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The Crystal Structure of Acetohydroxamic Acid Hemihydrate

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The structure of acetohydroxamic acid hemihydrate, $CH_3.CO.NHOH.\frac{1}{2}H_2O$, has been determined using three-dimensional X-ray intensities measured with a proportional counter. Atomic positions and anisotropic vibrational parameters have been refined using full-matrix least squares, and a vibrational analysis carried out. The final R value is 0·052, the principal interatomic distances (Å) within the molecule are C–C, 1·505 (6); C–O, 1·245 (6); C–N, 1·333 (6); N–O, 1·400 (5).

Introduction

This study of acetohydroxamic acid,

$$CH_3.CO.NHOH.\tfrac{1}{2}H_2O,$$

is part of a series of investigations of the crystal structures of simple amides. The authors' interest lay in comparing the geometries of the amidic and hydroxamic groups, and in a comparison of the hydrogen bonding arrangement with those found in typical amides.

Crystal data were gathered, and the approximate structure determined, at the University of Birmingham. Three-dimensional refinement was started, using Rollett's block-diagonal least-squares program, on the MERCURY computer at the University of Oxford. Refinement was later recommenced and completed by one of the authors (BHB) at the Atomic Energy Research Establishment, Harwell. The second refinement used a full-matrix least-squares program, and the results of this second refinement are reported here.

Experimental

Acetohydroxamic acid was prepared by heating equivalent proportions of acetamide and hydroxylamine hydrochloride to 100°C. Ethyl acetate was used as a solvent for extracting and recrystallizing the product. During preliminary crystallographic work, it became evident that the acid obtained in this way is hydrated, and the composition was checked by quantitative analysis with the following results:

	%C	%H	%N
Expected for $CH_3CONHOH$	32·0	6·7	18·7
Expected for $CH_3CONHOH.\frac{1}{2}H_2O$	28·6	7·1	16·7
Found	27·4	7·1	16·9

Weissenberg photographs showed that the crystal is orthorhombic. The systematic absences, $h0l$ when $(h+l)=2n+1$ and $0kl$ when $(k+l)=2n+1$, indicated the space group to be either $Pnnm$ or $Pnn2$. A statistical analysis of X-ray intensities in the $0kl$ zone (Howells, Phillips & Rogers, 1950) indicated that this zone is acentric, and the space group is therefore, $Pnn2$. The cell dimensions were obtained from a least-squares fit to 19 interplanar spacings, measured on the three-circle diffractometer using Cu $K\alpha$ radiation ($\lambda=1·5418$ Å). The density was measured by flotation in a mixture of chlorobenzene and bromobenzene. Crystal data are given in Table 1.

Table 1. *Crystal data*

Acetohydroxamic acid hemihydrate $C_2NO_2H_5.\frac{1}{2}H_2O$

$a = 8·406 \pm 0·002$ Å
$b = 12·046 \pm 0·004$
$c = 4·037 \pm 0·001$
$V = 408·8 \pm 0·02$ Å^3
$Z = 4C_2NO_2H_5 + 2H_2O$
$D_{obs} = 1·36$ g.cm^{-3}
$D_{calc} = 1·36$ g.cm^{-3}

Initially, two-dimensional intensity data were measured, using photographic and counter methods, for the $hk0$ and $0kl$ zones. After determination of the approximate structure, three-dimensional intensities were measured on the diffractometer described by Small & Travers (1961) using Cu $K\alpha$ radiation. In all, 456 out of a possible 498 intensities were measurable. No attempt was made to correct these data for absorption.

Determination of the structure

The x and y coordinates were obtained by interpretation of a sharpened Patterson synthesis projected onto (001) and trial and error methods were used to determine the z coordinates from $0kl$ data.

In the non-centrosymmetric space group $Pnn2$, an arbitrary specification of the origin in the z direction is required. In this case, the oxygen atoms of the water molecules, lying in the special positions $(0,0,z)$ and $(\frac{1}{2},\frac{1}{2},\frac{1}{2}+z)$ were given a fixed z coordinate of $z=0$.

Two-dimensional refinement was carried out by Fourier and least-squares methods, until the discrepancy indices were $R_{hk0}=0\cdot11$ and $R_{0kl}=0\cdot16$. $R=\sum||F_o|-|F_c||/\sum|F_o|$.

