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The Crystal Structures of Two Oxy-Acids of Iodine.
II. An X-ray Diffraction Study of Anhydro-iodic Acid, HI_3O_8

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Anhydro-iodic acid, HI_3O_8, crystallizes in the monoclinic system, space group $P2_1/n$. The cell dimensions are $a = 7{\cdot}55 \pm 0{\cdot}01$, $b = 7{\cdot}68 \pm 0{\cdot}01$, $c = 11{\cdot}40 \pm 0{\cdot}015$ Å, $\beta = 90{\cdot}1 \pm 0{\cdot}1°$. There are four units of HI_3O_8 per cell.

Refinement has been carried out by the method of least squares with allowance for anisotropic vibration for the iodine atoms. R decreased to 0·056 for 2412 independent *hkl* reflexions obtained by X-ray diffraction. The structure consists of units HIO_3 and I_2O_5 which show a strong intermolecular interaction. Many intermolecular I · · · O distances are as short as 2·6 Å; the thermal motion of the atoms is small. The I_2O_5 group with structure $O_2I–O^*–IO_2$ has four 'double' bonds of 1·79 Å and two single bonds (I–O*) of 1·96 Å, e.s.d. 0·013 Å. The length of the two 'double' bonds in the $HO'–IO_2$ group is 1·80 Å, the distance I–O′ is 1·90 Å. Indirect evidence for the position of the hydrogen atom was obtained.

Introduction

It has been concluded from infrared spectra of HI_3O_8 and I_2O_5 that HI_3O_8 is an addition compound of I_2O_5 and HIO_3 (Dupuis & Lecomte, 1961), and that solid I_2O_5 consists of molecules $O_2I–O–IO_2$ (Duval & Lecomte, 1960). It was thought to be worth while to verify these conclusions and to determine bond lengths and valence angles in the I_2O_5 and HIO_3 groups by X-ray analysis. The configuration of the HIO_3 molecules in solid α-iodic acid has been determined by neutron diffraction (Garrett, 1954).

Experimental

Anhydro-iodic acid was obtained by dehydration of iodic acid at 110°C (Brauer, 1960, p.281) and by oxidation of iodine with fuming nitric acid (Moles & Parts, 1935). The acid was purified by recrystallizing it from a 55% solution of nitric acid in water. The composition HI_3O_8 was checked by chemical analysis. Crystals with diameters $\leq 0{\cdot}2$ mm perpendicular to the axis of rotation were used during the X-ray work.

The crystallographic data are listed in Table 1. The dimensions of the monoclinic unit cell were determined from a diffractogram of a flat powder specimen; powder lines of CaF_2 were used as a reference.

Table 1. *Crystallographic data*

Monoclinic, space group $P2_1/n$
$\lambda(\text{Cu } K\alpha) = 1{\cdot}5418$ Å
$a = 7{\cdot}548 \pm 0{\cdot}01$ Å
$b = 7{\cdot}680 \pm 0{\cdot}01$
$c = 11{\cdot}402 \pm 0{\cdot}015$
$\beta = 90{\cdot}1 \pm 0{\cdot}1°$
Four units HI_3O_8 per cell
$d(\text{calc}) = 5{\cdot}04$, $d(\text{obs}) = 4{\cdot}97$ g.cm^{-3}.

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Morphological data of the crystals have been published by Garrido (1933). Garrido's axes a', b' and c' can be obtained from our axes a, b and c by the transformation $\mathbf{a}'=-\frac{1}{2}\mathbf{a}+\frac{1}{2}\mathbf{c}$, $\mathbf{b}'=\mathbf{b}$ and $\mathbf{c}'=\frac{1}{2}\mathbf{a}+\frac{1}{2}\mathbf{c}$. The axial ratio $a':b':c'=0{\cdot}896:1:0{\cdot}892$ and $\beta'=113{\cdot}6°$ calculated from our cell dimensions may be compared with Garrido's values, $a':b':c'=0{\cdot}901:1:0{\cdot}891$, $\beta'=112{\cdot}9$.

The intensities of the reflexions hkl with $k=0$ to 13 were measured on integrated equi-inclination Weissenberg photographs about the b axis. The photographs were taken with Zr-filtered Mo radiation, and the multiple film technique with Ilford Industrial G, B and C films was used. The intensities of different k layer lines were put on the same relative scale by recording side by side on one film small parts of integrated equi-inclination Weissenberg photographs of the different layer lines. The scaling factors were checked by comparing the values obtained for $|F(0kl)|^2$ and $|F(hk0)|^2$ from the different k levels with those obtained from integrated Weissenberg photographs of the zero layer-lines about the a and c axis.

The intensities of more than 2500 independent reflexions were measured. They were corrected for Lorentz and polarization effects and for absorption (absorption coefficient 141 cm^{-1}). The latter corrections were calculated on the Mercury computer at Kjeller (Norway). Use was made of Holte's absorption correction program devised for zero-layer line reflexions from crystals with a uniform cross section perpendicular to the rotation axis (Holte, 1961). For the reflexions $h0l$ the program could be used without modification. For the upper layer-line reflexions about the b axis the crystal measuring $0{\cdot}2\times0{\cdot}5\times0{\cdot}2$ mm was assumed to be infinitely long; the increase in path length due to increasing values of the inclination angle μ was taken into account by multiplying the absorption coefficient by sec μ (Bond, 1959). For the reflexions $0kl$ and $hk0$ which were obtained from crystals with non-uniform cross sections, Holte's program was modified so that the integration could be done for the whole volume of the crystal.

Determination of the structure

Approximate positions of the three independent iodine atoms could easily be determined from the Patterson syntheses of the three projections. The signs of nearly all reflexions were obtained from the contribution $F_{c,\mathrm{I}}$ of the iodine atoms to the calculated structure factors. The positions of the eight independent oxygen atoms were obtained by trial and error, use being made of F_o– and F_o–$F_{c,\mathrm{I}}$ syntheses of the [010] and [100] projections.

