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Metal Complexes with Mixed Ligands

8. The Crystal Structure of Diperchlorato-tetraimidazolo-copper(II); $Cu(C_3H_4N_2)_4(ClO_4)_2$

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The crystal structure of diperchlorato-tetraimidazolo-copper(II) has been determined using three-dimensional X-ray diffraction data. The crystals are monoclinic, space group $P2_1/n$, with two formula units in the unit cell. The cell dimensions with corresponding standard deviations are $a = 8.198 \pm 0.001$ Å, $b = 16.293 \pm 0.002$ Å, $c = 9.353 \pm 0.002$ Å and $\beta = 125.81 \pm 0.01°$. The intensities were collected and measured with a linear diffractometer and Mo$K\alpha$-radiation. The structure was solved by routine heavy-atom methods and refined by a full-matrix least squares method with anisotropic temperature factors for all nonhydrogen atoms to a final R-value of 0.060. The structure is built up of uncharged octahedral complexes $Cu(C_3H_4N_2)_4(ClO_4)_2$. The complexes are held together through hydrogen and van der Waals bonds. The distances between copper and imidazole nitrogens are 1.997 Å and 2.007 Å and the two distances between copper and perchlorate oxygens are 2.625 Å. The hydrogen atoms were located from a difference Fourier map.

The present investigation forms part of an X-ray crystal structure study of crystalline phases obtained from hydrolyzed aqueous $Cu^{2+} - C_3H_4N_2 - ClO_4^-$-solutions. Hitherto the phases $Cu_3(C_3H_3N_2)_2(C_3H_4N_2)_8(ClO_4)_4$ [1] and $Cu(C_3H_4N_2)_4(ClO_4)_2$ have been prepared and ivestigated by X-ray methods. The crystal structure of the former was presented in part 2 of this series and the crystal structure of the latter will be presented and discussed in the present paper. The equilibrium investigations by Sjöberg [2] provide the solution chemistry background to the present crystal structure study.

EXPERIMENTAL

Crystal preparation and analyses. The crystals used were prepared by adding 5.8 ml of a solution with the composition: $[Na^+] = 3.495$ M; $[ClO_4^-] = 3.000$ M; $[OH^-] = 0.495$ M to 5.6 ml of a solution with the composition $[Na^+] = 2.430$ M; $[ClO_4^-] = 3.000$ M; $[Cu^{2+}] =$

0.034 M; $[H^+] = 0.357$ M and $[C_3H_5N_2^+] = 0.179$ M. After mixing welldefined blue-violet prismatic crystals were formed within a few days.

The copper content of the crystals was determined electrolytically and the nitrogen content was determined using the Kjeldahl method. (Found: Cu, 11.47; N, 20.7. Calc. for $Cu(C_3H_4N_2)_4(ClO_4)_2$: Cu, 11.88; N, 21.0.)

Crystal data and space group. From rotation photographs around the *c*- and *b*-axis and the corresponding Weissenberg photographs (zero, first, and second layer lines) taken with Cu$K\alpha$-radiation it was concluded that the crystals were monoclinic. The cell dimensions were refined from a Guinier photograph giving 67 lines. The following parameters and their corresponding standard deviations were obtained: $a = 8.198 \pm 0.001$ Å, $b = 16.293 \pm 0.002$ Å, $c = 9.353 \pm 0.002$ Å, $\beta = 125.81 \pm 0.01°$, $V = 1011.79$ Å^3.

By the flotation method (using bromoform and acetone) the density was determined to be 1.75 g/cm^3. With two of the above mentioned formula units in the cell the calculated density is 1.755 g/cm^3. Systematic extinctions were found for $h0l$, $h+l = 2n+1$ and $0k0$, $k = 2n+1$. This is characteristic for the space group $P2_1/n$ (a transformation of $P2_1/c$, No. 14 in International Tables).[3]

Collection and reduction of intensity data. Single-crystal intensity data were collected and measured with an automatic linear diffractometer (PAILRED). The radiation was Mo$K\alpha$ with a LiF monochromator. The crystal was rotated along the *c*-axis and intensities for $hk0 - hk8$ were measured ($\sin\theta_{MoK\alpha} \leq 0.54$). The half scan intervals were for $hk0 - hk3$ equal to 0.6° for $\theta > 20°$ and equal to 1.2° for $\theta < 20°$. Corresponding values for $hk4 - hk8$ were 1.0° and 2.0°. The scan speed used was 0.5°/min. Weak reflexions (counts less than 1000) were measured up to three times. Background radiation was measured during 20 sec intervals on each side of the reflexion.

