Table 5. *Hydrogen-bond distances and angles*

i	*j*	*k*	*D*(*ij*)	*D*(*jk*)	∠*ijk*
N	Cl	O(*W*)	3·07 Å	3·23 Å	95·1°
N	Cl	O′(*W*)^a	3·07	3·25	67·8
O(*W*)	Cl	O′(*W*)^a	3·23	3·25	71·2
N′	Cl′	O′(*W*)	3·11	3·13	94·7
N′	Cl′	O(*W*)^b	3·11	3·23	70·3
O′(*W*)	Cl′	O(*W*)^b	3·13	3·23	73·1
Cl	O(*W*)	Cl′^{a}	3·23	3·22	106·9
Cl′	O′(*W*)	Cl^b	3·13	3·25	108·5

Intermolecular non-bonded distances less than 3·5 Å

i	*j*	*D*(*ij*)	*i*	*j*	*D*(*ij*)
F(1)	O(*A*)^c	3·45 Å	O(*A*)	C′(20)^e	3·23 Å
F(1)	C(*A*)1^c	3·28	F′(1)	O′(*A*)^d	3·15
F(1)	C(*A*)3^c	3·30	O′(*W*)	C(19)^b	3·50
F(3)	F′(3)^c	3·09	O′(*W*)	C(21)^b	3·39
O(*W*)	C(16)^d	3·50	O′(*A*)	C(20)^f	3·43
O(*W*)	C′(21)^b	3·44	O′(*A*)	C′(20)^g	3·45
O(*A*)	C(8)	3·46			

Symmetry code

Superscript							
None	x,	y,	z	(*d*)	x,	y,	$-1+z$
(*a*)	$1-x$,	$-\frac{1}{2}+y$,	$-z$	(*e*)	$-x$,	$\frac{1}{2}+y$,	$-z$
(*b*)	$1-x$,	$\frac{1}{2}+y$,	$-z$	(*f*)	$-x$,	$\frac{1}{2}+y$,	$1-z$
(*c*)	$1+x$,	y,	$1+z$	(*g*)	x,	y,	$1+z$

for kindly supplying the crystal and for many interesting discussions.

References

Bordner, J., Stanford, R. H. Jr & Zieger, H. E. (1973). *Acta Cryst.* B**29**, 313–318.

Brennan, T., Putkey, E. F. & Sundaralingam, M. (1971). *J. Chem. Soc.* (*D*), pp. 1490–1491.

Brinkman, A. W., Gordon, M., Hawey, R. G., Rabideau, P. W., Stothers, J. B. & Ternay, A. L. (1970). *J. Amer. Chem. Soc.* **92**, 5912–5916.

Chu, S. S. C. (1973). *Structure Interpretation Programs.* Institute of Technology, Southern Methodist Univ.

Cotton, F. A. & Norman, J. G. (1972). *J. Amer. Chem. Soc.* **94**, 5697–5702.

Cruickshank, D. W. J. (1965). *Computing Methods in Crystallography*, edited by J. S. Rollett, pp. 112–116. New York: Pergamon Press.

Domino, E. F. (1967). *Psychopharmacology, a Review of Progress*, edited by D. H. Efron, pp. 1045–1056.

Fowler, P. J., Tedeschi, D. H., Zirkle, C. & Macko, E. (1971). *Pharmacologist*, **13**, 235.

Germain, G., Main, P. & Woolfson, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

International Tables for X-ray Crystallography (1962). Vol. III, pp. 201–207. Birmingham: Kynoch Press.

Johnson, C. K. (1965). *ORTEP.* Oak Ridge National Laboratory Report ORNL-3794.

Johnston, D. L., Rohrbaugh, W. L. & Horrocks, W. D. Jr (1971). *Inorg. Chem.* **10**, 1474–1479.

Phelps, D. W. & Cordes, A. W. (1974). *Acta Cryst.* B**30**, 2812–2816.

Shiono, R. (1971). Technical Report 49, Crystallography Department, Univ. of Pittsburgh.

