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Crystal structure of potassium hydrogen bis((*E*)-2-[4-[3-(thiophen-3-yl)acryloyl]phenoxy]acetate)

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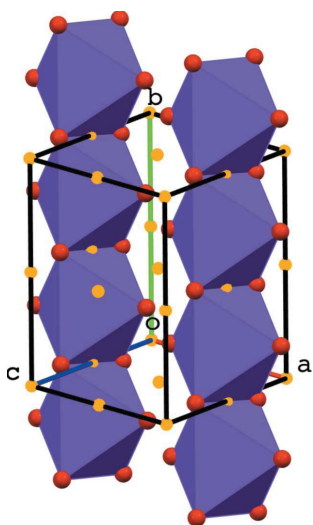
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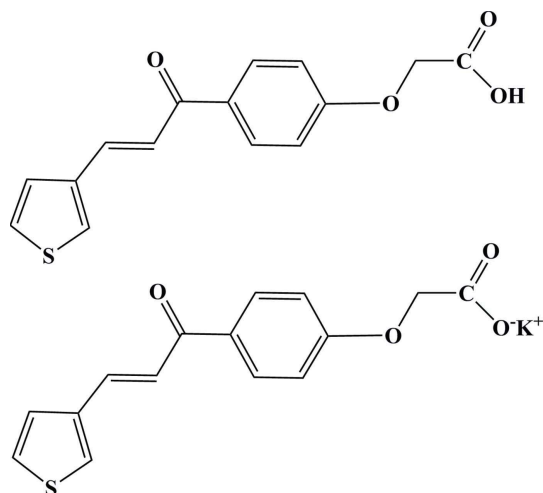
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The synthesis and spectroscopic data of (*E*)-2-[4-[3-(thiophen-3-yl)acryloyl]phenoxy]acetic acid are described. Crystallization from an ethanol–water mixture resulted in the title compound, $C_{30}H_{23}KO_8S_2$ or $[K(C_{15}H_{11}O_4S)(C_{15}H_{12}O_4S)]_n$, containing one molecule of the acid and one molecule of the potassium salt in the asymmetric unit. Both molecules share the H atom between their carboxyl groups and a potassium ion. The C=C bonds display an *E* configuration. The thiophene and phenyl rings in the two molecules are inclined by 43.3 (2) and 22.7 (2)°. The potassium ion is octahedrally coordinated by six O atoms. This distorted octahedron shares on opposite sides two oxygen atoms with inversion-related octahedra, resulting in chains of octahedra running in the [010] direction, which form ladder-like chains by C–H·· π interactions. A Hirshfeld surface analysis indicates that the highest contributions to the surface contacts arise from interactions in which H atoms are involved, with the most important contribution being from H··H (31.6 and 31.9% for the two molecules) interactions.



1. Chemical context

Over the last two decades, water-soluble polythiophenes and their derivatives have been of particular importance among conjugated polyelectrolytes owing to a unique combination of high conductivity, environmental stability and structural versatility, allowing derivatization of the π -conjugated backbone in view of numerous technological applications (Wang *et al.*, 2015; Chayer *et al.*, 1997; Wang *et al.*, 2013). Many regioregular polythiophenes with pendant carboxylic acid functionality have been studied (Ewbank *et al.*, 2004; McCullough *et al.*, 1997; Wu *et al.*, 2015; Janáky *et al.*, 2010). The increased alkyl side-chain length allows for increased coplanarity of the main-chain thiophene rings to advance regioregular polythiophene backbones (Vu Quoc *et al.*, 2019*a*). A lot of synthetic research has been conducted with a view to increasing the side-chain length of thiophene rings (Vu Quoc *et al.*, 2020). Crystal studies of thiophene monomers have also been reported (Vu Quoc *et al.*, 2017, 2018, 2019*b*).



(*E*)-2-[4-[3-(thiophen-3-yl)acryloyl]phenoxy]acetic acid are reported. This compound is considered to be a good monomer for the synthesis of water-soluble polythiophene-based conjugated polyelectrolytes. A single-crystal structure determination indicates that after crystallization, crystals were obtained containing one molecule of the acid and one molecule of the potassium salt in the asymmetric unit.

2. Structural commentary

The title compound crystallizes in the triclinic space group $P\bar{1}$ as a complex formed between the acid and the potassium salt of the acid, as illustrated in Fig. 1. In the following discussion, molecule *A* includes atoms S1–O20 and molecule *B* atoms S21–O40. Both molecules share hydrogen atom H19 between their carboxyl groups and a potassium ion, K41. Atom H19 is involved in hydrogen-bonding interactions with atoms O39

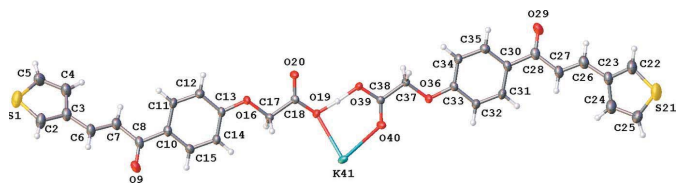


Figure 1
The molecular structure of the title compound, showing the atom-labelling scheme and displacement ellipsoids at the 30% probability level.

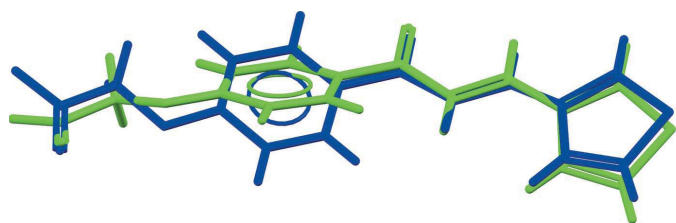


Figure 2
Overlay diagram of the two molecules *A* (green) and *B* (blue), comprising the asymmetric unit. H atoms are hidden for clarity (Mercury; Macrae *et al.*, 2020).

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

Cg3 and Cg4 are the centroids of rings C10–C15 and C30–C35, respectively.

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
O19–H19...O39	1.19 (4)	1.27 (4)	2.463 (3)	174 (4)
O19–H19...O40	1.19 (4)	2.47 (4)	3.259 (3)	122 (2)
C6–H6...O9	0.93	2.52	2.834 (6)	100
C26–H26...O29	0.93	2.49	2.814 (6)	100
C17–H17A...Cg4 ⁱ	0.97	2.76	3.509 (4)	134
C37–H37B...Cg3 ⁱⁱ	0.97	2.82	3.535 (4)	131

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

and O40 (Table 1). The dihedral angle between the thiophene and phenyl rings is 43.3 (2)° for molecule *A* and 22.7 (2)° for molecule *B*. The C=C bonds display an *E* configuration, resulting in short intramolecular C6–H6...O9 and C26–H26...O29 interactions (Table 1). The terminal thiophene groups are involved in intense thermal motion.

Fig. 2 shows an overlay diagram of the two molecules *A* and *B* [r.m.s. deviation 0.5622 Å as calculated using Mercury (Macrae *et al.*, 2020)]. The largest differences are caused by the different orientation of the phenyl groups.