The intensities of approixmately 3400 reflexions were measured. Reflexions with a relative counting statistical error of $\Delta I/I > 1.0$ were omitted, which left 1612 observed intensities. The intensities were corrected for Lorentz and polarization factors and absorption. The linear absorption coefficient was calculated to be 14.45 cm^{-1}. The absorption correction gave differences in the transmission between 0.81 – 0.93.

The computer programs used were the same as in a paper previously published by Ivarsson *et al.*[1]

STRUCTURE DETERMINATION AND REFINEMENT

The copper, chlorine, and oxygen atoms were located from a three dimensional Patterson synthesis and the other nonhydrogen atoms were found by standard Fourier methods.

The structure was refined by full-matrix least squares techniques. The weighting scheme of Cruickshank [4] was applied to the observed reflexions. The atomic scattering factors for Cu^{2+}, Cl^-, O, N, C, and H were used.[5] With individual isotropic temperature factors the refinement converged to a conventional *R*-value of 0.146; the hydrogen atoms were not included. Anisotropic temperature factors were then applied to all nonhydrogen atoms and the *R*-value dropped to 0.065. At this stage of the refinement a difference Fourier map was made in order to try to find the hydrogen positions. The maxima that were found could be explained by hydrogen atoms. Subsequent least squares cycles with these hydrogen atom positions included also reduced the *R*-value and a final *R*-value of 0.060 was obtained. The decreace in *R*-value was significant on the 0.1 %-level according to the Hamilton test as approximated by Pawley.[6] All parameter shifts in the final cycle were less than 10 % of the estimated standard deviation. A final difference Fourier synthesis was calculated in which no abnormalities could be detected. The final atomic coordinates and vibrational parameters are given in Tables 1 and 2. A comparison between the observed and calculated structure factors is given in Table 3.

Table 1. The final atomic positional fractional coordinates and vibrational parameters and their estimated standard deviations (σ in parenthesis). All values multiplied by 10^4. Anisotropic temperature factors have been calculated according to the formula exp $[-(h^2B_{11}+k^2B_{22}+l^2B_{33}+hkB_{12}+hlB_{13}+klB_{23})]$.

	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Cu	0	0	0	145 (2)	19(0)	131 (2)	−16 (1)	168 (3)	13 (1)
Cl	1186 (2)	1176(1)	−2843 (2)	221 (3)	31(0)	197 (4)	37 (2)	312 (6)	31 (2)
O1	−50 (7)	659(3)	−2587 (6)	249(11)	33(2)	225(11)	11 (7)	314(19)	−41 (7)
O2	872(11)	987(4)	−4458 (8)	595(26)	78(4)	243(15)	76(16)	586(33)	43(11)
O3	3217(10)	1022(7)	−1481(11)	236(17)	189(9)	398(22)	88(19)	331(32)	18(22)
O4	673(16)	1988(3)	−2833(16)	1266(50)	25(2)	1225(49)	35(17)	2334(95)	21(16)
N1	2790 (6)	410(3)	1850 (6)	158(10)	25(1)	133 (9)	−25 (6)	172(16)	−5 (6)
N2	6037 (8)	426(3)	3861 (8)	171(12)	42(2)	224(13)	7 (9)	167(21)	13 (9)
N3	−1170 (7)	1022(2)	238 (6)	175(10)	23(2)	152(10)	−5 (6)	210(17)	13 (6)
N4	−2540 (9)	1870(3)	1037 (9)	325(17)	33(2)	265(15)	−52 (9)	453(28)	19 (8)
C1	4441(10)	−37(4)	2748(11)	231(16)	34(2)	268(17)	−13(11)	313(27)	−12(11)
C2	5431(10)	1212(5)	3684(10)	207(16)	45(3)	216(17)	38(12)	144(26)	35(11)
C3	3417(11)	1191(4)	2433(11)	249(17)	26(2)	255(17)	−5(10)	166(28)	37(10)
C4	−1806(11)	1122(4)	1228 (9)	304(18)	33(2)	188(15)	−38(11)	355(28)	−6 (9)
C5	−2353(11)	2282(4)	−115(11)	340(20)	29(2)	267(18)	−76(11)	445(34)	−30(10)
C6	−1523(11)	1756(4)	−617(10)	344(20)	28(2)	229(16)	−55(11)	435(32)	−32 (9)

