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THE CRYSTAL STRUCTURE OF TWO NEW OXYAMINE SALTS "DOACl & DOABr"

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UNITED STATES AIR FORCE
EDWARDS, CALIFORNIA

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and

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FOREWORD

The synthesis of new propellant ingredients to obtain desirable chemical and physical characteristics is a continuing effort at this laboratory. Molecular structure is fundamental to the nature of any substance and X-ray diffraction analysis provides a rapid and unequivocal method of determining this structure in many cases. Knowing the precise structure of a new compound enables one to predict the feasibility of synthesis of homologs and an estimate of their stability.

This report describes the determination of the crystal and molecular structure of two salts of a new potential propellant material.

Reviewed and approved for publication by:

W. S. ANDERSON, Chief
Chemical & Materials Branch
Propellant Division

[redacted]

CONFIDENTIAL ABSTRACT

The crystal and molecular structures of two new oxyamine salts, "DOACl and DOABr" have been determined by X-ray diffraction methods. Bond lengths and angles are all normal suggesting that the analogous tris and tetrakis compounds can be made.

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SECTION I

INTRODUCTION

C - methylene bis(oxyamine hydrochloride), "DOACL" and methylene bis(oxyamine hydrobromide), "DOABr" were prepared at AFRPL under an in-house exploratory synthesis program. Details of the synthesis will be reported at a later date. Structural studies of the perchlorate salt are now in progress and the results of those stuides will be presented when complete.

SECTION II

EXPERIMENTAL

Clear colorless needle-shaped crystals of DOACL & DOABR were grown by slow evaporation of a water solution.

The crystals were mounted on glass fibers parallel to their needle axis (a_o). X-ray precision photographs established in the crystal symmetry of both crystals as monoclinic. Crystal data are:

	DOABR	DOAC1
ao	4.051 Å ±.008	3.941 ±.002
bo	12.25 ±.01	11.885 ±.010
co	12.94 ±.01	12.486 ±.010
β	90°40' ± 20'	90.48° ±.05°
pobs	not measured	1.66 g/cm^3
ρX-ray	2.48 g/cm^3	1.77 g/cm^3
Z	4	4
Space group	$P2_1/c$ (C^5_{2n})	Pz_1/c (C^5_{2n})
a_o: b_o: c_o	0.3306:1: 1.056	0.3316:1: 1.051

Lattice parameter measurements on DOABR were made from precission photographs taken with unfiltered molybdenum radiation (λ = 0.71069). Initial intensity data was taken with a Weissenberg camera using the multiple film technique with three films per pack and exposures of 23 and 3 hours and tube power at 40 K.V. and 30 M.A.. Initial intensity data were collected using Ni - filtered Cu radiation (λ = 1.54178). Reflection intensities were visually estimated by comparison with a calibrated intensity scale.

Lattice parameter and final intensity data on DOACL were derived from data taken on an automated Picker FACS-1 diffractometer. The lattice parameter data were obtained with a least squares lattice parameter refine program supplied with the FACS-1 system using twelve reflections ($\lambda = 1.54178$). A LiF monochromator was used for both the intensity data collection and the lattice parameter data.

All unique data were collected to 128° 2θ, however, because of an interruption caused by the full circle, data from two otherwise unique quadrants were used. Absorption corrections were made on the final DOACL data and were found to be appreciable even though the crystal was small (0.047 m.m. diameter X 0.62 m.m. long, $\mu = 92.7 cm^{-1}$). Peak intensity was estimated using the $\theta - 2\theta$ scanning method over a two degree range. Background was measured for 10 seconds on each side of the peak and a weighing scheme modeled after that suggested by Stout and Jensen was derived. A reflection was considered to be observed if the counts accumulated during the scan were greater than the estimated background.

SECTION III
SOLUTION AND REFINEMENT OF THE STRUCTURES

The structures were solved using the symbolic addition method of Karle and Karle (1). The DOABR structure was solved first by taking advantage of the short a_o axis and solving the structure in its okl projection. A somewhat equivocal E-map yielded Br atom positions which were introduced into the least squares program ORFLS[2]. Several cycles of least squares varying the 2 independent Bromine Y and Z coordinates reduced the conventional agreement factor R to 26 percent. A Fourier calculation using phases derived from the bromide atom coordinates clearly showed the positions of the carbon, oxygen and nitrogen atoms. With the addition of the other non-hydrogen atoms to the least squares refinement and the inclusion of a scale factor and individual isotropic thermal parameters, the residual fell to 12 percent. Unit weights were used throughout this refinement.

