 \cdot 4 \cdot 0 \cdot 0^{3.9} \cdot 0^{4.8}$]decan-2-one, C₁₀H₉Cl₃O, M.W. 251.54.

Monoclinic, space group $P2_1/c$, from systematic absences: h0l absent for l=2n+1, 0k0 absent for k=2n+1.

Z=4 $a=7.562 (\sigma=0.002), b=13.183 (\sigma=0.004), c=10.233$ $(\sigma=0.003) \text{ Å}.$ $\beta=94.40 (\sigma=0.03)^{\circ}.$ $D_m=1.683 (\sigma=0.007) \text{ g.cm}^{-3}$ $D_x=1.643 \text{ g.cm}^{-3}$ $\mu_{Cu \ K\alpha}=77.89 \text{ cm}^{-1}.$





Fig.1. View of the molecule, and identification of the atoms.

Since the space group is centrosymmetric and the molecule non-centrosymmetric, the crystal structure must be racemic.

Experimental

The intensity data were measured on a Picker fourcircle automated single-crystal diffractometer with Cu $K\alpha$ radiation. The crystal showed the morphological forms {110} and {011}. Its dimensions, which were measured on an optical goniometer with a ruled grating, were between 0.11 and 0.22 mm. The crystal faces were analytically described in a Cartesian coordinate system with $\mathbf{x}||\mathbf{a}^*, \mathbf{y}||\mathbf{b}, \mathbf{z}||\mathbf{c}$, by the following equations:

$$\{110\}: S_h \ 0.867x + S_k \ 0.498y = 0.098 \ mm$$

{011}:
$$S_l 0.057x + S_k 0.613y + S_l 0.788z = 0.056 \text{ mm.}$$

 $S_h = \text{sign}(h) \quad S_k = \text{sign}(k) \quad S_l = \text{sign}(l).$

The coefficients of x, y, z (*i.e.* the direction cosines of the face normal) were calculated with the use of the measured lattice constants.

The intensities were measured up to $\sin \theta / \lambda = 0.588$ Å⁻¹, ($\theta = 65^{\circ}$), using the diffractometer in the $\theta - 2\theta$ scanning mode. The scanning range was 1° in θ , with a speed of 1° per minute. The background was measured by a stationary count for 20 seconds on either side of the peak. Most of the high-order reflections were weak, since the intensities decreased rapidly with θ . These weak intensities were remeasured with a scanning speed of $\frac{1}{3}^{\circ}$ per minute and the background was measured for 40 seconds. Nevertheless, an appreciable proportion of the intensities were unobserved. All the accessible symmetry related reflections hkl and $h\bar{k}l$ were measured and the mean value was used to calculate the structure amplitudes. The intensities were corrected for absorption using a general absorption program for an IBM 1620 computer (Craven, 1963). There was no indication that extinction corrections were required. Of the 1691 independent reflections accessible on the diffractometer with Cu Ka radiation, 1168 were observed above the background.

Determination of the structure

The structure amplitudes (uncorrected for absorption) were converted to normalized $|E_{hkl}|$ values and used in the IBM 1620 sign correlation procedure developed by Beurskens (1963). This led to two different sets of signs for 360 structure factors, one of which was strongly favored by the probability criterion of Cochran & Woolfson (1955). The three-dimensional E map calculated with this set showed one very high peak, which was also an obvious solution of the Patterson E^2-1 synthesis, but no peaks in the Patterson map corresponding to other weaker peaks in the E map could be identified. The E map calculated with the less probable set of signs, however, showed three strong

peaks that were identified as the positions of the chlorine atoms and explained nearly all features of the Patterson synthesis.

A Fourier synthesis (Shiono, 1965) with signs determined by these chlorine positions revealed all the carbons and the oxygen, and a subsequent structure factor calculation gave an R value of 0.35 for all observed reflections. The refinement was then carried out by least squares (Busing, Martin & Levy, 1962), using weights $w = 1/\sigma^2$ with $\sigma(|F|) = \frac{1}{2}|F_{\min}| + 0.05|F|, |F_{\min}| = \frac{1}{2}|F_{\min}| + 0.05|F|$ 50 (Table 4). After two cycles of isotropic and two cycles of anisotropic refinement, the R value was 0.09. A difference Fourier synthesis showed several strong peaks, but did not reveal the hydrogen atoms unambigously. The structure factors were therefore corrected for absorption as described above. The square roots of the calculated transmission factors lay between 1.93 and 1.40. An anisotropic least-squares refinement with these corrected data gave no change in the R value and variations in the atomic positions of the order of σ . However, the difference map now showed nine strongest peaks which could be identified as the hydrogen atoms.

Two more least-squares cycles, refining the positional and anisotropic thermal parameters of chlorine, carbon, and oxygen, and the positional parameters of hydrogen reduced the *R* value to 0.073. The thermal parameters of the hydrogens were assumed to be the same as those of the carbon atoms to which they were attached and they were not varied. The positional parameters of the hydrogens did not change more than 0.5σ in the second cycle. The atomic positional and thermal parameters are given in Tables 1, 2 and 3, the observed and calculated structure factors in Table 4.

