

O2—C1'—O5'	112.95 (15)	C1—O5—C5	112.48 (15)
C1'—O5'—C5'	115.57 (15)	O1—C1—O5	107.9 (2)
C1'—O2—C2	118.02 (15)	C1—O1—C7	111.9 (2)
O5'—C1'—O2—C2	59.0 (2)	O5—C1—O1—C7	-69.9 (2)
C1'—O2—C2—C1	95.0 (2)		

Table 2. Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ )

D—H...A	D—H	H...A	D...A	D—H...A
O3—H3b...O4 <sup>i</sup>	0.95 (4)	1.90 (4)	2.789 (2)	154 (3)
O4 <sup>i</sup> —H4 <sup>i</sup> b...O4 <sup>ii</sup>	0.96 (4)	1.99 (4)	2.854 (2)	149 (3)
O4—H4b...O2 <sup>iii</sup>	0.92 (4)	1.85 (4)	2.762 (2)	171 (3)
O2 <sup>i</sup> —H2 <sup>i</sup> b...O6 <sup>iv</sup>	0.89 (4)	1.77 (4)	2.653 (2)	172 (3)
O6 <sup>i</sup> —H6 <sup>i</sup> c...O6 <sup>v</sup>	0.86 (5)	1.81 (5)	2.666 (2)	174 (5)
O6—H6c...O3 <sup>vi</sup>	0.91 (4)	2.02 (4)	2.874 (2)	157 (3)
O3 <sup>i</sup> —H3 <sup>i</sup> b...O5 <sup>vii</sup>	0.90 (4)	1.95 (4)	2.840 (2)	172 (3)

Symmetry codes: (i)  $x - \frac{1}{2}, \frac{3}{2} - y, 1 - z$ ; (ii)  $\frac{1}{2} + x, \frac{3}{2} - y, 1 - z$ ; (iii)  $\frac{1}{2} - x, 1 - y, z - \frac{1}{2}$ ; (iv)  $1 - x, \frac{1}{2} + y, \frac{3}{2} - z$ ; (v)  $\frac{1}{2} + x, \frac{1}{2} - y, 1 - z$ ; (vi)  $x, y - 1, z$ .

All non-H atoms were refined with anisotropic displacement parameters using a 'rigid-bond' restraint to  $U_{ij}$  of two bonded atoms (Rollett, 1970), implemented as the *DELU* instruction in *SHELXL93* (Sheldrick, 1993). The H atoms were positioned geometrically and allowed to ride during the least-squares refinements. The torsion angles containing H atoms are calculated with geometrically placed H atoms, thereby the e.s.d.'s of these are of little significance since the e.s.d.'s of the H-atom positions are related to those of the parent atom. The absolute configuration of the title compound was determined by its constituent monosaccharides that have the D configuration. This absolute configuration is in agreement with the obtained value of the Flack parameter which indicates the correct absolute configuration.

Data collection: *DIF4* (Stoe & Cie, 1991a). Cell refinement: *DIF4*. Data reduction: *REDU4* (Stoe & Cie, 1991b). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93*. Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1034). Services for accessing these data are described at the back of the journal.

## References

- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.  
 Hassel, O. & Ottar, B. (1947). *Acta Chem. Scand.* **1**, 929–942.  
 Jansson, P.-E., Kenne, L., Persson, K. & Widmalm, G. (1990). *J. Chem. Soc. Perkin Trans. 1*, pp. 591–598.  
 North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.  
 Rollett J. S. (1970). *Crystallographic Computing*, edited by F. R. Ahmed, S. R. Hall & C. P. Huber, pp. 167–181. Copenhagen: Munksgaard.  
 Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.  
 Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.  
 Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.  
 Stoe & Cie (1991a). *DIF4. Diffractometer Control Program*. Version 7.08. Stoe & Cie, Darmstadt, Germany.  
 Stoe & Cie (1991b). *REDU4. Data Reduction Program*. Version 7.08. Stoe & Cie, Darmstadt, Germany.  
 Thøgersen, H., Lemieux, R. U., Bock, K. & Meyer, B. (1982). *Can. J. Chem.* **60**, 44–57.  
 Wiberg, K. B., Murcko, M. A., Laidig, K. E. & MacDougall, P. J. (1990). *J. Phys. Chem.* **94**, 6956–6959.  
 Wolfe, S. (1972). *Acc. Chem. Res.* **5**, 102–111.

