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X-ray Crystal Structure Analysis of Bis(pyridine *N*-oxide)copper(II) Nitrate, $Cu(C_5H_5NO)_2(NO_3)_2$

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The crystals of $Cu(C_5H_5NO)_2(NO_3)_2$ are monoclinic with $a=11{\cdot}820$, $b=14{\cdot}862$, $c=8{\cdot}005$ Å, $\beta=95{\cdot}50°$, space group $P2_1/n$, with four formula units in the unit cell. A three-dimensional X-ray crystal structure analysis has shown that the compound is a dimeric complex, formula $[Cu(C_5H_5NO)_2(NO_3)_2]_2$, composed of two centrosymmetrically related monomers. Two copper atoms within the complex are joined by two bridging oxygen atoms (from C_5H_5NO groups) with a Cu–Cu distance of 3·458 Å. Two nitrate groups, acting as monodentate ligands, and two oxygen atoms (from two C_5H_5NO groups) are bonded to each copper atom by short (from 1·951 to 1·968 Å) and nearly coplanar bonds. The fifth Cu–O bond is long (2·439 Å); it's oxygen atom belongs to the C_5H_5NO group which is connected with the other copper atom (from the dimer) by short bonds. Thus each copper atom has a distorted tetragonal pyramidal environment of five oxygen atoms, and the dimeric complex contains two pyramidal polyhedra with one common pyramidal edge. The distances in the nitrate and pyridine *N*-oxide groupings do not deviate significantly from the known values. The shortest intermolecular distances correspond to van der Waals interactions.

Introduction

Bis(pyridine *N*-oxide)copper(II) nitrate,

$$Cu(C_5H_5NO)_2(NO_3)_2\,,$$

was isolated and characterized by infrared spectra, magnetic susceptibility and molecular conductance measurements by Quagliano, Fujita, Franz, Phillips, Walmsley & Tyree (1961). On the basis of the NO (from C_5H_5NO) frequency band at 1205 cm^{-1} and the strong absorption bands at 1292 and 1019 cm^{-1} it was concluded that the pyridine *N*-oxide and the nitrate ligand made up the first coordination sphere of the copper atom. This is in agreement with a stiochiometric composition of the molecule, the usual coordination number of the copper(II) ion, and the property of pyridine *N*-oxide to coordinate only through an oxygen atom (since the oxygen atom is a negative end of a dipole). The conclusion of Quagliano *et al.* (1961) was confirmed by the present work of X-ray crystal structure analysis. Besides this some new, unpredictable, properties of the structure were revealed which may be of interest in a study of the structural chemistry of transition metal complexes of pyridine *N*-oxide; these include, among others, the geometry of coordination of the oxygen donor ligand, its role as a bridging ligand, the 'deficient' coordination number of copper(II) in the presence of two unattached oxygen atoms per NO_3 group and the absence of metal–metal interactions. A starting hypothesis of an octahedral coordination for the copper(II) atom with two nitrate groups, acting as bidentate ligands in the equatorial plane, and two pyridine *N*-oxide groups in axial positions had to be abandoned.

The main features of the crystal structure of bis(pyridine *N*-oxide) copper(II) nitrate, with some interatomic distances and angles within the copper polyhedron, were described by Šćavničar & Matković (1967). At that time the structure was solved by least-squares refinement with isotropic temperature factors. About 220 reflexions (mostly weak, slightly above film ground and the strongest which indicated that extinction corrections were necessary) were omitted in these calculations. The present paper describes in detail the structure derived from a three-dimensional anisotropic refinement with all (collected) reflexions and corrections for secondary extinction.

Experimental

Crystals of $Cu(C_5H_5NO)_2(NO_3)_2$ were prepared by R. L. Carlin,* and kindly supplied to D. Grdenić.† Intensities of X-ray reflexions were recorded photographically by the equi-inclination Weissenberg method (Ni-filtered Cu $K\alpha$ radiation) from a crystal mounted about the *b* axis (layers *h*0*l* to *h*7*l* inclusive) and about the *a* and *c* axes (zero layer reflexions only). Of 1917 attainable symmetry-independent reflexions, 1359 were measured with an optical densitometer, another 200 (slightly above background) were estimated visually and 358 reflexions were unobserved. Lorentz and polarization, but not absorption corrections were applied. The calculated absorption coefficient, μ, of 27·5 cm^{-1}, and

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the dimensions of crystals used indicated that μs was limited to between 0·8 and 1·0, where s was the maximum path length of an X-ray within the crystal. Unit-cell dimensions were obtained from the oscillation photographs taken with Cu $K\alpha$ radiation.

Crystal data

The well developed crystals are bounded by the faces of the {010}, {110}, {021}, {011} and {$\bar{1}$01} forms of the monoclinic holosymmetric class. The crystals are transparent and of an intense emerald green colour. They are thick plate-like crystals of [010] habit slightly elongated in the a-axis direction.

M.W. for formula unit $Cu(C_5H_5NO)_2(NO_3)_2$ = 377·76,

Monoclinic,

$a = 11{\cdot}820 \pm 0{\cdot}005$, $b = 14{\cdot}862 \pm 0{\cdot}01$,

$c = 8{\cdot}005 \pm 0{\cdot}005$ Å; $\beta = 95{\cdot}5 \pm 0{\cdot}2°$.

$V = 1399{\cdot}6$ Å^3, $Z = 4$,

$D_c = 1{\cdot}792$ and $D_m = 1{\cdot}78$ g.cm^{-3} (determined by the flotation method in a mixture of bromoform and benzene.

Systematic absences of reflexions occur for $h0l: h+l = 2n+1$ and $0k0: k = 2n+1$. These uniquely determine the space group as $P2_1/n$. The asymmetric unit comprises one molecule; all the atoms are in the fourfold general positions of the set $\pm(x,y,z; \frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z)$. An alternative choice of a axis along the [$\bar{1}$01] direction, providing a space group symbol in the more usual $P2_1/a$ form, was not accepted because of a larger repeat period (13·590 Å) in this direction and an inconveniently large monoclinic angle (120·1°).

Structure determination

Two Patterson projections $P(xy)$ and $P(yz)$ were interpreted to give a set of coordinates for the copper atom in the asymmetric unit. A three-dimensional Fourier synthesis, with the phases of the diffracted beams calculated from the positions of the copper atom, yielded the locations of all the other non-hydrogen atoms. The structure was then refined by several cycles of full-matrix least-squares refinement with isotropic temperature factors. At this point low order strong reflexions were found to have F_o consistently less than F_c, which indicated extinction. To handle this, Zachariasen's (1963) formulation of the extinction problem was adopted and the formula $F_{corr} = 1/K.F_{obs}(1 + \beta_{2\theta}C.I_{obs})$ was employed. The resultant value of the C parameter was $0{\cdot}24.10^{-3}$ for intensities on a relative scale. Prior to beginning the anisotropic refinement the scale factors, K, among the various hkl levels of the data were adjusted, again to give the best fit to the calculated structure factors (the values of K obtained were between 0·32 and 0·36). Five cycles of full-matrix least-squares refinement with anisotropic temperature factors for all non-hydrogen atoms reduced the reliability index to $R_1 = 10{\cdot}1\%$ (including unobserved reflexions) and $R_2 = 7{\cdot}5\%$ (omitting unobserved reflexions). The weighting scheme adopted was: $w = 1$ for all reflexions whose intensities were determined with an optical densitometer, $w = 0{\cdot}25$ for reflexions which were estimated visually (slightly above film background) and $w = 0$ for unobserved reflexions. Neutral-atom scattering factors were taken from *International Tables for X-ray Crys-*

Table 1. *Final atomic coordinates* ($\times 10^4$) *and thermal parameters* ($\times 10^5$)

Standard deviations are in parentheses.

