Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

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

	x	y	z	$U_{\rm eq}$
Co	0.06270 (3)	0.70990 (1)	0.21777 (1)	0.01459 (8)
O1	0.1196 (2)	0.63839 (7)	0.13433 (6)	0.0172 (2)
O2	0.0433 (2)	0.59265 (7)	0.26132 (6)	0.0214 (3)
O4	0.3525 (2)	0.71039 (8)	0.25060 (7)	0.0234 (3)
O31	−0.2305 (2)	0.69943 (7)	0.19339 (6)	0.0219 (3)
O32	−0.2591 (2)	0.67768 (9)	0.08381 (6)	0.0277 (3)
O33	−0.4908 (2)	0.66064 (10)	0.13891 (7)	0.0377 (4)
O41	0.5135 (3)	0.61839 (9)	0.35835 (8)	0.0464 (4)
O42	0.3803 (2)	0.66325 (11)	0.44164 (9)	0.0442 (4)
O43	0.6743 (2)	0.67614 (8)	0.44304 (7)	0.0271 (3)
N1	0.0629 (2)	0.83134 (8)	0.24956 (7)	0.0166 (3)
N2	0.1011 (2)	0.78547 (8)	0.13168 (7)	0.0171 (3)
N3	0.1183 (2)	0.74738 (8)	0.07250 (7)	0.0186 (3)
N5	−0.0056 (2)	0.71906 (8)	0.32189 (7)	0.0171 (3)
N6	−0.0484 (2)	0.65175 (8)	0.35236 (7)	0.0188 (3)
N30	−0.3253 (2)	0.67869 (8)	0.13768 (7)	0.0192 (3)
N40	0.5192 (2)	0.65225 (9)	0.41420 (8)	0.0263 (3)
C1	0.0917 (2)	0.88720 (10)	0.20562 (8)	0.0184 (3)
C2	0.0993 (3)	0.96466 (10)	0.22493 (10)	0.0250 (4)
C3	0.0757 (3)	0.98311 (11)	0.29109 (10)	0.0293 (4)
C4	0.0454 (3)	0.92527 (10)	0.33635 (10)	0.0245 (4)
C5	0.0405 (2)	0.84939 (10)	0.31363 (8)	0.0180 (3)
C6	0.1135 (2)	0.85909 (10)	0.13628 (8)	0.0179 (3)
C7	0.1460 (3)	0.91358 (11)	0.08089 (10)	0.0265 (4)
C8	0.1201 (2)	0.66956 (10)	0.07828 (8)	0.0162 (3)
C9	0.0075 (2)	0.78218 (10)	0.35629 (8)	0.0179 (3)
C10	−0.0031 (3)	0.79068 (11)	0.43062 (9)	0.0242 (4)
C11	−0.0245 (2)	0.58782 (9)	0.31523 (8)	0.0175 (3)
C14	−0.0737 (2)	0.51282 (9)	0.34347 (8)	0.0184 (3)
C15	−0.1204 (3)	0.50505 (10)	0.40934 (9)	0.0219 (3)
C16	−0.1596 (3)	0.43302 (11)	0.43378 (10)	0.0260 (4)
C17	−0.1529 (3)	0.36828 (11)	0.39295 (11)	0.0289 (4)
C18	−0.1078 (3)	0.37541 (11)	0.32725 (10)	0.0287 (4)
C19	−0.0682 (3)	0.44743 (11)	0.30279 (9)	0.0235 (4)
C24	0.1237 (2)	0.62412 (10)	0.01502 (8)	0.0188 (3)
C25	0.2236 (3)	0.55565 (11)	0.01946 (9)	0.0234 (4)
C26	0.2239 (3)	0.50948 (11)	−0.03771 (10)	0.0288 (4)
C27	0.1237 (3)	0.53175 (12)	−0.09957 (10)	0.0298 (4)
C28	0.0261 (3)	0.60009 (12)	−0.10441 (9)	0.0289 (4)
C29	0.0249 (2)	0.64686 (11)	−0.04725 (9)	0.0222 (3)

