

Crystal structure of (1,4,7,10,13,16-hexaoxacyclooctadecane- κ^6O)potassium- μ -oxalato-triphenylstannate(IV), the first reported 18-crown-6-stabilized potassium salt of triphenyloxalatostannate

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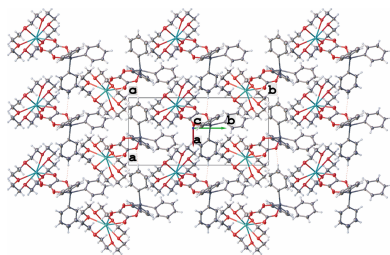
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The title complex, (1,4,7,10,13,16-hexaoxacyclooctadecane- $1\kappa^6O$)(μ -oxalato- $1\kappa^2O^1, O^2; 2\kappa^2O^1, O^2$)triphenyl- $2\kappa^3C$ -potassium(I)tin(IV), $[\text{KSn}(\text{C}_6\text{H}_5)_3(\text{C}_2\text{O}_4)(\text{C}_{12}\text{H}_{24}\text{O}_6)]$ or $\text{K}[18\text{-Crown-6}][(\text{C}_6\text{H}_5)_3\text{SnO}_4\text{C}_2]$, was synthesized. The complex consists of a potassium cation coordinated to the six oxygen atoms of a crown ether molecule and the two oxygen atoms of the oxalotriphenylstannate anion. It crystallizes in the monoclinic crystal system within the space group $P2_1$. The tin atom is coordinated by one chelating oxalate ligand and three phenyl groups, forming a *cis*-trigonal-bipyramidal geometry around the tin atom. The cations and anions form ion pairs, linked through carbonyl coordination to the potassium atoms. The crystal structure features $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds between the oxygen atoms of the oxalate group and the hydrogen atoms of the phenyl groups, resulting in an infinite chain structure extending along *a*-axis direction. The primary inter-chain interactions are van der Waals forces.

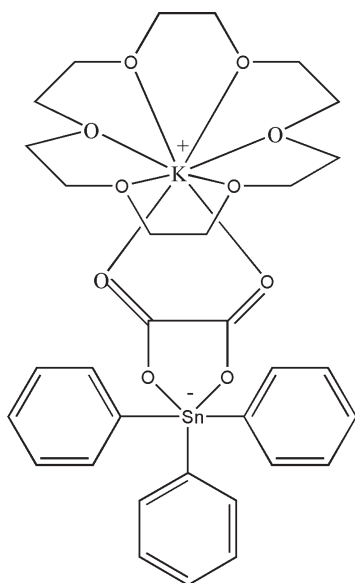
1. Chemical context

Organotin carboxylates are one of the most significant classes of compounds, valued not only for their theoretical and structural properties but also for their industrial and agricultural applications (Zuckermann *et al.*, 1976). Organotin(IV) carboxylates are particularly notable for their diverse and important biological activities, serving as anticancer, antiviral, antibacterial, and antifungal agents, as well as wood preservatives and pesticides (Davies & Smith, 1980; Smith *et al.*, 1978; Thayer *et al.*, 1984; Blunden *et al.*, 1985; Evans & Karpel, 1985; Angham *et al.*, 2019; Talebi *et al.*, 2023). Metal complexes of dicarboxylic acids, such as oxalic acid, have garnered significant interest due to their promising magnetic and electrochemical properties. The appeal of oxalate-based coordination compounds lies in their high structural diversity, attributed to the oxalate ligand's ability to adopt 17 different coordination modes and function as a mono-, bi-, tri-, or tetradentate ligand (Krishnamurty & Harris, 1961; Rao *et al.*, 2004). This results in a vast, yet largely unexplored, compositional area. Notably, there are very few reports of organotin complexes of oxalic acid in the literature.

The author has been interested in designing and preparing ionic organotin complexes to improve aqueous solubility through ionization. Since the pioneering work of Pedersen (Pedersen, 1988; Izatt, 2017), crown ethers and their complexes with metal cations have attracted considerable attention. Their remarkable selectivity on metal cations,



especially alkali and alkaline earth metal cations, is a topic of fundamental interest in both coordination chemistry and biological chemistry (Bajaj *et al.*, 1988; Hay & Rustad, 1994; Lehn *et al.*, 1988; Lee *et al.*, 1996). Literature reports show that crown ethers can be utilized in solid-solid and solid-liquid processes to capture alkali metal and ammonium cations in extended hydrogen-bonded networks formed by inorganic acid anions, such as hydrogen sulfate and di-hydrogen phosphate, as well as organic acid anions (Braga *et al.*, 2005, 2007, 2008, 2009). In this context, we present and discuss the crystal structure of a crown ether-stabilized potassium salt of oxalato-triphenylstannate, **1**.



2. Structural commentary

The stannate anionic unit of the title compound **1** features a *cis*-tbp $[\text{Ph}_3\text{Snox}]^-$ anion, with Sn1–O1 measuring 2.071 (5) Å and Sn1–O2 measuring 2.290 (6) Å, and an O1–Sn1–O2 bond angle of 73.4 (2)° (Fig. 1). This anion is coordinated *via* its two oxalate carbonyl groups (O3 and O4) to a K[18-crown-6] cation, with K1–O3 at 2.785 (7) Å, K1–O4 at 2.654 (6) Å, and an O3–K1–O4 bond angle of 61.3 (2)° (Fig. 1). The oxalate acts as a bidentate ligand to both tin and potassium, forming two five-membered chelate rings that are coplanar, with a dihedral angle of approximately 0°.

In the $[\text{Ph}_3\text{Snox}]^-$ portion, the axial Sn–O bond [Sn1–O2 at 2.290 (6) Å] is significantly longer than the equatorial Sn–O bond [Sn1–O1 at 2.071 (5) Å]. The bite angle of 73.4 (2)° is similar to those found in other chelated oxalato triphenylstannates (Ng *et al.*, 1992; Ng & Kumar Das, 1993; Ng, 1996). The axial Sn–C bond [Sn1–C7 at 2.188 (7) Å] is somewhat longer than the equatorial Sn–C bonds [Sn1–C1 at 2.136 (7) Å and Sn1–C13 at 2.138 (7) Å]. The axial structure is notably bent, with an O2–Sn1–C7 angle of 160.8 (3)°, and the Sn atom is displaced out of the equatorial plane [Σ angles at Sn = 354.9 (3)°] towards the axial C7 atom by 0.119 Å.