Stanford, R. H. Jr (1973). *Acta Cryst.* B**29**, 2849–2855.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Zirkle, C. L. & Kaiser, C. (1970). *Medicinal Chemistry*, 3rd ed., edited by A. Burger, Part II, pp. 1410–1497. New York: Wiley–Interscience.

Zirkle, C. L. & Kaiser, C. (1974). Private communication.

Acta Cryst. (1976). B**32**, 842

The Crystal and Molecular Structure of Tris(biguanidato)chromium(III) Monohydrate

By Leandro Coghi, Mario Nardelli and Giancarlo Pelizzi

Istituto di Chimica Generale ed Inorganica, Università di Parma, Centro di Studio per la Strutturistica Diffrattometrica del C.N.R., Parma, Italy

(*Received* 11 *July* 1975; *accepted* 6 *August* 1975)

Crystals of the title compound are monoclinic ($P2_1/c$) with unit-cell dimensions: $a=9{\cdot}260$, $b=10{\cdot}616$, $c=15{\cdot}928$ Å, $\beta=106{\cdot}0°$, $Z=4$. The structure has been determined from diffractometer data by direct methods and refined by block-diagonal least-squares calculations to $R=3{\cdot}6\%$ for 2451 independent reflexions. The main feature in this compound is the deprotonation of the organic ligand, which causes an increase of the π conjugation along the C–N–C system reducing the bond angle on nitrogen to the theoretical value of 120°. Coordination around metal is octahedral and involves six nitrogen atoms of the biguanide molecules, which act as bidentate ligands. The water molecule is involved in four hydrogen bonds with nitrogen atoms of the biguanide molecules.

Introduction

Biguanide (BG) and its substituted derivatives ethylenebisbiguanide (EBG) or 2-aminoethylbiguanide (AEBG) are interesting ligands which form highly coloured complexes with many transition metals, the following X-ray determinations of which have already been reported: $Ni(BG)_2Cl_2$ (Creitz, Gsell & Wampler, 1969), $Cr(BG)_3$-*d*-10-camphorsulphonic acid.$3H_2O$ (Brubaker & Webb, 1969), $Co(BG)_3Cl_3.H_2O$ (Snow, 1974), $Cu(EBG)Cl_2.H_2O$ (Mathew & Kunchur, 1970), $Ni(EBG)Cl_2.H_2O$ (Coghi, Mangia, Nardelli & Pelizzi, 1969; Holian & Marsh, 1970; Ward, Caughlan & Smith, 1971), Ag(EBG)$(NO_3)_3$ (Kunchur, 1968), Ag(EBG)$(ClO_4)_3$ (Simms, Atwood & Zatko, 1973), [Ag(EBG)]$SO_4HSO_4.H_2O$ (Coghi & Pelizzi, 1975), [Cu(AEBG) $(C_2H_4N_4)$]$SO_4.H_2O$ (Coghi, Mangia, Nardelli, Pelizzi & Sozzi, 1968), [Cu(AEBG) (NCS)]SCN (Andreetti, Coghi, Nardelli & Sgarabotto, 1971).

The present paper deals with the structural properties of $Cr(BG)_3.H_2O$, which is the first example of a complex in which the organic ligand occurs in a deprotonated form.

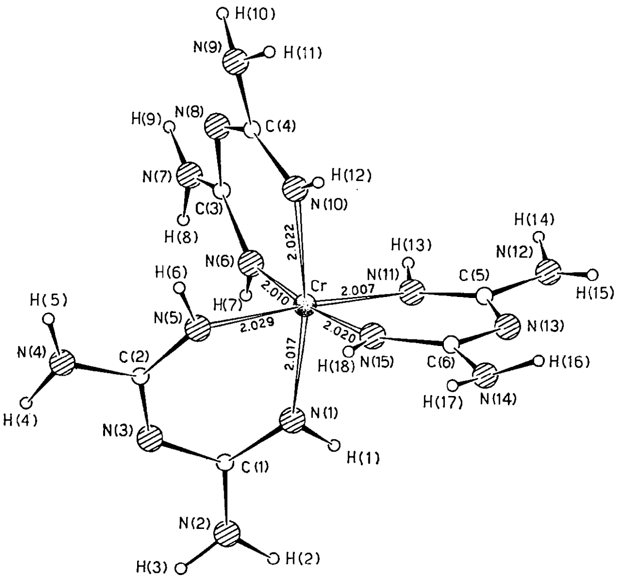

Fig. 1. Clinographic projection of the structure.

