Monoclinic
$P2_1$
a = 9.873 (1) Å
b = 6.129 (1) Å
c = 11.594 (1) Å
β = 108.23 (1)°
V = 666.4 (1) Å^3
Z = 2
D_x = 1.232 Mg m^{-3}

Cell parameters from 40 reflections
θ = 10–12.5°
μ = 0.08 mm^{-1}
T = 293 K
Block
0.8 × 0.8 × 0.3 mm
Colourless, transparent
Crystal source: cyclohexane/ethyl acetate

Data collection

Stoe four-circle diffractometer
$\omega/2\theta$ scans with profile fitting (Clegg, 1981)
Absorption correction: none
1434 measured reflections
1364 independent reflections
1328 observed reflections [$F > 3\sigma(F)$]

$R_{\rm int}$ = 0.03
$\theta_{\rm max}$ = 25°
$h = -11 \rightarrow 11$
$k = -7 \rightarrow 1$
$l = 0 \rightarrow 13$
3 standard reflections monitored every 100 reflections
intensity variation: none

Refinement

Refinement on F
R = 0.032
wR = 0.045
S = 1.79
1328 reflections
172 parameters
Position of H(N) refined, positions of H(C) fixed
$w = 1/[\sigma^2(F) + 0.0005F^2]$
$(\Delta/\sigma)_{\rm max}$ = 0.03

$\Delta\rho_{\rm max}$ = 0.12 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.14 e Å^{-3}
Extinction correction: $F^* = F[1 + (0.002\chi \times F^2/\sin 2\theta)]^{-1/4}$
Extinction coefficient: χ = 0.06 (2)
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
O(1)	0.7943 (1)	0.5299†	0.3704 (1)	0.046 (1)
C(2)	0.8848 (2)	0.4104 (4)	0.4567 (2)	0.040 (1)
C(3)	0.8346 (2)	0.3288 (4)	0.5593 (2)	0.041 (1)
N(4)	0.6795 (1)	0.3046 (3)	0.5190 (1)	0.041 (1)
C(5)	0.5920 (2)	0.4421 (4)	0.4433 (1)	0.040 (1)
C(6)	0.6641 (2)	0.6148 (4)	0.3879 (2)	0.042 (1)
O(21)	1.0006 (1)	0.3696 (3)	0.4496 (1)	0.052 (1)
O(51)	0.4612 (1)	0.4351 (4)	0.4190 (1)	0.051 (1)
C(61)	0.5720 (2)	0.6963 (5)	0.2644 (2)	0.055 (1)
C(62)	0.5353 (2)	0.5156 (6)	0.1702 (2)	0.070 (1)
C(63)	0.6456 (3)	0.8885 (6)	0.2243 (3)	0.082 (1)
C(1′)	0.8902 (2)	0.4804 (5)	0.6695 (2)	0.050 (1)
C(2′)	0.8540 (2)	0.4041 (5)	0.7804 (2)	0.046 (1)
C(3′)	0.8989 (2)	0.2054 (5)	0.8338 (2)	0.056 (1)
C(4′)	0.8727 (2)	0.1453 (5)	0.9402 (2)	0.068 (1)
C(5′)	0.8000 (3)	0.2826 (6)	0.9929 (2)	0.071 (1)
C(6′)	0.7529 (2)	0.4813 (6)	0.9397 (2)	0.069 (1)
C(7′)	0.7787 (2)	0.5400 (5)	0.8333 (2)	0.056 (1)

† Coordinate fixed to define origin.