Table 2. The final fractional coordinates and their estimated standard deviation for the hydrogen atoms. (All values multiplied by 10^3). The hydrogen atoms were given an isotropic thermal parameter of 5.0 which was not refined.

	x	y	z
H_1	448(10)	−61(4)	268 (9)
H_2	735(10)	25(4)	477(10)
H_3	652(10)	160(4)	448 (9)
H_4	258(10)	159(4)	202 (9)
H_5	−192(10)	57(4)	201 (9)
H_6	−302(10)	209(4)	165 (9)
H_7	−300(10)	285(4)	−46 (9)
H_8	−128(10)	181(4)	−134 (9)

DESCRIPTION AND DISCUSSION OF THE STRUCTURE

The structure is built up of uncharged octahedral complexes $Cu(C_3H_4N_2)_4(ClO_4)_2$. The complexes are held together through hydrogen and van der Waals bonds. A drawing of the complex is shown in Fig. 1 and in Fig. 2 the packing of the complexes is illustrated.

The $Cu(C_3H_4N_2)_4(ClO_4)_2$ complex. In the complex the four imidazole nitrogens and the Cu^{2+}-ion are on the basis of symmetry, placed in the same plane and form a slightly distorted plane quadratic nitrogen coordination around Cu^{2+}. The Cu−N distances (2.010 Å and 1.998 Å) are quite normal

Table 3. Observed and calculated structure factors (× 10). Values marked with an asterisk were not included in the final refinement.