*Acta Cryst.* (1997). **C53**, 1107–1111

## (1RS,3SR,4RS,5RS,7SR,9SR)-4,7-Diacetyl-9-hydroxy-1,3,5,9-tetramethyl-2,6,8-trioxatri-cyclo[3.2.1.1<sup>3,7</sup>]nonane: a Tricyclic System Formed Under Cathodic Conditions

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## Abstract

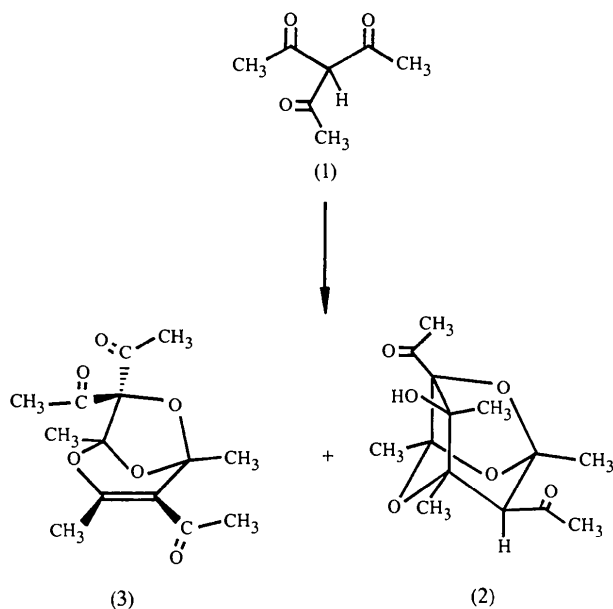
The title compound,  $C_{14}H_{20}O_6$ , generated from a bicyclic precursor under electrochemical conditions, crystallizes with six identical molecules in the asymmetric unit. Comparison of equivalent bond lengths and angles reveals no major structural differences between the molecules. However, the intermolecular distances suggest that the molecular packing is due to pairwise attractive interactions between the species, resulting in three different types of dimers. The crystal lattice can be described as a layer structure with strata either perpendicular or parallel to the *ac* plane.

## Comment

During our work in the field of the electrochemistry of substituted 4,7,8-trioxabicyclo[3.2.1]oct-2-enes [(3), see scheme below] we obtained the title compound (2) as a side product following the method described in the *Experimental* section. Isolation of (2) from the reaction mixture provides evidence that bicyclooctenes like (3) serve as precursors to tricyclic products under cathodic conditions. Evaluation of the reaction mechanism (Thomas, Wellen, Simons & Raabe, 1993; Simons 1992) critically depends on a reliable determination of the structures of the products.

Compound (3) was obtained as a racemate. Moreover, the fact that (2) was also formed as a racemate indicates that the two-electron reduction of (3) is stereoselective in the sense that one enantiomer of (3) yields a single enantiomer of (2). The title compound (2) has six chiral centres (C6, C11, C4, C3, C10 and C13, which are C1, C3, C5, C4, C7 and C9 according to IUPAC numbering). While the chirality of the first five C atoms could be determined by spectroscopic

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methods, there are no such means for determining the absolute configuration at C13 (C9). The structure was therefore elucidated using X-ray crystallography. For the enantiomer shown in Fig. 1, the absolute configuration at C13 (C9) is *S*, and the complete description of the chirality of the molecule is therefore *1R,3S,4R,5R,7S,9S*. Since (2) was obtained as a racemate its full name is (*1RS,3SR,4RS,5RS,7SR,9SR*)-4,7-diacetyl-9-hydroxy-1,3,5,9-tetramethyl-2,6,8-trioxatricyclo[3.2.1.1<sup>3,7</sup>]nonane.

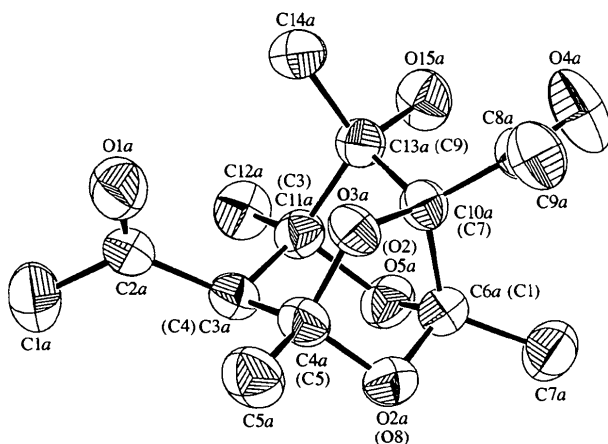


Fig. 1. One out of six members of the asymmetric unit. The ellipsoids are plotted at the 50% probability level. H atoms have been omitted for clarity. The numbers in parentheses correspond to IUPAC nomenclature.

