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Diaquabis[4-methyl-2-(4-methylphenylsulfonamido)pentanoato-κO]calcium(II)

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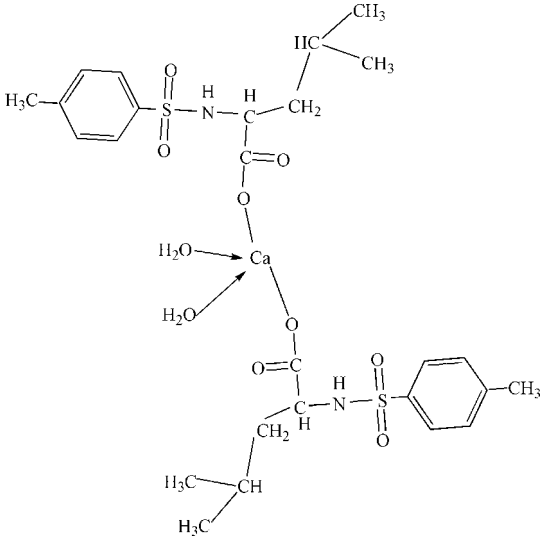
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.007 Å; R factor = 0.051; wR factor = 0.112; data-to-parameter ratio = 15.0.

In the title compound, $[Ca(C_{13}H_{18}NO_4S)_2(H_2O)_2]$, a distorted CaO_4 tetrahedron arises from the coordination of the two ligands and two water molecules. A network of hydrogen bonds helps to establish the crystal packing.

Related literature

For related literature, see: Tai *et al.* (2005).



Experimental

Crystal data

$[Ca(C_{13}H_{18}NO_4S)_2(H_2O)_2]$
M_r = 644.80
Orthorhombic, $P2_12_12_1$
a = 5.1575 (11) Å
b = 17.430 (2) Å
c = 35.147 (4) Å
V = 3159.5 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.39 mm^{-1}
T = 298 (2) K
0.58 × 0.40 × 0.38 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.808, T_{max} = 0.868
14074 measured reflections
5549 independent reflections
4532 reflections with $I > 2\sigma(I)$
R_{int} = 0.048

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.112
S = 1.02
5549 reflections
370 parameters
90 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Absolute structure: Flack (1983), with 2309 Friedel pairs
Flack parameter: 0.04 (5)

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BV2062).

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Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
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Tai, X.-S., Liu, W.-Y., Liu, Y.-Z. & Li, Y.-Z. (2005). *Acta Cryst.* E**61**, o389–o390.

supplementary materials

Acta Cryst. (2007). E**63**, m1935 [**doi:10.1107/S1600536807029327**]

Diaquabis[4-methyl-2-(4-methylphenylsulfonamido)pentanoato-*κO*]calcium(II)

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Comment

As part of our ongoing studies of metal coordination complexes with multidentate ligands (Tai *et al.*, 2005), the synthesis and structure of the title compound, (I), is reported.

Two O-monodentate ligands and two water molecules are attached to the calcium atom, resulting in a distorted CaO_4 tetrahedron (Fig. 1). The identical S2=O8 [1.437 (3) Å], S2=O7 [1.430 (3) Å], C14=O6 [1.243 (5) Å] and S1=O3 [1.427 (3) Å], S1=O4 [1.423 (3) Å], C1=O2 [1.254 (5) Å] bonds lengths imply double bond character. The dihedral angle between the two benzene ring mean planes (C7—C12 and C20—C25) is 129.9 (3) °.

Two molecules of water complete the structure of (I) and a network of hydrogen bonds helps to establish the crystal packing.

Experimental

1 mmol of calcium perchlorate was added to a solution of 4-toluenesulfonyl chloride-*L*-leucine (2 mmol) in 10 ml of CH_3OH/H_2O (*v*/*v* 1:1). The mixture was continuously stirred for 4 h at refluxing temperature, evaporating some methanol, then, upon cooling, the solid product was collected by filtration and dried *in vacuo* (yield 69%). Clear blocks of (I) were obtained by evaporation from a methanol solution after a week.

Refinement

The water H atoms were located in a difference map and refined as riding in their as-found relative positions with $U_{iso}(H) = 1.2U_{eq}(O)$. Other H atoms were placed geometrically (C—H = 0.93–0.97 Å, O—H = 0.82 Å, N—H = 0.86 Å) and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C,N)$ or $1.5U_{eq}(O)$.