H	K		
L=	0		
1	21	71	46
2	20	128	115
1	19	134	125
3	18	104	107
2	18	115	115
0	18	324	294
1	17	233	206
3	17	46	45
4	16	88	82
2	16	249	241
0	16	383	365
1	15	210	206
2	15	70	55
3	15	243	225
5	15	130	118
6	15	81	72
6	14	115	104
4	14	319	299
3	14	73	62
2	14	361	347
1	14	158	151
0	14	63	65
1	13	96	98
2	13	112	108
3	13	289	272
5	13	263	255
6	13	61	48
6	12	150	152
4	12	274	277
3	12	65	56
2	12	302	278
0	12	87	97
1	11	427	409
2	11	210	194
3	11	131	120
4	11	131	127
5	11	161	153
7	11	85	101
7	10	58	45
6	10	123	126
5	10	87	81
4	10	57	49
3	10	90	90
2	10	317	325
1	10	232	224
0	10	727	751
1	9	495	502
2	9	118	113
3	9	44	12*
4	9	105	116
6	9	79	61
7	9	101	101
6	8	114	123
5	8	136	123
4	8	54	26*
3	8	253	238
2	8	407	375
1	8	43	37
0	8	532	554
1	7	52	67
3	7	97	108
4	7	81	70
5	7	73	89
7	7	124	104
8	6	52	49
7	6	108	109
6	6	116	106
5	6	122	126
4	6	369	366
3	6	210	198
2	6	316	301
1	6	417	434
0	6	89	74
1	5	59	21*
3	5	452	436
5	5	414	403
6	5	46	35
7	5	121	95
8	4	48	42
6	4	78	91
4	4	714	713
3	4	318	318
2	4	384	366
1	4	495	482
0	4	580	580
1	3	234	241
2	3	119	143
3	3	640	649
4	3	328	327
5	3	411	421
6	3	121	108
7	3	152	141
8	3	59	17*
8	2	74	95
6	2	179	178
5	2	161	154
4	2	238	242
3	2	144	187
2	2	798	822
1	2	260	255
1	1	853	887
2	1	112	124
3	1	404	404
4	1	320	313
5	1	113	106
6	1	73	69
7	1	256	249
8	1	48	18*
8	0	157	170
6	0	143	153
4	0	120	79
2	0	319	288
L=	1		
0	21	118	122
-1	20	116	119
0	20	86	91
1	20	107	105
2	19	156	148
1	19	122	114
0	19	113	109
-2	19	92	93
-4	19	97	82
-3	18	74	75
-1	18	139	137
0	18	91	94
1	18	189	175
2	18	40	13*
3	18	137	113
4	17	103	100
2	17	204	199
0	17	193	186
-1	17	58	60
-3	17	85	73
-4	17	130	123
-5	16	134	129
-4	16	64	66
-3	16	147	150
-2	16	135	134
1	16	231	216
2	16	88	106
3	16	160	153
4	16	63	64
5	16	113	121
5	15	73	70
4	15	160	158
3	15	82	72
2	15	228	230
0	15	93	78
-1	15	125	116
-2	15	154	148
-3	15	128	135
-4	15	79	89
-5	15	54	47
-6	15	109	109
-5	14	129	139
-4	14	122	121
-3	14	359	361
-2	14	71	70
-1	14	225	223
0	14	77	63
1	14	62	63
2	14	101	103
3	14	101	107
5	14	133	143
6	13	117	123
4	13	218	203
2	13	55	70
1	13	41	37
0	13	249	249
-1	13	220	233
-2	13	531	527
-3	13	102	89
-4	13	84	80
-6	13	46	27
-7	12	53	58
-6	12	73	51
-5	12	173	160
-4	12	153	160
-3	12	449	444
-2	12	144	130
-1	12	381	389
1	12	153	165
2	12	134	127
3	12	62	55
4	12	106	109
5	12	138	138
6	12	57	48
6	11	114	103
5	11	142	141
4	11	149	156
3	11	105	107
2	11	258	254
1	11	282	236
0	11	409	411
-1	11	111	101
-3	11	207	203
-5	11	112	105
-6	11	126	127
-7	10	136	137
-6	10	107	98
-5	10	237	230
-4	10	169	156
-2	10	128	118
-1	10	54	44
0	10	74	56
1	10	509	493
2	10	43	40
3	10	167	170
4	10	129	113
5	10	118	117
6	10	72	57
5	9	82	66
4	9	213	205
3	9	148	139
2	9	597	587
1	9	214	191
0	9	348	342
-1	9	194	184
-2	9	202	192
-3	9	106	92
-4	9	192	180
-5	9	50	12*
-6	9	295	290
-7	9	57	39
-8	9	93	94
-7	8	168	149
-5	8	401	381
-4	8	65	62
-3	8	277	252
-2	8	202	174
-1	8	288	275
0	8	354	341
1	8	464	473
2	8	110	86
3	8	410	412
5	8	153	168
6	8	52	20*
7	8	54	36
6	7	109	104
5	7	153	144
4	7	239	233
3	7	109	110
2	7	558	568
1	7	290	288
0	7	255	258
-1	7	179	144
-2	7	317	321
-3	7	227	227
-4	7	176	180
-5	7	176	161
-6	7	282	268
-7	7	129	138
-8	7	92	83
-7	6	168	165
-6	6	141	136
-5	6	170	178
-4	6	262	250
-3	6	498	490
-2	6	570	543
-1	6	731	738
0	6	179	188
1	6	281	302
2	6	121	110
3	6	130	158
4	6	165	120
5	6	198	217
6	6	97	97
7	6	114	106
6	5	261	245
