(Etter, Adsmond & Britton, 1990), except that the proton from the carboxylic acid group of DNBA is located on the adjacent ring N atom of 3-AT [N(2B)] as is the case in the 2-aminopyrimidine–oxalic acid adduct (Etter, 1991). One water molecule [OW(1)] is hydrogen-bonded to triazole N atoms N(1B) [2.71 (1) Å: $-x$, $-1-y$, $1-z$] and N(2B) [2.90 (1) Å: x, $-1+y$, z], and to the second water OW(2) [2.87 (1) Å]. OW(2) forms an eight-membered hydrogen-bonded ring with O(11A) [2.77 (1) Å: $-1+x$, y, z] and O(12A) [2.83 (1) Å] and is also hydrogen bonded to N(31B) [2.87 (1) Å: $-x$, $1-y$, $1-z$].

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Structure of *meso-(R,R,S,S)-trans*-4,5,4′,5′-Tetrahydroxy-2,2′-bi(1,3-thiaoxolane)–Dimethylacetamide (1:2)

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Abstract. $C_6H_{10}O_6S_2.2C_4H_9NO$, $M_r = 416.5$, monoclinic, $P2_1/c$, $a = 11.782$ (2), $b = 9.058$ (2), $c = 9.766$ (2) Å, $\beta = 109.30$ (1)°, $V = 983.7$ (4) Å^3, $Z = 2$, D_m (by flotation) = 1.40, $D_x = 1.405$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, $\mu = 2.788$ mm^{-1}, $F(000) = 444$, $T = 293$ K, $R = 0.0291$, $wR = 0.0285$ for 1125 unique diffractometer data $[I > 1\sigma(I)]$. The crystal structure and the synthesis of this heterocycle are new. The molecule $C_6H_{10}O_6S_2$ lies on a centre of symmetry. Two solvate molecules, dimethylacetamide, are attached to it by hydrogen bonds (2.695 Å). The solvate molecules and the heterocycle molecules are arranged in the crystal on different parallel layers (100), which are linked by the hydrogen bonds. The heterocycle has an envelope conformation.

Introduction. Formaldehyde is used in the mineral-oil industry as an H_2S scavenger. Its toxicity requires that it be substituted by a less poisonous substance, for example glyoxal. An aqueous alkaline solution of glyoxal absorbs H_2S and a colourless powder deposits. Chemical analysis shows that three parts of glyoxal react with two parts of H_2S. It was not possible to solve the structure of the product by spectroscopic methods. It was, however, possible to dissolve this colourless powder in dimethylacetamide and to grow crystals.

Experimental. A crystal of dimensions 0.3 × 0.3 × 0.2 mm was sealed in a Lindemann-glass capillary. 25 reflections with $2\theta > 30°$ were used for the determination of the cell parameters. One standard reflection ($\overline{2}2\overline{1}$) showed no decomposition by X-rays. 2450 reflections were collected ($-12 < h < 12$; $-9 < k < 0$; $-10 < l < 10$), 1126 were unique, $R_{int} = 0.013$, and 1125 with intensities $I > \sigma(I)$ were used for the structure analysis; Siemens $R3m/V$ computer-controlled diffractometer, $2\theta/\theta$ scan, 2θ range 2–105°; scan speed 4–15° min^{-1}; no correction for extinction; empirical absorption correction according to the ψ method (Kopfmann & Huber, 1968; North, Phillips & Mathews, 1968), T_{min}, T_{max} = 0.85, 1.0. The phase problem was solved by direct methods (Sheldrick, 1990); all the non-H atoms were located in the first electron density synthesis (E map). After a few cycles

$C_6H_{10}O_6S_2 \cdot 2C_4H_9NO$

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement coefficients* ($\text{Å}^2 \times 10^3$)

Equivalent isotropic U is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
C(1)	4797 (2)	555 (2)	5441 (2)	34 (1)
C(2)	5440 (2)	847 (3)	8277 (2)	41 (1)
C(3)	4123 (2)	588 (2)	7429 (2)	39 (1)
C(4)	1240 (3)	3894 (5)	7755 (4)	83 (2)
C(5)	593 (2)	2845 (3)	6570 (3)	56 (1)
C(6)	−1223 (3)	2049 (5)	4726 (4)	80 (1)
C(7)	−1243 (4)	4351 (5)	6118 (5)	92 (2)
N(1)	−563 (2)	3063 (2)	5893 (2)	63 (1)
O(1)	4084 (1)	−188 (1)	6147 (1)	37 (1)
O(2)	6008 (2)	−365 (2)	9104 (2)	54 (1)
O(3)	3544 (2)	1947 (2)	7126 (2)	47 (1)
O(4)	1125 (1)	1794 (2)	6231 (2)	64 (1)
S(1)	6068 (1)	1385 (1)	6872 (1)	44 (1)

