

The cationic unit and the contacts are illustrated in Fig. 1 and are very similar to those observed in the structures of $C_6H_4S_2P^+ \cdot AlCl_4^-$ (Burford *et al.*, 1988) and $Me_2Si(N^tBu)_2P^+ \cdot AlCl_4^-$ (Veith, Bertsch & Huch, 1988). The cation is planar [maximum deviation 0.088 Å at C(7)] with P—N bond lengths [mean 1.643 (6) Å] comparable to other phosphonium cations (Cowley *et al.*, 1978; Pohl, 1979) and metallophosphonium complexes (Hutchins, Campana & Paine, 1980; Hutchins, Duesler & Paine, 1984; Hutchins, Reisacher, Wood, Duesler & Paine, 1987), indicating a substantial degree of $p\pi-p\pi$ bonding between the P and N centres. The endocyclic C—N distances [mean 1.379 (9) Å] are significantly shorter than the exocyclic counterparts [mean 1.476 (9) Å]. The heterocyclic bond angles appear to be defined by the cyclic geometry (Hutchins *et al.*, 1980, 1984, 1987; Burford *et al.*, 1988). The molecular geometry of the benzene ring exhibits no unusual features, and the anion adopts a tetrahedral geometry comparable to that observed in other tetrachloroaluminate salts (Cardinal, Gillespie, Sawyer & Vekris, 1982).

The structural features are consistent with an efficient π -delocalized 10π -electron, aromatic cation, illustrating further isolobal diversity for framework (1), while maintaining naphthalenic uniformity.

We thank the Natural Sciences and Engineering Research Council of Canada (NB and PSW), the Donors of the Petroleum Research Fund of the American Chemical Society (NB) and the Killam Foundation (BWR) for financial support.

References

- BURFORD, N. & ROYAN, B. W. (1989). *J. Chem. Soc. Chem. Commun.* pp. 19–21.
- BURFORD, N., ROYAN, B. W., LINDEN, A. & CAMERON, T. S. (1988). *J. Chem. Soc. Chem. Commun.* pp. 842–844.
- BURFORD, N., ROYAN, B. W., LINDEN, A. & CAMERON, T. S. (1989). *Inorg. Chem.* **28**, 144–150.
- CARDINAL, G., GILLESPIE, R. J., SAWYER, J. F. & VEKRIS, J. E. (1982). *J. Chem. Soc., Dalton Trans.* pp. 765–779.
- COWLEY, A. H., CUSHNER, M. C. & SZOBOTA, J. C. (1978). *J. Am. Chem. Soc.* **100**, 7785–7787.
- FRIEDRICH, P., HUTTNER, G., LUBER, J. & SCHMIDPETER, A. (1978). *Chem. Ber.* **111**, 1558–1563.
- GABE, E. J., LEE, F. L. & LE PAGE, Y. (1985). In *Crystallographic Computing 3: Data Collection, Structure Determination, Proteins, and Databases*, edited by G. M. SHELDRICK, C. KRUGER & R. GODDARD, pp. 167–174. Oxford: Clarendon.
- HAAS, A. (1984). *Adv. Inorg. Chem.* **28**, 168–202.
- HOFFMANN, R. (1982). *Angew. Chem. Int. Ed. Engl.* **21**, 711–724.
- HUTCHINS, L. D., CAMPANA, C. F. & PAINE, R. T. (1980). *J. Am. Chem. Soc.* **102**, 4521–4523.
- HUTCHINS, L. D., DUESLER, E. N. & PAINE, R. T. (1984). *Organometallics*, **3**, 399–403.
- HUTCHINS, L. D., REISACHER, H.-U., WOOD, G. L., DUESLER, E. N. & PAINE, R. T. (1987). *J. Organomet. Chem.* **355**, 229–237.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- JENNINGS, W. B., RANDALL, D., WORLEY, S. D. & HARGLO, J. H. (1981). *J. Chem. Soc. Perkin Trans. I*, pp. 1411–1416.
- JOHNSON, C. K. (1976). *ORTEPII. A Fortran Thermal-Ellipsoid Plot Program for Crystal Structure Illustrations*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
- POHL, S. (1979). *Chem. Ber.* **112**, 3159–3165.
- VEITH, M., BERTSCH, B. & HUCH, V. (1988). *Z. Anorg. Allg. Chem.* **559**, 73–88.

