$2\theta/\omega$ scans
Absorption correction: none
2333 measured reflections
2199 independent reflections
1501 reflections with $I > 2\sigma(I)$

$h = -7 \rightarrow 7$
$k = -11 \rightarrow 11$
$l = 0 \rightarrow 12$
1 standard reflection
frequency: 60 min
intensity decay: 1.5%

Refinement

Refinement on F^2
$R(F) = 0.0691$
$wR(F^2) = 0.2508$
$S = 1.027$
2193 reflections
162 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.1321P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = -0.040$
$\Delta\rho_{max} = 1.074$ e Å^{-3}
$\Delta\rho_{min} = -1.119$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Mn1—N2	2.062 (6)	Mn1—Cl2	2.255 (2)
Mn1—N1	2.062 (6)	Mn1—O1	2.288 (6)
Mn1—Cl3	2.209 (2)	Mn1—Cl1	2.520 (2)
N2—Mn1—N1	78.5 (2)	Cl3—Mn1—O1	92.1 (2)
N2—Mn1—Cl3	171.4 (2)	Cl2—Mn1—O1	90.0 (2)
N1—Mn1—Cl3	94.0 (2)	N2—Mn1—Cl1	86.5 (2)
N2—Mn1—Cl2	94.2 (2)	N1—Mn1—Cl1	88.3 (2)
N1—Mn1—Cl2	171.6 (2)	Cl3—Mn1—Cl1	97.61 (9)
Cl3—Mn1—Cl2	92.96 (10)	Cl2—Mn1—Cl1	95.49 (8)
N2—Mn1—O1	83.1 (2)	O1—Mn1—Cl1	168.6 (2)
N1—Mn1—O1	85.1 (2)		

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
O1—H11···Cl1^{i}	0.79 (7)	2.39 (7)	3.143 (7)	160 (6)
O1—H12···Cl2^{ii}	0.76 (9)	2.50 (9)	3.250 (7)	172 (9)

Symmetry codes: (i) $1 + x, y, z$; (ii) $1 - x, -y, -z$.

The aqua H atoms were located from difference maps and refined isotropically. The aromatic H atoms were included in calculated positions as riding atoms with *SHELXL*93 (Sheldrick, 1993) default parameters.

Data collection: *STADI*4 (Stoe & Cie, 1995*a*). Cell refinement: *STADI*4. Data reduction: *XRED* (Stoe & Cie, 1995*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93. Molecular graphics: *PLATON*/*PLUTON* (Spek, 1990). Software used to prepare material for publication: *SHELXL*93.

This work has been supported by the Swiss National Science Foundation and the Spanish Interministerial Committee for Science and Technology.

Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: AV1000). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 31–37.
Chen, X.-M., Shi, K.-L., Mak, T. C. W. & Luo, B.-S. (1995). *Acta Cryst.* C**51**, 358–361.
Lubben, M., Meetsma, A. & Feringa, B. L. (1995). *Inorg. Chim. Acta*, **230**, 169–172.
Lumme, P. O. & Lindell, E. (1988). *Acta Cryst.* C**44**, 463–465.
Perlepes, S. P., Blackmann, A. G., Huffman, J. C. & Christou, G. (1991). *Inorg. Chem.* **30**, 1665–1668.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.
Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.
Stoe & Cie (1995*a*). *STADI4. Diffractometer Control Program for Windows.* Stoe & Cie, Darmstadt, Germany.
Stoe & Cie (1995*b*). *XRED. Data Reduction Program for Windows.* Stoe & Cie, Darmstadt, Germany.
Weighardt, K. (1989). *Angew. Chem. Int. Ed. Engl.* **28**, 1153–1172.