Figures

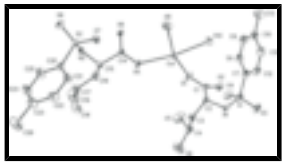


Fig. 1. The complex molecule in (I) with 50% probabiility ellipsoids (arbitrary spheres for the H atoms).

Diaquabis[4-methyl-2-(4-methylphenylsulfonamido)pentanoato-κ*O*]calcium(II)

Crystal data

$[Ca(C_{13}H_{18}NO_4S)_2(H_2O)_2]$ $D_x = 1.356$ Mg m^{-3}

M_r = 644.80	Mo $K\alpha$ radiation λ = 0.71073 Å
Orthorhombic, $P2_12_12_1$	Cell parameters from 4003 reflections
a = 5.1575 (11) Å	θ = 2.3–23.3º
b = 17.430 (2) Å	μ = 0.39 mm^{-1}
c = 35.147 (4) Å	T = 298 (2) K
V = 3159.5 (8) $Å^3$	Colourless, block
Z = 4	0.58 × 0.40 × 0.38 mm
F_{000} = 1368	

Data collection

Bruker CCD area-detector diffractometer	5549 independent reflections
Radiation source: fine-focus sealed tube	4532 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.048
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 1.3º
Absorption correction: multi-scan (SADABS; Bruker, 1997)	$h = -6 \rightarrow 6$
T_{min} = 0.808, T_{max} = 0.868	$k = -20 \rightarrow 14$
14074 measured reflections	$l = -39 \rightarrow 41$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.051	$w = 1/[\sigma^2(F_o^2) + (0.0424P)^2 + 1.9751P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.112	$(\Delta/\sigma)_{max} < 0.001$
S = 1.02	$\Delta\rho_{max}$ = 0.26 e $Å^{-3}$
5549 reflections	$\Delta\rho_{min}$ = −0.28 e $Å^{-3}$
370 parameters	Extinction correction: none
90 restraints	Absolute structure: Flack (1983)
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.04 (5)
Secondary atom site location: difference Fourier map	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-

factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ca1	0.85026 (15)	0.64157 (4)	0.06135 (2)	0.02487 (19)
N1	1.6802 (6)	0.46340 (18)	0.13168 (10)	0.0321 (8)
H1	1.7678	0.4925	0.1149	0.039*
N2	0.0086 (6)	0.84341 (17)	0.11043 (9)	0.0298 (8)
H2	−0.0864	0.8062	0.0994	0.036*
O1	1.0994 (5)	0.56081 (16)	0.09914 (8)	0.0379 (8)
O2	1.5023 (6)	0.56391 (15)	0.07629 (8)	0.0369 (7)
O3	1.9911 (6)	0.36387 (18)	0.11820 (10)	0.0559 (9)
O4	1.5574 (6)	0.32834 (17)	0.14181 (10)	0.0549 (10)
O5	0.5959 (5)	0.73580 (15)	0.09304 (8)	0.0341 (7)
O6	0.2036 (5)	0.72545 (15)	0.06731 (8)	0.0369 (7)
O7	0.1982 (6)	0.91204 (16)	0.05345 (7)	0.0386 (7)
O8	−0.2625 (5)	0.92264 (17)	0.06999 (8)	0.0451 (8)
O9	0.6424 (8)	0.69671 (18)	0.00867 (8)	0.0613 (10)
H9A	0.4981	0.7103	0.0182	0.074*
H9B	0.6140	0.6662	−0.0097	0.074*
O10	1.0908 (6)	0.57833 (18)	0.01260 (9)	0.0545 (9)
H10A	1.0654	0.5833	−0.0112	0.065*
H10B	1.2449	0.5779	0.0211	0.065*
S1	1.7171 (2)	0.37528 (6)	0.11838 (4)	0.0408 (3)
S2	0.0025 (2)	0.91630 (6)	0.08220 (3)	0.0324 (3)
C1	1.3381 (8)	0.5440 (2)	0.10069 (11)	0.0292 (9)
C2	1.4202 (8)	0.4970 (2)	0.13530 (11)	0.0312 (10)
H2A	1.2950	0.4553	0.1389	0.037*
C3	1.4144 (10)	0.5481 (3)	0.17045 (13)	0.0488 (13)
H3A	1.5502	0.5863	0.1681	0.059*
H3B	1.2499	0.5751	0.1710	0.059*
C4	1.4492 (10)	0.5064 (3)	0.20805 (13)	0.0532 (13)
H4	1.6039	0.4740	0.2058	0.064*
C5	1.2227 (13)	0.4545 (5)	0.21638 (18)	0.106 (2)
H5A	1.0701	0.4849	0.2205	0.159*
H5B	1.2586	0.4248	0.2388	0.159*
H5C	1.1949	0.4207	0.1952	0.159*
C6	1.4963 (17)	0.5625 (4)	0.24014 (17)	0.100 (2)
H6A	1.3482	0.5954	0.2429	0.150*
H6B	1.6467	0.5929	0.2345	0.150*
H6C	1.5237	0.5348	0.2634	0.150*
C7	1.6044 (9)	0.3669 (2)	0.07123 (13)	0.0397 (11)
C8	1.3987 (9)	0.3197 (2)	0.06278 (15)	0.0471 (12)
H8	1.3225	0.2899	0.0817	0.057*
C9	1.3070 (11)	0.3169 (3)	0.02595 (15)	0.0544 (14)
H9	1.1670	0.2852	0.0205	0.065*