Table 2. *Selected geometric parameters* (Å, °)

O(1)—C(2)	1.333 (2)	O(1)—C(6)	1.459 (2)
C(2)—C(3)	1.510 (2)	C(2)—O(21)	1.197 (2)
C(3)—N(4)	1.461 (2)	C(3)—C(1′)	1.535 (3)
N(4)—C(5)	1.323 (2)	C(5)—C(6)	1.526 (2)
C(5)—O(51)	1.233 (2)	C(6)—C(61)	1.520 (3)
C(61)—C(62)	1.518 (4)	C(61)—C(63)	1.531 (4)
C(1′)—C(2′)	1.512 (2)	C(2′)—C(3′)	1.376 (3)
C(2′)—C(7′)	1.381 (3)	C(3′)—C(4′)	1.387 (3)
C(4′)—C(5′)	1.368 (4)	C(5′)—C(6′)	1.379 (5)
C(6′)—C(7′)	1.384 (3)		
C(6)—O(1)—C(2)	120.3 (1)	C(3)—C(2)—O(1)	117.7 (1)
O(21)—C(2)—O(1)	119.5 (1)	O(21)—C(2)—C(3)	122.8 (2)
N(4)—C(3)—C(2)	110.5 (1)	C(1′)—C(3)—C(2)	109.6 (1)
C(1′)—C(3)—N(4)	113.2 (1)	C(5)—N(4)—C(3)	123.3 (1)
C(6)—C(5)—N(4)	115.3 (1)	O(51)—C(5)—N(4)	123.3 (2)
O(51)—C(5)—C(6)	121.4 (2)	C(5)—C(6)—O(1)	111.0 (1)
C(61)—C(6)—O(1)	106.2 (1)	C(61)—C(6)—C(5)	113.8 (1)
C(62)—C(61)—C(6)	112.1 (2)	C(63)—C(61)—C(6)	109.7 (2)
C(63)—C(61)—C(62)	111.8 (2)	C(2′)—C(1′)—C(3)	113.7 (2)
C(3′)—C(2′)—C(1′)	121.9 (2)	C(7′)—C(2′)—C(1′)	119.6 (2)
C(7′)—C(2′)—C(3′)	118.4 (2)	C(4′)—C(3′)—C(2′)	120.8 (2)
C(5′)—C(4′)—C(3′)	120.2 (2)	C(6′)—C(5′)—C(4′)	119.6 (2)
C(7′)—C(6′)—C(5′)	119.9 (2)	C(6′)—C(7′)—C(2′)	121.0 (2)

The structure was solved by direct methods. All H atoms were located using a difference Fourier synthesis and were refined with fixed individual displacement parameters [U(H) = 1.2 × $U_{\rm eq}$(C, N)] using a riding model with C—H = 0.96 Å. The coordinates of the amide H atom were refined with the N—H distance restrained to 0.96 Å. Anisotropic full-matrix refinement was carried out using *XLS* (Sheldrick, 1987).

We thank Professor U. Schöllkopf (University of Göttingen) for providing the sample.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: SH1078). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Clegg, W. (1981). *Acta Cryst.* A**37**, 22–28.
Maywald, V. (1987). PhD thesis, Univ. of Göttingen, Germany.
Sheldrick, G. M. (1987). *XLS. Expanded Version of SHELX76.* Univ. of Göttingen, Germany.

Acta Cryst. (1994). C**50**, 1118–1122

Four *N*-Saccharinaliphatic Acids

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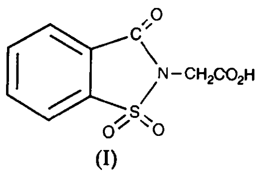
Abstract

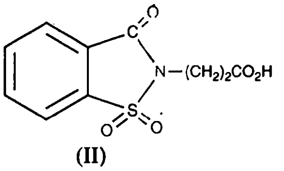
The crystal structures of *N*-saccharinacetic acid (3-oxo-2*H*-1,2-benzisothiazol-2-acetic acid 1,1-dioxide),

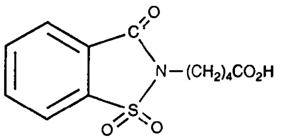
$C_9H_7NO_5S$, *N*-saccharinpropanoic acid, $C_{10}H_9NO_5S$, *N*-saccharinpentanoic acid, $C_{12}H_{13}NO_5S$, and *N*-saccharinhexanoic acid, $C_{13}H_{15}NO_5S$, have been determined. The carboxylic acid groups are hydrogen bonded to form the familiar eight-membered centrosymmetric ring dimer motif in each of the four structures.

