

Table 5. Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ )

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
RT O1—H1 $\cdots$ O2 <sup>i</sup>	1.08 (4)	1.55 (4)	2.623 (2)	174 (3)
LT O1—H1 $\cdots$ O2 <sup>i</sup>	1.13 (3)	1.49 (3)	2.614 (2)	174 (2)

Symmetry codes: (i)  $2 - x, y, -\frac{1}{2} - z$ .

Scan widths were  $(1.45 + 0.35 \tan\theta)^\circ$  in  $\omega$  (RT) and  $(1.50 + 0.35 \tan\theta)^\circ$  in  $\omega$  (LT), with background to scan time ratios of 0.5. The data were corrected for Lorentz and polarization effects. For each of the crystals, the Laue group assignment, systematic absences and intensity statistics consistent with centrosymmetry indicated space group  $C2/c$  and since refinement proceeded well, it was adopted in each case. Fourier difference methods were used to locate the H-atom positions. Full-matrix least-squares refinement was performed. In the later stages of refinement, ring H atoms were made canonical:  $C-H = 0.98 \text{\AA}$  and  $U_{iso} = 1.2U_{eq}$  of the attached atom. The carboxyl H atom was refined isotropically. For the crystal of RT, the maximum effect of extinction was 57% of  $F_o$  for 020 and for the crystal of LT, it was 7.6% of  $F_o$  for 020.

For both compounds, data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1989); program(s) used to solve structures: *MITHRIL* (Gilmore, 1983); program(s) used to refine structures: *TEXSAN*; molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *TEXSAN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1218). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## *N*-Saccharinperacetic Acid Monohydrate and *N*-Saccharinpentanoic Acid Monohydrate

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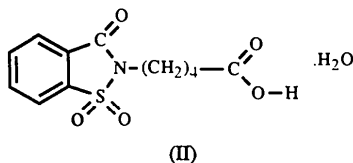
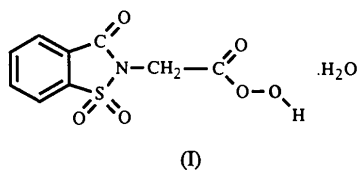
## Abstract

Hydrogen bonds involving water molecules dominate the crystal structures of *N*-saccharinperacetic acid monohydrate (3-oxo-2*H*-1,2-benzisothiazole-2-peroxyethanoic acid 1,1-dioxide monohydrate,  $C_9H_7NO_6S.H_2O$ ) and *N*-saccharinpentanoic acid monohydrate (3-oxo-2*H*-1,2-benzisothiazole-2-pentanoic acid 1,1-dioxide monohydrate,  $C_{12}H_{13}NO_5S.H_2O$ ). In both structures, molecules are held together by these hydrogen bonds to form two-dimensional blocks. Interdigitation of saccharin groups between adjacent blocks completes the three-dimensional structure.

## Comment

As part of a general comparative study of the hydrogen-bond networks formed in organic carboxylic acids and peracids, we have previously reported on the crystal structures of a number of *N*-saccharin acids (Feeder & Jones, 1994*a*) and *N*-saccharin peracids (Feeder & Jones, 1994*b*). We report here the structures of *N*-saccharinperacetic acid monohydrate, (I), and *N*-saccharinpentanoic acid monohydrate, (II). With these structures it has been possible to characterize the structural features relating to the additional hydrogen-bonding provided by the water molecules.

The bond lengths and angles within the saccharin groups are similar to those found for the series of *N*-saccharin acids (Feeder & Jones, 1994*a*) and *N*-saccharin peracids (Feeder & Jones, 1994*b*), as well as



to those of saccharin (Okaya, 1969), *N*-cyanomethylsaccharin (Junii, Guilan, Zhongyuan & Liu, 1989) and (*R*)-(+)-supidimide (Winter, Graudums & Frankus, 1983). The saccharin groups (C1–C7, N1, S1, O7) are planar [to within 0.030 (2) Å for (I) and 0.035 (2) Å for (II)] and the two sulfone O atoms (O1 and O2) lie approximately 1.2 Å above and below these planes. The bond lengths and angles associated with the peracid function of (I) are not significantly different to those previously reported for two *N*-saccharin peracids (Feeder & Jones, 1994*b*), a series of  $\omega$ -phthalimidoaliphatic peracids (Feeder & Jones, 1996*a*), a series of *p*-amidoperbenzoic acids (Feeder & Jones, 1996*b*), peroxyperlargonic acid (Belitskus & Jeffrey, 1965), *o*-nitroperoxybenzoic acid (Sax, Beurskens & Chu, 1965) and *p*-nitroperoxybenzoic acid (Kim, Chu & Jeffrey, 1970). The peracid group (C9, O90–O92) is planar to within 0.007 (2) Å.