Experimental

An aqueous solution of $KCr(SO_4)_2.12H_2O$ was added gradually with stirring to a hot alkaline solution of biguanide sulphate. On cooling in ice, small non-pleochroic orange red prismatic crystals were obtained which were then recrystallized from water containing a small amount of biguanide sulphate and sodium hydroxide.

Accurate cell dimensions were obtained by least-squares calculations based on θ,χ,φ values measured on an automated single-crystal Siemens diffractometer. Crystal data are listed in Table 1. 3954 independent reflexions within the range 5·4–58·0° in 2θ were examined by single-crystal diffractometry (Mo $K\alpha$ radiation, $\lambda=0{\cdot}7107$ Å); of these 1503 were considered 'unobserved', having $I<2\sigma(I)$. The intensities were corrected for Lorentz and polarization effects, but no correction for absorption was applied as the sample used for data collection showed low absorbance ($\mu r=0{\cdot}09$).

Table 1. *Crystallographic data*

$C_6H_{18}CrN_{15}.H_2O$, F. W. 370·4
Monoclinic, space group $P2_1/c$
$a=9{\cdot}260$ (4), $b=10{\cdot}616$ (4), $c=15{\cdot}928$ (6) Å, $\beta=106{\cdot}0$ (1)°,
$U=1504{\cdot}7$ Å^3, $Z=4$, $D_m=1{\cdot}61$ g cm^{-3}, $D_x=1{\cdot}63$ g cm^{-3}
μ(Mo $K\alpha$) = 8·3 cm^{-1}, $F(000)=772$

The structure was solved by direct methods with the weighted numerical addition procedure (Andreetti, 1973). The 1284 reflexions with $|E|\geq 1{\cdot}001$ and the most significant 2000 phase relationships were considered. An E map, computed with the phases from the highest figure of merit, revealed the positions of all

Table 2. *Fractional coordinates* ($\times 10^4$) *and thermal parameters* ($\times 10^2$ Å^2), *with estimated deviations in parentheses, for non-hydrogen atoms*

Anisotropic thermal parameters are in the form $\exp[-\frac{1}{4}(B_{11}h^2a^{*2}+B_{22}k^2b^{*2}+B_{33}l^2c^{*2}+2B_{12}hka^*b^*+2B_{13}hla^*c^*+2B_{23}klb^*c^*)]$