Table 2. *Bond lengths* (Å) *and angles* (°)

C(1)—O(1)	1.421 (3)	C(1)—S(1)	1.838 (2)
C(1)—C(1A)	1.501 (5)	C(2)—C(3)	1.516 (3)
C(2)—O(2)	1.396 (3)	C(2)—S(1)	1.828 (3)
C(3)—O(1)	1.423 (3)	C(3)—O(3)	1.391 (3)
C(4)—C(5)	1.496 (5)	C(5)—N(1)	1.317 (3)
C(5)—O(4)	1.243 (3)	C(6)—N(1)	1.470 (4)
C(7)—N(1)	1.472 (5)		

O(1)—C(1)—S(1)	106.8 (1)	O(1)—C(1)—C(1A)	108.2 (2)
S(1)—C(1)—C(1A)	112.1 (2)	C(3)—C(2)—O(2)	113.7 (2)
C(3)—C(2)—S(1)	103.1 (1)	O(2)—C(2)—S(1)	114.1 (2)
C(2)—C(3)—O(1)	106.6 (2)	C(2)—C(3)—O(3)	108.6 (2)
O(1)—C(3)—O(3)	112.3 (2)	C(4)—C(5)—N(1)	118.1 (3)
C(4)—C(5)—O(4)	121.3 (2)	N(1)—C(5)—O(4)	120.6 (2)
C(5)—N(1)—C(6)	119.3 (2)	C(5)—N(1)—C(7)	124.6 (2)
C(6)—N(1)—C(7)	115.8 (2)	C(1)—O(1)—C(3)	110.0 (1)
C(1)—S(1)—C(2)	91.8 (1)		

of least-squares refinement the H atoms could be included from a difference electron density synthesis. The parameter refinement was performed by the cascade least-squares method (Sheldrick, 1990): anisotropic atomic displacement parameters of the non-H atoms were refined, least-squares refinement on $|F|$, 174 parameters, 951 degrees of freedom, $w = 1/\sigma^2(|F|)$ based on counting statistics, $R = 0.0291$, $wR = 0.0285$, $S = 3.0$, maximum $\Delta/\sigma = 0.002$. The ten largest peaks in the final difference electron density synthesis were between 0.19 and 0.11 e Å^{-3}. All calculations and drawings were performed on a MicroVAX II computer with the *SHELXTL-Plus* program system (Sheldrick, 1990); the scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99, 149). The final atomic positional parameters are given in Table 1, the bond lengths and bond angles in Table 2.*

Discussion. Fig. 1. shows the structure of the molecule, which is formed when H_2S passes a concentrated aqueous solution of glyoxal, very probably according to the following reaction scheme:
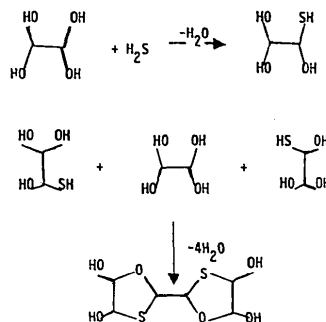
The structure of the heterocycle (Cambridge Structural Database, 1991), and even of the molecule itself, are to our knowledge not reported in the literature. Each C atom of the five-membered rings has chirality; the configuration of one five-membered
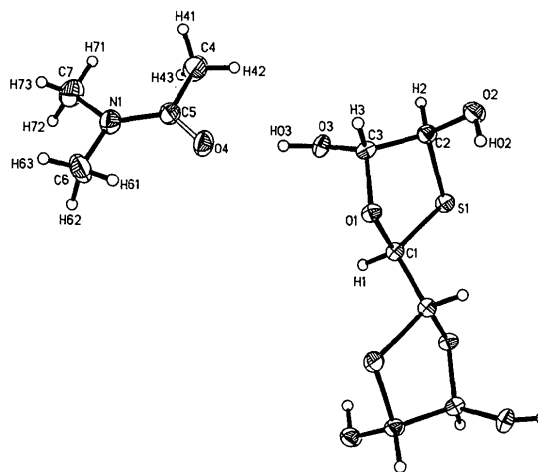

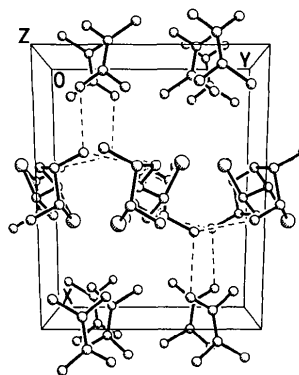Fig. 1. Molecular structures, showing atomic numbering scheme.