The interpretation of the Fourier maps was severely hampered by the presence of spurious maxima. In the F_o synthesis these maxima arise from series termination effects. Series termination also affected the first few difference maps where $F_{c,\mathrm{I}}$ was based on too large a value for the thermal parameter B, 0·80 Å^2 instead of the final value 0·55 Å^2; moreover the initial values obtained for the scale factor by comparison of observed and calculated structure factors were incorrect because of the large errors in B and the strong extinction of some low-order reflexions.

When approximate positions for most of the oxygen atoms had been obtained, the parameters of the iodine atoms could be refined by isotropic least-squares analysis with fixed oxygen positions. The final difference maps in Fig. 1 clearly show the positions of the oxygen atoms. Final R values for the observed reflexions are 0·078 for $h0l$, 0·086 for $0kl$ and 0·099 for $hk0$.

For the three-dimensional refinement 2412 independent reflexions with accurately measured intensities were available. The least-squares analysis started with some isotropic cycles on the electronic computer Zebra (Schoone, 1961). Refinement proceeded by five cycles on the Pegasus computer at Leeds. Cruickshank's program (Cruickshank & Pilling, 1961) was used with anisotropic thermal parameters for iodine and isotropic parameters for oxygen. The weighting scheme was $w=\text{constant}/|F_o|$; $\langle w(F_o-F_c)^2\rangle$ appeared to be reasonably constant for different values of sin θ/λ and $|F|$. The scattering factors used were those recommended in *International Tables for X-ray Crystallography* (1962). The residual R decreased to 0·056. The final parameters are listed in Tables 2 and 3; in Table 4 the calculated and observed structure factors are compared.

The thermal motion of the atoms is exceedingly low; average values are $B(\mathrm{I})=0{\cdot}55$ Å^2 and $B(\mathrm{O})=1{\cdot}09$ Å^2.

Accuracy

The standard deviations as estimated from the least-squares residuals are listed in Table 5; inaccuracies in

Table 2. *Final coordinates and thermal parameters for the oxygen atoms*

Atom	x	y	z	B(Å^2)
I(1)	0·2415	0·2274	0·0953	
I(2)	0·3376	0·7362	0·1035	
I(3)	0·7558	0·0473	0·1915	
O(1)	0·4585	0·3023	0·0532	1·25
O(2)	0·1291	0·4303	0·0872	0·92
O(3)	0·1737	0·8232	0·0076	1·20
O(4)	0·4406	0·9404	0·1375	1·11
O(5)	0·1822	0·7453	0·2397	0·88
O(6)	0·7870	0·4550	0·1566	1·24
O(7)	0·8031	0·8411	0·1246	1·19
O(8)	0·9797	0·1147	0·2253	0·87

Table 3. *Thermal parameters* U_{ij} (Å^2) *for the iodine atoms*

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{23}	U_{13}
I(1)	0·0070	0·0075	0·0067	−0·0013	0·0004	0·0034
I(2)	0·0082	0·0057	0·0064	−0·0007	0·0007	0·0016
I(3)	0·0061	0·0074	0·0081	−0·0022	−0·0007	0·0014

Table 4. *Observed and calculated structure factors*

Reflexions marked with an *N* were not taken into account in the anisotropic refinement; low order reflexions for which F_o is omitted were caught by the beam stop.