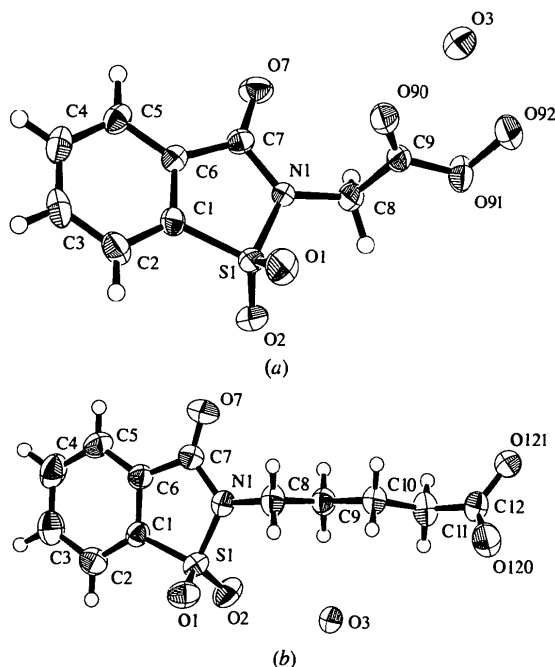


Fig. 1. ORTEP (Johnson, 1965) plots of (a) (I) and (b) (II) showing the atomic numbering schemes and 50% probability displacement ellipsoids.

Although the acid/peracid and water protons could not be located, the hydrogen-bond networks present in the structures of (I) and (II) can be identified from O...O intermolecular contacts (Tables 2 and 4). In both structures, the hydrogen-bond networks are complex but share similar features. For (II), the hydrogen-bond network extends parallel to the *a* axis (Fig. 2). Hydrogen bonds also extend parallel to the *b* axis and link molecules into blocks. A similar two-dimensional structure is found for (I), parallel to the (10 $\bar{1}$ ) plane. For (II), interdigitation of saccharin groups between adjacent blocks related by translation along the *c* axis completes the three-dimensional structure. A similar interaction between blocks is observed in the structure of (I).

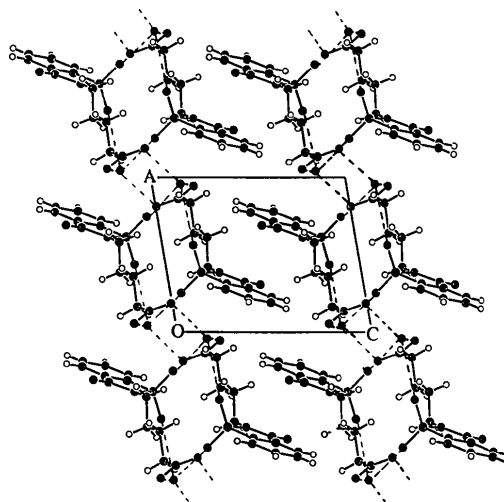


Fig. 2. Packing diagram for (II) viewed down the *b* axis. Hydrogen bonds extend parallel to the *a* axis and also parallel to the *b* axis (into the plane of the paper). O...O contacts are represented by dashed lines.

## Experimental

Both title compounds were supplied by Solvay Interlox. The hydrate crystals of (I) and (II) were grown by slow evaporation of ethyl acetate/water and ethanol/water solutions, respectively.