Since the crystallographic similarity between DOACl and DOABr was so striking a least squares refinement calculation was made using the okl data for DOACl and the atom coordinates derived from the DOABr structure. The residual stabilized at 12 percent after two cycles varying the same parameters that had been varied in the DOABr two-dimensional refinement.

The X coordinates for the COACl structure were derived by performing a Sigma-two calculation on the three dimensional DOACl data with all normalized structure factors greater than 1.0.

In addition to the okl reflections whose signs were known from the projected structure, the sign of the 131 $|E| = 2.13$ reflection was arbitrarily set positive thus fixing the three-dimensional origin. From the 72 reflections used as a starting set, 101 additional signs were

determined after three iterations of the data. A three-dimensional E map was computed and from this the relative X coordinates were determined.

Heavy atom coordinates derived from photographic data were used to start the refinement of the diffractometer data on DOACl. Several cycles of least squares varying positional and anisotropic least squares thermal parameters using the program ORFLS (2) brought the residual for observed data to 8 percent. Form factors used for heavy atoms were taken from the International Tables (3). The form factors for hydrogen were taken from the work of Stewart, Davidon and Simpson (4). A difference Fourier was completed and all hydrogen atoms were easily located. Also it was noted that the chlorine form factors used were those for neutral atoms producing a ring-shaped area of positive electron density in the difference Fourier (cf Stout & Jensen) (5). Accordingly, the form factors for Cl^- were introduced along with the hydrogen atoms to the next full matrix refinement. Two additional cycles of refinement brought the residual for non zero observed F's to 6.5 percent. Another difference Fourier was computed showing no anomalies and the refinement was terminated. Thermal parameters for the hydrogen atoms were fixed at the values derived for the attached heavy atoms and no attempt was made to refine that value.

Tables of computed and observed structure factors are presented in Appendix A.

Positional and thermal parameters for DOABr and DOACl are presented in Table I.

TABLE I. POSITIONAL THERMAL PARAMETERS FOR DOABr AND DOACl.

Anistropic thermal parameters are of the form Texptl - $(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + \beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl$ Error terms as determined from the least squares refinement are enclosed in ( ). Atoms from the DOABr molecule are enclosed in square brackets[ ].

Atom	x	y	z	B	β_{11}	β_{22}	β_{33}	$2\beta_{12}$	$2\beta_{13}$	$2\beta_{23}$
Cl 1	-0.3894(4)	0.4555(1)	0.1390(1)		0.0221(9)	0.0052(1)	0.0039(1)	-0.0000(3)	0.0005(2)	0.0001(1)
[Br 1]	-	0.4555(4)	0.1408(4)	1.0(1)						
Cl 2	-0.4514(4)	0.1280(1)	0.0864(1)		0.0240(9)	0.0047(1)	0.0048(1)	0.0018(3)	0.0007(3)	0.0003(1)
[Br 2]	-	0.1286(4)	0.0890(4)	1.2(0.1)						
O 1	0.1938(10)	0.3445(4)	0.3417(4)		0.0154(25)	0.0048(4)	0.0054(3)	-0.0002(8)	0.0018(7)	0.0004(3)
[O 1]	-	0.3454(28)	0.3422(27)	0.9(7)						
O 2	-0.1332(10)	0.1976(4)	0.4069(4)		0.0156(24)	0.0049(4)	0.0048(3)	-0.0008(8)	-0.0002(7)	0.0005(3)
[O 2]	-	0.2008(33)	0.4109(32)	2.0(9)						
N 1	-0.0497(14)	0.4308(5)	0.3620(5)		0.0252(34)	0.0043(5)	0.0049(4)	-0.0004(10)	-0.0004(10)	-0.0004(4)
[N 1]	-	0.4269(37)	0.3648(35)	1.2(10)						
N 2	0.1112(13)	0.1643(5)	0.4866(5)		0.0225(33)	0.0047(5)	0.0046(4)	-0.0002(10)	0.0008(9)	0.0004(4)
[N 2]	-	0.1643(32)	0.4865(30)	0.6(0.8)						
C	0.0337(17)	0.2420(6)	0.3158(6)		0.0347(42)	0.0056(6)	0.0043(5)	-0.0004(13)	-0.0002(11)	-0.0002(4)
[C]	-	0.2413(43)	0.3123(40)	1.3(1.0)						
H 1	0.853 (17)	0.258 (6)	0.255 (5)							
H 2	0.242 (17)	0.185 (6)	0.286 (5)							
H 3	0.820 (17)	0.441 (6)	0.289 (6)							
H 4	0.078 (17)	0.492 (6)	0.385 (5)							
H 5	0.808 (17)	0.411 (6)	0.429 (5)							
H 6	0.257 (17)	0.106 (6)	0.457 (5)							
H 7	0.223 (17)	0.270 (6)	0.012 (5)							
H 8	-0.22 (18)	0.370 (6)	0.037 (6)							