Table 1. Fractional atomic coordinates

The estimated standard deviations given in parentheses refer to the last decimal position.

	x	У	Z
C(1)	0.4782 (7)	0.1209 (4)	0.3231 (6)
C(2)	0.4485 (7)	0.0064 (4)	0.3136 (5)
C(3)	0.3043 (7)	-0.0032(3)	0.2015 (5)
C(4)	0.1323 (6)	0.0497 (4)	0.2397 (5)
C(5)	0.1670 (7)	0.0895 (4)	0.3819 (5)
C(6)	0.3010 (7)	0.1723 (4)	0.3601 (5)
C(7)	0.2127 (8)	0.2293 (4)	0.2423 (6)
C(8)	0.1687 (8)	0.1408 (4)	0.1492 (5)
C(9)	0.3408 (7)	0.0885 (4)	0.1091 (6)
C(10)	0.5078 (8)	0.1393 (5)	0.1771 (6)
0	0.5260 (5)	-0.0608(3)	0.3715 (4)
Cl(3)	0.2887 (2)	-0·1260 (1)	0.1356 (2)
Cl(4)	-0.0664 (2)	-0·0172 (1)	0.2068(2)
Cl(5)	-0.0291 (2)	0.1406 (1)	0.4453 (2)
H(1)	0.583 (8)	0.132 (5)	0.390 (6)
H(5)	0.214 (8)	0.038 (5)	0.454 (6)
H(6)	0.326 (8)	0.212 (5)	0.440 (6)
H(7–1)	0.112 (9)	0.272 (5)	0.273 (6)
H(7–2)	0.310 (9)	0.277 (5)	0.210 (6)
H(8)	0.081 (9)	0.156 (5)	0.077 (7)
H(9)	0.355 (8)	0.069 (5)	0.011 (7)
H(10-1)	0.630 (9)	0.108 (5)	0.151 (7)
H(10-2)	0.539 (9)	0.215 (6)	0.154 (7)

Table 2. Anisotropic thermal parameters The temperature factor expression used was $B = \sum_{i,j} \beta_{ij} h_i h_j$.

The estimated standard deviations given in parentheses refer to the last decimal position.

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C(1)	0.0145 (10)	0.0055 (4)	0.0107 (6)	-0.0004(5)	-0.0013 (6)	0.0003 (4
C(2)	0.0173 (10)	0.0056 (4)	0.0087 (6)	0.0020 (5)	-0.0016 (6)	-0.0001 (4
C(3)	0.0188 (10)	0.0033(3)	0.0094 (6)	0.0010 (4)	-0.0013 (6)	-0.0008 (3)
C(4)	0.0132 (9)	0.0040 (3)	0.0105 (6)	-0.0006(4)	-0·0008 (6)	0.0012 (3
C(5)	0.0157 (10)	0.0047 (3)	0.0084 (5)	0.0016 (4)	0.0003 (6)	-0.0004 (3)
C(6)	0.0147 (9)	0.0049 (3)	0.0104 (6)	-0.0007(5)	-0.0005(6)	-0.0010 (4
C(7)	0.0201(12)	0.0042(3)	0.0119 (7)	0.0008 (5)	0.0006 (7)	0.0000 (4)
C(8)	0.0204(12)	0.0050 (3)	0.0095 (6)	0.0005 (5)	-0.0007(7)	0.0003 (4
C(9)	0.0203(12)	0.0054 (4)	0.0093 (6)	0.0011(5)	0.0018(7)	0.0007 (4
C(10)	0.0181 (11)	0.0068 (4)	0.0109 (7)	0.0002 (6)	0.0029 (7)	0·0006 (4
0`´	0.0228 (9)	0.0065 (3)	0.0117(5)	0.0046 (4)	-0.0027(5)	0.0007 (3
Cl(3)	0.0314 (4)	0·0043 (Ì)	0.0125 (2)	0·0015 (1)	-0.0007(2)	-0·0018 (1
Cl(4)	0.0162 (3)	0.0057 (1)	0.0160 (2)	-0.0026(1)	-0.0034(2)	0·0000 (1
Cl(5)	0.0171 (3)	0.0058 (1)	0.0151 (2)	0·0010 (1)	0.0042 (2)	-0.0005 (1)

Table 3. R.m.s. displacements U along principal axes of thermal ellipsoids

	U_1	U_2	U_3
C(1)	0·1952 Å	0·2190 Å	0∙2489 Å
C(2)	0.1895	0.2167	0.2532
C(3)	0.1645	0.2081	0.2522
C(4)	0.1778	0.1907	0.2482
C(5)	0.1865	0.2080	0.2303
C(6)	0.1887	0.2145	0.2430
C(7)	0.1904	0.2400	0.2524
C(8)	0.2028	0.2196	0.2516
C(9)	0.2081	0.2219	0.2487
C(10)	0.2156	0.2391	0.2544
0	0.1807	0.2548	0.2987
Cl(3)	0.1812	0.2580	0.3105
Cl(4)	0.1810	0.2400	0.3064
Cl(5)	0.2022	0.2344	0.2876

Description of the structure

The configuration of the molecule (Fig. 1) is as proposed by Stedman *et al.* (1966). It has a ten-carbon cage structure containing one four-membered, two five-membered and two six-membered saturated rings. The C(5)-Cl(5) bond points away from the carbonyl group, in the conformation of least steric repulsion with respect to the substituents of the C(1)-C(2)-C(3)-C(4)-C(5)-C(6) ring.

The cyclobutane ring C(3)-C(4)-C(8)-C(9) is a planar square within the limits of accuracy. The deviations of the angles from 90° are equal to σ , and the distances of the atoms from the least-squares plane are less than 0.003 Å. The cyclopentane rings C(1)-C(2)-C(3)-C(9)-C(10) and C(4)-C(5)-C(6)-C(7)-C(8) are puckered with internal angles varying between 97 and 106°. The angles tend to be greater in the latter ring than in the former, possibly owing to the repulsion of Cl(5) and H(7-1). The two cyclohexane rings are in boat form with internal angles close to tetrahedral (108 to 111°), except for those at the atoms C(2) and C(5), C(7) and C(10), which are close to 100° . The angles of the C(1)-C(6)-C(7)-C(8)-C(9)-C(10) ring are all greater than those of the C(1)-C(2)-C(3)-C(4)-C(5)-C(6) ring, owing to the repulsion of H(7-2) and

H(10-2). The carbonyl group is planar within the limits of accuracy, and the sum of bond angles around C(2) is 359.9° .