## Compound (I)

### Crystal data

C<sub>9</sub>H<sub>7</sub>NO<sub>6</sub>S.H<sub>2</sub>O

$M_r = 275.23$

Monoclinic

$P2_1/n$

$a = 9.013 (3) \text{ \AA}$

$b = 7.055 (4) \text{ \AA}$

$c = 18.118 (2) \text{ \AA}$

$\beta = 98.51 (2)^\circ$

$V = 1139.4 (7) \text{ \AA}^3$

$Z = 4$

$D_x = 1.604 \text{ Mg m}^{-3}$

$D_m$  not measured

Mo  $K\alpha$  radiation

$\lambda = 0.71069 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 15\text{--}20^\circ$

$\mu = 0.312 \text{ mm}^{-1}$

$T = 173 (2) \text{ K}$

Rod

$0.40 \times 0.15 \times 0.15 \text{ mm}$

Colourless

**Data collection**

Rigaku AFC-7R diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction: none  
 2141 measured reflections  
 2006 independent reflections  
 1585 observed reflections  
 $[I > 2\sigma(I)]$

$R_{\text{int}} = 0.0191$   
 $\theta_{\text{max}} = 24.99^\circ$   
 $h = 0 \rightarrow 10$   
 $k = 0 \rightarrow 8$   
 $l = -21 \rightarrow 21$   
 3 standard reflections  
 monitored every 200 reflections  
 intensity decay: 0.8%

**Triclinic**

$P\bar{1}$   
 $a = 8.412(3) \text{ \AA}$   
 $b = 9.190(3) \text{ \AA}$   
 $c = 10.880(6) \text{ \AA}$   
 $\alpha = 66.74(3)^\circ$   
 $\beta = 88.86(3)^\circ$   
 $\gamma = 64.67(3)^\circ$   
 $V = 687.1(5) \text{ \AA}^3$   
 $Z = 2$   
 $D_x = 1.456 \text{ Mg m}^{-3}$   
 $D_m$  not measured

**Cell parameters from 25 reflections**

$\theta = 9\text{--}14^\circ$   
 $\mu = 0.26 \text{ mm}^{-1}$   
 $T = 295(2) \text{ K}$   
 Plate  
 $0.50 \times 0.50 \times 0.20 \text{ mm}$   
 Colourless

**Refinement**

Refinement on  $F^2$   
 $R(F) = 0.0466$   
 $wR(F^2) = 0.1340$   
 $S = 1.046$   
 2001 reflections  
 165 parameters  
 H-atom  $U$ 's refined  
 $w = 1/[\sigma^2(F_o^2) + (0.0649P)^2 + 0.8779P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = -0.001$   
 $\Delta\rho_{\text{max}} = 0.541 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.293 \text{ e \AA}^{-3}$   
 Extinction correction: none  
 Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

**Data collection**

Enraf-Nonius CAD-4 diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction: none  
 2538 measured reflections  
 2402 independent reflections  
 1903 observed reflections  
 $[I > 2\sigma(I)]$

$R_{\text{int}} = 0.014$   
 $\theta_{\text{max}} = 24.98^\circ$   
 $h = -9 \rightarrow 9$   
 $k = -10 \rightarrow 9$   
 $l = -9 \rightarrow 12$   
 2 standard reflections  
 monitored every 100 reflections  
 intensity decay: 3.8%

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ ) for (I)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}$
S1	0.91106(8)	0.21943(12)	0.84378(4)	0.0339(3)
O1	0.8963(3)	0.3899(4)	0.80109(13)	0.0542(7)
O2	0.9379(3)	0.0474(4)	0.80692(12)	0.0498(7)
O3	0.3683(3)	0.8067(3)	0.85038(12)	0.0439(6)
O7	0.6899(2)	0.1841(3)	1.00258(11)	0.0409(6)
O90	0.5281(2)	0.4588(3)	0.86484(12)	0.0446(6)
O91	0.3954(2)	0.2617(3)	0.78276(13)	0.0439(6)
O92	0.2856(2)	0.4179(4)	0.77485(13)	0.0480(6)
N1	0.7608(3)	0.1927(4)	0.88669(12)	0.0300(6)
C1	1.0299(3)	0.2488(4)	0.9287(2)	0.0306(7)
C2	1.1825(3)	0.2778(5)	0.9380(2)	0.0420(8)
C3	1.2540(4)	0.2995(5)	1.0109(2)	0.0472(9)
C4	1.1758(4)	0.2914(5)	1.0708(2)	0.0435(8)
C5	1.0217(4)	0.2632(4)	1.0598(2)	0.0346(7)
C6	0.9489(3)	0.2412(4)	0.9874(2)	0.0272(6)
C7	0.7870(3)	0.2044(4)	0.9637(2)	0.0286(6)
C8	0.6166(3)	0.1425(5)	0.8447(2)	0.0373(8)
C9	0.5123(3)	0.3115(5)	0.8328(2)	0.0325(7)

Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ) for (I)

S1—O2	1.423(3)	O91—O92	1.474(3)
S1—O1	1.425(3)	O3...O92 <sup>i</sup>	2.599(3)
S1—N1	1.669(2)	O3...O90	2.839(3)
O90—C9	1.189(4)	O3...O7 <sup>ii</sup>	2.791(3)
O91—C9	1.332(4)		
O2—S1—O1	118.27(15)	C7—N1—S1	115.7(2)
O2—S1—N1	109.15(14)	O90—C9—O91	126.1(3)
O1—S1—N1	109.33(14)	O90—C9—C8	126.3(3)
N1—S1—C1	92.15(13)	O91—C9—C8	107.5(3)
C9—O91—O92	108.7(2)		
C7—N1—C8—C9	82.8(3)	O92—O91—C9—C8	175.1(2)
S1—N1—C8—C9	-103.3(3)	N1—C8—C9—O90	-14.8(4)
O92—O91—C9—O90	-1.5(4)	N1—C8—C9—O91	168.6(2)

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

**Compound (II)****Crystal data**

$\text{C}_{12}\text{H}_{13}\text{NO}_5\text{S}\cdot\text{H}_2\text{O}$   
 $M_r = 301.31$

Mo  $K\alpha$  radiation  
 $\lambda = 0.71073 \text{ \AA}$

**Refinement**

Refinement on  $F^2$   
 $R(F) = 0.0455$   
 $wR(F^2) = 0.1473$   
 $S = 1.121$   
 2387 reflections  
 186 parameters  
 H-atom  $U$ 's refined  
 $w = 1/[\sigma^2(F_o^2) + (0.0843P)^2 + 0.2655P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.154$   
 $\Delta\rho_{\text{max}} = 0.436 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.480 \text{ e \AA}^{-3}$   
 Extinction correction: none  
 Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 3. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ ) for (II)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}$
S1	0.38639(9)	0.57878(9)	0.17691(7)	0.0403(2)
N1	0.3861(3)	0.3867(3)	0.2714(2)	0.0440(6)
O1	0.2543(3)	0.6740(3)	0.0561(2)	0.0548(6)
O2	0.5632(3)	0.5519(3)	0.1589(2)	0.0591(6)
O7	0.3093(4)	0.2486(3)	0.4702(2)	0.0718(7)
O120	0.8126(3)	-0.1996(3)	0.0065(2)	0.0618(6)
O121	0.9453(3)	-0.4102(3)	0.2152(2)	0.0501(5)
C1	0.3143(3)	0.6618(4)	0.2981(3)	0.0374(6)
C2	0.2803(4)	0.8272(4)	0.2882(3)	0.0477(7)
C3	0.2203(5)	0.8640(5)	0.3969(4)	0.0580(8)
C4	0.1937(5)	0.7433(5)	0.5071(3)	0.0607(9)
C5	0.2261(4)	0.5799(4)	0.5145(3)	0.0542(8)
C6	0.2869(4)	0.5395(4)	0.4081(3)	0.0403(6)
C7	0.3252(4)	0.3737(4)	0.3934(3)	0.0462(7)
C8	0.4181(4)	0.2559(4)	0.2165(3)	0.0504(7)
C9	0.6138(4)	0.1278(4)	0.2397(3)	0.0430(7)
C10	0.6419(4)	-0.0010(4)	0.1779(3)	0.0434(7)
C11	0.8361(4)	-0.1131(4)	0.1835(3)	0.0520(8)
C12	0.8634(4)	-0.2433(4)	0.1256(3)	0.0447(7)
O3	0.0418(3)	0.6576(3)	0.8514(2)	0.0590(6)

Table 4. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ) for (II)

S1—O2	1.422(2)	O3...O121 <sup>i</sup>	2.606(4)
S1—O1	1.430(2)	O3...O120 <sup>ii</sup>	2.783(4)
S1—N1	1.656(3)	O3...O1 <sup>iii</sup>	2.971(2)
O120—C12	1.219(4)	O3...O2 <sup>iv</sup>	3.073(2)
O121—C12	1.311(4)		

O2—S1—O1	116.58 (14)	C7—N1—S1	115.1 (2)
O2—S1—N1	110.51 (14)	O120—C12—O121	122.6 (3)
O1—S1—N1	109.88 (13)	O120—C12—C11	123.5 (3)
N1—S1—C1	93.07 (12)	O121—C12—C11	113.9 (3)
C7—N1—C8—C9	-101.8 (3)	C10—C11—C12—O120	64.7 (4)
S1—N1—C8—C9	89.2 (3)	C10—C11—C12—O121	-114.4 (3)

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

In both structures, the phenyl and alkyl H atoms were placed geometrically at the expected positions, riding on the attached C atoms, and refined with isotropic displacement parameters. The other H atoms could not be located.