Acta Cryst. (1989). **C45**, 1487–1490

Structure of Dimagnesium(II) *o*-Phenylenediaminetetraacetate, [Mg₂(C₁₄H₁₂N₂O₈)(H₂O)₆]

BY NORIYUKI NAKASUKA*

Faculty of Science, Nagoya University, Chikusa, Nagoya 464-01, Japan

AND MOTOO SHIRO

Shionogi Research Laboratories, Shionogi & Co. Ltd, Fukushima, Osaka 553, Japan

(Received 14 January 1989; accepted 27 February 1989)

Abstract. [Mg₂(C₁₄H₁₂N₂O₈)(H₂O)₆], $M_r = 493.0$, triclinic, $P\bar{1}$, $a = 9.774$ (2), $b = 11.466$ (1), $c = 9.760$ (2) Å, $\alpha = 97.25$ (1), $\beta = 105.10$ (1), $\gamma = 98.49$ (1)°, $V = 1029.0$ (3) Å³, $Z = 2$, $D_m = 1.592$, D_x

$= 1.591$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.54178$ Å, $\mu = 17.6$ cm⁻¹, $F(000) = 516$, $T = 298$ K, $R = 0.035$ for 2832 observed reflections. The title compound, 1,1,1,1,1,2-hexaqua- μ -(*o*-phenylenediamine-*N,N,N',N'*-tetraacetato-*O*¹:*N,N',O*²,*O*³,*O*⁴,*O*⁵)-dimagnesium(II), has a dinuclear structure, which consists of a

* To whom correspondence should be addressed.

chelated seven-coordinate Mg(1) moiety and a pentahydrated Mg(2) moiety in a nearly O_h environment. The two groups are bridged through acetate O(2), and every water molecule is directly coordinated to either magnesium ion. All the oxygen atoms except O(2) are hydrogen bonded, constituting a three-dimensional network.

Introduction. Hydrated magnesium(II) ion frequently serves as a large counter cation in metal complexes of aminopolycarboxylates like edta, e.g. in $Mg(H_2O)_6Cd(edta)H_2O$ where cadmium(II) ion alone is chelated and seven-coordinated (Pozhidaev, Polinova & Porai-Koshits, 1972; Solans, Gali, Font-Altaba, Oliva & Herrera, 1983). Hoard *et al.* have determined the structure of an interesting compound which consists of hexahydrated magnesium(II) cation and uncomplexed diprotonated H_2edta^{2-} anion (Julian, Day & Hoard, 1973). In some cases magnesium(II) ions act as a cationic center in a MgO_6^{2+} polyhedron and at the same time as an anionic center of $MgY(H_2O)^{2-}$ where Y denotes edta (Pozhidaev, Polinova, Porai-Koshits & Logvinenko, 1973; Passer, White & Cheng, 1977). Similar situations have been found for *o*-phenylenediaminetetraacetate ($phdta^{4-}$) complexes of cobalt(II) (McCandlish, Michael, Neal, Lingafelter & Rose, 1978), manganese(II) (Nakasuka, Azuma, Katayama, Honda, Tanaka & Tanaka, 1985) and cadmium(II) ions (Nakasuka, Azuma & Tanaka, 1986b). This reagent gave a well defined crystal with magnesium(II) ion, and preliminary chemical analyses indicated a possibility of the dinuclear structure as mentioned above.

