C8	0.065 (3)	0.666 (2)	0.9024 (7)	0.106 (6)
C9	−0.0347 (17)	0.790 (3)	0.9116 (8)	0.132 (9)
C10	0.045 (2)	0.880 (2)	0.9522 (7)	0.097 (5)
C11	−0.0330 (15)	1.088 (2)	0.8357 (6)	0.076 (4)
C12	−0.0356 (14)	0.984 (2)	0.7916 (6)	0.072 (3)
C13	0.0948 (16)	1.008 (2)	0.7563 (5)	0.076 (4)
C14	0.1798 (17)	1.132 (2)	0.7803 (8)	0.096 (5)
C15	0.098 (2)	1.182 (2)	0.8274 (8)	0.095 (5)
C16	0.4959 (12)	1.1428 (10)	0.9166 (4)	0.046 (2)
C17	0.5283 (14)	1.274 (1)	0.8853 (5)	0.058 (3)
C18	0.6580 (16)	1.363 (1)	0.9014 (6)	0.075 (3)
C19	0.7545 (16)	1.326 (1)	0.9462 (6)	0.080 (4)
C20	0.7213 (17)	1.196 (1)	0.9759 (5)	0.077 (3)
C21	0.5940 (13)	1.105 (1)	0.9611 (4)	0.058 (3)

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

*Cp*I denotes the centre of the cyclopentadienyl ring I (C1–C5), *Cp*II that of ring II (C6–C10) and *Cp*III that of ring III (C11–C15).

Np—O	2.136 (7)	Np—C10	2.71 (1)
Np—C1	2.73 (1)	Np—C11	2.74 (1)
Np—C2	2.75 (1)	Np—C12	2.74 (1)
Np—C3	2.72 (1)	Np—C13	2.74 (1)
Np—C4	2.74 (1)	Np—C14	2.71 (1)
Np—C5	2.73 (1)	Np—C15	2.76 (1)
Np—C6	2.72 (1)	O—C16	1.326 (12)
Np—C7	2.76 (1)	Np· · ·*Cp*I	2.460 (7)
Np—C8	2.73 (2)	Np· · ·*Cp*II	2.477 (8)
Np—C9	2.73 (1)	Np· · ·*Cp*III	2.470 (7)
O—Np· · ·*Cp*I	97.6 (4)	*Cp*I· · ·Np· · ·*Cp*II	116.4 (6)
O—Np· · ·*Cp*II	102.3 (5)	*Cp*I· · ·Np· · ·*Cp*III	116.1 (5)
O—Np· · ·*Cp*III	101.7 (4)	*Cp*II· · ·Np· · ·*Cp*III	117.7 (6)
Np—O—C16	159.8 (6)		

Table 3. *Short interligand distances (Å)*

O· · ·C contacts (< 3.1 Å)			
O· · ·C2	2.95 (2)		
C· · ·C contacts (< 3.4 Å)			
C1· · ·C13	3.17 (2)	C4· · ·C8	3.28 (2)
C1· · ·C14	3.24 (2)	C5· · ·C13	3.22 (2)
C2· · ·C16	3.38 (2)	C9· · ·C11	3.20 (2)
C3· · ·C7	3.31 (2)	C9· · ·C12	3.30 (2)
C4· · ·C7	3.19 (2)	C10· · ·C11	3.36 (2)

In the final difference Fourier map there are four substantial peaks with heights in the range 0.86–1.12 e Å^{-3}, close to the Np atom (0.94–1.18 Å).

