The Crystal Structure of the Bis-(5,5'-diethylbarbiturato)–bis-(picoline) Dihydrate Complex of Copper(II)

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The crystal structure of the title compound, $C_{28}H_{36}$ $CuN_6O_6 \cdot 2H_2O$ has been determined from single crystal X-ray diffraction data collected on a four-circle diffractometer. The analysis was carried out using 1133 independent reflections in Patterson and Fourier syntheses and the structure was refined by full-matrix least-squares methods to a final R factor of 0.035. The complex crystallizes in the space group $P2_1/c$ with a = 11.196(5), b = 15.124(5), c = 9.449(5) Å, β = 102.0(2)°, D_m = 1.37 and contains 2 molecules per unit cell (D_c = 1.381 g cm^{-3}).

The copper atom is bonded in square planar arrangement to the deprotonated nitrogen atoms of the barbital anions and to the nitrogens of the picolines with Cu–N bond lengths of 1.980(5) and 2.018(3) Å respectively. Of interest is the hydrogen bonding (C=O···H–OH and N–H···OH$_2$) in which the water molecules form a layer-like linkage with molecules of the complex. Off-the z-axis co-ordination is postulated between oxygen atoms of the barbital moieties and the copper atom, the Cu–O interatomic distance being 2.857(4) Å.

Introduction

Complexes of the general formula M(II)(barb)$_2$L$_2$ where M is a transition metal, barb is the anion of a substituted barbituric acid and L is an organic base are of interest because of their potential use in the clinical detection and estimation of barbiturate drugs. There has been some doubt as to the exact location of the co-ordinating site on the barbiturate anion. Levi and Hubley[1] have suggested on the basis of i.r. spectral data on a series of complexes of the type Cu(barb)$_2$ (pyridine)$_2$ that the barbiturate ligand is bound to the copper though an oxygen atom. Wang and Craven[2] have elucidated the structures of Co(barb)$_2$(imidazole)$_2$ and Zn(barb)$_2$(imidazole)$_2$ and have shown, in both cases, that the metal atom is bonded to a deprotonated nitrogen atom of the barbiturate anion and not to an oxygen atom as might have been expected. Very recently, Caira, Fazakerley, Linder and Nassimbeni[3] have shown in an X-ray structural investigation

at this university that in the complex Cu(barbital)$_2$ (pyridine)$_2$ the chelating site of the barbiturate anion is again a deprotonated nitrogen atom. We report now the structure of a similar complex which is thus the second in our series of crystal structure determinations of M(II)(barb)$_2$L$_2$ complexes. This program of X-ray analyses has been undertaken to establish whether the barbiturate donor atom in such complexes is nitrogen or oxygen and also to investigate the nature of any hydrogen bonding which might prevail.

Experimental

Preparation of the Complex

The complex was formed by mixing dilute aqueous solutions of CuCl$_2 \cdot 2H_2O$, sodium barbital and β-picoline in the molar ratio 1:2:2 at room temperature. The resulting solution was light purple in colour and had a pH of 7.2. It was allowed to stand for several days after which purple monoclinic prisms crystallized out.

Analysis	%C	%H	%N	%Cu
calculated (without water of crystallization):	54.54	6.18	13.63	10.30
calculated (with water of crystallization):	51.57	6.13	12.89	9.75
experimentally found:	50.9	6.1	12.7	10.6

Calculated and experimentally determined figures agreed closely enough to suggest that the complex had the composition $C_{28}H_{36}CuN_6O_6$ corresponding to the formulation Cu(II)(5,5'-diethylbarbiturato)$_2$(β-picoline)$_2$. During refinement of the structure however, water of crystallization was detected giving the complex the composition $C_{28}H_{36}CuN_6O_6 \cdot 2H_2O$.

Crystal Data

A single crystal of dimensions 0.15 × 0.30 × 0.25 mm was selected for the X-ray investigation. Its density was determined using a density gradient column containing the components *m*-xylene (ϱ = 0.86 g cm^{-3})

and carbon tetrachloride ($\varrho = 1.60$ g cm^{-3}), which was precalibrated with aqueous caesium chloride solutions. The lattice constants were obtained from a least squares analysis of the settings of 25 reflections measured on a four-circle diffractometer with MoK$_\alpha$ radiation.

Reflections of the type $h0l:l = 2n+1$ and $0k0:k = 2n+1$ were systematically absent corresponding to the monoclinic space group P2$_1$/c (2nd setting).

a	=	11.196(5) Å
b	=	15.124(5) Å
c	=	9.449(5) Å
β	=	102.0 (2)°
V	=	1565.23 Å^3
D$_m$	=	1.37(2) g cm^{-3}
D$_c$	=	1.381 g cm^{-3} for Z = 2
μ	=	7.79 cm^{-1} for MoK$_\alpha$ radiation.

Figures in parentheses are the e.s.ds calculated from several measured values of the parameters.

Intensity Data

The intensity data were collected on a Philips PW 1100 computer-controlled four-circle diffractometer operating in the ω-scan mode (scan width = 0.8° and scan speed = 0.02° sec^{-1}). The background and scan times were both 40 seconds. The monochromator to crystal distance was 17 cm and the crystal to detector distance was 22 cm. The receiving aperture at the counter was 6 mm wide and 1,5 mm high. The incident beam take off angle was 6°.

Using Zr-filtered MoK$_\alpha$ radiation ($\lambda = 0.7107$ Å), 1631 reflections up to $2\Theta = 40°$ were measured. Of these, 80 were systematically absent while 106 were redundant due to space group equivalence. A reflection was taken as being above background if $I_{rel} > 2\sigma$ (I_{rel}). 312 further reflections did not satisfy this criterion and were omitted as unobserved.

