Orotate Complexes

Synthesis and Crystal Structure of Lithium Orotate(−I) Monohydrate and Magnesium Bis[orotate(−I)] Octahydrate

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Neutralization of orotic acid (OrH_2) by lithium or magnesium hydroxide in aqueous medium yields the crystalline metal orotate hydrates $Li(OrH) \cdot H_2O$ (**1**) and $Mg(OrH)_2 \cdot (H_2O)_8$ (**2**). By single-crystal X-ray diffraction studies it has been shown that **1** (space group $P1$) forms a layer structure, with lithium in a tetrahedral environment of oxygen atoms from three different orotate(−I) anions (one carboxylate and two uracil oxygen atoms) and the water molecule. **2** (space group $P2_1/c$) has been shown to feature a hexaquo complex of magnesium, the $Mg(H_2O)_6^{2\oplus}$ cations being associated with two hydrated $OrH^{\ominus}$ ions only through hydrogen bonds. The anions are also engaged in "base-pairing" hydrogen bonds between the planar uracil ring systems of different units. The results are relevant for applications of magnesium orotates in magnesium therapy.

Orotic acid (6-uracilic acid, 1,2,3,6-tetrahydro-2,6-dioxo-4-pyrimidinecarboxylic acid, OrH_2) is of great significance in biological systems as a precursor of pyrimidine nucleosides[1,2]. Its importance is also obvious from its role as "vitamin B_{13}" (for pyrimidine synthesis) in the metabolism[3]. The biosynthesis of orotic acid is based on the condensation of aspartic acid and carbamoyl phosphate followed by dehydration and dehydrogenation steps[1−4].

In aqueous solution, OrH_2 acts as a dibasic acid, with the acid function localized at the exocyclic carboxylate group ($pK = 2.09$) and the 1-imino position ($pK = 9.28$)[5,6]. In the intermediate pH range (5−9) neutralization of orotic acid therefore yields orotate(−I) salts containing the ion $OrH^{\ominus}$. It is only in strongly alkaline solutions or with powerful coordination centers that orotates(−II) containing the $Or^{2\ominus}$ ion become relevant. Further deprotonation appears to be irrelevant for aqueous systems.

The ligand properties of orotic acid and its anions are largely unexplored[7,8]. Observation of a surprisingly low solubility of *sodium* and *potassium* orotates(−I) has led to some exploratory applications in analytical chemistry[9,10]. The corresponding *ammonium* salt shows much greater solubility, probably owing to efficient ion solvation. The crystal structure of $NH_4^{\oplus}OrH^{\ominus} \cdot H_2O$ has been determined, and it indeed reveals an extended system of hydrogen bonding[11]. From basic solutions a *magnesium* orotate(−II) pentahydrate has been isolated, whose formula should be given as $[Mg(Or)(H_2O)_4] \cdot H_2O$ according to the crystal structure[12]. The structures of orotate complexes containing *copper*[13,14], *cobalt*[15], and *uranium*[16] have also been determined. As part of research programs oriented towards stoichiometric and structural aspects of complexes relevant for chemotherapy of cancer, a large number of coordination compounds of orotic acid have been screened mainly involving the group VIII (18) metal ions *platinum, palladium,* and *nickel*. Crystal structures have been determined of $[Ni(Or)(H_2O)_4] \cdot H_2O$[17], *cis*-$[Pt(Or)(NH_3)_2]$[18], and [Pt(3-MeOr)DACH][19], where $3\text{-}MeOr^{2\ominus}$ and DACH represent 3-methylorotate(−II) and 1,2-diaminocyclohexane ligands, respectively. The stability constants of some orotate complexes have been derived from potentiometric measurements, but information is often still of preliminary nature[20].