Thermal parameters are of the form:

$$T = \exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)].$$

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Cu(1)	1060 (1)	801 (1)	311 (2)	250 (8)	170 (10)	760 (20)	10 (8)	−30 (8)	40 (13)
O(2)	−582 (5)	740 (5)	533 (7)	294 (30)	228 (40)	874 (72)	−18 (28)	29 (22)	−147 (48)
O(3)	2608 (5)	604 (5)	−263 (8)	320 (36)	305 (41)	976 (81)	8 (22)	23 (32)	135 (45)
O(4)	1430 (5)	547 (6)	2712 (8)	459 (41)	348 (45)	869 (72)	127 (36)	130 (47)	29 (43)
O(5)	2479 (7)	1023 (6)	4905 (10)	761 (55)	541 (58)	1103 (99)	−75 (44)	−308 (60)	−28 (53)
O(6)	2131 (7)	1918 (7)	2802 (11)	1193 (73)	214 (37)	1640 (129)	−93 (52)	228 (82)	66 (65)
O(7)	755 (6)	1230 (6)	−2013 (8)	585 (45)	248 (39)	866 (78)	61 (36)	−79 (50)	61 (56)
O(8)	536 (7)	2514 (7)	−811 (10)	859 (61)	202 (38)	1968 (132)	−76 (46)	356 (73)	−315 (68)
O(9)	496 (7)	2423 (6)	−3539 (10)	917 (60)	425 (52)	1527 (113)	132 (49)	−135 (56)	482 (65)
N(10)	−995 (6)	1166 (6)	1853 (8)	261 (39)	148 (37)	681 (84)	47 (34)	−18 (28)	−120 (53)
N(11)	3382 (6)	212 (6)	879 (9)	242 (38)	294 (52)	1090 (105)	68 (37)	28 (43)	15 (41)
N(12)	2034 (7)	1190 (7)	3498 (10)	350 (44)	384 (53)	900 (97)	9 (27)	−4 (32)	−124 (69)
N(13)	568 (7)	2094 (8)	−2117 (10)	301 (41)	261 (49)	1413 (121)	32 (38)	−46 (60)	134 (83)
C(14)	−1089 (8)	708 (8)	3270 (11)	575 (78)	219 (37)	913 (98)	4 (29)	−42 (48)	70 (64)
C(15)	−1545 (9)	1150 (9)	4580 (13)	691 (89)	316 (47)	1157 (108)	134 (57)	187 (62)	172 (70)
C(16)	−1872 (9)	2037 (9)	4425 (13)	437 (61)	371 (52)	1156 (111)	−58 (36)	61 (51)	−185 (69)
C(17)	−1770 (8)	2496 (9)	2921 (12)	427 (58)	184 (33)	1382 (127)	45 (34)	60 (53)	−116 (65)
C(18)	−1313 (8)	2034 (8)	1617 (11)	448 (59)	184 (31)	1270 (122)	47 (34)	25 (39)	−248 (77)
C(19)	4309 (8)	717 (9)	1445 (11)	329 (43)	319 (45)	1297 (120)	−34 (37)	−62 (45)	−209 (82)
C(20)	5156 (9)	313 (9)	2559 (13)	468 (62)	428 (59)	1523 (132)	82 (49)	−133 (59)	−110 (66)
C(21)	5027 (9)	−580 (9)	3024 (13)	521 (68)	339 (57)	1403 (136)	163 (61)	−237 (84)	−230 (79)
C(22)	4062 (9)	−1081 (9)	2427 (14)	644 (85)	265 (44)	1519 (133)	211 (59)	−167 (66)	62 (58)
C(23)	3252 (8)	−637 (9)	1327 (13)	403 (52)	233 (40)	1518 (128)	−37 (33)	−81 (52)	20 (50)