Three-dimensional refinement was carried out using a modified version of the full-matrix least-squares program of Gantzel, Sparks & Trueblood (ACA No. 317), on the IBM 7030 and 360/65 computers at Aldermaston and Harwell. This program minimizes the quantity $\sum w(|F_o|-|F_c|)^2$. Refinement of the positions and anisotropic vibrational parameters of the C, N and O atoms, with all reflexions given equal weight, converged with $R_{hkl}=0\cdot078$. At this point, the summation of a three-dimensional (F_o-F_c) synthesis gave the positions of the six hydrogen atoms. These were included in the structure model with isotropic temperature parameters based on the vibrational amplitudes of their parent atoms. In the final cycles of refinement, the positions of the hydrogen atoms (but not their vibrational parameters) were refined. The ultimate weighting scheme was

$$w=\{1+[(|F_o|-6\cdot0)/8\cdot0]^2\}^{-1}$$

and a final reliability index of $R_{hkl}=0\cdot052$ was obtained. An (F_o-F_c) synthesis revealed no significant features.

For all refinement calculations, atomic scattering factors as given in *International Tables for X-ray Crystallography* (1962) were used. The final positional and vibrational parameters are shown in Tables 2 and 3 respectively, while the atom labelling scheme is illustrated in Fig. 1. Table 4 shows a list of final observed and calculated structure amplitudes.

Table 2. *Fractional atomic coordinates*

The estimated standard deviations in parentheses refer to the last decimal positions of respective values.

	x	y	z
C(1)	0·42260 (45)	0·26020 (30)	−0·0202 (18)
C(2)	0·42346 (66)	0·13746 (35)	−0·0809 (23)
N	0·29498 (41)	0·31228 (26)	−0·1311 (18)
O(1)	0·27495 (34)	0·42586 (24)	−0·0801 (18)
O(2)	0·53290 (35)	0·30813 (23)	0·1219 (18)
O(3)	0·0 (0)	0·0 (0)	0·0 (0)
H(1)	0·3258 (82)	0·1027 (48)	−0·208 (19)
H(2)	0·4294 (85)	0·1090 (53)	0·124 (20)
H(3)	0·5301 (82)	0·1122 (49)	−0·180 (22)
H(4)	0·2123 (72)	0·2792 (45)	−0·239 (18)
H(5)	0·3345 (80)	0·4420 (54)	−0·207 (19)
H(6)	0·0107 (71)	0·0598 (42)	−0·144 (22)

In order to assess librational corrections to the coordinates of the C, N and O atoms, a vibrational analysis was carried out using the equations given by

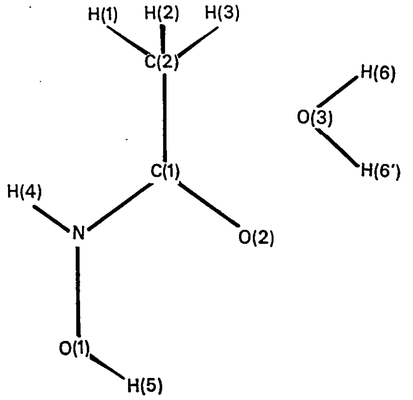

Fig. 1. Atom labelling system for acetohydroxamic acid and water. H(6) and H(6′) are symmetrically related.

Table 3. *Vibrational parameters*

The temperature factor expressions used were, for C, N and O atoms, $\exp[-10^{-4}(h^2b_{11}+k^2b_{22}+c^2b_{33}+hkb_{12}+hlb_{13}+klb_{23})]$ and for H atoms, $\exp(-B\sin^2\theta/\lambda^2)$. The estimated standard deviations in parentheses refer to the last digit of respective values.

	b_{11}	b_{22}	b_{33}	b_{12}	b_{13}	b_{23}
C(1)	86 (5)	43 (2)	422 (26)	15 (6)	−12 (23)	10 (17)
C(2)	144 (7)	38 (3)	727 (43)	−13 (7)	−77 (37)	−29 (21)
N	82 (5)	38 (2)	698 (35)	4 (5)	−94 (23)	19 (16)
O(1)	103 (5)	44 (2)	788 (32)	19 (5)	−15 (23)	7 (16)
O(2)	99 (4)	47 (2)	832 (30)	20 (5)	−230 (21)	−94 (14)
O(3)	138 (7)	36 (2)	571 (34)	6 (7)	0 (0)	0 (0)