Potassium ion K41 is octahedrally coordinated by six O atoms with K–O distances between 2.672 (2) and 2.906 (3) Å (Fig. 3) and an octahedral volume of 21.871 Å³. The coordination sphere can be extended with atoms O16 and O36, but K...O distances are much longer [K41...O16^{iv} = 3.245 (3), K41...O36ⁱⁱ = 3.347 (3) Å, symmetry codes: (ii) $-x + 2, -y + 2, -z + 1$, (iv) $-x + 1, -y + 1, -z + 1$].

3. Supramolecular features

In the crystal packing, the potassium ion K41 interacts with six molecules of which two occur in the carboxylic acid form

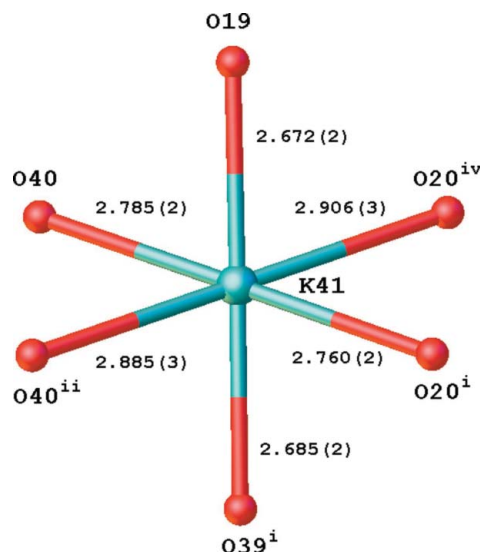


Figure 3
Octahedral coordination around K in the title compound [symmetry codes: (i) $x + 1, y, z$, (ii) $-x + 2, -y + 2, -z + 1$, (iv) $-x + 1, -y + 1, -z + 1$].

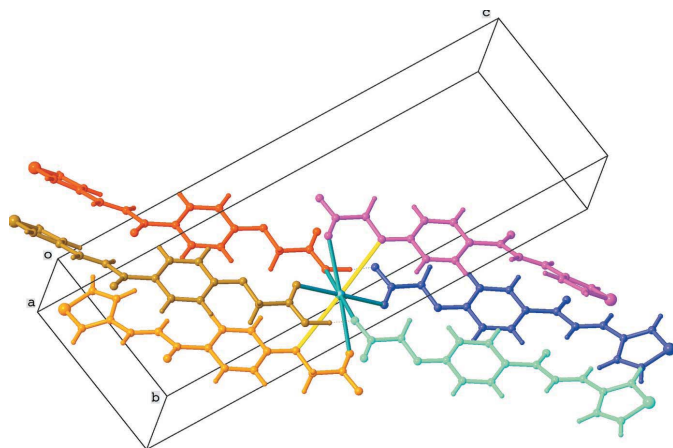


Figure 4
Potassium atom K41 is surrounded by six different molecules. The six $K \cdots O$ interactions participating in the distorted octahedral coordination are shown in turquoise, the two longer ones in yellow.

(Fig. 4). The distorted octahedron around K41 shares on opposite sides two oxygen atoms [at one side O40 and O40ⁱⁱ, at the other side O20ⁱ and O20^{iv}; symmetry codes: (i) $x + 1, y, z$, (ii) $-x + 2, -y + 2, -z + 1$, (iv) $-x + 1, -y + 1, -z + 1$] with inversion-related octahedra (Figs. 3 and 5). This results in parallel chains of octahedra running in the [010] direction and situated in the (002) plane. The $K \cdots K$ distances in the chains are 4.8084 (15) (for $K41 \cdots K41^{ii}$) and 4.8353 (14) Å [for $K41 \cdots K41^v$; symmetry code: (v) $-x + 2, -y + 1, -z + 1$].

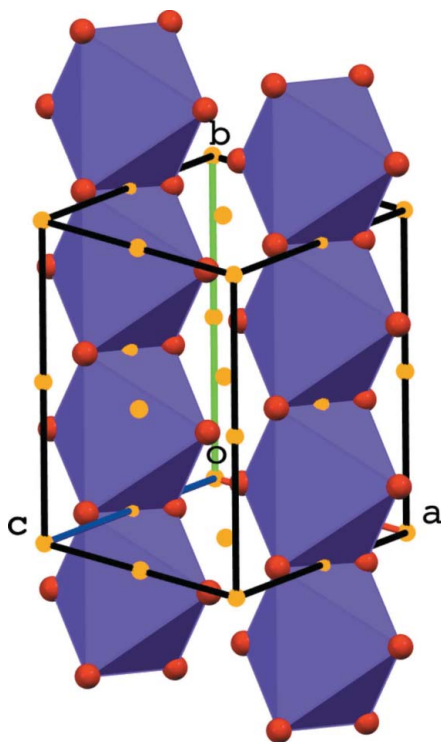


Figure 5
Parallel chains of K–O octahedra running in the [010] direction. Inversion centers are shown as yellow spheres.

Table 2
Percentage contributions of interatomic contacts to the Hirshfeld surfaces of the two molecules.

Molecule *A* includes atoms S1–O20, molecule *B* atoms S21–O40.

Contact	Molecule <i>A</i>	Molecule <i>B</i>
H···H	31.6	31.9
C···H/H···C	21.1	20.0
O···H/H···O	17.4	17.3
S···H/H···S	8.8	9.9
O···C/C···O	5.8	5.5
K···O/O···K	4.8	4.7
C···C	4.9	4.8
S···C/C···S	2.0	2.3
S···S	0.9	1.0
K···H/H···K	0.7	0.6

Despite the presence of many aromatic rings, the crystal packing of the title compound does not show π any– π interactions. The shortest centroid–centroid distance is 4.735 (3) Å between thiophene rings S1/C2–C5 and S21/C22–C25 with an

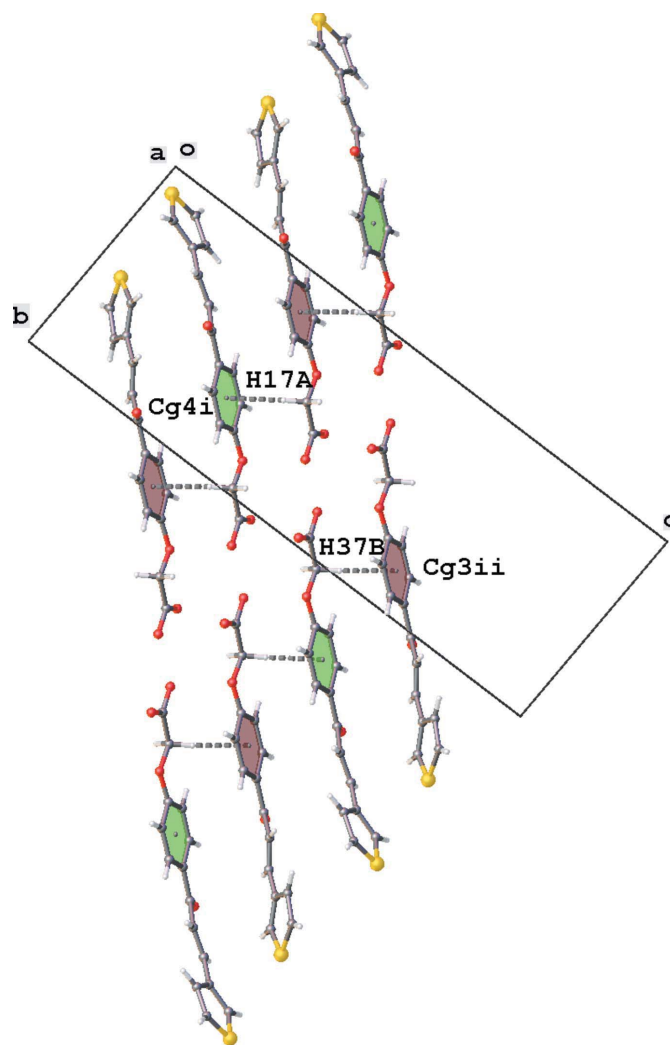


Figure 6
A view down the *a* axis of the intermolecular C–H··· π interactions of the title compound. Colour codes used: magenta for ring C10–C15, green for ring C30–C35. Cg3 and Cg4 are the centroids of the C10–C15 and C30–C35 rings, respectively. K atoms have been omitted.