Table 2. *Observed*, Fo, *and calculated* Fc, *structure factors*

H	K	L	100Fo	100Fc
0	1	1	13512	12731
0	1	2	2239	2317
0	1	3	5478	5592
0	1	4	2498	-2230
0	1	5	906	995
0	1	6	1170	1432
0	1	7	00	221
0	1	8	2666	-2622
0	1	9	769	-779
0	1	10	00	16
0	2	1	7742	-7460
0	2	2	6714	6836
0	2	3	6799	-7119
0	2	4	4339	4773
0	2	5	4133	-3987
0	2	6	768	674
0	2	7	4387	-4342
0	2	8	399	679
0	2	9	2187	-2234
0	2	10	313	-456
0	3	1	7766	6502
0	3	2	3109	-2902
0	3	3	5287	4892
0	3	4	6834	-6941
0	3	5	2672	-2491
0	3	6	4955	-5273
0	3	7	684	936
0	3	8	782	-1003
0	3	9	00	-252
0	3	10	558	-583
0	4	1	4068	-3672
0	4	2	3524	-3920
0	4	3	2924	-3544
0	4	4	4012	-4233
0	4	5	4411	-4548
0	4	6	3101	-3301
0	4	7	2516	-2494
0	4	8	00	236
0	4	9	1630	-1752
0	5	1	6747	-6130
0	5	2	3107	-2692
0	5	3	3562	-3645
0	5	4	2469	-2183
0	5	5	2373	-2410
0	5	6	1429	-1211
0	5	7	484	909
0	5	8	2219	-2119
0	5	9	00	209
0	6	1	4264	-4011
0	6	2	7888	-7733
0	6	3	2455	2516
0	6	4	3754	-3889
0	6	5	2197	-2373
0	6	6	2857	-3044
0	6	8	00	156
0	6	9	00	305
0	7	1	9716	-8538
0	7	2	771	-720
0	7	3	7484	-6793
0	7	4	00	66
0	7	5	1866	-1672
0	7	6	1547	1168
0	7	7	2135	-2096
0	7	8	1257	1234
0	7	9	799	753
0	8	1	00	228
0	8	2	3179	-3510
0	8	3	4667	5022
0	8	4	3670	-3802
0	8	5	1941	1874
0	8	6	570	-674
0	8	7	3897	4003
0	8	8	00	-375
0	8	9	1092	1406
0	9	1	4136	-4347
0	9	2	2097	2434
0	9	3	4193	-4768
0	9	4	3498	3609
0	9	5	1469	1797
0	9	6	3068	3239
0	9	7	2247	-2310
0	9	8	2034	2157
0	10	1	1647	1938
0	10	2	1468	1538
0	10	3	765	820
0	10	4	1676	1839
0	10	5	2907	3292
0	10	6	00	482
0	10	7	2365	2303
0	10	8	00	-210
0	11	1	2779	3171
0	11	2	1321	1059
0	11	3	2154	2163
0	11	4	1614	1611
0	11	5	00	259
0	11	6	2443	2487
0	11	7	00	235
0	11	7	00	235
0	11	8	1208	1554
0	12	1	685	-1204
0	12	2	5665	6184
0	12	3	771	-580
0	12	4	1138	1040
0	12	5	627	411
0	12	6	767	698
0	12	7	00	-121
0	12	8	00	277
0	13	1	4474	4953
0	6	7	814	1142
0	13	2	944	-1059
0	13	3	4762	4739
0	13	4	484	-432
0	13	5	481	1034
0	13	6	770	-893
0	13	7	484	523
0	14	1	00	83
0	14	2	2745	3111
0	14	3	1438	-1770
0	14	4	00	400
0	14	5	1852	-1842
0	14	6	1148	1447
0	14	7	860	-800
0	15	1	2187	2228
0	15	2	2247	-2533
0	15	3	00	600
0	15	4	2398	-2840
0	15	5	00	251
0	15	6	1786	-2124
0	16	1	1030	-1333
0	16	2	1350	-1516
0	16	3	1884	-2029
0	16	4	00	-220
0	16	5	1384	-1692
0	17	1	1554	-1619
0	17	2	00	-455
0	17	3	280	-609
0	17	4	1568	-1731
0	18	1	571	-624
0	18	2	1623	-1869
0	18	3	00	-263
0	19	1	1347	-2014
0	2	0	9576	7991
0	4	0	4301	-4218
0	6	0	22188	-21972
0	8	0	5159	-5188
0	10	0	2099	1551
0	12	0	2589	2419
0	14	0	2037	1859
0	16	0	2777	3249
0	18	0	1860	-2147
1	1	0	8640	9804
2	1	0	5452	-5612
3	1	0	8084	-8341
4	1	0	8075	-7755
5	1	0	10161	-11499
6	1	0	2996	-2972
7	1	0	2527	-2492
8	1	0	2654	2485
9	1	0	3956	3815
10	1	0	821	-852
11	1	0	00	301
12	1	0	307	-534
13	1	0	3076	-2952
14	1	0	00	313
15	1	0	782	-624
1	2	0	7933	-9346
2	2	0	491	87
3	2	0	1347	1185
4	2	0	4382	-4339
5	2	0	4847	-4437
6	2	0	1745	-1404
7	2	0	5651	5784
8	2	0	5378	5341
9	2	0	00	408
10	2	0	2066	1906
11	2	0	1606	-1211
12	2	0	1874	1767
13	2	0	432	-501
14	2	0	818	-856
15	2	0	00	455
1	3	0	8631	-9355
2	3	0	7006	-6554
3	3	0	3825	3546
4	3	0	6353	6466
5	3	0	4651	-4338
6	3	0	8435	8446
7	3	0	1267	1526
8	3	0	4095	3934
9	3	0	379	534
10	3	0	1391	-1283
11	3	0	00	-398
12	3	0	4639	-4932
13	3	0	632	-611
14	3	0	315	-444
1	4	0	7135	-7677
2	4	0	2867	2898
3	4	0	2400	-2165
4	4	0	5042	4840
5	4	0	4064	3933
6	4	0	7424	6875
7	4	0	7127	7379
8	4	0	2491	-2528
9	4	0	852	-864
10	4	0	3516	-3470
11	4	0	1961	-1686
12	4	0	00	-239
13	4	0	1677	-1618
14	4	0	692	685
1	5	0	5960	-6536
2	5	0	4079	-4380
3	5	0	3045	2954
4	5	0	4072	-3947
5	5	0	6815	6777
6	5	0	3935	3633
7	5	0	854	-694
8	5	0	00	-393
9	5	0	3488	-3193
10	5	0	1585	-1431
11	5	0	2098	-1882
12	5	0	2063	-1941
13	5	0	1418	1061
14	5	0	00	-64
1	6	0	1980	-2267
2	6	0	5383	-4962
3	6	0	2469	-2504
4	6	0	5963	5697
5	6	0	00	623
6	6	0	6932	6974
7	6	0	1747	1749
8	6	0	1484	-1024
9	6	0	1107	-1089
10	6	0	2026	-1966
11	6	0	1139	986
12	6	0	00	-173
13	6	0	379	-423
14	6	0	2107	2246
1	7	0	7200	-7934
2	7	0	3748	3664
3	7	0	4765	4241
4	7	0	4714	4378
5	7	0	6559	6170
6	7	0	627	510
7	7	0	1012	-573
8	7	0	2003	-1828
9	7	0	2418	-2135
10	7	0	00	125
11	7	0	00	-86
12	7	0	00	201
13	7	0	2654	2506
14	7	0	00	-221
1	8	0	1263	1548
2	8	0	1553	1171
3	8	0	2685	2303
4	8	0	2162	1864
5	8	0	3052	2760
6	8	0	1422	1267
7	8	0	3515	-3180
8	8	0	3348	-3110
9	8	0	00	-621
10	8	0	913	-917
11	8	0	2127	1874
12	8	0	00	407
13	8	0	440	773
1	9	0	2988	2153
2	9	0	4065	3607
3	9	0	00	403
4	9	0	2131	-2301
5	9	0	2360	2548
6	9	0	4865	-4467
7	9	0	1810	-1490
8	9	0	2716	-2544
9	9	0	00	-416
10	9	0	533	469
11	9	0	00	230
12	9	0	2617	2657
13	9	0	00	440
1	10	0	2850	2774
2	10	0	2101	-1905
3	10	0	2781	2834
4	10	0	2491	-2265
5	10	0	1585	-1392
6	10	0	2197	-2044
7	10	0	5519	-5467
8	10	0	00	527
9	10	0	00	-107
10	10	0	1712	1526
11	10	0	2878	2664
12	10	0	442	-516
1	11	0	2426	2192
2	11	0	4026	3602
3	11	0	629	-995
4	11	0	00	-410
5	11	0	1801	-1316
6	11	0	4547	-4177
7	11	0	00	219
8	11	0	00	434
9	11	0	2648	2596
10	11	0	630	622
11	11	0	00	771
12	11	0	1846	1744
1	12	0	00	165
2	12	0	1553	1961
3	12	0	2686	2350
4	12	0	2811	-2438
5	12	0	00	-122
6	12	0	2749	-2479
7	12	0	2100	-1765
8	12	0	00	481
9	12	0	00	276
10	12	0	1226	1222
11	12	0	315	352
1	13	0	2616	2272
2	13	0	627	-605
3	13	0	439	682
4	13	0	2458	-2401
5	13	0	3985	-3731
6	13	0	00	205
7	13	0	00	481
8	13	0	1617	1561
9	13	0	2286	2086
10	13	0	1395	-1379
11	13	0	00	774
1	14	0	915	-1200
2	14	0	853	932
3	14	0	2291	-2698
4	14	0	2586	-2238
5	14	0	2391	2209
6	14	0	2032	-1607
7	14	0	1835	1699
8	14	0	944	781
9	14	0	00	299
10	14	0	1005	824
1	15	0	2391	2290
2	15	0	1162	-1371
3	15	0	695	-975
4	15	0	630	-676
5	15	0	1808	-1522
6	15	0	2713	2556
7	15	0	00	-295
8	15	0	1647	1502
9	15	0	630	494
1	16	0	1229	-1378
2	16	0	00	-387
3	16	0	2647	-2586
4	16	0	00	143
5	16	0	631	493
6	16	0	00	-112
7	16	0	2425	2042
8	16	0	950	-891
1	17	0	690	-922
2	17	0	621	-926
3	17	0	00	408
4	17	0	00	-588
5	17	0	879	777
6	17	0	534	620
1	18	0	695	-727
2	18	0	537	-677
3	18	0	00	-188
4	18	0	1168	1364
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9	4	4	1683	-1879
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12	4	6	00	-236
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6	4	8	1611	-1703
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13	4	-2	3622	-3706
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9	4	-3	1180	-1148
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11	4	-3	991	-1043
12	4	-3	1950	-1784
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14	4	-3	626	-622
1	4	-4	2334	2106
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3	4	-4	2821	-2819
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6	4	-4	3121	2943
7	4	-4	3957	3920
8	4	-4	00	321
9	4	-4	2117	1964
10	4	-4	1071	1078
11	4	-4	00	-618
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14	4	-4	00	29
1	4	-5	3048	-2799
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Table 2 (*cont.*)