	B
H(1)	3·57
H(2)	3·57
H(3)	3·57
H(4)	2·94
H(5)	3·30
H(6)	3·03

Table 4. *List of observed and calculated structure amplitudes*

H	K	L	FO	FC
0	0	2	3242	3359
0	0	4	556	543
0	1	1	748	756
0	1	3	586	612
0	2	0	2466	2762
0	2	2	630	653
0	2	4	200	188
0	3	1	2473	2558
0	3	3	505	528
0	4	0	423	406
0	4	2	1083	1093
0	4	4	520	532
0	5	1	1014	1025
0	5	3	880	913
0	6	0	313	321
0	6	2	671	684
0	6	4	392	400
0	7	1	3221	3310
0	7	3	678	686
0	8	0	1114	1145
0	8	2	700	692
0	8	4	643	657
0	9	1	929	919
0	9	3	113	97
0	10	0	760	707
0	10	2	481	479
0	11	1	649	676
0	11	3	638	584
0	12	0	439	507
0	12	2	561	567
0	13	1	254	265
0	14	0	281	263
0	14	2	308	294
1	0	1	3950	4147
1	0	3	520	535
1	0	5	177	227
1	1	0	2042	1942
1	1	1	3963	4277
1	1	2	820	815
1	1	3	867	864
1	1	4	102	153
1	2	0	2394	2612
1	2	1	3780	3993
1	2	2	1514	1528
1	2	3	414	406
1	2	4	146	191
1	3	0	130	85
1	3	1	1358	1332
1	3	2	1521	1495
1	3	3	402	402
1	3	4	334	334
1	4	0	748	730
1	4	1	1184	1145
1	4	2	663	668
1	4	3	95	150
1	4	4	230	217
1	5	0	3533	3952
1	5	1	897	875
1	5	2	1017	939
1	5	3	333	319
1	6	0	2025	1996
1	6	1	658	671
1	6	2	1132	1095
1	6	3	793	800
1	6	4	168	162
1	7	0	1444	1469
1	7	1	570	575
1	7	2	243	223
1	7	3	407	401
1	7	4	314	321
1	8	0	958	909
1	8	1	858	833
1	8	2	595	558
1	8	3	557	535
1	8	4	110	125
1	9	0	284	277
1	9	1	880	847
1	9	2	404	403
1	9	3	468	423
1	9	4	253	255
1	10	0	1027	1074
1	10	1	657	676
1	10	2	783	779
1	10	3	657	665
1	11	0	967	1040
1	11	1	501	503
1	11	2	232	240
1	11	3	225	238
1	12	0	106	24
1	12	1	278	250
1	12	2	322	308
1	12	3	135	66
1	13	2	450	398
1	14	0	133	56
1	14	1	270	265
2	0	0	1684	1551
2	0	2	2568	2593
2	0	4	308	327
2	1	0	3080	3224
2	1	1	3929	3889
2	1	2	1823	1847
2	1	3	950	913
2	1	4	312	306
2	2	0	1411	1368
2	2	1	807	802
2	2	2	745	739
2	2	3	103	136
2	2	4	282	275
2	3	0	576	535
2	3	1	2330	2304
2	3	2	307	332
2	3	3	656	713
2	3	4	271	272
2	4	0	1504	1489
2	4	1	735	675
2	4	2	943	894
2	4	3	109	119
2	4	4	304	310
2	5	0	379	389
2	5	1	1362	1308
2	5	2	503	499
2	5	3	278	248
2	5	4	212	256
2	6	0	1859	1814
2	6	1	1343	1256
2	6	2	653	649
2	6	3	499	490
2	6	4	78	107
2	7	0	704	686
2	7	1	1059	1022
2	7	2	681	663
2	7	3	369	385
2	7	4	231	219
2	8	0	1754	1752
2	8	1	557	577
2	8	2	1781	1755
2	8	3	261	282
2	8	4	272	256
2	9	0	567	570
2	9	1	343	344
2	9	2	494	491
2	9	3	338	338
2	9	4	164	182
2	10	0	184	175
2	10	1	441	451
2	10	2	735	731
2	10	3	305	318
2	11	0	506	480
2	11	1	586	590
2	11	2	282	266
2	11	3	307	319
2	12	0	255	220
2	12	1	290	300
2	12	2	219	211
2	13	1	485	479
2	13	2	127	76
2	14	1	311	265
3	0	1	2785	2682
3	0	3	709	698
3	1	0	342	185
3	1	1	143	127
3	1	2	1004	1009
3	1	3	859	832
3	1	4	201	223
3	2	0	4289	4241
3	2	1	1841	1840
3	2	2	1245	1270
3	2	3	312	318
3	2	4	430	439
3	3	0	556	516
3	3	1	1349	1314
3	3	2	1379	1302
3	3	3	759	746
3	3	4	80	136
3	4	0	517	525
3	4	1	544	517
3	4	2	633	643
3	4	3	287	256
3	4	4	300	332
3	5	0	1562	1617
3	5	1	563	571
3	5	2	1073	1030
3	5	3	487	462
3	5	4	181	156
3	6	0	491	512
3	6	1	450	404
3	6	2	324	353
3	6	3	217	254
3	6	4	290	258
3	7	0	637	589
3	7	1	1204	1267
3	7	2	789	759
3	7	3	133	150
3	7	4	124	113
3	8	1	1056	1059
3	8	2	263	228
3	8	3	464	484
3	8	4	116	117
3	9	0	721	732
3	9	1	1819	1825
3	9	2	338	367
3	9	3	459	486
3	10	0	923	922
3	10	1	511	517
3	10	2	386	396
3	10	3	218	247
3	11	0	217	188
3	11	1	147	178
3	11	2	484	472
3	11	3	126	98
3	12	0	234	223
3	12	1	352	393
3	12	2	213	232
3	14	0	531	510
3	14	1	267	241
4	0	0	3368	3350
4	0	2	401	437
4	0	4	297	273
4	1	0	1147	1059
4	1	1	751	715