Data collection: Rigaku AFC-7R software for (I); CAD-4 Software (Enraf-Nonius, 1989) for (II). Cell refinement: Rigaku AFC-7R software for (I); SETANG (Enraf-Nonius, 1989) for (II). For both compounds, data reduction: TEXSAN (Molecular Structure Corporation, 1995); program(s) used to solve structures: SHELXS86 (Sheldrick, 1990); program(s) used to refine structures: SHELXL93 (Sheldrick, 1993); molecular graphics: TEXSAN; software used to prepare material for publication: SHELXL93.

The authors acknowledge support from the SERC (studentship for NF) and are grateful to Solvay Interlox (and in particular Dr A. James) for the provision of samples and numerous discussions.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: HA1157). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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*Acta Cryst.* (1996). **C52**, 2326–2332

## Ammonium Salts of Phosphoglycolic Acid (H<sub>3</sub>PG): NH<sub>4</sub>.H<sub>2</sub>PG, 3NH<sub>4</sub>.H<sub>2</sub>PG.HPG, 2NH<sub>4</sub>.HPG and 3NH<sub>4</sub>.PG.H<sub>2</sub>O at 150 K

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## Abstract

The structures have been determined of four ammonium phosphoglycolate (PG) salts: ammonium phosphoglycolate(1<sup>-</sup>), NH<sub>4</sub>.H<sub>2</sub>PG (I), triammonium phosphoglycolate(1<sup>-</sup>) phosphoglycolate(2<sup>-</sup>), 3NH<sub>4</sub>.H<sub>2</sub>PG.HPG (II), diammonium phosphoglycolate(2<sup>-</sup>), 2NH<sub>4</sub>.HPG (III) and triammonium phosphoglycolate(3<sup>-</sup>) monohydrate, 3NH<sub>4</sub>.PG.H<sub>2</sub>O (IV) (PG = C<sub>2</sub>H<sub>2</sub>O<sub>6</sub>P<sup>3-</sup>). The P—O(ester) bond in the doubly ionized phosphate group of (IV) [1.625 (2) Å] is longer than those in mono-ionized phosphate groups of (I)–(III) [1.580 (2)–1.600 (1) Å]. There are extensive networks of hydrogen bonds in all four crystals in which every O- and N-bonded H atom is involved.

## Comment

This work is part of a systematic study of the structures of phosphoglycolate residues in different ionization states and different chemical environments (Lis, 1993, 1995). The triammonium and other phosphoglycolate and phosphoenolpyruvate salts are now the subject of investigations aimed at finding the experimental electron density in organic phosphate esters (Lis & Starynowicz, 1995). The structures of (I), (II), (III) and (IV) are reported here. The same atom-numbering scheme has been used for each phosphoglycolate (PG) residue.

Crystals of (I) are composed of phosphoglycolate H<sub>2</sub>PG<sup>-</sup> monoanions (Fig. 1) and ammonium cations. As in most PG moieties, the carboxylic hydroxyl group is *trans* to the O2 ester atom (Lis, 1993), although in potassium phosphoglycolate these groups are *cis* to one another. All O- and N-bonded H atoms are utilized in intermolecular hydrogen bonding (Table 2, Fig. 2). The O4 atoms (as donors) form infinite chains with O1<sup>i</sup> atoms along *a*. The O6 atoms (as donors) form infinite chains with O1<sup>ii</sup> along the (101) direction. The NH<sub>4</sub><sup>+</sup> cations hydrogen-bond through carboxylic O5 and phosphate O3 atoms to four symmetry-related PG moieties.

The asymmetric unit of (II) contains three ammonium cations, a phosphoglycolate monoanion [denoted *A*; Fig. 3(a)] and a phosphoglycolate dianion [denoted *B*;