Fig. 2. Crystal structure; dashed lines represent hydrogen bonds.

ring is all-S or all-R. This means that the molecule has the *meso-(R,R,S,S)*-configuration. The molecule $C_6H_{10}O_6S_2$ lies on a crystallographic centre of symmetry. The five-membered rings have the envelope conformation; C(3) is 0.602 Å out of the plane of the other four atoms. The perpendicular distance of the best planes through the two rings in the molecule $C_6H_{10}O_6S_2$ is 1.057 Å. The hydroxy groups in the ring are in *trans* positions.

The molecule $C_6H_{10}O_6S_2$ crystallizes with two molecules of dimethylacetamide. Sheets of the five-membered ring molecules alternate with those of dimethylacetamide, linked by hydrogen bonds [O(4)⋯HO(3) 2.695 Å] parallel to the x axis (Fig. 2).

There is another hydrogen bond between the five-membered ring molecules [O(2)H⋯O(3) 2.841 Å] parallel to the y axis. In this manner the hydrogen-bond system controls the architecture of the crystal.

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Structure of N-{2-[2,4-Bis(1,1-dimethylpropyl)phenoxy]ethyl}-1-hydroxy-2-naphthalenecarboxamide

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Abstract. $C_{29}H_{37}NO_3$, $M_r = 447.62$, monoclinic, $P2_1/n$, $a = 13.391$ (2), $b = 28.817$ (3), $c = 6.667$ (2) Å, $\beta = 97.256$ (17)°, $V = 2552$ (2) Å^3, $Z = 4$, $D_x = 1.165$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 0.69$ cm^{-1}, $F(000) = 968$, $T = 295$ K, $R = 0.072$ for 2432 observed reflections $[I > \sigma(I)]$. The carbonyl O atom is intramolecularly hydrogen bonded to the hydroxyl proton. Intermolecular hydrogen bonding is absent. Disorder is evident in the *p-tert*-pentyl group. The molecules are packed in (010) sheets bounded on each surface by *tert*-pentyl groups and by naphthol groups. There is no intermolecular overlap of naphthol groups.

Introduction. The title compound (I) is a photographic coupler and reacts with oxidized color developer to yield cyan indoaniline image dyes in color films (Tong, 1977). The usual method of incorporating this compound in a sensitized film layer is to prepare a (high-vapor-pressure solvent and low-vapor-pressure plasticizer) solution of it and then to emulsify it as an oil-in-water emulsion (submicron particle sizes). After emulsification, the high-vapor-pressure solvent is removed by evapor-

ation or washing procedures. The resulting amorphous particles generally consist of coupler and plasticizer (*e.g.* di-*n*-butyl phthalate). Intraparticle nucleation of coupler can lead to microcrystallite formation, with dramatic effects on image dye formation kinetics. The definition of this crystalline structure (molecular packing) provides a basis for extending our understanding (Luss, Garti & Texter, 1992) of crystalline ripening and growth phenomena in photographic coupler dispersions (Sakamoto, Tanaka, Hanzawa & Kasai, 1972).

OH CONH(CH₂)₂—O—⟨⟩—C₅H₁₁—*tert* / C₅H₁₁—*tert*

(I)
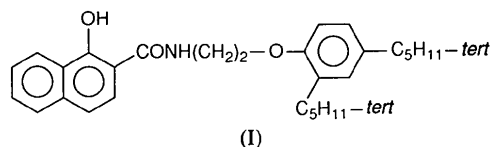
Experimental. The title compound was obtained from Mr W. F. Coffey of the Photographic Research Laboratories, Eastman Kodak Company. Large crystals were grown in saturated ethyl acetate solution at room temperature. One of these crystals was cut to a size of 0.27 × 0.28 × 0.38 mm for data col-