4	1	2	676	635
4	1	3	469	486
4	1	4	539	573
4	2	0	700	692
4	2	1	2213	2163
4	2	2	1194	1185
4	2	3	526	521
4	2	4	336	352
4	3	0	651	600
4	3	1	1476	1449
4	3	2	744	700
4	3	4	369	382
4	4	0	373	410
4	4	1	696	649
4	4	2	260	216
4	4	3	584	554
4	5	0	117	82
4	5	1	375	683
4	5	2	404	416
4	5	3	684	638
4	5	4	235	223
4	6	0	428	424
4	6	1	293	273
4	6	2	555	534
4	6	3	470	466
4	6	4	223	153
4	7	0	1849	1781
4	7	1	980	966
4	7	2	458	496
4	7	3	195	140
4	8	0	1172	1180
4	8	1	258	281
4	8	2	260	245
4	8	3	209	190
4	9	0	1905	1968
4	9	1	633	637
4	9	2	740	706
4	9	3	229	211
4	10	0	970	983
4	10	1	638	635
4	10	2	264	286
4	10	3	167	41
4	11	0	361	332
4	11	1	142	133
4	11	2	120	115
4	12	0	313	285
4	12	1	261	287
4	12	2	340	311
4	13	0	135	155
4	13	1	309	270
4	14	0	223	222
5	0	1	294	344
5	0	3	989	997
5	1	0	2431	2341
5	1	1	1067	1074
5	1	2	922	892
5	1	3	815	780
5	1	4	166	183
5	2	0	305	275
5	2	1	344	319
5	2	2	628	599
5	2	3	428	457
5	2	4	118	144
5	3	0	941	909
5	3	1	588	593
5	3	2	522	494
5	3	3	266	286
5	3	4	479	412
5	4	0	1463	1369
5	4	1	752	666
5	4	2	372	315
5	4	3	441	429
5	4	4	110	209
5	5	0	881	903
5	5	1	566	582
5	5	2	209	191
5	5	3	207	205
5	5	4	134	165
5	6	0	117	97
5	6	1	128	145
5	6	2	662	705
5	6	3	245	222
5	6	4	161	175
5	7	0	238	184
5	7	1	729	734
5	7	2	368	395
5	8	0	524	524
5	8	1	1583	1577
5	8	2	423	445
5	8	3	392	399
5	9	0	386	321
5	9	1	504	463
5	9	3	105	169
5	10	0	116	161
5	10	1	397	425
5	10	2	446	402
5	10	3	229	261
5	11	0	635	675
5	11	1	403	410
5	11	2	153	182
5	12	0	352	353
5	12	1	202	198
5	12	2	268	265
5	13	0	151	141
6	0	0	714	726
6	0	2	1056	1045
6	0	4	238	283
6	1	0	370	404
6	1	1	498	508
6	1	2	1176	1203
6	1	3	236	265
6	1	4	93	109
6	2	0	523	527
6	2	1	963	932
6	2	2	771	725
6	2	3	472	462
6	2	4	315	343
6	3	0	742	717
6	3	1	767	723
6	3	2	185	161
6	3	3	208	170
6	3	4	179	143
6	4	0	430	457
6	4	1	349	782
6	4	2	186	164
6	4	3	207	199
6	5	0	346	369
6	5	1	206	198
6	5	2	297	268
6	5	3	408	406
6	6	0	1041	1019
6	6	1	646	719
6	6	2	496	534
6	6	3	400	392
6	7	0	141	129
6	7	1	541	559
6	7	2	621	586
6	7	3	326	326
6	8	0	357	349
6	8	1	258	267
6	8	3	165	136
6	9	0	120	161
6	9	1	273	257
6	9	2	613	641
6	10	0	729	725
6	10	1	495	491
6	10	2	316	313
6	11	0	374	330
6	11	1	255	262
6	11	2	142	133
6	12	1	214	324
7	0	1	2050	2079
7	0	3	692	685
7	1	0	220	194
7	1	1	763	766
7	1	2	299	312
7	1	3	91	122
7	2	0	512	457
7	2	1	321	309
7	2	2	348	327
7	3	0	398	382
7	3	1	308	341
7	3	2	197	278
7	3	3	193	192
7	4	1	276	259
7	4	2	675	677
7	4	3	394	362
7	5	1	245	192
7	5	2	242	255
7	5	3	109	129
7	6	0	383	389
7	6	1	122	155
7	6	2	297	333
7	6	3	242	186
7	7	0	1052	1073
7	7	1	422	382
7	7	2	803	772
7	7	3	203	213
7	8	0	278	281
7	8	1	330	330
7	9	0	380	348
7	9	1	323	323
7	9	2	194	160
7	10	0	194	200
7	10	1	105	113
7	11	0	278	229
7	11	1	186	214
8	0	0	507	513
8	0	2	347	320
8	1	0	1408	1436
8	1	2	405	394
8	1	3	212	141
8	2	0	821	823
8	2	1	596	547
8	2	2	497	510
8	2	3	161	174
8	3	2	328	298
8	3	3	99	129
8	4	1	333	339
8	4	3	242	308
8	5	1	632	664
8	5	2	123	227
8	6	0	145	39
8	6	1	559	608
8	6	2	242	248
8	7	0	283	302
8	7	1	201	160
8	7	2	168	168
8	8	1	303	310
8	9	0	448	440
8	10	0	242	219
9	0	1	186	227
9	1	0	526	556
9	1	1	738	759
9	1	2	356	364
9	2	0	230	247
9	2	1	132	116
9	2	2	189	178
9	3	1	401	402
9	3	2	330	287
9	4	0	689	669
9	4	1	214	217
9	5	0	135	95
9	5	2	85	95
9	6	0	707	672
9	6	1	244	245
9	7	1	90	63
9	8	0	135	187
10	0	0	314	302
10	1	0	414	408
10	1	1	148	130
10	2	1	433	376
10	3	0	528	491
10	3	1	247	239
10	4	0	180	147
10	4	1	336	310