Acta Cryst. (1997). C**53**, 432–434

Sodium *trans*-Bis(methyliminodiacetato-*O*,*N*,*O*)chromate(III), $Na[Cr(C_5H_7NO_4)_2]$

JUNG-SUN SUH,[a] SUNG-JIN PARK,[a] KYU-WANG LEE,[a]
IL-HWAN SUH,[b] JIN-HO LEE,[b] JU-HO SONG[b] AND
MI-RAN OH[b]

[a]*Department of Chemistry, Myongji University, Yongin, Kyonggi-Do, 449-928, Korea, and* [b]*Department of Physics, Chungnam National University, Taejon, 305-764, Korea. E-mail: ihsuh@hanbat.chungnam.ac.kr*

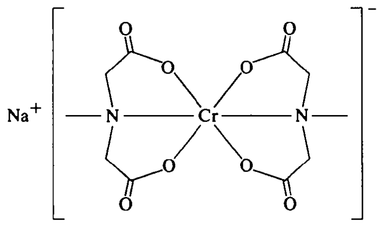
(*Received 25 June 1996; accepted 23 October 1996*)

Abstract

In the title compound, $Na[Cr^{III}\{RN(CH_2CO_2)_2\}_2]$, where R is CH_3, the methyliminodiacetate anions function as tridentate ligands defining a slightly distorted octahedral coordination at the Cr^{III} ion, with the N atoms in a *trans* configuration. The Cr—N distance of 2.060 (2) Å is slightly shorter than the values found in the complexes where R is bulkier, *i.e* an isopropyl or *tert*-butyl group, whereas the Cr—O bond lengths vary little as the size of R is changed. The methyliminodiacetato derivative appears to be the most symmetrical of the substituted iminodiacetato complexes so far studied.

Comment

Alkyliminodiacetatochromium(III) complexes, $[Cr^{III}\{R\text{-}N(CH_2CO_2)_2\}]^-$, are good models for structural and ligand-field analyses since the size of the R group affects both the Cr—N bond distance and the configuration at the Cr atom. The smallest substituent (R = H) yields a *cis* configuration, but when R is isopropyl, the metal adopts a *trans* configuration (Mootz & Wunderlich, 1980*a*,*b*). The relationship between the Cr—N bond length and ligand-field strength is also of interest. We report here the X-ray crystal structure of the title complex, (I), with $R = CH_3$, and compare it with those of other alkyliminodiacetatochromium(III) complexes. The spectroscopic properties of these complexes will also be considered.



(I)

A displacement ellipsoid plot of (I) is shown in Fig. 1, with the shaded bonds indicating the asymmetric unit of the structure. The Cr atom lies on an inversion center and the Na atom lies on a twofold axis. As predicted from the spectroscopic work of Hoggard & Schmidtke (1972), the title complex shows a *trans* configuration. Its high symmetry confirms predictions from low-temperature luminescence and excitation spectra (Flint & Matthews, 1975; Park, 1995). Bond angles at the Cr atom deviate by no more than 6.6° from ideal octahedral values of 90 and 180°.

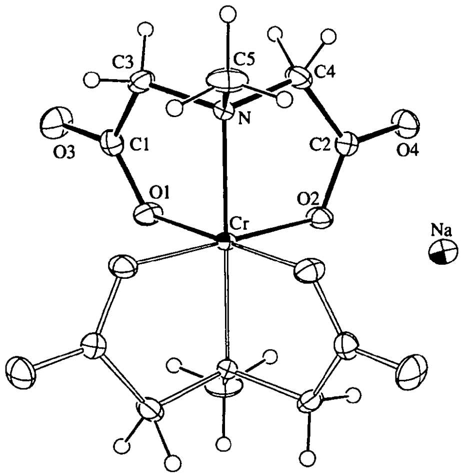


Fig. 1. Displacement ellipsoid plot (40% probability level) of the title complex with the numbering scheme. The asymmetric unit is indicated with shaded bonds. H atoms are represented as spheres of arbitrary radii.

The Cr—O bond lengths [1.963 (2) and 1.948 (2) Å] agree to within 0.01 Å with those for other structures in this series (Fürst, Gouzerh & Jeannin, 1979). The Cr—N distance [2.060 (2) Å] is slightly shorter than the distances found in the Cr^{III} complexes with bulkier ligands, *i.e* 2.118 (R = isopropyl) and 2.152 Å (R = *tert*-butyl) (Mootz & Wunderlich, 1980*a*,*b*). This is in agreement with the ligand-field strengths of these complexes (Wernike, Schmidtke & Hoggard, 1977).

