Crystal structure of tetra(aqua)(μ -2-mercaptoacetato-*S,O,O*)calcium, $\text{Ca}(\text{SCH}_2\text{COO})(\text{H}_2\text{O})_4$

B. Guillaume^I, S. Cendrowski-Guillaume^{II} and M. Nierlich^{*,III}^I Reckitt Benckiser, 98 Route de Sours, F-28011 Chartres, France^{II} ENSCPB-Université de Bordeaux I, CNRS(UMR 5629), Laboratoire de Chimie des Polymères Organiques, 16 avenue Pey-Berland, F-33607 Pessac Cedex, France^{III} CEA Saclay, DSM/DRECAM/SCM, Bât. 125, F-91191 Gif-sur-Yvette Cedex, France

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0.5–z], one S atom of the thioacetato ligands and four O atoms (O3–O6) of water molecules. The calcium coordination may be described as a distorted dodecahedron with the pentagonal plane defined by O1, O2, O5, S and O2A (at ± 0.311 Å), O3 being at 1.98 Å, O4 and O6 at -2.38 Å and -2.37 Å, respectively from this plane. Two calcium cations are bridged by one mercaptoacetato anion to form polymeric chains. The Ca–O2A–CaA bridge is asymmetric with Ca–O2A distance of 2.458 Å and $d(\text{CaA}—\text{O2A}) = 2.549$ Å: these distances are similar to those found for acetate calcium salts [7]. The Ca–O(water) distances averaged to 2.44 Å are within the range found for other calcium salts: for example, 2.404 Å for $[\text{Ca}(\text{CH}_3\text{COO})_3(\text{NO}_3)(\text{H}_2\text{O})_2]_n$ [4] and 2.42 Å for $\text{Ca}(\text{C}_{14}\text{H}_{14}\text{N}_2\text{O}_6\text{S}_2)(\text{H}_2\text{O})_4$ [8].

Abstract

$\text{C}_2\text{H}_{10}\text{CaO}_6\text{S}$, orthorhombic, *Pbca* (No. 61), $a = 8.527(2)$ Å, $b = 10.986(2)$ Å, $c = 16.328(3)$ Å, $V = 1529.6$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.019$, $wR_{\text{ref}}(F^2) = 0.050$, $T = 123$ K.

Source of material

Calcium 2-mercaptoacetate was prepared from a mixture of potassium 2-mercaptoacetate (4% weight) and calcium hydroxide (2.9% weight) in a liquid crystal emulsion [1,2]. Crystals were obtained as clear colorless needles upon raising the temperature of the full emulsion in an aluminum tube from 278 K to 291 K over 5 days.

Discussion

Calcium 2-mercaptoacetate is derived from 2-mercaptoacetic acid (HSCH_2COOH), which represents the first member of the mercaptocarboxylic acid series. The mercaptocarboxylic acid, commercially developed in the early 1940s, is used in cosmetology as an active material for cold wave permanents and depilatories [3]. A mixture of potassium 2-mercaptoacetate and calcium hydroxide in a liquid crystal emulsion leads to crystals. This liquid crystal emulsion which has been previously fully described [1,2] contains many compounds that are likely to crystallize. X-ray analysis was performed in order to identify the formula of the crystallized compound and in particular to clarify the nature of the cation (K or Ca). There are relatively few structural studies of mercaptoacetato complexes [4,5] and there is only one example with a bridging chelating mercaptoacetato ligand [6].

The crystal structure determination leads to the formula $\text{Ca}(\text{SCH}_2\text{COO})(\text{H}_2\text{O})_4$. Each Ca cation is eight coordinated, involving three O atoms [O1, O2, O2A; symmetry code 0.5+x, y,

Table 1. Data collection and handling.

Crystal:	colourless, irregular, size $0.10 \times 0.15 \times 0.20$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	10.70 cm^{-1}
Diffraction, scan mode:	Nonius Kappa CCD, $\Delta\varphi = 2^\circ$
$2\theta_{\text{max}}$:	49.42°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	9644, 1264
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1164
$N(\text{param})_{\text{refined}}$:	101
Programs:	SHELXS-86 [9], SHELXL-93 [10], SHELXTL [11], PLATON [12]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(3A)	8c	0.0530	0.8374	0.2112	0.028(6)
H(3B)	8c	0.2037	0.7801	0.2023	0.036(6)
H(4A)	8c	-0.2385	0.5221	0.3775	0.034(6)
H(4B)	8c	-0.1579	0.4634	0.4352	0.036(6)
H(5A)	8c	-0.1530	0.7813	0.4125	0.030(6)
H(5B)	8c	-0.0891	0.8580	0.3555	0.027(5)
H(6A)	8c	0.1645	0.3587	0.3883	0.050(7)
H(6B)	8c	0.2293	0.3763	0.3080	0.030(6)
H(2B)	8c	0.4030	0.5139	0.4581	0.027(5)
H(2A)	8c	0.4996	0.6289	0.4548	0.018(5)

* Correspondence author (e-mail: nierlich@drecam.cea.fr)

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Ca	8c	0.05574(4)	0.61299(3)	0.31913(2)	0.0078(2)	0.0081(2)	0.0092(2)	0.0001(1)	−0.0003(1)	0.0002(1)
S	8c	0.23243(5)	0.68634(3)	0.45994(2)	0.0118(2)	0.0145(2)	0.0091(2)	0.0011(1)	−0.0005(1)	−0.0007(2)
O(1)	8c	0.0616(1)	0.5246(1)	0.17841(6)	0.0099(6)	0.0121(6)	0.0150(6)	0.0020(4)	−0.0017(4)	−0.0022(4)
O(2)	8c	−0.1623(1)	0.61224(9)	0.21220(6)	0.0083(6)	0.0127(6)	0.0110(5)	0.0002(4)	0.0006(4)	−0.0016(4)
O(3)	8c	0.1210(1)	0.79233(9)	0.23633(6)	0.0103(6)	0.0123(5)	0.0140(5)	0.0021(4)	0.0011(4)	0.0023(4)
O(4)	8c	−0.1467(1)	0.5192(1)	0.39792(7)	0.0113(6)	0.0182(6)	0.0183(6)	−0.0025(4)	0.0004(5)	0.0062(5)
O(5)	8c	−0.1146(1)	0.77911(9)	0.36418(6)	0.0132(6)	0.0109(6)	0.0105(6)	0.0004(4)	0.0025(4)	−0.0006(4)
O(6)	8c	0.1469(1)	0.40138(9)	0.34103(7)	0.0132(6)	0.0119(6)	0.0144(6)	0.0020(4)	0.0014(4)	0.0015(5)
C(1)	8c	−0.0666(2)	0.5772(1)	0.1587(1)	0.0090(8)	0.0052(7)	0.0144(8)	−0.0035(6)	0.0000(6)	−0.0008(6)
C(2)	8c	0.4032(2)	0.5955(2)	0.43191(9)	0.0108(9)	0.0196(8)	0.0123(8)	0.0037(6)	−0.0023(6)	0.0019(7)

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