The C-C bond lengths vary between 1.517 and 1.574 Å, *i.e.* over a range of about 7σ , with a mean value of 1.545 Å. There are some systematic differences which lie in the probably significant range from 2 to 4σ , and might arise from the angular distortions necessary to form the cage. The bonds in the cyclobutane ring are all longer than the mean value and one of them, C(3)–C(9), is longer by about 4σ . The longest bond observed, C(1)-C(6), at 1.574 Å, is that which is opposite to the cyclobutane ring. Together with two sides of that ring, it links the two cyclopentane rings to form the cage. The bonds C(3)-C(4) and C(4)-C(5)are both longer than the mean at 1.552 and 1.550 Å and both have adjacent C-Cl bonds at both ends. The other seven C-C bonds are either equal to or less than the mean value. No systematic correlation could be found between these variations in distances and the angular distortions from 109°28'. The C-O bond has a normal value. Of the three C-Cl bonds, C(5)-Cl(5)is 0.04 Å longer than the other two, for which the only distinction is that it involves one of the carbon atoms with a hydrogen atom attached.

The molecules are hexagonal close-packed as shown in Fig.2, with the layers parallel to the plane (001). The ideal hexagonal close packing of spheres (space group $P6_3/mmc$) can be described in $P2_1/c$, a subgroup of $P6_3/mmc$, by the coordinates $\frac{1}{4}$, $\frac{1}{12}$, $\frac{1}{4}$. The axial ratio of the monoclinic cell with $\beta = 90^{\circ}$ is then a:b:c =0.5774:1:0.9428. The coordinates of the center of gravity of the molecule in this structure are 0.22, 0.05, 0.26and the axial ratio is 0.5734:1:0.7762. The molecules therefore pack as oblate ellipsoids rather than spheres.

Analysis of thermal motion

The structure appears to be well suited to an analysis of the thermal motion assuming rigid body motion of the molecules about their center of gravity (Cruickshank, 1956). The tensor **T** for the translational motion

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Table 4. Observed and calculated structure factors