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

Co—O4	2.129 (1)	Co—N2	2.200 (1)
Co—O31	2.140 (1)	Co—N1	2.202 (1)
Co—O1	2.153 (1)	Co—O2	2.224 (1)
Co—N5	2.193 (1)		
O4—Co—O31	173.44 (5)	O4—Co—N1	87.02 (5)
O4—Co—O1	86.88 (5)	O31—Co—N1	96.12 (5)
O31—Co—O1	94.19 (5)	N5—Co—N1	69.70 (5)
O4—Co—N5	93.49 (5)	N2—Co—N1	69.77 (5)
O31—Co—N5	82.25 (5)	O4—Co—O2	90.01 (5)
O4—Co—N2	90.27 (5)	O31—Co—O2	83.87 (5)
O31—Co—N2	96.22 (5)	O1—Co—O2	78.36 (4)
O1—Co—N2	72.02 (5)	N5—Co—O2	70.55 (5)

All H atoms were located in a difference Fourier synthesis and were refined isotropically. A hemisphere of frames (0.3° in ω) was collected. The first 50 frames were remeasured at the end of data collection to monitor instrument and crystal stability.

Data collection: *SMART* (Siemens, 1995). Cell refinement: *SMART* and *SAINT* (Siemens, 1995). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1995). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1270). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Triaqua(oxydiacetato-*O*,*O*′,*O*″)strontium(II) Monohydrate

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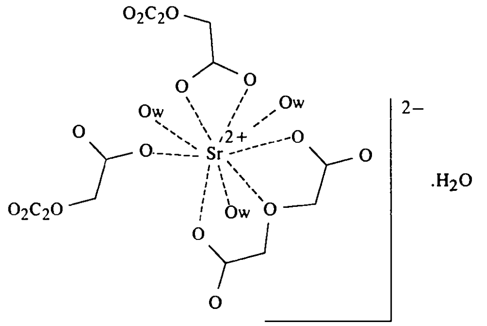
Abstract

The title complex, $[Sr(C_4H_4O_5)(H_2O)_3]_n.nH_2O$, consists of polymeric zigzag chains formed by SrO_9 units bridged by SrO_2Sr groups along the b axis, with Sr···Sr separations of 4.518 (1) Å. Each Sr atom is associated with three O atoms from a tridentate oxydiacetate ligand, three O atoms from adjacent carboxylate groups, one monodentate and one chelating, and three O atoms from metal-bound water molecules. The chains in the crystal are linked through an extensive hydrogen-bonding system into a complex three-dimensional network.

Comment

The study of new strontium(II) carboxylate complexes is of current interest as a result of their potential applications in precursor systems for strontium-containing ceramic materials. Recently, strontium acetate and oxalate have been used for this purpose in different chemical-processing methods (Lee, Doi & Yuasa, 1994; Alconchel, Ulla & Lombardo, 1994; Marta, Zaharescu, Ciontea & Petrisor, 1993). There are few crystallographic studies of strontium(II) carboxylates and among those reported have been strontium(II) acetates (Trunov, Chubinidze, Efremov & Velikodnyi, 1984; Trunov, Endeladze & Efremov, 1988; Groombridge, Harris, Packer, Hursthouse & Walker, 1985) and strontium(II) oxalate (Sterling, 1965). Among related carboxylate ligands, oxydiacetate (oda; $^-O_2CCH_2OCH_2CO_2^-$) has been shown to be a good complexing agent for a variety of metal ions. Structural data of hydrated oda complexes have been reported with cadmium(II) (Boman, 1977*a*,*b*,*c*), calcium(II) (Uchtman & Oertel, 1973), copper(II) (Whitlow & Davey, 1975), erbium(III) (Elding, 1977), lanthanum(III) (Baggio, Garland, Perec & Vega, 1996), zinc(II) (Baggio, Garland & Perec, 1996) and zirconium(IV) (Baggio, Garland, Perec & Vega, 1995), and different types of structures with the ligand bonded in mono-, bi- and tridentate modes have also been observed. The results of the present study include the preparation, characterization and X-ray structure determination of the novel polymeric strontium complex $[Sr(C_4H_4O_5)(H_2O)_3]_n.nH_2O$, (1).



