

Discussion. Final atomic coordinates are given in Table 1.* The molecular structure and atom numbering are shown in Fig. 1. Interatomic distances, valence angles and hydrogen-bond geometry are given in Table 2. The 3-amino-3-phosphonopropionic acid (α -AspP) exists as a zwitterion with the α -amino N protonated and the phosphonic acid group negatively charged; the P—O bond lengths, 1.509 (3) and 1.500 (3) Å, indicate that the charge is equally distributed between O(2) and O(3) — as in β -AspP (Sawka-Dobrowolska, Głowiak, Siatecki & Soroka, 1985). There is extensive hydrogen bonding,

* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles involving H atoms, torsion angles, H-atom parameters and the full synthesis of the title compound have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54317 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

in which all potential donor and acceptor atoms participate.

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References

- GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* **A27**, 368–376.
 HAMILTON, W. C. (1965). *Acta Cryst.* **18**, 502–510.
 JOHNSON, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
 KAFARSKI, P. & MASTALERZ, P. (1984). *Beitr. Wirkst. Forsch.* **21**, 1–39.
 KOWALIK, J., SAWKA-DOBROWOLSKA, W. & GŁOWIAK, T. (1984). *J. Chem. Soc. Chem. Commun.* pp. 446–447.
 SAWKA-DOBROWOLSKA, W., GŁOWIAK, T., SIATECKI, Z. & SOROKA, M. (1985). *Acta Cryst.* **C41**, 453–456.
 SYNTAX (1976). *XTL/XTLE Structure Determination System*. Syntax Analytical Instruments, Cupertino, California, USA.

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Structure of 12,14-Dihydro-2H-dibenzo[*d,i*][1,3,7,6,8]dioxathiadiazecine

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Abstract. $C_{13}H_{12}N_2O_4S$, $M_r = 292.309$, monoclinic, $P2_1/n$, $a = 4.907$ (2), $b = 13.797$ (6), $c = 19.474$ (8) Å, $\beta = 97.20$ (3)°, $V = 1308.02$ (95) Å³, $Z = 4$, $D_x = 1.4843$, $D_m = 1.477$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.54178$ Å, $\mu = 22.24$ cm⁻¹, $F(000) = 608$, room temperature, final $R = 0.0494$ for 2121 reflections with $I > 3\sigma(I)$. The ten-membered ring is in a chair conformation. There are two intramolecular and one intermolecular hydrogen bonds of N—H...O type. The molecules form chains in the [100] direction.

Introduction. Bearing in mind that sulfamides find wide application in medicine we tried to obtain and study a new class of related compounds, cyclic sulf-

amides. Some methods of obtaining benzo- and dibenzosulfadiazines on the basis of the reaction of aryldiamines with sulfamide, or sulfur chloride have been described (Knollmuller, 1971, 1974). Following these methods we have carried out the synthesis by using an aminoaryl ether, methylenedioxybis(aminobenzene), and sulfamide. We have obtained the title compound (1) and used it for preliminary screening in antibacterial properties. The pharmacological test showed it had comparatively weak antibacterial activity. The formula of (1) has been confirmed by elemental, IR, NMR and MS methods.

This structure determination has been undertaken to give more detailed information about the bond

Table 1. Final positional ($\times 10^4$) and equivalent isotropic thermal ($\times 10^4$) parameters with e.s.d.'s in parentheses

$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	x	y	z	U_{eq} (\AA^2)
S1	1202 (1)	9670 (0)	1706 (0)	423 (2)
O1	4003 (3)	9420 (1)	1907 (1)	580 (6)
O2	-489 (4)	9104 (1)	1218 (1)	564 (5)
N1	-308 (4)	9664 (1)	2408 (1)	429 (6)
N2	1329 (4)	10771 (1)	1400 (1)	529 (6)
C7	-2160 (9)	12373 (2)	2568 (2)	766 (12)
O3	-2357 (4)	11413 (1)	2794 (1)	615 (6)
O4	-538 (4)	12415 (1)	2011 (1)	699 (7)
C1	962 (5)	10157 (2)	3009 (1)	477 (7)
C2	3193 (6)	9728 (2)	3413 (1)	625 (9)
C3	4361 (9)	10198 (3)	4006 (2)	903 (14)
C4	3312 (10)	11078 (4)	4194 (2)	1013 (17)
C5	1109 (9)	11487 (3)	3800 (2)	871 (13)
C6	-78 (6)	11040 (2)	3199 (1)	575 (8)
C8	-1940 (5)	12105 (2)	1391 (1)	568 (8)
C9	-4090 (7)	12629 (2)	1047 (2)	773 (12)
C10	-5402 (7)	12323 (3)	423 (2)	821 (12)
C11	-4564 (7)	11502 (3)	124 (2)	781 (11)
C12	-2373 (6)	10978 (2)	449 (1)	623 (8)
C13	-1037 (4)	11271 (2)	1083 (1)	493 (6)