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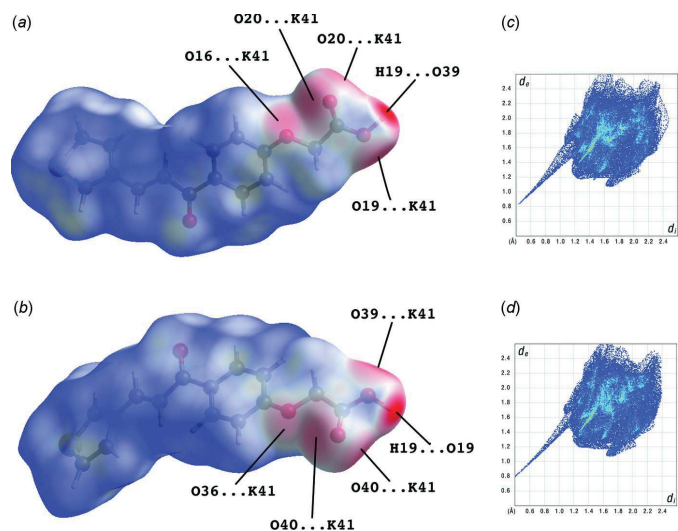


Figure 7
The Hirshfeld surfaces of molecules (a) *A* and (b) *B* mapped over d_{norm} in the colour ranges -1.0475 to 1.0150 and -1.1101 to 1.0300 a.u., respectively, together with the full two-dimensional fingerprint plots for molecules (c) *A* and (d) *B*.

angle between the rings of $52.3(3)^\circ$. However, $\text{C}-\text{H}\cdots\pi$ interactions are present and give rise to a ladder-like chain also running in the $[010]$ direction (Table 1, Fig. 6). In addition, neighbouring chains interact by short $\text{C}28=\text{O}29\cdots\text{C}g1^v$ contacts [$\text{O}29\cdots\text{C}g1^v = 3.652(4)$ Å; $\text{C}g1$ is the centroid of thiophene ring $\text{S}1/\text{C}2-\text{C}5$; symmetry code: (v) $-x, -y + 1, -z + 1$].

The packing does not show any residual solvent-accessible voids.

4. Hirshfeld surface analysis

The Hirshfeld surface analysis (Spackman & Jayatilaka, 2009) and the associated two-dimensional fingerprint plots (McKinnon *et al.*, 2007) were performed using *CrystalExplorer* (Turner *et al.*, 2017). The Hirshfeld surfaces of molecules *A* and *B* mapped over d_{norm} are given in Fig. 7*a* and *b*, respectively. The relative distributions from the different interatomic contacts to the Hirshfeld surfaces are presented in Table 2. The bright-red spots at atoms O19, H19 and O39 are indicative of the $\text{O}19-\text{H}19\cdots\text{O}39$ hydrogen bond between the molecules. The additional faint-red spots near atoms O16, O19, O20, O36, O39 and O40 concern the $\text{K}\cdots\text{O}$ interactions in the crystal structure. It should be noted that the Hirshfeld surfaces are almost identical for the two molecules. The same is true for the fingerprint plots (Fig. 7*c* and *d*). The sharp tips at $d_e + d_i \approx 1.4$ Å arise from the $\text{O}19-\text{H}19\cdots\text{O}39$ hydrogen bond. The principal contribution to the Hirshfeld surfaces involves $\text{H}\cdots\text{H}$ contacts at 31.6 and 31.9% for molecules *A* and *B*, respectively. These are followed by $\text{C}\cdots\text{H}/\text{H}\cdots\text{C}$ (21.1 and 20.0%) $\text{O}\cdots\text{H}/\text{H}\cdots\text{O}$ (17.4 and 17.3%) and $\text{S}\cdots\text{H}/\text{H}\cdots\text{S}$ (8.8 and 9.9%) contacts.

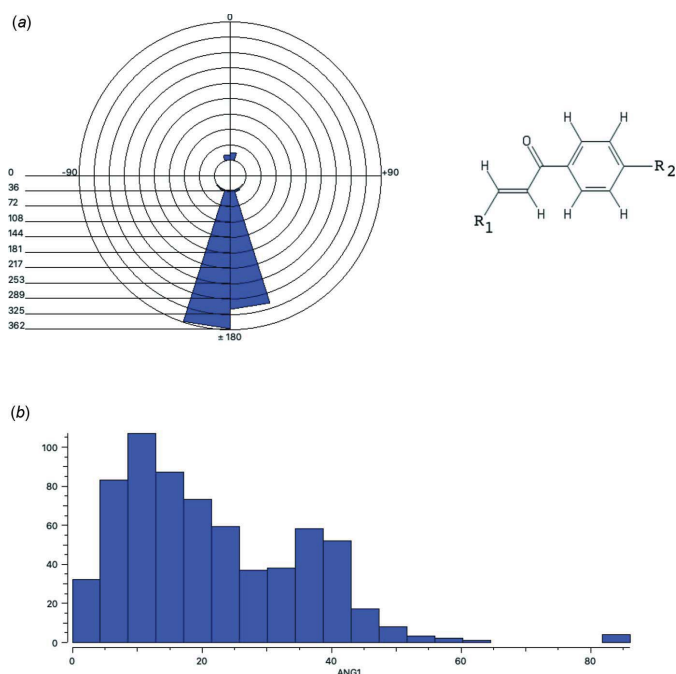


Figure 8
(a) Polar histogram of torsion angle $R_1-\text{C}=\text{C}-\text{C}$. (b) Histogram of the dihedral angle between the planes of the double bond and the phenyl ring.

5. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.42, last update February 2021; Groom *et al.*, 2016) for the fragment $R_1-\text{CH}=\text{CH}-\text{C}(=\text{O})-p-\text{C}_6\text{H}_4-R_2$ gave 619 hits (with C atoms double-bond acyclic). For only 33 cases (5.3%), the double bond has the *Z* configuration (Fig. 8*a*). The histogram of the dihedral angle between the planes of the double bond and the phenyl ring shows values between 0.0 and 86.2° (Fig. 8*b*). A search with thiophene as R_1 resulted in only four hits (CSD refcodes XOLJUG, XOLKAN, XOLKER and XOLKIV; Vu Quoc *et al.*, 2019*c*) displaying the *E* configuration and dihedral angles in the range 6.7 to 15.8° (smaller than in the title compound). Only one structure was found for which R_2 is the same as in the title compound (OCH₂COOH; CSD refcode TAMJID; Abdul Ajees *et al.*, 2017). In this monohydrate, a water molecule makes hydrogen

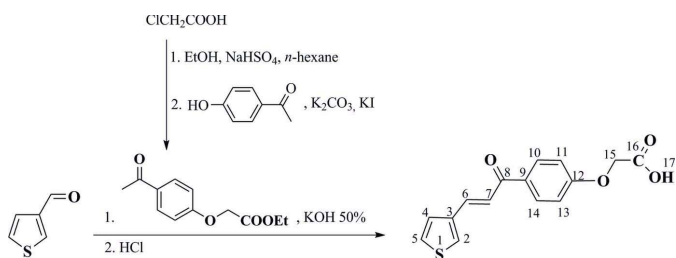


Figure 9
Reaction scheme for (*E*)-2-[4-[3-(thiophen-3-yl)acryloyl]phenoxy]acetic acid.

bonds to the carboxyl groups of two neighbouring molecules and in addition to a carbonyl of a third neighbouring enone moiety.

6. Synthesis and crystallization

The synthetic pathway to synthesize the target compound, (*E*)-2-[4-[3-(thiophen-3-yl)acryloyl]phenoxy]acetic acid, is given in Fig. 9 (numbering on chemical formulas is only used for NMR spectroscopic analysis).

A mixture of ethyl 2-(4-acetylphenoxy)acetate (5 mmol), 3-thiophenecarbaldehyde (5 mmol) and 50 mL of ethanol was stirred in ice-cold water for 20 minutes. Then, 5 mL of 50% KOH solution was added dropwise to the reaction mixture, which was then stirred continuously for 5 h. At the end of the reaction, water was added to the reaction mixture and stirring was continued until all solids in the mixture were dissolved. Concentrated HCl was slowly added to the obtained solution until the solution changed from brown to yellow. The solution was then heated until crystals appeared. The solid then began to crystallize when the solution temperature started to decrease. The crystallized solid was filtered off, washed thoroughly with water and recrystallized from an ethanol–water mixture to give 2-[4-[3-(thiophen-3-yl)acryloyl]phenoxy]acetic acid (yield 62%) in the form of pale-yellow crystals (m.p. 455 K).

IR (Shimadzu FTIR-8400S, KBr, cm^{-1}): 1017, 980 ($\text{C}=\text{C}$ bend), 1597 ($\text{C}=\text{C}$), 1659 ($\text{C}=\text{O}$), 3457 (*broad*, OH).

^1H NMR [Bruker XL-500, 500 MHz, d_6 -DMSO, (ppm), J (Hz)]: 7.60 (*d*, 1H, H^2), 7.42 (*m*, 1H, $^5J = 5.0$, H^4), 7.38 (*t*, 1H, $^4J = 5.0$, H^5), 7.81 (*d*, 1H, $^7J = 15.5$, H^6), 7.34 (*d*, 1H, $^6J = 15.5$, H^7), 8.03 (*t*, 2H, $J = 9.0$, H^{10} and H^{14}), 7.02 (*m*, 2H, H^{11} and H^{13}), 4.77 (*s*, 2H, H^{15}).

^{13}C NMR [Bruker XL-500, 125 MHz, d_6 -DMSO, (ppm)]: 121.81 (C2), 128.75 (C3), 127.01 (C4), 125.41 (C5), 132.67 (C6), 130.87 (C7), 171.85 (C8), 169.73 (C9), 138.39 (C10 and C14), 137.96 (C11 and C13), 114.65 (C12), 64.68 (C15), 189.09 (C16). Calculation for $\text{C}_{15}\text{H}_{11}\text{O}_4\text{S}$: $M = 287$ au.

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. Hydrogen atom H19 was located from a difference electron-density map and refined freely with an $U_{\text{iso}}(\text{H})$ value of $1.5U_{\text{eq}}$ of the parent atom O19. The other H atoms were placed in idealized positions and included as riding contributions with an $U_{\text{iso}}(\text{H})$ values of $1.2U_{\text{eq}}$ of the parent atom, with C–H distances of 0.93 (aromatic) and 0.97 Å (CH_2). In the final cycles of refinement, 12 outliers with $|\text{error}/\text{e.s.d.}| > 5.0$ were omitted.

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Author contributions are as follows. Conceptualization, LNN and TVQ; synthesis, LPT, DDB and DTTT; IR, NMR spectra measurements and analysis, LDK, HHM and KLV; writing

Table 3

Experimental details.

Crystal data	
Chemical formula	$[\text{K}(\text{C}_{15}\text{H}_{11}\text{O}_4\text{S})(\text{C}_{15}\text{H}_{12}\text{O}_4\text{S})]$
M_r	614.70
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	293
a, b, c (Å)	6.0036 (3), 9.6432 (5), 25.0966 (16)
α, β, γ (°)	92.412 (4), 90.548 (4), 105.808 (4)
V (Å ³)	1396.42 (14)
Z	2
Radiation type	Mo $K\alpha$
μ (mm ⁻¹)	0.39
Crystal size (mm)	0.4 × 0.2 × 0.05
Data collection	
Diffractometer	Rigaku Oxford Diffraction SuperNova, Single source at offset/far, Eos
Absorption correction	Multi-scan (<i>CrysAlis PRO</i> ; Rigaku OD, 2018)
$T_{\text{min}}, T_{\text{max}}$	0.863, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	15169, 4742, 2836
R_{int}	0.037
$(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹)	0.588
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.068, 0.211, 1.02
No. of reflections	4742
No. of parameters	373
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³)	0.59, -0.47

Computer programs: *CrysAlis PRO* (Rigaku OD, 2018), *SHELXT* (Sheldrick, 2015a), *SHELXL2016/4* (Sheldrick, 2015b) and *OLEX2* (Dolomanov *et al.*, 2009).

(review and editing of the manuscript), CNT, HT and LVM; crystal-structure determination and validation, LVM.

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Trung Vu Quoc, Linh Phan Thuy, Dai Do Ba, Duong Tran Thi Thuy, Linh Nguyen Ngoc, Chinh Nguyen Thuy, Linh Duong Khanh, Hung Ha Manh, Hoang Thai, Khoe Le Van and Luc Van Meervelt

Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2018); cell refinement: *CrysAlis PRO* (Rigaku OD, 2018); data reduction: *CrysAlis PRO* (Rigaku OD, 2018); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2016/4* (Sheldrick, 2015b); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

Potassium hydrogen bis((*E*)-2-{4-[3-(thiophen-3-yl)acryloyl]phenoxy}acetate)

Crystal data

[K(C₁₅H₁₁O₄S)(C₁₅H₁₂O₄S)]

$M_r = 614.70$

Triclinic, $P\bar{1}$

$a = 6.0036$ (3) Å

$b = 9.6432$ (5) Å

$c = 25.0966$ (16) Å

$\alpha = 92.412$ (4)°

$\beta = 90.548$ (4)°

$\gamma = 105.808$ (4)°

$V = 1396.42$ (14) Å³

$Z = 2$

$F(000) = 636$

$D_x = 1.462$ Mg m⁻³

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

Cell parameters from 3156 reflections

$\theta = 3.2\text{--}24.6^\circ$

$\mu = 0.39$ mm⁻¹

$T = 293$ K

Plate, colourless

$0.4 \times 0.2 \times 0.05$ mm

Data collection

Rigaku Oxford Diffraction SuperNova, Single source at offset/far, Eos diffractometer