(1)

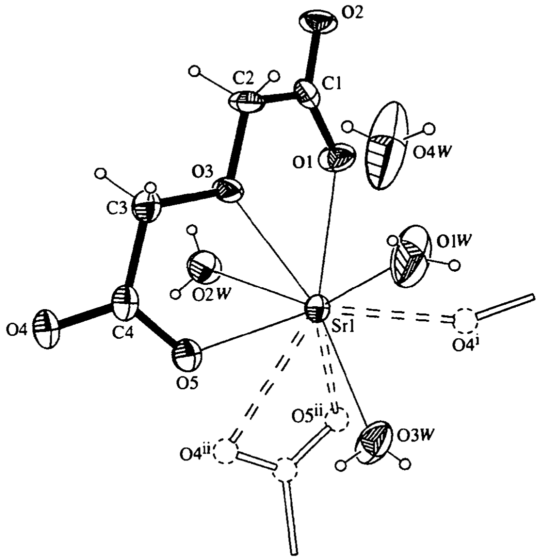


Fig. 1. Schematic diagram showing the structure of the building unit of (1) with atoms labels. Displacement ellipsoids are drawn at the 50% probability level.

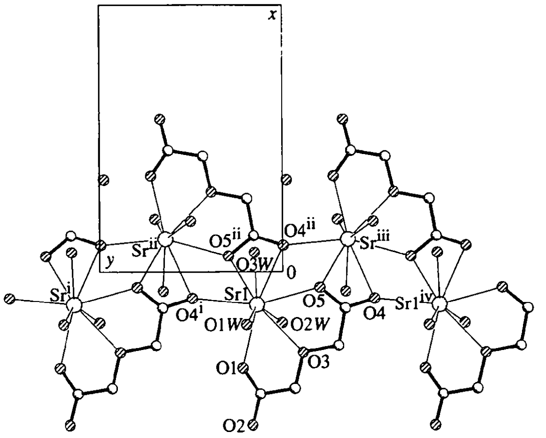


Fig. 2. A packing view along z showing the way in which chains are formed. Symmetry codes: (i) $x, 1+y, z$; (ii) $2-x, \frac{1}{2}+y, \frac{1}{2}-z$; (iii) $2-x, -\frac{1}{2}+y, \frac{1}{2}-z$; (iv) $x, -1+y, z$.

The X-ray structure determination of (1) reveals the complex to be a polymeric zigzag chain along the *b* axis built up from greatly distorted nine-coordinate strontium units, each of which is surrounded by an O_9 donor set. A schematic drawing of the full Sr coordination polyhedron is shown in Fig. 1, while Fig. 2 presents the packing pattern with a comprehensive labelling system we will recall in describing the polymeric interactions.

The Sr1 ion is nine coordinate, with one oda ligand completely deprotonated, coordinating two carboxylate O atoms [Sr1—O1 2.560 (5), Sr1—O5 2.613 (4) Å and O1—Sr1—O5 119.25 (14)°] and the ether O atom [Sr1—O3 2.658 (4) Å], one outer carboxylate O atom of an adjacent oda ligand [Sr1—O4^i 2.561 (4) Å], two O atoms of an unsymmetrically chelated carboxylate of a second adjacent oda ligand [Sr1—O4^{ii} 2.880 (5), Sr1—O5^{ii} 2.709 (5) Å and O4^{ii}—Sr1—O5^{ii} 46.15 (11)°; symmetry codes: (i) $x, 1+y, z$; (ii) $2-x, \frac{1}{2}+y, \frac{1}{2}-z$] and three aqua O atoms [Sr1—O1*W* 2.582 (5), Sr1—O2*W* 2.616 (4) and Sr1—O3*W* 2.703 (4) Å]. The Sr1—O bond distances in (1), which range from 2.560 (5) to 2.880 (5) Å, with an average value of 2.652 (32) Å, are similar to the corresponding value found for this bond in the eight- and nine-coordinate carboxylates noted above.