Experimental. Transparent crystals grown from an aqueous ethanol solution containing nearly equimolar amounts of $Mg(ClO_4)_2$ and $Na_2H_2(phdta)$, and recrystallized from water at pH 6. Elemental analysis: calcd for $C_{14}H_{24}Mg_2N_2O_{14}$: C 34.11, N 5.68, H 4.91%; found: C 33.97, N 5.58, H 4.78%. Crystal of dimensions $0.3 \times 0.3 \times 0.2$ mm. D_m by flotation in $C_6H_{12}/C_2H_2Br_4$. Rigaku AFC-5R diffractometer, graphite-monochromated Cu $K\alpha$ radiation. Cell dimensions from 2θ for 20 reflections ($40 \leq 2\theta \leq 42^\circ$). Intensities measured up to $2\theta = 120^\circ$ in $h - 10/10$, $k - 12/0$, $l - 10/10$. $R_{int} = 0.025$. Three standard reflections monitored after every 100 scans, no variation in intensity. Total of 3038 unique reflections measured. Absorption corrections applied by an empirical method (Walker & Stuart, 1983), relative absorption factors 0.881–1.164. Structure solved by the heavy-atom method. Positional parameters of all atoms and anisotropic thermal parameters of non-H atoms refined by the block-diagonal least-squares technique. H atoms located on a difference electron density map. Temperature factor

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic temperature factors ($\text{\AA}^2 \times 10^2$)

$$B_{eq} = \frac{1}{3} \sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

	x	y	z	B_{eq}
Mg(1)	663.8 (5)	6685.1 (4)	7513.7 (5)	140 (1)
Mg(2)	5226.4 (5)	6200.4 (5)	12819.1 (5)	148 (1)
O(1)	2699 (1)	6483 (1)	8681 (1)	188 (3)
O(2)	4255 (1)	6479 (1)	10787 (1)	196 (3)
O(3)	-1013 (1)	6005 (1)	8466 (1)	208 (3)
O(4)	-2383 (1)	6317 (1)	9926 (1)	265 (3)
O(5)	1465 (1)	6771 (1)	5685 (1)	235 (3)
O(6)	2842 (1)	7747 (1)	4554 (1)	288 (3)
O(7)	-1032 (1)	7321 (1)	6271 (1)	281 (3)
O(8)	-1752 (1)	8677 (1)	4952 (2)	321 (3)
O(9)	318 (1)	4806 (1)	6730 (1)	241 (3)
O(10)	3281 (1)	5687 (1)	13293 (1)	180 (3)
O(11)	6322 (1)	6003 (1)	14863 (1)	326 (3)
O(12)	7274 (1)	6710 (1)	12536 (1)	247 (3)
O(13)	5075 (1)	4438 (1)	12057 (1)	237 (3)
O(14)	5325 (1)	8009 (1)	13689 (1)	237 (3)
N(1)	1125 (1)	7787 (1)	9940 (1)	141 (3)
N(2)	1639 (1)	8776 (1)	7604 (1)	144 (3)
C(1)	1977 (2)	8983 (1)	10199 (2)	150 (4)
C(2)	2507 (2)	9661 (2)	11580 (2)	223 (4)
C(3)	3357 (2)	10789 (2)	11806 (2)	252 (4)
C(4)	3713 (2)	11235 (2)	10672 (2)	252 (4)
C(5)	3181 (2)	10573 (1)	9292 (2)	202 (4)
C(6)	2289 (2)	9454 (1)	9047 (2)	144 (3)
C(7)	1965 (2)	6998 (1)	10775 (2)	175 (4)
C(8)	3064 (2)	6634 (1)	10043 (2)	148 (3)
C(9)	-270 (2)	7761 (1)	10258 (2)	187 (4)
C(10)	-1306 (2)	6596 (1)	9502 (2)	170 (4)
C(11)	2682 (2)	8759 (2)	6750 (2)	240 (4)
C(12)	2291 (2)	7680 (2)	5569 (2)	183 (4)
C(13)	384 (2)	9279 (2)	6863 (2)	223 (4)
C(14)	-892 (2)	8348 (1)	5941 (2)	196 (4)