In recent years, metal orotates have attracted growing attention as they find application as components of drugs for a large number of medicinal indications; e.g. carrier function of orotic acid and its anions for magnesium ions is suggested to be responsible for the successful use of magnesium orotate in *magnesium therapy*. As judged from analytical data on this agent, it should be formulated as the tetrahydrate $[Mg(OrH)_2 \cdot (H_2O)_4]$, but the structure of the "complex" has not yet been reported. Other areas of application for metal orotates are quite generally mixed preparations for electrolyte substitution (in heart and liver protection) as well as uricosurica (for enhanced excretion of uric acid)[21].

In connection with current work on the structural aspects of other magnesium pharmaceuticals[22], mainly magnesium hydrogen L-aspartates and -glutamates[23−26], we became also interested in the general nature of the function of orotic acid as a ligand to metals. With magnesium orotates as the initial targets, a number of crystalline phases have been isolated from the aqueous system and characterized by analytical, spectroscopic, and diffraction methods. In this paper we report on the preparation and crystal structure of the most water-rich phase, which seems to be of prime interest owing to its closest relation with the structure of the species present in aqueous solution. In an extension of this study to the pharmacologically equally relevant lithium orotate system, the lithium orotate(−I) monohydrate phase has also been prepared and its crystal structure determined.

Preparation and Properties of the Complexes

The two title compounds are readily prepared by neutralization of lithium or magnesium hydroxide with one or two equivalents of orotic acid, respectively, in water at room temperature. Clear solutions are obtained which, upon concentration in a vacuum and cooling, give high yields of colorless crystalline products.

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$$LiOH + OrH_2 \xrightarrow{[H_2O]} Li(OrH) \cdot H_2O \quad \mathbf{1} \qquad (1)$$

$$Mg(OH)_2 + 2\,OrH_2 \longrightarrow Mg(OrH)_2 \cdot (H_2O)_8 \quad \mathbf{2} \qquad (2)$$

The lithium salt crystallizes as the monohydrate **1**, the magnesium salt as the octahydrate **2** under these conditions. **1** has a solubility of $8.0 \cdot 10^{-3}$ mol $\cdot$ l^{-1} and a pH of 7.53, while aqueous solutions of **2** feature a pH of 6.18 at a maximum concentration of $5.2 \cdot 10^{-3}$ mol $\cdot$ l^{-1}.

Description of the Crystal Structures

The single-crystal structure determinations by X-ray diffraction at ambient temperature give very different results for the two compounds. While the lithium compound **1** has been shown to form a two-dimensional layer structure (of the rare space group $P1$), in which the orotate(−I) ions $OrH^{\ominus}$ are directly associated with the lithium cations (Figure 1), the magnesium compound **2** has turned out to be a conventional hexaquomagnesium complex with two monohydrated $OrH^{\ominus}$ species as counter ions (Figure 2). The water-rich phase $Mg(OrH)_2 \cdot (H_2O)_8$ (**2**) therefore is not a true magnesium "orotate complex", but should be regarded as $[Mg(H_2O)_6]^{2\oplus}(OrH \cdot H_2O)_2^{\ominus}$.

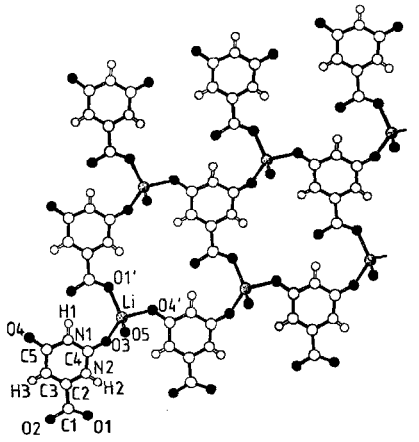

Figure 1. Layer structure of lithium orotate(−I) hydrate (**1**) with atomic numbering. [The hydrogen atoms of the water molecule (O5) have been omitted. Oxygen atoms are represented by black circles, lithium cations by shaded circles, carbon and nitrogen atoms by larger and smaller white circles, respectively.]