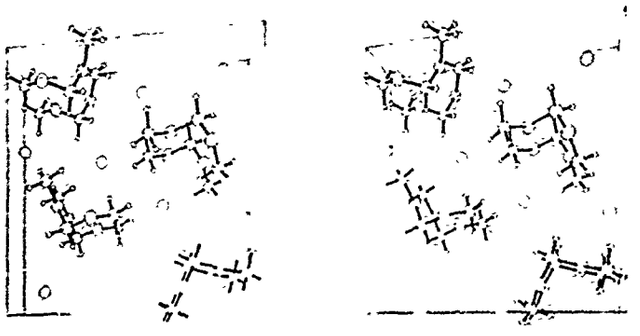

Figure 1. Stereo Diagram of The Unit Cell of DOACl Showing The Molecular Structure. Single Unbonded Balls in The Diagram Are The Cl Ions.

TABLE II. IMPORTANT INTERATOMIC DISTANCES, ANGLES AND THEIR STANDARD ERRORS

Atoms	Distance (Å)	Bond Angles, (deg)
$C-O_1$	1.407(8)	
$C-O_2$	1.418(8)	
$O_1 - N_1$	1.428(6)	
$O_2 - N_2$	1.435(7)	
$Cl_1 - Cl_2$	3.953(4)	
$O_1 - C - O_2$		110.4(5)
$N_1 - O_1 - C$		110.2(4)
$C - H_1$	1.04(5)	
$C - H_2$	1.12(5)	
$H_1 - C - H_2$		110(1)

TABLE III. DISTANCE AND ANGLES EVOLVED IN HYDROGEN BONDING

Atoms	N-Cl(Å)	N-H(Å)	H---Cl(Å)	AN-H. . . Cl(deg)
$N_1 - H_3 . . Cl_1$	3.09	1.05	2.05	173.8 deg
$N_1 - H_4 . . Cl_2$	3.13	0.92	2.23	168.8
$N_1 - H_5 . . Cl_2$	3.13	1.02	2.29	173.4
$N_2 - H_6 . . Cl_1$	3.13	0.98	2.21	157.0
$N_2 - H_7 . . Cl_2$	3.25	0.94	2.31	172.5
$N_2 - H_8 . . CL_1$	3.10	0.88	2.23	173.3

SECTION IV

DISCUSSION

The configuration of the DOACl molecular structure and its relationship to the unit cell chosen, is shown in the stereodiagram Figure I. The stereoplots were made with the program ORTEP (6).

Important interatomic distances and angles and their estimated standard errors were calculated with the program ORFEE (7) and are listed in Tables II and III.

From the inspection of the bond distances and angles about the carbon atom it is apparent that tetrhedral symmetry has been maintained. Further, the DOA molecule has within the limits of error a two-fold axis of symmetry. The difference between the carbon oxygen bonds is 0.011A $\pm$0.012 Å. The difference between the oxygen-nitrogen bonds is 0.007A $\pm$0.009Å. The difference between the two carbon-oxygen-nitrogen bond angles is 0.9 $\pm$ 0.7 .