Table 5. *Principal axes of thermal ellipsoids*

U_i is the root-mean-square displacement of the *i*th principal axis of the ellipsoid. c_{ia}, c_{ib}, c_{ic} are the direction cosines, multiplied by 10^4, of the angles between the *i*th axis and the crystallographic axes.

		u_i	c_{ia}	c_{ib}	c_{ic}
C(1)	1	0·188	4148	6367	6500
	2	0·187	−5570	−3872	7348
	3	0·164	7195	−6668	1940
C(2)	1	0·252	−4669	−0457	8831
	2	0·221	8674	−2180	4473
	3	0·165	1721	9749	1414
N	1	0·245	−2529	0630	9654
	2	0·170	6335	7650	1160
	3	0·161	7313	−6409	2333
O(1)	1	0·255	− 417	190	9989
	2	0·200	8423	5384	250
	3	0·171	−5373	8425	−384
O(2)	1	0·285	−4071	−2614	8752
	2	0·177	−1614	9637	2128
	3	0·162	8990	547	4345
O(3)	1	0·222	9977	685	0
	2	0·217	0	0	10000
	3	0·163	−685	9976	0

Cruickshank (1956, 1961). It was assumed that the molecule is a rigid body librating about its centre of mass. The program of Gantzel, Coulter & Trueblood (ACA No. 232), modified to give librational corrections, was used for this purpose. Table 5 shows the principle axes of vibration, and Table 6, the rigid-body tensors **T** and **ω**.