of each H atom set to B_{eq} of the bonded atom. $\sum(w|\Delta F|^2)$ minimized, $w = [\sigma^2(F_o) + 0.00177|F_o|^2]^{-1}$ for reflections with $w^{1/2}|F_o| > 3$ and $w^{1/2}|\Delta F| < 4$, $w = 0$ otherwise. Final $R = 0.035$, $wR = 0.052$, $S = 1.127$ for 2832 reflections. $(\Delta/\sigma)_{max} = 0.4$, $\Delta\rho_{max} = 0.17 \text{ e \AA}^{-3}$. Atomic scattering factors calculated by $\sum[a_i \exp(-b_i \lambda^{-2} \sin^2 \theta)]$ ($i = 1-4$) (*International Tables for X-ray Crystallography*, 1974). Calculations performed on a FACOM M340R computer at Shionogi Research Laboratories.

Discussion. Atomic coordinates and equivalent isotropic temperature factors of non-H atoms are given in Table 1.* Bond lengths and angles are listed in Table 2. The numbering system and molecular structure appear in Fig. 1, and the crystal packing is illustrated in Fig. 2; figures were drawn with the aid of the program *PLUTO* (Motherwell & Clegg, 1978). *Phdta* acts as a hexadentate ligand but the central ion Mg(1) is further hydrated to give rise to heptacoordination. The other pentahydrated metal ion Mg(2) is in a nearly octahedral environment. The two polyhedra are linked together *via* one carboxylate oxygen atom O(2).

* Lists of structure factors, anisotropic thermal parameters, and atomic coordinates and isotropic temperature factors B of H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51986 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Selected bond lengths (Å), bond angles (°) and hydrogen-bond lengths (Å)

Mg(1)—O(1)	2.081 (2)	Mg(1)—O(3)	2.181 (2)
Mg(1)—O(5)	2.135 (2)	Mg(1)—O(7)	2.070 (2)
Mg(1)—O(9)	2.139 (2)	Mg(1)—N(1)	2.434 (2)
Mg(1)—N(2)	2.427 (2)	Mg(2)—O(2)	2.051 (2)
Mg(2)—O(10)	2.091 (2)	Mg(2)—O(11)	2.062 (2)
Mg(2)—O(12)	2.100 (2)	Mg(2)—O(13)	2.033 (2)
Mg(2)—O(14)	2.118 (2)	O(1)—C(8)	1.265 (2)
O(2)—C(8)	1.252 (2)	O(3)—C(10)	1.264 (2)
O(4)—C(10)	1.242 (2)	O(5)—C(12)	1.257 (3)
O(6)—C(12)	1.250 (3)	O(7)—C(14)	1.257 (2)
O(8)—C(14)	1.242 (3)	N(1)—C(1)	1.450 (2)
N(1)—C(7)	1.488 (2)	N(1)—C(9)	1.472 (2)
N(2)—C(6)	1.450 (2)	N(2)—C(11)	1.476 (3)
N(2)—C(13)	1.487 (3)	C(1)—C(2)	1.394 (3)
C(1)—C(6)	1.390 (3)	C(2)—C(3)	1.388 (3)
C(3)—C(4)	1.378 (3)	C(4)—C(5)	1.386 (3)
C(5)—C(6)	1.395 (3)	C(7)—C(8)	1.515 (3)
C(9)—C(10)	1.522 (3)	C(11)—C(12)	1.509 (3)
C(13)—C(14)	1.511 (3)		