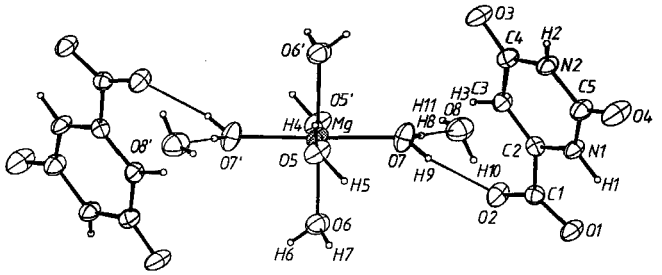

Figure 2. Representative unit in the crystal structure of hexaquomagnesium bis[orotate(−I) hydrate] (**2**) with atomic numbering. The magnesium cation occupies a crystallographical center of inversion. Only one pair of hydrogen bonds (O2−H9−O7) is shown. The orotate(−I) ions form pairs through double hydrogen bonds (via H1) originating from O1 and N1

Structural Details of the Lithium Orotate(−I) Monohydrate 1

$LiOrH \cdot H_2O$ crystallizes in the triclinic space group $P1$ with one molecule in the unit cell. The lithium cation is located in the center of a tetrahedron of oxygen atoms, one of which belongs to the water molecule. Among the remaining three, one is a carboxylate oxygen atom, while the other two are identified – surprisingly – as carbonyl oxygen atoms of the uracil ring, with all three belonging to different $OrH^{\ominus}$ groups. To the best of our knowledge this is the first case where direct interaction of a metal ion with these uracil oxo functions of orotic acid has been demonstrated by structure determination. In all other cases the carboxyl group and the nitrogen centers are the preferred monodentate or chelating functions. The Li−O distances are longest for H_2O and shortest for carboxylate, bracketing the carbonyl oxygen's contacts (Table 1). The uracil rings of the $OrH^{\ominus}$ ions are planar, and the carboxylate plane forms an angle of only 3.3° with this heterocyclic plane. The uracil rings are present in the double keto tautomeric form (with both nitrogen atoms protonated). Bond distances and angles are very similar to those found in previous studies of orotate(−I) salts. Each $OrH^{\ominus}$ ion is associated with three different lithium cations (through one carboxylate and the two carbonyl oxygen atoms). In this way a layer structure is adopted, representing a two-dimensional coordination polymer with large twenty-membered rings as a basic pattern (Figure 1). The water molecule is engaged in a network of hydrogen bonding which crosslinks the layers (Table 2).

Table 1. Selected interatomic distances [Å] and angles [°] in lithium orotate(−I) hydrate (**1**) (see Figure 1)

O1	-- C1	1.244(2)		O1	-- LI	1.882(3)	
O2	-- C1	1.248(2)		O3	-- C4	1.219(2)	
O3	-- LI	1.903(3)		O4	-- C5	1.242(2)	
O4	-- LI	1.943(3)		N1	-- C4	1.375(2)	
N1	-- C5	1.380(2)		N2	-- C2	1.369(2)	
N2	-- C4	1.357(2)		C1	-- C2	1.525(2)	
C2	-- C3	1.348(2)		C3	-- C5	1.433(2)	
O5	-- LI	1.991(3)					
C1	-O1 -LI	133.7(1)		C4	-O3 -LI	143.7(1)	
C5	-O4 -LI	132.6(1)		C4	-N1 -C5	125.2(1)	
C2	-N2 -C4	123.1(1)		O1	-C1 -O2	127.4(2)	
O1	-C1 -C2	115.4(1)		O2	-C1 -C2	117.2(1)	
N2	-C2 -C1	114.4(1)		N2	-C2 -C3	120.8(1)	
C1	-C2 -C3	124.8(1)		C2	-C3 -C5	119.6(1)	
O3	-C4 -N1	121.9(1)		O3	-C4 -N2	122.5(1)	
N1	-C4 -N2	115.5(1)		O4	-C5 -N1	119.8(1)	
O4	-C5 -C3	124.5(1)		N1	-C5 -C3	115.7(1)	
O1	-LI -O3	115.7(2)		O1	-LI -O4	100.6(1)	
O3	-LI -O4	119.0(2)		O1	-LI -O5	117.4(2)	
O3	-LI -O5	99.2(1)		O4	-LI -O5	105.6(1)	