TABLE IV. BOND DISTANCES FOR SOME COMPOUNDS SIMILAR TO DOACl

Compound	Distance O-C	Distance O-N	Reference
CH_3ONH_3	1.46 ± 0.04	1.42 ± 0.02	Laurent & Rerat (1964)
CH_3ONH_2	1.44 ± 0.02	1.37 ± 0.02	Brockway, Beach & Pauling (1935)
$(CH_3)_3$ NO HCl		1.425 ± 0.011	Caron & Donohue (1966)
$CH_2O_2(NH_3)_2^{++}Cl_2^{--}$	1.418 ± 0.011	1.431 ± 0.009+	This work

As shown in Table IV, the average carbon-oxygen and oxygen-nitrogen bonds for DOACl are in good agreement with distances determined for similarly bonded molecules, and preliminary work on the structure of the DOA perchlorate salt shows similar molecular symmetry and bond distances.

The separation of the chlorine atoms at 3.95 Å is only slightly greater than one would have predicted on the basis of the usual van der Waals radii of 1.80.

Hydrogen bonding between the amine groups and the chlorine ions seems to be in good agreement with other work (See Table V.).

TABLE V. SELECTED HYDROGEN BONDING INFORMATION

Compound	N...Cl(Å)	N-H(Å)	H...Cl(Å)	AN-H...Cl(deg)	Method of Determination	Reference
Methoxyamine hydrochloride	3.10	-	-	-	X-ray	Laurent & Rerat (1964)
	3.17	-	-	-		
Hydroxylammonium chloride	3.202(6)	1.024(11)	2.256(13)	153.2(9)	Neutron	Padmanabhan(1967)
		1.017(9)	2.248(16)	157.3(8)		
		1.019(9)	2.260(14)	156.2(9)		
DOACL (Averages)	3.14	0.97	2.22	170	X-ray	This work

SECTION V

SUMMARY

In summary, the structure shows unequivocally that the synthesis of the bis oxyamine cation was successful and that there is no reason to believe on stereochemical grounds, that the tris and tetrakis oxyamine radicals cannot also be made.

ACKNOWLEDGEMENTS

The authors wish to acknowledge the substantial help they received from Mr. Stephen Rose in programing many of the required calculations for our computers.

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APPENDIX A-1

DOACL observed and computed structure factors.