Disorder or librational motion of the cyclopentadienyl rings may explain the excessive vibrational anisotropy of C2 and C9: for these atoms the ratio of the maximum to minimum mean-square atomic displacement is greater than 10:1.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *SDP* (Frenz, 1983). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP* (Johnson, 1965).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1209). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Disodium Aqua[2,5,8-tris(carboxymethyl)-12-phenyl-2,5,8-triaza-11-oxa-1,9-dodecanedicarboxylato(5−)]europate Sesquihydrate

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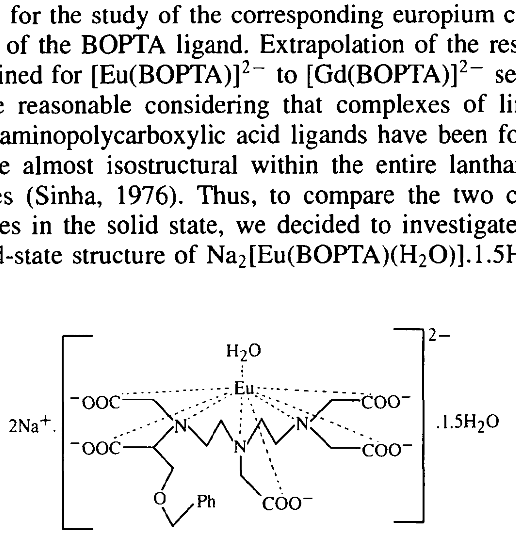
Abstract

The structure of the disodium salt of the [$Eu(C_{22}H_{26}N_3O_{11})(H_2O)]^{2-}$ complex anion is isomorphous with that of the corresponding gadolinium(III) compound. The Eu^{III} atom is nine-coordinate with the donor atoms disposed at the vertices of a tricapped trigonal prism, the cap positions being occupied by two N atoms of the ligand and the O atom of the ligated water molecule.

Comment

The bis(*N*-methylglucammonium) salt of [Gd(BOPTA)]$^{2-}$ [international non-proprietary name (INN): gadobenate; BOPTA is the pentanion of 2,5,8-tris(carboxymethyl)-12-phenyl-2,5,8-triaza-11-oxa-1,9-dodecanedicarboxylic acid] is a promising contrast agent for magnetic resonance imaging (MRI) which is currently under clinical development (Cavagna, Daprá, Maggioni, de Haën & Felder, 1991; Rosati, Pirovano & Spinazzi, 1994). Owing to the potential importance of such a compound, we thought it necessary to investigate thoroughly its structure both in the solid state and in solution. Although crystallization of the bis(*N*-methylglucammonium) salt of $[Gd(BOPTA)]^{2-}$ has so far been unsuccessful, crystals suitable for X-ray diffraction analysis have been obtained for the corresponding disodium salt and its solid-state structure has been reported recently (Uggeri *et al.*, 1995). The paramagnetic character of Gd^{III} does not allow investigation of the structure of $[Gd(BOPTA)]^{2-}$ in solution by NMR techniques; however, such techniques can be used successfully for the study of the corresponding europium complex of the BOPTA ligand. Extrapolation of the results obtained for $[Eu(BOPTA)]^{2-}$ to $[Gd(BOPTA)]^{2-}$ seems quite reasonable considering that complexes of linear polyaminopolycarboxylic acid ligands have been found to be almost isostructural within the entire lanthanide series (Sinha, 1976). Thus, to compare the two complexes in the solid state, we decided to investigate the solid-state structure of $Na_2[Eu(BOPTA)(H_2O)].1.5H_2O$, (I).



The solid-state structure of (I), which is made up of $[Eu(C_{22}H_{26}N_3O_{11})(H_2O)]^{2-}$ complex anions, sodium counterions and water molecules of crystallization, is isomorphous with that of the corresponding Gd^{III} compound (Uggeri *et al.*, 1995). Therefore, in this case also, the arrangement of the donor atoms around the metal ion gives rise to a distorted tricapped trigonal prism, according to Guggenberger & Muetterties (1976), who indicate that this kind of geometry is the preferred one for ML_9-type complexes. In fact, the values of the dihedral angles between the mean least-squares planes forming the faces of the coordination prism, reported in Table 3, compare favorably with those calculated for perfect D_{3h} geometry (Guggenberger & Muetterties, 1976). The cap positions are occupied by the O12 atom of the coordinated water molecule and the terminal N1 and N3 atoms of the polyaminopolycarboxylate ligand, while atoms N2, O6, O10 and O2, O5, O8 constitute the vertices of the two triangular faces of the coordination polyhedron (Fig. 1).