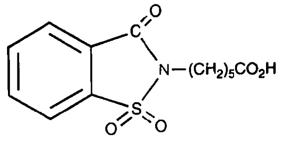
Comment

As part of a general study of the stability of organic peracids and peracid salts (Kariuki & Jones, 1990) and in particular the role of hydrogen bonding in controlling the crystal structure of peracid and acid amides (Feeder & Jones, 1992), we report here the structures of four *N*-saccharinaliphatic acids: *N*-saccharinacetic acid (I), *N*-saccharinpropanoic acid (II), *N*-saccharinpentanoic acid (III) and *N*-saccharinhexanoic acid (IV).


(I)


(II)


(III)


(IV)

The acidic protons were located for (II) and (IV) but could not be found for (I) and (III). The bond lengths and angles found for the saccharin groups in each of the molecules (I)-(IV) are similar to those reported for saccharin (Okaya, 1969), *N*-cyanomethylsaccharin (Junii, Guilan, Zhongyuan & Liu, 1989) and (*R*)-(+)-supidimide (Winter, Graudums & Frankus, 1983). Short C=O and long C—O bonds have been measured for (III) suggesting a disordered model for the carboxylic acid group. No such disorder is seen in the other three structures. The saccharin groups [C1-C6,C7,O7,N1,S1] are planar for each of the four structures [maximum deviations: (I) 0.023 (2), (II) 0.026 (2), (III) 0.049 (2), (IV) 0.037 (2) Å] and the sulfone O atoms lie approximately 1.2 Å out of this plane. The COOH groups of (II) and (IV) are planar to within 0.028 (9) and 0.012 (8) Å, respectively. Hydrogen bonding in each of the four structures produces the familiar eight-membered centrosymmetric ring dimer motif commonly observed in carboxylic acids (Leiserowitz, 1976) (see Table 5). The packing of the dimer in (I) is shown in Fig. 2. The dimers are held together by a number of short C—H···O contacts involving the sulfone and keto O atoms.