H	K	L	100Fo	100Fc	H	K	L	100Fo	100Fc	H	K	L	100Fo	100Fc	H	K	L	100Fo	100Fc	H	K	L	100Fo	100Fc
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8	4	-5	3253	3309	6	5	5	991	-1112	3	5	-6	955	-926	7	6	6	00	79	5	6	-7	1222	1307	6	7	8	855	-764
9	4	-5	00	-169	7	5	5	2992	3259	4	5	-6	2035	-1757	8	6	6	2746	-3135	6	6	-7	00	-289	1	7	9	1024	957
10	4	-5	00	128	8	5	5	2298	-2245	5	5	-6	2391	2397	9	6	6	00	357	7	6	-7	934	849	2	7	9	944	1246
11	4	-5	1900	-1795	9	5	5	00	-181	6	5	-6	00	-207	10	6	6	00	-102	8	6	-7	00	-530	3	7	9	00	269
12	4	-5	1901	-1951	10	5	5	1722	-1892	7	5	-6	3407	3101	1	6	7	2225	2156	9	6	-7	00	-232	1	7	-1	459	-203
13	4	-5	921	-787	11	5	5	00	-728	8	5	-6	987	825	2	6	7	1471	-1407	10	6	-7	332	344	2	7	-1	1592	-1437
1	4	-6	2497	2465	1	5	6	785	527	9	5	-6	1536	-1400	3	6	7	2271	2407	11	6	-7	1857	-1890	3	7	-1	6018	6186
2	4	-6	00	-465	2	5	6	2026	-1982	10	5	-6	1846	1813	4	6	7	1326	1276	1	6	-8	788	801	4	7	-1	4928	5398
3	4	-6	1882	-1743	3	5	6	2831	2730	11	5	-6	2603	-2462	5	6	7	00	-450	2	6	-8	1362	-1464	5	7	-1	1258	-1297
4	4	-6	00	-21	4	5	6	1138	1161	12	5	-6	719	-720	6	6	7	00	290	3	6	-8	1857	-1893	6	7	-1	7047	6883
5	4	-6	4592	-4640	5	5	6	00	636	1	5	-7	1854	1825	7	6	7	2674	-2872	4	6	-8	1821	-1720	7	7	-1	1321	-992
6	4	-6	1138	1001	6	5	6	1137	1356	2	5	-7	2963	-2787	8	6	7	816	1021	5	6	-8	00	462	8	7	-1	922	-1034
7	4	-6	1017	-1145	7	5	6	00	-315	3	5	-7	00	-461	1	6	8	994	-1027	6	6	-8	2126	1897	9	7	-1	1500	-1338
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2	5	1	6073	-6184	1	5	-2	6458	-6394	14	6	1	00	-61	1	6	-3	8660	-8747	3	7	3	877	923	8	7	-4	2303	-2255
3	5	1	2496	1949	2	5	-2	915	-992	1	6	2	1123	1133	2	6	-3	992	1052	4	7	3	4531	4335	9	7	-4	1056	-970
4	5	1	2303	2456	3	5	-2	00	333	2	6	2	2401	2184	3	6	-3	3078	2662	5	7	3	1332	-1266	10	7	-4	1377	-1447
5	5	1	00	141	4	5	-2	1912	-1767	3	6	2	00	334	4	6	-3	1572	-1223	6	7	3	1114	1017	11	7	-4	2846	-2933
6	5	1	3872	3900	5	5	-2	5534	5769	4	6	2	00	597	5	6	-3	6978	7131	7	7	3	00	302	12	7	-4	993	820
7	5	1	2341	2369	6	5	-2	1312	1132	5	6	2	00	122	6	6	-3	1287	1189	8	7	3	4053	-4322	13	7	-4	537	472
8	5	1	1302	1346	7	5	-2	3083	3109	6	6	2	2659	2778	7	6	-3	3947	4126	9	7	3	00	-390	1	7	-5	1318	-1031
9	5	1	00	17	8	5	-2	2576	2719	7	6	2	1207	879	8	6	-3	1223	-1379	10	7	3	1469	-1334	2	7	-5	4907	-4903
10	5	1	2639	-2718	9	5	-2	2331	-2316	8	6	2	3459	-3788	9	6	-3	00	267	11	7	3	1301	1203	3	7	-5	00	360
11	5	1	1937	-2235	10	5	-2	00	723	9	6	2	00	-364	10	6	-3	00	-08	12	7	3	1105	873	4	7	-5	1147	1168
12	5	1	00	62	11	5	-2	795	-753	10	6	2	00	-385	11	6	-3	3387	-3500	1	7	4	2968	-2608	5	7	-5	00	-334
13	5	1	1487	-1539	12	5	-2	2580	-2560	11	6	2	00	523	12	6	-3	00	204	2	7	4	1027	-745	6	7	-5	4017	4168
14	5	1	548	837	13	5	-2	876	932	12	6	2	2104	2087	13	6	-3	00	396	3	7	4	3447	3156	7	7	-5	00	-389
1	-5	2	5912	-5937	14	5	-2	775	764	13	6	2	00	-278	14	6	-3	00	-152	4	7	4	857	807	8	7	-5	1138	1236
2	5	2	6134	-6379	1	5	-3	3031	-2956	1	6	3	5712	-5873	1	6	-4	1268	-1211	5	7	4	533	654	9	7	-5	00	-283
3	5	2	5076	5277	2	5	-3	6290	-6582	2	6	3	2087	-1877	2	6	-4	1266	-1182	6	7	4	2388	-2342	10	7	-5	1388	-1497
4	5	2	1892	-1472	3	5	-3	790	-948	3	6	3	2378	2772	3	6	-4	722	460	7	7	4	1107	-895	11	7	-5	00	-459
5	5	2	3430	3058	4	5	-3	4871	4732	4	6	3	623	-687	4	6	-4	3530	3513	8	7	4	1619	-1754	12	7	-5	828	-844
6	5	2	3393	3433	5	5	-3	2242	-1904	5	6	3	3408	3284	5	6	-4	00	130	9	7	4	1077	-1050	1	7	-6	00	-392
7	5	2	2303	-1939	6	5	-3	2417	2335	6	6	3	881	551	6	6	-4	6168	6036	10	7	4	1225	1176	2	7	-6	860	894