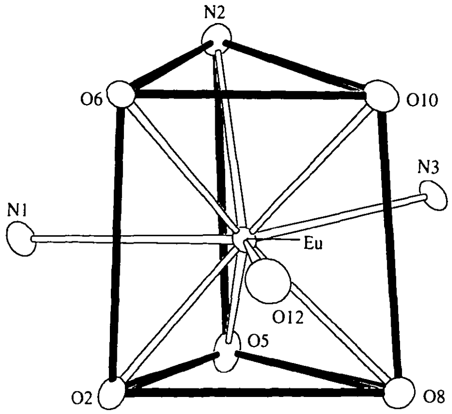


Fig. 1. The coordination polyhedron in $Na_2[Eu(BOPTA)(H_2O)].1.5H_2O$.

It is noteworthy that the two M—$N_{capping}$ distances are significantly different (see Table 2). The coordinating O atoms belonging to the chelating arms directly linked to the most distant N atom (N1) are located on the shortest edge of the rectangular face of the coordination prism. This feature is common to other europium and gadolinium complexes having a tricapped trigonal prism as the coordination polyhedron, particularly, $[Eu(DTPA\text{-}dienH^+)]_4^{4+}$ (Franklin & Raymond, 1994), $[Gd(DTPA\text{-}BEA)(H_2O)]$ (Konings, Dow, Love, Raymond, Quay & Rocklage, 1990) and $[Gd(BOPTA)(H_2O)]^{2-}$ (Uggeri *et al.*, 1995). The mean Eu—N (2.70 Å) and Eu—$O_{carboxylate}$ (2.38 Å) distances compare well with those reported for europium complexes having these kinds of donors (Grenthe, 1971; Ciampolini, Dapporto & Nardi, 1979; Spirlet, Rebizant, Desreux & Loncin, 1984; Chin, Morrow, Lake & Churchill, 1994; Franklin & Raymond, 1994), while the Eu—O_{water} distance [2.46 (1) Å] is slightly longer with respect to those previously published (Hansson, 1973; Spirlet, Rebizant, Desreux & Loncin, 1984; Chin, Morrow, Lake & Churchill, 1994). As is the case for the Gd^{III} ion in $[Gd(BOPTA)(H_2O)]^{2-}$, the europium ion is near the center of the prism, though shifted slightly towards the face capped by the water molecule and the triangular face composed of O donor atoms. This shift is about 0.2 Å in both cases.

Finally, the arrangement of the ligand around the metal ion is almost identical to that observed in the isostructural $[Gd(BOPTA)(H_2O)]^{2-}$ complex, with the O atoms not involved in the europium coordination and with the benzyloxymethyl arm pointing away from the metal-ion environment (Fig. 2). In the crystal lattice, sodium ions are octahedrally coordinated by the carboxylic O atoms of four different ligand molecules and a water molecule of crystallization (O13). Atoms Na1 and Na2, which are 3.75 (1) Å apart, are cross-linked by two carboxylic O atoms provided by two different molecules of the ligand. The contacts $O12\cdots O9(1-x,\ 1-y,\ 1-z)$ 2.89 (1), $O13\cdots O4$ 2.78 (1), $O13\cdots O7(1+x,\ 1+y,\ z)$ 2.85 (1) and $O14\cdots O4$ 2.87 (3) Å are indicative of $O—H\cdots O$ hydrogen bonding. In addition, the water O14 atom,