	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Cr	1289 (1)	5319 (1)	3081 (0)	149 (1)	146 (1)	149 (1)	−1 (1)	31 (1)	−3 (1)
O	5003 (3)	3535 (2)	568 (2)	392 (10)	402 (11)	392 (11)	−42 (9)	152 (9)	−95 (9)
N(1)	−686 (2)	4699 (2)	3227 (1)	195 (7)	234 (8)	167 (8)	7 (7)	43 (6)	23 (8)
N(2)	−3108 (2)	3899 (2)	2810 (1)	194 (8)	238 (9)	242 (9)	−34 (7)	65 (7)	13 (7)
N(3)	−2269 (2)	4918 (2)	1771 (1)	203 (8)	247 (9)	191 (8)	−30 (7)	35 (7)	1 (7)
N(4)	−1827 (3)	6052 (3)	659 (2)	343 (11)	554 (16)	216 (10)	−111 (11)	−23 (9)	130 (10)
N(5)	−53 (2)	6174 (2)	2008 (1)	214 (8)	189 (8)	203 (8)	−19 (7)	42 (7)	28 (7)
N(6)	1502 (2)	3755 (2)	2414 (1)	200 (8)	188 (8)	227 (9)	−19 (7)	67 (7)	−32 (7)
N(7)	2357 (4)	2368 (3)	1556 (1)	556 (16)	230 (11)	769 (19)	−91 (11)	461 (15)	−194 (12)
N(8)	3397 (3)	4327 (2)	1728 (2)	237 (9)	220 (9)	332 (11)	−9 (7)	139 (8)	−26 (8)
N(9)	4596 (3)	6211 (2)	1771 (2)	273 (10)	215 (9)	451 (13)	1 (8)	189 (9)	66 (9)
N(10)	3090 (2)	5925 (2)	2710 (2)	195 (8)	202 (9)	251 (9)	−28 (7)	53 (7)	2 (7)
N(11)	2479 (2)	4465 (2)	4178 (1)	232 (8)	186 (9)	212 (9)	19 (7)	16 (7)	7 (7)
N(12)	4059 (3)	4197 (2)	5582 (1)	240 (9)	273 (9)	196 (9)	−11 (7)	27 (7)	67 (8)
N(13)	2791 (3)	6055 (2)	5263 (1)	315 (10)	214 (9)	183 (8)	9 (8)	42 (7)	6 (7)
N(14)	1787 (2)	8019 (2)	5141 (2)	492 (14)	270 (11)	251 (10)	59 (10)	53 (10)	−85 (8)
N(15)	1406 (2)	6832 (2)	3866 (1)	256 (9)	183 (8)	175 (8)	28 (7)	13 (7)	−8 (7)
C(1)	−1965 (3)	4530 (2)	2620 (2)	188 (8)	138 (9)	221 (9)	31 (7)	69 (7)	−10 (8)
C(2)	−1338 (3)	5716 (2)	1517 (2)	225 (10)	203 (9)	165 (9)	15 (8)	48 (8)	6 (7)
C(3)	2392 (3)	3521 (2)	1924 (2)	202 (9)	189 (9)	254 (11)	21 (8)	60 (8)	3 (8)
C(4)	3649 (3)	5475 (2)	2104 (2)	144 (8)	202 (10)	275 (11)	28 (8)	43 (8)	51 (8)
C(5)	3072 (3)	4925 (2)	4963 (2)	179 (8)	211 (10)	191 (9)	−37 (7)	63 (7)	46 (7)
C(6)	1986 (3)	6929 (2)	4715 (2)	249 (10)	204 (10)	207 (10)	−8 (8)	62 (8)	−17 (8)

non-hydrogen atoms including a water molecule not expected on the basis of the conventional chemical analysis data. The refinement was by block-diagonal least-squares calculations with anisotropic thermal parameters, minimizing $\sum w(\Delta|F|)^2$, to an R value of 5·3 %. At this point an F_o-F_c synthesis was used to locate all the H atoms; their contributions were allowed for in the calculations and were refined isotropically. The final R was 3·6 %.

Final atomic and thermal parameters are given in Tables 2 and 3.* Scattering factors for non-hydrogen atoms were from Cromer & Mann (1968); those for H were from Stewart, Davidson & Simpson (1965).

Table 3. *Fractional coordinates* ($\times 10^3$) *and isotropic thermal parameters* (Å^2) *for hydrogen atoms*