C10	1.4153 (9)	0.3596 (3)	−0.00300 (14)	0.0487 (12)
C11	1.6259 (11)	0.4066 (3)	0.00626 (15)	0.0527 (13)
H11	1.7040	0.4360	−0.0126	0.063*
C12	1.7187 (9)	0.4097 (2)	0.04283 (14)	0.0459 (12)
H12	1.8597	0.4409	0.0485	0.055*
C13	1.3128 (12)	0.3565 (3)	−0.04291 (15)	0.0747 (17)
H13A	1.1532	0.3282	−0.0433	0.112*
H13B	1.4372	0.3316	−0.0591	0.112*
H13C	1.2823	0.4077	−0.0519	0.112*
C14	0.3589 (8)	0.7525 (2)	0.09079 (11)	0.0287 (9)
C15	0.2574 (8)	0.8097 (2)	0.12076 (11)	0.0311 (10)
H15	0.3851	0.8507	0.1241	0.037*
C16	0.2253 (10)	0.7668 (3)	0.15842 (13)	0.0485 (12)
H16A	0.0999	0.7259	0.1547	0.058*
H16B	0.3896	0.7430	0.1648	0.058*
C17	0.1382 (12)	0.8154 (3)	0.19202 (13)	0.0525 (12)
H17	−0.0259	0.8400	0.1850	0.063*
C18	0.3295 (13)	0.8783 (4)	0.20161 (17)	0.090 (2)
H18A	0.3598	0.9093	0.1795	0.135*
H18B	0.4898	0.8558	0.2098	0.135*
H18C	0.2601	0.9097	0.2216	0.135*
C19	0.0868 (19)	0.7650 (4)	0.22628 (16)	0.110 (3)
H19A	0.2438	0.7392	0.2335	0.166*
H19B	−0.0435	0.7277	0.2200	0.166*
H19C	0.0275	0.7961	0.2471	0.166*
C20	0.0724 (8)	0.9945 (2)	0.11172 (12)	0.0324 (10)
C21	0.2778 (9)	1.0421 (2)	0.10399 (13)	0.0413 (11)
H21	0.3907	1.0310	0.0841	0.050*
C22	0.3151 (11)	1.1071 (2)	0.12623 (13)	0.0494 (13)
H22	0.4537	1.1395	0.1210	0.059*
C23	0.1494 (11)	1.1245 (2)	0.15617 (13)	0.0506 (13)
C24	−0.0499 (11)	1.0745 (3)	0.16340 (14)	0.0567 (15)
H24	−0.1618	1.0846	0.1835	0.068*
C25	−0.0886 (10)	1.0103 (3)	0.14186 (13)	0.0492 (13)
H25	−0.2244	0.9772	0.1476	0.059*
C26	0.1876 (14)	1.1967 (3)	0.17961 (16)	0.081 (2)
H26A	0.1332	1.2405	0.1651	0.121*
H26B	0.3675	1.2019	0.1861	0.121*
H26C	0.0862	1.1933	0.2025	0.121*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ca1	0.0183 (4)	0.0281 (4)	0.0282 (4)	−0.0003 (4)	−0.0001 (4)	0.0012 (3)
N1	0.0190 (19)	0.0360 (18)	0.041 (2)	0.0032 (16)	−0.0011 (17)	0.0109 (15)
N2	0.0227 (18)	0.0241 (17)	0.043 (2)	0.0018 (15)	−0.0004 (18)	−0.0088 (14)
O1	0.0176 (16)	0.0478 (18)	0.0483 (19)	0.0029 (14)	−0.0024 (13)	0.0210 (14)
O2	0.0287 (16)	0.0385 (17)	0.0434 (17)	−0.0011 (14)	0.0052 (15)	0.0130 (13)

