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The Crystal Structure of the Bis-(5,5′-diethylbarbiturato)bispicoline Complex of Zinc(II)

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The crystal structure of the title compound has been determined from single-crystal X-ray diffraction data collected on a four-circle diffractometer. The analysis was carried out with 3343 independent reflexions by Patterson and Fourier methods and the structure refined by full-matrix least-squares methods to an R of 0·029. The complex crystallizes in the space group $P\bar{1}$ with $a=13{\cdot}013$ (5), $b=11{\cdot}748$ (5), $c=10{\cdot}842$ (5) Å, $\alpha=114{\cdot}4$ (2), $\beta=90{\cdot}9$ (2), $\gamma=90{\cdot}3$ (2)°, $Z=2$. The zinc atom is tetrahedrally bonded to the deprotonated nitrogen atoms of the barbital anions [Zn–N, 2·006 (3) and 1·987 (3) Å] and to the nitrogen atoms of the picolines [Zn–N, 2·095 (3) and 2·069 (3) Å]. The molecules are linked by N–H···O hydrogen bonds.

Introduction

The structure of the title compound is the fourth in our series of investigations of compounds of the general formula M(II) $(\text{barb})_2\text{L}_2$, where M is a transition metal, barb is the anion of a substituted barbituric acid and L is an organic base, such compounds being important in the clinical detection and identification of drugs. The first X-ray determination on compounds of this type was carried out by Wang & Craven (1971) who elucidated the structures of the bis-(5,5-diethylbarbiturato)bisimidazole complexes of cobalt(II) and zinc(II). We have subsequently reported the crystal structures of the bis-(5,5′-diethylbarbiturato)bispyridine complex of copper(II) (Caira, Fazakerley, Linder & Nassimbeni, 1973), the bis-(5,5′-diethylbarbiturato)-bispicoline dihydrate complex of copper(II) (Fazakerley, Linder, Nassimbeni & Rodgers, 1974*a*) and the bis-[5-allyl-5-(2-bromoallyl)barbiturato]bispyridine dihydrate complex of copper(II) (Fazakerley, Linder, Nassimbeni & Rodgers, 1974*b*).

Levi & Hubley (1956) have suggested on the basis of infrared spectral data on a series of complexes of the type $\text{Cu(barb)}_2(\text{pyridine})_2$ that the barbiturate ligand is bound to the copper through an oxygen atom. However, all the above crystal structure analyses have shown the coordinating site to be a deprotonated nitrogen atom.

Hydrogen bonding has been a prominent feature in the crystal structures of barbiturates and their salts (Berking & Craven, 1971; Craven, Vizzini & Rodrigues, 1969) as well as the complexes mentioned above.

The present analysis was undertaken as part of a general programme established to ascertain the exact coordination site on the barbiturate and to investigate the nature of any hydrogen bonding which might prevail.

Experimental

The complex was formed by mixing dilute aqueous solutions of ZnSO_4, sodium barbital and β-picoline in the molar ratio 1:2:2 at room temperature. After a few days, 2 drops of 1*M* NaOH were added. Large, block-like transparent crystals formed after a further 24 hours. Microanalysis showed that they had the composition $\text{ZnC}_{28}\text{H}_{36}\text{N}_6\text{O}_6$ corresponding to the formulation Zn(II) (5,5′-diethylbarbiturato)$_2$(β-picoline)$_2$.

A single crystal of dimensions 0·25 × 0·25 × 0·375 mm was selected for the X-ray investigation. Its density was determined with a density gradient column containing the components *m*-xylene ($\varrho=0{\cdot}86$ g cm^{-3}) and carbon tetrachloride ($\varrho=1{\cdot}60$ g cm^{-3}), which was precalibrated with aqueous caesium chloride solutions. The lattice constants (Table 1) were obtained from a least-squares analysis of the settings of 25 reflexions measured on a four-circle diffractometer with Mo $K\alpha$ radiation ($\lambda=0{\cdot}7107$ Å).

Table 1. *Crystal data*

Molecular formula	$\text{ZnC}_{28}\text{H}_{36}\text{N}_6\text{O}_6$
Molecular weight	617·4
Space group	Triclinic, $P\bar{1}$
$a=13{\cdot}013$ (5) Å	$V=1509{\cdot}16$ Å^3
$b=11{\cdot}748$ (5)	$D_m=1{\cdot}368$ g cm^{-3}
$c=10{\cdot}842$ (5)	$D_c=1{\cdot}341$ g cm^{-3} for $Z=2$
$\alpha=114{\cdot}4$ (2)°	$\mu(\text{Mo }K\alpha)=8{\cdot}92$ cm^{-1}
$\beta=90{\cdot}9$ (2)	$F(000)=648$
$\gamma=90{\cdot}3$ (2)	

The intensities were collected at the National Physics Research Laboratory of the CSIR on a Philips PW 1100 computer-controlled four-circle diffractometer operating in the ω-scan mode (scan width = 1·20° and scan speed = 0·04°s^{-1}).

With Zr-filtered Mo $K\alpha$ radiation 3687 reflexions up to $2\theta=44°$ were measured. A reflexion was taken as being above background if $I_{\text{rel}}>1{\cdot}65\sigma(I_{\text{rel}})$. 344 reflexions did not satisfy this criterion and were omitted as unobserved. Three reference reflexions were measured after every 57 reflexions to monitor stability of operation and crystal decomposition. The variation in intensity of a reference reflexion was observed to be less than 2% of its mean value. Lorentz–polarization corrections were applied. However, absorption corrections were ignored as the variation in μR for the crystal selected was between 0·11 and 0·20 with corresponding A^* values of 1·18 and 1·40 for the θ range scanned. This was regarded as insignificant.

Solution and refinement of the structure

The position of the zinc atom was determined from a Patterson map. From the subsequent Fourier synthesis all 41 non-hydrogen atoms of the molecule were located. Scattering factors were those of Cromer & Mann (1968). The zinc was treated as Zn^0 and the anomalous dispersion correction ($\Delta f' = 0{\cdot}3$) was applied. Each reflexion was assigned unit weight. Before refinement R was 0·245. After three cycles, the isotropic refinement [*CRYLSQ*: X-RAY system (1972)] terminated at an R of 0·089. Anisotropic temperature factors were then introduced and after 2 cycles of refinement R dropped to 0·054. For the anisotropic refinement the thermal parameters were of the form

$$T=\exp\left[-2\pi^2(U_{11}h^2a^{*2}+U_{22}k^2b^{*2}+U_{33}l^2c^{*2}\right.$$
$$\left.+2U_{12}hka^*b^*+2U_{13}hla^*c^*+2U_{23}klb^*c^*)\right].$$

All 36 hydrogen atoms were located in difference syntheses and were assigned the isotropic temperature factors of the atoms to which they were bonded. After a further four cycles of non-hydrogen refinement, the average e.s.d. in the positional and anisotropic temperature factors was about 12 times the average parameter shift. After the last refinement cycle in which the hydrogen atomic positions were varied, the average e.s.d. in these parameters was about three times the average parameter shift. The last cycle of refinement yielded an R of 0·029. A final difference map was practically featureless. The positional and thermal parameters for the non-hydrogen atoms are listed in Table 2 and the positional parameters for the hydrogen atoms in Table 3. Table 4 lists the observed and calculated structure factors.

Table 2. *Non-hydrogen atoms: fractional atomic coordinates and their e.s.d.'s* ($\times 10^4$) *and anisotropic temperature factors and their e.s.d.*'s ($\times 10^3$)