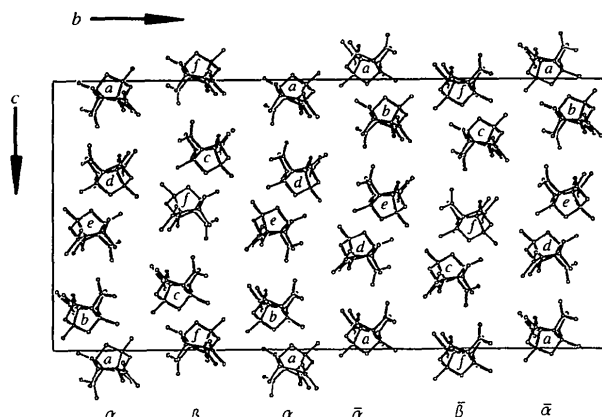
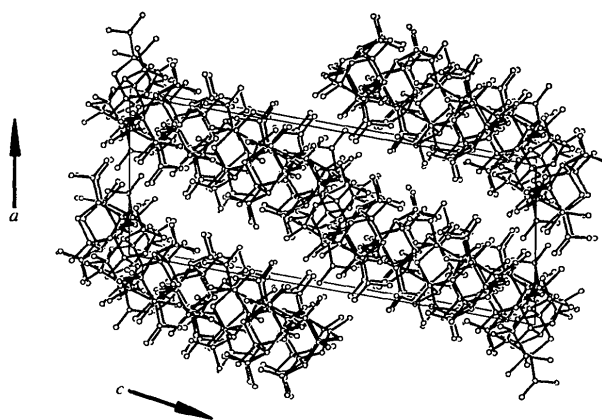
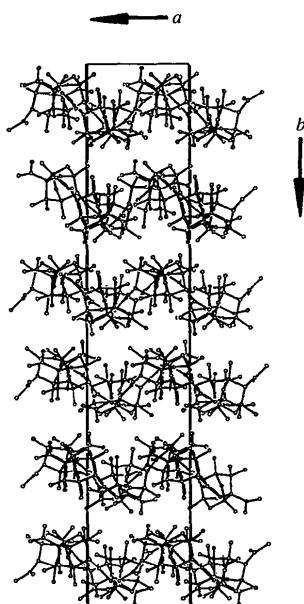
The most striking feature of the structure is the presence of six molecules (*a–f*) in the asymmetric unit. Comparison of corresponding bond lengths and angles reveals no major structural differences between the six molecules (*cf.* Tables 1 and 2). The space group *P2<sub>1</sub>/n* was indicated by the systematically absent

intensities. Later tests on the solved structure using *MISSYM* (Le Page, 1988) and *BUNYIP* (Hester & Hall, 1996) failed to detect higher crystallographic symmetry. Thus, it is reasonable to assume that the existence of six symmetrically independent molecules is caused by specific interactions between the individuals.

All but three H atoms could be located in a difference Fourier map, and hydrogen bonds of different strength are obviously responsible for the molecular packing. According to the intermolecular distances (Bondi, 1964), the members of the asymmetric unit cluster in pairs (*a*···*b*, which requires  $1-x, 1-y, 2-z$  at *b*; *e*···*d*, which requires  $1+x, y, z$  at *e*; and *c*···*f*, which requires  $\frac{1}{2}+x, \frac{3}{2}-y, \frac{1}{2}+z$  at *f*). The molecules in each dimer are linked by one classical (O—H···O) and two most likely weaker non-classical (C—H···O) hydrogen bonds (Taylor & Kennard, 1982; Jeffrey & Saenger, 1991).

The stronger O—H···O links are those between the hydroxylic proton H15 and O15 of a neighbouring molecule. The strongest of these bonds is probably that between molecules *a* and *b* [H15*a*···O15*b* 2.450 (2), O15*a*···O15*b* 3.037 (3) Å and O15*a*—H15*a*···O15*b* 133.1°], while according to bond lengths and angles those between *c* and *f* [H15*f*···O15*c* 2.899 (2), O15*c*···O15*f* 3.191 (3) Å and O15*f*—H15*f*···O15*c* 100.9°], and *d* and *e* [H15*e*···O15*d* 3.103 (2), O15*d*···O15*e* 3.341 (4) Å and O15*e*—H15*e*···O15*d* 104.5°] are somewhat weaker. The non-classical hydrogen bonds occur pairwise between O15 and one H atom at C7. Thus, *a* and *b* [O15*a*···H7*b*1 2.484 (2), H7*a*3···O15*b* 2.626 (2), O15*a*···C7*b* 3.406 (4) and O15*b*···C7*a* 3.417 (4) Å], *d* and *e* [O15*d*···H7*e*2 2.306 (2), H7*d*3···O15*e* 2.433 (3), O15*d*···C7*e* 3.268 (4) and O15*e*···C7*d* 3.324 (5) Å], as well as *c* and *f* [H7*c*1···O15*f* 2.437 (2), O15*c*···H7*f*1 2.613 (2), O15*f*···C7*c* 3.245 (4) and O15*c*···C7*f* 3.452 (4) Å] are linked by these probably weaker but still bonding C—H···O—C interactions.