The oxalate group in the $[\text{Ph}_3\text{Snox}]^-$ ion consists of two similar carboxylate ($-\text{COO}^-$) entities. Both bind to the Sn1

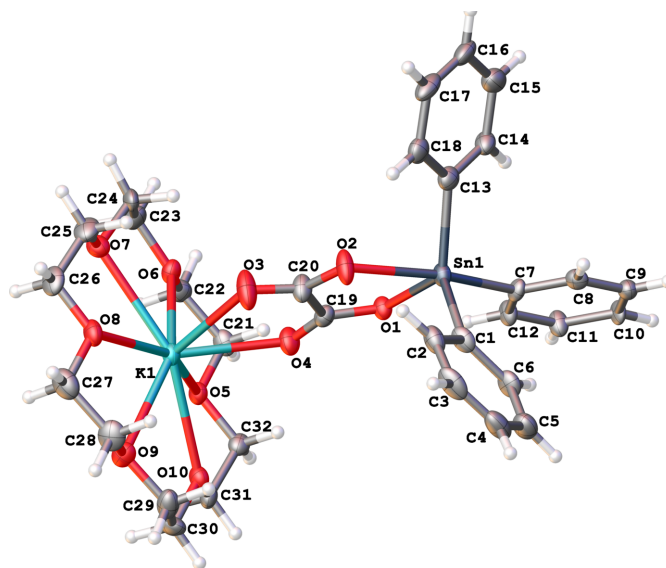


Figure 1

The asymmetric unit and molecular structure of crystal $[(18\text{-crown-6})\text{K}][\text{SnPh}_3(\text{ox})]$ (**1**) with anisotropic displacement ellipsoids set to the 50% probability level.

and K1 atoms, with slightly different C–O bond lengths: those bonded to tin [C–O = 1.266 (11) and 1.303 (10) Å] are slightly longer than those bonded to potassium [C–O = 1.233 (11) and 1.202 (10) Å]. The two negative charges appear to be delocalized over the four oxygen atoms in the oxalate group.

In the $[\text{K}(18\text{-crown-6})]^+$ complex cation, the potassium atom deviates by 0.614 Å from the root-mean-square plane of the six oxygen atoms in the 18-crown-6 ligand towards the

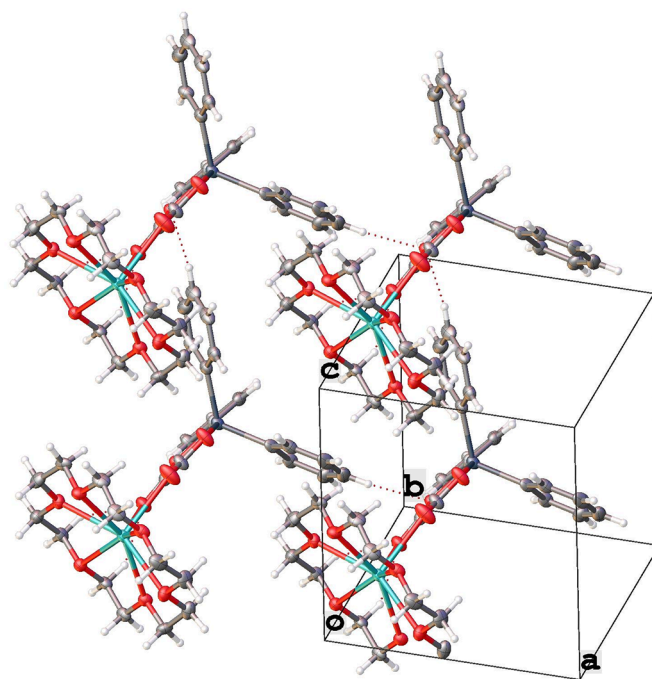


Figure 2

Crystal packing in the crystal structure showing C–H...O hydrogen bonds, denoted by dashed lines, between neighboring molecules.

Table 1
Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C4-H4\cdots O1^i$	0.95	2.42	3.360 (11)	172
$C16-H16\cdots O1^{ii}$	0.95	2.41	3.332 (9)	165

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

oxalate group. This deviation is due to the coordination of two oxygens from the oxalate group. A similar observation has been reported in the literature (Gjikaj *et al.*, 2005; Liebing *et al.*, 2016; Sellin & Malischewski, 2019) when potassium has axial coordination to other heteroatoms. The K–O bond lengths with the 18-crown-6 ligand range from 2.802 (6) to 2.976 (6) Å, which are slightly longer than those reported for other $[K(18\text{-crown-6})]^+$ complexes in the literature. The increased average K–O bond length with 18-crown-6 is attributed to the strong coordination with the oxalate group, where the two K–O bond lengths with the oxalate group are 2.654 (6) Å and 2.785 (7) Å. The coordination number of the K^+ cation is 8. The coordination polyhedron of the potassium cation can be described as a distorted hexagonal pyramid with a bifurcated vertex at the O3 and O4 atoms.

3. Supramolecular features

The title complex **1** exhibits a supramolecular structure that is consolidated by two weak intermolecular hydrogen bonds: $C4-H4\cdots O1$ and $C16-H16\cdots O1$, with $C\cdots O$ distances of 3.360 (11) and 3.332 (9) Å, respectively (Fig. 2; symmetry codes as in Table 1). These intermolecular hydrogen bonds result in the formation of a ‘shoulder-to-shoulder’ arrangement of the complex molecules, creating a supramolecular layer parallel to the (001) plane, as depicted in Figs. 3 and 4.