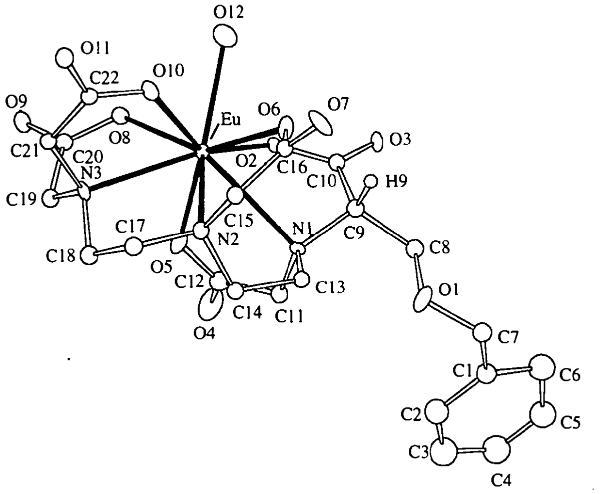


Fig. 2. An *ORTEP*II (Johnson, 1976) view of the $[Eu(BOPTA)(H_2O)]^{2-}$ complex anion. Displacement ellipsoids are plotted at the 20% probability level.

having a population parameter of 0.5, has some interaction with an H atom of the methylenic C17 atom and those bound to the aromatic C2 and C4 atoms. Other possible intermolecular C—H···O hydrogen bonds are shown in Table 2.

Experimental

The BOPTA ligand was synthesized by the method previously reported by Uggeri *et al.* (1995). BOPTA (4.87 g, 9.5 mmol) was suspended in H_2O (50 ml) and dissolved by the addition of 2 *N* NaOH (9.5 ml). After addition of Eu_2O_3 (1.65 g, 4.7 mmol), the suspension was stirred at 353 K for 1 h. The formation of the complex was monitored by both high-performance liquid chromatography and titration of the free ligand. The cloudy solution was filtered through a Millipore Durapore membrane (0.45 µ) filter and evaporated to dryness, affording the [Eu(BOPTA)] disodium salt in almost quantitative yield. Crystals of $Na_2[Eu(BOPTA)(H_2O)].1.5H_2O$ suitable for X-ray diffraction analysis were obtained by slow crystallization from a 1:1 (*v*/*v*) DMSO/H_2O solution at 323 K.

Crystal data

$Na_2[Eu(C_{22}H_{26}N_3O_{11})(H_2O)].1.5H_2O$
$M_r = 751.44$
Triclinic
$P\bar{1}$
$a = 9.099\ (4)$ Å
$b = 9.520\ (3)$ Å
$c = 16.753\ (6)$ Å
$\alpha = 102.46\ (3)°$
$\beta = 92.34\ (3)°$
$\gamma = 102.22\ (3)°$
$V = 1379.20\ (93)$ Å^3
$Z = 2$
$D_x = 1.809$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 9.30–13.35°$
$\mu = 2.378$ mm^{-1}
$T = 298$ K
Parallelepiped
0.45 × 0.25 × 0.20 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: refined from ΔF (Walker & Stuart, 1983)
$T_{min} = 0.49$, $T_{max} = 0.61$
4822 measured reflections
4642 independent reflections

2723 observed reflections $[F > 4\sigma(F)]$
$R_{int} = 0.042$
$\theta_{max} = 25°$
$h = -10 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = 0 \rightarrow 19$
2 standard reflections frequency: 60 min intensity decay: none

Refinement

Refinement on F^2
$R(F) = 0.066$
$wR(F^2) = 0.148$
$S = 1.14$
2723 reflections
269 parameters
H-atom parameters not refined
Weighting scheme based on measured e.s.d.'s

$(\Delta/\sigma)_{max} = 0.564$
$\Delta\rho_{max} = 1.25$ e Å^{-3}
$\Delta\rho_{min} = -1.16$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2A, 2.2C and 2.3.1)