Table 2. Interatomic distances (\AA) and angles ($^\circ$), and torsion angles ($^\circ$) of the heterocyclic ring with e.s.d.'s in parentheses

S1—O1	1.424 (2)	C1—C6	1.389 (4)
S1—O2	1.416 (2)	C2—C3	1.384 (5)
S1—N1	1.635 (2)	C3—C4	1.386 (7)
S1—N2	1.636 (2)	C4—C5	1.367 (6)
N1—C1	1.428 (3)	C5—C6	1.385 (5)
N2—C13	1.423 (3)	C8—C9	1.381 (4)
C7—O3	1.403 (3)	C8—C13	1.395 (4)
C7—O4	1.425 (5)	C9—C10	1.369 (5)
O3—C6	1.384 (3)	C10—C11	1.361 (6)
O4—C8	1.381 (3)	C11—C12	1.382 (5)
C1—C2	1.397 (4)	C12—C13	1.384 (3)
N1—S1—N2	110.6 (1)	C1—C6—C5	119.1 (3)
O1—S1—N2	108.3 (1)	O3—C6—C5	122.7 (3)
O2—S1—N1	105.6 (1)	O3—C6—C1	118.2 (2)
O1—S1—N2	104.2 (1)	O4—C8—C13	118.8 (2)
O1—S1—N1	107.0 (1)	O4—C8—C9	121.9 (2)
O1—S1—O2	121.0 (1)	C9—C8—C13	119.2 (2)
S1—N1—C1	119.1 (2)	C8—C9—C10	121.0 (3)
S1—N2—C13	122.8 (2)	C9—C10—C11	120.2 (4)
O3—C7—O4	110.1 (2)	C10—C11—C12	120.0 (4)
C7—O3—C6	116.6 (3)	C11—C12—C13	120.7 (3)
C7—O4—C8	112.9 (3)	C8—C13—C12	119.0 (2)
N1—C1—C6	119.9 (2)	N2—C13—C12	121.0 (2)
N1—C1—C2	119.4 (2)	N2—C13—C8	119.9 (2)
C2—C1—C6	120.7 (2)	C3—C4—C5	120.5 (4)
C1—C2—C3	119.0 (3)	C4—C5—C6	120.7 (4)
C2—C3—C4	120.1 (4)		
N1—S1—N2—C13	-71.03 (22)	N2—S1—N1—C1	-64.88 (21)
S1—N2—C13—C8	115.20 (24)	S1—N1—C1—C6	104.99 (26)
N2—C13—C8—O4	-1.52 (36)	N1—C1—C6—O3	0.39 (38)
C13—C8—O4—C7	-115.25 (29)	C1—C6—O3—C7	-128.56 (29)
C8—O4—C7—O3	76.75 (32)	C6—O3—C7—O4	76.67 (33)

system and the conformation of the heterocyclic ring of the first compound of this new class.

Experimental. Colourless crystals were obtained from ethanol at room temperature. Large needle-shaped

crystal with a specimen size of $0.3 \times 0.3 \times 0.3$ mm. D_m obtained by flotation. Diffraction data measured on a four-circle KM-4 diffractometer (KUMA Diffraction, Wrocław, Poland), Cu $K\alpha$ radiation, graphite monochromatization, $\omega/0.8\theta$ scan type. Unit-cell parameters were obtained by least-squares treatment of 25 reflections with $\theta_{max} = 43^\circ$. Cu data collected to $\theta_{max} = 75^\circ$, total of 5115 non-zero reflections measured, not corrected for absorption, range of h, k and l : 0 to 5, -16 to 16 and -23 to 23, respectively, standard reflections 244 and 266 monitored every 100 reflections, count variations less than 5%. 2101 reflections were considered observed by the criterion $I > 3\sigma(I)$ and used in calculations. Solution by direct methods using *SHELX76* (Sheldrick, 1976); H atoms located from difference Fourier map, refinement by full-matrix least-squares procedure on F magnitudes, 230 parameters; anisotropic thermal parameters for non-H atoms and isotropic for H atoms. Refinement to final $R = 0.0494$, $wR = 0.0571$, $S = 1.1014$; $w = 1/[\sigma^2(F) + 0.00427F^2]$. Max. shift/e.s.d. = 0.054, largest peaks on a final difference map 0.413 and $-0.582 \text{ e \AA}^{-3}$. Atomic scattering factors from *SHELX76*. The geometry of the molecule was calculated by *PARST* program from *CRYRULER* (Rizzoli, Sangermano, Calestani & Andreotti, 1976).