Table 2. Prominent hydrogen bond crosslinks in the layer structure of lithium orotate(−I) hydrate (**1**)

X - H ··· Y	X-H	H···Y	X···Y	X-H···Y
N1-H1 ··· O2(a)	0.979	1.919	2.896	175.6
N2-H2 ··· O5(b)	0.871	2.128	2.923	151.8
O5-H4 ··· O4(c)	0.867	2.066	2.900	161.1

Symmetry positions of atom Y: (a): $x - 1, y + 1, z$; (b): $x - 1, y, z$; (c): $x + 1, y - 1, z - 1$.

The structure of **1** is thus very similar to that of lithium hydrogen L-aspartate hydrate, where the lithium cations are also tetracoordinated exclusively by oxygen atoms in an infinite three-dimensional coordination polymer[27].

Structural Details of Hexaquomagnesium Bis[orotate(−I) hydrate] (2)

$Mg(H_2O)_6^{2\oplus}$ $(OrH \cdot H_2O)_2^{\ominus}$ crystallizes in the monoclinic space group $P2_1/c$ with two formula units in the unit cell. The hexaquo-coordinated magnesium cation occupies a crystallographic center of inversion, which relates the two orotate(−I) counter ions and the remaining two anion-bound water molecules. The Mg−O bonds in the almost perfectly octahedral dication are similar to those found in other hexaquo complexes of magnesium.

The uracil rings of the anions in **2** are planar, with the carboxylate plane inclined by only 6.1° and with geometrical parameters very similar to the anions in **1** (Table 3).

Table 3. Selected interatomic distances [Å] and angles [°] in hexaquomagnesium bis[orotate(−I) hydrate] (**2**) (see Figure 2)

MG	-- O5		2.025(2)	MG	-- O6		2.094(2)
MG	-- O7		2.083(2)	O1	-- C1		1.250(3)
O2	-- C1		1.240(3)	O3	-- C4		1.233(3)
O4	-- C5		1.221(3)	N1	-- C2		1.376(3)
N1	-- C5		1.362(4)	N2	-- C4		1.376(4)
N2	-- C5		1.386(4)	C1	-- C2		1.515(4)
C2	-- C3		1.346(4)	C3	-- C4		1.437(4)
O5	-MG	-O6	90.4(1)	O5	-MG	-O7	93.5(1)
O6	-MG	-O7	90.6(1)	O5	-MG	-O5	180.0(1)
O6	-MG	-O6	180.0(1)	C2	-N1	-C5	123.2(2)
C4	-N2	-C5	126.4(2)	O1	-C1	-O2	127.0(3)
O1	-C1	-C2	115.8(2)	O2	-C1	-C2	117.3(2)
N1	-C2	-C1	115.5(2)	N1	-C2	-C3	121.0(3)
C1	-C2	-C3	123.5(3)	C2	-C3	-C4	119.7(3)
O3	-C4	-N2	119.5(3)	O3	-C4	-C3	125.3(3)
N2	-C4	-C3	115.2(2)	O4	-C5	-N1	122.8(3)
O4	-C5	-N2	122.8(3)	N1	-C5	-N2	114.4(3)

Table 4. Prominent hydrogen bonds in the structure of hexaquomagnesium bis[orotate(−I) hydrate] (**2**)

X - H ··· Y	X-H	H···Y	X···Y	X-H···Y
N1-H1 ··· O3(g)	0.916	2.135	3.009	159.5
N2-H2 ··· O1(c)	0.895	2.059	2.892	154.7
O5-H4 ··· O4(d)	0.933	1.805	2.714	164.0
O5-H5 ··· O1(e)	1.021	1.661	2.661	165.4
O6-H6 ··· O8(f)	0.813	2.009	2.820	175.4
O6-H7 ··· O2(b)	0.958	1.849	2.741	153.9
O7-H8 ··· O8(a)	1.022	1.820	2.835	172.1
O7-H9 ··· O2(a)	0.870	1.941	2.773	159.6
O8-H10··· O6(f)	0.816	2.338	2.820	143.5
O8-H11··· O3(f)	0.863	2.029	2.886	172.1