O(1)—Mg(1)—O(3)	112.1 (1)	O(1)—Mg(1)—O(5)	88.4 (1)
O(1)—Mg(1)—O(7)	163.9 (1)	O(1)—Mg(1)—O(9)	85.4 (1)
O(1)—Mg(1)—N(1)	73.1 (1)	O(1)—Mg(1)—N(2)	88.9 (1)
O(3)—Mg(1)—O(5)	150.3 (1)	O(3)—Mg(1)—O(7)	81.3 (1)
O(3)—Mg(1)—O(9)	78.9 (1)	O(3)—Mg(1)—N(1)	69.9 (1)
O(3)—Mg(1)—N(2)	126.2 (1)	O(5)—Mg(1)—O(7)	83.0 (1)
O(5)—Mg(1)—O(9)	81.6 (1)	O(5)—Mg(1)—N(1)	139.0 (1)
O(5)—Mg(1)—N(2)	73.2 (1)	O(7)—Mg(1)—O(9)	106.7 (1)
O(7)—Mg(1)—N(1)	104.9 (1)	O(7)—Mg(1)—N(2)	75.7 (1)
O(9)—Mg(1)—N(1)	131.0 (1)	O(9)—Mg(1)—N(2)	154.3 (1)
N(1)—Mg(1)—N(2)	70.2 (1)	O(2)—Mg(2)—O(10)	94.4 (1)
O(2)—Mg(2)—O(11)	176.1 (1)	O(2)—Mg(2)—O(12)	90.7 (1)
O(2)—Mg(2)—O(13)	90.6 (1)	O(2)—Mg(2)—O(14)	91.1 (1)
O(10)—Mg(2)—O(11)	89.1 (1)	O(10)—Mg(2)—O(12)	174.9 (1)
O(10)—Mg(2)—O(13)	86.5 (1)	O(10)—Mg(2)—O(14)	90.9 (1)
O(11)—Mg(2)—O(12)	85.8 (1)	O(11)—Mg(2)—O(13)	91.3 (1)
O(11)—Mg(2)—O(14)	87.1 (1)	O(12)—Mg(2)—O(13)	94.0 (1)
O(12)—Mg(2)—O(14)	88.4 (1)	O(13)—Mg(2)—O(14)	177.0 (1)
Mg(1)—O(1)—C(8)	121.2 (1)	Mg(2)—O(2)—C(8)	140.3 (1)
Mg(1)—O(3)—C(10)	123.8 (1)	Mg(1)—O(5)—C(12)	122.4 (1)
Mg(1)—O(7)—C(14)	122.8 (1)	Mg(1)—N(1)—C(1)	114.5 (1)
Mg(1)—N(1)—C(7)	99.6 (1)	Mg(1)—N(1)—C(9)	107.9 (1)
C(1)—N(1)—C(7)	110.3 (1)	C(1)—N(1)—C(9)	113.4 (1)
C(7)—N(1)—C(9)	110.2 (1)	Mg(1)—N(2)—C(6)	114.5 (1)
Mg(1)—N(2)—C(11)	105.0 (1)	Mg(1)—N(2)—C(13)	104.8 (1)
C(6)—N(2)—C(11)	112.2 (1)	C(6)—N(2)—C(13)	109.6 (1)
C(11)—N(2)—C(13)	110.4 (1)	N(1)—C(1)—C(2)	121.4 (2)
N(1)—C(1)—C(2)	119.2 (2)	C(2)—C(1)—C(6)	119.4 (2)
C(1)—C(2)—C(3)	120.3 (2)	C(2)—C(3)—C(4)	120.2 (2)
C(3)—C(4)—C(5)	119.9 (2)	C(4)—C(5)—C(6)	120.2 (2)
N(2)—C(6)—C(1)	118.7 (2)	N(2)—C(6)—C(5)	121.5 (2)
C(1)—C(6)—C(5)	119.8 (2)	N(1)—C(7)—C(8)	109.6 (1)
O(1)—C(8)—O(2)	123.4 (2)	O(1)—C(8)—C(7)	116.8 (2)
O(2)—C(8)—C(7)	119.8 (2)	N(1)—C(9)—C(10)	110.6 (2)
O(3)—C(10)—O(4)	125.3 (2)	O(3)—C(10)—C(9)	116.9 (2)
O(4)—C(10)—C(9)	117.7 (2)	N(2)—C(11)—C(12)	113.0 (2)
O(5)—C(12)—O(6)	124.5 (2)	O(5)—C(12)—C(11)	118.2 (2)
O(6)—C(12)—C(11)	117.2 (2)	N(2)—C(13)—C(14)	114.3 (2)
O(7)—C(14)—O(8)	125.2 (2)	O(7)—C(14)—C(13)	117.6 (2)
O(8)—C(14)—C(13)	117.0 (2)		