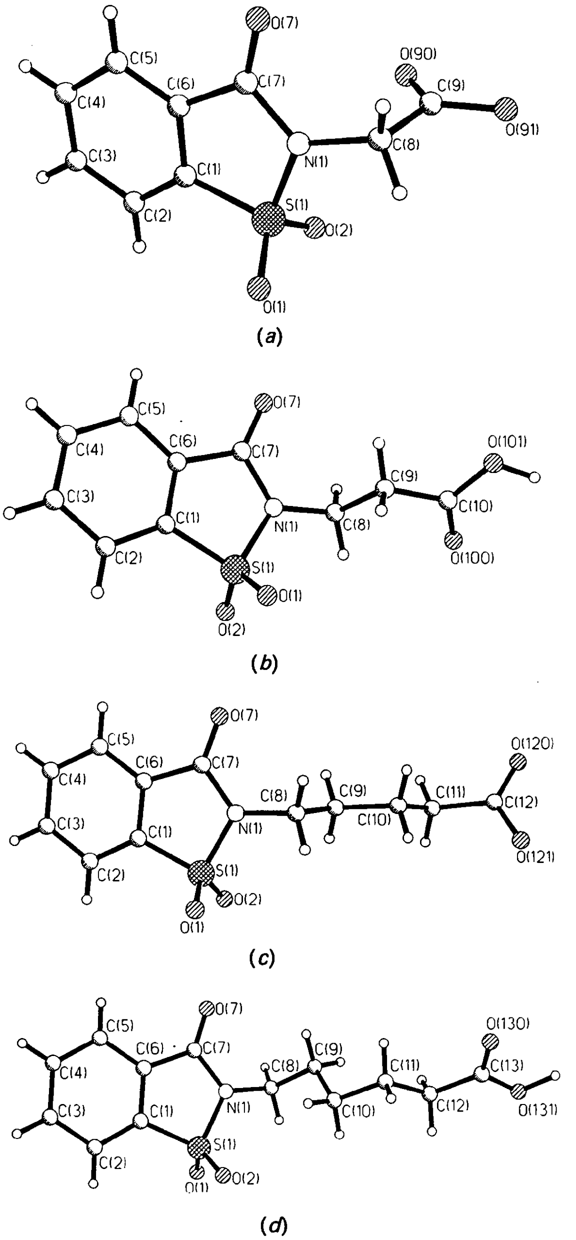


Fig. 1. (*a*) Atomic numbering scheme for (I), (*b*) atomic numbering scheme for (II), (*c*) atomic numbering scheme for (III) and (*d*) atomic numbering scheme for (IV).

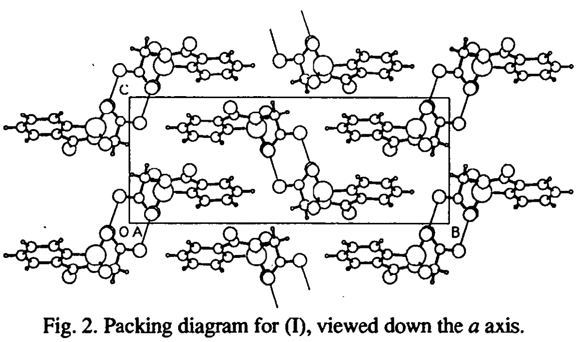


Fig. 2. Packing diagram for (I), viewed down the *a* axis.

Experimental

Compound (I)

Crystal data

$C_9H_7NO_5S$
$M_r = 241.22$
Monoclinic
$P2_1/c$
$a = 6.938\ (8)$ Å
$b = 19.326\ (7)$ Å
$c = 7.621\ (4)$ Å
$\beta = 94.96\ (6)°$
$V = 1018.1\ (13)$ Å^3
$Z = 4$
$D_x = 1.574$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 8\text{–}14°$
$\mu = 0.323$ mm^{-1}
$T = 295\ (2)$ K
Rod
$0.50 \times 0.30 \times 0.10$ mm
White

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1932 measured reflections
1790 independent reflections
1069 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int} = 0.0295$
$\theta_{\rm max} = 24.98°$
$h = -8 \rightarrow 8$
$k = 0 \rightarrow 22$
$l = 0 \rightarrow 9$
2 standard reflections monitored every 100 reflections intensity variation: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0460$
$wR(F^2) = 0.1269$
$S = 1.237$
1790 reflections
147 parameters
Only H-atom U's refined
Calculated weights $w = 1/[\sigma^2(F_o^2) + (0.0820P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = -0.005$
$\Delta\rho_{\rm max} = 0.452$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.240$ e Å^{-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 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for compound* (I)

$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	$U_{\rm eq}$
S1	0.41922 (13)	0.10438 (4)	0.24964 (12)	0.0518 (3)
O1	0.3073 (4)	0.07522 (13)	0.3780 (4)	0.0709 (8)
O91	0.9525 (4)	−0.03138 (11)	0.2116 (3)	0.0562 (7)
N1	0.6509 (4)	0.10848 (13)	0.3296 (4)	0.0518 (7)
O90	0.8799 (4)	0.06739 (12)	0.0717 (3)	0.0608 (7)
C3	0.2589 (7)	0.3034 (2)	0.1856 (5)	0.0714 (12)
C5	0.5874 (6)	0.2962 (2)	0.3095 (5)	0.0602 (10)
C6	0.5707 (5)	0.2247 (2)	0.3033 (4)	0.0440 (8)
C8	0.7679 (6)	0.0467 (2)	0.3547 (5)	0.0637 (11)
O7	0.8853 (4)	0.1861 (2)	0.4165 (4)	0.0824 (9)
C1	0.3995 (5)	0.1941 (2)	0.2389 (4)	0.0453 (8)
C9	0.8730 (5)	0.0290 (2)	0.1981 (4)	0.0475 (8)
O2	0.4059 (4)	0.07169 (14)	0.0836 (3)	0.0763 (8)
C7	0.7236 (5)	0.1748 (2)	0.3568 (4)	0.0475 (8)
C4	0.4286 (7)	0.3352 (2)	0.2493 (5)	0.0713 (12)
C2	0.2399 (5)	0.2330 (2)	0.1777 (4)	0.0596 (10)