Symmetry positions of atom Y: (a): x, y, z; (b): $-x, y + 0.5, -z + 0.5$; (c): $-x + 1, y + 0.5, -z + 1.5$; (d): $-x + 1, -y, -z + 1$; (e): $x, -y - 0.5, z - 0.5$; (f): $-x, -y, -z + 1$; (g): $-x + 1, y - 0.5, -z + 1.5$.

In the crystals of compound **2** there is an extensive network of hydrogen bonds which connect not only cations and anions, but also adjacent anions through the carboxylate groups, the amide functions of the uracil rings, and the water molecules. The most prominent contacts are summarized in Table 4. The direct uracil/uracil interactions are clearly reminiscent of the ubiquitous "base-pair" contacts between pyrimidine functions in many biological systems.

There is no experimental evidence that species other than OrH_2 or $OrH^{\ominus}$ prevail in acidic or neutral aqueous solutions and in crystalline hydrates of orotic acid and alkali/alkaline earth orotates(−I) obtained from such solutions. Due to the solid-state structure of **2** it is also extremely unlikely that aqueous solutions of magnesium bis[orotate(−I)] hydrates contain "magnesium orotate complexes", which would require the $OrH^{\ominus}$ ions to enter the inner coordination sphere of the metal by removing water molecules from their coordination sites. We could show, in fact, that if commercially available $Mg(OrH)_2(H_2O)_4$ is dissolved in water and recrystallized, only the octahydrate **2** is obtained at ambient temperature.

For comparison, it is interesting to mention that from aqueous solutions of magnesium *citrate* a crystalline hydrate $[Mg_3(Ci)_2(H_2O)_8](H_2O)_2$ [where $Ci^{3\ominus}$ represents citrate(−III)] can be crystallized which contains both $[Mg(H_2O)_6]^{2\oplus}$ ions and hydrated magnesium citrate complexes $[Mg(Ci)(H_2O)]^{\ominus}$ in the ratio 1:2[28]. On the other hand, crystalline magnesium hydrogen L-*aspartate* chloride trihydrate $[Mg(L\text{-}AspH)Cl(H_2O)_2] \cdot H_2O$ has been shown to be a genuine magnesium complex with the *O,O*-chelating L-$AspH^{\ominus}$ ion functioning as a ligand to *all* magnesium cations. With magnesium orotate(−I), hydrogen L-aspartate chloride, and citrate representing the most common preparations used in magnesium therapy, it therefore appears that both simple aquo complexes as well as true complexes with an organic carrier are successfully employed for similar medication. In none of these cases the therapeutic role of the various components has been elucidated, however, and very specific effects may be operative in each system.

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Experimental

All reactions were carried out in pure, desalinated water. Reagents were of p.a. quality. A Knick apparatus (AgCl/KCl) was used for pH readings. Elemental analyses were performed in the microanalytical laboratory of this Institute by standard combustion methods.

Lithium Orotate(−I) Hydrate (**1**): A suspension of orotic acid (7.8 g, 50 mmol) in water (75 ml) was treated with lithium hydroxide monohydrate (2.1 g, 50 mmol) with stirring at room temp. As soon as an almost clear solution was obtained, the reaction mixture was filtered and concentrated in vacuo to a volume of ca. 20 ml. A microcrystalline product precipitated during ca. 12 h, which was recrystallized from hot concentrated aqueous solution. The transparent colorless crystals dissolved in water to give a solution with pH = 7.53.