H	K	L	FOB	FCA
-4	0	-6	60	57
-4	0	-4	44	49
-4	0	-2	349	324
-4	0	0	222	188
-4	0	2	307	285
-4	0	4	176	189
-4	0	6	235	243
-4	1	-6	95	103
-4	1	-5	139	141
-4	1	-4	107	104
-4	1	-3	277	277
-4	1	-2	103	102
-4	1	-1	124	137
-4	1	0	162	142
-4	1	1	323	294
-4	1	2	68	65
-4	1	3	40	35
-4	1	4	187	180
-4	1	5	60	56
-4	1	6	163	171
-4	1	7	106	106
-4	2	-6	72	67
-4	2	-5	206	201
-4	2	-4	155	156
-4	2	-3	145	151
-4	2	-2	0	1
-4	2	-1	118	135
-4	2	0	114	108
-4	2	1	11	14
-4	2	2	184	173
-4	2	3	32	45
-4	2	4	84	95
-4	2	5	88	84
-4	2	6	161	157
-4	3	-6	15	17
-4	3	-5	8	12
-4	3	-4	0	13
-4	3	-3	44	42
-4	3	-2	48	47
-4	3	-1	111	116
-4	3	0	75	85
-4	3	1	225	216
-4	3	2	202	198
-4	3	3	304	308
-4	3	4	88	96
-4	3	5	36	36
-4	3	6	263	246
-4	4	-5	34	38
-4	4	-4	221	213
-4	4	-3	198	196
-4	4	-2	175	179
-4	4	-1	120	119
-4	4	0	122	127
-4	4	1	183	179
-4	4	2	212	204
-4	4	3	92	94
-4	4	4	52	50
-4	4	5	174	164
-4	5	-4	16	24
-4	5	-3	97	99
-4	5	-2	335	337
-4	5	-1	88	96
-4	5	0	32	29
-4	5	1	9	21
-4	5	2	108	103
-4	5	3	107	105
-4	5	4	37	28
-4	6	-3	29	30
-4	6	-2	78	81
-4	6	-1	114	111
-4	6	0	82	74
-4	6	1	246	251
-4	6	2	0	11
-4	6	3	95	89
-3	0	-10	120	121
-3	0	-8	76	84
-3	0	-6	114	125
-3	0	-4	63	76
-3	0	-2	137	132
-3	0	0	206	180
-3	0	2	528	456
-3	0	4	101	106
-3	0	6	272	302
-3	0	8	95	106
-3	0	10	147	154
-3	1	-10	62	62
-3	1	-9	242	257
-3	1	-8	58	70
-3	1	-7	73	81
-3	1	-6	101	112
-3	1	-5	348	389
-3	1	-4	212	214
-3	1	-3	152	156
-3	1	-2	19	7
-3	1	-1	305	298
-3	1	0	318	279
-3	1	1	0	2
-3	1	2	65	66
-3	1	3	136	128
-3	1	4	306	298
-3	1	5	197	210
-3	1	6	181	203
-3	1	7	29	50
-3	1	8	82	94
-3	1	9	309	333
-3	1	10	51	55
-3	1	11	5	12
-3	2	-10	0	3
-3	2	-9	26	29
-3	2	-8	150	155
-3	2	-7	214	235
-3	2	-6	127	143
-3	2	-5	0	6
-3	2	-4	169	178
-3	2	-3	292	308
-3	2	-2	209	216
-3	2	-1	289	281
-3	2	0	239	220
-3	2	1	67	56
-3	2	2	160	150
-3	2	3	184	185
-3	2	4	135	129
-3	2	5	112	125
-3	2	6	158	183
-3	2	7	11	24
-3	2	8	62	70
-3	2	9	64	71
-3	2	10	112	118
-3	3	-10	108	109
-3	3	-9	0	20
-3	3	-8	67	72
-3	3	-7	76	77
-3	3	-6	19	26
-3	3	-5	33	34
-3	3	-4	67	83
-3	3	-3	269	281
-3	3	-2	12	13
-3	3	-1	169	179
-3	3	0	366	356
-3	3	1	200	195
-3	3	2	36	35
-3	3	3	201	196
-3	3	4	404	434
-3	3	5	68	80
-3	3	6	29	32
-3	3	7	223	246
-3	3	8	185	202
-3	3	9	100	108
-3	3	10	6	3
-3	4	-10	108	111
-3	4	-9	130	133
-3	4	-8	141	143
-3	4	-7	118	121
-3	4	-6	52	66
-3	4	-5	197	213
-3	4	-4	323	367
-3	4	-3	92	95
-3	4	-2	0	5
-3	4	-1	0	18
-3	4	0	281	284
-3	4	1	251	252
-3	4	2	47	50
-3	4	3	86	96
-3	4	4	97	104
-3	4	5	149	170
-3	4	6	45	54
-3	4	7	194	206
-3	4	8	215	226
-3	4	9	126	117
-3	4	10	67	72
-3	5	-9	73	68
-3	5	-8	286	286
-3	5	-7	108	115
-3	5	-6	257	282
-3	5	-5	130	143
-3	5	-4	152	169
-3	5	-3	0	17
-3	5	-2	81	97
-3	5	-1	66	81
-3	5	0	65	72

H	K	L	FOB	FCA
-3	5	1	155	171
-3	5	2	39	47
-3	5	3	110	128
-3	5	4	58	59
-3	5	5	83	86
-3	5	6	116	132
-3	5	7	108	112
-3	5	8	126	125
-3	5	9	7	11
-3	6	-8	7	9
-3	6	-7	0	12
-3	6	-6	74	85
-3	6	-5	44	49
-3	6	-4	125	133
-3	6	-3	130	149
-3	6	-2	0	4
-3	6	-1	193	225
-3	6	0	172	178
-3	6	1	324	357
-3	6	2	29	24
-3	6	3	151	170
-3	6	4	11	5
-3	6	5	348	377
-3	6	6	80	86
-3	6	7	22	37
-3	6	8	0	11
-3	6	9	190	177
-3	7	-8	81	77
-3	7	-7	20	27
-3	7	-6	176	179
-3	7	-5	66	66
-3	7	-4	156	168
-3	7	-3	166	187
-3	7	-2	317	360
-3	7	-1	0	2
-3	7	0	92	99
-3	7	1	201	231
-3	7	2	108	112
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-3	7	4	148	152
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-3	8	-7	90	86
-3	8	-6	78	74
-3	8	-5	8	24
-3	8	-4	216	223
-3	8	-3	118	134
-3	8	-2	57	77
-3	8	-1	0	14
-3	8	0	102	114
-3	8	1	0	2
-3	8	2	128	134
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-3	8	4	0	13
-3	8	5	42	42
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-2	9	2	105	116
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-1	0	12	54	54
-1	0	14	173	144
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-1	13	2	131	114
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0	0	10	0	8
0	0	12	118	134
0	0	14	172	162