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Zn	2266 (0·3)	2347 (0·3)	3761 (0·3)	23 (0·2)	23 (0·2)	25 (0·2)	−3 (0·1)	−2 (0·1)	11 (0·2)
N(1)	1968 (2)	2109 (2)	5449 (2)	20 (1)	28 (1)	24 (1)	−9 (1)	−6 (1)	14 (1)
C(2)	1208 (2)	1269 (3)	5331 (3)	21 (2)	28 (2)	27 (2)	−3 (1)	−4 (1)	14 (1)
N(3)	1087 (2)	849 (2)	6340 (3)	29 (1)	43 (2)	35 (2)	−19 (1)	−11 (1)	25 (1)
C(4)	1703 (2)	1168 (3)	7474 (3)	34 (2)	47 (2)	36 (2)	−8 (2)	−6 (2)	26 (2)
C(5)	2554 (2)	2126 (3)	7681 (3)	30 (2)	44 (2)	30 (2)	−14 (2)	−14 (1)	20 (2)
C(6)	2581 (2)	2623 (3)	6581 (3)	27 (2)	32 (2)	29 (2)	−7 (1)	−2 (1)	13 (1)
C(7)	2405 (3)	3228 (4)	9072 (4)	80 (3)	62 (3)	33 (2)	−23 (2)	−15 (2)	20 (2)
C(8)	1407 (5)	3928 (5)	9192 (5)	134 (5)	83 (4)	56 (3)	29 (3)	23 (3)	14 (3)
C(9)	3594 (3)	1507 (4)	7678 (4)	37 (2)	80 (3)	73 (3)	−9 (2)	−17 (2)	53 (3)
C(10)	3812 (3)	363 (5)	6412 (6)	58 (3)	103 (4)	106 (4)	33 (3)	17 (3)	66 (4)
O(2)	614 (2)	888 (2)	4349 (2)	32 (1)	53 (1)	35 (1)	−21 (1)	−14 (1)	25 (1)
O(4)	1571 (2)	702 (3)	8269 (3)	64 (2)	97 (2)	61 (2)	−37 (2)	−23 (1)	63 (2)
O(6)	3183 (2)	3471 (2)	6707 (2)	52 (2)	55 (2)	41 (1)	−36 (1)	−16 (1)	25 (1)
N(11)	3715 (2)	3051 (2)	3593 (2)	23 (1)	30 (1)	30 (1)	−4 (1)	−1 (1)	12 (1)
C(12)	4571 (2)	2865 (3)	4186 (3)	31 (2)	38 (2)	37 (2)	3 (2)	0 (2)	17 (2)
C(13)	5512 (2)	3326 (3)	4028 (3)	22 (2)	50 (2)	44 (2)	0 (2)	−6 (2)	16 (2)
C(14)	5586 (2)	3986 (3)	3244 (3)	24 (2)	46 (2)	40 (2)	−9 (2)	−1 (1)	12 (2)
C(15)	4715 (2)	4171 (3)	2602 (3)	33 (2)	37 (2)	33 (2)	−11 (1)	−2 (1)	13 (2)
C(16)	3803 (2)	3687 (3)	2817 (3)	26 (2)	36 (2)	32 (2)	−7 (1)	−6 (1)	14 (2)
C(17)	4754 (3)	4871 (4)	1714 (4)	52 (2)	84 (3)	78 (3)	−28 (2)	−13 (2)	57 (3)
N(18)	2124 (2)	734 (2)	2140 (2)	26 (1)	27 (1)	25 (1)	−2 (1)	−5 (1)	8 (1)
C(19)	1279 (2)	404 (3)	1302 (3)	31 (2)	31 (2)	28 (2)	−4 (1)	−7 (1)	9 (1)
N(20)	1130 (2)	−836 (2)	417 (3)	45 (2)	27 (2)	44 (2)	−3 (1)	−24 (1)	2 (1)
C(21)	1770 (3)	−1788 (3)	262 (4)	67 (3)	27 (2)	42 (2)	−2 (2)	−17 (2)	8 (2)
C(22)	2783 (3)	−1454 (3)	1061 (3)	46 (2)	27 (2)	37 (2)	4 (2)	−5 (2)	12 (2)
C(23)	2829 (2)	−117 (3)	2136 (3)	32 (2)	34 (2)	32 (2)	−2 (1)	−4 (1)	16 (2)
C(24)	2966 (3)	−2370 (3)	1730 (4)	76 (3)	38 (2)	68 (3)	−1 (2)	−16 (2)	30 (2)
C(25)	2172 (5)	−2298 (5)	2744 (6)	125 (5)	100 (4)	105 (4)	−20 (3)	3 (4)	77 (4)
C(26)	3672 (3)	−1601 (4)	71 (4)	68 (3)	54 (2)	49 (2)	19 (2)	12 (2)	16 (2)
C(27)	3647 (4)	−713 (5)	−609 (4)	74 (3)	86 (3)	60 (3)	9 (3)	17 (2)	38 (3)
O(19)	642 (2)	1173 (2)	1323 (2)	37 (1)	33 (1)	45 (1)	4 (1)	−20 (1)	2 (1)
O(21)	1554 (2)	−2860 (2)	−520 (3)	102 (2)	28 (1)	87 (2)	0 (1)	−46 (2)	1 (1)
O(23)	3526 (2)	209 (2)	2993 (2)	43 (1)	43 (1)	52 (2)	0 (1)	−22 (1)	17 (1)
N(28)	1488 (2)	3916 (2)	3870 (2)	26 (1)	28 (1)	33 (1)	0 (1)	4 (1)	15 (1)
C(29)	894 (3)	3960 (3)	2866 (3)	38 (2)	40 (2)	42 (2)	2 (2)	−3 (2)	22 (2)
C(30)	440 (3)	5047 (4)	2966 (4)	55 (3)	55 (2)	62 (3)	16 (2)	−4 (2)	34 (2)
C(31)	604 (3)	6126 (3)	4114 (4)	44 (2)	43 (2)	74 (3)	14 (2)	11 (2)	32 (2)
C(32)	1222 (2)	6116 (3)	5163 (4)	32 (2)	28 (2)	54 (2)	−2 (1)	18 (2)	8 (2)
C(33)	1645 (2)	4976 (3)	4992 (3)	30 (2)	32 (2)	41 (2)	−1 (1)	5 (1)	13 (2)
C(34)	1444 (4)	7279 (4)	6412 (4)	82 (3)	34 (2)	71 (3)	3 (2)	2 (2)	3 (2)

All computations were performed on a Univac 1106 computer.

Description of the structure and discussion

The structure of the molecule is shown in Fig. 1 (*ORTEP*: Johnson, 1965). The intramolecular bond lengths and angles and their associated e.s.d.'s are given in Tables 5 and 6 respectively. The parameters were calculated with the X-RAY system (1972) program *BONDLA*. Table 7 lists least-squares planes with the distances of various atoms from these planes as well as their intersection angles. The four independent moieties of the molecule have been numbered 1 to 4.

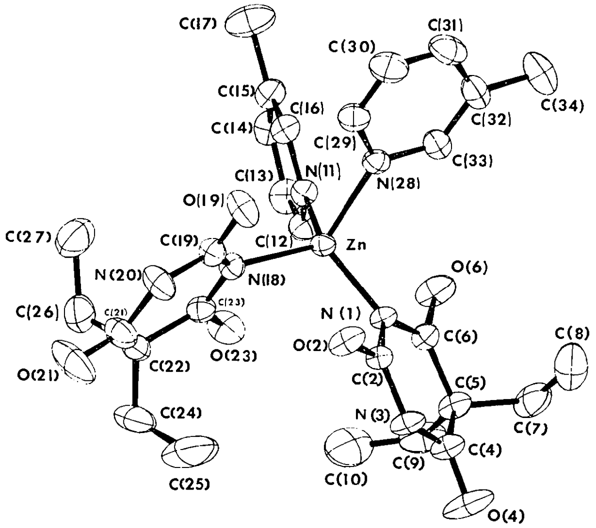

Fig. 1. Structure of the molecule.

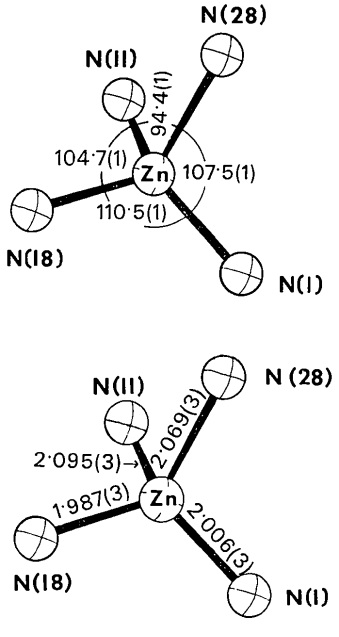

Fig. 2. Bond lengths and angles in the zinc coordination sphere. N(1)-Zn-N(11) = 118·4(1)° and N(18)-Zn-N(28) = 121·1 (1)°.

Table 3. *Hydrogen atoms: fractional atomic coordinates* (× 10^3) *and their e.s.d.'s* (× 10^3)

	x	*y*	*z*
H(3)	60 (2)	32 (3)	620 (3)
H(7,1)	243 (3)	287 (3)	975 (4)
H(7,2)	293 (3)	382 (3)	924 (4)
H(8,1)	137 (4)	427 (4)	859 (5)
H(8,2)	82 (4)	325 (4)	907 (5)
H(8,3)	142 (4)	460 (4)	8 (5)
H(9,1)	414 (3)	217 (3)	782 (4)
H(9,2)	359 (3)	132 (3)	844 (4)
H(10,1)	333 (3)	−28 (4)	627 (4)
H(10,2)	379 (3)	56 (4)	570 (4)
H(10,3)	448 (3)	4 (4)	643 (4)
H(12)	449 (2)	238 (3)	469 (3)
H(13)	609 (2)	318 (3)	445 (3)
H(14)	622 (2)	434 (3)	315 (3)
H(16)	318 (2)	385 (3)	246 (3)
H(17,1)	430 (3)	550 (4)	199 (4)
H(17,2)	539 (3)	512 (3)	161 (4)
H(17,3)	456 (3)	435 (4)	86 (4)
H(20)	62 (2)	−100 (3)	−11 (3)
H(24,1)	302 (3)	−329 (3)	93 (4)
H(24,2)	369 (3)	−220 (3)	215 (4)
H(25,1)	205 (4)	−140 (5)	349 (5)
H(25,2)	138 (4)	−243 (5)	237 (5)
H(25,3)	233 (4)	−285 (5)	306 (5)
H(26,1)	434 (3)	−145 (3)	55 (4)
H(26,2)	366 (3)	−247 (3)	−58 (4)
H(27,1)	302 (3)	−75 (4)	−99 (4)
H(27,2)	371 (3)	30 (4)	17 (4)
H(27,3)	427 (3)	−89 (4)	−113 (4)
H(29)	78 (2)	315 (3)	206 (3)
H(30)	−1 (3)	515 (3)	230 (4)
H(31)	29 (3)	695 (3)	426 (4)
H(33)	207 (2)	487 (3)	572 (3)
H(34,1)	65 (3)	777 (4)	689 (4)
H(34,2)	166 (3)	704 (4)	707 (4)
H(34,3)	154 (3)	797 (4)	627 (4)

Environment of the zinc atom

The zinc atom is tetrahedrally coordinated to the barbital anions *via* the deprotonated nitrogen atoms N(1) and N(18) and to the *β*-picoline moieties *via* the nitrogen atoms N(11) and N(28). The respective bond lengths are 2·006(3) and 1·987(3) Å for Zn–N(barb) and 2·095(3) and 2·069(3) Å for Zn–N(picoline). These are in good agreement with the analogous Zn–N distances [2·009(2) and 2·023(2) Å respectively] reported by Wang & Craven (1971), and the bond angles of the complex lie within the same range as those found by the latter authors. All bond lengths and angles involving the zinc coordination sphere are shown in Fig. 2.