O3	0.0298 (17)	0.0490 (19)	0.089 (3)	0.0144 (17)	−0.0061 (19)	0.0041 (19)
O4	0.049 (2)	0.0426 (18)	0.073 (2)	−0.0043 (16)	−0.0045 (19)	0.0275 (17)
O5	0.0152 (16)	0.0380 (16)	0.0493 (18)	0.0023 (13)	−0.0013 (13)	−0.0079 (13)
O6	0.0211 (16)	0.0387 (16)	0.0509 (19)	0.0002 (13)	−0.0084 (14)	−0.0155 (14)
O7	0.0358 (18)	0.0503 (17)	0.0296 (15)	−0.0021 (15)	0.0089 (14)	−0.0053 (13)
O8	0.0283 (17)	0.0517 (18)	0.055 (2)	0.0098 (15)	−0.0129 (15)	−0.0051 (16)
O9	0.076 (3)	0.075 (2)	0.0337 (18)	0.022 (2)	−0.0120 (19)	0.0049 (16)
O10	0.044 (2)	0.077 (2)	0.0420 (18)	0.0081 (19)	0.0030 (16)	−0.0154 (17)
S1	0.0280 (6)	0.0339 (6)	0.0606 (8)	0.0043 (5)	−0.0029 (6)	0.0105 (5)
S2	0.0246 (5)	0.0365 (6)	0.0362 (6)	0.0038 (5)	−0.0019 (5)	−0.0051 (5)
C1	0.023 (2)	0.027 (2)	0.037 (2)	−0.003 (2)	−0.003 (2)	0.0047 (17)
C2	0.022 (2)	0.037 (2)	0.035 (2)	0.0023 (19)	0.0007 (19)	0.0125 (19)
C3	0.045 (3)	0.054 (3)	0.048 (3)	0.017 (2)	−0.002 (2)	0.006 (2)
C4	0.045 (3)	0.073 (3)	0.041 (3)	0.013 (3)	−0.004 (2)	0.008 (2)
C5	0.077 (5)	0.171 (6)	0.070 (4)	−0.023 (5)	−0.002 (4)	0.050 (4)
C6	0.130 (6)	0.111 (5)	0.059 (4)	0.029 (5)	−0.010 (4)	−0.013 (4)
C7	0.033 (2)	0.030 (2)	0.056 (3)	0.004 (2)	0.005 (2)	0.003 (2)
C8	0.044 (3)	0.040 (3)	0.058 (3)	0.001 (2)	0.010 (3)	0.002 (2)
C9	0.047 (4)	0.044 (3)	0.073 (4)	−0.008 (3)	0.000 (3)	−0.007 (3)
C10	0.045 (3)	0.044 (3)	0.058 (3)	0.003 (3)	−0.007 (3)	−0.002 (3)
C11	0.052 (3)	0.045 (3)	0.061 (3)	−0.003 (3)	0.008 (3)	0.009 (2)
C12	0.037 (3)	0.036 (2)	0.065 (3)	−0.007 (2)	−0.002 (3)	0.006 (2)
C13	0.079 (5)	0.075 (4)	0.069 (4)	−0.007 (4)	−0.010 (3)	−0.004 (3)
C14	0.021 (2)	0.026 (2)	0.039 (2)	−0.0007 (19)	0.002 (2)	0.0000 (18)
C15	0.030 (2)	0.029 (2)	0.034 (2)	0.0027 (18)	−0.002 (2)	−0.0069 (18)
C16	0.051 (3)	0.047 (3)	0.047 (3)	0.012 (2)	0.006 (2)	0.004 (2)
C17	0.058 (3)	0.058 (3)	0.041 (3)	0.010 (3)	0.011 (3)	0.002 (2)
C18	0.096 (5)	0.104 (4)	0.070 (4)	0.007 (4)	−0.001 (4)	−0.037 (3)
C19	0.170 (7)	0.101 (5)	0.060 (4)	0.024 (5)	0.042 (4)	0.023 (3)
C20	0.026 (2)	0.032 (2)	0.039 (2)	0.0051 (19)	−0.002 (2)	0.0017 (19)
C21	0.039 (3)	0.040 (2)	0.044 (3)	−0.004 (2)	0.007 (2)	−0.001 (2)
C22	0.057 (3)	0.041 (3)	0.050 (3)	−0.015 (3)	−0.001 (3)	0.001 (2)
C23	0.066 (4)	0.034 (2)	0.052 (3)	−0.008 (3)	0.005 (3)	−0.002 (2)
C24	0.069 (4)	0.052 (3)	0.049 (3)	−0.010 (3)	0.022 (3)	−0.013 (3)
C25	0.051 (3)	0.043 (3)	0.054 (3)	−0.010 (2)	0.023 (3)	−0.005 (2)
C26	0.125 (6)	0.045 (3)	0.072 (4)	−0.007 (4)	0.012 (4)	−0.016 (3)