Table 6. *Rigid-body translational and librational tensors*

Elements T_{ij} of the mean-square translational tensor, and those, ω_{ij} of the librational tensor, are expressed in Å^2 and radians^2 respectively. All values have been multiplied by 10^4. Estimated standard deviations, referring to the last digit of respective values, are in parentheses.

T_{11}	277 (25)	ω_{11}	73 (18)
T_{22}	272 (21)	ω_{22}	231 (36)
T_{33}	371 (37)	ω_{33}	49 (12)
T_{12}	−4 (21)	ω_{12}	−44 (21)
T_{13}	−8 (28)	ω_{13}	7 (12)
T_{23}	−6 (24)	ω_{23}	1 (23)

The molecular arrangement

The molecular arrangement of acetohydroxamic acid, projected onto (001), is illustrated in Fig. 2. In this diagram, molecules labelled *A* and *B* are roughly at $z=0$, while those labelled *C* and *D* are at $z=\frac{1}{2}$.

There are three types of hydrogen bond in the structure. The first type connects the O(2) and N atoms of molecules *A* and *C* respectively: the relevant interatomic distances are O· · ·N, 2·80; O· · ·H, 1·91; N–H, 0·90 Å, giving an O–H–N angle of 169·8°. This first type of hydrogen bond links molecules into chains, parallel to $(10\bar{1})$ for molecules *A* and *C*, and to (101) for *B* and *D*. The closest non-bonding approach in each chain is 2·69 Å, between H(3*a*) and H(4*c*). The angle between the $(10\bar{1})$ plane and the plane through molecule *A* is 8·7°.

The other two types of hydrogen bond connect each water molecule to four acid molecules, giving roughly a tetrahedral arrangement of hydrogen bonds round each O(3). The O(1)H(5) groups of molecules *A* and *C* form hydrogen bonds with the water oxygen at position $(\frac{1}{2},\frac{1}{2},-\frac{1}{2})$. Relevant distances are: O· · ·O, 2·69; O· · ·H, 1·95 and O–H, 0·75 Å. The O–H–O angle is 169·2°. The third type of hydrogen bond connects the water oxygen atom at $(\frac{1}{2},\frac{1}{2},\frac{1}{2})$ with atoms O(2*c*) and O(2*d*), using the water hydrogen atoms. The interatomic distances are: O· · ·O, 2·78; O· · ·H, 1·86; O–H, 0·93 Å, giving an O–H–O angle of 169·6°.

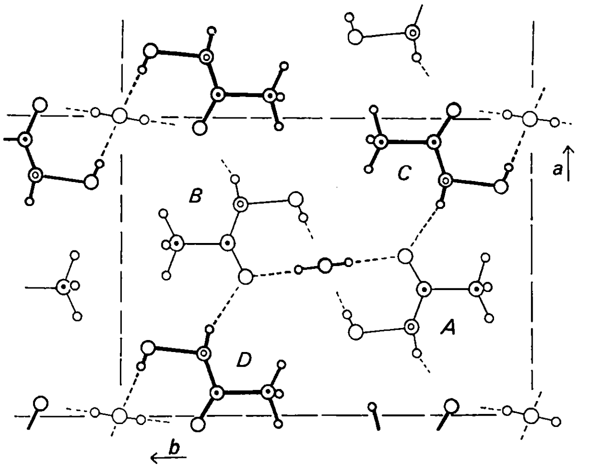

Fig. 2. The crystal structure projected onto (001). Molecules *A* and *B* are at height $z=0$; *C* and *D* are at $z=\frac{1}{2}$. Large and small open circles represent O and H atoms respectively. Circles containing dots and rings represent C and N atoms respectively. Hydrogen bonds are shown by dotted lines.