8	5	2	3009	3157	7	5	-3	1424	1358	7	6	3	2464	-2091	7	6	-4	00	536	11	7	4	831	776	3	7	-6	2191	-2255
9	5	2	903	-1245	8	5	-3	1176	1318	8	6	3	813	786	8	6	-4	697	-411	12	7	4	796	747	4	7	-6	00	383
10	5	2	00	-29	9	5	-3	1946	1523	9	6	3	2647	-2614	9	6	-4	920	787	1	7	5	3332	2990	5	7	-6	3705	3729
11	5	2	00	511	10	5	-3	2902	-2721	10	6	3	00	33	10	6	-4	2270	-2068	2	7	5	4650	4531	6	7	-6	1303	1368
12	5	2	1705	-2078	11	5	-3	00	225	11	6	3	00	822	11	6	-4	632	-710	3	7	5	1024	1032	7	7	-6	2084	2413
13	5	2	1042	955	12	5	-3	802	-876	12	6	3	00	413	12	6	-4	2302	-1876	4	7	5	1347	1242	8	7	-6	1217	-1104
1	5	3	1956	-1599	13	5	-3	1993	-1905	13	6	3	2530	2718	13	6	-4	591	652	5	7	5	3329	-3421	9	7	-6	00	-289
2	5	3	3676	3385	14	5	-3	00	272	1	6	4	5067	-4859	1	6	-5	2428	-2506	6	7	5	1754	-1709	10	7	-6	00	-182
3	5	3	2559	-2239	1	5	-4	3431	-3225	2	6	4	3943	3667	2	6	-5	00	-133	7	7	5	1474	1362	11	7	-6	2151	-2362
4	5	3	3495	3653	2	5	-4	3412	-3123	3	6	4	989	-1160	3	6	-5	3391	-3555	8	7	5	1952	-2190	1	7	-7	2004	-2106
5	5	3	882	799	3	5	-4	3250	-3169	4	6	4	3194	2953	4	6	-5	1608	-1689	9	7	5	00	-166	2	7	-7	796	-690
6	5	3	745	-951	4	5	-4	5570	-5503	5	6	4	2290	2367	5	6	-5	3491	3594	10	7	5	992	993	3	7	-7	1255	1276
7	5	3	3245	3331	5	5	-4	4601	4732	6	6	4	1457	-1205	6	6	-5	1145	991	11	7	5	499	539	4	7	-7	00	-235
8	5	3	3543	-4001	6	5	-4	3426	3352	7	6	4	00	625	7	6	-5	4132	4078	1	7	6	445	481	5	7	-7	363	319
9	5	3	2699	-2769	7	5	-4	2390	2145	8	6	4	4581	-4708	8	6	-5	1047	-920	2	7	6	00	-383	6	7	-7	1322	1163
10	5	3	1143	-1369	8	5	-4	1820	1557	9	6	4	00	-557	9	6	-5	1873	-1925	3	7	6	2464	2434	7	7	-7	00	-378
11	5	3	1195	-1124	9	5	-4	00	-281	10	6	4	00	-554	10	6	-5	1762	1534	4	7	6	1068	-1122	8	7	-7	1582	1501
12	5	3	3617	3658	10	5	-4	00	-00	11	6	4	00	-217	11	6	-5	3456	-3419	5	7	6	00	282	9	7	-7	00	-558
13	5	3	00	-83	11	5	-4	2568	-2437	12	6	4	988	970	12	6	-5	622	-721	6	7	6	746	-487	10	7	-7	1309	-1186
1	5	4	2996	-2784	12	5	-4	896	-797	1	6	5	307	120	13	6	-5	551	-566	7	7	6	3177	-3523	1	7	-8	1561	-1468
2	5	4	1960	-2197	13	5	-4	731	-861	2	6	5	00	-297	1	6	-6	779	789	8	7	6	1178	1004	2	7	-8	616	-951
3	5	4	3474	3452	1	5	-5	1202	1119	3	6	5	3526	3695	2	6	-6	2644	-2880	9	7	6	2141	-2291	3	7	-8	2721	-2657
4	5	4	4579	4428	2	5	-5	2997	-2681	4	6	5	623	-842	3	6	-6	00	187	10	7	6	1318	1741	4	7	-8	696	438
5	5	4	2345	2039	3	5	-5	840	-981	5	6	5	620	874	4	6	-6	582	750	1	7	7	00	-610	5	7	-8	865	982
6	5	4	2834	2822	4	5	-5	1291	1103	6	6	5	310	-619	5	6	-6	2408	-2229	2	7	7	2882	2771	6	7	-8	602	473
7	5	4	4119	-4081	5	5	-5	2687	-2316	7	6	5	2938	-3177	6	6	-6	2938	2974	3	7	7	565	-644	7	7	-8	824	635
8	5	4	00	60	6	5	-5	3947	3784	8	6	5	853	781	7	6	-6	00	99	4	7	7	2145	2134	8	7	-8	00	-306
9	5	4	2136	-2206	7	5	-5	616	611	9	6	5	1940	-2213	8	6	-6	1051	1042	5	7	7	2106	-2218	9	7	-8	00	258
10	5	4	1512	-1567	8	5	-5	1600	1157	10	6	5	1260	-1472	9	6	-6	00	261	6	7	7	1614	-1773	1	7	-9	448	-517
11	5	4	1199	1324	9	5	-5	1646	1589	11	6	5	1397	1535	10	6	-6	1812	-1757	7	7	7	00	-404	2	7	-9	1061	-965
12	5	4	00	-587	10	5	-5	1813	-1863	1	6	6	2080	2128	11	6	-6	00	-348	8	7	7	2003	-2043	3	7	-9	442	-292
1	5	5	4546	-4598	11	5	-5	1357	-1221	2	6	6	3560	3768	12	6	-6	2339	-2159	1	7	8	1952	1912	4	7	-9	771	-694
2	5	5	2145	2119	12	5	-5	2398	-2047	3	6	6	763	-847	1	6	-7	00	-87	2	7	8	899	961	5	7	-9	525	512
3	5	5	688	606	13	5	-5	1712	-1371	4	6	6	00	374	2	6	-7	00	-420	3	7	8	1592	1717	6	7	-9	966	1084
4	5	5	3713	3919	1	5	-6	2832	-2733	5	6	6	00	130	3	6	-7	1250	-1066	4	7	8	613	-722					