In the *β*-picoline moieties the average C–C and C–H bond lengths are 1·380 and 0·97 Å respectively. Both rings are planar (planes V and VI), the greatest deviations being C(14) and C(29) at 0·007 and 0·005 Å respectively. The two planes intersect at an angle of 76·43°.

Table 4. *Observed and calculated structure factors*

The values listed are $10F_o$ and $10\ F_c$.

	F_o	F_c
H,0,0		
-13	203	204
-12	227	-231
-11	110	-100
-10	278	-287
-9	182	181
-8	212	208
-7	408	-398
-6	182	-184
-5	126	128
-4	462	-450
-3	767	-777
-2	984	-1034
2	994	-1036
3	771	-777
4	465	-450
5	124	128
6	183	-184
7	412	-398
8	219	208
9	180	181
10	282	-287
11	110	-100
12	233	-231
13	203	204
H,1,0		
13	95	-91
12	194	191
11	43	18
10	130	-129
9	148	-153
8	576	582
7	59	54
6	651	-613
5	369	-368
4	223	-201
3	714	720
2	757	-761
-2	442	-441
-3	72	-94
-4	501	-512
-5	982	979
-6	385	384
-7	507	-521
-8	360	-353
-9	169	154
-10	250	248
-11	30*	26
-12	301	-303
-13	128	-126
H,2,0		
-13	299	-297
-12	9*	-4
-11	82	74
-10	170	172
-9	346	-347
-8	180	-193
-7	280	275
-6	538	531
-5	202	-213
-4	812	-787
-3	558	548
-2	1671	1696
-1	186	161
0	457	-466
1	48	40
2	418	424
3	59	-40
4	567	-557
5	720	-703
6	152	154
7	226	221
8	40	-45
9	284	-274
10	91	-97
11	275	277
12	9*	5
13	60	-50
H,3,0		
13	45	-11
12	77	-70
11	61	52
10	242	247
9	117	114
8	259	-273
7	151	-159
6	347	344
5	476	472
4	629	-612
3	479	-478
2	499	-489
1	447	-429
0	182	167
-1	203	-210
-2	651	631
-3	936	971
-4	198	-176
-5	865	-861
-6	220	-237
-7	474	467
-8	383	397
-9	22*	-34
-10	309	-309
-11	45	53
-12	23*	-29
-13	96	83
H,4,0		
-12	35*	47
-11	155	-163
-10	131	-129
-9	163	169
-8	231	230
-7	161	-184
-6	54	27
-5	392	333
-4	882	889
-3	69	79
-2	821	-803
-1	43	-93
0	136	149
1	386	-368
2	568	-566
3	394	-402
4	471	-474
5	584	569
6	176	-162
7	258	-255
8	96	92
9	236	236
10	39	41
11	196	-138
12	24*	-29
H,5,0		
12	111	95
11	53	-56
10	99	-103
9	24*	32
8	321	325
7	62	70
6	282	-288
5	292	-286
4	617	608
3	393	399
2	486	-448
1	590	-602
0	20*	-4
-1	199	170
-2	291	-232
-3	309	-305
-4	202	-205
-5	486	494
-6	133	191
-7	195	-192
-8	168	-202
-9	230	226
-10	189	196
-11	46	-52
-12	104	-104
H,6,0		
-11	249	241
-10	118	116
-9	190	-198
-8	138	-134
-7	59	-61
-6	414	418
-5	190	-191
-4	373	-377
-3	195	132
-2	301	309
-1	18*	-6
0	492	-503
1	718	-719
2	108	122
3	730	716
4	35	38
5	22*	9
6	174	186
7	285	277
8	63	-33
9	271	-266
10	196	-196
11	204	191
H,7,0		
10	30*	-28
9	168	-176
8	81	-79
7	191	191
6	204	281
5	73	79
4	317	-320
3	57	-46
2	493	497
1	68	81
0	305	-299
-1	491	-496
-2	13*	-7
-3	329	326
-4	26*	-31
-5	410	-402
-6	25*	0
-7	239	241
-8	119	117
-9	155	-160
-10	128	-123
H,8,0		
-9	74	73
-8	65	77
-7	0*	-16
-6	300	-298
-5	170	-172
-4	226	235
-3	292	239
-2	357	-351
-1	174	-175
0	348	350
1	436	438
2	19*	3
3	271	-279
4	20*	-5
5	176	173
6	36	59
7	84	-88
8	23*	-22
9	15*	-23
H,9,0		
8	87	81
7	110	-118
6	260	-250
5	102	113
4	362	364
3	194	137
2	232	-240
1	195	-177
0	26*	35
-1	103	101
-2	170	-160
-3	26*	5
-4	199	195
-5	68	64
-6	76	-61
-7	256	-256
-8	109	-108
H,10,0		
-6	217	207
-5	62	-67
-4	208	-213
-3	112	-117
-2	323	326
-1	337	337
0	54	59
1	157	-148
2	22*	49
3	19*	0
4	62	-51
5	70	-85
6	63	-71
H,11,0		
2	182	175
1	29*	32
0	190	-189
-1	35*	-18
-2	85	84
-3	200	193
H,-11,1		
-4	138	-192
-3	47	78
-2	138	-194
-1	193	-180
0	173	161
1	229	221
2	61	58
3	160	-159
4	45	-49
H,-10,1		
7	151	-155
6	204	-204
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-7	95	-90
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-4	102	109
-5	29*	-32
-6	294	-294
-7	0*	-5
H,-9,3		
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-8	31*	30
-7	281	-291
-6	18*	12
-5	112	107
-4	273	268
-3	164	-162
-2	179	-180
-1	66	-66
0	170	171
1	180	178
2	390	-385
3	391	-391
4	194	205
5	160	151
6	135	-131
7	87	-97
8	75	83
9	265	271
H,-8,3		
10	187	-187
9	82	95
8	232	223
7	72	53
6	87	-97
5	55	-54
4	209	208
3	162	154
2	184	-192
1	222	-230
0	114	-112
-1	332	306
-2	40	-41
-3	415	-423
-4	293	-254
-5	288	294
-6	206	210
-7	29*	25
-8	171	-177
-9	80	60
-10	281	283
H,-7,3		
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-10	15*	-10
-9	214	-219
-8	110	-105
-7	219	212
-6	278	281
-5	174	-160
-4	489	-496
-3	193	-194
-2	389	390
-1	6*	0
0	196	-204
1	397	-327
2	308	317
3	97	105
4	134	-126
5	163	-175
6	151	167
7	216	217
8	18*	-7
9	166	-157
10	22*	4
11	157	157
H,-6,3		
11	111	-129
10	215	216
9	89	94
8	197	-193
7	20*	-5
6	266	272
5	237	236
4	136	112
3	237	224
2	55	62
1	348	354
0	270	-267
-1	473	-486
-2	344	-337
-3	282	281
-4	68	-71
-5	360	-370
-6	72	-77
-7	232	234
-8	178	184
-9	103	-92
-10	217	-221
-11	42	-34
H,-5,3		
-12	16*	-1
-11	261	-270
-10	27*	-5
-9	151	150
-8	189	194
-7	201	-191
-6	394	-388
-5	60	59
-4	233	244
-3	283	-280
-2	118	-127
-1	50	-47
0	701	703
1	161	171
2	612	-588
3	167	-184
4	534	476
5	428	407
6	53	-51
7	297	-297
8	150	-145
9	102	97
10	216	-216
11	164	-167
12	60	52
H,-4,3		
12	159	157
11	157	-153
10	554	-540
9	101	-106
8	294	293
7	129	127
6	61	22
5	207	-199
4	448	443
3	247	258
2	353	-359
1	186	171
0	344	341
-1	239	239
-2	518	516
-3	364	-380
-4	372	-358
-5	421	419
-6	174	-180
-7	501	-504
-8	310	-308
-9	106	106
-10	229	221
-11	19*	4
-12	308	-314
H,-3,3		
-13	192	-196
-12	45	-43
-11	198	197
-10	118	117
-9	365	-358
-8	369	-369
-7	244	235
-6	586	577
-5	88	90
-4	573	-567
-3	318	302
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-1	366	-339
0	264	-276
1	69	-95
2	185	187
3	567	570
4	394	-377
5	500	-496
6	263	294
7	163	164
8	43	-100
9	398	-389
10	291	-318
11	178	170
12	240	254
13	47	-30
H,-2,3		
13	110	-103
12	30*	12
11	152	156
10	154	158
9	116	-131
8	463	-452
7	351	-397
6	51	81
5	397	384
4	356	-344
3	271	251
2	44	-36
1	163	-192
0	321	-295
-1	101	73
-2	1178	1195
-3	1266	1292
-4	9*	-11
-5	797	-782
-6	205	-215
-7	39	-26
-8	84	94
-9	331	-325
-10	153	-155
-11	129	127
-12	129	128
-13	84	-74
H,-1,3		
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-12	129	122
-11	192	-187
-10	390	-395
-9	187	186
-8	378	389
-7	14*	12
-6	179	-193
-5	294	277
-4	95	-82
-3	399	407
-2	452	448
-1	449	-447
0	363	369
1	224	-228
2	861	-852
3	865	-852
4	239	224
5	332	342
6	130	120
7	529	-528
8	35	22
9	258	261
10	23*	44
11	24*	7
12	34*	-56
13	192	136
H,0,3		
13	186	182
12	185	185
11	84	91
10	171	-177
9	218	221
8	297	258
7	231	-239
6	330	-328
5	38	-32
4	193	-158
3	376	382
2	511	-504
1	293	-284
0	689	686
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-2	64	-74
-3	23*	1
-4	42	-43
-5	631	619
-6	13*	30
-7	349	-345
-8	192	138
-9	293	280
-10	24*	10
-11	227	-230
-12	175	-188
-13	94	102
H,1,3		
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-12	117	-121
-11	182	189
-10	324	323
-9	0*	-25
-8	92	-92
-7	156	-160
-6	181	197
-5	159	194
-4	128	-135
-3	119	-137
-2	251	244
-1	213	222
0	673	-655
1	463	-450
2	285	298
3	426	425
4	431	-384
5	553	-500
6	81	81
7	256	255
8	266	273
9	135	-137
10	152	143
11	167	172
12	292	238
H,2,3		
12	202	-207
11	252	258
10	226	227
9	173	175
8	300	-303
7	219	217