$C_5H_5LiN_2O_4$ (179.52) Calcd. C 33.25 H 2.81 N 15.69
Found C 33.45 H 2.81 N 15.60

Hexaquomagnesium Bis[orotate(−I) hydrate] (**2**): A suspension of orotic acid (312.1 mg, 2.00 mmol) in water (75 ml) was treated

with magnesium hydroxide (58.40 mg, 1.00 mmol) with stirring at room temp. The reaction mixture was filtered after 5 h, the filtrate concentrated in vacuo to a volume of ca. 25 ml, and cooled to 2°C. Transparent, colorless needles were obtained after 3 d. When dissolved in water, the pH = 6.18.

$C_{10}H_{22}MgN_4O_{16}$ (478.64) Calcd. C 25.09 H 4.63 N 11.71
Found C 24.04 H 4.73 N 11.68

Crystal Structure Determination: A summary of the data collection and structure refinement parameters is given in Table 5. Further details of the individual determinations are described below. The final coordinates are presented in Tables 6 and 7.

Table 5. Crystal data collection and structure refinement parameters for compounds **1** and **2**

	$Mg(OrH)_2 \cdot 8\ H_2O$	$Li(OrH) \cdot H_2O$
Formula	$C_{10}H_{22}MgN_4O_{16}$	$C_5H_5LiN_2O_5$
M_r	478.609	180.045
Space group	$P2_1/c$ (No. 14)	$P1$ (No. 1)
a [Å]	10.984(1)	4.998(1)
b [Å]	12.901(1)	5.345(1)
c [Å]	6.944(1)	6.784(1)
α [°]	90.0	90.18(1)
β [°]	98.51(1)	103.15(1)
γ [°]	90.0	98.75(1)
V [Å^3]	973.16	174.29
Z	2	1
$\sigma_{calcd.}$ [g/cm^3]	1.633	1.715
μ (Mo-K_α) [cm^{-1}]	1.7	1.0
T [°C]	23	23
$(\sin\Theta/\lambda)_{max}$ [Å^{-1}]	0.571	0.638
hkl range	±12, 14, 7	±6, ±6, ±8
Number of reflections		
measured	1674	1520
unique	1509	1514
observed	1322	1459
Refined parameters	186	115
R[a]	0.043	0.026
R_w[b]	0.041	0.028
$\Delta\sigma_{fin.}$ [e Å^{-3}]	+0.24/−0.24	+0.49/−0.37

[a] $R = \Sigma(F_o - F_c)/\Sigma F_o$. — [b] $R_w = [\Sigma(F_o - F_c)^2/\Sigma w F_o^2]^{1/2}$, $w = 1/\sigma^2(F_o)$.

Table 6. Fractional atomic coordinates and thermal displacement parameters of compound **1**

ATOM	X/A	Y/B	Z/C	U(eq.)
O1	0.49960	0.81290	0.79300	0.051
O2	0.7877(4)	0.8650(3)	1.0998(2)	0.050
O3	-0.1371(4)	1.3822(4)	0.7422(3)	0.051
O4	0.3444(3)	1.5458(3)	1.3937(2)	0.050
N1	0.1108(4)	1.4676(4)	1.0658(3)	0.036
N2	0.2179(4)	1.1672(3)	0.8631(3)	0.033
C1	0.5862(5)	0.9119(4)	0.9665(3)	0.033
C2	0.4265(4)	1.1144(4)	1.0190(3)	0.032
C3	0.4783(4)	1.2363(4)	1.2011(3)	0.036
C4	0.0526(4)	1.3415(4)	0.8804(3)	0.030
C5	0.3137(4)	1.4239(4)	1.2318(3)	0.031
O5	0.9545(4)	0.8390(3)	0.5045(3)	0.050
LI	0.6616(7)	0.6387(6)	0.6206(5)	0.049