Proton donor	Acceptor	$R_{D \cdots O}$	$R_{H \cdots O}$
O(9)H	O(12) (1 - x, 1 - y, 2 - z)	3.117 (2)	2.28 (3)
O(9)H'	O(5) (-x, 1 - y, 1 - z)	2.778 (2)	1.89 (3)
O(10)H	O(6) (x, y, 1 + z)	2.668 (2)	1.81 (3)
O(10)H'	O(3) (-x, 1 - y, 2 - z)	2.744 (2)	1.76 (3)
O(11)H	O(10) (1 - x, 1 - y, 3 - z)	2.808 (2)	1.93 (3)
O(11)H'	O(7) (1 + x, y, 1 + z)	2.708 (2)	1.71 (3)
O(12)H	O(4) (1 + x, y, z)	2.647 (2)	1.71 (3)
O(12)H'	O(8) (1 + x, y, 1 + z)	2.900 (2)	2.11 (3)
O(13)H	O(1) (1 - x, 1 - y, 2 - z)	2.770 (2)	1.88 (3)
O(13)H'	O(4) (-x, 1 - y, 2 - z)	2.769 (2)	1.80 (3)
O(14)H	O(8) (1 + x, y, 1 + z)	2.748 (2)	1.82 (3)
O(14)H'	O(6) (x, y, 1 + z)	2.758 (2)	1.90 (3)

Structurally similar anions have been found in $\text{Na}_2[\text{Mg}(\text{H}_2\text{O})\text{Y}]\cdot 5\text{H}_2\text{O}$ (Stezowski, Countryman & Hoard, 1973) and $[\text{Mg}(\text{H}_2\text{O})_6][\text{Mg}(\text{H}_2\text{O})\text{Y}]\cdot 2\text{H}_2\text{O}$ (Passer *et al.*, 1977), where Y denotes the edta anion. The chelated anion is heptacoordinated in both

cases, but the geometry is better described in first approximation as monocapped trigonal prismatic for the former complex, while it is very close to the pentagonal bipyramid for the latter complex. Coplanarity of the phenylenediamine chelate ring may be apt to give rise to a monocapped trigonal prism (Fig. 1), but some distortion from it is found in the present complex, for which the sum of interior angles for the chelate ring [Mg(1)—N(1)—C(1)—C(6)—N(2)—Mg(1)] is 537.1° , in contrast to 539.9° for the aforementioned Cd-phdta complex (Nakasuka *et al.*, 1986b) and to 539.8° for a Cu-phdta complex (Nakasuka *et al.*, 1986a).

In accordance with the previous findings for the phdta complexes, metal-N distances (2.434 and 2.427 Å) are again longer than those in the