Geometric parameters (Å, °)

Ca1—O2^{i}	2.308 (3)	C6—H6C	0.9600
Ca1—O1	2.323 (3)	C7—C8	1.375 (6)
Ca1—O9	2.345 (3)	C7—C12	1.378 (6)
Ca1—O6^{ii}	2.346 (3)	C8—C9	1.379 (7)
Ca1—O5	2.379 (3)	C8—H8	0.9300
Ca1—O10	2.385 (3)	C9—C10	1.378 (7)
Ca1—H9A	2.6516	C9—H9	0.9300
Ca1—H10B	2.7147	C10—C11	1.399 (7)
N1—C2	1.469 (5)	C10—C13	1.500 (7)
N1—S1	1.617 (3)	C11—C12	1.372 (7)

N1—H1	0.9000	C11—H11	0.9300
N2—C15	1.458 (5)	C12—H12	0.9300
N2—S2	1.612 (3)	C13—H13A	0.9600
N2—H2	0.9000	C13—H13B	0.9600
O1—C1	1.267 (5)	C13—H13C	0.9600
O2—C1	1.254 (5)	C14—C15	1.542 (5)
O2—Ca1^{ii}	2.308 (3)	C15—C16	1.529 (6)
O3—S1	1.427 (3)	C15—H15	0.9800
O4—S1	1.423 (3)	C16—C17	1.521 (6)
O5—C14	1.259 (5)	C16—H16A	0.9700
O6—C14	1.243 (5)	C16—H16B	0.9700
O6—Ca1^{i}	2.346 (3)	C17—C18	1.513 (8)
O7—S2	1.430 (3)	C17—C19	1.514 (7)
O8—S2	1.437 (3)	C17—H17	0.9800
O9—H9A	0.8500	C18—H18A	0.9600
O9—H9B	0.8499	C18—H18B	0.9600
O10—H10A	0.8498	C18—H18C	0.9600
O10—H10B	0.8498	C19—H19A	0.9600
S1—C7	1.762 (5)	C19—H19B	0.9600
S2—C20	1.750 (4)	C19—H19C	0.9600
C1—C2	1.526 (5)	C20—C21	1.373 (6)
C2—C3	1.523 (6)	C20—C25	1.374 (6)
C2—H2A	0.9800	C21—C22	1.389 (6)
C3—C4	1.519 (6)	C21—H21	0.9300
C3—H3A	0.9700	C22—C23	1.389 (7)
C3—H3B	0.9700	C22—H22	0.9300
C4—C5	1.506 (8)	C23—C24	1.371 (7)
C4—C6	1.512 (8)	C23—C26	1.516 (6)
C4—H4	0.9800	C24—C25	1.366 (6)
C5—H5A	0.9600	C24—H24	0.9300
C5—H5B	0.9600	C25—H25	0.9300
C5—H5C	0.9600	C26—H26A	0.9600
C6—H6A	0.9600	C26—H26B	0.9600
C6—H6B	0.9600	C26—H26C	0.9600
O2^{i}—Ca1—O1	86.82 (10)	H6A—C6—H6B	109.5
O2^{i}—Ca1—O9	93.70 (12)	C4—C6—H6C	109.5
O1—Ca1—O9	162.26 (12)	H6A—C6—H6C	109.5
O2^{i}—Ca1—O6^{ii}	161.64 (11)	H6B—C6—H6C	109.5
O1—Ca1—O6^{ii}	84.08 (10)	C8—C7—C12	119.8 (4)
O9—Ca1—O6^{ii}	99.80 (12)	C8—C7—S1	120.5 (4)
O2^{i}—Ca1—O5	82.51 (10)	C12—C7—S1	119.7 (4)
O1—Ca1—O5	117.11 (11)	C7—C8—C9	119.2 (5)
O9—Ca1—O5	80.48 (11)	C7—C8—H8	120.4
O6^{ii}—Ca1—O5	87.49 (9)	C9—C8—H8	120.4
O2^{i}—Ca1—O10	107.26 (11)	C10—C9—C8	122.4 (5)
O1—Ca1—O10	80.97 (11)	C10—C9—H9	118.8
O9—Ca1—O10	81.95 (12)	C8—C9—H9	118.8

