Fig. 3. Molecules of 2,5-dimethylhexane-2,5-diol. A is at a general position, B at an inversion centre.

C(5)–C(6) (mean 115·4°) are *ca* 3° larger than the value of about 112° found in aliphatic hydrocarbons (Bodor, Bednowitz & Post, 1967). As observed in other glycols (Jeffrey & Shen, 1972), in the groups –C(CH₃)₂OH the angles C–C–O are generally smaller than the angles C–C–C. Due to the repulsions mentioned above the bonds of type C(4)–C(5) and C(5)–C(6) (mean 1·532 Å) have a tendency to be longer than the bonds of type C–CH₃ (mean 1·528 Å). A similar trend has been observed by Jeffrey & Shen (1972) for 2,5-dimethylhexane-2,5-diol tetrahydrate.

The C–O lengths can be compared with the values observed by Ruysink & Vos (1974) for the C–O bonds in *trans* (1·436 and 1·452 Å) and *cis* (1·432 and 1·440 Å) 2,5-dimethyl-3-hexene-2,5-diol and with the value of 1·459 Å reported by Jeffrey & Shen (1972). No explanation has yet been found for the variation in C–O lengths.

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5-Mesylamino-2H-1,2,3-triazole-4-carbonitrile Monohydrate

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Abstract. $C_4H_5N_5O_2S.H_2O$, triclinic $P\bar{1}$; $a = 5\cdot846$ (1), $b = 5\cdot222$ (1), $c = 13\cdot700$ (2) Å, $\alpha = 88\cdot87$ (3), $\beta = 94\cdot41$ (4), $\gamma = 84\cdot10$ (2)°, $V = 414\cdot62$ Å³; $Z = 2$, $D_x = 1\cdot643$, $D_m = 1\cdot645$ g cm⁻³; $\mu(\text{Mo } K\bar{\alpha}, \lambda = 0\cdot7107$ Å$) = 3\cdot19$ cm⁻¹. The structure was solved by the heavy-atom and Fourier techniques. An R of 0·035 was obtained for 1633 observed reflexions after least-squares refinement. The endocyclic H atom is linked to N(2).

Introduction. Intensities of 1825 independent reflexions were collected on a Syntex $P2_1$ computer-controlled four-circle diffractometer with Mo $K\alpha$ radiation from a graphite monochromator. After data reduction 192 reflexions with $|F| - 5\sigma(F) < 0$ were taken as unobserved. The position of the S atom was located in a Patterson map ($R = 0\cdot52$). Structure factor and Fourier calculations gave the positions of all non-hydrogen

atoms ($R = 0.28$). Prior to the refinement the positions of the methyl H atoms were generated assuming a regular tetrahedron with C–H constrained to 1·08 Å. This moiety was refined as a rigid group. Blocked full-matrix refinement of atomic coordinates with isotropic thermal parameters reduced R to 0·113. At this stage the four missing H atoms were located in a difference map. Anisotropic refinement for the non-hydrogen atoms gave a final R of 0·035 ($R_G = [\sum w\Delta^2/\sum w|F_o|^2]^{1/2} = 0.044$) for the observed reflexions ($R_{tot} = 0.045$ for all 1825 reflexions). The weights were given by $w = k/[\sigma^2(F_o) + gF_o^2]$ where k and g refined to 1·0 and 0·0, respectively. A bonded H atom scattering factor was employed (Stewart, Davidson & Simpson, 1965) with complex neutral atom scattering factors for the remaining atoms (Cromer & Mann, 1968; Cromer & Liberman, 1970). All calculations were performed with the program *SHELX* (Sheldrick, 1976). The final co-ordinates and thermal parameters for non-hydrogen atoms are given in Table 1, the parameters for the H atoms in Table 2, and the bond distances and angles in Fig. 1.*

Discussion. Stadler, Anschütz, Regitz, Keller, Van Assche & Fleury (1975) have summarized their results for the tosyl azide–malodinitrile adduct. The structure of this compound, described as 5-tosylamino-1H-1,2,3-triazole-4-carbonitrile, was confirmed by a structure analysis made by Hooge & Fischer.† Regitz (1967) has discussed the triazole *vs* triazene structure. At that time we synthesized the mesyl azide–malodinitrile adduct [yellow crystals, m.p. 180° (decomp.)]. The IR, NMR and mass spectral data (Horváth & Tamás, 1972) of this compound and those of the dimethyl derivative corroborated neither the triazene nor any of the pos-

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31754 (11 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

† Unpublished results, referred to in Stadler *et al.* (1975).