Throughout the course of the data collection process, three standard reflections were measured after every 44 reflections to ensure stability of operation and to monitor any crystal decomposition. The variation in intensity of a standard reflection was observed to be less than 2.8% of its net intensity. Lorentz Polarization corrections were applied to the data. However absorption corrections were ignored as the variation in μR for the crystal selected was between 0.06 and 0.12 with corresponding A* values of 1.10 and 1.20 for the Θ range scanned. This was regarded as insignificant.

All computations were performed on a Univac 1106 computer system.

Structure Determination and Refinement

With the number of molecules per unit cell having been determined as 2, symmetry considerations re-

quired the copper atoms to occupy special positions 0,0,0 and 0,$^1/_2$,$^1/_2$ with the rest of the centrosymmetric molecule occupying general positions. Since the real space location of the copper atom was 0,0,0 the Patterson synthesis, with copper at the unit cell origin, yielded a map resembling the electron density map which would have been obtained with copper at 0,0,0. Consequently the 3 dimensional Patterson was interpreted as such, but, because it had P2/m symmetry, a mirror image of the molecule was generated and care had to be exercised in distinguishing real molecule peaks from the mirror image peaks. Nevertheless, the 9 atoms of the trioxopyrimidine ring were located and the resulting Fourier synthesis, phased on the positions of these atoms and the position of the copper atom enabled all the other non-hydrogen atoms of the complex to be located. Least-squares refinement of atomic positions and isotropic temperature factors brought the residual index, R, down to 0.20 where $R = \Sigma(||F_o| - |F_c||)/\Sigma|F_o|$. At this stage a difference Fourier synthesis revealed the presence of a hydrate oxygen atom. Further least-squares refinement of atomic positions and anisotropic temperature factors was followed by difference Fourier syntheses in which all hydrogen atom positions were located.

All atomic parameters were then further refined by full-matrix least-squares methods (PROGRAM ORFLS)[4] in which the hydrogen atoms were given the same anisotropic temperature factors as the atoms to which they were attached. The function minimized was $\Sigma w(|F_o| - |F_c|)^2$, where w is the weight of a reflection. In the refinement each reflection was assigned a weight of unity thereby making the weighted R equal to the conventional R defined above. For the anisotropic refinement the thermal parameters were of the form

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

After the last cycle of refinement R had settled at 0,035 and the average e.s.d. in the positional parameters of the atoms was about five times the average parameter shift. In the structure factor calculations, scattering factors for the copper, carbon, nitrogen, oxygen and hydrogen atoms were those obtained from Hanson et al.[5] The copper was treated as Cu^0 and the anomalous dispersion correction ($\Delta f' = 0.3$ for MoK$_\alpha$ radiation) was applied to the scattering curve. Extinction was not regarded as being significant and therefore no correction was made.

Final atomic positional and thermal parameters for the non hydrogen atoms are given in Table I while the atomic positional parameters of the hydrogen atoms are given in Table II. Table VIII lists observed and calculated structure factors.

TABLE I. Non Hydrogens Atoms. Fractional Atomic Co-ordinates $(X10^4)$ and Thermal Parameters $(X10^4)$ and Their e.s.ds.

Atom	X	Y	Z	β_{11}	β_{22}	β_{33}	β_{12}	β_{23}	β_{31}
Cu	0	0	0	30(1)	19(1)	52(1)	−2(1)	−2(1)	−2(1)
C(2)	1450(4)	280(3)	2754(5)	42(6)	20(3)	99(9)	0(3)	13(6)	−7(4)
C(4)	3506(4)	844(3)	3606(5)	37(6)	40(3)	55(8)	−10(4)	4(5)	−7(4)
C(5)	3654(4)	940(3)	2063(5)	39(5)	30(3)	45(8)	−2(4)	4(5)	−7(4)
C(6)	2535(4)	632(3)	946(5)	55(6)	16(3)	51(8)	7(3)	0(6)	−2(4)
C(7)	3863(5)	1926(4)	1767(7)	71(7)	37(4)	110(10)	−13(4)	2(6)	−2(5)
C(8)	2863(7)	2531(4)	1996(9)	119(9)	38(4)	194(13)	1(5)	18(8)	−5(6)
C(9)	4768(5)	394(4)	1868(6)	47(6)	49(4)	82(9)	3(4)	3(6)	−4(5)
C(10)	4604(7)	−598(5)	1982(7)	95(8)	57(5)	103(10)	34(5)	19(7)	8(5)
C(12)	54(5)	1864(4)	−847(6)	65(7)	46(4)	130(10)	1(4)	8(7)	5(5)
C(13)	−281(6)	2741(4)	−951(7)	128(10)	30(4)	193(13)	−2(5)	1(9)	17(6)
C(14)	−1240(6)	3018(5)	−352(7)	136(10)	34(4)	176(13)	22(5)	−9(9)	−8(6)
C(15)	−1875(5)	2422(4)	332(6)	90(7)	40(4)	118(11)	22(5)	−6(7)	−5(5)
C(16)	−1471(5)	1548(4)	385(6)	63(6)	34(4)	97(9)	8(4)	10(6)	−1(5)
C(17)	−2917(8)	2690(7)	967(11)	172(14)	85(8)	236(19)	50(8)	68(13)	1(9)
N(1)	1525(3)	345(2)	1354(4)	45(5)	25(2)	48(6)	−7(3)	4(4)	2(3)
N(3)	2440(4)	512(3)	3842(4)	50(5)	46(3)	48(6)	−15(3)	15(5)	−9(3)
N(11)	−536(3)	1276(2)	−181(4)	37(4)	24(3)	87(7)	−3(3)	−5(5)	−7(3)
O(2)	507(3)	19(3)	3090(4)	68(4)	59(3)	140(6)	−16(3)	35(4)	−10(4)
O(4)	4293(3)	1079(3)	4617(3)	74(4)	80(3)	60(6)	−26(3)	−11(4)	−8(3)
O(6)	2591(3)	660(2)	−349(3)	75(4)	52(3)	54(5)	−3(3)	1(4)	−2(3)
O(18)	1926(3)	4826(3)	1650(4)	86(4)	60(3)	85(6)	19(3)	31(4)	13(3)

TABLE II. Hydrogen Atoms. Fractional Atomic Coordinates $(X10^3)$ and Their e.s.ds.