A new feature in the present structure is the asymmetric coordination mode of the oda ligand; one carboxylate end is monodentate through a short bond [Sr—O1 2.560 (5) Å], whereas the second end is bonded to three adjacent Sr atoms in an arrangement involving both *anti–anti* bridging [$Sr1^{iv}$—O4 2.561 (4) and Sr1—O5 2.613 (4) Å] and unsymmetrical chelating [$Sr1^{iii}$—O5 2.709 (5) and $Sr1^{iii}$—O4 2.880 (5) Å; symmetry codes: (iii) $2-x$, $y-\frac{1}{2}$, $\frac{1}{2}-z$; (iv) x, $y-1$, z] modes. The coordination of the latter carboxylates results in an infinite polymeric chain along the *b* axis, with an Sr···Sr non-interacting distance of 4.518 (1) Å. Similar ambidentate functions of carboxylate groups have been reported in the polymeric strontium acetates. The CCOCC frame of the oda ligand is bent about the Sr1—O3 bond vector leading to two planar halves (C1, C2, Sr1, O3 and C3, C4, Sr1, O3) with a dihedral angle of 17.3 (2)° between them. The carboxylate groups are further rotated about their C—C bonds by 7.6 (4) and 15.9 (4)° for the monodentate and the bridging and chelating carboxylates, respectively. The combined effect is that of a twisting of the oda ligand as previously reported in two cadmium oxydiacetate compounds (Boman, 1977*a*,*b*). The departure from planarity of the Sr(oda) unit is best described by the mean deviation from the least-squares plane through the ten non-H atoms (0.22 Å), with a maximum deviation of 0.45 Å for the O4 atom. Bond distances and angles within the ligand are consistent with the values reported for other metal–oxydiacetate compounds.

The crystal structure of (1) is a complex three-dimensional network of parallel chains linked together through hydrogen bonds involving all coordinated and non-coordinated water molecules, and the O2 and O5 atoms of the oda ligands. A summary of bond distances and angles involving hydrogen-bonding contacts is given in Table 3. It is of interest to note that the present structure differs significantly from that reported for the related $[Ca(oda)(H_2O)_5(H_2O)]$ (Uchtman & Oertel, 1973), which is mononuclear, with the Ca^{2+} ion in an eightfold coordination geometry and a tridentate oda ligand in a nearly planar Ca(oda) group. The $Ca(oda)(H_2O)_5$ units are linked only by hydrogen bonding involving the sixth water molecule, but without metal-bridging complexation.

Thermogravimetric analysis measurements show that compound (1) is thermally stable up to 325 K, at which temperature loss of water commences. The removal of the crystallization and coordination water molecules occurs in four overlapping stages in the range 325–574 K. The subsequent degradation occurs at 648–748 K, the residual mass at 748 K being consistent with the formation of $SrCO_3$ (49%) as confirmed by the matching of its powder XRD diffraction pattern with file No. 05-0418 of the JCPDS (1992).