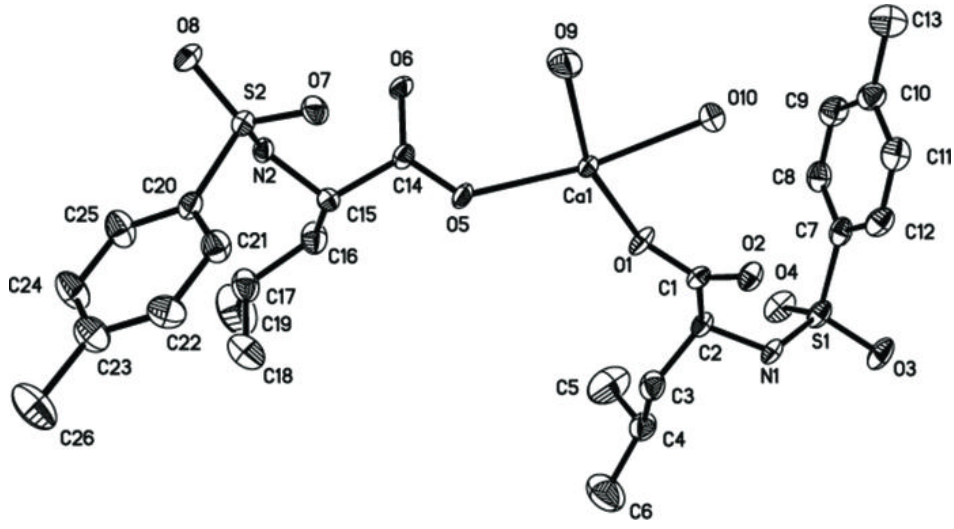
O6^{ii}—Ca1—O10	87.03 (11)	C9—C10—C11	117.3 (5)
O5—Ca1—O10	160.42 (11)	C9—C10—C13	121.9 (5)
O2^{i}—Ca1—H9A	82.1	C11—C10—C13	120.8 (5)
O1—Ca1—H9A	168.4	C12—C11—C10	120.8 (5)
O9—Ca1—H9A	18.3	C12—C11—H11	119.6
O6^{ii}—Ca1—H9A	107.6	C10—C11—H11	119.6
O5—Ca1—H9A	65.0	C11—C12—C7	120.5 (4)
O10—Ca1—H9A	98.9	C11—C12—H12	119.7
O2^{i}—Ca1—H10B	117.5	C7—C12—H12	119.7
O1—Ca1—H10B	68.6	C10—C13—H13A	109.5
O9—Ca1—H10B	95.7	C10—C13—H13B	109.5
O6^{ii}—Ca1—H10B	73.7	H13A—C13—H13B	109.5
O5—Ca1—H10B	159.9	C10—C13—H13C	109.5
O10—Ca1—H10B	17.7	H13A—C13—H13C	109.5
H9A—Ca1—H10B	113.6	H13B—C13—H13C	109.5
C2—N1—S1	120.7 (3)	O6—C14—O5	125.4 (4)
C2—N1—H1	106.9	O6—C14—C15	118.7 (4)
S1—N1—H1	106.7	O5—C14—C15	115.8 (4)
C15—N2—S2	119.2 (3)	N2—C15—C16	108.5 (3)
C15—N2—H2	107.2	N2—C15—C14	112.9 (3)
S2—N2—H2	107.1	C16—C15—C14	108.2 (3)
C1—O1—Ca1	134.6 (3)	N2—C15—H15	109.1
C1—O2—Ca1^{ii}	147.5 (3)	C16—C15—H15	109.1
C14—O5—Ca1	131.7 (3)	C14—C15—H15	109.1
C14—O6—Ca1^{i}	142.8 (3)	C17—C16—C15	115.6 (4)
Ca1—O9—H9A	101.7	C17—C16—H16A	108.4
Ca1—O9—H9B	115.1	C15—C16—H16A	108.4
H9A—O9—H9B	108.9	C17—C16—H16B	108.4
Ca1—O10—H10A	125.3	C15—C16—H16B	108.4
Ca1—O10—H10B	103.7	H16A—C16—H16B	107.5
H10A—O10—H10B	119.5	C18—C17—C19	110.9 (5)
O4—S1—O3	119.7 (2)	C18—C17—C16	112.6 (4)
O4—S1—N1	108.1 (2)	C19—C17—C16	110.2 (4)
O3—S1—N1	104.49 (19)	C18—C17—H17	107.6
O4—S1—C7	107.8 (2)	C19—C17—H17	107.6
O3—S1—C7	108.1 (2)	C16—C17—H17	107.6
N1—S1—C7	108.15 (18)	C17—C18—H18A	109.5
O7—S2—O8	117.66 (18)	C17—C18—H18B	109.5
O7—S2—N2	112.35 (18)	H18A—C18—H18B	109.5
O8—S2—N2	105.22 (19)	C17—C18—H18C	109.5
O7—S2—C20	108.30 (19)	H18A—C18—H18C	109.5
O8—S2—C20	108.24 (19)	H18B—C18—H18C	109.5
N2—S2—C20	104.17 (18)	C17—C19—H19A	109.5
O2—C1—O1	124.2 (4)	C17—C19—H19B	109.5
O2—C1—C2	120.4 (4)	H19A—C19—H19B	109.5
O1—C1—C2	115.3 (4)	C17—C19—H19C	109.5
N1—C2—C3	108.7 (3)	H19A—C19—H19C	109.5