Atom	X	Y	Z
H(3)	228(4)	53(4)	473(5)
H(7 1)	461(5)	204(4)	234(6)
H(7 2)	411(5)	193(4)	67(6)
H(8 1)	270(6)	257(4)	298(7)
H(8 2)	195(6)	238(4)	156(7)
H(8 3)	294(6)	316(4)	174(7)
H(9 1)	547(5)	60(4)	268(6)
H(9 2)	495(5)	58(4)	87(5)
H(10 1)	389(5)	−82(4)	127(6)
H(10 2)	441(5)	−78(4)	295(6)
H(10 3)	527(5)	−93(4)	175(6)
H(12)	67(5)	162(4)	−134(6)
H(13)	17(6)	314(4)	−132(7)
H(14)	−151(6)	363(4)	−27(7)
H(16)	−187(5)	107(4)	90(6)
H(17 1)	−337(7)	237(5)	110(8)
H(17 2)	−259(7)	310(5)	179(8)
H(17 3)	−337(6)	312(5)	22(8)
H(18 1)	98(5)	488(4)	165(6)
H(18 2)	212(5)	462(4)	238(6)

TABLE III. Intramolecular Bond Lengths and Their e.s.ds in Å

Cu–N(1)	1.980(5)	N(3)–H(3)	0.90(5)
N(1)–C(2)	1.346(6)	C(7)–H(7 1)	0.91(5)
C(2)–N(3)	1.391(6)	C(7)–H(7 2)	1.13(6)
N(3)–C(4)	1.356(7)	C(8)–H(8 1)	0.99(7)
C(4)–C(5)	1.508(7)	C(8)–H(8 2)	1.05(6)
C(5)–C(6)	1.534(6)	C(8)–H(8 3)	0.98(6)
C(6)–N(1)	1.341(6)	C(9)–H(9 1)	1.02(5)
C(2)–O(2)	1.229(6)	C9–H(9 2)	1.04(5)
C(4)–O(4)	1.214(6)	C10–H(10 1)	0.99(6)
C(6)–O(6)	1.239(6)	C10–H(10 2)	1.02(6)
C(5)–C(7)	1.544(8)	C10–H(10 3)	0.97(6)
C(7)–C(8)	1.50(1)	C12–H(12)	0.99(6)
C(5)–C(9)	1.539(8)	C13–H(13)	0.90(6)
C(9)–C(10)	1.52(1)	C14–H(14)	0.99(6)
Cu–N(11)	2.018(3)	C16–H(16)	1.02(5)
N(11)–C(12)	1.340(7)	C17–H(17 1)	0.74(8)
C(12)–C(13)	1.376(9)	C17–H(17 2)	1.00(8)
C(13)–C(14)	1.38(1)	C17–H(17 3)	1.01(8)
C(14)–C(15)	1.39(1)		
C(15)–C(16)	1.396(9)		
C(16)–N(11)	1.335(7)	O(18)–H(18 1)	1.06(6)
C(15)–C(17)	1.48(1)	O(18)–H(18 2)	0.74(5)

Description of the Structure and Discussion

The structure of the molecule is shown in Figure 1 (ORTEP).[6] The intramolecular bond lengths and angles and their associated e.s.ds are given in Tables III

and IV respectively. These parameters were calculated using the function and error program ORFFE.[4] Table V lists computed least-squares planes with their equations and the distances of various atoms from these planes.
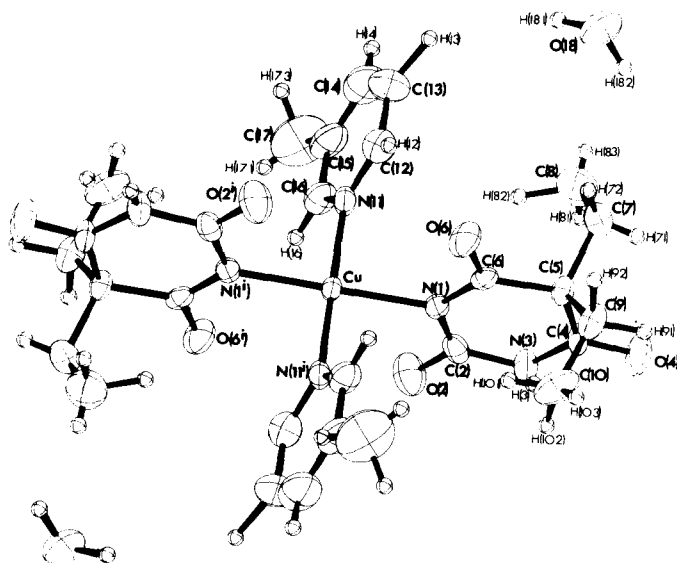
Figure 1. The molecular structure of the complex. The superscripted atoms are centrosymmetrically related to the corresponding atoms without superscripts.

Environment of the Copper Atom

The copper atom is co-ordinated to the barbital anions via their deprotonated nitrogen atoms N(1) and N(1^i) and to the β-picoline moieties via their nitrogen atoms N(11) and N(11^i) in square planar arrangement, the bond angles N(1)–Cu–N(11) and N(1)–Cu–N(11^i) being 90.3(1)° and 89.7(1)° respectively. The bond length for Cu–N(1) is 1.980(5) and compares very favourably with the 1.983(5) Å found by Caira et al.[3] The Cu–N(11) distance is 2.018(3) Å as opposed to 2.032(5) Å found by Caira and 1.989(6) Å reported by Davey and Stephens[7] in the complex trans-Bis[(chloroacetato)-(α-picoline)] copper(II).