APPENDIX A-2

DOABR observed and computed structure factors.

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0	1	10	0	23
0	1	11	204	231
0	1	12	0	67
0	1	13	0	34
0	1	14	0	11
0	2	0	20	7
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0	2	4	429	456
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0	2	7	220	217
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0	2	9	425	442
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0	2	11	58	60
0	2	12	153	161
0	2	13	245	228
0	2	14	174	146
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0	3	5	304	308
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0	5	2	101	89
0	5	3	114	110
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0	5	7	95	92
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0	5	9	79	84
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0	6	7	150	149
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0	6	10	47	54
0	6	11	55	60
0	6	12	94	75
0	6	13	106	83
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0	7	2	0	5
0	7	3	215	212
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0	7	5	252	256
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0	7	7	108	108
0	7	8	207	214
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0	7	10	139	131
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0	7	12	140	120
0	8	0	33	28
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2	6	7	35	43
2	6	8	126	126
2	6	9	94	95
2	6	10	118	102
2	6	11	169	155
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2	7	3	138	151
2	7	4	235	257
2	7	5	164	178
2	7	6	200	216
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2	7	8	272	271
2	7	9	76	79
2	7	10	126	108
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2	8	1	75	88
2	8	2	170	191
2	8	3	53	61
2	8	4	144	161
2	8	5	0	3
2	8	6	258	278
2	8	7	122	125
2	8	8	0	11
2	8	9	48	42
2	8	10	218	190
2	9	0	309	343
2	9	1	18	30
2	9	2	172	190
2	9	3	94	106
2	9	4	120	130
2	9	5	65	62
2	9	6	115	110
2	9	7	0	5
2	9	8	0	1
2	9	9	48	39
2	10	0	59	63
2	10	1	163	178
2	10	2	86	89
2	10	3	196	202
2	10	4	17	23
2	10	5	0	14
2	10	6	115	109
2	10	7	143	132
2	11	0	35	36
2	11	1	114	113
2	11	2	66	64
2	11	3	15	29
2	11	4	7	20
2	11	5	250	223
2	11	6	49	46
2	12	0	112	109
2	12	1	0	5
2	12	2	192	171
2	12	3	27	27