Columns are: Index H, $|F_{obs}|$, F_{cal} . *=unobserved

,K=	0 L≠ 0	4 51	28-	-4 1	197 185-	× ۲	= 12 l	.= 1	-7	424	8 I	_K=	10	141-	3	420	420-	-4	52	83-
2	417 421	6 108	3 117~		42 • 26	lĭ	234	247	-8	68	89-	ĭ	42 •	19-	4	40+	220 9-		123	130
3	162 183	K= 12	2 L= 0	6	52 53	-1	178	178	K	2 4	L= 2	-1	180	178-	-4	339	350	~5	131	124
4 4	448 464	0 120	0 111-	-6	73 49-	2	133	108	0	827	803-	2	42•	1	5	83	81-	6	51	62
5	346 353-	1 40	• 9-	1 7	74 50-	-2	40+	30	1	154	159-	-2	286	287-	-5	329	342	-6	37+	6-
6	163 172	2 40	3	1-7	37• 48-	1_3	40	34	-1	1187	1182-	-1	42*	28-	-6	150	156-	K	= 10 I	.= 3
é	111 130-		3 103-	-8	440 10	- 4	40#	20	-2	336	113-	- 5	124	129-	7	73	66	ĭ	237	233-
κ _≖ ,	1 L= 0	5 4	5. 5	К≖	6 L= 1	-4	40+	26-	3	200	197-	-4	43+	12-	-7	167	153	-1	123	109-
1	367 389-	K= 1	3 L= 0	0	176 190	5	40.	14	-3	403	408-	5	90	95	8	75	65	2	52	107-
2	190 198-	1 172	2 172-	1	10+ 41-	-5	40•	5-	4	296	286	-5	91	101-	-8	107	94	-2	408	418-
3	10+ 15	2 38	3• 19-	-1	194 192	K	= 13 1	.= 1	-4	376	385-	6	51	45	K	- 4 L	= 3	3	89	81
4	323 333	3 40)• 42-	2	737 733	0	90	84-	2	136	144	-6	42*	4 -	0	124	124	-3	129	132-
2	83 60	K-10	5 214-	1 2	497 487	1	49.	33-	- 5	184	167		237	237	-1	22	103-		41	15
7	41+ 10	0 160	- 183	-3	307 295-	1 2	39+	14	-6	41.	3-	ĭ	51	52-	2	164	179	5	51	35-
8	108 105	1 58	8 51	4	53 35	-2	49+	31-	7	113	97	-1	81	81-	-2	191	189	-5	42+	8
K≖	2 L≭ 0	2 7	5 72	-4	132 112	3	69	27-	-7	42	· 31-	2	72	68	3	37+	24-	6	104	110
0 1	067 1003	3 44	4 - 14	5	85 81	- 3	85	84-	8	171	158	-2	36+	45-	-3	52	33-	-6	42+	22-
1	76 74	K= 19	5 L= 0	-5	53 30-	4	35*	11	-8	38	12	3	.97	95	4	310	293	ĸ	* 11 1	.= 3
2	409 500 50 53-	1 100	5 94-	-6	140 142 87 88-		- 14 1	- 1	`	= 7 535	511-	-,	104	124	-4	414	105-	, i	50	41-
4	155 148-	0 10	7 86-	7	53 60-	1 0	34+	26-	ĭ	358	341	-4	41+	2	-5	104	102-	-i	96	111
5.	234 247-	1 834	4 877-	-7	218 205-	li	59	51	-i	10.	15	5	51	24-	6	104	94	2	181	182-
6	100 103-	-1 65	3 672	8	45+ 9-	1-1	۱50	187	2	143	146	-5	176	168-	-6	37+	2-	-2	103	92
7	82 72-	2 142	2 140-	-8	78 83-	2	212	218	-2	141	130-	-6	52	38-	7	98	89	3	102	116-
8	106 101-	-2 99	2 1036-	K=	7 L= 1	-2	93	101	3	269	276-	K=	12	L= 2	-7	96	71	-3	121	132
, K =	5 1 4	3 6/1	8 /1/	12	63 47	1.3	594	50-	-3	348	327-	, o	40*	153	- 8	68	22	-4	121	114
2	210 218-	4 4	9. 46	1-1	132 134-	1 - x	= 15 1	= 1	-4	200	202-	-1	222	225-	- к	5,1	= 3	5	41+	10
3	404 413-	-4 41	1 424-	12	111 93	0	39+	- 11 I	5	80	77	2	40+	14-	0	46*	6	-5	41+	46
4	244 250	5 13	3 144	-2	705 673-	1	68	88-	-5	112	118	-2	40+	40-	1	39+	37	ĸ	= 12 4	.= 3
5	143 144	-5 47	2 482-	3	44• 13	-1	34 •	0	6	112	89-	3	101	103-	-1	446	447-	0	81	70-
6	72 66	6 17	6 207	1-3	509 495-	K	= 01	= 2	-6	421	32	-3	35+	31-	2	441	425-	1	40+	27
7	51 39-	-6 36	6 371-	4	54 50	1 ?	1043	1087-		140	143-	-	35+	39	-2	440	17	-1	49#	14 p1
° + -	41 - 05	-7	c 02-	1	107 10/~		738	767	-/	501			61	51	-1	427	414	-2	40+	22
0	714 687-	8 7	3 68-	1-5	205 200-	2	370	384	-8	66	52	-5	35+	36-	4	79	86-	3	160	166
ĩ	202 192	-8 4	2+ 11	6	171 158	-2	183	183-	[°] к	= 6	L= 2	K≃	13	L= 2	-4	173	182	-3	40*	6
2	53 53	K= ;	2 L= 1	-6	247 236-	3	658	676	0	219	212-	0	39+	1	5	110	110-	4	128	136
3	184 192-	0 66	2 616-	17	76 49-	-3	172	168	1	1.19	121	1	49+	7	-5	170	172	-4	60	56-
4	315 331-	1 23	3 232	-7	39 4	1 4	127	122-	-1	73	207-	-1	494		6	13	49-	->_	- 12 1	- 37-
2	316 315	2 60	0 620-	1	0 L= 1		306	322	-5	190	181	-2	79	69	- 7	137	132	່ດົ	49.	
7	137 139-	-2 1	0 21	lĭ	89 81	-5	324	323	3	62	80	3	99	110-	-7	107	85	Ĩ	35+	26
8	111 110-	3 5	0* 48-	-i	151 145	6	134	143	-3	118	123-	-3	148	152-	-8	38•	19	-1	110	83-
К=	5 L≖ 0	-3 69	9 708	2	242 225-	-6	118	129	4	178	182	4	40•	8	K=	= 6 L	= 3	2	70	68-
1	576 583-	4 7	1 64-	-2	186 180	17	82	68	-4	38+	24-	-4	80	84-	0	125	121	-2	49+	. 4
2	164 171-	-4 20	2 199	3	67 52-	-7	159	159	2	239	248	K*	14	L= 2		414	401	-3	47*	74
4	271 247-	5 12	0 19/-	-3	1/9 104 54 49-	1-6	157	160	~ 7	53	37	l i	39.	18	1 2	216	219		40.	45-
5	301 292-	6 46	2 460+	-4	55 30	Ч°к	= í i i	= 2	-6	137	135	-i	124	154	-2	196	179	K	= 14 1	L= 3
6	114 99-	-6 10	2 94	5	54 46-	0	559	585	1	52	51	2	68	62	3	113	112-	0	172	166-
7	119 118-	7 4	6• 56-	-5	89 76	1	546	545	-7	76	66	-2	84	93	-3	129	110-	1	56	70
8	70 66-	-7 9	7 90	6	193 184-	-1	42*	5	-8	168	164	3	77	78	1 4	256	242	-1	104	109-
_K≖	6 1 0	88	1 96-	-6	440 /	1 - 2	100	66	× ا	≖ ,,,	L= 2		48*	28		321	239-	1 - 5	48.	30
ĩ	360 361	- ° K = 1	3 1 = 1	1-7	44+ 27-	1 3	510	520	l ĭ	178	183	0^-	34+	15-	-5	42+	6	-3	58	51-
2	289 292	0 24	7 235	K≖	9 L= 1	-3	322	328	-i	79	46	1 i	11	104	6	128	116	ĸ	= 0 I	= 4
3	311 313	1 109	3 1091	0	221 219	4	210	220	2	189	188	-1	59	88	-6	47•	57	0	553	570-
4	135 135	-1 15	0 164	11	140 151-	-4	171	163	-2	401	28-	K=	1	L= 3	1	95	84		645	650
5	99 98-	2 63	7 659	1-1	53 92	5	61	52	3	251	254	0	621	630-	- ' .		- 29-	-1	626	103
2	40.0 11	1 1 19	2 10C A 399	-2	317 306-	1 6	108	92	4	53	62	_i	363	376-	0	582	568-	-2	453	475
8	102 95	- 3 38	5 385	3	282 278	-6	114	121-	-4	182	177-	2	122	116	1	304	295-	3	555	555-
К=	7 L= 0	4 33	0 349	- 3	43* 33-	1	171	174	5	85	45	-2	216	214	-1	111	112-	-3	563	577
1	282 277	-4 12	9 149-	4	148 159	- 7	41+	15	-5	43	5	3	383	389-	2	107	109-	1	356	358-
2	454 23	23	2 236	-4	38* 45- 142 13-	8	72	70-		429	21-	1-2	211	212-	1-2	74	60-	5	205	207-
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2	201 193	- -4 37	7 376	5	145 155-	-7	41•	3	-6	55	47	3	182	178-	3	74	57-	4	490	16
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4	130 128	-5 8	y 99-	6	144 169-	-8	64	47	-/,	-112	102	-4	138	128-	-4	130	132-		202	212
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2 0	22	030-	-3 454 52	-3 803 304	1 222 220	1 0 138 140		-6 71 6	5 2 69 58-
-2 1	23	110-	4 60 67	4 114 97	1 223 224		-3 265 44		9 -2 409 17
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Table 4 (cont.)