Two centrosymmetrically related oxygen atoms O(2) and O(2^i) lie above and below the plane through atoms Cu,N(1), N(11), N(1^i) and N(11^i) (plane I) at a distance of 2.857(4) Å from the Cu atom. This is less than the sum of the van der Waals radii of 3.4 Å for copper and oxygen and it would therefore appear that O(2) and O(2^i) are involved in 'off-the-z-axis' co-ordination with Cu(Figure 2). The z axis is taken as the normal to plane I giving the 'off-the-z-axis' angle as 39.0°. Davey and Stephens[7] also found this type of co-ordination with a Cu–O distance of 2.707 Å while Caira reports this length as 2.723(5) Å. The respective angles are 36.1° (Davey and Stephens) and 35.7° (Caira et al.). A second, but probably weaker interaction exists between oxygen atoms O(6) and O(6^i) and copper. Here the Cu–O(6) distance is 3.149(4) Å.

All interatomic distances and angles involved in this 'off-the-z-axis' co-ordination are given in Table VI.
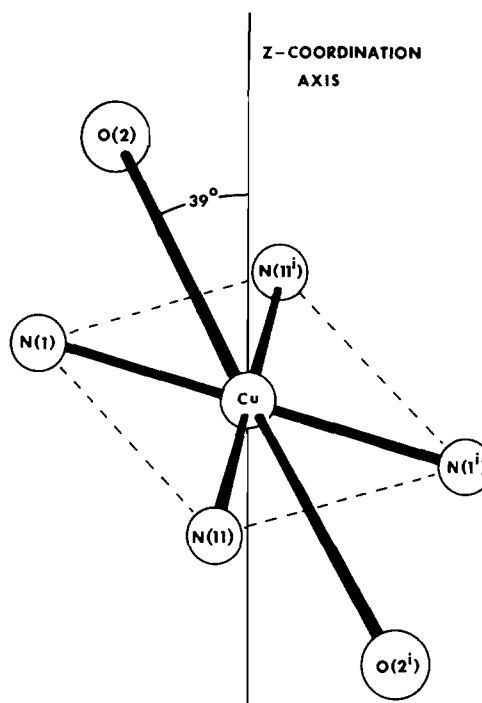

Figure 2. The environment of the copper atom showing the 'off-the-z-axis' co-ordination involving O(2) and O(2^i).

In the β-picoline moiety, the average C–C and C–H bond lengths within the ring are 1.38 Å and 0.98 Å respectively. The ring is planar, the greatest deviation from the least-squares plane through the six atoms

TABLE IV. Intramolecular Bond Angles (in degrees).

N(1)–Cu–N(11)	90.3(1)	C(7)–C(5)–C(9)	109.8(4)
N(1)–Cu–N(11^i)	89.7(1)	C(6)–C(5)–C(7)	107.5(4)
N(1)–C(2)–N(3)	120.4(4)	C(6)–C(5)–C(9)	108.7(4)
C(2)–N(3)–C(4)	124.4(4)	C(5)–C(7)–C(8)	114.7(5)
N(3)–C(4)–C(5)	118.2(4)	C(5)–C(9)–C(10)	114.1(5)
C(4)–C(5)–C(6)	113.5(4)	N(11)–C(12)–C(13)	121.2(6)
C(5)–C(6)–N(1)	121.1(4)	C(12)–C(13)–C(14)	119.2(6)
C(6)–N(1)–C(2)	122.3(4)	C(13)–C(14)–C(15)	120.7(6)
N(1)–C(2)–O(2)	120.6(4)	C(14)–C(15)–C(16)	116.0(5)
N(3)–C(2)–O(2)	119.0(4)	C(15)–C(16)–N(11)	123.4(5)
N(3)–C(4)–O(4)	120.0(4)	C(16)–N(11)–C(12)	119.2(4)
C(5)–C(4)–O(4)	121.9(4)	C(14)–C(15)–C(17)	122.4(6)
C(5)–C(6)–O(6)	118.0(4)	C(16)–C(15)–C(17)	121.5(6)
N(1)–C(6)–O(6)	120.9(4)		
C(4)–C(5)–C(9)	108.6(4)		
C(4)–C(5)–C(7)	108.6(4)	H(18 1)–O(18)–H(18 2)	97(5)

TABLE V. Least-squares Planes. The equations of the planes are expressed in orthogonalised space as $lx + my + nz = d$.

Plane I	square planar co-ordination around copper atom						
Atoms defining the plane	Cu	N(1)	N(11)	N(1^i)	N(11^i)		
Atoms not included in the plane						O(2)	O(6)
Distance from the plane, Å	0.0	0.0	0.0	0.0	0.0	2.221	–2.226
Equation	$-0.62671l - 0.11646m + 0.77050n = 0.00$						

Plane II	through the 6 atoms of the β picoline ring					
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 plane, Å	0.003	–0.001	–0.003	0.005	–0.003	–0.001	–0.015
Equation	$0.46166l + 0.18499m + 0.86755n = -0.05210$					

Plane III	through the 6 pyrimidine ring atoms							
Atoms defining the plane	N(1)	C(2)	N(3)	C(4)	C(5)	C(6)		
Atoms not included in plane							O(2)	O(4)	O(6)
Distance from the plane, Å	0.008	0.011	–0.017	0.006	0.011	–0.018	0.043	0.060	–0.060
Equation	$-0.36411l + 0.93093m - 0.02822n = -0.08297$							

Plane IV	through the trioxopyrimidine ring atoms								
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	0.018	–0.003	–0.046	–0.015	0.017	0.003	0.019	0.025	–0.017
Equation	$-0.36090l + 0.93147m - 0.04599n = -0.11030$								

Plane V	through the atoms of the diethyl group and C(5)				
Atoms defining the plane	C(5)	C(7)	C(8)	C(9)	C(10)
Distance from the plane, Å	0.026	–0.040	0.006	0.044	–0.036
Equation	$0.20985l + 0.09277m + 0.97332n = 2.73546$				

Intersection Angles

Planes				Planes		
	I and II	69.05°			II and III :	91.17°
	I and III	84.37°			III and IV :	1.30°
	I and IV	85.28°			III and V :	91.00°

TABLE VI. Interatomic Distances (Å) and Bond Angles (degrees) and Their e.s.ds Involved in the "Off-the-Z-Axis Co-ordination".