The hydrogen atoms of the water molecule at $(\frac{1}{2},\frac{1}{2},-\frac{1}{2})$ are 2·30 and 2·69 Å away from atom H(5*a*), while the distances between atom O(1*a*) and the hydrogens of the water molecule at $(\frac{1}{2},\frac{1}{2},\frac{1}{2})$ are 2·66 and 2·99 Å.

The molecule

Bond lengths and angles, corrected for libration, are given in Table 7.

Table 7. *Bond lengths and bond angles*

The figures in parenthesis are estimated standard deviations referred to the last place of decimals.

(*a*) Bond lengths (Å) before librational corrections.

C(1)-C(2)	1·498 (6)
C(1)-N	1·321 (6)
C(1)-O(2)	1·234 (6)
N—O(1)	1·393 (5)

(*b*) Bond lengths (Å) after librational corrections.

C(1)-C(2)	1·505 (6)	C(2)-H(1)	1·05 (7)
C(1)-N	1·333 (6)	C(2)-H(2)	0·89 (8)
C(1)-O(2)	1·245 (6)	C(2)-H(3)	1·02 (8)
N—O(1)	1·400 (5)	N—H(4)	0·90 (7)
		O(1)-H(5)	0·75 (8)
O(3)-H(1)	0·93 (7)		

(*c*) Bond angles (°) before librational corrections.

C(2)-C(1)-N	114·6 (4)
C(2)-C(1)-O(2)	122·3 (4)
N—C(1)-O(2)	123·1 (4)
C(1)-N—O(1)	120·9 (4)

(*d*) Bond angles (°) after librational corrections.

C(2)-C(1)-N	114·3 (4)
C(2)-C(1)-O(2)	122·4 (4)
N—C(1)-O(2)	123·3 (4)
C(1)-N—O(1)	120·6 (4)
C(1)-C(2)-H(1)	118 (3)
C(1)-C(2)-H(2)	102 (5)
C(1)-C(2)-H(3)	111 (4)
H(1)-C(2)-H(2)	110 (7)
H(2)-C(2)-H(3)	102 (7)
H(1)-C(2)-H(3)	112 (6)
C(1)-N—H(4)	125 (4)
O(1)-N—H(4)	114 (3)
N—O(1)-H(5)	94 (5)
Water H-O-H	103 (7)

Atoms C(1), C(2), N and O(2) are coplanar within the limits of experimental error, atom O(1) lying 0·056 Å from this plane. The equation of the plane is given in Table 8.

Table 8. *Deviations from molecular plane*

Deviations of the atoms from the plane through C(1), C(2), N and O(2) are given in Å. The equation of the plane, referred to axes *a*, *b*, *c*, is $-0{\cdot}4528x-0{\cdot}1480y+0{\cdot}8793z+2{\cdot}1444=0$.

C(1)	0·0006
C(2)	−0·0002
N	−0·0002
O(1)	0·056
O(2)	−0·0002

A full discussion of the significance of these interatomic distances in relation to those observed in other simple derivatives of acetamide will be given later, elsewhere.

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Die Kristall- und Molekülstruktur von $[Co_3(NH_3)_8(OH)_2(NO_2)_2(CN)_2](ClO_4)_3.NaClO_4.2H_2O$

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The structure of $[Co_3(NH_3)_8(OH)_2(NO_2)_2(CN)_2]\ (ClO_4)_3.NaClO_4.2H_2O$ has been determined by X-ray methods. The compound crystallizes in the monoclinic space group $A2\ (C_2^3)$ with $a=14{\cdot}11$, $b=7{\cdot}86$, $c=14{\cdot}84$ Å, $\beta=112{\cdot}3°$, $Z=2$. The cation is of symmetry C_2. At the central Co atom there are two CN^- ligands in the *cis* position. This Co atom is linked to the two $Co(NH_3)_4$ groups by one OH and one NO_2 bridge each. The N atoms of the two NO_2 bridges are coordinated to the central Co atom *trans* to each other, while the O atoms of the OH bridges are in the *cis* position.

Einleitung

Kürzlich wurden Darstellung und Eigenschaften von cyanidhaltigen dreikernigen Kobalt(III)-amminen beschrieben (Siebert & Schiedermaier, 1968), welche u.a. das Kation $[Co_3(NH_3)_8(OH)_2(NO_2)_2(CN)_2]^{3+}$ enthalten. Aufgrund der chemischen Reaktionen und der Ultrarotspektren konnte nur festgestellt werden, dass das