Table 4 (*cont.*)

	Fo	Fc
	H,2,3	
6	423	429
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3	160	167
2	291	289
1	513	505
0	793	-791
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-2	902	907
-3	221	232
-4	358	-361
-5	211	-219
-6	49	-47
-7	432	419
-8	152	144
-9	123	-131
-10	69	70
-11	95	90
-12	67	51
	H,3,3	
-12	61	51
-11	49	36
-10	120	-115
-9	105	99
-8	319	318
-7	18*	7
-6	338	-345
-5	310	-321
-4	113	119
-3	202	207
-2	190	-150
-1	865	-873
0	818	810
1	930	923
2	315	-289
3	444	-443
4	63	-61
5	390	374
6	164	171
7	342	-341
8	19*	-11
9	363	362
10	80	95
11	139	-130
	H,4,3	
11	371	-372
10	181	-181
9	225	218
8	566	564
7	155	-153
6	413	-422
5	132	140
4	418	413
3	139	139
2	73	-85
1	240	229
0	193	138
-1	253	263
-2	751	-752
-3	103	-113
-4	103	107
-5	277	288
-6	185	-189
-7	453	-447
-8	52	30
-9	289	293
-10	52	58
-11	171	-176
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-10	172	155
-9	40	49
-8	306	-298
-7	219	-218
-6	328	335
-5	272	273
-4	216	-223
-3	363	-359
-2	249	270
-1	196	157
0	133	-122
1	333	-331
2	193	196
3	358	354
4	101	113
5	235	-238
6	66	-63
7	178	172
8	54	-21
9	161	-164
10	305	-305
	H,6,3	
9	250	-292
8	267	-273
7	22*	-41
6	389	383
5	190	191
4	398	-407
3	124	116
2	309	306
1	85	-78
0	135	-125
-1	51	-52
-2	323	330
-3	316	323
-4	219	-215
-5	167	-177
-6	150	195
-7	48	-47
-8	175	-184
-9	187	-184
	H,7,3	
-8	96	92
-7	85	88
-6	80	81
-5	61	66
-4	142	143
-3	141	134
-2	379	-377
-1	225	-229
0	117	112
1	285	282
2	68	83
3	381	-383
4	69	-67
5	321	318
6	99	97
7	225	-227
8	192	-191
	H,8,3	
6	268	-269
5	32*	40
4	391	392
3	61	77
2	237	-227
1	32*	-59
0	161	152
-1	15*	15

	Fo	Fc
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-2	118	-119
-3	32*	-43
-4	253	256
-5	297	238
-6	95	-101
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-4	79	-76
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-1	179	176
0	93	-93
1	314	-315
2	70	-67
3	301	297
	H,-12,4	
-3	175	172
-2	45	45
-1	226	-226
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1	192	181
2	110	102
3	196	-152
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6	44	-32
5	236	236
4	101	101
3	138	-132
2	185	-184
1	160	161
0	315	313
-1	190	-136
-2	340	-352
-3	122	121
-4	136	151
-5	50	49
-6	259	-254
	H,-10,4	
-8	151	-194
-7	166	-166
-6	207	206
-5	90	83
-4	206	194
-3	388	-388
-2	298	-292
-1	295	297
0	308	307
1	98	-103
2	292	-294
3	118	115
4	191	182
5	139	-152
6	395	-398
7	110	114
8	189	187
	H,-9,4	
9	163	-154
8	171	177
7	212	211
6	3*	5
5	392	-393
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3	225	227
2	93	80
1	129	-129
0	157	-155
-1	175	181
-2	215	218
-3	166	-172
-4	444	-451
-5	124	124
-6	171	172
-7	192	184
-8	220	-212
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-10	89	-81
-9	73	-67
-8	114	113
-7	88	93
-6	110	107
-5	407	-402
-4	337	-348
-3	395	394
-2	205	196
-1	202	-210
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1	193	195
2	376	391
3	58	-59
4	384	-382
5	55	49
6	154	157
7	80	80
8	167	-160
9	120	-108
10	53	64
	H,-7,4	
11	177	-173
10	158	154
9	225	233
8	53	57
7	222	-228
6	42	41
5	307	293
4	226	-271
3	302	-305
2	181	168
1	274	281
0	123	127
-1	171	-180
-2	523	-534
-3	209	214
-4	347	351
-5	206	-209
-6	111	-115
-7	26*	-14
-8	189	189
-9	42	47
-10	215	-204
-11	65	-84
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-11	44	-47
-10	68	71
-9	141	130
-8	0*	1
-7	261	-259
-6	71	-75
-5	197	203
-4	22*	13
-3	117	-117
-2	108	-104
-1	501	504

	Fo	Fc
	H,-6,4	
0	578	570
1	320	-322
2	453	-434
3	126	111
4	387	377
5	217	219
6	303	-305
7	232	-232
8	44	20
9	72	-67
10	256	-267
11	63	-48
	H,-5,4	
12	127	119
11	195	193
10	103	-107
9	227	-225
8	290	293
7	264	278
6	157	-159
5	469	-464
4	136	137
3	532	518
2	199	-138
1	416	-407
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-1	293	302
-2	178	191
-3	27*	-13
-4	397	-406
-5	26*	39
-6	433	433
-7	85	-87
-8	406	-402
-9	30*	-5
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-11	224	238
-12	122	-129
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-11	120	112
-10	142	-132
-9	461	-454
-8	94	-92
-7	499	488
-6	451	458
-5	170	-185
-4	37	-22
-3	498	495
-2	201	205
-1	167	130
0	582	-560
1	133	129
2	237	234
3	96	-91
4	663	-656
5	153	-198
6	285	287
7	294	295
8	193	-196
9	411	-427
10	33*	-22
11	224	229
12	65	70
	H,-3,4	
12	101	-107
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10	196	199
9	111	117
8	212	-215
7	101	-108
6	129	127
5	421	428
4	35	-35
3	569	-566
2	96	82
1	443	434
0	472	-457
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-2	143	110
-3	858	852
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-5	534	-521
-6	629	-612
-7	64	-56
-8	352	341
-9	42	100
-10	179	-172
-11	32*	-23
-12	168	167
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-12	83	-95
-11	250	-249
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-9	323	315
-8	94	56
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-6	35	17
-5	298	309
-4	590	579
-3	265	253
-2	592	-587
-1	211	-187
0	518	498
1	207	-183
2	874	-861
3	461	-435
4	209	196
5	38	46
6	70	43
7	118	-122
8	259	252
9	277	271
10	144	-139
11	136	-132
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	H,-1,4	
11	197	194
10	307	-311
9	253	-294
8	317	325
7	177	187
6	106	94
5	391	294
4	132	131
3	294	309
2	437	-421
1	907	-888
0	78	-56
-1	537	537
-2	98	124
-3	209	-224
-4	494	-490
-5	150	164
-6	334	329
-7	180	-184
-8	293	-295
-9	116	112