Cu–N(1)	: 1.980(5)	N(1)–Cu–O(2)	:	51.3(1)
Cu–N(11)	: 2.018(3)	N(11)–Cu–O(2)	:	94.00(5)
Cu–O(2)	: 2.857(4)	N(1^i)–Cu–O(2)	:	128.7(1)
N(1)–O(2)	: 2.237(5)	N(11^i)–Cu–O(2)	:	86.00(5)
N(11)–O(2)	: 3.611(3)			
N(1^i)–O(2)	: 4.376(7)			
N(11^i)–O(2)	: 3.380(4)			
N(1)–N(11)	: 2.833(6)			
N(1)–N(11^i)	: 2.820(5)			

(plane II) being that of atom C(14) at a distance of 0.005 Å. The β methyl group lies at a distance 0.015 Å from this plane. Planes I and II intersect at an angle 69.05°.

The Barbital Ligand Structure

The conformation of the trioxopyrimidine ring is shown in Figure 3 in which the dotted line is a trace of the least-squares plane through the ring atoms only (Plane III). The ring is almost planar, the greatest deviation from this plane being that of atom N(3) at a distance of 0.017 Å. However, slight puckering of the ring is evident. In the structure reported by Caira the puckering is more pronounced with the largest deviation from the plane being that of atom C5. Its distance from the plane is 0.1 Å. when the three exocyclic oxygens are included in the least-squares plane calculation, it is seen (Plane IV) that the entire trioxopyrimidine moiety is virtually planar, the largest deviation from the plane being that of atom N(3) at a distance of 0.04 Å. Planes III and IV intersect at an angle of 1.30° indicating the coplanarity of the exocyclic oxygen atoms and the pyrimidine ring atoms.

Atoms C(7), C(8), C(9) and C(10) of the two ethyl groups as well as atom C(5) to which they are attached lie in the same plane (Plane V) which is virtually perpendicular (91.0°) to the pyrimidine ring plane (Plane III). The hydrogen atoms of the two ethyl groups are in the staggered configuration. Planes I and III intersect at 84.37°, (Caira 86.7°) i.e. the
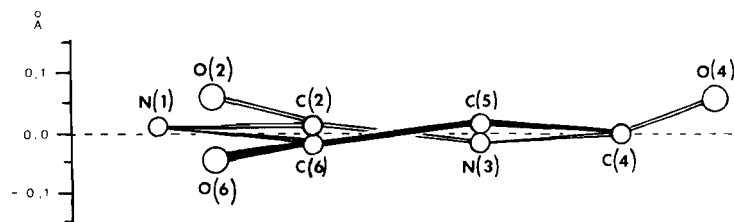

Figure 3. The conformation of the trioxopyrimidine ring. The dotted line is a trace of the best least-squares plane through the six ring atoms only (plane III). The vertical scale is approximately five time the horizontal scale.
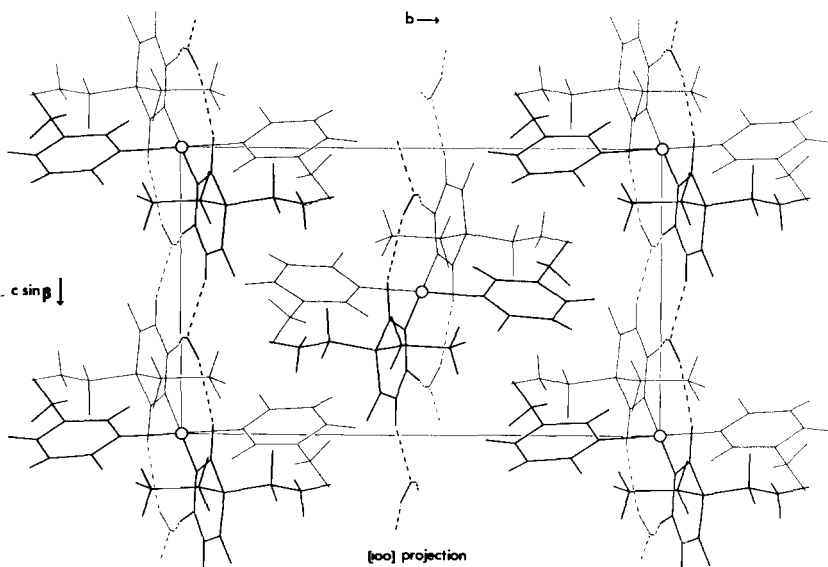

Figure 4. The [100] projection of the structure.

plane Cu, N(1), N(11) and the pyrimidine ring plane are almost perpendicular. Similarly the planes through the two different moieties of the molecule, *i.e.* through the picoline ring (Plane II) and the pyrimidine ring (Plane III), are at right angles to each other (91.17°).

The internal ringle angle C(2)–N(1)–C(6) is 122.3(4)°, which compares favourably with the 121.6° found in Zn(II)(barb)$_2$(imidazole)$_2$. The corresponding value in the barbital molecule is 126.4° while in the barbital ion it is 119.2°.[8] Interest is centred on this angle as deprotonation occurs at N(1).