	x	y	z	B
H(1)	−70 (3)	437 (3)	373 (2)	2·9 (6)
H(2)	−307 (3)	380 (3)	336 (2)	3·9 (7)
H(3)	−407 (3)	390 (3)	241 (3)	3·5 (6)
H(4)	−280 (3)	594 (3)	38 (2)	4·0 (7)
H(5)	−123 (3)	661 (3)	41 (2)	4·1 (7)
H(6)	44 (3)	672 (3)	172 (2)	3·1 (6)
H(7)	89 (3)	312 (3)	244 (2)	3·4 (7)
H(8)	156 (3)	179 (3)	161 (2)	4·0 (7)
H(9)	297 (3)	220 (3)	123 (2)	4·8 (8)
H(10)	526 (3)	585 (3)	152 (2)	4·1 (7)
H(11)	505 (3)	687 (3)	210 (2)	4·4 (7)
H(12)	343 (3)	670 (3)	293 (2)	3·6 (7)
H(13)	290 (3)	368 (3)	407 (2)	3·4 (7)
H(14)	453 (3)	342 (3)	539 (2)	4·4 (7)
H(15)	468 (3)	462 (3)	604 (2)	3·9 (7)
H(16)	253 (3)	822 (3)	567 (2)	4·4 (7)
H(17)	132 (3)	864 (3)	482 (2)	4·9 (8)
H(18)	95 (3)	753 (3)	365 (2)	3·7 (7)
H(19)	591 (4)	382 (4)	83 (2)	7·4 (9)
H(20)	460 (3)	377 (3)	93 (2)	4·9 (8)

All calculations were performed on the CDC 6600 computer of the Centro di Calcolo Elettronico Interuniversitario dell'Italia Nord-Orientale.

Results and discussion

A drawing of the structure, including the Cr–N distances, is shown in Fig. 1, while a complete list of bond lengths and angles is given in Table 4.

The Cr atom is surrounded by three biguanide molecules which act as bidentate ligands giving rise to an octahedral arrangement. The Cr–N bond lengths (2·017, 2·029, 2·010, 2·022, 2·007, 2·020 Å) are all equivalent within the experimental errors and fall within the range (1·98–2·16 Å) of lengths found in octahedral Cr^{III} complexes. The N–Cr–N bond angles which involve N atoms belonging to the same biguanide ligand are all nearly equal and significantly smaller (83·04, 84·22, 84·11°) than the others (88·54–95·68°). This is a consequence of the rigidity of the ligand which always shows the same 'bite' distance [N(1)···N(5) = 2·682, N(6)···N(10) = 2·704, N(11)···N(15) = 2·697 Å] and coordinates to metal through equal N–Cr interactions.

The ligand geometry closely resembles that observed in the structurally known biguanide derivatives, in which the organic molecule behaves as a bidentate ligand. The only important and significant difference is that the ligand is now deprotonated and the deprotonation involves the imine group. This situation appears to be an exception to the results of the theoretical calculations of Wasson & Zacharopoulos (private communication) who found, on the basis of the stabilization energies, that the deprotonated ligand site should be one of the two bonding sites employed in coordination.

Table 4. *Bond lengths* (Å) *and angles* (°)