Compound (II)

Crystal data

$C_{10}H_9NO_5S$
$M_r = 255.24$
Monoclinic
$P2_1/c$
$a = 7.435\ (7)$ Å
$b = 8.926\ (5)$ Å
$c = 16.883\ (4)$ Å
$\beta = 102.89\ (5)°$
$V = 1092.2\ (12)$ Å^3
$Z = 4$
$D_x = 1.552$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 8\text{–}14°$
$\mu = 0.306$ mm^{-1}
$T = 295\ (2)$ K
Cuboid
$0.40 \times 0.30 \times 0.20$ mm
White

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1992 measured reflections
1922 independent reflections
1311 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int} = 0.0157$
$\theta_{\rm max} = 24.97°$
$h = -8 \rightarrow 8$
$k = 0 \rightarrow 10$
$l = 0 \rightarrow 20$
2 standard reflections monitored every 100 reflections intensity variation: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0397$
$wR(F^2) = 0.1099$
$S = 1.244$
1922 reflections
161 parameters
Only H-atom U's refined
Calculated weights $w = 1/[\sigma^2(F_o^2) + (0.0661P)^2 + 0.0498P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = -0.002$
$\Delta\rho_{\rm max} = 0.243$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.264$ e Å^{-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 2. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for compound* (II)

$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	$U_{\rm eq}$
S1	0.13419 (10)	0.21259 (8)	0.64577 (4)	0.0512 (3)
O7	0.4533 (3)	0.5233 (2)	0.62499 (12)	0.0628 (6)
O101	0.4086 (3)	0.6258 (2)	0.91888 (13)	0.0737 (7)
C2	−0.0256 (4)	0.2064 (3)	0.4784 (2)	0.0534 (7)
C7	0.3396 (4)	0.4254 (3)	0.61308 (15)	0.0440 (6)
O100	0.4680 (4)	0.3819 (2)	0.92508 (12)	0.0782 (7)
O1	−0.0150 (3)	0.2508 (3)	0.68129 (12)	0.0729 (7)
C1	0.0987 (3)	0.2654 (3)	0.54344 (14)	0.0427 (6)
C6	0.2168 (3)	0.3800 (3)	0.53516 (14)	0.0411 (6)
C8	0.3972 (4)	0.3517 (3)	0.75956 (15)	0.0496 (7)
C10	0.4064 (4)	0.4942 (3)	0.8873 (2)	0.0512 (7)
O2	0.2061 (4)	0.0640 (2)	0.65832 (13)	0.0800 (7)
N1	0.3010 (3)	0.3357 (2)	0.67470 (12)	0.0458 (6)
C5	0.2141 (4)	0.4389 (3)	0.4596 (2)	0.0507 (7)
C3	−0.0259 (5)	0.2662 (3)	0.4024 (2)	0.0611 (8)
C4	0.0915 (4)	0.3798 (3)	0.3934 (2)	0.0586 (8)
C9	0.3245 (4)	0.4853 (3)	0.79797 (15)	0.0565 (8)

Compound (III)

Crystal data

$C_{12}H_{13}NO_5S$
$M_r = 283.29$
Triclinic
$P\overline{1}$
$a = 8.247$ (2) Å
$b = 8.332$ (2) Å
$c = 10.536$ (2) Å
$\alpha = 87.67$ (3)°
$\beta = 74.07$ (3)°
$\gamma = 67.64$ (3)°
$V = 642.2$ (3) Å^3
$Z = 2$
$D_x = 1.465$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 8$–15°
$\mu = 0.268$ mm^{-1}
$T = 295$ (2) K
Cuboid
0.40 × 0.30 × 0.20 mm
White