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$ \begin{array}{c} 1 \\ -1 \\ 2 \\ -3 \\ -3 \\ -4 \\ -5 \\ -1 \\ 2 \\ -2 \\ -3 \\ -4 \\ -5 \\ -5 \\ -4 \\ -5 \\ -5 \\ -5 \\ -4 \\ -5 \\ -5 \\ -5 \\ -5 \\ -5 \\ -5 \\ -5 \\ -5$	80 69 39 40 68 49 57 50 57 57 57 57 57 57 57 57 57 57	82- 67- 28- 46- 46- 46- 45 7 30 101 20- 59- 64 114- 25- 42- 8- 18	1 -1 -2 -3 -3 -4 -5 K= 0 1 -2 -2 -3 -4 K= 0	78 69 35* 111 35* 69 36* 76 36* 78 85 60 85 60 85 60 87 120 78 85 60 87 12 78 85 40* 60 84 85 40* 60 84 85 69	69- 56 14- 136 75- 82- 82 21 92 88 24 123 76 32 32 32 32 32 32 32 32 32 32 32 32 32	$ \begin{array}{c} 2 \\ -2 \\ 3 \\ -3 \\ -3 \\ -1 \\ 2 \\ -2 \\ -3 \\ -1 \\ -1 \\ -1 \\ -1 \\ 2 \\ -1 \\ -1 \\ 2 \\ -1 \\ -1 \\ 2 \\ -1 \\ -1 \\ 2 \\ -1 \\ -1 \\ 2 \\ -1 \\ -1 \\ 2 \\ -1 \\ -1 \\ -1 \\ 2 \\ -1 \\ -1 \\ -1 \\ -1 \\ -1 \\ -1 \\ -1 \\ -1$	60 34* 40* 9 L 44* 44* 39* 49* 35* 10 L 34* 61 10* 10* 136	53- 21 36- 34- 2- 74 21- 12- 13- 13- 567- 10 65- 110- 110- 1	3 -3 4 -4 -5 K= 0 1 -1 2 -3 -3 4 -4 -5 K= 0 1 -1	34+ 59 38+ 62 34+ 1 L= 38+ 28+ 38+ 38+ 38+ 38+ 38+ 38+ 38+ 20+ 48+ 2138 48+ 28+	26 41- 87- 26 10 29 12- 67- 74- 8 36 50- 71 5 10 143 18 31	-2 3 -3 -4 -4 -5 0 1 -1 2 -3 -3 -4 -5 0 1 -1 -5	10• 33= 68 38• 28• 34• 48• 108 48• 91 47• 48• 59 39• 59 39• 59 39• 59 39• 48• 59 39• 48• 59 39• 48•	45- 359 46 15- 25- 94- 34 56 94- 170 33	$ \begin{array}{c} -2 \\ 3 \\ -3 \\ -4 \\ 0 \\ 1 \\ -1 \\ 2 \\ -3 \\ -4 \\ K = \\ 0 \\ 1 \\ -1 \\ 2 \\ -3 \\ -4 \\ -1 \\ -1 \\ -3 \\ -4 \\ -4 \\ -3 \\ -4 \\ -4 \\ -3 \\ -4 \\ -4 \\ -4 \\ -4 \\ -4 \\ -4 \\ -4 \\ -4$	63 48* 115 28* 55L= 83 83 48* 38* 48* 38* 48* 39* 48* 39* 48* 39* 48* 39* 68	74 51 109 7 10 65- 85 36 27 30 10- 26 48- 10 1- 12 30- 24 33 37-	0 1 -1 2 -3 K= 0 1 -1 -2 K= 0 1 -1 -2 -3 K= 0 1 -1 -2 -3 K= 0 1 -1 -2 -3 K= 0 1 -1 -3 K= 0 1 -1 -3 K= 0 1 -1 -3 K= 0 1 -1 -3 K= 0 1 -1 -3 K= 0 1 -1 -3 K= 0 1 -1 -3 K= 0 -1 -1 -3 K= 0 -1 -1 -3 K= 0 -1 -1 -3 K= 0 -1 -1 -3 K= -3 C 	47* 38* 38* 59* 8 L= 47* 48* 10* 47* 48* 61 47* 48* 61 45* 28* 28* 28* 28* 48*	33- 38 39- 0 57 31 10- 25 26- 11 28- 39 91 8- 7- 11 50 32-	2 -2 -3 K= 0 1 -1 2 -3 K= 0 1 -1 -2 -3 K= 0 1 -1 -1 -2 -3 L -1 -1 -1 -2 -3 L -1 -1 -1 -2 -3 -3 -3 -3 -3 -3 -3 -3 -3 -3 -3 -3 -3	57 28* 20* 38* 28* 34* 88 20* 4 L* 39* 38* 39* 28* 28* 28* 28* 28* 38* 38* 38* 38* 38*	97 13 11 17 20 20 10 20 11 13 5 21 311 7 26 54
K≓ 0	6 L:	- 9	1	49 .	49-	-2	77	60-	2	67	50	2	38+	3-	Г-, к=	48• 7 L=	10	-1	48+ 20+	32- 85	-1	48• 58	54- 82-