In this regard it is of interest to note that in the (CH<sub>4</sub>···H<sub>2</sub>O) complex, in spite of H···O and C···O distances of 2.555 and 3.648 Å, the energy of the H<sub>3</sub>C—H···OH<sub>2</sub> bond was calculated to be  $-6.2$  kJ mol<sup>-1</sup> at a reasonably high level of quantum-chemical *ab initio* theory (MP4/6-311G\*\*) (Raabe, 1995). Previously, an even lower value of  $-7.2$  kJ mol<sup>-1</sup> was reported (Seiler *et al.*, 1987). The dimers are arranged in two different kinds of wavy chains  $\alpha$  (···*abedabed*···) and  $\beta$  (···*cfcfcfcf*···) (Fig. 2), propagating approximately parallel to the *c–a* vector (Fig. 3) and combining to result in layers of parquet-like appearance when viewed along the *a* axis (··· $\beta\alpha\bar{\alpha}\bar{\beta}\bar{\alpha}$ ···, where  $\bar{\alpha}$  and  $\bar{\beta}$  are the inverse of  $\alpha$  and  $\beta$ , respectively) (Fig. 2). Thus, each of the six independent species ‘sees’ a different environment resulting in different energetical conditions. Depending on the direction of view, the packing in the crystal appears either as structure with layers perpendicular (Fig. 3) or parallel (Fig. 4) to the *ac* plane.

Fig. 2. Packing of the molecules in the lattice. View along the *a* axis.Fig. 3. Packing diagram showing the layers perpendicular to the *ac* plane.Fig. 4. Packing diagram showing the layers parallel to the *ac* plane.

## Experimental

When triacetylmethane, (1), was subjected to anodic oxidation in an undivided electrolysis cell employing acetonitrile containing traces of triethylamine as solvent, the title compound (1*RS*,3*SR*,4*RS*,5*RS*,7*SR*,9*SR*)-4,7-diacetyl-9-hydroxy-1,3,5,9-tetramethyl-2,6,8-trioxatricyclo[3.2.1.1<sup>3,7</sup>]nonane, (2), was obtained in the form of colourless needles as the minor product, together with a 92% yield of (1*RS*,5*RS*)-2,6,6-triacetyl-1,3,5-trimethyl-4,7,8-trioxabicyclo[3.2.1]oct-2-ene, (3) (Thomas *et al.*, 1993).

### Crystal data

$C_{14}H_{20}O_6$   
 $M_r = 284.3$   
 Monoclinic  
 $P2_1/n$   
 $a = 8.676(1) \text{ \AA}$   
 $b = 44.948(2) \text{ \AA}$   
 $c = 22.143(2) \text{ \AA}$   
 $\beta = 99.369(5)^\circ$   
 $V = 8520(1) \text{ \AA}^3$   
 $Z = 24$   
 $D_x = 1.33 \text{ Mg m}^{-3}$   
 $D_m$  not measured

Cu  $K\alpha$  radiation

$\lambda = 1.54179 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 13.3\text{--}33.6^\circ$

$\mu = 0.831 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Irregular

$0.53 \times 0.49 \times 0.21 \text{ mm}$

Colourless

### Data collection

Enraf–Nonius CAD-4  
 diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction: none  
 17 485 measured reflections  
 16 793 independent reflections  
 9141 reflections with  
 $I_{net} > 2\sigma(I_{net})$

$R_{int} = 0.02$

$\theta_{max} = 74.99^\circ$

$h = -10 \rightarrow 10$

$k = 0 \rightarrow 56$

$l = 0 \rightarrow 27$

3 standard reflections

frequency: 60 min

intensity decay: none

### Refinement

Refinement on  $F$   
 $R = 0.059$   
 $wR = 0.067$   
 $S = 2.529$   
 9129 reflections  
 1082 parameters  
 H-atom treatment: see below  
 Weighting scheme based  
 on measured e.s.d.'s  
 $(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.458 \text{ e \AA}^{-3}$   
 $\Delta\rho_{min} = -0.65 \text{ e \AA}^{-3}$

Extinction correction:

Zachariasen (Zachariasen,  
 1967; Larson, 1970)

Extinction coefficient:

$4.2(3) \times 10^3$

Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

All H atoms except one could be located in a difference Fourier map and were subjected to five cycles of isotropic refinement.