Data collection

Enraf-Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2391 measured reflections
2256 independent reflections
1323 observed reflections $[I > 2\sigma(I)]$
$R_{\rm int} = 0.0291$
$\theta_{\rm max} = 24.97°$
$h = -9 \rightarrow 9$
$k = -9 \rightarrow 9$
$l = 0 \rightarrow 12$
2 standard reflections monitored every 100 reflections intensity variation: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0453$
$wR(F^2) = 0.1242$
$S = 1.026$
2256 reflections
177 parameters
Only H-atom U's refined
Calculated weights $w = 1/[\sigma^2(F_o^2) + (0.0964P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.060$
$\Delta\rho_{\rm max} = 0.229$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.237$ e Å^{-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* (Å^2) *for compound* (III)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
S1	0.43336 (11)	0.58402 (11)	0.80608 (9)	0.0532 (3)
O120	1.4753 (3)	0.0022 (3)	0.8419 (2)	0.0608 (7)
O7	0.7957 (3)	0.6972 (4)	0.5721 (3)	0.0739 (8)
O1	0.3382 (3)	0.6986 (3)	0.9226 (2)	0.0663 (7)
O2	0.4457 (3)	0.4099 (3)	0.8169 (3)	0.0694 (7)
C6	0.4926 (4)	0.7202 (4)	0.5831 (3)	0.0488 (8)
N1	0.6382 (3)	0.5897 (3)	0.7431 (3)	0.0509 (7)
O121	1.2656 (3)	0.0868 (4)	1.0368 (3)	0.0752 (8)
C5	0.4599 (5)	0.7993 (5)	0.4708 (4)	0.0651 (10)
C10	1.0467 (4)	0.3033 (4)	0.8626 (4)	0.0548 (9)
C12	1.3121 (5)	0.0640 (4)	0.9112 (4)	0.0551 (9)
C1	0.3592 (4)	0.6764 (4)	0.6704 (3)	0.0508 (8)
C8	0.7728 (4)	0.5380 (4)	0.8193 (4)	0.0562 (9)
C7	0.6605 (4)	0.6722 (4)	0.6262 (3)	0.0520 (8)
C2	0.1926 (5)	0.7096 (5)	0.6485 (4)	0.0660 (10)
C11	1.1626 (5)	0.1115 (5)	0.8443 (4)	0.0615 (10)
C9	0.9049 (4)	0.3514 (4)	0.7860 (3)	0.0536 (9)
C3	0.1622 (5)	0.7896 (5)	0.5359 (4)	0.0742 (11)
C4	0.2927 (6)	0.8341 (5)	0.4484 (4)	0.0714 (11)

Compound (IV)

Crystal data

$C_{13}H_{15}NO_5S$
$M_r = 297.32$
Orthorhombic
$Pbcn$
$a = 20.694$ (6) Å
$b = 7.415$ (4) Å
$c = 17.693$ (7) Å
$V = 2714.9$ (20) Å^3
$Z = 8$
$D_x = 1.455$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 9$–13°
$\mu = 0.257$ mm^{-1}
$T = 295$ (2) K
Rod
0.60 × 0.20 × 0.20 mm
White