of the mass center and the tensor ω for the rotational motion were determined by least squares from the observed anisotropic temperature factors given in Table 2,

thus assuming that the molecules can be regarded as independent rigid bodies (Coulter, Ganztel & Trueblood, 1962). T and ω , obtained by assuming the same



Fig.2. Hexagonal close-packing of the molecules. The structure is viewed along c^* . The molecules at the corners and in the center of the hexagon lie in the same plane, the ones at the corners of the triangle in a different plane below. The sequence of these two planes is repeated by translation along c.

weights for all β_{ij} , are given in Table 7, relative to a Cartesian coordinate system with $\mathbf{x}||\mathbf{a}, \mathbf{y}||\mathbf{b}, \mathbf{z}||\mathbf{c}^*$, together with their e.s.d.'s determined by the leastsquares procedure. The atomic vibration parameters U_{ij} (Å²) calculated from T and ω are given relative to the same coordinate system in Table 8, together with the observed values. The mean square atomic vibration amplitude u^2 in a direction given by the direction cosines l_i is $u^2 = \Sigma U_{ij} l_i l_j$. The r.m.s. difference between $(U_{ij})_{obs}$ and $(U_{ij})_{calc}$ is 0.0061 Å², the standard devia-

C(6) C(5) Cl(5)

111.2 (4)

tion of the $(U_{ij})_{obs}$ estimated from the fit with the $(U_{ij})_{calc}$ therefore 0.0066 Å². The average e.s.d. of the $(U_{ij})_{obs}$ derived from the e.s.d.'s of the β_{ij} is 0.003 Å² for C and O, 0.001 Å² for Cl. Inspection of Table 8 therefore shows that there is a very significant difference between the $(U_{ij})_{obs}$ and $(U_{ij})_{calc}$ which is greatest for the Cl atoms.

The tensor components obtained by weighting the β_{ij} according to their e.s.d.'s (Stewart, 1966) differ from the ones in Table 7 by amounts up to 3σ . The differ-

ne estim	lated standa	ard deviations given i	n parentneses r	eler to the la	si decimai posi
i	j	D_{ij}	i	j	D_{ij}
C(1)	C(2)	1·528 (7) Å	C(1)	H(1)	1·02 (6) Å
C(1)	C(6)	1.574 (8)	C(5)	H(5)	1.05 (6)
C(1)	C(10)	1.547 (9)	C(6)	H(6)	0.98 (6)
C(2)	C(3)	1.526 (7)	C(7)	H(7-1)	1.02 (7)
C(3)	C(4)	1.552 (7)	C(7)	H(7-2)	1.04 (7)
C(3)	C(9)	1.572 (7)	C(8)	H(8)	0.98 (7)
C(4)	C(5)	1.550 (7)	C(9)	H(9)	1.05 (7)
C(4)	C(8)	1.554 (7)	C(10)	H(10-1)	1.07 (7)
C(5)	C(6)	1.517 (8)	C(10)	H(10-2)	1.06 (8)
C(6)	C(7)	1.530 (8)			
C(7)	C(8)	1.527 (8)			
C(8)	C(9)	1.555 (8)			
C(9)	C(10)	1.547 (8)			
C(2)	0	1.194 (6)			
C(3)	Cl(3)	1.754 (4)			
C(4)	Cl(4)	1.753 (5)			
C(5)	Cl(5)	1.795 (6)			

Table 5. Bond lengths

ated standard deviations given in parentheses refer to the last decimal position. Tł