5	5	132	136
4	5	390	395
3	5	35	33
1	5	201	211
0	5	682	668
-1	5	174	168
-2	5	706	715
-3	5	290	291
-4	5	312	313
-5	5	112	120
-6	5	96	87
-7	5	64	66
-8	5	86	99
-8	4	82	68
-7	4	79	79
-6	4	68	100
-5	4	236	226
-4	4	93	97
-3	4	685	674
-2	4	133	122
-1	4	1073	1084
1	4	45	13*
2	4	359	346
3	4	88	90
4	4	89	88
5	4	156	159
7	4	142	128
8	3	88	74
6	3	293	283
5	3	166	153
4	3	288	274
3	3	197	212
2	3	435	413
1	3	423	419
0	3	76	57
-1	3	764	782
-2	3	1130	1160
-3	3	310	300
-4	3	427	419
-7	3	107	95
-8	3	99	79
-9	3	63	65
-9	2	78	86
-8	2	85	88
-7	2	177	178
-6	2	98	84
-5	2	380	369
-4	2	245	253
-3	2	455	425
-2	2	566	542
1	2	405	406
2	2	960	1005
3	2	490	484
4	2	164	183
5	2	44	29
7	2	73	91
8	2	45	39
6	1	163	146
5	1	86	96
4	1	300	291
3	1	63	72
2	1	419	434
1	1	1215	1277
-2	1	640	623
-4	1	411	405
-5	1	108	124
-6	1	338	337
-7	1	111	121
-8	1	164	140
-9	0	80	74
-7	0	301	285
-5	0	361	373
-3	0	189	177
1	0	1230	1255
3	0	569	603
5	0	43	35
7	0	51	46
L=	2		
-2	21	47	46
-2	20	59	80
0	20	120	122
1	19	112	123
-1	19	126	130
-3	19	107	96
-4	19	68	72
-4	18	142	154
-2	18	110	119
0	18	76	74
1	18	59	28*
2	18	85	77
3	17	62	60
1	17	91	87
-1	17	118	124
-3	17	206	199
-5	17	130	128
-6	16	66	78
-4	16	211	217
-3	16	41	27
-2	16	143	143
-1	16	184	195
-1	15	36	35
0	16	49	38
1	16	95	96
2	16	51	33
3	16	54	49
4	16	127	131
3	15	110	110
2	15	154	152
1	15	104	117
0	15	92	92
-2	15	45	53
-3	15	225	215
-4	15	62	65
-5	15	123	109
-6	15	82	79
-7	15	109	96
-6	14	154	141
-5	14	50	46
-4	14	129	132
-3	14	169	164
-2	14	101	85
-1	14	206	219
-0	14	161	168
2	14	39	51
3	14	83	86
4	14	79	79
5	13	48	39
4	13	50	55
3	13	196	205
2	13	42	48
1	13	313	313
-1	13	281	287
-4	13	70	60
-5	13	54	35
-6	13	45	57
-7	13	133	137
-7	12	44	55
-6	12	194	204
-5	12	158	146
-3	12	79	79
-2	12	263	262
-1	12	209	200
0	12	504	493
1	12	64	62
2	12	96	79
3	12	115	119
4	12	67	54
5	12	66	63
4	11	63	93
3	11	179	186
2	11	214	206
1	11	499	511
0	11	180	171
-1	11	244	251
-2	11	133	146
-3	11	283	288
-4	11	41	15*
-6	11	150	148
-7	11	190	190
-6	10	112	90
-7	10	104	87
-6	10	123	126
-4	10	387	390
-3	10	42	30
-2	10	388	391
-1	10	417	409
0	10	294	291
1	10	61	49
2	10	122	144
3	10	105	107
4	10	186	196
6	10	63	83
5	9	147	153
3	9	257	264
4	9	62	59
1	9	422	451
0	9	103	99
-1	9	332	332
-2	9	120	128
-3	9	528	541
-4	9	43	38
-5	9	380	385
-7	9	83	70
-7	8	50	47
-6	8	161	167
-4	8	646	628
-3	8	43	2*
-2	8	506	518
-1	8	277	271
0	8	212	212
1	8	51	19*
2	8	243	272
3	8	157	154
4	8	345	325
5	8	68	78
6	8	139	144
6	7	47	53
5	7	165	204
4	7	194	203
3	7	340	346
2	7	95	87
1	7	416	396
0	7	129	136
-1	7	654	639
-2	7	304	333
-3	7	579	581
-4	7	408	402
-5	7	363	368
-6	7	101	81
-7	7	150	156
-9	7	66	56
-9	6	47	37
-8	6	149	145
-7	6	98	66
-6	6	371	368
-5	6	183	172
-4	6	362	341
-3	6	100	94
-2	6	443	432
-1	6	326	335
0	6	389	384
1	6	75	69
2	6	312	292
3	6	336	320
4	6	202	190
5	6	133	141
6	6	135	144
7	5	52	73
5	5	186	185
4	5	135	126
3	5	375	358
2	5	135	133
1	5	436	440
0	5	58	69
-1	5	529	522
-2	5	275	263
-3	5	360	320
-4	5	147	135
-5	5	107	126
-6	5	101	117
-7	5	252	254
-9	5	111	89
-8	4	262	258
-6	4	321	350
-5	4	190	194
-3	4	410	384
-2	4	86	74
-1	4	253	249
0	4	658	683
1	4	260	253
2	4	341	351
3	4	94	105
4	4	62	71
6	4	78	81
6	3	106	98
5	3	84	95
4	3	112	108
2	3	167	163
1	3	325	344
0	3	145	141
-1	3	869	886
-2	3	63	25*
-3	3	95	63
-5	3	170	213
-6	3	290	248
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Table 3. Continued.