Table 2. *Fractional coordinates* ($\times 10^3$) *and isotropic thermal parameters* ($Å^2 \times 10^3$) *for the hydrogen atoms*

	x	y	z	$\bar{u}$
H(W1)	490	760	300	170 (16)
H(W2)	603	855	258	170 (16)
H(N2)	571	276	77	39 (12)
H(N4)	−122	497	273	39 (12)
H(C11)	426	281	393	96 (8)
H(C12)	309	177	499	96 (8)
H(C13)	235	495	453	96 (8)

sible triazole isomeric structures. The present X-ray study revealed a 2H-1,2,3-triazole structure.

The position of the endocyclic H atom differs from those found in other 4,5-disubstituted 1H-1,2,3-triazoles determined by X-ray diffraction (Kálmán, Simon, Schawartz & Horváth, 1974; Purnell, Shepherd & Hodgson, 1975) but this does not significantly influence the bond lengths in the five-membered ring. For comparison, the mean values of the corresponding distances and angles in 5-amino-4-carbamoyl-1H-1,2,3-triazole (Kálmán *et al.*, 1974) and tetrachlorobis-2-[(5-amino-4-carboxamidium)-1H-1,2,3-triazole]copper
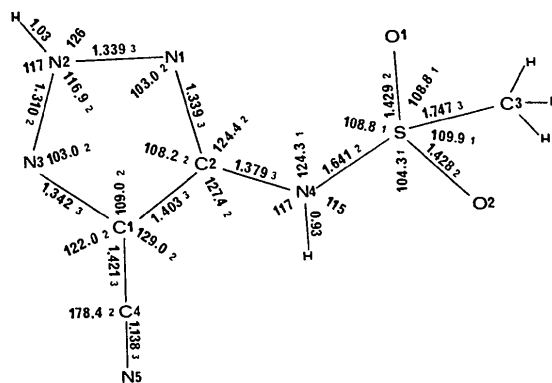

Fig. 1. Bond distances and angles with their e.s.d.'s for the non-hydrogen atoms. O(1)–S–O(2), 118·5 (1); C(3)–S–N(4), 105·9 (1)°.

Table 1. *Fractional coordinates and anisotropic thermal parameters* ($\times 10^4$)

Estimated standard deviations are given in parentheses. The anisotropic thermal parameters are in the form:
$$\exp\left(-2\pi^2 \sum_i \sum_j a_i^* a_j^* h_i h_j U_{ij}\right), \text{ with } U_{ij} \text{ in } Å^2.$$