Data collection: *CAD-4 Operators Guide* (Schagen, Straver, Van Meurs & Williams, 1988). Cell refinement: *CELDIM* (Rettig, 1988). Data reduction: *DIFDAT* (Stewart *et al.*, 1992), *SORTRF* (Hall, Spadaccini & Stewart, 1992) and *ADDRESS* (Davenport & Hall, 1992). Program(s) used to solve structure: *GENSIN* (Subramanian & Hall, 1992) and *GENTAN* (Hall, 1992a). Program(s) used to refine structure: *CRYLSQ* (Olthoff-Hazekamp, 1992). Molecular graphics: *ORTEP* (Davenport, Hall & Dreissig, 1992; Johnson, 1965). Software used to

Table 1. Corresponding bond lengths in the six symmetrically independent molecules of the asymmetric unit (Å)

 $\sigma^2$  is  $10^4$  times the dispersion (variance) calculated neglecting the standard deviations.

	<i>a</i>	<i>b</i>	<i>c</i>	<i>d</i>	<i>e</i>	<i>f</i>	$\sigma^2$
C1—C2	1.491 (6)	1.490 (6)	1.487 (7)	1.500 (6)	1.501 (6)	1.481 (6)	0.59
C2—O1	1.215 (5)	1.209 (5)	1.206 (5)	1.202 (5)	1.203 (5)	1.205 (5)	0.23
C2—C3	1.521 (5)	1.520 (5)	1.522 (5)	1.520 (5)	1.530 (5)	1.523 (5)	0.14
C3—C4	1.537 (5)	1.554 (5)	1.549 (5)	1.548 (5)	1.539 (5)	1.540 (5)	0.46
C3—C11	1.572 (4)	1.559 (5)	1.567 (5)	1.577 (5)	1.566 (5)	1.567 (5)	0.37
C4—O2	1.427 (4)	1.423 (4)	1.422 (4)	1.434 (4)	1.424 (4)	1.429 (4)	0.20
C4—O3	1.432 (4)	1.429 (4)	1.428 (4)	1.421 (4)	1.422 (4)	1.434 (5)	0.24
C4—C5	1.492 (5)	1.496 (5)	1.510 (5)	1.506 (5)	1.505 (5)	1.503 (5)	0.45
C6—O5	1.415 (4)	1.414 (4)	1.414 (4)	1.412 (4)	1.412 (4)	1.415 (4)	0.02
C6—O2	1.414 (4)	1.418 (4)	1.423 (4)	1.411 (4)	1.427 (4)	1.425 (4)	0.41
C6—C7	1.502 (5)	1.501 (5)	1.478 (5)	1.495 (5)	1.489 (5)	1.498 (5)	0.82
C6—C10	1.584 (5)	1.594 (5)	1.590 (5)	1.582 (5)	1.587 (5)	1.598 (5)	0.37
C8—O4	1.205 (4)	1.201 (4)	1.196 (5)	1.203 (5)	1.196 (5)	1.202 (4)	0.14
C8—C9	1.472 (5)	1.483 (6)	1.471 (7)	1.459 (6)	1.471 (6)	1.465 (6)	0.64
C8—C10	1.523 (5)	1.522 (5)	1.505 (5)	1.519 (5)	1.511 (5)	1.516 (5)	0.48
C10—O3	1.426 (4)	1.429 (4)	1.429 (4)	1.430 (4)	1.432 (4)	1.430 (4)	0.04
C10—C13	1.540 (4)	1.536 (5)	1.544 (5)	1.539 (5)	1.543 (5)	1.534 (5)	0.15
C11—O5	1.455 (4)	1.456 (4)	1.462 (4)	1.448 (4)	1.458 (4)	1.457 (4)	0.21
C11—C12	1.509 (5)	1.498 (5)	1.512 (4)	1.506 (5)	1.503 (5)	1.511 (5)	0.28
C11—C13	1.554 (5)	1.556 (5)	1.554 (5)	1.538 (5)	1.546 (5)	1.545 (5)	0.49
C13—O15	1.423 (4)	1.429 (4)	1.421 (4)	1.437 (4)	1.430 (4)	1.425 (4)	0.33
C13—C14	1.518 (5)	1.506 (5)	1.513 (5)	1.515 (5)	1.517 (5)	1.510 (5)	0.21

Table 2. Corresponding bond angles of the six symmetrically independent molecules in the asymmetric unit (°)

 $\sigma^2$  is the dispersion (variance) calculated neglecting the standard deviations.

