

Bis(dicyclohexylammonium 3-thienyl-
acetate)Seik Weng Ng,^a Suchada Chantrapromma,^{b*}
Ibrahim Abdul Razak^c and Hoong-Kun Fun^c^aInstitute of Postgraduate Studies, University of Malaya, 50603 Kuala Lumpur, Malaysia, ^bDepartment of Chemistry, Faculty of Science, Prince of Songkla University, Hat Yai, Songkla 90112, Thailand, and ^cX-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia
Correspondence e-mail: suchada@ratree.psu.ac.th

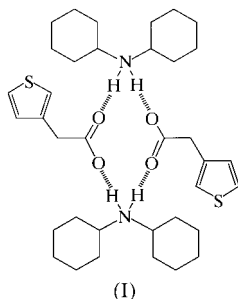
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The triclinic cell of the title compound contains $2C_{12}H_{24}N^{+} \cdot 2C_6H_5O_2S^{-}$ ion pairs that are linked by four hydrogen bonds [$N \cdots O = 2.728(3)$ and $2.758(3) \text{ \AA}$] across a centre of inversion.

Comment

The dicyclohexylammonium salts of a number of substituted acetic acids have been structurally documented; the acids include, for example, 2,4-dichlorophenoxyacetic acid (Subramanian *et al.*, 2000), various dithiocarbamylacetic acids (Ng & Hook, 1999) and trifluoroacetic acid (Ng *et al.*, 1999). With the exceptions of the trifluoroacetate, which adopts a zigzag hydrogen-bonded chain structure, and the *N,N*-dimethyldithiocarbamylacetate, which crystallizes as a monohydrate (Ng, 1992), the dicyclohexylammonium carboxylates exist as two ion pairs linked by $N \cdots O$ hydrogen bonds across a centre of inversion; the interactions give rise to the formation of an $H-N-H \cdots O-C-O \cdots H-N-H \cdots O-C-O \cdots$ ring. The short hydrogen bonds [$N \cdots O = 2.728(3)$ and $2.758(3) \text{ \AA}$] in the title compound, (I), are representative of the hydrogen bonds found in other dimeric dicyclohexylammonium carboxylates.



A displacement ellipsoid plot with the numbering scheme of the dimeric title compound is shown in Fig. 1.

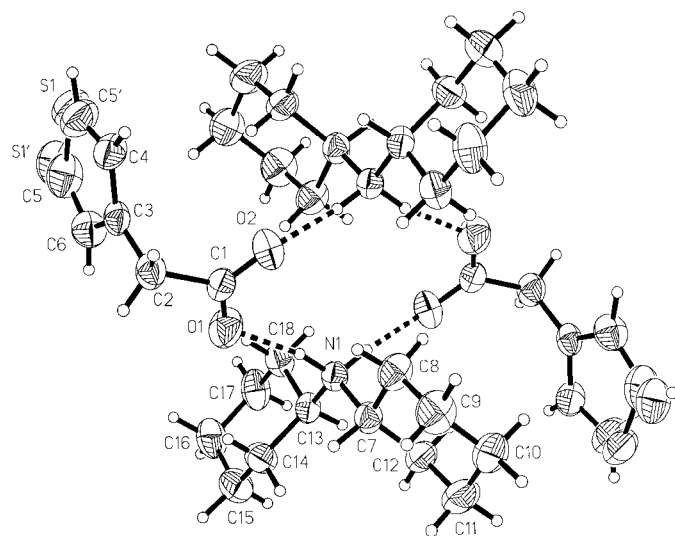


Figure 1
ORTEPII (Johnson, 1976) diagram of the dimeric title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme.

Experimental

Compound (I) was the unexpected product from the attempted condensation of dicyclohexylammonium hydrogen 3-thienylmalonate and dibutyltin oxide in ethanol. The ammonium hydrogen carboxylate was prepared by treating equimolar quantities of dicyclohexylamine and 3-thienylmalonic acid in ethanol; the reagent was heated with dibutyltin oxide (2:1 molar ratio) until the oxide dissolved completely. The title compound separated as crystals when the solution was cooled.

Crystal data

$2C_{12}H_{24}N^{+} \cdot 2C_6H_5O_2S^{-}$
 $M_r = 646.96$
Triclinic, $P\bar{1}$
 $a = 8.8444(3) \text{ \AA}$
 $b = 9.7581(2) \text{ \AA}$
 $c = 11.9220(3) \text{ \AA}$
 $\alpha = 112.009(1)^\circ$
 $\beta = 94.990(1)^\circ$
 $\gamma = 99.585(1)^\circ$
 $V = 927.98(4) \text{ \AA}^3$

$Z = 1$
 $D_x = 1.158 \text{ Mg m}^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 4250 reflections
 $\theta = 1.87\text{--}28.19^\circ$
 $\mu = 0.181 \text{ mm}^{-1}$
 $T = 298(2) \text{ K}$
Block, colourless
 $0.48 \times 0.44 \times 0.34 \text{ mm}$

Data collection

Siemens SMART CCD area-detector diffractometer
 ω scans
Absorption correction: empirical (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.918$, $T_{\max} = 0.941$
6549 measured reflections

4299 independent reflections
2833 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.047$
 $\theta_{\max} = 28.19^\circ$
 $h = -11 \rightarrow 11$
 $k = -12 \rightarrow 12$
 $l = -15 \rightarrow 7$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.060$
 $wR(F^2) = 0.162$
 $S = 0.926$
4299 reflections
317 parameters

H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0865P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.25 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.33 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

S1—C4	1.689 (2)	O2—C1	1.248 (2)
S1—C5	1.708 (8)	N1—C7	1.506 (2)
O1—C1	1.249 (2)	N1—C13	1.507 (2)
<hr/>			
C7—N1—C13	117.6 (1)	O2—C1—O1	125.9 (2)

Table 2

Hydrogen-bonding geometry (Å, °).

<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>
N1—H1A...O1	1.01 (3)	1.74 (3)	2.728 (3)	167 (2)
N1—H1B...O2 ⁱ	0.92 (2)	1.85 (2)	2.758 (3)	169 (2)

Symmetry code: (i) 1 - x, 1 - y, 1 - z.

All H atoms, except the H atoms attached to the disordered portion of the molecule, were located from difference maps and isotropically refined. Those attached to the disordered portion (H5 and H5') were fixed geometrically and allowed to ride on the parent atoms with $U_{\text{iso}} = 1.2U_{\text{eq}}$. The S atom at the 3-position of the thiophene ring is disordered with respect to the C atom at the 4-position. The occupancy refined to almost 3:1, and this was then fixed as 3:1. C—S distances were restrained to be equal by a *SADI*0.01 instruction in *SHELXTL* (Sheldrick, 1997). The displacement parameter of S1 was set to be equal to that of C5', and that of S1' to be equal to that of C5.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 1990).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1439). Services for accessing these data are described at the back of the journal.

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