In supramolecular chemistry, weak hydrogen bonds such as $C-H\cdots\pi$ and $\pi-\pi$ interactions play significant roles in the structural integrity of crystal structures (Meyer *et al.*, 2003; Nishio, 2004). The effectiveness of these interactions is primarily influenced by the distance between the hydrogen

atom of the C–H bond and the plane of the aromatic ring, which should be less than 2.9 Å (the combined van der Waals radii), and the $C-H\cdots\pi$ access angles, ideally ranging from 140 to 180° (Takahashi *et al.*, 2001).

In the crystal under investigation, relatively weak $C-H\cdots\pi$ interactions are observed. The analysis of these interactions reveals $C-H\cdots$ centroid phenyl distances of 2.936 and 2.937 Å (for $H26A\cdots C11$) and distances of 2.937 and 2.949 Å (for $H27B\cdots C13$). The corresponding access angles are 158° for C1 and 162° for C13. Despite the parallel alignment of phenyl groups (C7–C12) along the (101) direction, significant $\pi-\pi$ interactions are absent. This is attributed to a large separation distance of 7.006 Å between the planes, which greatly exceeds the critical distance of 4 Å, and an intercentroid distance of 9.406 Å, surpassing the 6 Å threshold (Ninković *et al.*, 2011).

4. Database survey

A survey of the Cambridge Structural Database (CSD; Groom *et al.*, 2016; Conquest version 2024.1.0, Build 401958; Bruno *et al.*, 2002) reveals thirteen reports of oxalotriphenylstannate compounds with ammonium ions as counter-ions. Examples include di-*iso*-propylammonium (Ng & Hook, 1999), di-*cyclo*-hexylammonium (Ng & Rae, 2000), dibenzylammonium (Gueye *et al.*, 2012), and di-*iso*-butylammonium (Thorpe *et al.*, 2013). A further search for metal salts of triphenylstannate came out with one hit, in which a sodium salt of triphenylstannate named sodium bis[2-(3',6',9'-trioxadecyl)-1,2-dicarba-closododecaboane-1-carboxylato]triphenylstannate was reported (Bregadze *et al.*, 2004). In this reported stannate, the sodium ion is stabilized by coordination to the carbonyl oxygen and five oxygen atoms of trioxadecyl substituents. In contrast, in the title compound **1**, the potassium salt of triphenylstannate is described for the first time, where the potassium ion is primarily stabilized through coordination to 18-crown-6.

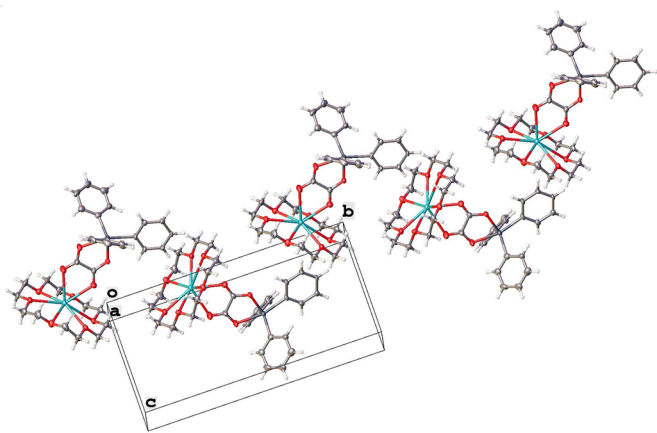


Figure 3
Partial packing plot of **1** along the b axis showing the 1-D chain formed through $C-H\cdots O$ hydrogen bonding.

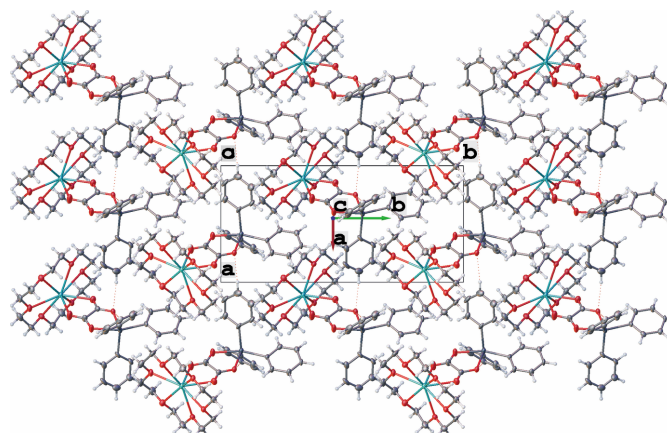


Figure 4
Packing plot of **1** viewed approximately along [001] showing a layer of molecules perpendicular to the c axis.

5. Synthesis and crystallization

The title coordination complex of triphenyltin was synthesized by reacting 1 mmol of oxalic acid, 1 mmol of potassium bicarbonate, 1 mmol of 18-crown-6, and 1 mmol of triphenyltin hydroxide in 30 mL of ethanol. The mixture was refluxed at 373 K with stirring for 1 h. The resulting solution, which was slightly cloudy, was filtered to yield a clear ethanol solution. This filtrate was then allowed to evaporate slowly at 300 K over the course of one week, resulting in colorless crystals suitable for X-ray diffraction analysis.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The H atoms in compound **1** were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C–H distances of 0.93 Å (ring H atoms) and 0.97 Å (methylene H atoms), and N–H distances of 0.98 Å, with $U_{\text{iso}}(\text{H})$ values of $1.2U_{\text{eq}}$ of the parent atoms. Reflections were merged by *SHELXL* according to the crystal class for the calculation of statistics and refinement. The Friedel fraction is defined as the number of unique Friedel pairs measured divided by the number that would be possible theoretically, ignoring centric projections and systematic absences. The crystal studied was refined as a two-component twin. Completeness statistics refer to single and composite reflections containing twin component 1 only.

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Table 2

Experimental details.