Table 3. *Interatomic distances (shorter than 3·35 Å) and bond angles*

Standard errors are in parentheses.

Small letters indicate symmetry transformations or cell translations:

a: $\bar{x}, \bar{y}, \bar{z}$; b: $1-x, \bar{y}, 1-z$; c: $\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$; d: $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$; e: $x, y, 1+z$.

(*a*) Distances within a dimeric molecule

Polyhedron around the copper atom			
Cu(1)–O(2)	1·968 (5)	O(3)—O(4)	2·873 (7)
Cu(1)–O(3)	1·951 (5)	O(3)—O(7)	2·653 (7)
Cu(1)–O(4)	1·966 (5)	O(2*a*)–O(2)	2·773 (6)
Cu(1)–O(7)	1·967 (5)	O(2*a*)–O(3)	3·110 (7)
Cu(1)–O(2*a*)	2·439 (6)	O(2*a*)–O(4)	3·305 (7)
O(2)—O(4)	2·862 (6)	O(2*a*)–O(7)	3·172 (7)
O(2)—O(7)	2·793 (7)		
Nitrate groups			
N(12)–O(4)	1·316 (9)	N(13)–O(7)	1·304 (8)
N(12)–O(5)	1·222 (9)	N(13)–O(8)	1·221 (8)
N(12)–O(6)	1·227 (10)	N(13)–O(9)	1·234 (9)
O(4)—O(5)	2·168 (8)	O(7)—O(8)	2·164 (10)
O(4)—O(6)	2·198 (10)	O(7)—O(9)	2·158 (9)
O(5)—O(6)	2·154 (10)	O(8)—O(9)	2·184 (7)
Pyridine *N*-oxide molecules			
N(10)–O(2)	1·362 (7)	N(11)–O(3)	1·361 (7)
N(10)–C(14)	1·337 (10)	N(11)–C(19)	1·369 (9)
N(10)–C(18)	1·352 (10)	N(11)–C(23)	1·325 (11)
C(14)–C(15)	1·389 (10)	C(19)–C(20)	1·410 (12)
C(15)–C(16)	1·376 (12)	C(20)–C(21)	1·391 (14)
C(16)–C(17)	1·399 (11)	C(21)–C(22)	1·407 (12)
C(17)–C(18)	1·400 (11)	C(22)–C(23)	1·402 (12)
Other distances shorter than 3·35 Å			
Cu(1)–N(10)	2·880 (4)	O(3)—O(6)	3·227 (9)
Cu(1)–N(11)	2·875 (6)	O(3)—O(12)	3·267 (7)
Cu(1)–N(12)	2·758 (6)	O(4)—N(10)	3·026 (7)
Cu(1)–N(13)	2·755 (6)	O(4)—N(11)	2·988 (6)
Cu(1)–O(6)	2·802 (7)	O(4)—C(14)	3·062 (8)
Cu(1)–O(8)	2·750 (7)	O(4)—C(23)	3·069 (10)
O(2)—O(3*a*)	3·110 (7)	O(8)—C(18)	3·143 (9)
O(2)—O(8)	3·182 (9)	N(11)–N(12)	3·112 (6)
O(2)—N(13)	3·309 (8)		

(*b*) Distances between dimeric molecules

O(3*e*)–C(17*c*)	3·291 (12)	O(9*c*)—C(19)	3·099 (12)
O(5)—C(21*b*)	3·307 (10)	O(9*e*)—C(15)	3·312 (11)
O(5)—C(17*c*)	3·324 (11)	O(9*e*)—C(16)	3·154 (10)
O(6)—C(22*d*)	3·289 (13)	O(9*e*)—C(17*c*)	3·332 (11)
O(8*e*)–C(16*c*)	3·124 (12)	N(13*e*)–C(17*c*)	3·202 (9)

(*c*) Bond angles (in degrees ±0·5°)

In nitrate groups			
O(4)–N(12)–O(5)	117·3	O(7)–N(13)–O(8)	117·9
O(4)–N(12)–O(6)	119·5	O(7)–N(13)–O(9)	116·4
O(5)–N(12)–O(6)	123·1	O(8)–N(13)–O(9)	125·6
In pyridine *N*-oxide molecules			
O(2)—N(10)–C(14)	119·1	O(3)—N(11)–C(19)	116·9
O(2)—N(10)–C(18)	116·8	O(3)—N(11)–C(23)	120·1
C(14)–N(10)–C(18)	124·1	C(19)–N(11)–C(23)	122·9
N(10)–C(14)–C(15)	118·0	N(11)–C(19)–C(20)	118·2
C(14)–C(15)–C(16)	120·7	C(19)–C(20)–C(21)	119·1
C(15)–C(16)–C(17)	119·9	C(20)–C(21)–C(22)	121·4
C(16)–C(17)–C(18)	118·3	C(21)–C(22)–C(23)	116·6
C(17)–C(18)–N(10)	119·0	C(22)–C(23)–N(11)	121·9
In a polyhedron around the copper atom			
O(2)–Cu(1)–O(4)	91·8	O(2*a*)–Cu(1)–O(2)	77·1
O(2)–Cu(1)–O(7)	90·4	O(2*a*)–Cu(1)–O(3)	89·5
O(3)–Cu(1)–O(4)	94·3	O(2*a*)–Cu(1)–O(4)	96·5
O(3)–Cu(1)–O(7)	85·2	O(2*a*)–Cu(1)–O(7)	91·5

Table 3 (*cont.*)

Angles on oxygen atoms

Cu(1)–O(2)–N(10)	118·6	Cu(1)—O(7)–N(13)	113·2
Cu(1)–O(3)–N(11)	119·4	Cu(1)—O(2)–Cu(1*a*)	102·9
Cu(1)–O(4)–N(12)	112·8	Cu(1*a*)–O(2)–N(10)	124·1

tallography (1962). The real, but not the imaginary part of the anomalous dispersion correction for the copper atom was applied. The attempt to locate hydrogen atoms by means of a difference-Fourier synthesis and to refine the calculated hydrogen positional parameters (with C–H ~ 1 Å) was unsuccessful.

Three-dimensional Fourier syntheses (Sly, Shoemaker & van den Hende, 1962) and several cycles of least-squares refinement (Busing, Martin & Levy, 1962) were carried out on the Washington State University IBM 709 computer. The least-squares refinement (Prewitt, 1964) was continued on the IBM 7040 computer at the University of Technology (Technische Hochschule), Vienna. Final refinement (Polić, 1968), the atomic distances and angles, and the mean planes (Zelenko, 1968) were computed on the Institute Ruder Bošković CAE 90·40 computer.

The final atomic coordinates and thermal parameters together with their estimated standard deviations are shown in Table 1, and the observed and calculated structure factors in Table 2. The bond lengths and angles together with other selected interatomic distances are given in Table 3 and Fig. 1.

Description and discussion of the structure

Fig. 2 represents the projection of the structure along [001]: atoms in the nitrate and pyridine *N*-oxide groups are connected by full heavy lines, as are copper–oxygen bonds; the coordination polyhedra of the copper atoms are drawn as full and broken lines. The only atoms numbered are those whose distances and angles are mentioned in Table 3.

The crystal is composed of discrete molecules which are dimeric, $[Cu(C_5H_5NO)_2(NO_3)_2]_2$. A three-dimensional arrangement is achieved by a fairly efficient molecular packing but no specific intermolecular interactions are indicated. The shortest distances correspond to normal van der Waals interactions. Dimeric species are placed in the monoclinic unit cell astride centres of symmetry at 0,0,0, and $\frac{1}{2},\frac{1}{2},\frac{1}{2}$, and so are composed of two centrosymmetrically related complex monomers. These two monomers are connected in such a way that two copper atoms [*e. g.* Cu(1) and Cu(1*a*) of Fig. 2] are bridged by two oxygen atoms [*e.g.* O(2) and O(2*a*)] which belong to two symmetrically equivalent pyridine *N*-oxide groups. In the monomeric unit the copper