H	K	L	FOB	FCA

H	K	L	FOB	FCA
0	0	2	96	72
0	0	4	308	389
0	0	6	23	22
0	0	8	100	86
0	0	10	14	4
0	0	12	78	79
0	0	14	107	103
0	0	16	64	69
0	1	1	10	0
0	1	2	87	80
0	1	3	110	99
0	1	4	150	116
0	1	5	118	86
0	1	6	82	68
0	1	7	224	214
0	1	8	93	93
0	1	9	25	29
0	1	10	28	30
0	1	11	191	172
0	1	12	0	10
0	1	13	0	6
0	1	14	29	42
0	1	15	91	88
0	1	16	34	33
0	2	0	103	97
0	2	1	18	17
0	2	2	66	52
0	2	3	195	172
0	2	4	181	160
0	2	5	247	236
0	2	6	102	86
0	2	7	130	115
0	2	8	123	115
0	2	9	209	231
0	2	10	131	121
0	2	11	31	29
0	2	12	72	61
0	2	13	190	148
0	2	14	122	85
0	2	15	39	43
0	2	16	0	5
0	3	1	174	183
0	3	2	233	286
0	3	3	210	203
0	3	4	56	52
0	3	5	186	158
0	3	6	111	96
0	3	7	41	35
0	3	8	39	43
0	3	9	0	14
0	3	10	29	23
0	3	11	0	9
0	3	12	34	24
0	3	13	98	76
0	3	14	55	47
0	3	15	0	3
0	3	16	59	72
0	4	0	144	137
0	4	1	203	186
0	4	2	144	124
0	4	3	161	153
0	4	4	43	42
0	4	5	152	130
0	4	6	185	168
0	4	7	55	43
0	4	8	62	52
0	4	9	127	109
0	4	10	131	119
0	4	11	0	11
0	4	12	126	117
0	4	13	90	65
0	4	14	28	24
0	4	15	64	60
0	4	16	44	70
0	5	1	180	180
0	5	2	59	50
0	5	3	56	47
0	5	4	175	165
0	5	5	155	124
0	5	6	114	98
0	5	7	46	40
0	5	8	214	226
0	5	9	35	36
0	5	10	148	125
0	5	11	68	59
0	5	12	166	128
0	5	13	38	42
0	5	14	62	51
0	5	15	0	9
0	6	0	29	27
0	6	1	197	202
0	6	2	53	48
0	6	3	209	223
0	6	4	11	22
0	6	5	105	97
0	6	6	0	12
0	6	7	106	106
0	6	8	0	4
0	6	9	31	27
0	6	10	33	21
0	6	11	42	38
0	6	12	30	40
0	6	13	15	19
0	6	14	0	4
0	6	15	80	103
0	7	1	141	156
0	7	2	37	46
0	7	3	85	92
0	7	4	134	144
0	7	5	140	134
0	7	6	77	83
0	7	7	44	45
0	7	8	148	143
0	7	9	16	6
0	7	10	102	88
0	7	11	73	58
0	7	12	105	101
0	7	13	60	58
0	7	14	65	78
0	8	0	22	27
0	8	1	92	103
0	8	2	57	58
0	8	3	25	33
0	8	4	0	12
0	8	5	121	128
0	8	6	171	156
0	8	7	0	9
0	8	8	105	98
0	8	9	117	86
0	8	10	150	129
0	8	11	59	40
0	8	12	97	94
0	8	13	65	63
0	8	14	40	54
0	9	1	106	129
0	9	2	149	187
0	9	3	45	59
0	9	4	75	81
0	9	5	79	79
0	9	6	94	97
0	9	7	53	49
0	9	8	87	56
0	9	9	0	25
0	9	10	0	9
0	9	11	14	17
0	9	12	0	22
0	9	13	43	50
0	10	0	150	172
0	10	1	49	51
0	10	2	30	22
0	10	3	96	101
0	10	4	109	116
0	10	5	53	53
0	10	6	69	74
0	10	7	85	69
0	10	8	63	62
0	10	9	126	116
0	10	10	73	63
0	10	11	0	9
0	10	12	0	8
0	11	1	0	14
0	11	2	84	81
0	11	3	96	98
0	11	4	0	15
0	11	5	57	54
0	11	6	0	16
0	11	7	163	152
0	11	8	0	10
0	11	9	34	34
0	11	10	25	14
0	11	11	112	138
0	12	0	153	176
0	12	1	0	16
0	12	2	34	24
0	12	3	0	3
0	12	4	148	152
0	12	5	32	41
0	12	6	68	65
0	12	7	0	9
0	12	8	27	32

H	K	L	FOB	FCA
0	12	9	44	45
0	12	10	0	6
0	13	1	0	26
0	13	2	117	115
0	13	3	86	90
0	13	4	37	36
0	13	5	0	2
0	13	6	33	38
0	13	7	109	106
0	13	8	20	29
0	13	9	0	25
0	14	0	28	28
0	14	1	0	2
0	14	2	0	22
0	14	3	54	57
0	14	4	0	23
0	14	5	59	69
0	14	6	53	54
0	14	7	40	50
0	15	1	71	89
0	15	2	92	104
0	15	3	29	36
0	15	4	0	3
0	15	5	27	54

H	K	L	FOB	FCA

H	K	L	FOB	FCA

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13. ABSTRACT

The crystal and molecular structures of two new oxyamine salts, "DOACl and DOABr" have been determined by x-ray diffraction methods. Bond lengths and angles are all normal suggesting that the analogous tris and tetrakis compounds can be made.

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