Crystal data	
Chemical formula	[KSn(C ₆ H ₅) ₃ (C ₂ O ₄)(C ₁₂ H ₂₄ O ₆)]
M_r	741.42
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	100
a, b, c (Å)	9.4060 (3), 19.3779 (4), 9.4225 (3)
β (°)	97.925 (2)
V (Å ³)	1701.02 (8)
Z	2
Radiation type	Mo $K\alpha$
μ (mm ⁻¹)	0.93
Crystal size (mm)	0.06 × 0.06 × 0.03 × 0.02 (radius)
Data collection	
Diffractometer	Rigaku XtaLAB Synergy-S dual wavelength Mo/Cu
Absorption correction	Multi-scan (<i>CrysAlis PRO</i> ; Rigaku OD, 2022)
$T_{\text{min}}, T_{\text{max}}$	0.913, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	15412, 7113, 6792
R_{int}	0.040
$(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹)	0.649
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.033, 0.105, 1.08
No. of reflections	7113
No. of parameters	398
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³)	1.28, -0.52
Absolute structure	Flack x determined using 2807 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons <i>et al.</i> , 2013)
Absolute structure parameter	-0.08 (3)

Computer programs: *CrysAlis PRO* (Rigaku OD, 2022), *SHELXT* (Sheldrick, 2015a), *SHELXL2018/3* (Sheldrick, 2015b) and *OLEX2* (Dolomanov *et al.*, 2009).

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Computing details

(1,4,7,10,13,16-Hexaoxacyclooctadecane-1 κ^6O)(μ -oxalato-1 κ^2O^1, O^2 :2 κ^2O^1, O^2)triphenyl-2 κ^3C -potassium(I)tin(IV)

Crystal data

[KSn(C₆H₅)₃(C₂O₄)(C₁₂H₂₄O₆)]

$M_r = 741.42$

Monoclinic, $P2_1$

$a = 9.4060$ (3) Å

$b = 19.3779$ (4) Å

$c = 9.4225$ (3) Å

$\beta = 97.925$ (2)°

$V = 1701.02$ (8) Å³

$Z = 2$

$F(000) = 760$

$D_x = 1.448$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 9822 reflections

$\theta = 2.9$ – 31.1 °

$\mu = 0.93$ mm⁻¹

$T = 100$ K

Block, colorless

$0.06 \times 0.06 \times 0.03 \times 0.02$ (radius) mm

Data collection

Rigaku XtaLAB Synergy-S dual wavelength

Mo/Cu

diffractometer

Radiation source: microfocus sealed X-ray tube,

Rigaku PhotonJet-S

Mirror optics monochromator

Detector resolution: 10.0000 pixels mm⁻¹

ω scans

Absorption correction: multi-scan

(CrysAlis PRO; Rigaku OD, 2022)

$T_{\min} = 0.913$, $T_{\max} = 1.000$

15412 measured reflections

7113 independent reflections

6792 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.040$

$\theta_{\max} = 27.5$ °, $\theta_{\min} = 2.9$ °

$h = -12 \rightarrow 11$

$k = -23 \rightarrow 25$

$l = -11 \rightarrow 10$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.033$

$wR(F^2) = 0.105$

$S = 1.08$

7113 reflections

398 parameters

1 restraint

Primary atom site location: structure-invariant

direct methods

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0708P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 1.28$ e Å⁻³

$\Delta\rho_{\min} = -0.52$ e Å⁻³

Absolute structure: Flack x determined using
2807 quotients $[(F^+)-(F^-)]/[(F^+)+(F^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: -0.08 (3)

Special details

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

Refinement. Refined as a 2-component twin.

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Sn1	0.41008 (5)	0.58376 (2)	0.45935 (5)	0.02209 (12)
O1	0.2517 (6)	0.5552 (3)	0.2950 (6)	0.0223 (10)
O2	0.3969 (8)	0.4658 (3)	0.4651 (8)	0.0369 (16)
O3	0.2847 (9)	0.3760 (4)	0.3470 (9)	0.0484 (19)
O4	0.1482 (7)	0.4720 (3)	0.1562 (7)	0.0312 (14)
C1	0.6233 (8)	0.5741 (5)	0.4077 (8)	0.0256 (19)
C2	0.7115 (10)	0.5176 (4)	0.4492 (9)	0.0306 (18)
H2	0.677139	0.480378	0.500564	0.037*
C3	0.8536 (11)	0.5166 (5)	0.4135 (11)	0.039 (2)
H3	0.913985	0.478341	0.441871	0.047*
C4	0.9046 (10)	0.5691 (5)	0.3399 (10)	0.040 (3)
H4	1.000617	0.568146	0.319164	0.048*
C5	0.8163 (11)	0.6238 (5)	0.2954 (12)	0.038 (2)
H5	0.850759	0.659627	0.240293	0.045*
C6	0.6788 (9)	0.6274 (4)	0.3295 (10)	0.0280 (17)
H6	0.620610	0.666331	0.300060	0.034*
C7	0.3592 (8)	0.6911 (4)	0.4013 (9)	0.0211 (15)
C8	0.4450 (8)	0.7424 (4)	0.4748 (8)	0.0237 (15)
H8	0.519593	0.729495	0.548454	0.028*
C9	0.4225 (8)	0.8126 (4)	0.4413 (8)	0.0232 (14)
H9	0.480264	0.846932	0.492962	0.028*
C10	0.3137 (8)	0.8314 (4)	0.3306 (8)	0.0225 (15)
H10	0.298609	0.878641	0.305833	0.027*
C11	0.2278 (9)	0.7806 (4)	0.2571 (9)	0.0265 (16)
H11	0.152693	0.793270	0.183718	0.032*
C12	0.2525 (9)	0.7106 (4)	0.2917 (9)	0.0246 (16)
H12	0.195505	0.676187	0.239377	0.030*
C13	0.3708 (7)	0.5795 (7)	0.6773 (8)	0.0273 (15)
C14	0.2974 (10)	0.6343 (5)	0.7297 (9)	0.0282 (17)
H14	0.262058	0.670815	0.667257	0.034*
C15	0.2754 (11)	0.6360 (5)	0.8732 (11)	0.038 (2)
H15	0.226304	0.673758	0.908593	0.046*
C16	0.3256 (8)	0.5824 (8)	0.9638 (8)	0.0354 (17)
H16	0.310981	0.583366	1.061508	0.042*