Experimental

Strontium carbonate (1.1 g, 7.5 mmol) was added to a hot solution of oxydiacetic acid (1.5 g, 11.25 mmol) in water (100 ml) in portions of 50 mg. After stirring for 6 h at 353 K, the resultant solution was passed through a glass filter and the filtrate allowed to stand at room temperature. After 4 d, colorless X-ray quality crystals of the product were collected and dried under vacuum for 4 h (yield 1.50 g, 70%). Analysis calculated (found) for $C_4H_{12}O_9Sr$: C 16.75 (16.65), H 4.03 (4.05)%. IR (KBr disk, cm^{-1}): 3386 (*s*, *br*), 2917 (*m*), 1653 (*s*), 1647 (*s*), 1619 (*s*), 1576 (*vs*), 1509 (*m*), 1466 (*m*), 1441 (*s*), 1422 (*s*), 1316 (*s*), 1119 (*s*), 1048 (*m*), 932 (*m*), 820 (*m*), 737 (*m*), 703 (*m*), 664 (*w*), 610 (*w*), 555 (*w*) and 465 (*w*). Oxydiacetic acid (Fluka) and strontium carbonate (Fluka) were purchased and used without further purification. Water was purified by a Millipore Milli-Q system yielding 18 MΩ cm water. The elemental analyses (C, H) were performed on a Carlo Erba EA 1108 instrument, the IR spectra were recorded as KBr disks with a Nicolet 510P FT-IR spectrophotometer, the thermogravimetric analyses were recorded on a Mettler TG-50 thermal analyzer under an atmosphere of oxygen at a heating rate of 5° min^{-1} and the powder X-ray diffraction (XRD) data were collected using graphite-monochromated Cu *Kα* radiation on a Phillips X'Pert diffractometer, while single-crystal data were gathered with graphite-monochromated Mo *Kα* radiation on a four-circle Siemens *R*3*m* diffractometer.

Crystal data

$[Sr(C_4H_4O_5)(H_2O)_3].H_2O$
$M_r = 291.76$
Monoclinic
$P2_1/c$
$a = 11.400\ (2)$ Å
$b = 7.1350\ (10)$ Å
$c = 13.113\ (2)$ Å
$\beta = 114.49\ (1)°$
$V = 970.6\ (3)$ Å^3
$Z = 4$
$D_x = 1.997$ Mg m^{-3}
D_m not measured

Mo *Kα* radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 7.5–12.5°$
$\mu = 5.58$ mm^{-1}
$T = 293\ (2)$ K
Block
0.40 × 0.38 × 0.18 mm
Colorless

Data collection

Siemens *R*3*m* diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (*XEMP* in *SHELXTL/PC*; Sheldrick, 1991)
$T_{min} = 0.14$, $T_{max} = 0.35$
1384 measured reflections
1133 independent reflections
911 observed reflections [$I > 2\sigma(I)$]

$R_{int} = 0.024$
$\theta_{max} = 24.98°$
$h = 0 \rightarrow 7$
$k = -1 \rightarrow 8$
$l = -15 \rightarrow 14$
2 standard reflections monitored every 98 reflections intensity decay: <2%

Refinement

Refinement on F^2
$R(F) = 0.034$
$wR(F^2) = 0.085$

Extinction correction: *SHELXL*93 (Sheldrick, 1993)

$S = 1.029$
1133 reflections
154 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0541P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = <0.001$
$\Delta\rho_{\max} = 0.41$ e Å^{-3}
$\Delta\rho_{\min} = -0.37$ e Å^{-3}
Extinction coefficient:
0.031 (2)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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}
Sr1	0.87638 (6)	0.14210 (6)	0.24560 (4)	0.0193 (4)
O1	0.6385 (5)	0.2213 (6)	0.1309 (3)	0.0320 (14)
O2	0.4274 (5)	0.1686 (5)	0.0646 (4)	0.0308 (14)
O3	0.6979 (4)	−0.1099 (5)	0.2297 (3)	0.0252 (14)
O5	0.9416 (4)	−0.2033 (5)	0.3106 (3)	0.0298 (13)
O4	0.9000 (4)	−0.5032 (5)	0.2772 (4)	0.037 (2)
C1	0.5430 (8)	0.1245 (7)	0.1216 (5)	0.022 (2)
C2	0.5648 (7)	−0.0632 (8)	0.1810 (5)	0.025 (2)
C3	0.7232 (8)	−0.3035 (7)	0.2495 (5)	0.025 (2)
C4	0.8635 (8)	−0.3409 (7)	0.2817 (5)	0.023 (2)
O1*W*	0.8033 (6)	0.1974 (7)	0.4050 (4)	0.069 (2)
O2*W*	0.8084 (4)	0.0043 (5)	0.0442 (3)	0.0326 (14)
O3*W*	1.0750 (5)	0.1567 (5)	0.4504 (3)	0.039 (2)
O4*W*	0.6548 (7)	0.0215 (7)	0.4819 (5)	0.092 (2)