Hydrogen Bonding

The intermolecular hydrogen bonding is shown as dotted lines in Figure 4, the [100] projection of the structure. Each discrete molecule of the complex is linked to two other molecules via four bridging water molecules. This gives a sheet-like structure in which the layers comprising the barbiturate anions and the water molecules are orientated approximately parallel to the x-z plane of the crystal lattice.

There are thus 3 crystallographically distinct hydrogen bonds emanating from each water molecule. The hydrogens of the water molecule H(18.1) and H(18.2) are each hydrogen-bonded to oxygens O(2) and O(6^i) respectively of the barbital moieties of a single molecule of the complex at (x,y,z). The hydrate oxygen is hydrogen-bonded to an N–H group of the molecule at (x,y,z−1).

The geometry of the interactions is shown in Figure 5. The hydrogen bond distances are all in good agreement with those listed by Donohue[9] for an assort-
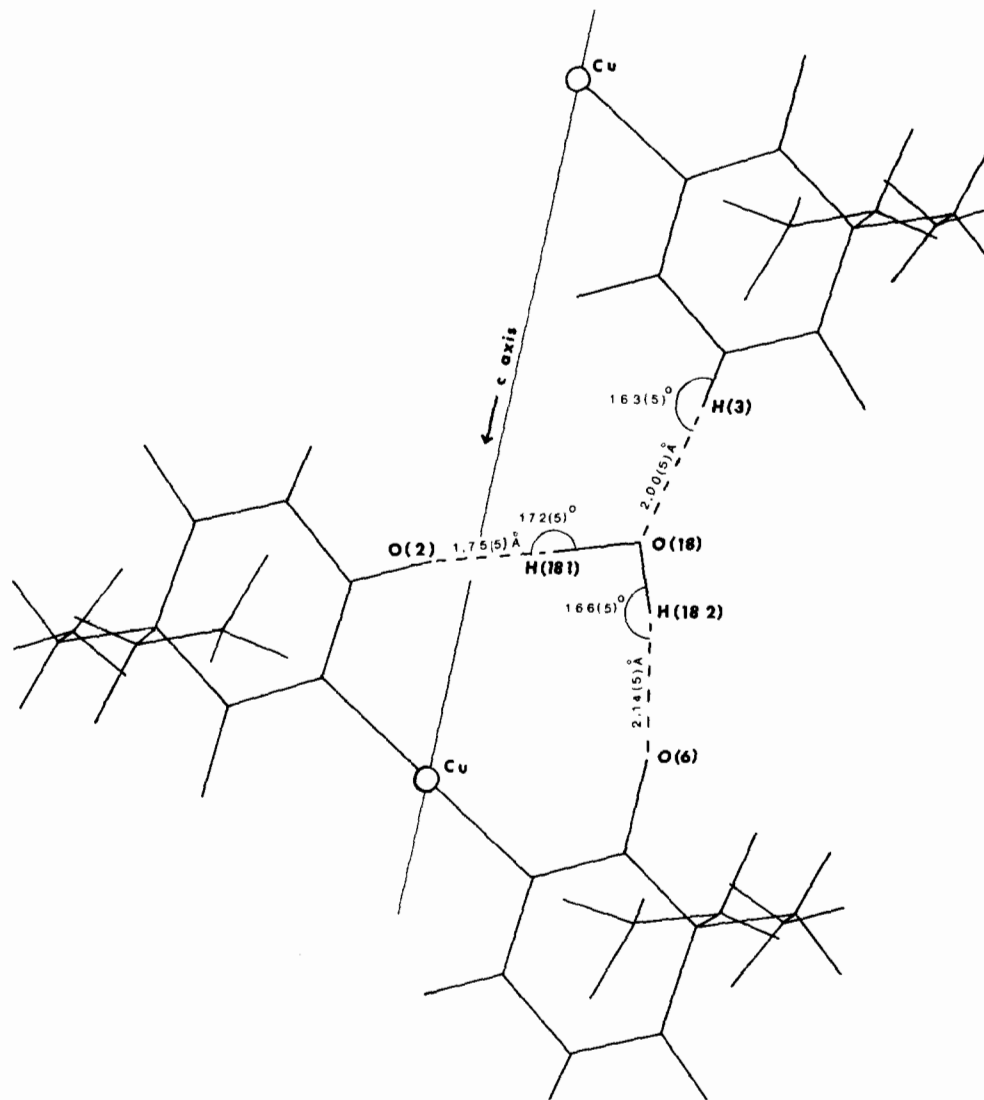

Figure 5. Schematic diagram of the geometry of the hydrogen bond interactions.

ment of hydrogen-bonded compounds. Figure 5 shows the near linearity of each of the 3 hydrogen bonds with bond angles well within the maximum allowed deviation from 180°.[9] All bond lengths and angles involved in the hydrogen bonding are listed in Table VII.

Intramolecular hydrogen bonding of the type reported in the complex Zn(II)(barb)$_2$(imidazole)$_2$ was found to be absent. In the structure Cu(II)(barb)$_2$ (pyridine)$_2$, which is not hydrated, the only intermolecular hydrogen bonding is of the type NH····O=C linking adjacent barbital moieties.

TABLE VII. Bond Lengths (Å) and Angles (degrees) and their e.s.ds Involved in the Hydrogen Bonding.