	H	K	L	FO	FC
	0	0	0	-	884
N	0	0	2	-	-29
N	0	0	4	231	-262
	0	0	6	168	-164
	0	0	10	266	288
	0	0	12	42	40
	0	0	14	137	-138
	0	0	16	55	-45
	0	0	20	106	107
	1	0	-19	58	-56
	1	0	-17	145	-155
	1	0	-15	91	89
	1	0	-13	174	185
	1	0	-9	46	-47
N	1	0	-7	311	-366
	1	0	-5	198	200
N	1	0	-3	298	410
N	1	0	-1	-	-38
N	1	0	1	-	-102
N	1	0	3	267	-371
N	1	0	7	291	247
	1	0	11	51	-47
	1	0	13	112	-112
	1	0	17	135	145
	2	0	-20	102	-106
	2	0	-18	80	82
	2	0	-16	79	72
	2	0	-14	112	114
	2	0	-12	112	-115
	2	0	-10	251	-266
	2	0	-8	125	124
	2	0	-6	155	158
	2	0	-4	144	143
	2	0	-2	138	-122
N	2	0	0	294	-463
N	2	0	2	193	215
N	2	0	4	180	204
	2	0	10	199	-205
	2	0	14	99	102
	2	0	20	59	-59
	3	0	-19	61	60
	3	0	-17	95	104
	3	0	-15	140	-146
	3	0	-13	124	-122
	3	0	-11	62	66
	3	0	-9	73	70
	3	0	-7	180	178
	3	0	-5	197	-200
	3	0	-3	180	-189
	3	0	-1	129	131
	3	0	1	82	-76
	3	0	3	113	116
	3	0	5	40	-36
	3	0	7	206	-218
	3	0	9	106	104
	3	0	17	86	-83
	4	0	-18	97	-98
	4	0	-12	95	89
	4	0	-10	107	109
	4	0	-8	173	-176
	4	0	-6	48	-44
	4	0	0	207	217
	4	0	2	178	-190
	4	0	6	93	89
	4	0	8	79	-71
	4	0	10	95	91
	4	0	12	62	-59
	5	0	-15	95	98
	5	0	-11	83	-85
	5	0	-9	53	55
	5	0	-7	74	-73
	5	0	-5	100	95
	5	0	-3	60	53
	5	0	-1	116	-114
	5	0	1	151	148
	5	0	3	119	-114
	5	0	7	66	65
	5	0	9	67	-67
	5	0	11	84	82
	5	0	15	66	-64
	6	0	-18	71	67
	6	0	-10	63	-61
	6	0	-8	95	95
	6	0	-6	97	-86
	6	0	-4	56	49
	6	0	-2	66	62
	6	0	0	105	-86
	6	0	2	67	77
	6	0	4	53	-52
	6	0	8	169	170
	6	0	10	61	-60
	6	0	16	96	102
	7	0	-17	55	56
	7	0	-9	68	-91
	7	0	-7	119	116
	7	0	1	175	-172
	7	0	3	135	125
	7	0	5	103	103
	7	0	7	64	-56
	7	0	11	120	-123
	7	0	13	54	53
	7	0	15	117	117
	8	0	-14	74	-84
	8	0	-10	92	94
	8	0	-6	65	66
	8	0	-4	123	-126
	8	0	-2	136	-137
	8	0	0	133	127
	8	0	2	55	51
	8	0	4	69	68
	8	0	6	86	-85
	8	0	8	173	-182
	8	0	10	103	121
	8	0	12	69	73
	9	0	-11	73	68
	9	0	-9	84	73
	9	0	-7	148	-173
	9	0	-5	77	-72
	9	0	-3	72	70
	9	0	-1	83	87
	9	0	1	129	131
	9	0	3	197	-209
	9	0	5	109	-106
	9	0	7	61	84
	9	0	9	71	56
	9	0	11	76	77
	9	0	13	[illegible]	-90
	9	0	15	92	-89
	10	0	-14	105	115
	10	0	-10	129	-134
	10	0	-4	151	162
	10	0	-2	67	73
	10	0	0	174	-181
	10	0	2	52	-55
	10	0	6	113	116
	10	0	8	96	101
	10	0	10	134	-129
	10	0	12	57	-52
	11	0	-11	90	-80
	11	0	-7	167	181
	11	0	-3	87	-95
	11	0	-1	64	-64
	11	0	3	153	167
	11	0	7	99	-105
	11	0	13	84	79
	12	0	-10	115	122
	12	0	-4	97	-102
	12	0	0	136	143
	12	0	6	54	-57
	12	0	10	109	108
	13	0	-7	112	-116
	13	0	-3	64	72
	13	0	3	87	-91
	13	0	7	62	64
	0	1	1	-	76
	0	1	2	42	-30
	0	1	3	146	-159
	0	1	5	142	152
	0	1	6	106	-100
	0	1	10	88	87
	0	1	11	69	64
	0	1	13	72	-76
	0	1	15	55	51
	0	1	16	51	-51
	1	1	-14	72	70
	1	1	-12	81	-87
	1	1	-11	41	28
	1	1	-10	55	54
	1	1	-8	39	33
	1	1	-7	59	-50
	1	1	-6	113	-121
	1	1	-5	72	75
	1	1	-4	157	177
	1	1	-2	109	-117
N	1	1	-1	-	69
N	1	1	1	-	-80
	1	1	2	96	96
	1	1	3	115	-123
	1	1	4	97	-101
	1	1	5	121	127
	1	1	6	98	104
	1	1	7	110	116
	1	1	9	60	56
	1	1	11	55	-58
	1	1	12	74	77
	1	1	13	95	-99
	1	1	14	83	-82
	1	1	17	76	86
	2	1	-15	44	-44
	2	1	-13	86	92
	2	1	-12	66	-64
	2	1	-11	81	-89
	2	1	-10	35	-35
	2	1	-7	77	78
	2	1	-6	103	109
	2	1	-5	101	-108
	2	1	-4	162	171
	2	1	-3	104	101
	2	1	-2	138	-145
	2	1	-1	97	-96
	2	1	0	131	-148
	2	1	1	45	-44
	2	1	2	43	43
	2	1	3	101	105
	2	1	4	173	193
	2	1	5	110	-115
	2	1	6	98	101
	2	1	7	81	75
	2	1	8	111	-113
	2	1	10	163	-170
	2	1	11	85	-88
	2	1	12	54	55
	2	1	13	70	75
	2	1	14	96	102
	2	1	16	64	71
	2	1	18	59	-63
	3	1	-15	57	-63
	3	1	-14	57	-71
	3	1	-13	54	-55
	3	1	-12	65	74
	3	1	-10	59	-55
	3	1	-9	157	166
	3	1	-7	79	80
	3	1	-6	81	79
	3	1	-5	160	-169
	3	1	-4	136	-141
N	3	1	-3	155	-208
	3	1	-2	67	64
	3	1	-1	37	32
N	3	1	1	207	194
	3	1	2	82	-81
	3	1	3	165	177
	3	1	4	158	175
	3	1	5	186	-203
	3	1	6	111	-105
	3	1	7	134	-139
	3	1	11	131	135
	3	1	12	55	-58
	3	1	13	91	96
	3	1	14	63	69
	3	1	15	91	-95
	3	1	17	109	-109
	4	1	-16	77	-83
	4	1	-13	71	-75
	4	1	-12	119	131
	4	1	-11	84	87
	4	1	-10	66	66
	4	1	-8	128	-132
	4	1	-7	65	-63
	4	1	-6	157	-161
	4	1	-5	93	92
	4	1	-3	143	-143
N	4	1	-2	244	295
	4	1	-1	70	69
	4	1	0	129	133
	4	1	1	91	87
	4	1	2	165	-179
	4	1	3	105	-108
N	4	1	4	194	-222
	4	1	5	79	7
	4	1	6	52	-46
	4	1	7	100	[illegible]
	4	1	8	174	166
	4	1	10	110	[illegible]
	4	1	11	65	71
	4	1	12	119	-124
	4	1	14	112	-114
	4	1	15	[illegible]	94
	5	1	-15	114	128
	5	1	-14	50	64
	5	1	-11	69	-77
	5	1	-10	62	63
	5	1	-9	159	-145
	5	1	-6	116	-113
N	5	1	-5	207	233
	5	1	-4	125	126
	5	1	-3	83	79
	5	1	-2	41	-44
	5	1	-1	145	-150
	5	1	0	55	50
N	5	1	1	211	-238
	5	1	4	120	-126
N	5	1	5	219	246
	5	1	6	105	103
	5	1	7	91	95
	5	1	9	77	-86
	5	1	11	128	-132
	5	1	14	69	-71
	5	1	15	106	115
	5	1	17	59	59
	6	1	-16	54	65
	6	1	-14	56	-62
	6	1	-13	65	78
	6	1	-12	143	-161
	6	1	-11	92	-89
	6	1	-8	140	163
	6	1	-7	46	43
	6	1	-6	100	97
	6	1	-4	69	-65
	6	1	-3	122	126
N	6	1	-2	216	-254
	6	1	-1	79	-77
N	6	1	2	192	219
	6	1	3	76	77
	6	1	4	112	118
	6	1	5	63	-72
	6	1	6	81	-72
	6	1	7	67	65
	6	1	8	159	-168
	6	1	11	52	-51
	6	1	12	123	133
	6	1	13	56	57
	7	1	-15	113	-127
	7	1	-14	51	-48
	7	1	-12	47	54
	7	1	-11	105	115
	7	1	-10	58	-62
	7	1	-9	118	125
	7	1	-7	78	-76
	7	1	-6	71	72
	7	1	-5	160	-175
	7	1	-4	101	-104
	7	1	-2	57	55
	7	1	-1	140	145
	7	1	1	110	108
	7	1	4	87	93
	7	1	5	147	-161
	7	1	6	66	-72
	7	1	9	110	107
	7	1	11	50	55
	7	1	12	48	-48
	7	1	14	63	61
	8	1	-14	78	84
	8	1	-13	56	-54
	8	1	-12	85	95
	8	1	-11	69	74
	8	1	-10	55	-58
	8	1	-8	118	-124
	8	1	-5	68	66
	8	1	-4	61	90
	8	1	-3	74	-80
	8	1	-2	124	127
	8	1	-1	48	48
	8	1	0	41	-40
	8	1	2	119	-126
	8	1	3	65	-68
	8	1	5	72	75
	8	1	7	62	-65
	8	1	8	65	72
	8	1	12	61	-61
	8	1	13	54	-53
	9	1	-15	60	64
	9	1	-11	63	-69
	9	1	-7	65	65
	9	1	-6	49	-50
	9	1	-5	85	91
	9	1	-4	75	81
	9	1	-2	65	72
	9	1	-1	48	-51
	9	1	1	41	-27
	9	1	4	68	-73
	9	1	5	44	45
	9	1	6	73	73
	10	1	-5	60	-59
	10	1	-3	68	75
	10	1	-1	44	-52
	10	1	5	54	-56
	11	1	-4	56	-65
	11	1	-2	48	50
N	0	2	0	-	-220
N	0	2	1	-	-113
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Table 4 (*cont.*)