Cr—N(1)	2·017 (2)	Cr—N(6)	2·010 (2)	Cr—N(11)	2·007 (2)
Cr—N(5)	2·029 (2)	Cr—N(10)	2·002 (2)	Cr—N(15)	2·020 (2)
N(1)–C(1)	1·319 (3)	N(6)–C(3)	1·306 (4)	N(11)–C(5)	1·313 (3)
C(1)–N(2)	1·355 (4)	C(3)–N(7)	1·354 (4)	C(5)—N(12)	1·381 (3)
C(1)–N(3)	1·367 (4)	C(3)–N(8)	1·362 (4)	C(5)—N(13)	1·343 (3)
N(3)–C(2)	1·348 (4)	N(8)–C(4)	1·350 (3)	N(13)–C(6)	1·349 (3)
C(2)–N(4)	1·363 (4)	C(4)–N(9)	1·385 (4)	C(6)—N(14)	1·379 (4)
C(2)–N(5)	1·323 (3)	C(4)–N(10)	1·305 (4)	C(6)—N(15)	1·314 (4)
N(1)–Cr—N(5)	83·04 (8)	N(5)–Cr—N(6)	92·92 (7)	N(6)—Cr—N(11)	88·67 (8)
N(1)–Cr—N(6)	90·89 (9)	N(5)–Cr—N(10)	88·54 (8)	N(6)—Cr—N(15)	171·00 (9)
N(1)–Cr—N(10)	170·05 (6)	N(5)–Cr—N(11)	175·63 (9)	N(10)–Cr—N(11)	95·68 (8)
N(1)–Cr—N(11)	92·86 (8)	N(5)–Cr—N(15)	94·68 (9)	N(10)–Cr—N(15)	91·15 (9)
N(1)–Cr—N(15)	94·81 (8)	N(6)–Cr—N(10)	84·22 (9)	N(11)–Cr—N(15)	84·11 (8)
Cr—N(1)–C(1)	128·3 (2)	Cr—N(6)—C(3)	130·0 (2)	Cr—N(11)–C(5)	130·2 (2)
N(1)–C(1)–N(2)	120·2 (2)	N(6)–C(3)—N(7)	119·2 (3)	N(11)–C(5)—N(12)	119·5 (2)
N(1)–C(1)–N(3)	125·7 (2)	N(6)–C(3)—N(8)	127·0 (2)	N(11)–C(5)—N(13)	126·8 (2)
N(2)–C(1)–N(3)	114·2 (2)	N(7)–C(3)—N(8)	113·9 (3)	N(12)–C(5)—N(13)	113·6 (2)
C(1)–N(3)–C(2)	120·8 (2)	C(3)–N(8)—C(4)	120·7 (3)	C(5)—N(13)–C(6)	120·7 (2)
N(3)–C(2)–N(4)	113·7 (3)	N(8)–C(4)—N(9)	112·9 (3)	N(13)–C(6)—N(14)	112·6 (3)
N(3)–C(2)–N(5)	126·8 (3)	N(8)–C(4)—N(10)	127·1 (3)	N(13)–C(6)—N(15)	127·5 (2)
N(4)–C(2)–N(5)	119·5 (2)	N(9)–C(4)—N(10)	119·9 (2)	N(14)–C(6)—N(15)	119·9 (2)
C(2)–N(5)–Cr	125·1 (2)	C(4)–N(10)–Cr	128·8 (2)	C(6)—N(15)–Cr	129·5 (2)

---

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31319 (17 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

The N–C lengths are worthy of some comment. As pointed out by Holian & Marsh (1970), there is a small but significant difference between these bonds according to whether the N atom is involved in coordination, is in the terminal position, or is in the ring. Table 5 lists the N–C lengths and the C–N–C bond angles, together with the final R values, for the above-mentioned biguanide compounds. Unfortunately, the molecular geometry for the $Cr(BG)^{3+}$ cation was not reported by Brubaker & Webb (1969). (Since no coordinates are given, it was also impossible carry out calculations.) Limiting our comparison to only the best refined ($R \leq 8{\cdot}5\%$) structures, it can be seen that the sequence: C–N(ligand) < C–N(terminal) < C–N (ring), which is always observed in the protonated ligands, is inverted for the two last bonds in the present compound. This inversion can be related to the deprotonation of the ring N atom, which produces an increase of π conjugation along the central C–N–C system in the ligand, reducing the bond angle on N to the theoretical value of 120° (in the protonated ligands this angle ranges from 125·7 to 128·8°).

All the biguanide ligands are nearly planar as a consequence of the π conjugation along them (Table 6). The displacements from planarity are a little larger for the three six-membered chelation rings; the dihedral angles formed by the ligand planes and the corresponding NCrN planes are 66·7, 85·1, and 86·1°.