	Fo	Fc
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-11	26*	31
-12	203	-199
	H,0,4	
-12	194	194
-11	334	324
-10	115	114
-9	218	-215
-8	124	-133
-7	43	47
-6	460	494
-5	302	-295
-4	409	-423
-3	194	197
-2	303	289
-1	206	-207
0	504	-494
1	38	36
2	194	199
3	33	11
4	211	-210
5	260	234
6	292	236
7	281	291
8	28*	-23
9	295	-302
10	106	114
11	218	209
12	87	-85
	H,1,4	
12	293	-294
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10	125	140
9	106	113
8	314	-315
7	264	-268
6	466	465
5	390	383
4	226	214
3	230	-218
2	61	63
1	186	201
0	23*	-39
-1	528	-531
-2	355	-338
-3	252	247
-4	227	234
-5	303	-334
-6	435	-442
-7	312	305
-8	182	190
-9	7*	-3
-10	257	-262
-11	52	-54
-12	229	225
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-9	233	242
-8	131	141
-7	274	-270
-6	482	-486
-5	21*	-17
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-3	277	-262
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0	494	473
1	29	12
2	193	-201
3	93	69
4	331	332
5	524	523
6	190	-192
7	374	-371
8	112	-119
9	37	24
10	72	-74
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	H,3,4	
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9	80	-86
8	288	292
7	13*	6
6	511	-511
5	356	-361
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3	314	310
2	178	156
1	175	154
0	114	-111
-1	298	324
-2	388	-380
-3	310	-319
-4	60	-63
-5	191	190
-6	236	254
-7	174	-169
-8	355	-350
-9	86	87
-10	234	236
-11	128	-130
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-10	121	120
-9	197	-235
-8	327	-339
-7	288	288
-6	273	276
-5	179	-167
-4	111	-111
-3	18*	-17
-2	313	330
-1	21*	26
0	135	-139
1	97	87
2	286	284
3	321	320
4	84	81
5	324	-325
6	346	-353
7	23*	-7
8	199	-205
9	34	-26
10	134	-133
	H,5,4	
9	27*	17
8	32*	12
7	290	-293
6	168	-167
5	43	45
4	130	-133
3	67	-73
2	281	274
1	101	119

	Fo	Fc
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0	63	-64
-1	233	-234
-2	162	164
-3	260	253
-4	28*	30
-5	292	-281
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-7	128	129
-8	94	91
-9	113	-98
-10	139	-134
	H,6,4	
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-7	54	51
-6	125	116
-5	189	201
-4	101	100
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-2	426	-427
-1	102	101
0	187	189
1	234	242
2	83	-69
3	318	-325
4	102	-106
5	61	43
6	151	-155
7	131	-123
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	H,7,4	
6	170	-161
5	157	-142
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3	75	80
2	65	-61
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-4	95	96
-5	197	197
-6	50	41
-7	168	-156
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-4	133	-129
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-2	129	128
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0	204	-211
1	196	-191
2	67	61
3	118	109
4	157	-155
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3	37*	2
2	300	-302
1	298	-303
0	253	250
-1	298	294
-2	34*	-21
-3	112	-105
	H,-11,5	
-6	167	164
-5	34*	32
-4	67	-74
-3	168	-171
-2	278	279
-1	491	438
0	113	-111
1	314	-309
2	55	-62
3	135	124
4	69	-65
5	161	-160
6	192	-135
	H,-10,5	
7	158	155
6	280	296
5	235	-237
4	182	-179
3	28*	7
2	212	214
1	106	-104
0	363	-363
-1	166	-161
-2	292	291
-3	312	316
-4	51	-41
-5	202	-194
-6	34*	-4
-7	250	244
	H,-9,5	
-9	28*	-22
-8	179	181
-7	163	166
-6	306	-309
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-4	289	295
-3	422	434
-2	271	-272
-1	301	-306
0	93	-92
1	191	154
2	95	97
3	175	-175
4	125	-124
5	326	321
6	101	101
7	14*	6
8	127	-115
9	37*	-34
	H,-8,5	
10	35*	42
9	135	127
8	138	138
7	59	-57
6	348	-352
5	152	153
4	335	341
3	128	-134
2	218	-219
1	69	-44
0	88	93
-1	49	-45
-2	409	-411
-3	253	-258
-4	568	577
-5	219	232
-6	90	-9
-7	349	-343

	Fo	Fc
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-8	33	74
-9	183	181
-10	31*	-28
	H,-7,5	
-10	204	207
-9	133	129
-8	310	-314
-7	48	-41
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-3	417	-425
-2	56	-54
-1	307	314
0	88	-79
1	329	-337
2	128	120
3	500	504
4	305	302
5	165	-164
6	59	-50
7	66	68
8	179	174
9	160	-154
10	242	-250
	H,-6,5	
11	249	249
10	103	106
9	151	-153
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3	219	216

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9	298	294
8	72	-72
7	268	-277
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5	226	231
4	82	89
3	595	-582
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5	195	189
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7	69	-68
8	195	192
9	78	65
10	118	-110
11	133	-128
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10	90	92
9	97	-96
8	154	161
7	283	283
6	176	-177
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3	268	262
2	10*	11
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2	377	379
3	49	-36
4	197	-203
5	211	-206
6	234	229
7	288	279
8	272	-275
9	139	-139
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4	39	41
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3	185	171
4	184	-179
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2	89	-81
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4	86	92
5	270	266
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7	76	76
6	297	-290
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2	85	-94
1	75	-71
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3	141	126
4	192	138
5	289	-289
6	186	194

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9	220	-211
	H,-7,6	
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8	249	-248
7	38	37
6	138	-132
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0	293	-293
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-2	268	264
-3	440	433
-4	86	89
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-6	201	-211
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-9	117	-114
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-6	211	-212
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-3	121	122
-2	94	-99
-1	160	-169
0	99	112
1	262	265
2	87	-91
3	331	-328
4	62	68
5	323	324
6	274	280
7	322	-325
8	367	-373
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10	214	226
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10	109	-107
9	322	332
8	114	-110
7	519	-514
6	116	-110
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3	62	54
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-6	212	-226
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7	173	182
8	206	204
9	80	-78
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	H,-3,6	
11	192	182
10	191	190
9	173	-183
8	179	-177
7	269	270
6	272	266
5	276	-285
4	252	-257
3	294	294
2	74	83
1	94	-87
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-3	388	386
-4	29*	19
-5	23*	-23
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-8	70	63
-9	196	-182
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-7	148	154
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-5	60	51
-4	400	409
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-2	268	-266
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1	255	253
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3	291	-309

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6	186	198
7	220	-219
8	238	-240
9	255	252
10	274	266
11	62	71
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9	307	312
8	185	191
7	163	-163
6	66	-65
5	176	182
4	477	460
3	177	-179
2	558	-567
1	26*	30
0	362	356
-1	119	117
-2	340	-333
-3	445	-450
-4	39	32
-5	430	438
-6	63	-81
-7	179	-178
-8	74	80
-9	216	211
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-11	134	-130
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-7	45	-31
-6	250	254
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-4	539	-533
-3	430	-436
-2	91	90
-1	297	289
0	14*	-22
1	325	-329
2	182	-174
3	585	574
4	234	212
5	206	-210
6	65	-53
7	196	198
8	253	250
9	59	63
10	230	-232
11	158	-170
	H,1,6	
10	65	-63
9	112	-107
8	42	-24
7	192	189
6	67	69
5	338	-335
4	291	-298
3	334	334
2	480	470
1	62	62
0	334	-327
-1	76	-80
-2	448	451
-3	14*	13
-4	277	-277
-5	175	-179
-6	91	86
-7	197	152
-8	51	-44
-9	281	-281
-10	14*	26
	H,2,6	
-10	196	-191
-9	31*	-34
-8	195	192
-7	103	107
-6	355	-370
-5	160	-161
-4	83	-79
-3	88	93
-2	126	130
-1	222	-228
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1	386	377
2	129	129
3	269	-280
4	150	-141
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6	477	484
7	69	65
8	266	-267
9	52	-54
	H,3,6	
9	210	216
8	23*	-15
7	233	-239
6	82	-93
5	10*	-9
4	179	-175
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2	137	-143
1	232	233
0	411	411
-1	12*	13
-2	285	-284
-3	68	-77
-4	259	254
-5	130	123
-6	218	-212
-7	170	-173
-8	183	181
-9	136	137
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-7	83	-90
-6	153	146
-5	158	155
-4	40	-31
-3	233	-228
-2	142	-148
-1	391	395
0	175	172
1	173	-170
2	19*	-19
3	106	107
4	93	94
5	205	-214
6	217	-227
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8	223	216

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1	55	-63
2	248	-244
3	59	-38
4	168	168
5	158	153
	H,-10,7	
6	86	-84
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4	103	105
3	259	252
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-2	61	50
-3	154	-153
-4	24*	-39
-5	177	180
-6	195	197
	H,-9,7	
-8	23*	19
-7	205	198
-6	154	156
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-2	226	234
-1	34*	-36
0	193	-192
1	30*	-30
2	150	154
3	73	-78
4	219	-221
5	94	-89
6	357	365
7	54	62
8	166	-175
	H,-8,7	
8	282	-273
7	355	-351
6	69	-67
5	295	295
4	213	-209
3	78	-75
2	189	192
1	338	342
0	194	194
-1	394	-391
-2	190	-192
-3	445	446
-4	55	50
-5	194	-196
-6	24*	-29
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-8	209	199
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0	287	282
1	21*	-24
2	268	-270
3	204	-211
4	233	237
5	233	228
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8	199	195
9	396	397
	H,-6,7	
10	170	-162
9	83	77
8	332	323
7	135	-134
6	365	-366
5	315	-321
4	370	373
3	256	258
2	122	-120
1	397	-402
0	24*	-27
-1	302	301
-2	258	250
-3	230	-226
-4	366	-364
-5	367	380
-6	31*	14
-7	28*	-12
-8	36*	-44
-9	321	318
-10	126	119