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	3	11	-2	35	33
	3	11	0	67	66
	3	11	3	39	-39
	3	11	4	135	-151
	3	11	8	63	56
	3	11	14	113	-108
	3	11	18	60	53
	4	11	-13	83	80
	4	11	-11	75	-72
	4	11	-9	67	-63
N	4	11	-6	30	27
	4	11	-5	39	25
	4	11	-4	32	32
	4	11	-3	125	129
	4	11	-2	37	-30
	4	11	-1	68	-67
	4	11	1	115	-120
	4	11	4	33	32
	4	11	7	99	108
	4	11	11	102	-108
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	5	11	-16	53	50
	5	11	-14	54	-51
	5	11	-10	57	-53
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	5	11	-6	116	115
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	5	11	-2	70	-63
	5	11	0	43	-38
	5	11	2	63	58
	5	11	4	125	136
	5	11	5	35	-33
	5	11	6	76	-69
	5	11	8	67	-65
	5	11	14	92	90
	5	11	18	53	-50
	6	11	-15	53	-48
	6	11	-13	66	-59
	6	11	-11	104	102
	6	11	-9	56	57
	6	11	-5	52	-44
	6	11	-3	89	-85
	6	11	-1	109	106
	6	11	1	94	92
	6	11	3	55	-57
	6	11	5	39	-36
	6	11	7	62	-64
	6	11	9	60	56
	6	11	11	72	81
	7	11	-14	79	76
	7	11	-8	85	-81
	7	11	-6	69	-66
	7	11	-4	112	108
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	7	11	4	75	-76
	7	11	6	63	82
	7	11	14	53	-54
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	15	11	0	49	46
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N	0	12	1	-	78
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	1	12	-10	69	67
	1	12	-9	57	-57
	1	12	-6	95	-100
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	1	12	4	84	-89
	1	12	5	24	25
	1	12	6	70	-75
	1	12	8	64	67
	1	12	9	37	31
	1	12	10	61	57
	1	12	11	44	-43
	1	12	14	53	-46
	1	12	15	41	43
	1	12	16	41	-42
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	2	12	-6	59	59
	2	12	-5	37	41
	2	12	-3	77	74
	2	12	-2	60	-58
	2	12	-1	37	37
	2	12	0	36	37
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	2	12	3	45	-45
	2	12	5	44	41
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	3	12	3	48	-47
	3	12	4	60	62
	3	12	5	41	-38
	3	12	8	58	-54
	4	12	-10	49	-42
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	4	12	8	50	50
	4	12	10	64	-56
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	2	13	13	65	-66
	2	13	15	37	39
	3	13	-12	61	-61
	3	13	-6	36	35
	3	13	-5	47	45
	3	13	-3	44	-46
	3	13	-2	63	-83
	3	13	0	51	-54
	3	13	2	54	50
	3	13	4	46	44
	3	13	6	45	45
	3	13	8	74	-75
	3	13	12	52	54
	4	13	-15	51	-50
	4	13	-9	78	74
	4	13	-5	100	-100
	4	13	-3	36	-31
	4	13	1	76	77
	4	13	3	49	52
	4	13	5	96	-102
	4	13	7	40	-37
	4	13	13	48	42
	4	13	15	67	-63
	5	13	-12	86	87
	5	13	-8	61	-64
	5	13	-6	43	-39
	5	13	-3	47	42
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	5	13	4	46	-47
	5	13	8	83	88
	6	13	-15	68	70
	6	13	-9	78	-77
	6	13	-1	50	-44
	6	13	1	64	-63
	6	13	5	91	96
	7	13	-12	77	-76
	7	13	-8	59	60
	7	13	-2	83	-83
	7	13	2	71	73
	7	13	8	52	-55
	8	13	-9	45	37
	8	13	-5	69	-69
	8	13	5	53	-56
	9	13	-2	42	43