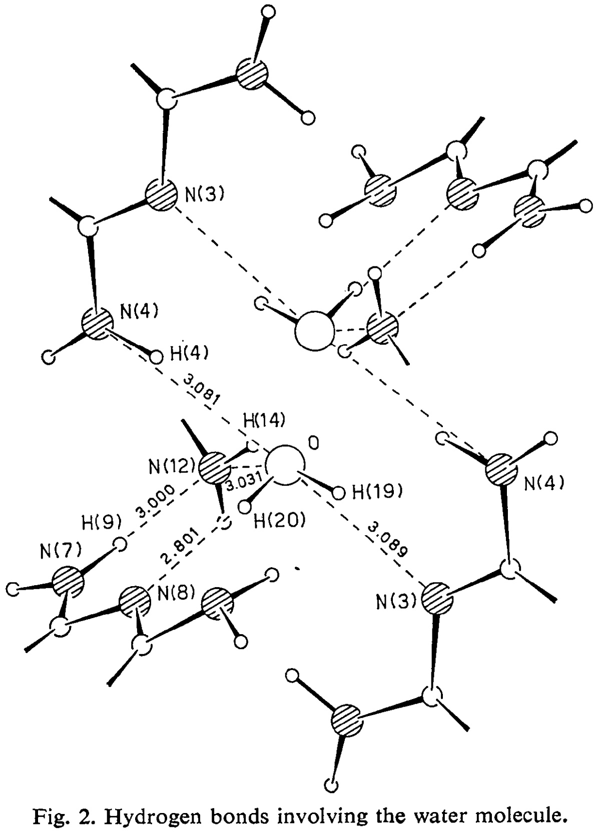

Fig. 2. Hydrogen bonds involving the water molecule.

Table 5. *A comparison between the (averaged) C–N bond lengths (Å) and the C–N–C bond angles (°) in the present compound and in other biguanide compounds, together with the conventional R values*

	$Co(BG)_3.H_2O$	$Ni(BG)_2Cl_2$	$Co(BG)_3Cl_3.H_2O$	$Ni(EBG)Cl_2.H_2O$			$Cu(EBG)Cl_2.H_2O$	$Ag(EBG)(NO_3)_3$	$Ag(EBG)(ClO_4)_3$	$[Ag(EBG)]SO_4HSO_4.H_2O$	$[Cu(AEBG)(C_2H_4N_4)]SO_4.H_2O$	$[Cu(AEBG)(NCS)]SCN$
	(*a*)	(*b*)	(*c*)	(*d*)	(*e*)	(*f*)	(*g*)	(*h*)	(*i*)	(*l*)	(*m*)	(*n*)
C–N (ligand	1·313	1·32	1·282	1·303	1·290	1·289	1·323	1·31	1·32	1·31	1·32	1·290
C–N (ring)	1·353	1·32	1·365	1·359	1·374	1·371	1·410	1·40	1·36	1·37	1·37	1·407
C–N (terminal)	1·370	1·35	1·353	1·343	1·348	1·349	1·372	1·30	1·35	1·33	1·35	1·345
C–N–C	120·7	127	127·2	126·5	126·8	126·9	126·5	125·7	–	128·8	127·9	125·7
R	3·6	12·3	2·1	9·2	4·8	3·1	8·4	10·5	8·1	4·0	11·2	8·5

(*a*) Present work. (*b*) Creitz, Gsell & Wampler (1969). (*c*) Snow (1974). (*d*) Coghi, Mangia, Nardelli & Pelizzi (1969). (*e*) Holian & Marsh (1970). (*f*) Ward, Caughlan & Smith (1971). (*g*) Mathew & Kunchur (1970). (*h*) Kunchur (1968). (*i*) Simms, Atwood & Zatko (1973). (*l*) Coghi & Pelizzi (1975). (*m*) Coghi, Mangia, Nardelli, Pelizzi & Sozzi (1968). (*n*) Andreetti, Coghi, Nardelli & Sgarabotto (1971).

Table 6. *Least-squares planes and deviations* (Å) *of atoms from the planes*

Equation of planes: $AX+BY+CZ=D$, where X, Y, Z are the coordinates in Å referred to orthogonal axes.