	Fo	Fc
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-10	155	147
-9	111	-114
-8	19*	-31
-7	120	-124
-6	391	396
-5	0*	-19
-4	389	-398
-3	100	-84
-2	223	220
-1	120	-124
0	261	-257
1	105	-104
2	282	265
3	371	371
4	483	-490
5	182	-178
6	121	124
7	220	216
8	4*	-22
9	42	-38
10	57	53
	H,-4,7	
10	189	197
9	197	193
8	174	-164
7	110	-102
6	473	471
5	193	195
4	322	-313
3	316	-300
2	234	236
1	230	229
0	255	-237
-1	259	-266
-2	100	-94
-3	245	238
-4	142	135
-5	145	-151
-6	121	-121
-7	100	98
-8	117	130
-9	0*	8
-10	33*	-33
	H,-3,7	
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-9	121	112
-8	49	47
-7	155	-155
-6	292	-287
-5	136	145
-4	196	198
-3	54	-66
-2	219	-219
-1	364	-357
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1	218	222
2	222	-235
3	53	-52
4	341	329
5	311	310
6	53	-64
7	262	-267
8	13*	-4
9	321	324
10	59	-58
	H,-2,7.	
10	323	-337
9	13*	-14
8	380	397
7	11*	15
6	231	-228
5	86	-81
4	384	387
3	405	403
2	211	-207
1	390	-386
0	218	212
-1	212	209
-2	105	-107
-3	134	-140
-4	65	-44
-5	177	178
-6	140	137
-7	292	-304
-8	191	-190
-9	35*	40
-10	64	57
	H,-1,7	
-10	31*	23
-9	135	-124
-8	181	-179
-7	82	83
-6	281	284
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-4	313	-322
-3	102	-94
-2	153	154
-1	395	399
0	357	-355
1	278	-272
2	335	338
3	389	385
4	168	-174
5	183	-173
6	13*	-7
7	275	287
8	120	129
9	314	-323
10	264	-274
	H,0,7	
9	194	-195
8	300	-294
7	43	-44
6	194	199
5	28*	-29
4	291	-291
3	25*	-24
2	572	574
1	306	308
0	293	-293
-1	318	-313
-2	320	321
-3	287	296
-4	26*	25
-5	226	-229
-6	22*	18
-7	91	72
-8	70	-70
-9	187	-185
-10	152	-152
	H,1,7	
-9	34*	56
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-6	194	-153
-5	64	-66
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-3	171	175

Table 4 (*cont.*)

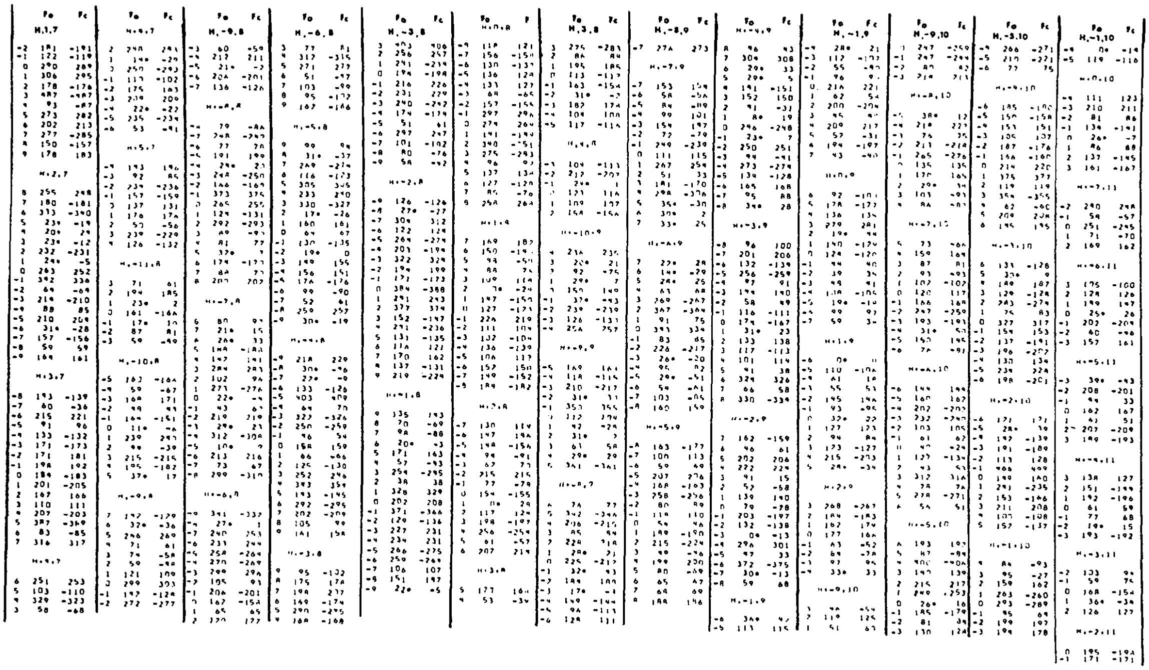

Table 5. *Intramolecular bond lengths and their e.s.d.'s in Å*

Zn—N(1)	2·006 (3)	Zn—N(18)	1·987 (3)
N(1)—C(2)	1·359 (4)	N(18)-C(19)	1·361 (4)
C(2)—N(3)	1·383 (5)	C(19)-N(20)	1·384 (4)
N(3)—C(4)	1·371 (4)	N(20)-C(21)	1·353 (5)
C(4)—C(5)	1·521 (5)	C(21)-C(22)	1·521 (5)
C(5)—C(6)	1·530 (6)	C(22)-C(23)	1·520 (4)
C(6)—N(1)	1·362 (4)	C(23)-N(18)	1·359 (4)
C(2)—O(2)	1·227 (4)	C(19)-O(19)	1·223 (4)
C(4)—O(4)	1·211 (6)	C(21)-O(21)	1·220 (4)
C(6)—O(6)	1·225 (4)	C(23)-O(23)	1·228 (4)
C(5)—C(7)	1·545 (4)	C(22)-C(24)	1·545 (7)
C(7)—C(8)	1·520 (8)	C(24)-C(25)	1·499 (8)
C(5)—C(9)	1·540 (5)	C(22)-C(26)	1·554 (6)
C(9)—C(10)	1·504 (6)	C(26)-C(27)	1·505 (8)
Zn—N(11)	2·095 (3)	Zn—N(28)	2·069 (3)
N(11)-C(12)	1·341 (4)	N(28)-C(29)	1·343 (5)
C(12)-C(13)	1·378 (5)	C(29)-C(30)	1·375 (6)
C(13)-C(14)	1·371 (6)	C(30)-C(31)	1·373 (5)
C(14)-C(15)	1·384 (5)	C(31)-C(32)	1·386 (6)
C(15)-C(16)	1·378 (5)	C(32)-C(33)	1·392 (5)
C(16)-N(11)	1·343 (5)	C(33)-N(28)	1·344 (4)
C(15)-C(17)	1·504 (7)	C(32)-C(34)	1·495 (5)
N(3)—H(3)	0·85 (3)	N(20)-H(20)	0·84 (3)
C(7)—H(7,1)	0·98 (5)	C(24)-H(24,1)	1·07 (3)
C(7)—H(7,2)	0·93 (4)	C(24)-H(24,2)	1·02 (4)
C(8)—H(8,1)	0·90 (6)	C(25)-H(25,1)	1·04 (4)
C(8)—H(8,2)	1·06 (5)	C(25)-H(25,2)	1·08 (5)
C(8)—H(8,3)	0·96 (4)	C(25)-H(25,3)	0·87 (6)
C(9)—H(9,1)	1·01 (4)	C(26)-H(26,1)	0·98 (4)
C(9)—H(9,2)	0·94 (5)	C(26)-H(26,2)	0·97 (3)
C(10)-H(10,1)	0·94 (5)	C(27)-H(27,1)	0·90 (4)
C(10)-H(10,2)	0·89 (6)	C(27)-H(27,2)	1·14 (4)
C(10)-H(10,3)	0·95 (5)	C(27)-H(27,3)	0·97 (4)
C(12)-H(12)	0·95 (4)	C(29)-H(29)	1·00 (3)
C(13)-H(13)	0·93 (3)	C(30)-H(30)	0·97 (4)
C(14)-H(14)	0·95 (3)	C(31)-H(31)	1·00 (4)
C(16)-H(16)	0·95 (3)	C(33)-H(33)	1·01 (3)
C(17)-H(17,1)	0·90 (4)	C(34)-H(34,1)	1·20 (4)
C(17)-H(17,2)	0·90 (4)	C(34)-H(34,2)	0·91 (5)
C(17)-H(17,3)	0·91 (3)	C(34)-H(34,3)	0·89 (5)

Table 6. *Intramolecular bond angles* (°)