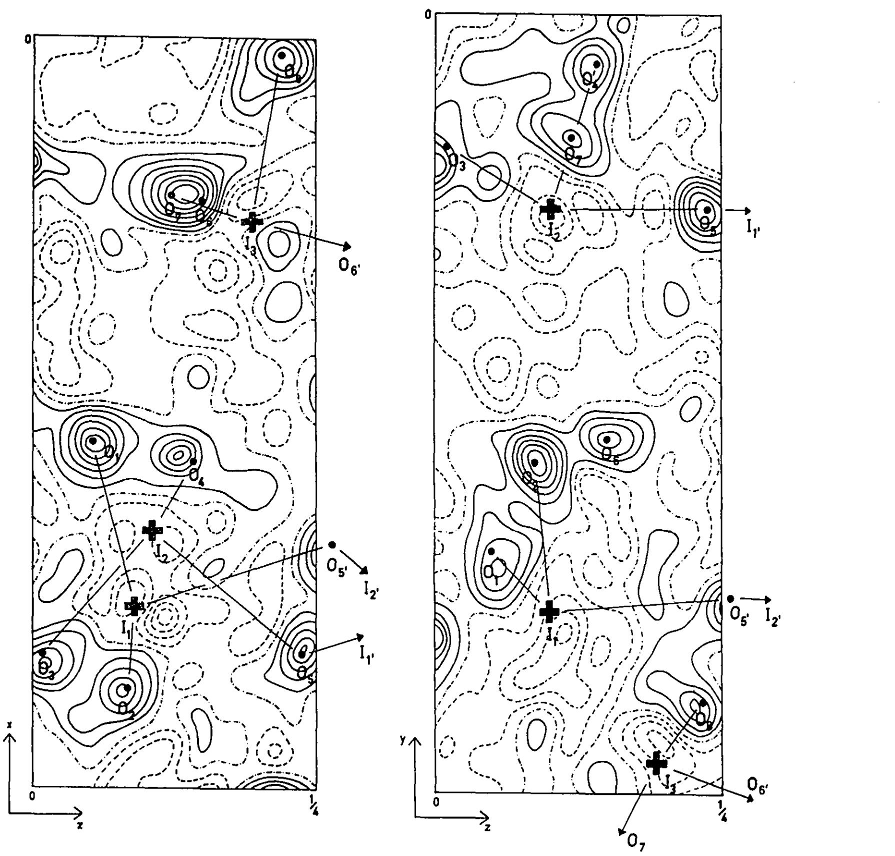


Fig. 1. $F_o - F_{cI}$ syntheses of the [010] and [100] projections after isotropic refinement of the iodine parameters. Contours are at equal intervals of arbitrary scale; dotted lines indicate negative areas.

Table 5. *Estimated standard deviations*

	s.d.		s.d.
I–O	0·01 Å	B(O)	0·17 Å^2
I–O–I	0·6°	U_{ii}(I)	0·0002
O–I–O	0·5°	U_{ij}(I)	0·0003

the cell dimensions may cause a systematic error of approximately 0·01 Å in the I–O bond lengths. It may be noted that owing to the exceedingly small thermal motion of the atoms in HI_3O_8 and the large number of reflexions recorded with molybdenum radiation, the present three-dimensional X-ray study has yielded more accurate I–O bond lengths than the two-dimensional neutron diffraction study of H_5IO_6 (Feikema, 1966), in spite of the relatively small scattering power of oxygen atoms for X-rays.

Description of the structure

The HIO_3 *and* I_2O_5 *groups*

The present investigation confirms that HI_3O_8 is an addition compound of HIO_3 and I_2O_5. In Fig. 2, which shows the structure in [100] projection, the units HIO_3 and I_2O_5 are easily recognized. The bond lengths and valence angles are shown in Fig. 3 and Table 6 respectively. In agreement with the infrared work by Duval & Lecomte (1960) the units I_2O_5 consist of two IO_3 pyramids which have one oxygen atom in common: $O_2I–O–IO_2$. The relative orientation of the pyramids is such that the oxygen atoms O(1), O(2), O(3) and O(4) are at the same side of the plane through I(1), O(5) and I(2). Inspection of Fig. 2 shows that the distance to this plane is much smaller for O(1) and O(3) than for O(2) and O(4). This implies that the group I_2O_5 in HI_3O_8 does not have the symmetry C_{2v} deduced from the infrared spectrum of solid I_2O_5 by Duval & Lecomte (1960). In HI_3O_8 the orientation of the pyramids as well as the values of the angles at iodine may be affected by the strong intermolecular interaction to be described in the next section. The angles O–I–O(5) which are expected to be equal in a free I_2O_5 group, are 97·3 and 88·1° respectively at I(1), whereas at I(2) equal values of 93·3° have been observed.

Table 6. *Valence angles*

O(5)–I(1)–O(2)	97·3°	I(1) –O(5)–I(2)	125·8°
O(5)–I(1)–O(1)	88·1	O(6)–I(3) –O(7)	95·4
O(2)–I(1)–O(1)	98·1	O(6)–I(3) –O(8)	94·2
O(5)–I(2)–O(3)	93·3	O(7)–I(3) –O(8)	99·0
O(5)–I(2)–O(4)	93·3	I(1) –O(2) · · · O(6)	120·1
O(3)–I(2)–O(4)	96·0	I(3) –O(6) · · · O(2)	117·1

The large value of the angle I(1)–O(5)–I(2) may be due to repulsion of the iodine atoms, the distance I(1) · · · I(2) being only 3·49 Å, *i.e.* 0·75 Å shorter than the van der Waals distance.

The bond lengths and angles will be discussed further in the next section.

The three-dimensional network

Most of the I · · · O distances indicated by dotted lines in Fig. 2 are approximately 2·6 Å, *i.e.* about 1 Å shorter than the sum of the van der Waals radii, 3·55 Å. Just as in the alkali iodates the iodine atoms, I(1) and I(2), of the I_2O_5 groups are pseudo-octahedrally surrounded by six oxygen atoms at short distances. Opposite to three oxygen atoms of the I_2O_5 group each iodine atom has three oxygen atoms of neighbouring groups at larger distances. The distances and angles in the pseudo-octahedra are listed in Table 7. The coordination of the iodine atom I(3) of the HIO_3 group is somewhat different. Here one corner of the octahedron is shared by two oxygen atoms, O(6) and O(7), which are at a distance of approximately 3·15 Å from I(3).