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}
Eu	0.25441 (8)	0.20141 (8)	0.35938 (5)	0.0203 (2)
Na1	0.6317 (6)	0.4295 (7)	0.4379 (4)	0.046 (2)
Na2	0.7823 (8)	0.1387 (7)	0.5130 (4)	0.056 (2)
O12	0.2996 (12)	0.2194 (12)	0.5071 (7)	0.058 (5)
O13	0.6837 (11)	0.5447 (11)	0.3257 (7)	0.052 (4)
O14	0.724 (2)	0.187 (2)	0.0438 (13)	0.062 (9)
O1	0.4216 (11)	−0.2814 (11)	0.1736 (7)	0.052 (4)
O2	0.4954 (9)	0.1640 (9)	0.3805 (5)	0.030 (3)
O3	0.6441 (9)	0.0034 (9)	0.3888 (6)	0.034 (3)
N1	0.3502 (11)	−0.0069 (11)	0.2389 (6)	0.026 (4)
O4	0.6009 (13)	0.3045 (12)	0.1921 (8)	0.074 (5)
O5	0.3876 (11)	0.2902 (10)	0.2559 (6)	0.045 (4)
N2	0.0302 (12)	0.0581 (12)	0.2456 (7)	0.032 (4)
O6	0.1459 (9)	−0.0385 (9)	0.3777 (6)	0.039 (3)
O7	−0.0533 (11)	−0.2210 (11)	0.3689 (7)	0.055 (4)
N3	0.1131 (10)	0.3862 (11)	0.3074 (7)	0.027 (4)
O8	0.3636 (9)	0.4619 (9)	0.4185 (5)	0.030 (3)
O9	0.3930 (10)	0.7000 (9)	0.4184 (6)	0.038 (4)
O10	0.0293 (9)	0.2296 (10)	0.4255 (6)	0.035 (3)
O11	−0.1562 (9)	0.3420 (10)	0.4586 (6)	0.037 (3)
C1	0.6194 (17)	−0.3570 (17)	0.0887 (10)	0.045 (4)
C2	0.637 (2)	−0.247 (2)	0.0446 (11)	0.066 (5)
C3	0.753 (2)	−0.218 (2)	0.0016 (14)	0.082 (6)
C4	0.869 (2)	−0.280 (2)	0.0091 (13)	0.081 (6)
C5	0.862 (2)	−0.378 (2)	0.0503 (11)	0.067 (5)
C6	0.744 (2)	−0.418 (2)	0.1019 (13)	0.074 (6)
C7	0.4833 (15)	−0.4016 (15)	0.1323 (9)	0.037 (3)
C8	0.5211 (14)	−0.1914 (14)	0.2435 (8)	0.032 (3)
C9	0.4370 (14)	−0.0810 (14)	0.2917 (8)	0.033 (3)
C10	0.5354 (13)	0.0322 (14)	0.3573 (8)	0.026 (3)
C11	0.4527 (14)	0.0721 (14)	0.1903 (8)	0.032 (3)
C12	0.4851 (16)	0.2333 (15)	0.2124 (9)	0.038 (3)
C13	0.2103 (13)	−0.1007 (13)	0.1913 (8)	0.025 (3)
C14	0.1051 (14)	−0.0057 (14)	0.1733 (8)	0.029 (3)
C15	−0.0625 (14)	−0.0564 (15)	0.2814 (8)	0.032 (3)
C16	0.0135 (13)	−0.1129 (14)	0.3425 (8)	0.028 (3)
C17	−0.0547 (14)	0.1623 (14)	0.2238 (8)	0.031 (3)
C18	0.0485 (13)	0.3149 (13)	0.2227 (7)	0.026 (3)
C19	0.2345 (13)	0.5226 (13)	0.3086 (7)	0.024 (3)
C20	0.3424 (13)	0.5695 (13)	0.3863 (8)	0.025 (3)
C21	0.0052 (13)	0.4305 (13)	0.3653 (7)	0.024 (3)
C22	−0.0493 (13)	0.3263 (13)	0.4169 (7)	0.024 (3)