The shortest contact between the Na^+ ion and an O atom of the complex anion is Na···O4 of 2.519 (2) Å, which is slightly longer than that observed in the structure of the complex where R is isopropyl (Mootz & Wunderlich, 1980*b*).

Experimental

Crystals of the title compound were prepared according to the method of Weyh & Hamm (1968). Two recrystallizations from aqueous solution at room temperature in the dark yielded light-red crystals. Sample purity was checked by low-temperature (12 K) luminescence spectroscopy.

Crystal data

$Na[Cr(C_5H_7NO_4)_2]$
$M_r = 365.22$
Monoclinic
$C2/c$
$a = 16.848 (2)$ Å
$b = 6.2610 (10)$ Å
$c = 17.0340 (10)$ Å
$\beta = 132.49 (3)°$
$V = 1325.0 (3)$ Å^3
$Z = 4$
$D_x = 1.831$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 13.3–17.9°$
$\mu = 0.942$ mm^{-1}
$T = 293 (2)$ K
Rectangular
0.2 × 0.2 × 0.1 mm
Light red

Data collection

Enraf–Nonius CAD-4 diffractometer
$w/2\theta$ scans
Absorption correction: none
1863 measured reflections
1013 independent reflections
910 reflections with $I > 2\sigma(I)$

$R_{int} = 0.0146$
$\theta_{max} = 24°$
$h = -18 \rightarrow 19$
$k = -7 \rightarrow 6$
$l = -19 \rightarrow 10$
3 standard reflections
frequency: 180 min
intensity decay: <1.0%

Refinement

Refinement on F^2
$R(F) = 0.0284$
$wR(F^2) = 0.0741$
$S = 0.166$
1013 reflections
130 parameters
All Hs refined
$w = 1/[\sigma^2(F_o^2) + (0.2434P)^2 + 188.1861P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.37$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U^{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Na	1/2	0.2375 (3)	1/4	0.0367 (4)
Cr	3/4	3/4	1/2	0.0177 (2)
O1	0.87005 (15)	0.5464 (3)	0.58352 (15)	0.0294 (5)
O2	0.66241 (15)	0.5467 (3)	0.38302 (14)	0.0286 (5)

O3	1.0339 (2)	0.4914 (4)	0.6467 (2)	0.0444 (6)
O4	0.6128 (2)	0.4501 (3)	0.2306 (2)	0.0365 (5)
N	0.8141 (2)	0.8278 (3)	0.4350 (2)	0.0185 (5)
C1	0.9473 (2)	0.5842 (4)	0.5872 (2)	0.0264 (6)
C2	0.6692 (2)	0.5552 (4)	0.3126 (2)	0.0235 (6)
C3	0.9287 (2)	0.7614 (5)	0.5155 (2)	0.0315 (7)
C4	0.7530 (3)	0.7032 (6)	0.3345 (2)	0.0397 (8)
C5	0.8072 (3)	1.0603 (5)	0.4125 (3)	0.0386 (8)

Table 2. *Selected geometric parameters (Å, °)*

Cr—O2	1.948 (2)	O4—C2	1.223 (3)
Cr—O1	1.963 (2)	N—C3	1.484 (3)
Cr—N	2.060 (2)	N—C4	1.490 (4)
O1—C1	1.283 (3)	N—C5	1.490 (4)
O2—C2	1.280 (3)	C1—C3	1.517 (4)
O3—C1	1.222 (3)	C2—C4	1.510 (4)
O2—Cr—O1	90.84 (9)	C5—N—Cr	113.1 (2)
O2—Cr—N	85.11 (8)	O3—C1—O1	123.8 (3)
O1—Cr—N	83.42 (8)	O3—C1—C3	119.8 (3)
C1—O1—Cr	115.5 (2)	O1—C1—C3	116.4 (2)
C2—O2—Cr	116.5 (2)	O4—C2—O2	123.8 (2)
C3—N—C4	111.9 (2)	O4—C2—C4	119.1 (2)
C3—N—C5	109.7 (2)	O2—C2—C4	117.1 (2)
C4—N—C5	110.0 (3)	N—C3—C1	112.4 (2)
C3—N—Cr	105.3 (2)	N—C4—C2	114.2 (2)
C4—N—Cr	106.7 (2)		

The title structure was refined by full-matrix least-squares techniques with anisotropic displacement parameters for non-H atoms. All H atoms were found from difference Fourier syntheses and refined isotropically.