N(1)—Zn—N(11)	118·4 (1)	N(11)-Zn—N(28)	94·4 (1)
N(1)—Zn—N(18)	110·5 (1)	N(11)-Zn—N(18)	104·7 (1)
N(1)—Zn—N(28)	107·5 (1)	N(18)-Zn—N(28)	121·1 (1)
Zn—N(1)—C(2)	115·8 (2)	Zn—N(18)-C(19)	123·5 (2)
N(1)—C(2)—N(3)	119·9 (3)	N(18)-C(19)-N(20)	119·1 (3)
C(2)—N(3)—C(4)	125·7 (3)	C(19)-N(20)-C(21)	126·1 (3)
N(3)—C(4)—C(5)	116·9 (3)	N(20)-C(21)-C(22)	117·1 (3)
C(4)—C(5)—C(6)	114·3 (3)	C(21)-C(22)-C(23)	113·3 (3)
C(5)—C(6)—N(1)	120·9 (3)	C(22)-C(23)-N(18)	120·9 (3)
C(6)—N(1)—C(2)	121·9 (3)	C(23)-N(18)-C(19)	122·1 (2)
C(6)—N(1)—Zn	121·7 (2)	C(23)-N(18)-Zn	112·6 (2)
N(1)—C(2)—O(2)	120·9 (3)	N(18)-C(19)-O(19)	121·7 (2)
N(3)—C(2)—O(2)	119·2 (3)	N(20)-C(19)-O(19)	119·2 (3)
N(3)—C(4)—O(4)	121·1 (3)	N(20)-C(21)-O(21)	121·1 (4)
C(5)—C(4)—O(4)	122·0 (3)	C(22)-C(21)-O(21)	121·8 (4)
C(5)—C(6)—O(6)	119·3 (3)	C(22)-C(23)-O(23)	119·7 (3)
N(1)—C(6)—O(6)	119·8 (3)	N(18)-C(23)-O(23)	119·4 (3)
C(4)—C(5)—C(7)	107·9 (3)	C(21)-C(22)-C(24)	109·3 (3)
C(6)—C(5)—C(7)	109·3 (3)	C(23)-C(22)-C(24)	109·7 (3)
C(5)—C(7)—C(8)	114·5 (3)	C(22)-C(24)-C(25)	113·6 (4)
C(4)—C(5)—C(9)	108·4 (3)	C(21)-C(22)-C(26)	109·0 (3)
C(6)—C(5)—C(9)	107·5 (3)	C(23)-C(22)-C(26)	107·4 (3)
C(7)—C(5)—C(9)	109·6 (3)	C(24)-C(22)-C(26)	108·1 (3)
C(5)—C(9)—C(10)	115·4 (3)	C(22)-C(26)-C(27)	115·0 (3)
Zn—N(11)-C(12)	123·4 (3)	Zn—N(28)-C(29)	124·3 (2)
N(11)-C(12)-C(13)	121·4 (4)	N(28)-C(29)-C(30)	121·8 (3)
C(12)-C(13)-C(14)	119·9 (3)	C(29)-C(30)-C(31)	119·5 (4)
C(13)-C(14)-C(15)	119·6 (3)	C(30)-C(31)-C(32)	120·2 (4)
C(14)-C(15)-C(16)	117·0 (4)	C(31)-C(32)-C(33)	116·9 (3)
C(15)-C(16)-N(11)	124·2 (3)	C(32)-C(33)-N(28)	123·2 (3)
C(16)-N(11)-C(12)	117·9 (3)	C(33)-N(28)-C(29)	118·4 (3)
C(16)-N(11)-Zn	118·7 (2)	C(33)-N(28)-Zn	117·2 (2)
C(14)-C(15)-C(17)	121·9 (3)	C(31)-C(32)-C(34)	121·7 (4)
C(16)-C(15)-C(17)	121·1 (3)	C(33)-C(32)-C(34)	121·4 (4)

The barbital ligand structure

The analogous bond lengths and angles for the two barbital ligands show excellent agreement with one another and with the values listed by Wang & Craven.

The conformations of the two trioxopyrimidine rings are shown in Fig. 3. The dotted line is a trace of the least-squares plane through the six ring atoms only (planes I and II). Each pyrimidine ring is almost planar but slight puckering is evident. This is a little more pronounced than that previously observed in the complexes Cu(barbital)$_2$(pyridine)$_2$ and Cu(barbital)$_2$(β-picoline)$_2$.2H$_2$O. Planes I and II intersect at 69·96°. The exocyclic oxygen atoms and the pyrimidine ring atoms are almost coplanar as can been gauged by the small intersection angles (1·65 and 2·32°) of planes I and III, and II and IV. (Planes III and IV are those calculated for the atoms of the trioxopyrimidine rings.)

C(7), C(8), C(9) and C(10) of the two ethyl groups as well as C(5) to which they are attached lie in the same plane (plane VII) which is almost perpendicular (88·68°) to the pyrimidine ring plane (plane I) with which it is associated. Similarly, C(24), C(25), C(26) and C(27) as well as C(22) lie in the same plane (plane VIII) which is at 88·77° to the pyrimidine ring plane (plane II) with which it is associated. These results are consistent with those found in our earlier structures. The hydrogen atoms of the ethyl groups are in the staggered configuration.

Table 7. *Least-squares planes*

The equations of the planes are expressed in orthogonalized space as $PI+QJ+RK=S$.

Plane I	Through the 6 atoms of pyrimidine ring (1)								
Atoms defining the plane	N(1)	C(2)	N(3)	C(4)	C(5)	C(6)			
Atoms not included in the plane							O(2)	O(4)	O(6)
Distance from the plane, Å	0·039	0·005	−0·032	0·015	0·024	−0·052	−0·013	0·047	−0·158
Equation	$0{\cdot}65107I-0{\cdot}73344J-0{\cdot}19539K=-1{\cdot}05638$								
Plane II	Through the 6 atoms of pyrimidine ring (2)								
Atoms defining the plane	N(18)	C(19)	N(20)	C(21)	C(22)	C(23)			
Atoms not included in the plane							O(19)	O(21)	O(23)
Distance from the plane, Å	−0·007	−0·033	0·011	0·044	−0·076	0·062	−0·080	0·123	0·204
Equation	$-0{\cdot}49553I-0{\cdot}19789J+0{\cdot}84575K=0{\cdot}16436$								
Plane III	Through the 9 atoms of the trioxopyrimidine ring (1)								
Atoms defining the plane	N(1)	C(2)	N(3)	C(4)	C(5)	C(6)	O(2)	O(4)	O(6)
Distance from the plane, Å	0·082	0·011	−0·056	−0·003	0·048	0·002	−0·017	0·002	−0·070
Equation	$0{\cdot}66810I-0{\cdot}71424J-0{\cdot}20856K=-1{\cdot}07862$								
Plane IV	Through the 9 atoms of the trioxopyrimidine ring (2)								
Atoms defining the plane	N(18)	C(19)	N(20)	C(21)	C(22)	C(23)	O(19)	O(21)	O(23)
Distance from the plane, Å	−0·021	−0·008	0·022	0·004	−0·162	−0·006	−0·006	0·075	0·102
Equation	$-0{\cdot}52778I-0{\cdot}17888J+0{\cdot}83033K=0{\cdot}07560$								
Plane V	Through the 6 atoms of the β-picoline ring (3)								
Atoms defining the plane	N(11)	C(12)	C(13)	C(14)	C(15)	C(16)			
Atoms not included in the plane							C(17)		
Distance from the plane, Å	0·006	−0·005	−0·002	0·007	−0·005	−0·001	−0·018		
Equation	$-0{\cdot}18200I+0{\cdot}85223J+0{\cdot}49049K=3{\cdot}09456$								
Plane VI	Through the 6 atoms of the β-picoline ring (4)								
Atoms defining the plane	N(28)	C(29)	C(30)	C(31)	C(32)	C(33)			
Atoms not included in the plane							C(34)		
Distance from the plane, Å	−0·002	0·005	−0·003	−0·003	0·005	−0·003	0·046		
Equation	$0{\cdot}80921I+0{\cdot}21277J-0{\cdot}54763K=1{\cdot}12472$								
Plane VII	Through the 4 atoms of the diethyl group and C(5) to which it is attached								
Atoms defining the plane	C(5)	C(7)	C(8)	C(9)	C(10)				
Distance from the plane, Å	−0·024	0·019	0·004	−0·024	0·025				
Equation	$-0{\cdot}43932I-0{\cdot}59964J+0{\cdot}66890K=2{\cdot}14875$								
Plane VIII	Through the 4 atoms of the diethyl group and C(22) to which it is attached								
Atoms defining the plane	C(22)	C(24)	C(25)	C(26)	C(27)				
Distance from the plane, Å	−0·042	0·005	0·021	−0·014	0·030				
Equation	$0{\cdot}54010I+0{\cdot}67677J+0{\cdot}50028K=1{\cdot}86887$								

Intersection angles

Planes		Planes	
I and II	69·96	II and IV	2·32
I and III	1·65	II and V	70·34
I and V	32·92	II and VI	25·01
I and VI	61·46	II and VIII	88·77
I and VII	88·68	V and VI	76·43

In the structure Zn(barbital)$_2$(imidazole)$_2$, Wang & Craven reported that each barbiturate ring is almost coplanar with an adjacent imidazole ligand and that this configuration is stabilized by C–H$\cdots$O intramolecular interactions involving carbon atoms of imidazole and carbonyl oxygen atoms of barbital. In the structure reported here neither of these two effects was observed.

Hydrogen bonding

The intermolecular hydrogen bonding is shown as dotted lines in Fig. 4; the [100] projection of the structure and the geometry of the interactions are shown in Fig. 5. All relevant bond lengths and angles are listed in Table 8. Each discrete molecule of the complex is linked to two other molecules by N–H$\cdots$O hydrogen bonds which form pairs of double links along **c**. The hydrogen bonds between H(3) and $O(2^{i})$, and $H(3^{i})$ and O(2) constitute one such double link while the other arises from the bonding between H(20) and $O(19^{i})$, and $H(20^{i})$ and O(19). There are thus four crystallographically distinct hydrogen bonds emanating in pairs from each molecule. Any one pair links adjacent molecules along **c**.