C17	0.3963 (10)	0.5280 (6)	0.9124 (10)	0.038 (2)
H17	0.429710	0.491448	0.975628	0.045*
C18	0.4207 (9)	0.5247 (5)	0.7686 (9)	0.0307 (18)
H18	0.469574	0.486627	0.734140	0.037*
C19	0.2277 (8)	0.4908 (4)	0.2599 (9)	0.0227 (15)
C20	0.3101 (10)	0.4379 (4)	0.3672 (10)	0.0287 (16)
K1	0.10867 (17)	0.33895 (8)	0.09631 (17)	0.0219 (3)
O5	-0.0911 (6)	0.4275 (3)	-0.0971 (6)	0.0229 (11)
O6	-0.1562 (6)	0.3828 (3)	0.1682 (6)	0.0248 (12)
O7	-0.0356 (7)	0.2587 (4)	0.2852 (7)	0.0275 (15)
O8	0.2230 (7)	0.2108 (3)	0.2005 (7)	0.0292 (12)
O9	0.2918 (8)	0.2593 (4)	-0.0621 (8)	0.0294 (16)
O10	0.1753 (6)	0.3845 (3)	-0.1714 (6)	0.0250 (12)
C21	-0.1744 (9)	0.4682 (4)	-0.0123 (9)	0.0258 (16)
H21A	-0.241853	0.497889	-0.075143	0.031*
H21B	-0.110542	0.498315	0.053253	0.031*
C22	-0.2558 (11)	0.4209 (6)	0.0720 (11)	0.029 (2)
H22A	-0.319867	0.447871	0.126044	0.035*
H22B	-0.315664	0.389015	0.006764	0.035*
C23	-0.2230 (12)	0.3420 (6)	0.2648 (12)	0.035 (2)
H23A	-0.286849	0.307414	0.211248	0.042*
H23B	-0.281701	0.371518	0.319785	0.042*
C24	-0.1089 (10)	0.3064 (4)	0.3645 (9)	0.0276 (17)
H24A	-0.040179	0.340835	0.411697	0.033*
H24B	-0.152538	0.281731	0.439726	0.033*
C25	0.0662 (11)	0.2172 (5)	0.3752 (10)	0.035 (2)
H25A	0.016438	0.190038	0.442444	0.042*
H25B	0.137948	0.247248	0.431986	0.042*
C26	0.1389 (11)	0.1701 (4)	0.2836 (10)	0.0339 (19)
H26A	0.201246	0.137366	0.344216	0.041*
H26B	0.066709	0.143313	0.219869	0.041*
C27	0.2983 (11)	0.1707 (5)	0.1108 (11)	0.039 (2)
H27A	0.229728	0.144494	0.041901	0.047*
H27B	0.361344	0.137377	0.168962	0.047*
C28	0.3872 (10)	0.2180 (5)	0.0311 (11)	0.036 (2)
H28A	0.449335	0.247442	0.099552	0.044*
H28B	0.449059	0.190529	-0.024455	0.044*
C29	0.3694 (10)	0.3065 (5)	-0.1418 (11)	0.035 (2)
H29A	0.439272	0.280956	-0.191382	0.043*
H29B	0.422566	0.340498	-0.076177	0.043*
C30	0.2618 (11)	0.3427 (5)	-0.2491 (10)	0.032 (2)
H30A	0.311547	0.371631	-0.313522	0.038*
H30B	0.201563	0.308548	-0.307951	0.038*
C31	0.0763 (10)	0.4239 (5)	-0.2660 (10)	0.027 (2)
H31A	0.010250	0.392884	-0.326933	0.032*
H31B	0.128454	0.452511	-0.329063	0.032*
C32	-0.0073 (9)	0.4696 (4)	-0.1780 (8)	0.0253 (16)
H32A	0.059398	0.498412	-0.112634	0.030*