Data collection, cell refinement and data reduction: *SDP* (Frenz, 1985). Program used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program used to refine structure: *SHELXL*93 (Sheldrick, 1993). Preparation of materials for publication and molecular graphics: *ORTEP*II (Johnson, 1976) and *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). Software used to prepare material for publication: *NRCVAX*.

K-WL acknowledges the Korea Science and Engineering Foundation for financial support.

Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: MU1280). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Flint, C. D. & Matthews, A. P. (1975). *J. Chem. Soc. Faraday Trans.* 2, **71**, 379–386.
Frenz, B. A. (1985). *Enraf–Nonius Structure Determination Package.* Enraf–Nonius, Delft, The Netherlands.
Fürst, W., Gouzerh, H. & Jeannin, Y. (1979). *J. Coord. Chem.* **8**, 237–243.
Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl Cryst.* **22**, 384–387.
Hoggard, P. E. & Schmidtke, H.-H. (1972). *Ber. Bunsenges. Phys. Chem.* **76**, 1013–1021.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Mootz, D. & Wunderlich, H. (1980*a*). *Acta Cryst.* B**36**, 445–447.
Mootz, D. & Wunderlich, H. (1980*b*). *Acta Cryst.* B**36**, 721–722.
Park, S.-J. (1995). MS dissertation, Myongji University, Korea.
Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures.* University of Göttingen, Germany.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.
Wernike, R., Schmidtke, H.-H. & Hoggard, P. E. (1977). *Inorg. Chim. Acta*, **24**, 145–148.
Weyh, J. A. & Hamm, R. E. (1968). *Inorg. Chem.* **7**, 2431–2435.

Acta Cryst. (1997). C**53**, 434–436

trans-Bis[4-amino-3,5-bis(2-pyridyl)-1,2,4-triazole-N^1,N']copper(II) Bis(tetrafluoroborate)

ALAN J. JIRCITANO,[a] SHAUN O. SOMMERER[b] AND KHALIL A. ABBOUD[c]

[a]*Division of Science, The Pennsylvania State University at Erie, The Behrend College, Station Road, Erie, Pennsylvania 16563-1200, USA,* [b]*Department of Physical Sciences, Barry University, 11300 N. E. 2nd Avenue, Miami Shores, Florida 33161-6695, USA, and* [c]*Department of Chemistry, The University of Florida, Gainesville, Florida 32611-2046, USA. E-mail: a0j@psu.edu*

(*Received 24 June 1996; accepted 3 December 1996*)

Abstract

The title complex, $[Cu(C_{12}H_{10}N_6)_2](BF_4)_2$, contains square-planar mononuclear Cu^{II}–bis[4-amino-3,5-bis(2-pyridyl)-1,2,4-triazole] cations counterbalanced by semi-coordinated BF_4^- anions. The Cu atoms reside on inversion centers and have a distorted octahedral coordination sphere. The amino group is hydrogen bridged to two of the F atoms of an adjacent BF_4^- anion.

Comment

Dipyridyl ligands give structural chemists access to a wide variety of transition metal complexes and unusual geometries. Simple variation of the central metal atom and associated counterions has resulted in the isolation of novel dimers (Sommerer, Westcott, Jircitano & Abboud, 1995) and metal-containing extended structures (Sommerer, Westcott, Jircitano & Abboud, 1996). The ligand 4-amino-3,5-bis(2-pyridyl)-1,2,4-triazole (abpt) exemplifies this behavior and has been used to isolate dinuclear Ni^{II} (Keij, de Graaff, Haasnoot & Reedijk, 1984), Cu^{II} (van Koningsbruggen *et al.*, 1995) and Mn^{II} (Faulmann, van Koningsbruggen, de Graaff, Haasnoot & Reedijk, 1990) species, as well as mononuclear Rh^{II} (García *et al.*, 1986), Ru^{II} (Rheingold, Saisuwan & Thomas, 1993) and Cu^{II} (Cornelissen *et al.*, 1992). An