|	x	y	z	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
S	528 (1)	1715 (1)	3635 (1)	313 (3)	271 (3)	339 (3)	44 (2)	93 (2)	22 (2)
O(1)	1353 (4)	−782 (3)	3322 (1)	547 (12)	240 (8)	523 (11)	24 (7)	134 (9)	39 (8)
O(2)	−1533 (3)	1980 (4)	4137 (1)	371 (10)	481 (11)	489 (11)	104 (8)	176 (9)	24 (8)
O(W)	−3880 (3)	7374 (3)	2880 (2)	447 (10)	431 (9)	925 (14)	85 (9)	193 (9)	102 (8)
N(1)	3358 (3)	2396 (3)	1855 (1)	329 (9)	302 (8)	356 (8)	63 (7)	82 (7)	102 (7)
N(2)	4202 (3)	3423 (3)	1072 (1)	332 (10)	350 (9)	353 (8)	59 (7)	106 (7)	144 (7)
N(3)	3026 (3)	5492 (4)	663 (1)	345 (10)	335 (8)	338 (8)	60 (7)	87 (7)	125 (7)
N(4)	−64 (3)	3648 (3)	2670 (1)	313 (9)	323 (8)	359 (8)	67 (6)	107 (6)	96 (6)
N(5)	−1928 (4)	9639 (4)	896 (1)	465 (11)	424 (10)	476 (10)	122 (8)	127 (8)	198 (8)
C(1)	1246 (3)	5901 (4)	1223 (1)	292 (10)	280 (9)	277 (9)	25 (7)	55 (7)	68 (7)
C(2)	1439 (4)	3966 (4)	1959 (1)	290 (10)	241 (9)	283 (9)	6 (7)	27 (7)	53 (7)
C(3)	2754 (5)	2970 (6)	4336 (2)	410 (15)	512 (16)	422 (14)	−12 (12)	−18 (12)	−31 (12)
C(4)	−500 (4)	7990 (4)	1033 (1)	342 (11)	319 (9)	331 (8)	47 (7)	88 (7)	75 (8)
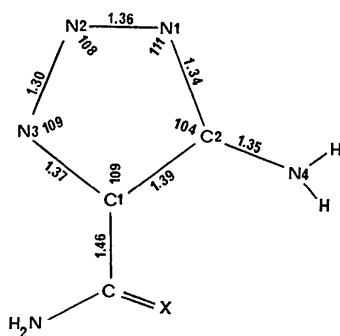
Fig. 2. The mean bond distances and angles for the 1,2,3-triazole rings measured in 5-amino-4-carbamoyl-1*H*-1,2,3-triazole (Kálmán *et al.*, 1974) [X = O] and tetrachlorobis-2-[(5-amino-4-carboxamidium)-1*H*-1,2,3-triazole]copper monohydrate (Purnell *et al.*, 1975) [X = NH$_2^+$].

monohydrate (Purnell *et al.*, 1975) are given in Fig. 2. The mean bond lengths, except C(1)–N(3), agree well with the corresponding ones in the title compound. The shortest N–N multiple bond (mean value 1·304 Å) in all three rings, independently of the position of the endocyclic H atom, is between N(2) and N(3). The angle at N(2) in the title compound is significantly greater than the corresponding mean value at N(1) for the other compounds. This can be explained by the neighbourhood of two equally low angles at N(1) and N(3), which do not take part in the formation of any hydrogen bonds. Therefore it may be assumed that these low angles are determined by the repulsion of the lone pairs localized on the N atoms. Angles at C(2) are presumably influenced by the character of the exocyclic C(2)–N(4) bonds. This bond is somewhat longer in the title compound than in the others which corresponds to a delocalized $d\pi$–$p\pi$ bond system formed on the C(2)–N(4)–S moiety. The S^{VI}–N(4) length of 1·641 Å agrees well with the value predicted by Kálmán (1974) for this sort of system. The geometry of the *N*-mesyl group is normal (Klug, 1968).

The C$_4$H$_5$N$_5$O$_2$S.H$_2$O units [except O(1), $\varDelta_O = -0.70$ Å and the mesyl groups, $\varDelta_C = -1.80$ Å)] lie approximately in the plane $(0.5543X + 0.6600Y + 0.5425Z - 3.3425 = 0$, in which the constants are referred to crystal axes) and are linked together within the parallel sheets by three different hydrogen bonds (Table 3). The hydrogen bond (No. 3), due to the deviation of O(1),

is somewhat out of the best plane given above. Owing to the limited number of *active* H atoms, a three-dimensional hydrogen-bond network, which is fairly complicated in the other 4,5-disubstituted 1*H*-1,2,3-triazoles, cannot be formed between the independent sheets.

Table 3. *The parameters of the hydrogen bonds*

No.	X–H$\cdots Y$	$X\cdots Y$ (Å)	H$\cdots Y$ (Å)	X–H$\cdots Y$ (°)
1	N(2)—H(N2)$\cdots$N(5)^i	2·88	2·02	139·2
2	N(4)—H(N4)$\cdots$O(*W*)	2·84	1·92	176·8
3	O(*W*)–H(*W*1)$\cdots$O(1)^{ii}	2·97	2·03*	180·0

Symmetry code Superscript		
None	$x,$	$y,$	z
(i)	$x+1,$	$y-1,$	z
(ii)	$x-1,$	$y+1,$	z

* After correction of the O(*W*)–H(*W*1) distance.

The X-ray analysis of the dimethyl derivative (m.p. 105°C) prepared from the title compound with methyl iodide is now in progress.

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