Radiation source: micro-focus sealed X-ray tube, SuperNova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 15.9631 pixels mm⁻¹

ω scans

Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2018)

$T_{\min} = 0.863$, $T_{\max} = 1.000$

15169 measured reflections

4742 independent reflections

2836 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.037$

$\theta_{\max} = 24.7^\circ$, $\theta_{\min} = 2.4^\circ$

$h = -7 \rightarrow 7$

$k = -11 \rightarrow 11$

$l = -29 \rightarrow 29$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.068$

$wR(F^2) = 0.211$

$S = 1.02$

4742 reflections

373 parameters
 0 restraints
 Primary atom site location: dual
 Hydrogen site location: mixed
 H atoms treated by a mixture of independent
 and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.111P)^2 + 0.2689P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.59 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.47 \text{ e } \text{\AA}^{-3}$

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.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	−0.2860 (3)	−0.46353 (17)	0.04899 (7)	0.0970 (6)
C2	−0.0473 (9)	−0.4037 (5)	0.08824 (19)	0.0737 (15)
H2	0.067798	−0.451127	0.090654	0.088*
C3	−0.0407 (8)	−0.2770 (5)	0.11636 (17)	0.0590 (13)
C4	−0.2427 (9)	−0.2331 (6)	0.1050 (2)	0.0793 (16)
H4	−0.271387	−0.150647	0.120601	0.095*
C5	−0.3963 (9)	−0.3270 (6)	0.06777 (19)	0.0773 (16)
H5	−0.536358	−0.315058	0.055580	0.093*
C6	0.1494 (8)	−0.2019 (5)	0.15232 (17)	0.0590 (13)
H6	0.278677	−0.237405	0.152768	0.071*
C7	0.1570 (8)	−0.0887 (5)	0.18434 (16)	0.0564 (12)
H7	0.028240	−0.052797	0.186141	0.068*
C8	0.3652 (8)	−0.0173 (4)	0.21754 (16)	0.0492 (11)
O9	0.5535 (6)	−0.0384 (3)	0.20858 (12)	0.0645 (9)
C10	0.3407 (7)	0.0826 (4)	0.26258 (15)	0.0415 (10)
C11	0.1313 (7)	0.0763 (4)	0.28700 (16)	0.0481 (11)
H11	−0.003355	0.010363	0.273775	0.058*
C12	0.1206 (7)	0.1658 (4)	0.33031 (16)	0.0453 (10)
H12	−0.019503	0.157608	0.347103	0.054*
C13	0.3197 (6)	0.2689 (4)	0.34915 (14)	0.0345 (9)
C14	0.5284 (6)	0.2788 (4)	0.32503 (15)	0.0399 (9)
H14	0.661673	0.347244	0.337605	0.048*
C15	0.5386 (7)	0.1867 (4)	0.28211 (15)	0.0424 (10)
H15	0.679617	0.193833	0.265864	0.051*
O16	0.2876 (4)	0.3529 (2)	0.39231 (10)	0.0382 (6)
C17	0.4768 (6)	0.4721 (4)	0.40807 (14)	0.0319 (8)
H17A	0.535908	0.527277	0.377332	0.038*
H17B	0.600512	0.438362	0.423326	0.038*
C18	0.3947 (6)	0.5661 (4)	0.44896 (14)	0.0306 (8)
O19	0.5596 (4)	0.6761 (2)	0.46588 (10)	0.0377 (6)
H19	0.510 (5)	0.754 (3)	0.4992 (13)	0.057*
O20	0.1957 (4)	0.5391 (3)	0.46318 (11)	0.0438 (7)
K41	0.99969 (12)	0.75075 (8)	0.50093 (4)	0.0469 (3)

S21	1.2169 (3)	1.89226 (19)	0.97523 (6)	0.1039 (7)
C22	0.9574 (9)	1.8038 (5)	0.9458 (2)	0.0810 (16)
H22	0.815783	1.786740	0.962611	0.097*
C23	0.9853 (9)	1.7605 (5)	0.89334 (18)	0.0594 (13)
C24	1.2196 (10)	1.8010 (6)	0.8802 (2)	0.0792 (16)
H24	1.273298	1.780776	0.846982	0.095*
C25	1.3664 (9)	1.8759 (5)	0.9226 (2)	0.0764 (16)
H25	1.526468	1.910936	0.920654	0.092*
C26	0.7955 (8)	1.6804 (5)	0.85850 (17)	0.0609 (13)
H26	0.647616	1.682595	0.868474	0.073*
C27	0.8094 (8)	1.6051 (4)	0.81437 (16)	0.0544 (12)
H27	0.955220	1.604702	0.802281	0.065*
C28	0.6044 (8)	1.5216 (4)	0.78335 (16)	0.0507 (11)
O29	0.4111 (6)	1.5305 (4)	0.79430 (13)	0.0712 (10)
C30	0.6352 (7)	1.4222 (4)	0.73870 (15)	0.0417 (10)
C31	0.8468 (7)	1.4318 (4)	0.71402 (16)	0.0511 (11)
H31	0.978632	1.501336	0.726665	0.061*
C32	0.8639 (7)	1.3410 (4)	0.67160 (16)	0.0471 (11)
H32	1.005424	1.351138	0.655226	0.057*
C33	0.6704 (6)	1.2342 (4)	0.65314 (15)	0.0362 (9)
C34	0.4595 (7)	1.2207 (4)	0.67660 (15)	0.0415 (10)
H34	0.329159	1.149650	0.664132	0.050*
C35	0.4435 (7)	1.3150 (4)	0.71934 (15)	0.0437 (10)
H35	0.301130	1.305726	0.735190	0.052*
O36	0.7068 (4)	1.1507 (2)	0.61025 (10)	0.0382 (6)
C37	0.5203 (6)	1.0305 (4)	0.59364 (14)	0.0337 (9)
H37A	0.396265	1.063516	0.578152	0.040*
H37B	0.460285	0.974311	0.624094	0.040*
C38	0.6055 (6)	0.9381 (4)	0.55283 (13)	0.0294 (8)
O39	0.4427 (4)	0.8279 (2)	0.53558 (10)	0.0377 (6)
O40	0.8052 (4)	0.9662 (3)	0.53874 (11)	0.0437 (7)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.1100 (13)	0.0799 (10)	0.0923 (11)	0.0172 (9)	-0.0278 (10)	-0.0307 (9)
C2	0.096 (4)	0.058 (3)	0.065 (3)	0.021 (3)	-0.022 (3)	-0.016 (3)
C3	0.075 (3)	0.054 (3)	0.048 (3)	0.019 (3)	-0.006 (2)	-0.009 (2)
C4	0.085 (4)	0.082 (4)	0.074 (3)	0.033 (3)	-0.012 (3)	-0.030 (3)
C5	0.071 (3)	0.104 (4)	0.062 (3)	0.035 (3)	-0.012 (3)	-0.020 (3)
C6	0.071 (3)	0.056 (3)	0.051 (3)	0.022 (2)	-0.009 (2)	-0.018 (2)
C7	0.064 (3)	0.059 (3)	0.047 (2)	0.021 (2)	-0.005 (2)	-0.011 (2)
C8	0.066 (3)	0.043 (2)	0.040 (2)	0.017 (2)	0.000 (2)	-0.007 (2)
O9	0.067 (2)	0.070 (2)	0.0599 (19)	0.0291 (18)	0.0033 (17)	-0.0232 (17)
C10	0.051 (3)	0.033 (2)	0.040 (2)	0.0112 (19)	-0.0026 (19)	-0.0040 (18)
C11	0.043 (2)	0.046 (2)	0.048 (2)	0.0037 (19)	-0.005 (2)	-0.017 (2)
C12	0.038 (2)	0.045 (2)	0.049 (2)	0.0065 (19)	0.0049 (19)	-0.007 (2)
C13	0.041 (2)	0.0283 (19)	0.035 (2)	0.0125 (17)	-0.0021 (18)	-0.0055 (17)