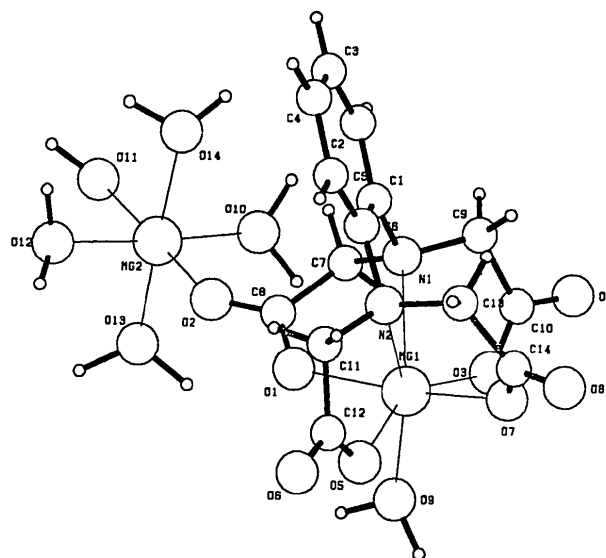


Fig. 1. A perspective view of the molecular structure of dimagnesium(II) *o*-phenylenediaminetetraacetate. Pentahydrated Mg(2) is in an O_h environment, while Mg(1) is heptacoordinated. The molecular geometry of the Mg(1)—phdta moiety is close to a water-capped trigonal prism, for which one triangle is defined by N(1), O(1) and O(3), and the other by N(2), O(5) and O(7). The rectangle approximately defined by the four carboxylate oxygen atoms is nearly perpendicular to a twofold axis passing through Mg(1) and bisecting the line N(1)—N(2).

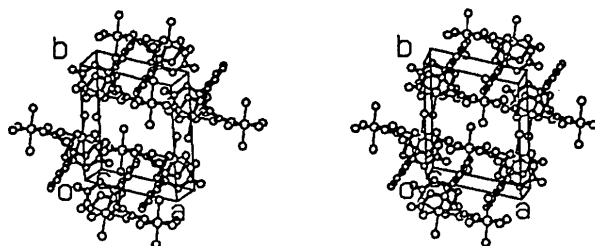


Fig. 2. Stereoscopic illustration of the molecular packing of dimagnesium(II) *o*-phenylenediaminetetraacetate, viewed approximately down the *c* axis. H atoms are omitted for clarity.

corresponding edta complexes {2.378 Å in $\text{Na}_2[\text{Mg}(\text{H}_2\text{O})\text{Y}]\cdot 5\text{H}_2\text{O}$ and 2.396 Å in $[\text{Mg}(\text{H}_2\text{O})_6][\text{Mg}(\text{H}_2\text{O})\text{Y}]\cdot 2\text{H}_2\text{O}$ }. This reflects the reduced basicity on the nitrogen atoms in phdta compared with those in edta. Configuration around the N atoms is well described as tetrahedral, since the sum of the three C—N—C bond angles is 333.9 (3)° for N(1) and 332.2 (3)° for N(2), respectively, although our preliminary work shows a quasiplanar configuration in neutral ligand H_4phdta and exclusive protonation on the carboxylate oxygens atoms alone (no zwitterion).

The pentahydrated $\text{Mg}(2)\text{O}_6$ moiety may be approximately described as octahedral, though the bond lengths $\text{Mg}(2)\text{—O}$ are somewhat more divergent than those in the other edta complexes having a discrete hexahydrated moiety; *i.e.* the $\text{Mg}(2)\text{—O}$ bond ranges from 2.033 to 2.118 Å (2.076 Å on the average) in $[\text{Mg}(\text{H}_2\text{O})_5][\text{Mg}(\text{H}_2\text{O})\text{phdta}]$, while it ranges from 2.047 to 2.087 Å (ave. 2.062 Å) in $[\text{Mg}(\text{H}_2\text{O})_6][\text{Mg}(\text{H}_2\text{O})\text{Y}]$ (Passer *et al.*, 1977) and from 2.053 to 2.087 Å (ave. 2.076 Å) in $[\text{Mg}(\text{H}_2\text{O})_6][\text{H}_2\text{Y}]$ (Julian *et al.*, 1973). These values may be compared with the mean Mg—O distance of 2.044 Å found for an aqueous solution (2 molar in MgCl_2 and CaCl_2) by an X-ray diffraction study (Caminiti, Licheri, Piccaluga & Pinna, 1977). All the water molecules are hydrated in this Mg—phdta complex, differing from those in the dimeric Mg—edta complex. These water molecules act as proton donor in a three-dimensional hydrogen-bonding network, as summarized in Table 2.