O(2)–H(18 1)		1.75(5)
O(2)–O(18)		2.801(5)
O(6)–H(18 2)		2.14(5)
O(6)–O(18)		2.874(5)
H(3)–O(18)		2.00(5)
N(3)–O(18)		2.875(6)
O(2)–H(18 1)–O(18)	:	172(5)
O(6)–H(18 2)–O(18)	:	166(5)
N(3)–H(3)–O(18)	:	163(5)

TABLE VIII. Observed and Calculated Structure Factors.

h	k	F_o	F_c
		ℓ = 0	
2	0	225	216
3	0	286	302
4	0	816	807
5	0	709	695
6	0	442	432
7	0	503	499
8	0	428	430
10	0	128	-124
7	1	283	276
5	1	111	-115
4	1	140	129
3	1	198	180
2	1	1038	1019
1	2	293	282
2	2	443	438
4	2	655	645
5	2	414	413
6	2	253	259
7	2	113	121
8	2	331	342
9	2	194	197
10	2	115	-95
9	3	226	-236
7	3	118	117
4	3	229	223
3	3	330	332
1	3	1315	-1327
0	4	1319	1351
1	4	1041	1047
3	4	150	144
4	4	796	776
5	4	343	340
6	4	595	592
7	4	371	374
8	4	240	243
9	4	147	130
10	4	318	299
9	5	291	-309
8	5	105	-98
7	5	132	143
6	5	129	110
1	5	990	-985
0	6	72	76
1	6	1224	1208
2	6	908	908
3	6	125	132
4	6	118	111
5	6	444	447
6	6	481	484
7	6	316	318
8	6	211	195
9	6	173	145
8	7	150	-148
7	7	132	-116
6	7	150	-143
4	7	138	150
3	7	157	144
0	8	235	250
1	8	815	726
3	8	387	392
4	8	201	189
5	8	339	345
6	8	379	384
7	8	174	177
8	8	175	-189
8	9	81	-58
6	9	286	283
4	9	191	205
3	9	82	77
2	9	318	-323
0	10	229	217
1	10	321	313
2	10	628	628
3	10	572	580
5	10	431	432
6	10	247	238
7	10	91	103
5	11	269	254
4	11	325	329
3	11	245	-248
0	12	232	231
1	12	203	199
2	12	147	138
3	12	438	405
4	12	217	215
5	12	270	252
0	14	150	143
1	14	251	244
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-4	1	746	739
-3	1	1043	1031
-2	1	259	264
2	1	741	754
3	1	110	-82
4	1	546	574
5	1	130	137
6	1	473	472
7	1	361	360
8	1	425	429
9	1	232	-236
10	1	216	205
10	2	184	185
8	2	169	-170
7	2	266	271
6	2	68	59
4	2	162	177
3	2	399	382
2	2	153	158
-2	2	174	157
-3	2	504	-491
-4	2	520	506
-5	2	153	-145
-6	2	190	-181
-7	2	127	-127
-8	2	167	-172
-9	3	525	542
-8	3	453	448
-7	3	620	620
-6	3	95	103
-5	3	285	297
-4	3	583	579
-3	3	563	570
2	3	105	-93
3	3	91	117
4	3	224	243
6	3	472	475
1	4	83	-78
3	4	277	274
4	4	89	-82
7	3	337	332
0	4	417	-390
10	3	208	212
8	3	240	250
8	4	247	-238
7	4	64	51
5	4	73	77
-1	4	114	-116
-2	4	418	429
-3	4	452	-443
-4	4	69	-66
-5	4	127	125
-6	4	242	-234
-7	4	89	-74
-8	4	89	-96
-9	4	75	50
-7	5	410	409
-6	5	350	345
-5	5	690	698
-9	5	400	405
-10	5	260	243
-8	5	441	449
-4	5	287	314
-1	5	115	-116
2	5	429	432
5	5	454	465
8	6	320	-317
-2	3	857	862
-1	3	187	192
0	3	307	303
1	3	89	-61
4	6	68	-43
3	6	168	-165
2	6	145	-143
1	6	475	-475
0	6	318	-319
-1	6	284	274
-3	6	502	493
-4	6	151	163
-5	6	152	170
-6	6	77	-94
-3	5	518	528
-2	5	886	896
-1	5	115	-116
1	5	132	137
2	5	429	432
3	5	270	275
4	5	157	153
5	5	454	465
6	5	521	518
7	5	394	398
8	5	95	100
9	5	328	333
8	6	320	-317
5	6	114	-106
-9	7	177	174
-8	7	334	336
-7	7	368	369
-6	7	409	428
-5	7	309	331
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-3	7	505	532
-2	7	853	871
-1	7	367	364
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1	7	896	896
2	7	288	306
3	7	291	295
5	7	153	158
6	7	412	415
7	7	201	194
7	8	268	-259
5	8	153	169
4	8	67	50
3	8	191	179
2	8	188	191
-1	8	207	-215
-2	8	138	-137
-3	8	350	351
-4	8	170	-171
-5	8	124	142
-7	8	169	-170
-8	8	137	-131
-6	9	225	219
-6	9	346	347
-5	9	229	238
-4	9	404	418
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-2	9	269	281
-1	9	255	252
0	9	70	-43
2	9	352	348
3	9	218	218
5	9	357	360
6	9	188	201
7	10	201	-200
5	10	199	203
3	10	64	75
0	10	66	-64
-2	10	174	-187
-3	10	163	178
-5	10	171	-165
-6	10	185	-174
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-6	11	384	387
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-3	11	531	545
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0	11	198	175
1	11	276	272
2	11	428	414
3	11	273	265
4	11	208	189
5	11	351	350
5	12	192	188
4	12	202	206
1	12	117	145
0	12	70	-84
-1	12	101	83
-3	12	118	123
-4	12	89	116
-3	13	84	69
-1	13	112	107
0	13	224	215
1	13	248	257
3	13	157	105
4	13	110	85
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-1	14	125	-119
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-2	0	440	456
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0	0	718	712
1	0	1082	1077
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3	0	245	257
4	0	920	915
5	0	668	643
6	0	127	121
7	0	445	446
8	0	221	213
9	0	268	273
9	1	199	193
8	1	71	94
7	1	110	116
6	1	100	89
5	1	149	-146
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3	1	355	347
2	1	980	968
1	1	1067	1066
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-3	1	63	36
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-7	1	120	-124
-9	1	103	112
-10	2	175	178
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-8	2	352	348
-7	2	586	590
-6	2	257	-215
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-3	2	738	747
-2	2	211	242
-1	2	133	167
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1	2	137	-112
2	2	159	-132
3	2	1011	1014
4	2	1054	1038
5	2	508	503
6	2	172	161
7	2	213	198
8	2	380	384
9	2	304	306
9	3	119	132
8	3	112	-119
7	3	66	71
5	3	408	-400
4	3	239	226
3	3	475	472
2	3	121	-118
1	3	953	-975
0	3	334	332
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-10	4	335	345
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-8	4	390	408
-7	4	298	304
-6	4	388	387
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-4	4	73	-26
-3	4	665	624
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3	4	498	492
4	4	633	636
5	4	546	543
6	4	494	499
7	4	114	118
8	4	380	388
9	4	326	331
8	5	85	101
7	5	87	60