C14	0.040 (2)	0.033 (2)	0.044 (2)	0.0079 (18)	-0.0013 (19)	-0.0081 (18)
C15	0.042 (2)	0.045 (2)	0.043 (2)	0.0168 (19)	0.0027 (19)	-0.0065 (19)
O16	0.0329 (14)	0.0307 (13)	0.0467 (15)	0.0037 (11)	0.0042 (12)	-0.0143 (12)
C17	0.0271 (18)	0.0277 (18)	0.041 (2)	0.0080 (15)	0.0047 (16)	-0.0023 (17)
C18	0.034 (2)	0.0231 (18)	0.036 (2)	0.0108 (16)	0.0022 (17)	0.0031 (16)
O19	0.0281 (13)	0.0313 (13)	0.0503 (16)	0.0046 (11)	-0.0025 (12)	-0.0134 (12)
O20	0.0313 (15)	0.0346 (15)	0.0600 (17)	0.0013 (12)	0.0132 (13)	-0.0118 (13)
K41	0.0229 (5)	0.0337 (5)	0.0827 (7)	0.0078 (4)	-0.0027 (4)	-0.0122 (5)
S21	0.1230 (14)	0.1059 (13)	0.0789 (11)	0.0321 (11)	-0.0247 (10)	-0.0399 (10)
C22	0.087 (4)	0.082 (4)	0.067 (3)	0.017 (3)	-0.003 (3)	-0.030 (3)
C23	0.076 (3)	0.053 (3)	0.050 (3)	0.021 (3)	-0.006 (3)	-0.014 (2)
C24	0.086 (4)	0.081 (4)	0.061 (3)	0.011 (3)	0.000 (3)	-0.021 (3)
C25	0.062 (3)	0.068 (3)	0.089 (4)	0.006 (3)	-0.003 (3)	-0.024 (3)
C26	0.073 (3)	0.057 (3)	0.056 (3)	0.024 (3)	-0.002 (3)	-0.010 (2)
C27	0.067 (3)	0.053 (3)	0.043 (2)	0.018 (2)	-0.002 (2)	-0.011 (2)
C28	0.066 (3)	0.043 (2)	0.043 (2)	0.017 (2)	0.001 (2)	-0.002 (2)
O29	0.068 (2)	0.078 (2)	0.071 (2)	0.0307 (19)	0.0031 (18)	-0.0287 (18)
C30	0.053 (3)	0.038 (2)	0.036 (2)	0.018 (2)	0.0015 (19)	-0.0054 (18)
C31	0.045 (2)	0.048 (2)	0.055 (3)	0.007 (2)	-0.009 (2)	-0.020 (2)
C32	0.039 (2)	0.044 (2)	0.054 (3)	0.0077 (19)	0.000 (2)	-0.016 (2)
C33	0.040 (2)	0.0291 (19)	0.041 (2)	0.0136 (17)	0.0015 (18)	-0.0043 (17)
C34	0.042 (2)	0.031 (2)	0.048 (2)	0.0050 (17)	0.0015 (19)	-0.0088 (19)
C35	0.042 (2)	0.046 (2)	0.043 (2)	0.0130 (19)	0.0056 (19)	0.000 (2)
O36	0.0343 (14)	0.0296 (13)	0.0467 (15)	0.0042 (11)	0.0040 (12)	-0.0128 (12)
C37	0.0314 (19)	0.0255 (18)	0.043 (2)	0.0070 (16)	0.0019 (17)	-0.0045 (17)
C38	0.030 (2)	0.0262 (18)	0.034 (2)	0.0111 (16)	-0.0027 (17)	-0.0014 (16)
O39	0.0256 (13)	0.0333 (13)	0.0511 (16)	0.0053 (11)	-0.0033 (12)	-0.0141 (12)
O40	0.0317 (15)	0.0330 (14)	0.0632 (18)	0.0042 (12)	0.0130 (13)	-0.0073 (13)

Geometric parameters (Å, °)

S1—C2	1.682 (5)	K41—K41 ^{iv}	4.8084 (15)
S1—C5	1.680 (5)	K41—K41 ^v	4.8353 (14)
C2—H2	0.9300	K41—O36 ^{iv}	3.347 (3)
C2—C3	1.375 (6)	K41—O39 ⁱⁱⁱ	2.685 (2)
C3—C4	1.419 (6)	K41—O40 ^{iv}	2.885 (3)
C3—C6	1.455 (6)	K41—O40	2.785 (2)
C4—H4	0.9300	S21—C22	1.703 (5)
C4—C5	1.415 (6)	S21—C25	1.631 (5)
C5—H5	0.9300	C22—H22	0.9300
C6—H6	0.9300	C22—C23	1.390 (6)
C6—C7	1.318 (5)	C23—C24	1.400 (7)
C7—H7	0.9300	C23—C26	1.451 (6)
C7—C8	1.481 (6)	C24—H24	0.9300
C8—O9	1.223 (5)	C24—C25	1.413 (6)
C8—C10	1.489 (5)	C25—H25	0.9300
C10—C11	1.391 (5)	C26—H26	0.9300
C10—C15	1.399 (5)	C26—C27	1.314 (5)