Plane	A	B	C	D
I	0·3767	−0·8185	−0·4337	6·7994
II	0·5130	−0·8137	−0·2734	6·5480
III	−0·4494	0·4018	−0·7979	1·4326
IV	−0·5390	0·3911	−0·7460	1·4605
V	0·9333	0·2998	−0·1975	−0·6134
VI	0·9145	0·3418	−0·2164	−0·7454

	I	II		III	IV		V	VI
N(1)	−0·200	0·086	N(6)	−0·064	0·084	N(11)	−0·025	−0·087
C(1)	0·005	0·015	C(3)	−0·032	−0·014	C(5)	0·075	0·007
N(3)	0·265	0·082	N(8)	0·095	−0·003	N(13)	−0·022	−0·041
C(2)	0·108	−0·002	C(4)	0·094	0·007	C(6)	−0·050	−0·004
N(5)	−0·249	−0·103	N(10)	−0·102	−0·075	N(15)	0·023	0·092
Cr	0·072	0·582*	Cr	0·009	0·236*	Cr	−0·001	0·019*
N(2)	−0·004*	−0·106	N(7)	−0·132*	−0·142	N(12)	0·255*	0·125
N(4)	0·357*	0·028	N(9)	0·355*	0·142	N(14)	−0·178*	−0·090

* atoms not included in the least-squares calculation.

Table 7. *Hydrogen bonds and short contacts* (Å)

Symmetry code

(i) $\bar{x}$, $\frac{1}{2}+y$, $\frac{1}{2}-z$
(ii) x, $\frac{1}{2}-y$, $\frac{1}{2}+z$
(iii) $x-1$, y, z
(iv) $\bar{x}$, $1-y$, $\bar{z}$
(v) x, $\frac{3}{2}-y$, $\frac{1}{2}+z$
(vi) x, $1-y$, $1-z$
(vii) $1-x$, $1-y$, $1-z$

N(8)····O	2·801	N(2)····N(8^{iii})	3·252
N(1)····N(7^{i})	3·292	N(3)····N(9^{iii})	3·211
N(5)····N(6^{i})	3·292	N(4)····O^{iv}	3·081
N(10)···N(2^{i})	3·265	N(14)···N(9^{v})	3·233
N(12)···O^{ii}	3·031	N(13)···N(2^{vi})	3·002
N(12)···N(7^{ii})	3·000	N(12)···N(10^{vii})	3·229
N(3)····O^{iii}	3·089		

σ's are in the range 0·005–0·006 Å.

The water molecule forms four hydrogen bonds, two donor and two acceptor, with N atoms of four adjacent ligands (Fig. 2). These hydrogen bonds, with others of the N–H···N type (Table 7), are responsible for the packing of the structure.

References

Andreetti, G. D. (1973). VI Meeting Associazione Italiana di Cristallografia, Trieste.

Andreetti, G. D., Coghi, L., Nardelli, M. & Sgarabotto, P. (1971). *J. Cryst. Mol. Struct.* **1**, 147–154.

Brubaker, G. R. & Webb, L. E. (1969). *J. Amer. Chem. Soc.* **91**, 7199–7200.

Coghi, L., Mangia, A., Nardelli, M. & Pelizzi, G. (1969). *Ric. Sci.* **39**, 438–440.

Coghi, L., Mangia, A., Nardelli, M., Pelizzi, G. & Sozzi, L. (1968). *Chem. Commun.* pp. 1475–1477.

Coghi, L. & Pelizzi, G. (1975). *Acta Cryst.* **B31**, 131–134.

Creitz, T. C., Gsell, R. & Wampler, D. L. (1969). *Chem. Commun.* pp. 1371–1372.

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* **A24**, 321–324.

Holian, B. L. & Marsh, R. E. (1970). *Acta Cryst.* **B26**, 1049–1058.

Kunchur, N. R. (1968). *Nature, Lond.* **217**, 539–541.

Mathew, M. & Kunchur, N. R. (1970). *Acta Cryst.* **B26** 2054–2062.

Simms, M. L., Atwood, J. L. & Zatko, D. A. (1973). *Chem. Commun.* pp. 46–47.

Snow, M. R. (1974). *Acta Cryst.* **B30**, 1850–1856.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Ward, D. L., Caughlan, C. N. & Smith, G. D. (1971). *Acta Cryst.* **B27**, 1541–1549.