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

Eu—O2	2.320 (9)	Eu—O12	2.46 (1)
Eu—O5	2.36 (1)	Eu—N3	2.65 (1)
Eu—O6	2.379 (9)	Eu—N2	2.65 (1)
Eu—O10	2.405 (9)	Eu—N1	2.81 (1)
Eu—O8	2.437 (8)		
O2—Eu—O5	75.3 (3)	O10—Eu—N3	66.0 (3)
O2—Eu—O6	90.8 (3)	O8—Eu—N3	65.3 (3)
O5—Eu—O6	133.9 (3)	O12—Eu—N3	120.0 (3)
O2—Eu—O10	143.4 (3)	O2—Eu—N2	130.8 (3)
O5—Eu—O10	137.5 (3)	O5—Eu—N2	88.3 (3)
O6—Eu—O10	75.7 (3)	O6—Eu—N2	67.9 (3)
O2—Eu—O8	86.4 (3)	O10—Eu—N2	75.5 (3)
O5—Eu—O8	74.5 (3)	O8—Eu—N2	133.8 (3)
O6—Eu—O8	149.4 (3)	O12—Eu—N2	134.4 (4)
O8—Eu—O10	88.7 (3)	N3—Eu—N2	68.6 (3)
O12—Eu—O2	73.5 (3)	O2—Eu—N1	62.1 (3)
O12—Eu—O5	137.2 (4)	O5—Eu—N1	62.9 (3)
O12—Eu—O6	75.1 (4)	O6—Eu—N1	71.7 (3)
O12—Eu—O10	70.2 (3)	O10—Eu—N1	138.7 (3)
O8—Eu—O12	74.9 (3)	O8—Eu—N1	131.5 (3)
O2—Eu—N3	140.8 (3)	O12—Eu—N1	122.9 (3)
O5—Eu—N3	71.5 (3)	N3—Eu—N1	117.1 (3)
O6—Eu—N3	127.4 (3)	N2—Eu—N1	69.1 (3)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13*B*···O1	0.97 (2)	2.38 (2)	2.82 (2)	107 (1)
C19—H19*B*···O5	0.97 (2)	2.36 (2)	2.86 (2)	111 (1)

Table 3. *Dihedral angles (°) for the coordination polyhedron of* $[Eu(BOPTA)(H_2O)]^{2-}$

Face 1	Face 2	Angle (°)	Idealized angle (°) for D_{3h} geometry
O2—O5—O8	O6—N2—O10	176.90 (4)	180
O2—O5—O8	N1—N3—O12	173.6 (2)	180
O6—N2—O10	N1—N3—O12	173.3 (3)	180
O8—O10—O12	N1—O5—N2	138.4 (4)	146.4
O2—O6—O12	O5—N2—N3	141.3 (4)	146.4
O2—O6—N1	O8—O10—N3	139.6 (4)	146.4
O2—O6—O12	O2—O6—N1	19.2 (4)	26.4
O8—O10—O12	O8—O10—N3	22.8 (3)	26.4
O5—N2—N1	O5—N2—N3	17.5 (4)	26.4

The crystals exist as twins with a twinning plane perpendicular to the *c** reciprocal axis. Reciprocal matrices for both twins were found and the ratio between the intensities of the two twins was about 1.3. Corrections for the superimposed reflections were performed using a local program; reflections were considered to be superimposed when the distance was less than 0.012 $Å^{-1}$ in the reciprocal lattice. It was not possible to localize the water H atoms during the refinement process.

Data collection: *CAD*-4 *Software* (Enraf-Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *CAD*-4 *Software*. Program(s) used to solve structure: *SHELX*76 (Sheldrick, 1976). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including intermolecular contact distances, have been deposited with the IUCr (Reference: NA1216). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Five New Chlorochromates of Organic Bases

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Abstract

We have synthesized and characterized five new chlorochromate(VI) complexes of organic bases: 4,4′-bipyridinediium bis[chlorochromate(VI)], $(C_{10}H_{10}N_2)[CrO_3Cl]_2$, 4-methylquinolinium chlorochromate(VI), $(C_{10}H_{10}N)[CrO_3Cl]$, 6-methylquinolinium chlorochromate(VI), $(C_{10}H_{10}N)[CrO_3Cl]$, *N*,*N*′-ethylenediaminium chloride chlorochromate(VI), $(C_2H_{10}N_2)(Cl)[CrO_3Cl]$ and (5-chloro-2-pyridyl)(2-pyridyl)ammonium chloro-