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				-2	13	217	215	-10	3	159	153	9	10	198	191	-10	2	83	69	1	7	56	48	-5	12	44	25*
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0	4	245	240	0	13	201	204	-8	3	212	204	-2	10	327	326	-9	1	107	96	-1	6	95	99	-2	12	129	126
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-3	19	52	24*	2	7	176	174	-4	15	62	49	1	5	255	243	-2	11	180	181	-5	0	77	90	2	4	56	30*
0	19	91	80	4	7	124	127	-3	15	137	135	3	5	173	148	0	11	112	103	-7	0	142	141	2	3	54	21*
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-1	18	68	74	3	6	75	89	-1	15	109	91	2	4	160	163	1	10	124	125					-1	3	195	200
-2	18	48	10*	2	6	152	146	0	15	90	88	1	4	50	15*	-1	10	118	134	L=	8			-3	3	256	256
-3	18	109	113	1	6	182	172	1	15	60	74	0	4	248	266	-2	10	136	131					-5	3	71	82
-5	18	123	119	0	6	51	43	2	14	90	93	-1	4	56	54	-3	10	43	36	-5	19	81	60	-6	3	144	139
-7	18	75	63	-1	6	385	366	-2	14	53	23*	-4	4	262	272	-4	10	53	64	-6	18	107	106	-7	3	136	155
-6	17	98	110	-2	6	177	179	-3	14	83	70	-5	4	121	128	-5	10	42	56	-4	18	54	60	-7	3	124	135
-4	17	154	159	-3	6	516	498	-4	14	102	95	-6	4	340	348	-7	10	104	169	-5	17	136	137	-9	3	123	121
-3	17	48	36	-4	6	216	205	-5	14	81	83	-7	4	64	78	-9	10	105	120	-7	17	92	108	-10	3	68	58
-2	17	89	77	-5	6	247	240	-6	14	201	181	-8	4	245	230	-10	10	83	87	-8	16	91	82	-11	3	72	94
2	17	80	88	-6	6	280	284	-7	14	95	67	-11	3	62	57	-10	9	87	91	-6	16	121	110	-12	3	69	43
1	16	53	21*	-7	6	113	125	-8	14	86	74	-10	3	62	53	-9	9	68	57	-5	16	85	66	-9	2	75	102
0	16	51	40	-9	6	119	123	-8	13	70	32*	-8	3	140	128	-8	9	175	173	-4	16	103	99	-8	2	61	121*
-1	16	99	87	-10	6	83	67	-7	13	118	111	-7	3	289	281	-6	9	159	162	-3	16	90	74	-7	2	91	124
-2	16	97	102	-11	5	169	163	-3	13	50	62	-6	3	203	210	-4	9	89	93	-2	16	96	48*	-6	2	249	278
-3	16	188	182	-8	5	212	219	-2	13	57	17*	-5	3	192	182	-3	9	66	74	0	16	89	63	-5	2	151	175
-5	16	185	186	-6	5	128	130	1	13	159	157	-4	3	224	224	-2	9	150	140	-1	15	80	74	-3	2	83	95
-7	16	80	71	-5	5	69	98	3	13	85	59	-3	3	82	83	0	9	270	259	-3	15	134	135	-2	2	222	214
-7	15	86	75	-4	5	98	97	2	12	132	141	-2	3	189	192	1	9	69	22*	-4	15	68	67	-1	2	38	20*
-6	15	147	151	-3	5	370	370	0	12	103	96	-1	3	113	102	1	8	212	200	-5	15	84	88	0	2	126	117
-4	15	136	134	-2	5	569	551	-2	12	46	44	0	3	322	301	-1	8	187	176	-6	15	59	17*	1	2	78	62
-3	15	269	265	0	5	331	307	-5	12	222	221	1	3	286	276	-2	8	73	62	-8	15	58	37	2	2	119	105
-2	15	131	147	3	5	52	60	-6	12	224	234	3	3	141	147	-4	8	159	152	-8	14	91	78	1	1	119	114
2	15	88	72	4	5	68	67	-8	12	161	146	4	3	51	40	-6	8	119	103	-7	14	48	11*	0	1	55	67
3	15	64	27*	5	4	61	63	-9	11	115	112	5	2	79	59	-7	8	114	87	-5	14	87	72	-1	1	50	56
2	14	61	55	2	4	89	64	-7	11	119	127	2	2	64	44	-8	8	73	74	-4	14	112	121	-2	1	81	71
1	14	140	129	1	4	325	318	-6	11	178	167	1	2	54	53	-9	8	169	168	-3	14	60	64	-3	1	75	84
-1	14	235	229	0	4	103	110	-5	11	187	201	0	2	173	177	-10	7	96	104	-2	14	152	135	-5	1	450	498
-2	14	148	149	-1	4	482	485	-4	11	161	165	-1	2	148	146	-8	7	180	175	0	14	55	61	-7	1	256	289
-4	14	83	92	-2	4	157	156	-3	11	189	193	-2	2	180	178	-7	7	144	152	-1	13	134	136	-6	0	390	398
-5	14	139	148	-3	4	451	448	-2	11	86	90	-4	2	331	338	-6	7	142	154	-3	13	225	218	2	0	126	144
-8	13	159	164	-4	4	276	273	-1	11	137	128	-5	2	448	439	-5	7	49	51	-7	13	58	61				
-8	13	51	32	-5	4	201	202	0	11	111	102	-6	2	158	143	-4	7	94	109	-9	13	95	93				
-5	13	79	84	-6	4	63	75	1	11	143	132	-7	2	38	14*	-3	7	182	159	-10	12	67	114*				