Owing to the strong intermolecular interaction between neighbouring HIO_3 and I_2O_5 groups a firm three-dimensional network is formed. Fig. 2 shows the network in approximately half the unit cell in the *x* direction; the *x* coordinates of the iodine atoms shown are between 1·9 and 2·5 Å. The next layer of atoms is generated by the diagonal glide plane (for O(8) all equivalent positions in the unit cell are drawn). The strong three-dimensional network accounts for the small thermal motion of the atoms.

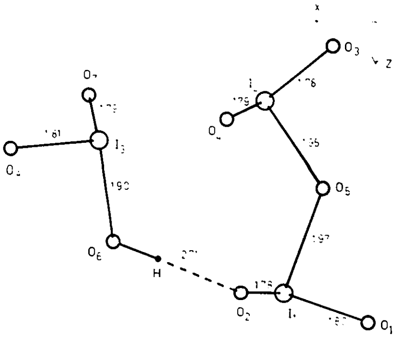


Fig. 3. Molecule of HI_3O_8 with bond lengths (Å).

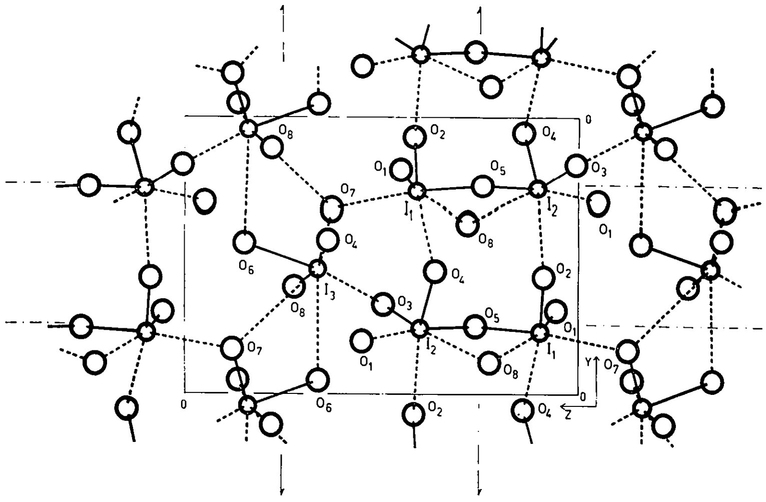


Fig. 2. The structure viewed along the *a* axis.

The arrangement of the IO_3 groups in HI_3O_8 shown in Fig. 4 may be compared with the arrangement of the HIO_3 molecules in α-iodic acid illustrated in Fig. 5. The lower parts of both figures show that all iodine atoms and two of the oxygen atoms of each IO_3 group are almost located in a plane perpendicular to this part of the figure. In HI_3O_8 the IO_3 groups are, however, at larger distances from each other in the y direction than in HIO_3. The translation $\frac{1}{2}b$ along the screw axis is 3·84 Å for HI_3O_8 and only 2·944 Å for α-iodic acid.

Location of the hydrogen atom

Although no direct information on the position of the hydrogen atom is available, the bond lengths in Fig. 3 indicate that the hydrogen atom is linked to O(6) of the HIO_3 group. To obtain further information on the position of this atom the distances of O(6) to the oxygen atoms of neighbouring molecules were calculated. As the observed value is shortest for O(6) · · · O(2) it is tentatively assumed that O(6) of the HIO_3 group is hydrogen bonded to O(2) of the I_2O_5 group (Fig. 3). The distance O(6) · · · O(2) is 2·71 Å, the angles I(3)–O(6) · · · O(2) and O(6) · · · O(2)–I(1) are 117 and 120° respectively. The assumed hydrogen bond is shorter than the hydrogen bond O(6)–H · · · O(7) (2·99 Å) which would be formed if the hydrogen atom were located between neighbouring HIO_3 molecules such as shown for α-iodic acid in Fig. 5 [hydrogen bond O(2)–H · · · O(1)].