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

Sr1—O1	2.560 (5)	O1—C1	1.252 (8)
Sr1—O4^{i}	2.561 (4)	O2—C1	1.256 (8)
Sr1—O1*W*	2.582 (5)	O3—C3	1.413 (6)
Sr1—O5	2.613 (4)	O3—C2	1.421 (7)
Sr1—O2*W*	2.616 (4)	O5—C4	1.273 (7)
Sr1—O3	2.658 (4)	O4—C4	1.240 (7)
Sr1—O3*W*	2.703 (4)	C1—C2	1.516 (7)
Sr1—O5^{ii}	2.709 (5)	C3—C4	1.499 (9)
Sr1—O4^{ii}	2.880 (5)		
C3—O3—C2	114.3 (5)	O3—C3—C4	109.8 (5)
O1—C1—O2	125.1 (5)	O4—C4—O5	121.9 (8)
O1—C1—C2	119.1 (6)	O4—C4—C3	119.5 (6)
O2—C1—C2	115.8 (6)	O5—C4—C3	118.6 (5)
O3—C2—C1	110.8 (5)		

Symmetry codes: (i) $x, 1 + y, z$; (ii) $2 - x, \frac{1}{2} + y, \frac{1}{2} - z$.

Table 3. *Hydrogen-bonding geometry*† (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4*W*—H4*WB*···O2^{i}	0.94 0.67 (4)	1.77	2.668 (6)	160
O1*W*—H1*WA*···O2*W*^{ii}	0.94 0.67 (6)	1.89	2.789 (7)	161
O2*W*—H2*WA*···O3*W*^{iii}	0.94 0.66 (4)	1.94	2.801 (6)	151
O2*W*—H2*WB*···O2^{iv}	0.94 0.67 (1)	1.86	2.758 (6)	159
O3*W*—H3*WB*···O5^{v}	0.94 0.67 (8)	2.40	3.234 (7)	148
O3*W*—H3*WA*···O4*W*^{v}	0.94 0.66 (3)	2.27	3.102 (8)	148
O3*W*—H3*WA*···O1*W*^{v}	0.94 0.66 (3)	2.42	3.118 (6)	132

Symmetry codes: (i) $1 - x, y - \frac{1}{2}, \frac{1}{2} - z$; (ii) $x, \frac{1}{2} - y, \frac{1}{2} + z$; (iii) $2 - x, y - \frac{1}{2}, \frac{1}{2} - z$; (iv) $1 - x, -y, -z$; (v) $2 - x, -y, 1 - z$.

† Values arising from refinement are quoted with e.s.d.'s, while values normalized according to Jeffrey & Lewis (1978) and Taylor & Kennard (1983) are quoted without e.s.d.'s.

The title structure was solved through the usual combination of direct methods and difference Fourier procedures. H atoms attached to C atoms were placed at idealized positions and allowed to ride, while those corresponding to water molecules were located in a difference Fourier synthesis and subsequently refined with restraints. For distance and angle calculations, H-atom positions were corrected by sliding them along the bond so as to have 'neutron-like' values for the O—H and C—H distances [*PARST* (Nardelli, 1983) after Jeffrey & Lewis (1978) and Taylor & Kennard (1983)].

Data collection: *P3/P4-PC* (Siemens, 1991). Cell refinement: *P3/P4-PC*. Data reduction: *XDISK* in *SHELXTL/PC* (Sheldrick, 1991). Program(s) used to solve structure: *XS* in *SHELXTL/PC*. Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *XP* in *SHELXTL/PC*. Software used to prepare material for publication: *CIFTAB* in *SHELXL93*, *PARST* and *CSD* (Allen, Kennard & Taylor, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1264). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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