H32B -0.070902 0.500531 -0.241853 0.030*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.0258 (2)	0.01459 (18)	0.0242 (2)	0.0024 (3)	-0.00227 (13)	0.0011 (3)
O1	0.026 (3)	0.016 (2)	0.024 (3)	0.002 (2)	-0.0018 (19)	0.001 (2)
O2	0.045 (4)	0.016 (3)	0.043 (4)	-0.001 (3)	-0.018 (3)	-0.001 (3)
O3	0.061 (5)	0.022 (3)	0.053 (5)	-0.004 (3)	-0.024 (3)	-0.002 (3)
O4	0.034 (3)	0.025 (3)	0.031 (3)	-0.003 (2)	-0.007 (2)	-0.002 (2)
C1	0.027 (3)	0.021 (5)	0.027 (3)	0.014 (3)	-0.005 (3)	-0.001 (3)
C2	0.038 (5)	0.020 (4)	0.030 (5)	0.006 (3)	-0.007 (3)	-0.003 (3)
C3	0.036 (5)	0.034 (5)	0.043 (5)	0.023 (4)	-0.008 (4)	-0.012 (4)
C4	0.030 (4)	0.046 (8)	0.044 (5)	0.001 (4)	0.005 (3)	-0.022 (5)
C5	0.039 (5)	0.032 (5)	0.043 (6)	0.002 (4)	0.013 (4)	-0.010 (4)
C6	0.028 (4)	0.020 (4)	0.035 (5)	0.012 (3)	0.000 (3)	-0.003 (3)
C7	0.022 (4)	0.012 (3)	0.030 (5)	0.002 (3)	0.004 (3)	0.005 (3)
C8	0.024 (4)	0.023 (4)	0.024 (4)	0.006 (3)	0.005 (3)	0.001 (3)
C9	0.028 (4)	0.020 (3)	0.023 (4)	-0.001 (3)	0.006 (3)	-0.001 (3)
C10	0.032 (4)	0.008 (3)	0.027 (4)	0.002 (3)	0.000 (3)	0.002 (3)
C11	0.035 (4)	0.018 (3)	0.024 (4)	0.002 (3)	-0.008 (3)	0.002 (3)
C12	0.030 (4)	0.013 (4)	0.028 (5)	0.004 (3)	-0.003 (3)	0.002 (3)
C13	0.026 (3)	0.028 (4)	0.027 (3)	-0.005 (5)	-0.001 (2)	0.001 (5)
C14	0.038 (5)	0.022 (4)	0.023 (4)	-0.004 (3)	-0.001 (3)	0.004 (3)
C15	0.039 (5)	0.040 (5)	0.038 (5)	-0.010 (4)	0.012 (4)	-0.005 (4)
C16	0.039 (4)	0.034 (4)	0.032 (4)	-0.016 (6)	0.004 (3)	0.009 (6)
C17	0.038 (5)	0.041 (5)	0.030 (4)	-0.015 (4)	-0.006 (3)	0.017 (4)
C18	0.027 (4)	0.029 (4)	0.035 (5)	-0.008 (3)	0.001 (3)	0.000 (4)
C19	0.021 (4)	0.020 (4)	0.027 (4)	0.001 (3)	0.003 (3)	0.004 (3)
C20	0.032 (5)	0.020 (4)	0.029 (5)	-0.001 (3)	-0.010 (3)	0.004 (3)
K1	0.0241 (7)	0.0168 (7)	0.0240 (8)	-0.0005 (5)	0.0001 (5)	-0.0004 (5)
O5	0.026 (3)	0.017 (2)	0.026 (3)	0.000 (2)	0.0027 (19)	-0.0001 (19)
O6	0.022 (3)	0.020 (3)	0.032 (3)	-0.002 (2)	0.003 (2)	0.001 (2)
O7	0.033 (4)	0.026 (3)	0.023 (3)	0.003 (3)	0.002 (2)	0.001 (3)
O8	0.039 (3)	0.017 (3)	0.030 (3)	0.000 (2)	-0.002 (2)	-0.001 (2)
O9	0.026 (3)	0.026 (3)	0.037 (4)	0.004 (3)	0.007 (3)	0.003 (3)
O10	0.030 (3)	0.018 (3)	0.026 (3)	0.002 (2)	0.002 (2)	-0.002 (2)
C21	0.023 (4)	0.021 (4)	0.031 (4)	0.009 (3)	-0.004 (3)	-0.001 (3)
C22	0.027 (5)	0.032 (5)	0.029 (5)	0.000 (4)	0.006 (4)	-0.002 (4)
C23	0.039 (5)	0.028 (5)	0.042 (5)	-0.007 (4)	0.018 (4)	-0.001 (4)
C24	0.041 (5)	0.015 (4)	0.027 (4)	-0.006 (3)	0.007 (3)	-0.004 (3)
C25	0.048 (5)	0.031 (4)	0.025 (4)	-0.002 (4)	0.001 (4)	0.012 (4)
C26	0.045 (5)	0.018 (4)	0.035 (5)	0.003 (4)	-0.005 (4)	0.011 (3)
C27	0.047 (6)	0.029 (5)	0.040 (5)	0.014 (4)	-0.004 (4)	-0.002 (4)
C28	0.028 (4)	0.030 (4)	0.049 (6)	0.014 (4)	0.001 (4)	0.003 (4)
C29	0.029 (4)	0.025 (4)	0.054 (6)	0.000 (4)	0.011 (4)	-0.005 (4)
C30	0.041 (5)	0.031 (5)	0.025 (4)	0.005 (4)	0.013 (4)	-0.003 (4)
C31	0.032 (5)	0.019 (4)	0.027 (5)	-0.007 (3)	0.000 (4)	0.001 (3)

C32	0.028 (4)	0.026 (4)	0.020 (4)	-0.003 (3)	-0.002 (3)	0.003 (3)
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Geometric parameters (Å, °)

Sn1—O1	2.071 (5)	K1—O7	2.845 (7)
Sn1—O2	2.290 (6)	K1—O8	2.827 (6)
Sn1—C1	2.136 (7)	K1—O9	2.878 (7)
Sn1—C7	2.188 (7)	K1—O10	2.823 (6)
Sn1—C13	2.138 (7)	O5—C21	1.430 (9)
O1—C19	1.303 (10)	O5—C32	1.425 (9)
O2—C20	1.266 (11)	O6—C22	1.418 (12)
O3—C20	1.233 (11)	O6—C23	1.417 (11)
O3—K1	2.785 (7)	O7—C24	1.426 (10)
O4—C19	1.202 (10)	O7—C25	1.435 (11)
O4—K1	2.654 (6)	O8—C26	1.425 (11)
C1—C2	1.397 (12)	O8—C27	1.408 (11)
C1—C6	1.411 (13)	O9—C28	1.415 (11)
C2—H2	0.9500	O9—C29	1.443 (12)
C2—C3	1.422 (14)	O10—C30	1.421 (11)
C3—H3	0.9500	O10—C31	1.419 (11)
C3—C4	1.356 (15)	C21—H21A	0.9900
C4—H4	0.9500	C21—H21B	0.9900
C4—C5	1.376 (14)	C21—C22	1.493 (13)
C5—H5	0.9500	C22—H22A	0.9900
C5—C6	1.377 (13)	C22—H22B	0.9900
C6—H6	0.9500	C23—H23A	0.9900
C7—C8	1.401 (11)	C23—H23B	0.9900
C7—C12	1.389 (11)	C23—C24	1.494 (14)
C8—H8	0.9500	C24—H24A	0.9900
C8—C9	1.406 (10)	C24—H24B	0.9900
C9—H9	0.9500	C25—H25A	0.9900
C9—C10	1.405 (11)	C25—H25B	0.9900
C10—H10	0.9500	C25—C26	1.486 (13)
C10—C11	1.396 (11)	C26—H26A	0.9900
C11—H11	0.9500	C26—H26B	0.9900
C11—C12	1.407 (10)	C27—H27A	0.9900
C12—H12	0.9500	C27—H27B	0.9900
C13—C14	1.394 (15)	C27—C28	1.508 (15)
C13—C18	1.406 (14)	C28—H28A	0.9900
C14—H14	0.9500	C28—H28B	0.9900
C14—C15	1.396 (13)	C29—H29A	0.9900
C15—H15	0.9500	C29—H29B	0.9900
C15—C16	1.385 (16)	C29—C30	1.502 (14)
C16—H16	0.9500	C30—H30A	0.9900
C16—C17	1.370 (18)	C30—H30B	0.9900
C17—H17	0.9500	C31—H31A	0.9900
C17—C18	1.407 (13)	C31—H31B	0.9900
C18—H18	0.9500	C31—C32	1.506 (13)