C11—H11	0.9300	C27—H27	0.9300
C11—C12	1.372 (5)	C27—C28	1.469 (6)
C12—H12	0.9300	C28—O29	1.220 (5)
C12—C13	1.394 (5)	C28—C30	1.490 (5)
C13—C14	1.378 (5)	C30—C31	1.400 (6)
C13—O16	1.372 (4)	C30—C35	1.388 (5)
C14—H14	0.9300	C31—H31	0.9300
C14—C15	1.380 (5)	C31—C32	1.372 (5)
C15—H15	0.9300	C32—H32	0.9300
O16—C17	1.417 (4)	C32—C33	1.387 (5)
O16—K41 ⁱ	3.245 (3)	C33—C34	1.377 (5)
C17—H17A	0.9700	C33—O36	1.372 (4)
C17—H17B	0.9700	C34—H34	0.9300
C17—C18	1.514 (5)	C34—C35	1.398 (5)
C18—O19	1.292 (4)	C35—H35	0.9300
C18—O20	1.212 (4)	O36—K41 ^{iv}	3.347 (3)
O19—H19	1.19 (3)	O36—C37	1.420 (4)
O19—K41	2.672 (2)	C37—H37A	0.9700
O20—K41 ⁱⁱⁱ	2.760 (2)	C37—H37B	0.9700
O20—K41 ⁱ	2.906 (3)	C37—C38	1.512 (5)
K41—O16 ⁱ	3.245 (3)	C38—O39	1.288 (4)
K41—O19	2.672 (2)	C38—O40	1.215 (4)
K41—H19	2.95 (3)	O39—H19	1.27 (3)
K41—O20 ⁱ	2.906 (3)	O39—K41 ⁱⁱ	2.685 (2)
K41—O20 ⁱⁱⁱ	2.760 (2)	O40—K41 ^{iv}	2.885 (3)
C5—S1—C2	94.1 (2)	K41 ^{iv} —K41—H19	73.6 (6)
S1—C2—H2	123.6	K41 ^{iv} —K41—K41 ^v	178.88 (4)
C3—C2—S1	112.8 (4)	O36 ^{iv} —K41—H19	116.2 (6)
C3—C2—H2	123.6	O36 ^{iv} —K41—K41 ^v	99.62 (4)
C2—C3—C4	110.3 (4)	O36 ^{iv} —K41—K41 ^{iv}	79.38 (5)
C2—C3—C6	123.5 (4)	O39 ⁱⁱⁱ —K41—O16 ⁱ	103.47 (7)
C4—C3—C6	126.2 (4)	O39 ⁱⁱⁱ —K41—H19	156.1 (7)
C3—C4—H4	123.2	O39 ⁱⁱⁱ —K41—O20 ⁱ	105.57 (8)
C5—C4—C3	113.5 (4)	O39 ⁱⁱⁱ —K41—O20 ⁱⁱⁱ	73.16 (7)
C5—C4—H4	123.2	O39 ⁱⁱⁱ —K41—K41 ^v	89.77 (5)
S1—C5—H5	125.3	O39 ⁱⁱⁱ —K41—K41 ^{iv}	90.48 (5)
C4—C5—S1	109.3 (4)	O39 ⁱⁱⁱ —K41—O36 ^{iv}	76.94 (7)
C4—C5—H5	125.3	O39 ⁱⁱⁱ —K41—O40	106.49 (8)
C3—C6—H6	116.7	O39 ⁱⁱⁱ —K41—O40 ^{iv}	74.93 (7)
C7—C6—C3	126.6 (4)	O40—K41—O16 ⁱ	70.70 (7)
C7—C6—H6	116.7	O40 ^{iv} —K41—O16 ⁱ	131.58 (7)
C6—C7—H7	119.1	O40 ^{iv} —K41—H19	97.8 (6)
C6—C7—C8	121.7 (4)	O40—K41—H19	51.1 (6)
C8—C7—H7	119.1	O40 ^{iv} —K41—O20 ⁱ	177.80 (7)
C7—C8—C10	118.2 (4)	O40—K41—O20 ⁱ	117.64 (8)
O9—C8—C7	121.6 (4)	O40 ^{iv} —K41—K41 ^v	147.81 (6)
O9—C8—C10	120.2 (4)	O40—K41—K41 ^v	148.16 (6)

C11—C10—C8	123.3 (4)	O40—K41—K41 ^{iv}	32.63 (6)
C11—C10—C15	118.1 (3)	O40 ^{iv} —K41—K41 ^{iv}	31.38 (5)
C15—C10—C8	118.5 (4)	O40 ^{iv} —K41—O36 ^{iv}	49.80 (6)
C10—C11—H11	119.5	O40—K41—O36 ^{iv}	110.51 (7)
C12—C11—C10	121.0 (4)	O40—K41—O40 ^{iv}	64.01 (9)
C12—C11—H11	119.5	C25—S21—C22	94.4 (2)
C11—C12—H12	120.0	S21—C22—H22	124.5
C11—C12—C13	120.0 (4)	C23—C22—S21	111.1 (4)
C13—C12—H12	120.0	C23—C22—H22	124.5
C14—C13—C12	120.0 (3)	C22—C23—C24	110.5 (4)
O16—C13—C12	114.8 (3)	C22—C23—C26	123.7 (5)
O16—C13—C14	125.2 (3)	C24—C23—C26	125.8 (4)
C13—C14—H14	120.2	C23—C24—H24	123.3
C13—C14—C15	119.6 (3)	C23—C24—C25	113.4 (5)
C15—C14—H14	120.2	C25—C24—H24	123.3
C10—C15—H15	119.4	S21—C25—H25	124.6
C14—C15—C10	121.2 (3)	C24—C25—S21	110.7 (4)
C14—C15—H15	119.4	C24—C25—H25	124.6
C13—O16—C17	116.7 (3)	C23—C26—H26	116.4
C13—O16—K41 ⁱ	127.8 (2)	C27—C26—C23	127.1 (5)
C17—O16—K41 ⁱ	106.87 (18)	C27—C26—H26	116.4
O16—C17—H17A	110.0	C26—C27—H27	118.6
O16—C17—H17B	110.0	C26—C27—C28	122.8 (4)
O16—C17—C18	108.7 (3)	C28—C27—H27	118.6
H17A—C17—H17B	108.3	C27—C28—C30	118.8 (4)
C18—C17—H17A	110.0	O29—C28—C27	121.1 (4)
C18—C17—H17B	110.0	O29—C28—C30	120.1 (4)
O19—C18—C17	112.2 (3)	C31—C30—C28	123.8 (4)
O20—C18—C17	122.6 (3)	C35—C30—C28	118.7 (4)
O20—C18—O19	125.2 (3)	C35—C30—C31	117.5 (3)
C18—O19—H19	116.3 (15)	C30—C31—H31	119.3
C18—O19—K41	142.1 (2)	C32—C31—C30	121.5 (4)
K41—O19—H19	91.2 (14)	C32—C31—H31	119.3
C18—O20—K41 ⁱⁱ	122.3 (2)	C31—C32—H32	120.0
C18—O20—K41 ⁱ	115.7 (2)	C31—C32—C33	120.0 (4)
K41 ⁱⁱ —O20—K41 ⁱ	117.15 (9)	C33—C32—H32	120.0
O16 ⁱ —K41—H19	63.9 (6)	C34—C33—C32	120.2 (3)
O16 ⁱ —K41—K41 ^v	79.07 (5)	O36—C33—C32	115.1 (3)
O16 ⁱ —K41—K41 ^{iv}	101.93 (5)	O36—C33—C34	124.7 (3)
O16 ⁱ —K41—O36 ^{iv}	178.61 (5)	C33—C34—H34	120.4
O19—K41—O16 ⁱ	76.94 (7)	C33—C34—C35	119.3 (3)
O19—K41—H19	23.9 (6)	C35—C34—H34	120.4
O19—K41—O20 ⁱ	74.97 (7)	C30—C35—C34	121.6 (4)
O19—K41—O20 ⁱⁱⁱ	107.07 (8)	C30—C35—H35	119.2
O19—K41—K41 ^v	90.67 (5)	C34—C35—H35	119.2
O19—K41—K41 ^{iv}	89.07 (5)	C33—O36—K41 ^{iv}	129.6 (2)
O19—K41—O36 ^{iv}	102.66 (7)	C33—O36—C37	117.0 (3)
O19—K41—O39 ⁱⁱⁱ	179.45 (8)	C37—O36—K41 ^{iv}	104.73 (18)