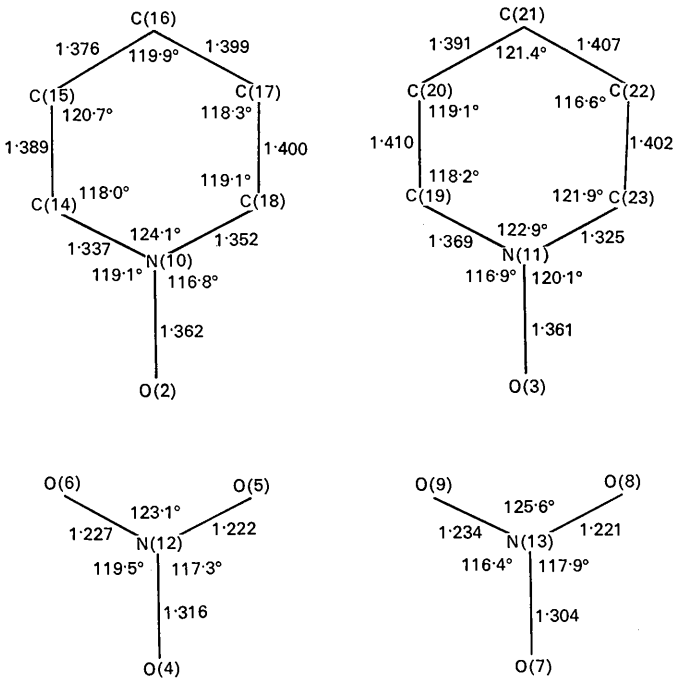

Fig. 1. Bond distances and angles in pyridine *N*-oxide and nitrate groups.

atom is at a centre of a nearly coplanar rectangular arrangement of four oxygen atoms which belong to two nitrate and two pyridine *N*-oxide groups. These groups occupy *trans* positions to one another in the coordination sphere of the copper atom. The structural arrangement is such that one of the four oxygen atoms mentioned [O(2)] participates also in the coordination sphere of the copper atom from the centrosymmetrically related monomer, and *vice versa*. Thus each copper atom has a distorted tetragonal pyramidal environment of five oxygen atoms. Short and nearly coplanar bonds (mean value, 1·963 Å) are directed from the copper atom Cu(1) to four oxygen atoms: O(2) from C_5H_5NO at 1·968 Å, O(3) from C_5H_5NO at 1·951 Å, O(4) from NO_3 at 1·966 Å and O(7) from NO_3 at 1·967 Å; the fifth (long) bond (2·439 Å) is approximately perpendicular to the plane of those four oxygen atoms and directed to the bridging oxygen atom [O(2*a*)]. Next closest atoms (to the copper atom), on the opposite side the pyramidal apex, are two oxygen atoms [O(6) at 2·802 Å and O(8) at 2·750 Å] and two nitrogen atoms [N(12) at 2·758 Å and N(13) at 2·755 Å] from two NO_3 groups of the same monomer. Their distances from the copper atom are too great to be considered in the copper coordination sphere. This conclusion is partly supported by a significant difference between N–O bond lengths in the NO_3 groups: bonds directed to the oxygen atoms [O(4) and O(7)] from the copper polyhedron are about 0·08 Å longer than the remaining nitrogen–oxygen bonds. It follows that nitrate groups act as monodentate ligands, presumably because of a steric hindrance which prevents one of them from completing, as a bidentate ligand, an octahedral copper coordination. In spite of being structurally independent both NO_3 groups coordinate with an equal Cu–O–N angle of 113°; this value is closer to the tetrahedral (109·5°) than to the trigonal angle (120°), making sp^3 hybridization more likely than sp^2 on the O(4) and O(7) atoms.

There is no metal–metal interaction in the present structure. The closest Cu–Cu approach is 3·458 Å and this occurs within a dimeric unit; it is greater than the separation in other bridged copper complexes (Porai-Koshits & Gilinskaya, 1966; Muetterties & Schunn, 1966; Sager, Williams & Watson, 1967).

The equation of the mean plane through the copper [Cu(1)] and four oxygen atoms [O(2), O(3), O(4) and

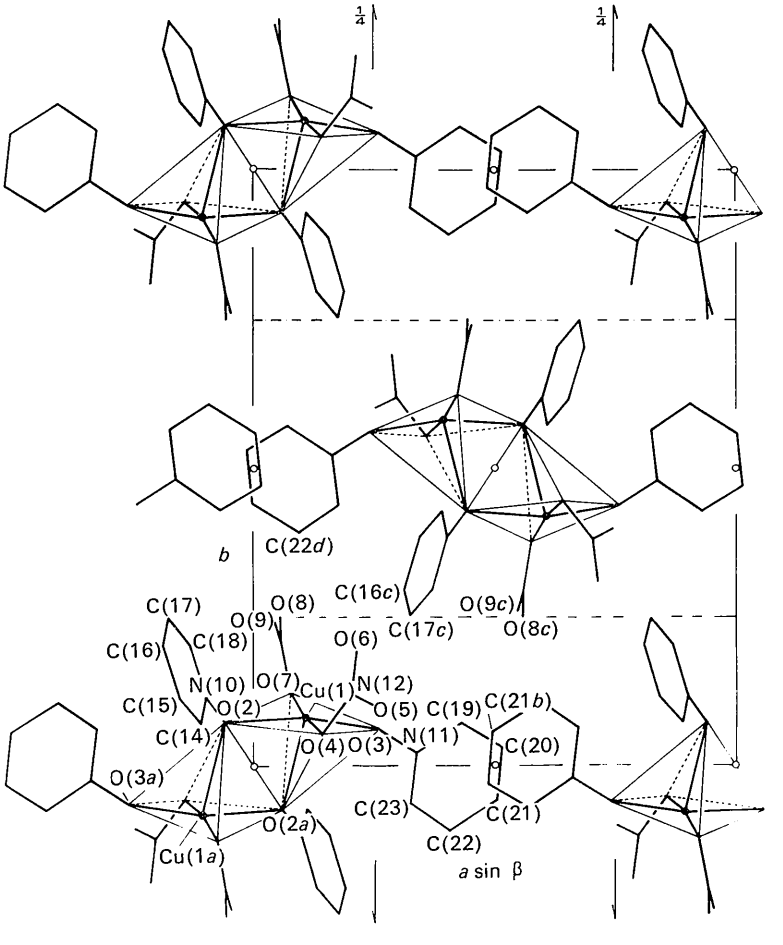

Fig. 2. The projection of the structure along [001].

O(7)], together with the distances of the atoms from this plane, are shown in Table 4. These data indicate a small tetrahedral distortion of a planar configuration of four oxygen atoms: the diagonal O(2)–O(3) of a quadrilateral does not intersect the second O(4)–O(7) diagonal; they are separated by 0·36 Å from one another. Table 4 also contains the equations of the mean planes through the atoms of the pyridine *N*-oxide and nitrate groups together with the deviations of the atoms from these planes; these deviations are so small (and the sums of the bond angles are very close to 360° or 720°) that all nitrate and pyridine *N*-oxide groups may be considered as planar.

The pyridine rings are twisted out of the Cu–O–N planes. The angle between the normal to the copper–oxygens plane and the normal to either pyridine ring is $64 \pm 0{\cdot}5°$, and the angle between the normals to two pyridine rings is $82 \pm 0{\cdot}5°$. From the lack of coincidence of the Cu–O–N plane and the pyridine ring it may be concluded that the interaction between the *p* orbital of the oxygen atom and the π system of the pyridine group was of little importance in determining the mutual orientation of ligands and the equatorial plane of the copper polyhedron.