We thank Professor M. Tanaka of Nagoya University for helpful discussions. NN is very grateful to

Professor N. Sakabe of the National Laboratory for High Energy Physics, Tsukuba, for his valuable advice and continuing encouragement, and to Dr T. Yamane of Nagoya University for his useful discussions. This research was partly financed by Grants-in-Aid for Scientific Research No. 63540449 from the Ministry of Education, Science and Culture, Japan.

References

- CAMINITI, R., LICHERI, G., PICCALUGA, G. & PINNA, G. (1977). *Chem. Phys. Lett.* **47**, 275–278.
- International Tables for X-ray Crystallography* (1974). Vol. IV, p. 99. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- JULIAN, M. O'D., DAY, V. W. & HOARD, J. L. (1973). *Inorg. Chem.* **12**, 1754–1757.
- MCCANDLISH, E. F. K., MICHAEL, T. K., NEAL, J. A., LINGAFELTER, E. C. & ROSE, N. J. (1978). *Inorg. Chem.* **17**, 1383–1394.
- MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
- NAKASUKA, N., AZUMA, S., KATAYAMA, C., HONDA, M., TANAKA, J. & TANAKA, M. (1985). *Acta Cryst.* **C41**, 1176–1179.
- NAKASUKA, N., AZUMA, S. & TANAKA, M. (1986a). *Acta Cryst.* **C42**, 1482–1485.
- NAKASUKA, N., AZUMA, S. & TANAKA, M. (1986b). *Acta Cryst.* **C42**, 1736–1739.
- PASSER, E., WHITE, J. G. & CHENG, K. L. (1977). *Inorg. Chim. Acta*, **24**, 13–23.
- POZHIDAEV, A. I., POLINOVA, T. N. & PORAI-KOSHITS, M. A. (1972). *Acta Cryst.* **A28**, S76.
- POZHIDAEV, A. I., POLINOVA, T. N., PORAI-KOSHITS, M. A. & LOGVINENKO, V. A. (1973). *Zh. Strukt. Khim.* **14**, 746–747.
- SOLANS, X., GALI, S., FONT-ALTABA, M., OLIVA, J. & HERRERA, J. (1983). *Acta Cryst.* **C39**, 438–440.
- STEZOWSKI, J. J., COUNTRYMAN, R. & HOARD, J. L. (1973). *Inorg. Chem.* **12**, 1749–1754.
- WALKER, N. & STUART, D. (1983). *Acta Cryst.* **A39**, 158–166.

Acta Cryst. (1989). **C45**, 1490–1493

Structure of *cis*-Bis(4-allyl-4*H*-1,2,4-triazole)bis(2,2'-bipyridine)ruthenium(II) Bis(hexafluorophosphate)

BY HU ZHEN-SHAN, LIN YONG-HUA AND JIN SONG-CHUN

Changchun Institute of Applied Chemistry, Academia Sinica, Changchun, Jilin, People's Republic of China

AND J. G. VOS

School of Chemical Sciences, Dublin City University, Dublin 9, Ireland

(Received 1 December 1988; accepted 27 February 1989)

Abstract. $[\text{Ru}(\text{C}_5\text{H}_7\text{N}_3)_2(\text{C}_{10}\text{H}_8\text{N}_2)_2](\text{PF}_6)_2$, $M_r = 921.74$, monoclinic, $P2_1/n$, $a = 13.472$ (4), $b = 20.278$ (6), $c = 13.995$ (4) Å, $\beta = 101.34$ (2)°, $V = 3748.6$ Å³, $Z = 4$, $D_x = 1.633$, $D_m = 1.66$ (1) Mg m^{-3} , $\lambda(\text{Mo K}\alpha) = 0.71069$ Å, $\mu = 0.819$ mm^{-1} , $F(000) = 1848.80$, $T = 298$ K, $R = 0.0489$ for 2635 unique reflections [$I > 3\sigma(I_o)$]. The planar 2,2'-bipyridine ligands are coordinated in a *cis* fashion to the central