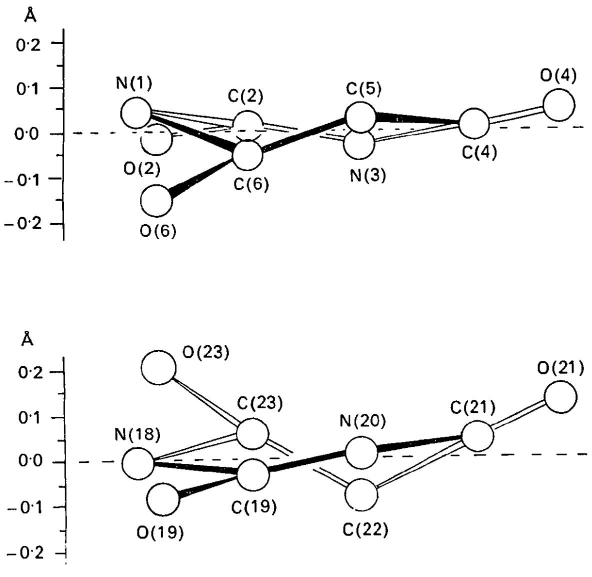

Fig. 3. The conformation of the two trioxopyrimidine rings. The dotted line is a trace of the least-squares plane through the six ring atoms only.

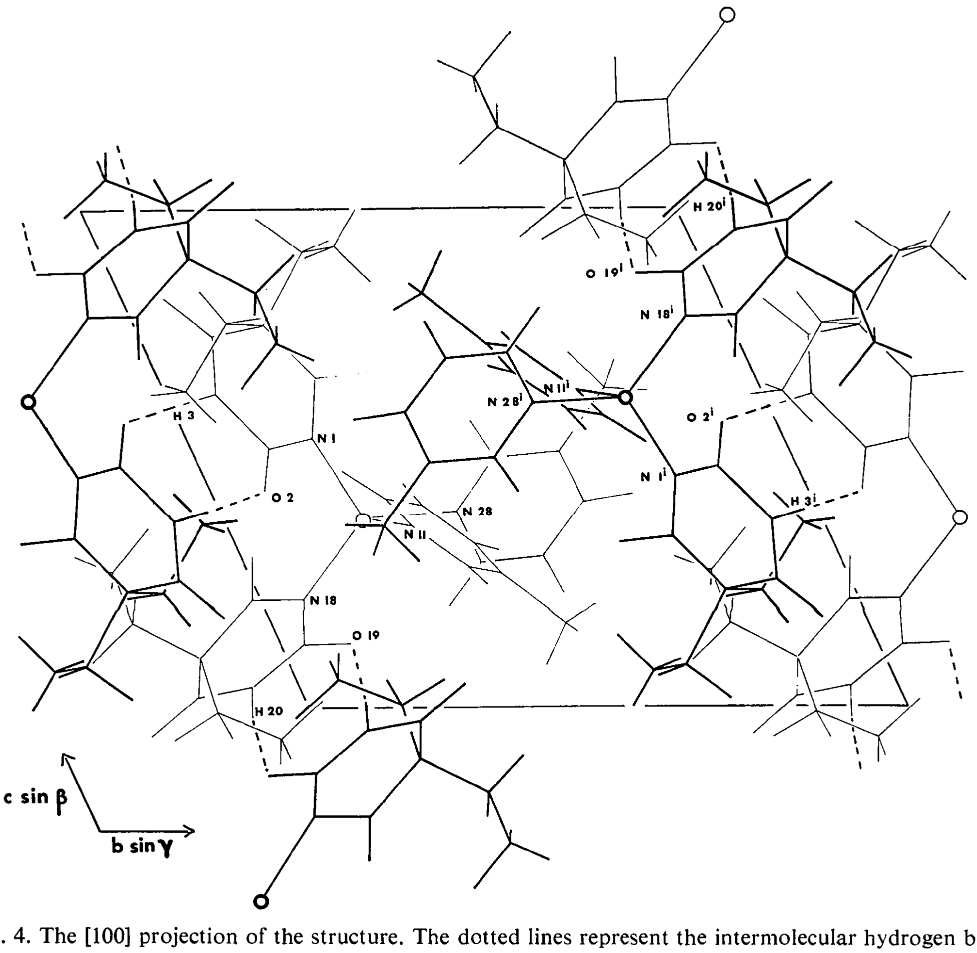

Fig. 4. The [100] projection of the structure. The dotted lines represent the intermolecular hydrogen bonding.

The hydrogen-bond distances are all in good agreement with the corresponding distances in hydrogen-bonded compounds containing donor ring-NH groups and acceptor carbonyl oxygen atoms while the hydrogen-bond angles are well within the maximum allowed deviation from 180° (Donohue, 1968).

Table 8. *Bond lengths* (Å) *and angles* (°) *and their e.s.d.'s involved in the hydrogen bonding*

N(3)–O(2^{i})	2·873 (4)	N(20)–O(19^{i})	2·874 (4)
H(3)–O(2^{i})	2·03 (3)	H(20)–O(19^{i})	2·04 (3)
N(3)–H(3)–O(2^{i})	172 (3)	N(20)–H(20)–O(19^{i})	173 (3)

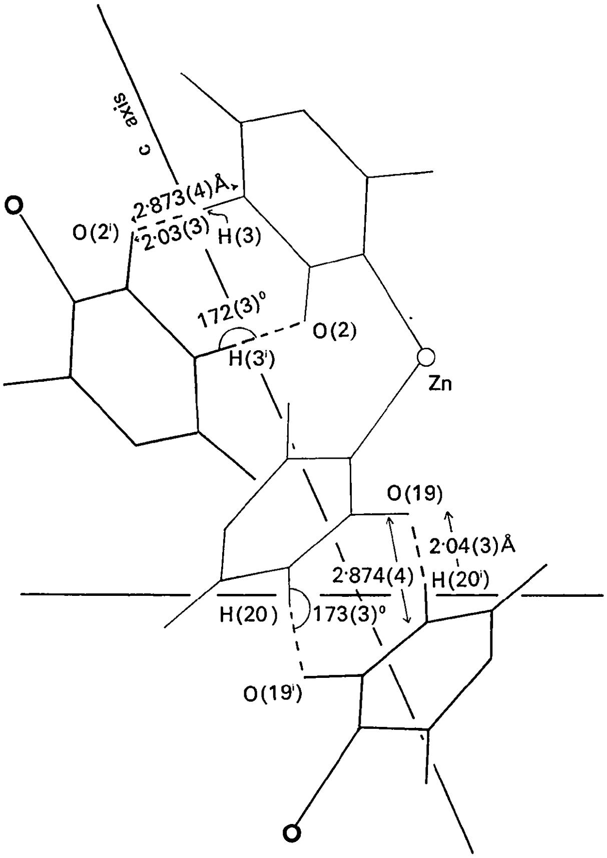

Fig. 5. The geometry of the hydrogen-bonding interactions.

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References

Berking, B. & Craven, B. M. (1971). *Acta Cryst.* **B27**, 1107–1115.
Caira, M. R., Fazakerley, G. V., Linder, P. W. & Nassimbeni, L. R. (1973). *Acta Cryst.* **B29**, 2898–2904.
Craven, B. M., Vizzini, E. A. & Rodrigues, M. M. (1969). *Acta Cryst.* **B25**, 1978–1993.
Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* **A24**, 321–324.
Donohue, J. (1968). *Acta Cryst.* **B24**, 1558–1560.
Fazakerley, G. V., Linder, P. W., Nassimbeni, L. R. & Rodgers, A. (1973). *Cryst. Struct. Commun.* **2**, 647–653.
Fazakerley, G. V., Linder, P. W., Nassimbeni, L. R. & Rodgers, A. (1974*a*). *Inorg. Chim. Acta.* In the press.
Fazakerley, G. V., Linder, P. W., Nassimbeni, L. R. & Rodgers, A. (1974*b*). Submitted to *Cryst. Struct. Commun.*
Johnson, C. K.(1965). *ORTEP*. Report ORNL-3794, Oak Ridge National Laboratory, Oak Ridge, Tennessee.
Levi, L. & Hubley, C. (1956). *Anal. Chem.* **28**, 1591–1605.
Wang, B. C. & Craven, B. M. (1971). *Chem. Commun.* pp. 290–291.
X-RAY system (1972). Technical Report TR-192 of the Computer Science Center, Univ. of Maryland.

Acta Cryst. (1974). **B30**, 1961

Crystallographic Studies of Metal–Nucleotide Base Complexes. III. Dichlorobis-(9-methyl-6-oxypurine)diaquocopper(II) Trihydrate

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$[(C_6H_6N_4O)_2CuCl_2.2H_2O].3H_2O$ crystallizes as deep-blue prisms from an acidic solution. The space group is $C2/c$ with $a=16·858$, $b=8·541$, $c=14·293$ Å, $\beta=91·02°$, $Z=4$. The structure determination is based on diffractometer data measured with Nb-filtered Mo $K\alpha$ radiation. Final R is 0·027 for the 2029 observed reflexions. The copper ion is located at a centre of symmetry and binds to the purine ring through nitrogen N(7); Cu–N = 2·054 Å. The copper coordination is (4+2) with a pair of centrosymmetrically related water molecules completing the equatorial coordination, Cu–O = 1·972 Å, and the axial sites occupied by chloride ions, Cu–Cl = 2·787 Å. The water molecules in the coordination sphere form intramolecular hydrogen bonds to the carbonyl substituent on C(6).

Introduction

The purine bases incorporated in the nucleic acids have several potential coordination sites. The nitrogen atoms N(1), N(3) and N(7) are more or less exposed, and so is the substituent in the 6-position. In the reversible unwinding and rewinding of DNA strands caused by certain transition elements, the coordination