	<i>a</i>	<i>b</i>	<i>c</i>	<i>d</i>	<i>e</i>	<i>f</i>	$\sigma^2$
O1—C2—C1	120.1 (3)	120.5 (3)	119.6 (4)	119.4 (4)	120.0 (4)	120.1 (4)	0.16
O1—C2—C3	122.4 (3)	122.5 (3)	122.9 (4)	122.8 (4)	122.4 (4)	122.8 (4)	0.05
C1—C2—C3	117.2 (3)	116.8 (3)	117.3 (4)	117.5 (3)	117.5 (4)	117.0 (4)	0.08
C2—C3—C4	114.3 (3)	113.8 (3)	113.5 (3)	113.5 (3)	113.5 (3)	114.2 (3)	0.14
C2—C3—C11	116.8 (3)	117.5 (3)	116.7 (3)	116.5 (3)	116.6 (3)	116.5 (3)	0.14
C4—C3—C11	108.3 (3)	108.5 (3)	108.1 (3)	108.1 (3)	108.5 (3)	108.3 (3)	0.03
O2—C4—O3	102.2 (2)	102.5 (3)	102.3 (2)	102.3 (2)	102.6 (2)	101.6 (2)	0.12
O2—C4—C5	110.1 (3)	109.0 (3)	108.8 (3)	108.6 (3)	109.1 (3)	110.1 (3)	0.43
O2—C4—C3	107.2 (3)	107.5 (3)	108.0 (3)	108.0 (3)	107.6 (3)	107.5 (3)	0.10
O3—C4—C5	110.8 (3)	111.5 (3)	110.6 (3)	110.7 (3)	110.0 (3)	110.1 (3)	0.29
O3—C4—C3	109.8 (3)	109.3 (3)	109.1 (3)	109.3 (3)	109.3 (3)	110.1 (3)	0.15
C5—C4—C3	115.9 (3)	116.1 (3)	117.0 (3)	116.9 (3)	117.1 (3)	116.4 (3)	0.25
O5—C6—O2	109.1 (3)	109.4 (3)	108.3 (3)	108.9 (3)	107.8 (3)	108.3 (3)	0.36
O5—C6—C7	110.0 (3)	110.3 (3)	110.9 (3)	110.2 (3)	109.9 (3)	110.9 (3)	0.19
O5—C6—C10	104.3 (2)	104.1 (3)	104.7 (3)	104.6 (3)	105.3 (3)	104.4 (3)	0.17
O2—C6—C7	109.4 (3)	109.5 (3)	109.9 (3)	110.4 (3)	110.3 (3)	109.5 (3)	0.19
O2—C6—C10	102.1 (2)	101.9 (3)	102.0 (3)	102.1 (2)	102.1 (2)	101.8 (3)	0.02
C7—C6—C10	121.2 (3)	120.9 (3)	120.2 (3)	120.1 (3)	120.6 (3)	121.0 (3)	0.20
O4—C8—C9	121.8 (4)	121.3 (4)	120.5 (4)	121.2 (4)	121.8 (4)	121.3 (4)	0.23
O4—C8—C10	119.7 (3)	120.0 (3)	121.2 (4)	119.9 (4)	119.9 (4)	119.9 (3)	0.30
C9—C8—C10	118.5 (3)	118.8 (3)	118.2 (3)	118.9 (3)	118.2 (3)	118.8 (3)	0.10