and the N – Cu – N angles are very near to 90°. Above and below this plane two oxygen atoms from the perchlorate groups are symmetrically placed and complete the octahedral coordination. The Cu – O distances are 2.625 Å and the angles O – Cu – O, O – Cu – N_1, and O – Cu – N_3 are 180°, 93°, and 94°, respectively. These angles and distances strongly indicate a nearly regular octahedral coordination around the copper atom.

The ligands imidazole and perchlorate. The dimensions of the imidazole

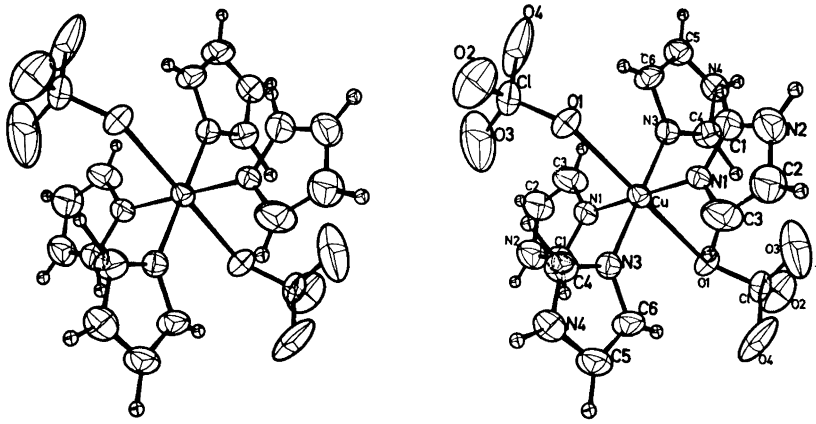

Fig. 1. Stereoscopic diagrams of the Cu-coordination with thermal ellipsoids of the non-hydrogen atoms scaled to enclose 50 % probability.

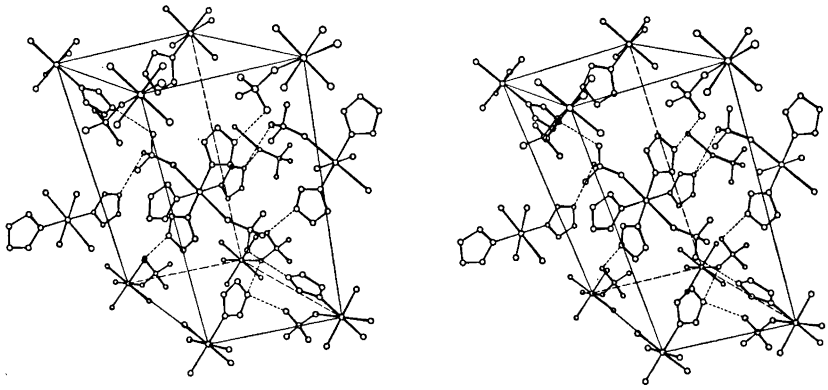

Fig. 2. Stereoscopic illustration of the molecular packing of $Cu(C_3H_4N_2)_4(ClO_4)_2$ in the unit cell. The cell is tilted to avoid overlaping. The broken lines mark hydrogen bonds.

rings are of the same size as found in other studies.[1,7–9] The planarity of the imidazole rings is shown in Table 5, where the planes of best fit are given.

No abnormalities could be found in the perchlorate group. The Cl – O distances range from 1.391 Å to 1.442 Å, the longest distance being to the copperbonded oxygen.

The hydrogen positions and hydrogen bonds. The positions of the hydrogen atoms are shown in Fig. 1 and the coordinates are given in Table 2.