O19—K41—O40	73.28 (7)	O36—C37—H37A	109.9
O19—K41—O40 ^{iv}	104.52 (8)	O36—C37—H37B	109.9
O20 ⁱ —K41—O16 ⁱ	50.51 (6)	O36—C37—C38	109.1 (3)
O20 ⁱⁱⁱ —K41—O16 ⁱ	109.72 (7)	H37A—C37—H37B	108.3
O20 ⁱ —K41—H19	82.6 (6)	C38—C37—H37A	109.9
O20 ⁱⁱⁱ —K41—H19	129.3 (6)	C38—C37—H37B	109.9
O20 ⁱⁱⁱ —K41—O20 ⁱ	62.85 (9)	O39—C38—C37	112.1 (3)
O20 ⁱ —K41—K41 ^{iv}	150.25 (6)	O40—C38—C37	122.9 (3)
O20 ⁱ —K41—K41 ^v	30.52 (5)	O40—C38—O39	125.0 (3)
O20 ⁱⁱⁱ —K41—K41 ^v	32.32 (6)	K41 ⁱⁱ —O39—H19	94.4 (13)
O20 ⁱⁱⁱ —K41—K41 ^{iv}	146.88 (7)	C38—O39—H19	112.4 (14)
O20 ⁱⁱⁱ —K41—O36 ^{iv}	69.08 (7)	C38—O39—K41 ⁱⁱ	142.1 (2)
O20 ⁱ —K41—O36 ^{iv}	128.11 (6)	K41—O40—K41 ^{iv}	115.99 (9)
O20 ⁱⁱⁱ —K41—O40 ^{iv}	115.51 (8)	C38—O40—K41 ^{iv}	116.9 (2)
O20 ⁱⁱⁱ —K41—O40	179.49 (8)	C38—O40—K41	121.3 (2)
K41 ^v —K41—H19	106.5 (6)		
S1—C2—C3—C4	1.2 (6)	K41 ^{iv} —O36—C37—C38	39.0 (3)
S1—C2—C3—C6	-178.9 (4)	S21—C22—C23—C24	0.6 (6)
C2—S1—C5—C4	0.4 (5)	S21—C22—C23—C26	178.8 (4)
C2—C3—C4—C5	-0.9 (7)	C22—S21—C25—C24	0.0 (4)
C2—C3—C6—C7	-172.8 (5)	C22—C23—C24—C25	-0.6 (7)
C3—C4—C5—S1	0.3 (6)	C22—C23—C26—C27	-161.5 (5)
C3—C6—C7—C8	-177.2 (4)	C23—C24—C25—S21	0.3 (6)
C4—C3—C6—C7	7.1 (9)	C23—C26—C27—C28	176.8 (4)
C5—S1—C2—C3	-0.9 (4)	C24—C23—C26—C27	16.4 (8)
C6—C3—C4—C5	179.2 (5)	C25—S21—C22—C23	-0.4 (4)
C6—C7—C8—O9	16.3 (7)	C26—C23—C24—C25	-178.8 (4)
C6—C7—C8—C10	-163.9 (4)	C26—C27—C28—O29	7.8 (7)
C7—C8—C10—C11	23.2 (6)	C26—C27—C28—C30	-170.8 (4)
C7—C8—C10—C15	-158.0 (4)	C27—C28—C30—C31	-19.3 (6)
C8—C10—C11—C12	176.3 (4)	C27—C28—C30—C35	162.1 (4)
C8—C10—C15—C14	-177.6 (4)	C28—C30—C31—C32	-177.4 (4)
O9—C8—C10—C11	-157.1 (4)	C28—C30—C35—C34	178.3 (4)
O9—C8—C10—C15	21.7 (6)	O29—C28—C30—C31	162.1 (4)
C10—C11—C12—C13	2.6 (6)	O29—C28—C30—C35	-16.5 (6)
C11—C10—C15—C14	1.2 (6)	C30—C31—C32—C33	-1.7 (7)
C11—C12—C13—C14	-1.5 (6)	C31—C30—C35—C34	-0.5 (6)
C11—C12—C13—O16	179.5 (3)	C31—C32—C33—C34	1.3 (6)
C12—C13—C14—C15	0.3 (6)	C31—C32—C33—O36	179.4 (4)
C12—C13—O16—C17	-171.3 (3)	C32—C33—C34—C35	-0.5 (6)
C12—C13—O16—K41 ⁱ	45.4 (4)	C32—C33—O36—K41 ^{iv}	-44.3 (4)
C13—C14—C15—C10	-0.2 (6)	C32—C33—O36—C37	173.4 (3)
C13—O16—C17—C18	169.6 (3)	C33—C34—C35—C30	0.1 (6)
C14—C13—O16—C17	9.7 (5)	C33—O36—C37—C38	-170.2 (3)
C14—C13—O16—K41 ⁱ	-133.6 (3)	C34—C33—O36—K41 ^{iv}	133.7 (3)
C15—C10—C11—C12	-2.4 (6)	C34—C33—O36—C37	-8.6 (5)
O16—C13—C14—C15	179.2 (3)	C35—C30—C31—C32	1.3 (6)

O16—C17—C18—O19	178.9 (3)	O36—C33—C34—C35	-178.4 (3)
O16—C17—C18—O20	-1.5 (5)	O36—C37—C38—O39	-179.0 (3)
C17—C18—O19—K41	-46.1 (5)	O36—C37—C38—O40	1.2 (5)
C17—C18—O20—K41 ⁱ	52.3 (4)	C37—C38—O39—K41 ⁱⁱ	44.9 (5)
C17—C18—O20—K41 ⁱⁱ	-153.2 (2)	C37—C38—O40—K41	153.2 (3)
O19—C18—O20—K41 ⁱⁱ	26.3 (5)	C37—C38—O40—K41 ^{iv}	-54.7 (4)
O19—C18—O20—K41 ⁱ	-128.2 (3)	O39—C38—O40—K41	-26.5 (5)
O20—C18—O19—K41	134.3 (3)	O39—C38—O40—K41 ^{iv}	125.6 (3)
K41 ⁱ —O16—C17—C18	-40.0 (3)	O40—C38—O39—K41 ⁱⁱ	-135.4 (3)

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

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

$Cg3$ and $Cg4$ are the centroids of rings C10–C15 and C30–C35, respectively.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O19—H19 \cdots O39	1.19 (4)	1.27 (4)	2.463 (3)	174 (4)
O19—H19 \cdots O40	1.19 (4)	2.47 (4)	3.259 (3)	122 (2)
C6—H6 \cdots O9	0.93	2.52	2.834 (6)	100
C26—H26 \cdots O29	0.93	2.49	2.814 (6)	100
C17—H17A \cdots Cg4 ^{vi}	0.97	2.76	3.509 (4)	134
C37—H37B \cdots Cg3 ⁱ	0.97	2.82	3.535 (4)	131

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (vi) $-x+1, -y+2, -z+1$.