Li(OrH) · H$_2$O (**1**): Enraf-Nonius CAD4 diffractometer; Mo-K_α radiation, λ = 0.71069 Å; graphite monochromator; Θ−Θscan; Lp correction was applied. From 1514 independent structure factors 1459 with $F_o \geq 4\ \sigma(F_o)$ were deemed "observed" and used for all calculations. The acentric space group $P1$ was assumed and confirmed by the successful refinement of the structure. Reduced-cell calculations did not indicate higher symmetry. The cell dimensions were confirmed by precession photographs. The structure was solved by direct methods (SHELXS-86) and completed by difference Fourier syntheses. Three hydrogen atoms were found, one hydrogen atom was calculated at idealized geometrical position. One hydrogen atom at the water molecule could not be located by difference Fourier syntheses nor was its idealized geometrical position unambiguously clear. This hydrogen atom was therefore neglected in final refinement cycles. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included in the refinement with fixed isotropic displacement parameters $U_{iso} = 0.05$ Å^2. Refinement of the inverse coordinate set yielded no differences in R values and geometrical parameters.

Table 7. Fractional atomic coordinates and thermal displacement parameters of compound **2**

ATOM	X/A	Y/B	Z/C	U(eq.)
MG	0.00000	0.00000	0.00000	0.019
O1	0.3350(2)	-0.3560(2)	0.5571(3)	0.021
O2	0.1926(2)	-0.2466(2)	0.4147(3)	0.021
O3	0.3899(2)	0.0954(2)	0.6689(3)	0.025
O4	0.6683(2)	-0.1569(2)	0.8717(4)	0.030
O5	0.1772(2)	0.0096(2)	-0.0470(3)	0.022
O6	0.0193(2)	0.1388(2)	0.1580(4)	0.028
O7	0.0443(2)	-0.0850(2)	0.2557(3)	0.037
O8	-0.1412(2)	-0.1647(2)	0.4583(4)	0.030
N1	0.4852(2)	-0.2040(2)	0.6985(4)	0.017
N2	0.5261(2)	-0.0314(2)	0.7685(4)	0.019
C1	0.2923(3)	-0.2677(2)	0.5155(4)	0.016
C2	0.3713(3)	-0.1778(2)	0.5998(4)	0.015
C3	0.3342(3)	-0.0783(2)	0.5849(4)	0.017
C4	0.4139(3)	0.0020(2)	0.6745(4)	0.019
C5	0.5676(3)	-0.1329(2)	0.7840(4)	0.019
H1	-0.515(3)	1.230(3)	-0.208(5)	0.05(1)
H2	0.586(3)	0.984(3)	0.200(5)	0.06(1)
H3	-0.252(2)	1.063(2)	0.495(4)	0.017(7)
H4	0.231(3)	1.064(3)	-0.010(5)	0.05(1)
H5	-0.232(3)	1.055(3)	0.027(5)	0.05(1)
H6	0.051(3)	1.145(3)	0.271(5)	0.05(1)
H7	-0.058(4)	1.171(3)	0.170(6)	0.08(1)
H8	-0.028(4)	0.887(4)	0.318(7)	0.10(2)
H9	0.101(3)	0.868(3)	0.282(5)	0.04(1)
H10	0.139(4)	1.273(3)	0.059(6)	0.06(1)
H11	-0.217(5)	0.854(4)	0.431(7)	0.10(2)

Mg(OrH)$_2$ · 8 H$_2$O (**2**): Enraf-Nonius CAD4 diffractometer; Mo-K_α radiation, λ = 0.71069 Å; graphite monochromator; ω scan. Lp correction was applied. After merging of equivalent data (R_{int} = 0.012) the remaining independent structure factors with $F_o \geq 1\ \sigma(F_o)$ were considered "observed" and used for all calculations. Reduced cell calculations did not indicate any higher symmetry (DELOS, LEPAGE). The structure was solved by direct methods (SHELXS-86) and completed by difference Fourier syntheses. The 11 hydrogen atoms in the asymmetric unit could be located in difference Fourier syntheses. The non-hydrogen atoms were refined anisotropically, the hydrogen atoms isotropically.

Further details of the crystal structure determinations are available on request from the Fachinformationszentrum Karlsruhe, Gesellschaft für wissenschaftlich-technische Information mbH, D-7514 Eggenstein-Leopldshafen 2, on quoting the depository number CSD-54 578, the names of the authors, and the journal citation.