Taking into account the probable error in the positional parameters, the differences between the corresponding bond lengths within two independent pyridine *N*-oxide molecules are not significant; mean values of nitrogen–oxygen (1·361 Å), nitrogen–carbon (1·345 Å) and carbon–carbon (1·396 Å) distances are in good agreement with published data (Horrocks, Templeton & Zalkin, 1968; Sager, Williams & Watson, 1967; Tsoucaris, 1961). Both pyridine *N*-oxide molecules coordinate with the same Cu–O–N angle of $119 \pm 0{\cdot}5°$ This value is very close to 120° and may suggest, as in the structure of $Ni(C_5H_7O_2)_2(C_5H_5NO)_2$ (Horrocks *et al.*, 1968), an sp^2 hybridization on oxygen, at least for a non-bridging oxygen atom; for a bridging oxygen atom [O(2)] the third weakest O(2)–Cu(1*a*) bond of 2·439 Å is out of the Cu(1)–O(2)–N(10) plane, with a corresponding reduction of the Cu(1)–O(2)–Cu(1*a*) angle to 103°.

In the crystal structure of $[(C_5H_5NO)CuCl_2]_2$ (Schafer, Morrow & Smith, 1965; Sager, Williams & Watson, 1967), which is also an oxygen bridged dimer, two Cu–O bonds are of similar strength (with lengths of 1·979 and 2·036 Å) and the distribution of 2 Cl and 2 O atoms is distorted square planar. Comparing this with the structure of 2:1 complex copper(II) nitrate it follows that in the formation of the present structure four strong Cu–O bonds were equally distributed among four structurally independent ligands (two C_5H_5NO and two NO_3 groups) which remain at the disposal of each copper ion, while the fifth (more remote) apical position was left for bridging purposes.

Crystal structures with a 4+1 coordination of the Cu^{II} ion were reviewed by Zemann (1961) and later by Muetterties & Schunn (1966). In all of them, independent of whether the formation of a pentacoordinate structure was achieved by dimerization or not, the fifth Cu–O bond is about 0·4 Å longer than the bonds from the equatorial plane; our data are in agreement with this. In the recently determined structure of $Cu(NO_3)_2.2CH_3CN$ (Duffin, 1968), the fifth Cu–O bond is a little shorter (2·31 Å). The values obtained for short copper–oxygen bonds fall within the range of expected values of 1·9 to 2·0 Å.

The shortest distances between oxygen and carbon atoms from adjacent dimeric molecules [O(9*c*)–C(19) = 3·10, O(8*e*) – C(16*c*) = 3·12, O(9*e*) – C(16) = 3·15 Å] are too long, which may indicate some significant intermolecular interactions which could account for hydrogen bonding. There are no additional atoms of closer approach, although the possible positions of some of the hydrogen atoms fall within this range.

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Table 4. *Least-squares planes and deviations of atoms from the planes*

x, *y*, *z* are fractional atomic coordinates.

Atoms and their distances (Å) from the plane	Equation of plane (fractional form)
Cu(1), −0·0348; O(2), 0·1852; O(3), 0·1917; O(4), −0·1622; O(7), −0·1799	$x/1{\cdot}21082 + y/0{\cdot}08960 + z/0{\cdot}68208 = 1$
O(4), 0·0005; O(5), 0·0005; O(6), 0·0005; N(12), −0·0015	$x/0{\cdot}01526 + y/-0{\cdot}03135 + z/-0{\cdot}04096 = 1$
O(7), 0·0054; O(8), 0·0063; O(9), 0·0062; N(13), −0·0179	$x/0{\cdot}10274 + y/0{\cdot}53835 + z/-4{\cdot}89626 = 1$
O(2), −0·0123; N(10), 0·0118; C(14), 0·0080; C(15), −0·0082; C(16), 0·0006; C(17), −0·0049; C(18), 0·0050	$x/-0{\cdot}01600 + y/-0{\cdot}03878 + z/-0{\cdot}08116 = 1$
O(3), −0·0316; N(11), 0·0252; C(19), 0·0143; C(20), −0·0011; C(21), −0·0201; C(22), −0·0026; C(23), 0·0158	$x/0{\cdot}24024 + y/-0{\cdot}34687 + z/-0{\cdot}24250 = 1$

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The Crystal and Molecular Structure of the Triethylammonium Salt of Cyclic Uridine-3′, 5′-Phosphate

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The triethylammonium salt of cyclic uridine-3′,5′-phosphate forms monoclinic crystals of space group $P2_1$, with $a=15{\cdot}51$, $b=11{\cdot}674$, $c=11{\cdot}044$ Å, $\beta=98{\cdot}6°$; there are two molecules of the salt in the asymmetric unit. Three-dimensional diffractometer data were collected with Cu $K\alpha$ radiation. The structure was determined by Patterson and Fourier syntheses in conjunction with the tangent formula, and refined by block-diagonal least squares. The final R value was 5·8% for the 2712 reflections used in the refinement and 6·5% for all 2866 data.

The nucleotides pack with the pyrimidine rings nearly normal to **c**, arranged in hydrogen-bonded ribbons with the base planes near $z=0$ and $z=\frac{1}{2}$. Adjacent anions are linked with a hydrogen bond between the C(2′) hydroxyl group of the ribose and a phosphate oxygen atom.

The geometry of the molecule is in accord with previous studies of nucleotides. The C(4)–O(4) carbonyl bond on the uracil ring is bent towards N(3), as was found in most earlier studies. The torsion angles of the bases with respect to the sugars are $-77°$ and $-58°$, both in the *anti* range. The ribose conformation is C(3′)-*endo* in both anions. The conformation about the C(4′)–C(5′) bond is *trans-gauche* rather than *gauche-gauche* because of the geometrical requirements for forming the cyclic phosphate.

Introduction

Cyclic uridine-3′,5′-phosphate (3′,5′-UMP) is an interesting member of the class of mononucleotides. Uridine is a component of ribonucleic acid, and cyclic 2′,3′-phosphate esters are intermediates in the breakdown of RNA. Cyclic 3′,5′-ribonucleotides were used by Smith & Khorana (1959) in the synthesis of the specific 3′,5′-phosphodiester linkage of a dinucleotide; some of these nucleotides (especially cyclic adenosine-3′,5′-phosphate) are physiologically active. No detailed structure determinations have been reported for cyclic nucleotides, and this study was undertaken to elucidate the crystal structure of 3′-5′-UMP. Preliminary notes have appeared for this structure (Coulter, 1968) and for the adenosine analog, 3′,5′-AMP (Watenpaugh, Dow, Jensen & Furberg, 1968). The chemical structure of 3′,5′-UMP is given in Fig. 1 with the conventional numbering of the atoms.

Experimental

The triethylammonium salt of 3,′5′-UMP was prepared and crystallized from an ethanol–water solution by Dr D. H. Rammler. The crystals were tabular plates, flattened on (001). They tended to fracture easily along (001). A roughly cube-shaped crystal 0·2 mm on a side was aligned about **a*** and used for the determination of cell constants and the initial data collection. To this end, the specimen was placed on a General Electric goniostat with a single-crystal orienter and scintillation counter, and irradiated with nickel-filtered copper radiation. Cell dimensions were derived by a least-squares fit of 31 careful 2θ measurements, and were found to be:

$$a=15{\cdot}51\pm0{\cdot}01\ b=11{\cdot}674\pm0{\cdot}007,\ c=11{\cdot}044\pm0{\cdot}006\ \text{Å}$$
$$\beta=98{\cdot}6°\pm0{\cdot}1;\ \text{Cu}\ K\alpha:\lambda=1{\cdot}5418\ \text{Å}.$$

Systematic extinctions ($0k0$, $k=2n+1$) suggested space