Data collection

Enraf-Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2387 measured reflections
2387 independent reflections
1378 observed reflections $[I > 2\sigma(I)]$
$\theta_{\rm max} = 24.97°$
$h = 0 \rightarrow 24$
$k = 0 \rightarrow 8$
$l = 0 \rightarrow 20$
2 standard reflections monitored every 100 reflections intensity variation: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0423$
$wR(F^2) = 0.0948$
$S = 1.105$
2387 reflections
191 parameters
Only H-atom U's refined
Calculated weights $w = 1/[\sigma^2(F_o^2) + (0.0436P)^2 + 1.8389P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.013$
$\Delta\rho_{\rm max} = 0.205$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.185$ e Å^{-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 4. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for compound* (IV)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
S1	0.11999 (4)	0.06458 (11)	0.65473 (4)	0.0466 (2)
O130	0.05022 (10)	0.9163 (3)	0.93568 (12)	0.0546 (6)
O131	−0.03286 (10)	0.7801 (3)	0.99019 (12)	0.0541 (6)
C1	0.19045 (14)	0.0401 (4)	0.6008 (2)	0.0404 (7)
N1	0.16116 (11)	0.1272 (3)	0.73122 (13)	0.0456 (6)
O1	0.08871 (10)	−0.1035 (3)	0.66920 (12)	0.0640 (7)
C6	0.24380 (14)	0.0774 (4)	0.6440 (2)	0.0435 (7)
C13	0.01534 (14)	0.7843 (4)	0.9430 (2)	0.0440 (7)
O2	0.08083 (10)	0.2083 (3)	0.62829 (12)	0.0653 (7)
C9	0.1259 (2)	0.3127 (4)	0.8410 (2)	0.0527 (8)
C7	0.2275 (2)	0.1256 (4)	0.7230 (2)	0.0470 (7)
O7	0.26452 (11)	0.1581 (3)	0.77351 (13)	0.0720 (7)
C10	0.0827 (2)	0.4400 (5)	0.7991 (2)	0.0584 (9)
C2	0.1944 (2)	−0.0110 (4)	0.5259 (2)	0.0546 (8)
C4	0.3089 (2)	0.0082 (5)	0.5382 (2)	0.0623 (9)
C3	0.2557 (2)	−0.0273 (5)	0.4955 (2)	0.0609 (9)
C5	0.3047 (2)	0.0625 (4)	0.6124 (2)	0.0565 (9)
C12	0.0247 (2)	0.6136 (4)	0.9003 (2)	0.0529 (9)
C8	0.1302 (2)	0.1262 (4)	0.8059 (2)	0.0554 (9)
C11	0.0726 (2)	0.6224 (5)	0.8375 (2)	0.0652 (10)

Table 5. *Selected interatomic distances* (Å) *and angles* (°) *for compounds* (I)-(IV)