C19—C20	1.567 (11)	C32—H32A	0.9900
K1—O5	2.976 (6)	C32—H32B	0.9900
K1—O6	2.802 (6)		
O1—Sn1—O2	73.4 (2)	O8—K1—O9	58.88 (19)
O1—Sn1—C1	114.0 (3)	O9—K1—O5	111.35 (18)
O1—Sn1—C7	87.6 (3)	O10—K1—O5	58.09 (15)
O1—Sn1—C13	120.4 (3)	O10—K1—O7	156.01 (19)
C1—Sn1—O2	88.5 (3)	O10—K1—O8	117.67 (18)
C1—Sn1—C7	101.9 (3)	O10—K1—O9	58.80 (19)
C1—Sn1—C13	120.5 (3)	C21—O5—K1	108.9 (4)
C7—Sn1—O2	160.8 (3)	C32—O5—K1	108.0 (4)
C13—Sn1—O2	85.5 (4)	C32—O5—C21	111.7 (6)
C13—Sn1—C7	102.5 (4)	C22—O6—K1	122.3 (5)
C19—O1—Sn1	121.9 (5)	C23—O6—K1	118.3 (6)
C20—O2—Sn1	116.1 (5)	C23—O6—C22	112.9 (7)
C20—O3—K1	117.5 (6)	C24—O7—K1	106.3 (5)
C19—O4—K1	121.4 (5)	C24—O7—C25	112.6 (7)
C2—C1—Sn1	123.1 (7)	C25—O7—K1	109.8 (5)
C2—C1—C6	118.1 (7)	C26—O8—K1	117.6 (5)
C6—C1—Sn1	118.8 (6)	C27—O8—K1	118.3 (5)
C1—C2—H2	120.5	C27—O8—C26	112.8 (6)
C1—C2—C3	119.1 (8)	C28—O9—K1	110.8 (5)
C3—C2—H2	120.5	C28—O9—C29	111.0 (7)
C2—C3—H3	119.3	C29—O9—K1	108.2 (5)
C4—C3—C2	121.4 (8)	C30—O10—K1	119.7 (5)
C4—C3—H3	119.3	C31—O10—K1	121.5 (5)
C3—C4—H4	120.2	C31—O10—C30	110.8 (7)
C3—C4—C5	119.5 (9)	O5—C21—H21A	110.0
C5—C4—H4	120.2	O5—C21—H21B	110.0
C4—C5—H5	119.5	O5—C21—C22	108.6 (7)
C4—C5—C6	121.0 (9)	H21A—C21—H21B	108.3
C6—C5—H5	119.5	C22—C21—H21A	110.0
C1—C6—H6	119.6	C22—C21—H21B	110.0
C5—C6—C1	120.8 (8)	O6—C22—C21	108.5 (7)
C5—C6—H6	119.6	O6—C22—H22A	110.0
C8—C7—Sn1	117.3 (5)	O6—C22—H22B	110.0
C12—C7—Sn1	123.7 (6)	C21—C22—H22A	110.0
C12—C7—C8	118.9 (7)	C21—C22—H22B	110.0
C7—C8—H8	119.5	H22A—C22—H22B	108.4
C7—C8—C9	121.1 (7)	O6—C23—H23A	110.0
C9—C8—H8	119.5	O6—C23—H23B	110.0
C8—C9—H9	120.3	O6—C23—C24	108.5 (8)
C10—C9—C8	119.3 (7)	H23A—C23—H23B	108.4
C10—C9—H9	120.3	C24—C23—H23A	110.0
C9—C10—H10	120.1	C24—C23—H23B	110.0
C11—C10—C9	119.8 (7)	O7—C24—C23	109.1 (7)
C11—C10—H10	120.1	O7—C24—H24A	109.9