N1—C2—C1	113.5 (3)	H19B—C19—H19C	109.5
C3—C2—C1	109.1 (3)	C21—C20—C25	119.8 (4)
N1—C2—H2A	108.5	C21—C20—S2	120.8 (3)
C3—C2—H2A	108.5	C25—C20—S2	119.3 (3)
C1—C2—H2A	108.5	C20—C21—C22	119.2 (4)
C4—C3—C2	115.0 (4)	C20—C21—H21	120.4
C4—C3—H3A	108.5	C22—C21—H21	120.4
C2—C3—H3A	108.5	C21—C22—C23	121.3 (5)
C4—C3—H3B	108.5	C21—C22—H22	119.3
C2—C3—H3B	108.5	C23—C22—H22	119.3
H3A—C3—H3B	107.5	C24—C23—C22	117.5 (4)
C5—C4—C6	111.6 (5)	C24—C23—C26	121.6 (5)
C5—C4—C3	111.4 (4)	C22—C23—C26	120.9 (5)
C6—C4—C3	111.0 (5)	C25—C24—C23	121.8 (5)
C5—C4—H4	107.6	C25—C24—H24	119.1
C6—C4—H4	107.6	C23—C24—H24	119.1
C3—C4—H4	107.6	C24—C25—C20	120.2 (4)
C4—C5—H5A	109.5	C24—C25—H25	119.9
C4—C5—H5B	109.5	C20—C25—H25	119.9
H5A—C5—H5B	109.5	C23—C26—H26A	109.5
C4—C5—H5C	109.5	C23—C26—H26B	109.5
H5A—C5—H5C	109.5	H26A—C26—H26B	109.5
H5B—C5—H5C	109.5	C23—C26—H26C	109.5
C4—C6—H6A	109.5	H26A—C26—H26C	109.5
C4—C6—H6B	109.5	H26B—C26—H26C	109.5
$O2^{i}$—Ca1—O1—C1	155.9 (4)	C7—C8—C9—C10	0.5 (7)
O9—Ca1—O1—C1	63.7 (6)	C8—C9—C10—C11	0.2 (7)
$O6^{ii}$—Ca1—O1—C1	−40.1 (4)	C8—C9—C10—C13	−179.6 (5)
O5—Ca1—O1—C1	−124.2 (4)	C9—C10—C11—C12	−0.2 (7)
O10—Ca1—O1—C1	47.9 (4)	C13—C10—C11—C12	179.6 (5)
$O2^{i}$—Ca1—O5—C14	−45.5 (4)	C10—C11—C12—C7	−0.5 (7)
O1—Ca1—O5—C14	−128.1 (3)	C8—C7—C12—C11	1.2 (7)
O9—Ca1—O5—C14	49.5 (4)	S1—C7—C12—C11	−177.0 (4)
$O6^{ii}$—Ca1—O5—C14	149.9 (4)	$Ca1^{i}$—O6—C14—O5	108.2 (5)
O10—Ca1—O5—C14	76.0 (5)	$Ca1^{i}$—O6—C14—C15	−69.9 (5)
C2—N1—S1—O4	−51.5 (4)	Ca1—O5—C14—O6	−7.3 (6)
C2—N1—S1—O3	179.9 (3)	Ca1—O5—C14—C15	170.9 (2)
C2—N1—S1—C7	64.9 (4)	S2—N2—C15—C16	156.8 (3)
C15—N2—S2—O7	36.1 (3)	S2—N2—C15—C14	−83.2 (4)
C15—N2—S2—O8	165.3 (3)	O6—C14—C15—N2	−18.4 (5)
C15—N2—S2—C20	−80.9 (3)	O5—C14—C15—N2	163.3 (3)
$Ca1^{ii}$—O2—C1—O1	107.7 (5)	O6—C14—C15—C16	101.7 (4)
$Ca1^{ii}$—O2—C1—C2	−71.6 (6)	O5—C14—C15—C16	−76.6 (5)
Ca1—O1—C1—O2	−9.3 (7)	N2—C15—C16—C17	−60.0 (5)
Ca1—O1—C1—C2	170.0 (3)	C14—C15—C16—C17	177.2 (4)
S1—N1—C2—C3	140.5 (3)	C15—C16—C17—C18	−61.5 (6)
S1—N1—C2—C1	−97.9 (4)	C15—C16—C17—C19	174.1 (5)