O3—C10—C8	109.8 (3)	110.0 (3)	110.2 (3)	109.0 (3)	110.0 (3)	110.5 (3)	0.26
O3—C10—C13	107.1 (2)	107.4 (2)	107.7 (3)	107.4 (2)	107.6 (3)	107.3 (2)	0.05
O3—C10—C6	103.8 (2)	103.5 (3)	103.3 (3)	103.8 (3)	103.4 (3)	103.2 (3)	0.06
C8—C10—C13	116.0 (3)	116.4 (3)	115.4 (3)	115.9 (3)	115.9 (3)	116.2 (3)	0.11
C8—C10—C6	115.8 (3)	115.4 (3)	116.4 (3)	116.8 (3)	116.7 (3)	115.6 (3)	0.35
C13—C10—C6	103.3 (3)	103.0 (3)	102.7 (3)	102.9 (3)	102.1 (3)	102.9 (3)	0.16
O5—C11—C12	108.7 (3)	108.9 (3)	107.8 (3)	108.3 (3)	107.6 (3)	108.0 (3)	0.26
O5—C11—C13	99.3 (2)	99.0 (2)	99.0 (3)	99.9 (3)	99.3 (3)	99.9 (2)	0.17
O5—C11—C3	104.3 (2)	104.5 (3)	104.9 (3)	104.8 (3)	104.8 (3)	104.4 (3)	0.06
C12—C11—C13	115.0 (3)	115.4 (3)	115.1 (3)	115.4 (3)	115.4 (3)	115.4 (3)	0.03
C12—C11—C3	113.9 (3)	113.8 (3)	114.5 (3)	114.5 (3)	114.7 (3)	114.1 (3)	0.14
C13—C11—C3	113.8 (3)	113.5 (3)	113.5 (3)	112.2 (3)	113.0 (3)	113.2 (3)	0.32
O15—C13—C14	107.0 (3)	108.0 (3)	108.0 (3)	107.5 (3)	106.9 (3)	107.7 (3)	0.23
O15—C13—C10	112.9 (2)	111.1 (2)	112.2 (3)	112.3 (3)	110.9 (3)	112.5 (3)	0.64
O15—C13—C11	107.6 (3)	107.5 (3)	107.1 (3)	105.7 (3)	108.9 (3)	106.3 (3)	1.24
C14—C13—C10	114.0 (3)	113.9 (3)	114.3 (3)	114.5 (3)	114.3 (3)	113.9 (3)	0.06
C14—C13—C11	118.1 (3)	118.4 (3)	117.4 (3)	118.8 (3)	117.9 (3)	118.2 (3)	0.22
C10—C13—C11	97.0 (3)	97.5 (3)	97.4 (3)	97.7 (3)	97.8 (3)	97.9 (3)	0.11
C6—O2—C4	103.4 (2)	103.2 (2)	103.4 (2)	103.1 (2)	103.1 (2)	103.6 (2)	0.04
C10—O3—C4	103.6 (2)	103.6 (2)	104.0 (2)	103.2 (2)	103.9 (2)	104.1 (2)	0.11
C6—O5—C11	104.8 (2)	105.2 (2)	104.9 (2)	105.2 (2)	104.6 (2)	105.0 (2)	0.06