C10—C11—H11	120.0	O7—C24—H24B	109.9
C10—C11—C12	120.0 (7)	C23—C24—H24A	109.9
C12—C11—H11	120.0	C23—C24—H24B	109.9
C7—C12—C11	120.9 (8)	H24A—C24—H24B	108.3
C7—C12—H12	119.6	O7—C25—H25A	109.9
C11—C12—H12	119.6	O7—C25—H25B	109.9
C14—C13—Sn1	118.1 (7)	O7—C25—C26	108.9 (7)
C14—C13—C18	119.9 (7)	H25A—C25—H25B	108.3
C18—C13—Sn1	122.0 (7)	C26—C25—H25A	109.9
C13—C14—H14	119.8	C26—C25—H25B	109.9
C13—C14—C15	120.5 (8)	O8—C26—C25	108.3 (7)
C15—C14—H14	119.8	O8—C26—H26A	110.0
C14—C15—H15	120.1	O8—C26—H26B	110.0
C16—C15—C14	119.7 (9)	C25—C26—H26A	110.0
C16—C15—H15	120.1	C25—C26—H26B	110.0
C15—C16—H16	120.0	H26A—C26—H26B	108.4
C17—C16—C15	120.0 (8)	O8—C27—H27A	109.9
C17—C16—H16	120.0	O8—C27—H27B	109.9
C16—C17—H17	119.1	O8—C27—C28	108.8 (8)
C16—C17—C18	121.7 (9)	H27A—C27—H27B	108.3
C18—C17—H17	119.1	C28—C27—H27A	109.9
C13—C18—C17	118.1 (9)	C28—C27—H27B	109.9
C13—C18—H18	121.0	O9—C28—C27	107.8 (8)
C17—C18—H18	121.0	O9—C28—H28A	110.1
O1—C19—C20	114.2 (7)	O9—C28—H28B	110.1
O4—C19—O1	124.3 (8)	C27—C28—H28A	110.1
O4—C19—C20	121.5 (8)	C27—C28—H28B	110.1
O2—C20—C19	113.8 (7)	H28A—C28—H28B	108.5
O3—C20—O2	128.3 (8)	O9—C29—H29A	110.2
O3—C20—C19	117.9 (8)	O9—C29—H29B	110.2
O3—K1—O5	128.47 (19)	O9—C29—C30	107.7 (8)
O3—K1—O6	99.3 (2)	H29A—C29—H29B	108.5
O3—K1—O7	83.7 (2)	C30—C29—H29A	110.2
O3—K1—O8	77.22 (19)	C30—C29—H29B	110.2
O3—K1—O9	104.4 (2)	O10—C30—C29	107.5 (7)
O3—K1—O10	119.8 (2)	O10—C30—H30A	110.2
O4—K1—O3	61.30 (19)	O10—C30—H30B	110.2
O4—K1—O5	68.16 (17)	C29—C30—H30A	110.2
O4—K1—O6	75.94 (18)	C29—C30—H30B	110.2
O4—K1—O7	117.6 (2)	H30A—C30—H30B	108.5
O4—K1—O8	138.06 (19)	O10—C31—H31A	110.0
O4—K1—O9	123.7 (2)	O10—C31—H31B	110.0
O4—K1—O10	81.05 (18)	O10—C31—C32	108.5 (7)
O6—K1—O5	57.71 (16)	H31A—C31—H31B	108.4
O6—K1—O7	59.86 (18)	C32—C31—H31A	110.0
O6—K1—O8	119.38 (18)	C32—C31—H31B	110.0
O6—K1—O9	154.6 (2)	O5—C32—C31	109.2 (6)
O6—K1—O10	115.80 (18)	O5—C32—H32A	109.8

O7—K1—O5	112.53 (18)	O5—C32—H32B	109.8
O7—K1—O9	113.74 (19)	C31—C32—H32A	109.8
O8—K1—O5	153.77 (17)	C31—C32—H32B	109.8
O8—K1—O7	59.61 (19)	H32A—C32—H32B	108.3
Sn1—O1—C19—O4	-171.7 (6)	K1—O3—C20—C19	6.8 (13)
Sn1—O1—C19—C20	9.4 (10)	K1—O4—C19—O1	179.9 (6)
Sn1—O2—C20—O3	179.5 (10)	K1—O4—C19—C20	-1.3 (11)
Sn1—O2—C20—C19	0.0 (11)	K1—O5—C21—C22	-59.3 (7)
Sn1—C1—C2—C3	-178.2 (6)	K1—O5—C32—C31	60.9 (7)
Sn1—C1—C6—C5	179.2 (7)	K1—O6—C22—C21	-35.7 (10)
Sn1—C7—C8—C9	178.1 (5)	K1—O6—C23—C24	29.9 (9)
Sn1—C7—C12—C11	-178.2 (7)	K1—O7—C24—C23	65.7 (7)
Sn1—C13—C14—C15	-176.6 (7)	K1—O7—C25—C26	-60.8 (8)
Sn1—C13—C18—C17	176.8 (6)	K1—O8—C26—C25	-37.5 (9)
O1—C19—C20—O2	-5.7 (12)	K1—O8—C27—C28	38.8 (9)
O1—C19—C20—O3	174.8 (9)	K1—O9—C28—C27	59.3 (8)
O4—C19—C20—O2	175.4 (9)	K1—O9—C29—C30	-64.4 (8)
O4—C19—C20—O3	-4.2 (14)	K1—O10—C30—C29	-34.4 (9)
C1—C2—C3—C4	-0.2 (13)	K1—O10—C31—C32	33.9 (8)
C2—C1—C6—C5	-0.1 (13)	O5—C21—C22—O6	63.9 (9)
C2—C3—C4—C5	-1.7 (14)	O6—C23—C24—O7	-65.8 (9)
C3—C4—C5—C6	2.7 (15)	O7—C25—C26—O8	66.3 (9)
C4—C5—C6—C1	-1.8 (14)	O8—C27—C28—O9	-66.0 (10)
C6—C1—C2—C3	1.1 (12)	O9—C29—C30—O10	66.4 (9)
C7—C8—C9—C10	-1.1 (11)	O10—C31—C32—O5	-64.5 (8)
C8—C7—C12—C11	-1.7 (13)	C21—O5—C32—C31	-179.4 (7)
C8—C9—C10—C11	1.1 (11)	C22—O6—C23—C24	-177.8 (7)
C9—C10—C11—C12	-1.5 (13)	C23—O6—C22—C21	173.2 (8)
C10—C11—C12—C7	1.8 (14)	C24—O7—C25—C26	-179.0 (7)
C12—C7—C8—C9	1.4 (12)	C25—O7—C24—C23	-174.0 (7)
C13—C14—C15—C16	-0.7 (14)	C26—O8—C27—C28	-178.2 (8)
C14—C13—C18—C17	-1.0 (12)	C27—O8—C26—C25	179.2 (8)
C14—C15—C16—C17	-0.1 (14)	C28—O9—C29—C30	173.8 (7)
C15—C16—C17—C18	0.3 (14)	C29—O9—C28—C27	179.6 (8)
C16—C17—C18—C13	0.2 (13)	C30—O10—C31—C32	-177.4 (7)
C18—C13—C14—C15	1.3 (13)	C31—O10—C30—C29	176.3 (7)
K1—O3—C20—O2	-172.6 (9)	C32—O5—C21—C22	-178.5 (7)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C4—H4 \cdots O1 ⁱ	0.95	2.42	3.360 (11)	172
C16—H16 \cdots O1 ⁱⁱ	0.95	2.41	3.332 (9)	165

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