Table 7. *Distances and angles in the coordination polyhedra*

		Distances and angles at I(1)			
I(1)–O(1)	1·80 Å	O(1)–I(1)–O(2)	98·1°	O(5)–I(1) · · · O(8)	72·7°
I(1)–O(2)	1·78	O(1)–I(1)–O(5)	88·1	O(4) · · · I(1) · · · O(7)	94·5
I(1)–O(5)	1·97	O(2)–I(1)–O(5)	97·3	O(4) · · · I(1) · · · O(8)	92·8
I(1) · · · O(4)	2·71	O(1)–I(1) · · · O(4)	78·6	O(7) · · · I(1) · · · O(8)	112·6
I(1) · · · O(7)	2·58	O(1)–I(1) · · · O(7)	85·6		
I(1) · · · O(8)	2·62	O(2)–I(1) · · · O(7)	93·8	O(1)–I(1) · · · O(7)	160·6
		O(2)–I(1) · · · O(8)	87·6	O(2)–I(1) · · · O(4)	170·8
		O(5)–I(1) · · · O(4)	74·1	O(5)–I(1) · · · O(7)	167·9
		Distances and angles at I(2)			
I(2)–O(3)	1·78 Å	O(3)–I(2)–O(4)	96·0°	O(5)–I(2) · · · O(8)	74·4°
I(2)–O(4)	1·79	O(3)–I(2)–O(5)	93·3	O(1) · · · I(2) · · · O(2)	102·0
I(2)–O(5)	1·95	O(4)–I(2)–O(5)	93·3	O(1) · · · I(2) · · · O(8)	100·3
I(2) · · · O(1)	2·38	O(3)–I(2) · · · O(1)	92·0	O(2) · · · I(2) · · · O(8)	92·7
I(2) · · · O(2)	2·83	O(3)–I(2) · · · O(2)	83·4		
I(2) · · · O(8)	2·56	O(4)–I(2) · · · O(1)	89·5	O(3)–I(2) · · · O(8)	167·6
		O(4)–I(2) · · · O(8)	83·5	O(4)–I(2) · · · O(2)	168·6
		O(5)–I(2) · · · O(2)	75·3	O(5)–I(2) · · · O(1)	173·7
		Distances and angles at I(3)			
I(3)–O(6)	1·90 Å	O(6)–I(3)–O(7)	95·4°	O(3) · · · I(3) · · · O(4)	96·1°
I(3)–O(7)	1·79	O(6)–I(3)–O(8)	94·2	O(3) · · · I(3) · · · O(6)	58·9
I(3)–O(8)	1·81	O(7)–I(3)–O(8)	99·0	O(3) · · · I(3) · · · O(7)	110·4
I(3) · · · O(3)	2·54	O(6)–I(3) · · · O(4)	86·6	O(4) · · · I(3) · · · O(6)	110·6
I(3) · · · O(4)	2·59	O(6)–I(3) · · · O(6)	119·8	O(4) · · · I(3) · · · O(7)	104·9
I(3) · · · O(6)	3·17	O(6)–I(3) · · · O(7)	68·4	O(6) · · · I(3) · · · O(7)	51·5
I(3) · · · O(7)	3·11	O(7)–I(3) · · · O(3)	85·6		
		O(7)–I(3) · · · O(4)	78·6	O(6)–I(3) · · · O(3)	177·3
		O(8)–I(3) · · · O(3)	83·2	O(7)–I(3) · · · O(6)	143·6
		O(8)–I(3) · · · O(6)	71·0	O(7)–I(3) · · · O(7)	162·8
		O(8)–I(3) · · · O(7)	77·5	O(8)–I(3) · · · O(4)	177·6

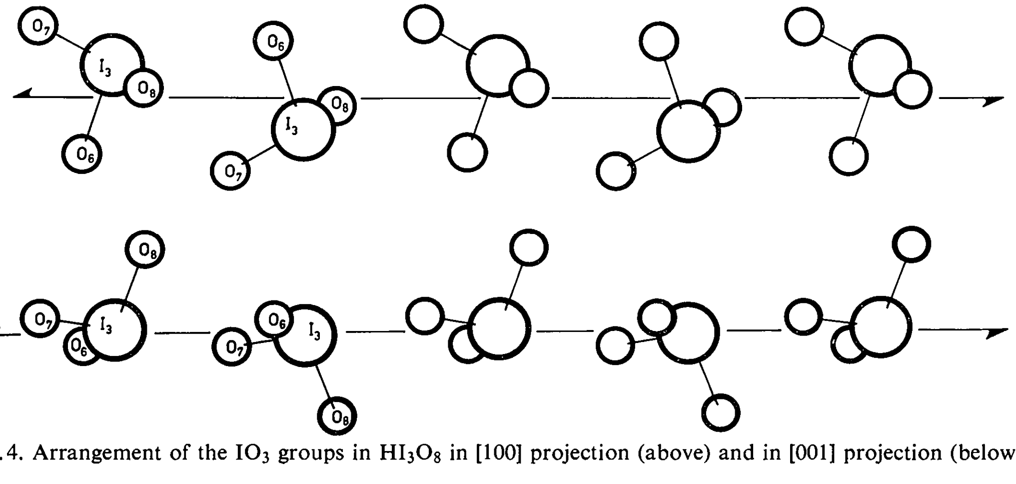
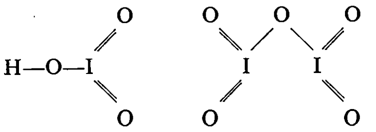


Fig. 4. Arrangement of the IO_3 groups in HI_3O_8 in [100] projection (above) and in [001] projection (below).

Discussion of the oxy-acids of iodine with known crystal structure

Comparison of the I–O *bond lengths*

The lengths of the I–O bonds in the oxy-acids of iodine are listed in Table 8. The short bonds in the last column of the table correspond to the double bonds in the structural formulae



and $O{=}I(OH)_5$

for HIO_3, I_2O_5 and H_5IO_6 respectively. It may be seen that the lengths of the 'double' bonds are equal within experimental error. The 'single' bonds in I_2O_5 are only slightly shorter than the value 1·98 Å estimated for the length of a single I–O bond from the sum of the covalent radii and the difference in electronegativity of oxygen and iodine (Pauling, 1960, p.229). Lengths which are approximately 0·1 Å shorter than the value 1·98 Å are found for the bonds I–OH.

Table 8. I–O *bond lengths* (Å) *in the oxy-acids of iodine with known crystal structure*

Group	Compound	I–O 'single'	I–O 'in I–OH'	I–O 'double'
HIO_3	α-HIO_3		1·90	1·80
HIO_3	HI_3O_8		1·90	1·80
I_2O_5	HI_3O_8	1·96		1·79
H_5IO_6	H_5IO_6		1·89	1·78

The coordination of the iodine atoms

The configurations of the HIO_3 groups in HI_3O_8 and in α-iodic acid show small differences only. Table 8 has illustrated that the corresponding bond lengths are equal within experimental error. In both cases a pyramidal configuration is observed with angles O–I–O of 94, 98 and 101° for α-iodic acid and 94, 95 and 99° for HIO_3 in HI_3O_8.

In all oxy-iodic acids investigated the iodine atoms have a tendency to be surrounded by six oxygen atoms. In the iodine(VII) acid H_5IO_6 the six oxygen atoms are directly linked to the iodine atom and give a slightly irregular octahedron in which the angles O–I–O range from 87 to 95°. Apart from the iodine atom of the HIO_3 group in HI_3O_8 which is surrounded by seven oxygen atoms, the iodine atoms of the iodine(V) acids HI_3O_8 and α-HIO_3 are pseudo-octahedrally coordinated to six oxygen atoms. Opposite to three short I–O distances, approximately 1·9 Å, three longer I–O distances, approximately 2·6 Å in HI_3O_8, are observed. A similar pseudo-octahedral coordination has also been found in several iodates ($NaIO_3$, MacGillavry & Panthaleon van Eck, 1943; $Ce(IO_3)_4$, Cromer & Larson, 1956; $Ce(IO_3)_4{\cdot}H_2O$, Ibers, 1956; Ibers & Cromer, 1958). $LiIO_3$, for which six equal I–O distances are reported (Zachariasen & Basta, 1931) has been investigated rather early, and an attempt will be made to determine its crystal structure more accurately. Table 7 shows that in HI_3O_8 the O–I–O angles in the pseudo-octahedra around I(1) and I(2) show quite large deviations from 90 and 180°, the values range from 74 to 113° and from 160 to 174° respectively.