Discussion. The positional parameters and equivalent values of the anisotropic temperature factors for non-H atoms are given in Table 1,* and bond lengths, bond angles and torsion angles of the heterocyclic ring are given in Table 2. The structure of the molecule with the atom-numbering scheme is shown in Fig. 1.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54397 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

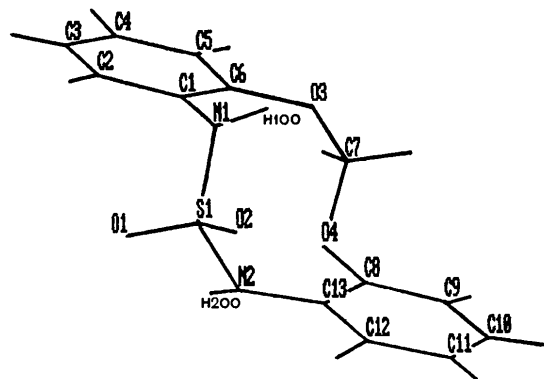


Fig. 1. The structure of the molecule with the atom-numbering scheme.

The ten-membered heterocyclic ring assumes a distorted chair conformation giving this conformation to the molecule as a whole. The ring is approximately symmetrical with respect to a pseudo-twofold axis through S1 and C7 and to a pseudo-mirror plane passing through the middle of bond C1—C6. The rotational symmetry is dominant: the asymmetry parameters (Duax & Norton, 1975) are $\Delta_2 = 13.8$ and $\Delta_m = 29.1$. The symmetry distortion mainly results from the large difference of the torsion angles of the pair S1—N1—C1—C6 and C1—C6—O3—C7. Two fused benzene rings are nearly planar with the maximum deviations from the least-squares planes 0.009 (4) for C5 and 0.013 (4) Å for C9. The dihedral angle of benzene-ring planes is 5.13 (10)°.

N1—H100 and N2—H200 bonds are intramolecular with torsion angles C6—C1—N1—H100 of -31.3 (2.2) and C8—C13—N2—H200 of -28.6 (2.6)°. The shortness of the N1...O3 and N2...O4 distances [2.755 (2) and 2.770 (2) Å, respectively] suggests there are two intramolecular hydrogen bonds, the N1—H100...O3 and N2—H200...O4

angles being 103.5 (2.5) and 95.6 (2.4)°, respectively. One of the two N atoms forms an intermolecular hydrogen bond N1—H100...O1, where O1 belongs to the molecule related by $x + 1, y, z$. The N1...O1 distance is 2.859 (3) Å and the N1—H100...O1 angle is 148.1 (2.8)°. Thus the crystal structure is built from chains of molecules linked by hydrogen bonds; these chains run in the [100] direction.

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References

- DUAX, W. L. & NORTON, D. A. (1975). *Atlas of Steroid Structure*, Vol. I. New York:IFI/Plenum.
 KNOLLMULLER, M. (1971). *Monatsh. Chem.* **102**, 1055–1064.
 KNOLLMULLER, M. (1974). *Monatsh. Chem.* **105**, 114–119.
 RIZZOLI, C., SANGERMANO, V., CALESTANI, G. & ANDRETTI, G. D. (1976). *CRYSRULER Package*. Version 1.2 (Polish version). Univ. degli Studi di Parma, Italy.
 SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

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Structure of (22*S*)-3β-Acetoxy-20-(3-isopropylisoxazolin-5-yl)-4,4,14α-trimethylpregn-8(9)-ene

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Abstract. C₃₂H₅₁NO₃, $M_r = 497.7$, orthorhombic, $P2_12_12_1$, $a = 7.577$ (2), $b = 10.510$ (2), $c = 35.399$ (7) Å, $V = 2819$ (1) Å³, $Z = 4$, $D_x = 1.173$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 0.69$ cm⁻¹, $F(000) = 1096$, $T = 153$ K, $R = 0.0497$ for 2235 observed reflections. The compound investigated is found to be a (22*S*)-epimer.

Introduction. Steroids having a lanostane skeleton and oxygen function in their side chain at C22 exhibit some useful biological properties (Kamernitzky & Reshetova, 1977). Isolation of such compounds from natural products and their

synthesis may be of considerable interest. We have synthesized two isomers [(22*R*) and (11*S*)] of 3β-acetoxy-20-(3-isopropylisoxazolin-5-yl)-4,4,14α-trimethylpregn-8(9)-ene (1). Biological activity of such steroid derivatives depends on the configuration of the C22 centre. Therefore the determination of chirality at the C22 atom in one epimer is of primary importance especially taking into account that further transformations proceed without inversion at the C22 atom.

Experimental. The title compound (1) was prepared from 3β-acetoxy-25,26,27-trinorlanosta-8,22-diene (Poyser, Hirtzbach & Ourisson, 1974; Akhrem, Khripach, Litvinovskaya & Baranovsky, 1989) via

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