Table 6. Bond angles

The o	estimate	ed standard	deviations given in	parentheses refer	to the	last decin	mal position.
i	j	k	Angle (ijk)	i	j	k	Angle (ijk)
C(2)	C(1)	C(6)	108·5 (4)°	C(2)	C(1)	H(1)	107 (4)°
C(2)	$\hat{C}(1)$	C(10)	97·2 (4)	C(6)	C(1)	H(1)	114 (4)
C(6)	C(1)	C(10)	110.7 (5)	C(10)	C(1)	H(1)	118 (4)
C(1)	C(2)	C(3)	102.9 (4)	C(4)	C(5)	H(5)	118 (4)
C(2)	C(3)	C(4)	109.8 (4)	C(6)	C(5)	H(5)	112 (4)
C(2)	C(3)	C(9)	103.9 (4)	Cl(5)	C(5)	H(5)	104 (4)
C(4)	C(3)	C(9)	89.7 (4)	C(1)	C(6)	H(6)	109 (4)
C(3)	C(4)	C(5)	107.7 (4)	C(5)	C(6)	H(6)	111 (4)
C(3)	C(4)	C(8)	90.4 (4)	C(7)	C(6)	H(6)	111 (4)
C(5)	C(4)	C(8)	105.8 (4)	C(6)	C(7)	H(7-1)	109 (4)
C(4)	C(5)	C(6)	99.9 (4)	C(8)	C(7)	H(7-1)	119 (4)
C(1)	C(6)	C(5)	108.5(5)	C(6)	C(7)	H(7-2)	106 (4)
C(1)	C(6)	C(7)	110.5 (4)	C(8)	C(7)	H(7-2)	113 (4)
C(S)	C(6)	C(7)	102.3(4)	H(/-1)	C(7)	H(7-2)	109 (5)
C(6)	C(7)	C(8)	100.2 (4)	C(4)	C(8)	$H(\delta)$	118 (4)
C(4)	C(8)	C(7)	105.0 (4)	C(7)			115 (4)
C(4)	C(8)	C(9)	90.3 (4)	C(9)	$C(\delta)$		115 (4)
C(n)	C(0)	C(9)	110.9(3)	C(3)	C(9)		113 (4)
C(3)	C(9)	C(0)	09.0 (4) 102.7 (4)	C(0)	C(9)		121(4) 112(4)
C(3)	C(9)	C(10)	$103^{\circ}7(4)$ 111.1(5)	C(10)	C(1)	$H(10_1)$	113(4)
C(0)	C(3)	C(10)	101.1(5)	C(1)	C(10)	H(10-1)	112(4)
	C(10)	C(9)	10111 (3)	C(1)	C(10)	H(10-2)	115 (4)
$\mathbf{C}(1)$	C(2)	0	129.6 (5)	C(9)	C(10)	H(10-2)	120(4)
C(3)	C(2)	ŏ	127.4(5)	H(10-1)	C(10)	H(10-2)	96 (5)
C(2)	C(3)	$\tilde{C}(3)$	112.9(4)		0(10)	()	
$\widetilde{C(4)}$	C	C(3)	118.6(3)				
$\tilde{C}(9)$	$\tilde{C}(3)$	C(3)	119.2(4)				
Č(5)	Č(4)	Cl(4)	115.5 (4)				
C(3)	C(4)	Cl(4)	116.6 (4)				
C(8)	C(4)	Cl(4)	117.5 (4)				
C(4)	C(5)	Cl(5)	112.4 (4)				

ences of the $(U_{ij})_{obs}$ and $(U_{ij})_{cale}$ remain about the same. They could be due to systematic errors in the β_{ij} or, contrary to expectation, the rigid body model is not applicable. Since a large part of the high order intensities were unobserved, the β_{ij} might well be biased, in which case the weighting is not meaningful. However, the results might give some qualitative information about the thermal motion of the molecule, in that the vibration tensors **T** and $\boldsymbol{\omega}$ are both nearly isotropic, and the r.m.s. amplitudes of the translational movement and of the rotation are about 0.2 Å and 3.4°, respectively.

The atomic positions were corrected for this motion (Cruickshank, 1961) and new bond lengths and angles calculated. They differ from those given in Tables 5 and 6 and discussed above by less than $\frac{1}{2}\sigma$.

Table 7. T and ω tensors for rigid body motion

APPENDIX

After this paper had been submitted for publication, Dr K. N. Trueblood kindly analyzed the thermal parameters for rigid body motion using a new program by Schomaker & Trueblood (1966). Besides the tensors T for translation and L for libration, the program calculates an additional tensor S to account for correlations between libration and translation (L is the same as Cruickshank's ω). The results obtained were substantially better than the ones mentioned above. The e.s.d. of the $(U_{ij})_{obs}$, derived from the differences ΔU_{ij} (Table 9) was found to be 0.0037 Å². Thus the rigid body model now explains the observed temperature parameters quite accurately.

Table 10. Results of the analysis of thermal motionby K.N. Trueblood

T and its estimated standard deviation is given in 10^{-2} Å², Principal axes of translation tensor T (Å) and libration tensor ω in (°)². L (°). 22 33 12 13 23 11 Direction cosines to a, b, c* Axes 4.57 0.30 -0.670.06 Т 4.38 3.37 -0.03000.27 0.18 0.19 0.20 Т 0.223 -0.61260.7898 e.s.d. (T) 0.21 0.23 0.3549 0.5811 0.6 0.195 0.7323 13.4 -0.7 -1.510.2 12.6 ω 1.9 -0.29760.9346 -0.19541.6 1.5 1.5 0.182 e.s.d. (ω) 2.6 $2 \cdot 2$

Table 8. Analysis of thermal parameters for rigid body motion. Observed and calculated U_{ij} (in 10^{-2} Å^2)