The hydrogen atoms which are bonded to the carbon atoms in the imidazole rings are placed in the expected positions. The hydrogen atoms on the pyrrole nitrogens (N_2 and N_4) take part in hydrogen bonds of which there are two

Table 4. Bond lengths and bond angles and their estimated standard deviations.

Bond	$l(\sigma(l)\times 10^3)$,Å	Angle	$\theta(10\sigma(\theta))°$	Bond	$l(\sigma(l)\times 10^3)$,Å
$Cu-O_1$	2.625(5)	N_1-Cu-N_3	90.3(2)		
$Cu-N_1$	2.010(4)	O_1-Cu-N_1	86.9(2)		
$Cu-N_3$	1.998(4)	O_1-Cu-N_3	90.7(2)		
N_1-C_1	1.319(8)	$N_1-C_1-N_2$	110.9(6)	C_1-H_1	0.938(70)
C_1-N_2	1.331(9)	$C_1-N_2-C_2$	108.6(6)	N_2-H_2	0.947(69)
N_2-C_2	1.348(9)	$N_2-C_2-C_3$	104.9(6)	C_2-H_3	0.988(70)
C_2-C_3	1.355(10)	$C_2-C_3-N_1$	110.7(6)	C_3-H_4	0.852(70)
C_3-N_1	1.361(8)	$C_3-N_1-C_1$	104.8(5)		
N_3-C_4	1.311(9)	$N_3-C_4-N_4$	110.9(6)	C_4-H_5	1.200(70)
C_4-N_4	1.325(8)	$C_4-N_4-C_5$	108.0(6)	N_4-H_6	0.937(70)
N_4-C_5	1.353(10)	$N_4-C_5-C_6$	106.2(6)	C_5-H_7	1.019(68)
C_5-C_6	1.339(10)	$C_5-C_6-N_3$	109.4(6)	C_6-H_8	0.834(71)
C_6-N_3	1.372(8)	$C_6-N_3-C_4$	105.5(5)		
$Cl-O_1$	1.442(5)	O_1-Cl-O_2	110.2(4)	$O_1\cdots H_2$	2.217(71)
$Cl-O_2$	1.409(7)	O_1-Cl-O_3	108.7(5)	$O_2\cdots H_2$	2.403(70)
$Cl-O_3$	1.406(7)	O_1-Cl-O_4	108.1(5)	$O_4\cdots H_6$	2.056(69)
$Cl-O_4$	1.391(6)	O_2-Cl-O_3	108.0(5)		
		O_2-Cl-O_4	110.4(6)		
		O_3-Cl-O_4	111.4(7)		

Table 5. (a) Planes of best fit (least squares).

Imidazole ring ($N_1C_1N_2C_2C_3$)
$0.6902x-0.1445y-0.7090z-2.9100=0$

Imidazole ring ($N_3C_4N_4C_5C_6$)
$-0.5774x+0.3185y-0.7517z-1.0066=0$

(b) Deviation (Å) from planes (max standard deviation of the atomic positional fractional coordinates $xyz \times 10^4$ in parentheses).

$N_1(6)$	0.003	Cu(0)	−0.016	$N_3(7)$	0.000	Cu(0)	−0.037
$C_1(10)$	−0.004	$H_1(100)$	0.047	$C_4(11)$	0.003	$H_5(100)$	−0.139
$N_2(8)$	0.003	$H_2(100)$	0.134	$N_4(9)$	−0.005	$H_6(100)$	0.041
$C_2(10)$	−0.001	$H_3(100)$	0.024	$C_5(11)$	0.005	$H_7(100)$	−0.094
$C_3(11)$	−0.002	$H_4(100)$	−0.003	$C_6(11)$	−0.003	$H_8(100)$	−0.053

kinds in the structure. A schematic drawing illustrating these hydrogen bonds is shown in Fig. 2. The two broken lines from an imidazole nitrogen to two different perchlorate groups show a suggested bifurcated hydrogen bond. Distances are given in Table 4. By comparing the distances there seems to be a stronger interaction to the copperbonded oxygen $O_1(O_1\cdots N_2=3.000$ Å) than to $O_2(O_2\cdots N_2=3.221$ Å). An interaction to O_2 cannot be excluded. The

distances between N_4 and O_4, where a single hydrogen bond is proposed, is 2.921 Å.

As can be seen from Fig. 2, hydrogen bonds connect the complexes forming coupled sheets within the structure.

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