O2—C1—C2—N1	−14.3 (5)	O7—S2—C20—C21	3.6 (4)
O1—C1—C2—N1	166.3 (3)	O8—S2—C20—C21	−125.0 (4)
O2—C1—C2—C3	107.1 (4)	N2—S2—C20—C21	123.4 (4)
O1—C1—C2—C3	−72.3 (5)	O7—S2—C20—C25	−179.7 (3)
N1—C2—C3—C4	−65.2 (5)	O8—S2—C20—C25	51.7 (4)
C1—C2—C3—C4	170.6 (4)	N2—S2—C20—C25	−59.9 (4)
C2—C3—C4—C5	−66.8 (6)	C25—C20—C21—C22	−1.9 (7)
C2—C3—C4—C6	168.2 (5)	S2—C20—C21—C22	174.9 (3)
O4—S1—C7—C8	−1.2 (4)	C20—C21—C22—C23	0.3 (7)
O3—S1—C7—C8	129.5 (4)	C21—C22—C23—C24	1.1 (7)
N1—S1—C7—C8	−117.9 (4)	C21—C22—C23—C26	−178.4 (5)
O4—S1—C7—C12	177.0 (3)	C22—C23—C24—C25	−0.9 (8)
O3—S1—C7—C12	−52.3 (4)	C26—C23—C24—C25	178.6 (5)
N1—S1—C7—C12	60.3 (4)	C23—C24—C25—C20	−0.7 (8)
C12—C7—C8—C9	−1.2 (7)	C21—C20—C25—C24	2.1 (7)
S1—C7—C8—C9	177.0 (4)	S2—C20—C25—C24	−174.7 (4)

Symmetry codes: (i) *x*−1, *y*, *z*; (ii) *x*+1, *y*, *z*.

Fig. 1


O8
S2
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O5
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C17
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C22
C2
S1
N1
O3
C23
C18
C5
C3
C4
C26
C6