	U_{11}		U_{22}		U_{33}		L	/12	U ₁₃		U	J ₂₃
	obs	calc	obs	calc	obs	calc	obs	calc	obs	calc	obs	calc
C (1)	4.31	4.85	4.84	5.08	5.65	6.30	-0.20	-0.41	-0.95	-1.07	0.19	-0.28
C(2)	5.14	4.63	4.96	4.70	4.60	5.73	1.00	0.74	0.97	- 1.00	- 0.09	0.13
$\tilde{C}(3)$	5.54	4.71	2.90	3.65	4.96	4.89	0.56	0.48	-0.88	-0.50	-0.57	-0.06
C(4)	3.92	4.40	3.50	3.57	5.55	4.73	-0.39	0.30	- 0.74	-0.72	0.85	0.05
$\tilde{C}(\tilde{S})$	4.56	5.02	4.10	3.88	4.45	4.73	0.81	0.40	-0.21	-0.54	-0.27	-0.11
Č(6)	4.31	5.74	4.28	3.85	5.48	5.51	-0.31	-0.09	-0.62	-0.78	0.66	-0.48
$\vec{C}(\vec{7})$	5.81	6.66	3.69	3.38	6.26	6.28	0.38	0.36	-0.24	-0.41	0.01	0.21
$\tilde{C}(8)$	5.99	5.51	4.39	3.85	5.00	5.02	0.23	0.54	-0.66	-0.71	0.17	0.49
Č(9)	5.79	5.44	4.73	4.43	4.92	5.08	0.51	0.15	0.33	-0.01	0.45	0.30
Č(10)	5.09	5.25	5.98	5.49	5.76	7.03	0.05	-0.74	0.68	0.21	0.40	0.21
0	6.80	5.79	5.73	6.02	6.15	7.01	2.28	2.78	-1.51	- 1.54	0.51	0.58
Čl(3)	9.19	7.14	3.79	3.98	6.60	6.34	0.84	0.78	-0.78	-0.50	-1.22	-0.80
Cl(4)	4.96	4.81	5.04	5.41	8.43	10.23	-1.29	-0.47	- 1.97	- 1.08	0.03	-0.28
CI(5)	4.75	6.20	5.14	5.79	7.97	9.47	0.55	1.19	1.02	1.29	-0.33	-0.50

Table 9. $\Delta U_{ij} = (U_{ij})_{obs} - (U_{ij})_{cale}$, obtained by K. N. Trueblood (in 10⁻² Å²)

	ΔU_{11}	ΔU_{22}	ΔU_{33}	ΔU_{12}	ΔU_{13}	ΔU_{23}
C(1)	0.11	-0.24	0.13	-0.19	-0.33	0.57
$\tilde{C}(2)$	0.27	0.23	-0.44	-0.08	-0.22	-0.10
$\overline{C}(\overline{3})$	0.12	-0.79	0.30	-0.06	-0.18	-0.41
C(4)	-0.66	0.04	0.37	-0.38	0.27	0.71
C(5)	0.09	0.37	-0.58	0.72	-0.03	-0.31
C(6)	-0.27	0.53	0.25	-0.19	-0.22	-0.23
C(7)	0.36	0.36	-0.04	0.22	0.09	-0.35
C(8)	0.55	0.49	-0.14	-0.13	0.39	-0.37
C(9)	-0.17	0.08	0.32	0.10	0.20	0.21
C(10)	0.06	0.32	-0.44	0.28	0.12	0.23
0`́	0.42	-0.28	-0.04	-0.05	-0.30	0.04
Cl(3)	0.17	-0.35	0.07	-0.16	0.10	-0.13
Cl(4)	-0.65	-0.20	0.01	-0.22	-0.09	0.25
Cl(5)	-0.39	-0.53	0.23	0.09	-0.10	-0.12

Table 10 (cont.)

L	4.00	0.4203	-0.8085	-0.4119
	3.59	0.1769	0.5184	-0.8366
	3.27	0.8900	0.2790	0.3606

Displacements of the libration axes from the origin (0,0,0) in Å, referred to the directions of the principal axes of **L**. Screw pitches of the libration axes in Å(°)⁻¹.

Displacement along axis

Axis	1	2	3	Screw pitch
1 2 3	-1.60 - 1.15	-1.55 -1.63	2·86 2·95	0.0043 - 0.0098 - 0.0052

The standard deviations of the r.m.s. amplitudes are 0.003 Å for T and 0.25° for L.

Table 10 describes the results in terms of three translations and three screw librations about three nonintersecting axes. Of the three screw pitches, only two are independent. The ones listed are derived by setting the trace of S equal to zero. T and L are not very different from the tensors listed in Table 7, and the screw pitches are quite small. However, the three libration axes do not intersect. The axes 2 and 3 are displaced by 0.45 Å parallel to axis 1.

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Acta Cryst. (1968). B24, 246 The Crystal Structure of Decammine-µ-peroxo-dicobalt Pentanitrate*

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The structure of decammine- μ -peroxo-dicobalt pentanitrate, $(NH_3)_5COO_2CO(NH_3)_5(NO_3)_5$, has been re-examined. The substance forms tetragonal crystals, a = 11.96, c = 8.08 Å; there are two formula units in the cell. We have collected a completely new set of data and, working in space group $P4_2/mnm$, we have refined the structure to an R index of 0.054 for 361 non-zero reflections. Every atom in the structure, except the cobalt atoms, appears to suffer some degree of disorder; for some of the nitrate groups this disorder is so severe as to prevent a satisfactory description of them. However, the detailed structure of the cation is clear: each cobalt atom is bonded to only one of the oxygen atoms of the bridging $-O_2$ - group, and the O-O axis is skewed to the Co-Co axis, just as was found in the salt $(NH_3)_5COO_2CO(NH_3)_5SO_4(HSO_4)_3$. The O-O distance and the planarity of the Co-O-O-Co atoms both indicate that the bridging group is a superoxide radical, rather than a peroxide ion.

Introduction

The crystal structure of decammine- μ -peroxo-dicobalt pentanitrate, $(NH_3)_5COO_2Co(NH_3)_5(NO_3)_5$, was first

investigated by Vannerberg & Brosset (1963; hereafter VB). They derived a structure, based on the space group $P4_{2}nm$, in which the bridging peroxide group was perpendicular to the Co-Co direction (I); this arrangement had been proposed earlier on theoretical grounds by Vlček (1960). On the other hand, we have found a skewed arrangement (II) for the cation [(NH₃)₅CoO₂Co(NH₃)₅]⁵⁺ crystallized as the monosulfate tris(bisulfate) salt (Schaefer & Marsh, 1966). We

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