	(I)	(II)	(III)	(IV)
Carboxyl group				
C(9)=O(90)	1.220 (4)	-	-	-
C(9)—O(91)	1.292 (4)	-	-	-
C(8)—C(9)	1.491 (5)	-	-	-
O(90)=C(9)—O(91)	124.5 (3)	-	-	-
C(8)—C(9)=O(90)	123.3 (3)	-	-	-
C(8)—C(9)—O(91)	112.2 (3)	-	-	-
C(10)=O(100)	-	1.222 (3)	-	-
C(10)—O(101)	-	1.288 (3)	-	-
C(9)—C(10)	-	1.497 (4)	-	-
O(100)=C(10)—O(101)	-	124.3 (2)	-	-
C(9)—C(10)=O(100)	-	120.2 (2)	-	-
C(9)—C(10)—O(101)	-	115.5 (2)	-	-
C(12)=O(120)	-	-	1.254 (4)	-
C(12)—O(121)	-	-	1.272 (4)	-
C(11)—C(12)	-	-	1.503 (5)	-
O(120)=C(12)—O(121)	-	-	123.3 (3)	-
C(11)—C(12)=O(120)	-	-	119.0 (3)	-
C(11)—C(12)—O(121)	-	-	117.7 (3)	-
C(13)=O(130)	-	-	-	1.223 (3)
C(13)—O(131)	-	-	-	1.302 (3)
C(12)—C(13)	-	-	-	1.487 (4)
O(130)=C(13)—O(131)	-	-	-	122.6 (3)
C(12)—C(13)=O(130)	-	-	-	123.4 (3)
C(12)—C(13)—O(131)	-	-	-	114.0 (3)
Saccharin group				
C(7)=O(7)	1.194 (4)	1.201 (3)	1.195 (4)	1.201 (3)
S(1)=O(1)	1.417 (4)	1.415 (2)	1.425 (3)	1.427 (2)
S(1)=O(2)	1.410 (4)	1.428 (2)	1.418 (2)	1.418 (2)
C(7)—N(1)	1.386 (4)	1.392 (3)	1.388 (4)	1.382 (4)
S(1)—N(1)	1.671 (3)	1.647 (2)	1.655 (3)	1.665 (2)
O(7)—C(7)—N(1)	123.0 (3)	123.2 (2)	123.8 (3)	123.6 (3)
C(7)—N(1)—S(1)	115.1 (2)	115.4 (2)	115.3 (2)	114.9 (2)
N(1)—S(1)—O(1)	109.5 (2)	108.84 (13)	110.0 (2)	109.25 (13)
N(1)—S(1)—O(2)	109.6 (2)	110.07 (14)	110.71 (15)	110.53 (13)
O(1)—S(1)—O(2)	116.3 (2)	117.64 (15)	116.9 (2)	117.14 (14)
Selected torsion angles for the alkyl chains				
S(1)—N(1)—C(8)—C(9)	91.2 (4)	−100.8 (3)	96.0 (3)	−116.9 (3)
N(1)—C(8)—C(9)—C(10)	-	173.8 (2)	178.0 (3)	65.7 (4)
C(8)—C(9)—C(10)—C(11)	-	-	172.1 (3)	174.4 (3)
C(9)—C(10)—C(11)—C(12)	-	-	175.8 (3)	−79.0 (4)
C(10)—C(11)—C(12)—C(13)	-	-	-	176.7 (3)
N(1)—C(8)—C(9)=O(90)	9.1 (5)	-	-	-
C(8)—C(9)—C(10)=O(100)	-	−23.3 (4)	-	-
C(10)—C(11)—C(12)=O(120)	-	-	−116.6 (4)	-
C(11)—C(12)—C(13)=O(130)	-	-	-	−10.3 (5)

Short intermolecular distances

(I)		(III)	
C(9)—O(90)···O(91^{i})	2.632 (4)	C(12)—O(120)···O(121^{iii})	2.639 (4)
C(9)—O(91)···O(90^{i})	2.632 (4)	C(12)—O(121)···O(120^{iii})	2.639 (4)
(II)		(IV)	
C(10)—O(100)···O(101^{ii})	2.590 (3)	C(13)—O(130)···O(131^{iv})	2.630 (3)
C(10)—O(101)···O(100^{ii})	2.590 (3)	C(13)—O(131)···O(130^{iv})	2.630 (3)

Symmetry codes: (i) $2-x,-y,-z$; (ii) $1-x,1-y,2-z$; (iii) $3-x,-y,2-z$; (iv) $-x,2-y,2-z$.

For each structure, data collection, cell refinement and data reduction were performed with *CAD*-4 *Software* (Enraf-Nonius, 1989). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL* (Sheldrick, 1994). Molecular graphics: *PLUTO* (Motherwell & Clegg, 1978). Software used to prepare material for publication: *SHELXL*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HA1083). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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[1*R*,2*R*,5(1′)*S*,9*R*,2′*R*,4′*R*,5′*S*]-2,10,10-Triméthyl-3-oxa-6-azatricyclo[7,1,1,$0^{2,7}$]undeca-5-spiro[1′(2′,4′,5′-triméthylcyclohexane)]-6-ène-4-one

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Abstract

In order to synthesize cyclic α-amino acids the dienophile 5-ethylidene-2,10,10-trimethyl-3-oxa-6-azatricyclo-[7.1.1.$0^{2,7}$]undeca-6-ene-4-one, (1), which has a pinane nucleus which acts as a chiral inductor, was prepared. The Diels-Alder reaction of (1) with 2,3-dimethyl-1,3-butadiene gives one diastereoisomer, the hydrogenation of which leads to an equimolecular mixture of the

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