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[1] L. Stryer, *Biochemie*, Vieweg, Braunschweig, Wiesbaden 1987.
[2] P. Karlson, *Kurzes Lehrbuch der Biochemie*, Thieme, New York 1988.
[3] A. L. Lehninger, *Prinzipien der Biochemie*, W. de Gruyter, Berlin 1987.
[4] J. D. Rawn, *Biochemistry*, Neil Patterson Publishers, Burlington, N. C., USA, 1989.

[5] J. J. Kaneti, E. Golovinski, *Chem.-Biol. Interact.* **3** (1971) 421.
[6] J. Maslowska, A. Dortabialski, *Pol. J. Chem.* **57** (1983) 1089.
[7] N. K. Davidenko, N. N. Zinich, *Koord. Khim.* **5** (1973) 3 [*Chem. Abstr.* **90** (1973) 110812g].
[8] N. K. Davidenko, N. N. Valasova, *Zh. Neorg. Khim.* **27** (1982) 385 [*Chem. Abstr.* **96** (1982) 209750w].
[9] B. C. Lewis, W. I. Stephen, *Anal. Chim. Acta* **36** (1966) 234.
[10] G. S. Pandey, P. C. Nigam, U. Agarwala, *J. Inorg. Nucl. Chem.* **39** (1977) 1877.
[11] J. Solbakk, *Acta Chem. Scand.* **25** (1971)3006.
[12] I.Mutikainen, *J. Inorg. Biochem.* **36** (1980) 183.
[13] I. Mutikainen, P. Lumme, *Acta Crystallogr., Sect. B,* **36** (1980) 2233.
[14] T. S. Khodashova, M. A. Portai-Koshits, N. K. Davidenko, N. N. Vlasova, *Koord. Khim.* **10** (1984) 262 [*Chem. Abstr.* **100** (1984) 148870n].
[15] I. Mutikainen, *Finn. Chem. Lett.* **1985**, 193.
[16] D. Mentzafos, N. Katsaros, A. Terzis, *Acta Crystallogr., Sect. C,* **43** (1987) 1905.
[17] M. Sabat, D. Zglinska, *Acta Crystallogr., Sect. B,* **36** (1980) 1187; A. Karapidis, B. Thomas, *Acta Crystallogr., Sect. C,* **42** (1986) 1705.
[18] T. Solin, K. Matsumoto, K. Fuwa, *Bull. Chem. Soc. Jpn.* **54** (1981) 3731.
[19] P. Castan, E. Colazio-Rodriguez, A. L. Beauchamp, S. Cros, S. Wimmer, *J. Inorg. Biochem.* **38** (1990) 225.
[20] J. Maslowska, A. Dorabialski, *Pol. J. Chem.* **57** (1983) 1089.
[21] D. Szeleny, J. Sos, *Arzneim.-Forsch.* **21** (1971) 777.
[22] H. Schmidbaur, H.-G. Classen, J. Helbig, *Angew. Chem.* **102** (1990), im Druck.
[23] H. Schmidbaur, G. Müller, J. Riede, G. Manninger, J. Helbig, *Angew. Chem.* **98** (1986) 1014; *Angew. Chem. Int. Ed. Engl.* **25** (1986) 1013.
[24] H. Schmidbaur, I. Bach, D. L. Wilkinson, G. Müller, *Chem. Ber.* **122** (1989) 1445.
[25] H. Schmidbaur, I. Bach, D. L. Wilkinson, G. Müller, *Chem. Ber.* **122** (1989) 1433.
[26] I. Bach, *Dissertation*, Techn. Univ. Munich 1989.
[27] H. Schmidbaur, I. Bach, D. L. Wilkinson, G. Müller, *Chem. Ber.* **122** (1989) 1227.
[28] C. K. Johnson, *Acta Crystallogr.* **18** (1965) 1004.

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