prepare material for publication: *BONDLA* (Dreissig, Doherty, Stewart & Hall, 1992), *ATABLE* (Englehardt & Hall, 1992) and *CIFIO* (Hall, 1992b).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: KA1225). Services for accessing these data are described at the back of the journal.

## References

- Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.
- Davenport, G. & Hall, S. R. (1992). *ADDREF. Xtal3.2 Reference Manual*, edited by S. R. Hall, H. D. Flack & J. M. Stewart, pp. 53–59. Universities of Western Australia, Australia, Geneva, Switzerland, and Maryland, USA.
- Davenport, G., Hall, S. R. & Dreissig, W. (1992). *ORTEP. Xtal3.2 Reference Manual*, edited by S. R. Hall, H. D. Flack & J. M. Stewart, pp. 219–224. Universities of Western Australia, Australia, Geneva, Switzerland, and Maryland, USA.
- Dreissig, W., Doherty, R., Stewart, J. M. & Hall, S. R. (1992). *BONDLA. Xtal3.2 Reference Manual*, edited by S. R. Hall, H. D. Flack & J. M. Stewart, pp. 69–71. Universities of Western Australia, Australia, Geneva, Switzerland, and Maryland, USA.
- Englehardt, L. & Hall, S. R. (1992). *ATABLE. Xtal3.2 Reference Manual*, edited by S. R. Hall, H. D. Flack & J. M. Stewart, p. 61. Universities of Western Australia, Australia, Geneva, Switzerland, and Maryland, USA.
- Hall, S. R. (1992a). *GENTAN. Xtal3.2 Reference Manual*, edited by S. R. Hall, H. D. Flack & J. M. Stewart, pp. 139–150. Universities of Western Australia, Australia, Geneva, Switzerland, and Maryland, USA.
- Hall, S. R. (1992b). *CIFIO. Xtal3.2 Reference Manual*, edited by S. R. Hall, H. D. Flack & J. M. Stewart, pp. 79–82. Universities of Western Australia, Australia, Geneva, Switzerland, and Maryland, USA.
- Hall, S. R., Spadaccini, N. & Stewart, J. M. (1992). *SORTRF. Xtal3.2 Reference Manual*, edited by S. R. Hall, H. D. Flack & J. M. Stewart, pp. 306–309. Universities of Western Australia, Australia, Geneva, Switzerland, and Maryland, USA.
- Hester, J. R. & Hall, S. R. (1996). *J. Appl. Cryst.* **29**, 474–478.
- Jeffrey, G. A. & Saenger, W. (1991). *Hydrogen Bonding in Biological Structures*, pp. 156–160. Berlin: Springer-Verlag.
- Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- Larson, A. C. (1970). *Crystallographic Computing*, edited by F. R. Ahmed, S. R. Hall & C. P. Huber, pp. 291–294. Copenhagen: Munksgaard.
- Le Page, Y. (1988). *J. Appl. Cryst.* **21**, 983–984.
- Olthof-Hazekamp, R. (1992). *CRYLSQ. Xtal3.2 Reference Manual*, edited by S. R. Hall, H. D. Flack & J. M. Stewart, pp. 93–104. Universities of Western Australia, Australia, Geneva, Switzerland, and Maryland, USA.
- Raabe, G. (1995). Unpublished results.
- Rettig, S. (1988). *CELDIM. Enraf-Nonius CAD-4 Operators Guide*. Enraf-Nonius Scientific Instrument Division, 2600 AL Delft, The Netherlands.
- Schagen, J. D., Straver, L., Van Meurs, F. & Williams, G. (1988). *Enraf-Nonius CAD-4 Operators Guide*. Enraf-Nonius Scientific Instrument Division, 2600 AL Delft, The Netherlands.
- Seiler, P., Weisman, G. R., Glendening, E. D., Weinhold, F., Johnson, V. B. & Dunitz, J. D. (1987). *Angew. Chem.* **99**, 1216–1218.
- Simons, J. (1992). Doctoral thesis, Rheinisch-Westfälische Technische Hochschule Aachen, Germany.
- Stewart, J., Merom, R., Holden, J., Doherty, R., Hall, S. R., Maslen, E. & Spadaccini, N. (1992). *DIFDAT. Xtal3.2 Reference Manual*, edited by S. R. Hall, H. D. Flack & J. M. Stewart, pp. 104–110. Universities of Western Australia, Australia, Geneva, Switzerland, and Maryland, USA.
- Subramanian, V. & Hall, S. R. (1992). *GENSIN. Xtal3.2 Reference Manual*, edited by S. R. Hall, H. D. Flack & J. M. Stewart, pp. 131–139. Universities of Western Australia, Australia, Geneva, Switzerland, and Maryland, USA.
- Taylor, R. & Kennard, O. (1982). *J. Am. Chem. Soc.* **104**, 5063–5070.
- Thomas, H. G., Wellen, U., Simons, J. & Raabe, G. (1993). *Synthesis*, pp. 1113–1120.
- Zachariasen, W. H. (1967). *Acta Cryst.* **23**, 558–564.

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## Tetramethylammonium Hydrogen Bis[bis(dimethyldithiocarbamyl)acetate]†

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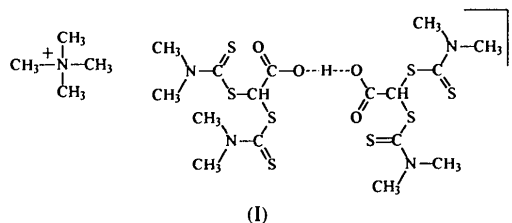
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### Abstract

The two carboxylate anions in the title compound,  $C_4H_{12}N^+ \cdot C_{16}H_{27}N_4O_4S_8^-$ , are linked through an H atom which lies on a center of inversion [ $O \cdots O$  2.484 (3) Å].

### Comment

In an earlier study, the C—O and C=O bonds [1.232 (2)–1.241 (3) Å] of the  $-CO_2$  group in both independent ion pairs of dicyclohexylammonium bis(*N,N*-dimethyldithiocarbamyl)acetate were found to be indistinguishable from one another (Ng, 1996). The anions are linked through the ammonium N atom across a center of inversion into a dimeric ion pair having an O—C—O $\cdots$ N $\cdots$ O—C—O $\cdots$ N ring. Such hydrogen bonding through N atoms is not possible with the title compound, (I), which crystallizes with a neutral acid molecule.



† Alternative name: tetramethylammonium hydrogen bis[bis(*N,N*-dimethyldithiocarbamylthio)acetate].