6	5	117	-115
5	5	176	-175
4	5	92	-102
3	5	141	140
0	5	234	-226
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-4	5	159	153
-5	5	324	307
-6	5	162	-167
-7	5	181	-186
-8	5	76	96
-9	5	181	177
-9	6	210	197
-8	6	253	246
-7	6	300	307
-5	6	68	45
-4	6	70	-43
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-2	6	391	403
2	6	611	606
3	6	619	622
4	6	367	370
5	6	358	362
6	6	392	387
7	6	292	286
8	6	431	429
6	7	218	-214
5	7	155	-162
4	7	78	-62
3	7	440	443
1	7	62	65
0	7	123	-128
-1	7	173	142
-2	7	223	-215
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-6	8	392	416
-5	8	412	413
-4	8	207	213
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-1	8	157	167
0	8	311	313
1	8	174	170
2	8	556	568
3	8	453	455
4	8	112	112
5	8	382	384
6	8	227	238
7	8	167	173
7	9	103	105
6	9	272	-255
5	9	147	136
1	9	200	155
0	9	277	-286
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-2	9	171	-163
-3	9	201	226
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-5	9	113	-124
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-7	9	216	-227
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1	10	145	146
2	10	481	440
3	10	290	279
4	10	370	368
5	10	224	228
6	10	155	152
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-2	11	71	-55
-5	11	129	-143
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2	12	281	264
3	12	217	189
4	12	230	219
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-1	14	193	169
0	14	263	302
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2	1	171	190
3	1	853	866
4	1	657	654
5	1	276	288
6	1	219	205
7	1	231	232
8	1	200	211
9	1	217	208
9	2	82	-80
8	2	157	145
6	2	177	167
3	2	739	718
2	2	82	102
1	2	138	145
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-2	2	150	148
-3	2	175	-170
-4	2	142	134
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-7	2	145	-154
-8	2	87	-92
-10	2	120	106
-10	3	297	297
-9	3	299	307
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-4	3	226	234
-3	3	399	403
-2	3	438	438
-1	3	672	674
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1	3	365	370
2	3	131	143
3	3	620	628
4	3	359	361
5	3	643	648
6	3	145	142
7	3	304	304
8	3	243	248
7	4	100	66
6	4	71	-61
5	4	149	-155
4	4	252	-250
3	4	109	110
2	4	124	137
1	4	180	-184
0	4	190	-188
-1	4	446	444
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-3	4	301	-294
-4	4	330	-303
-6	4	236	240
-7	4	100	-93
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-7	5	207	213
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-5	5	372	386
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-1	5	352	362
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1	5	514	530
2	5	573	569
3	5	538	529
4	5	439	437
5	5	548	553
6	5	279	283
7	5	356	352
8	5	142	140
8	6	89	-70
7	6	116	127
5	6	132	-144
4	6	121	-112
3	6	62	-41
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3	7	86	94
4	7	197	195
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4	8	101	86
2	8	279	277
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0	8	58	46
-1	8	58	79
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-5	8	118	127
-6	8	163	168
-8	8	86	-80
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-7	9	153	164
-5	9	210	208
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2	9	528	533
3	9	189	188
4	9	382	391
5	9	258	262
6	9	407	419
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-3	11	209	205
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0	11	222	226
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2	11	276	279
3	11	125	126
4	11	310	307
5	11	210	189
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-2	13	161	158
-1	13	203	180
0	13	445	434
1	13	234	215
2	13	228	235
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-4	0	833	812
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1	0	989	994
2	0	246	247
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5	0	219	220
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8	0	97	76
7	1	89	75
3	1	330	326
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0	1	439	427
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-4	1	125	110
-5	1	213	217
-6	1	211	-212
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-6	2	364	376
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4	2	176	190
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6	2	172	175
7	2	354	340
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8	3	84	-76
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-7	3	160	-152
-9	3	124	-145
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-1	4	168	161
0	4	67	58
1	4	372	391
2	4	327	326
4	4	260	256
5	4	232	223
6	4	336	334
7	4	286	287
8	4	202	190
5	5	137	-124
4	5	164	-167
3	5	193	-197
2	5	154	-153
1	5	149	132
0	5	64	71
-1	5	88	-78
-2	5	157	-154
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-4	5	352	344
-7	5	77	57
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-9	5	85	-73
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-6	6	283	261
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3	6	327	330
4	6	145	164
5	6	70	49
6	6	318	309
7	6	166	166
7	7	113	-108
5	7	73	69
3	7	84	-86
1	7	260	266
0	7	58	61
-1	7	124	-127
-3	7	87	-71
-4	7	176	178
-6	7	162	165
-8	8	267	248
-7	8	214	222
-6	8	276	264
-5	8	504	507
-4	8	437	448
-3	8	519	530
-2	8	292	295
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Acknowledgements

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