Description of the bonds

In the previous paper (Feikema, 1966) a covalent description of the bonds in H_5IO_6 has been given: $(HO)_5I{=}O$. In an analogous way the bonds in the iodine(V) oxy-compounds, *e.g.* HIO_3, may be described as covalent. In HIO_3 five outer *p* electrons of iodine are available for the formation of one single bond with OH and two coordinate bonds with the remaining oxygen atoms so as to form $HO{-}I^{2+}({-}O^{-})_2$. The pyramidal configuration at the iodine atom is easily

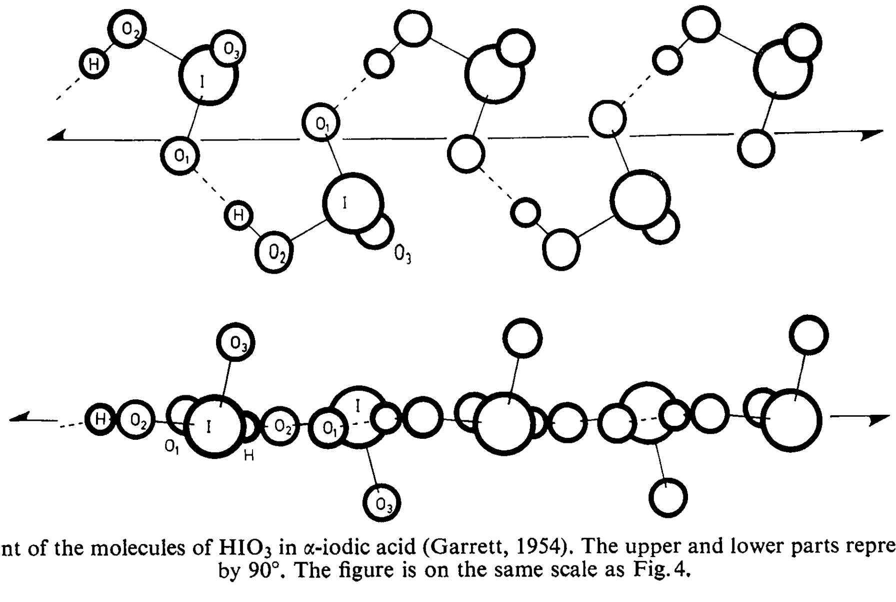
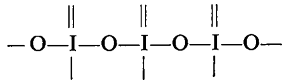


Fig. 5. Arrangement of the molecules of HIO_3 in α-iodic acid (Garrett, 1954). The upper and lower parts represent views differing by 90°. The figure is on the same scale as Fig. 4.

explained from the location of the three iodine orbitals used for the bonding: 5*p*, possibly mixed with some 5*s*. Just as in H_5IO_6 the short lengths of the coordinate bonds indicate, however, a substantial amount of π-bonding due to partial occupation of the iodine 5*d* orbitals by lone pairs of the O^- ions. HIO_3 and I_2O_5 are therefore represented best by the formulae with the double bonds shown before. It should be noted that corresponding bonds in HI_3O_8 and in H_5IO_6 (Table 8) have equal lengths, although different orbitals are assumed to be used for the σ bonds in the iodine(V) and iodine(VII) compounds.

In addition to the covalent character mentioned above, the I–O bonds will have some ionic character, the electronegativities of oxygen and iodine being 3·5 and 2·5 respectively (Pauling, 1960, p.90). The short intermolecular I · · · O distances have not been explained yet. Contributions of covalent structures like



$$-O-\overset{\|}{\underset{|}{I}}-O-\overset{\|}{\underset{|}{I}}-O-\overset{\|}{\underset{|}{I}}-O-$$

may play a part, in addition to dipole interaction, polarization effects and London dispersion forces.

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The Crystal Structure of Methyldicyanoarsine

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Methyldicyanoarsine, $CH_3As(CN)_2$, is monoclinic, space group $P2_1/c$, $a=6{\cdot}28$, $b=8{\cdot}19$, $c=11{\cdot}73$ Å, $\beta=121{\cdot}9°$, with four molecules in the unit cell. The molecules have approximate C_m symmetry with angles: As–C(1)–N(1) 177, As–C(2)–N(2) 173, C(1)–As–C(2) 89·4, C(2)–As–C(3) 93·5, C(3)–As–C(1) 98·3°, and bond lengths: As–C(1) 1·97, As–C(2) 1·99, As–C(3) 2·00, C(1)–N(1) 1·09, C(2)–N(2) 1·15 Å. An intermolecular interaction is apparent in a 2·94 Å As–N distance, which is considerably below the expected van der Waals distance of 3·5 Å. This distance is intermediate between the corresponding short distances observed in $As(CN)_3$, 2·74 Å and $(CH_3)_2AsCN$, 3·18 Å. A further refinement of the structure of $As(CN)_3$ (Emerson & Britton, 1963) is also reported.

In both cacodyl cyanide, $(CH_3)_2AsCN$ (Camerman & Trotter, 1963), and arsenic tricyanide, As $(CN)_3$, (Emerson & Britton, 1963. See also the Appendix to this paper), intermolecular As–N interactions are a recognizable feature of the crystal structure. However, the As–N distance in $As(CN)_3$ is significantly shorter than in $(CH_3)_2AsCN$. We report here the crystal structure of $CH_3As(CN)_2$, which has been determined to see whether the shortening represents a real trend. A comparison of the melting points of $CH_3As(CN)_2$, 126–