$C_{17}H_{11}ClO_4S$

| C6 | 0.9601 (2) | 1.4783 (4) | 1.1251 (2) | 3.14 (6) |
|----|-----------|-----------|-----------|----------|
| C7 | 0.7979 (2) | 1.1513 (4) | 1.1616 (2) | 3.12 (6) |
| C8 | 0.8430 (2) | 1.3044 (4) | 1.1448 (2) | 3.05 (6) |
| C9 | 1.1029 (2) | 0.9874 (4) | 1.1165 (2) | 3.32 (6) |
| C10 | 0.6511 (1) | 1.0519 (5) | 1.0851 (2) | 3.08 (6) |
| C11 | 0.6332 (2) | 1.1369 (5) | 0.9891 (2) | 3.79 (7) |
| C12 | 0.5939 (2) | 1.0260 (5) | 0.9129 (2) | 4.29 (7) |
| C13 | 0.5731 (2) | 0.8330 (5) | 0.9338 (2) | 3.96 (7) |
| C14 | 0.5912 (2) | 0.7459 (5) | 1.0283 (2) | 4.16 (7) |
| C15 | 0.6310 (2) | 0.8569 (5) | 1.1050 (2) | 3.68 (7) |
| C16 | 1.1789 (2) | 1.0203 (5) | 1.1060 (2) | 4.01 (7) |
| C17 | 1.2065 (2) | 1.2145 (5) | 1.0989 (2) | 4.31 (7) |

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

| | | | |
|----|----------|------------|----------|
| Cl—C13 | 1.733 (2) | C4—C5 | 1.459 (3) |
| S—O1 | 1.430 (2) | C5—C6 | 1.351 (3) |
| S—O2 | 1.442 (2) | C5—C8 | 1.451 (3) |
| S—C7 | 1.746 (2) | C7—C8 | 1.322 (3) |
| S—C10 | 1.764 (2) | C9—C16 | 1.365 (3) |
| O3—C2 | 1.378 (2) | C10—C11 | 1.385 (3) |
| O3—C6 | 1.337 (3) | C10—C15 | 1.380 (3) |
| O4—C4 | 1.228 (2) | C11—C12 | 1.378 (3) |
| C1—C2 | 1.384 (3) | C12—C13 | 1.374 (4) |
| C1—C17 | 1.370 (4) | C13—C14 | 1.375 (3) |
| C2—C3 | 1.382 (3) | C14—C15 | 1.387 (3) |
| C3—C4 | 1.469 (3) | C16—C17 | 1.391 (4) |
| C3—C9 | 1.403 (3) | | |
| O1—S—O2 | 118.8 (1) | C4—C5—C8 | 123.9 (2) |
| O1—S—C7 | 109.0 (1) | C6—C5—C8 | 116.8 (2) |
| O1—S—C10 | 108.7 (1) | O3—C6—C5 | 125.7 (2) |
| O2—S—C7 | 107.3 (1) | S—C7—C8 | 120.0 (2) |
| O2—S—C10 | 108.2 (1) | C5—C8—C7 | 127.5 (2) |
| C7—S—C10 | 103.9 (1) | C3—C9—C16 | 120.5 (2) |
| C2—O3—C6 | 118.5 (2) | S—C10—C11 | 119.9 (2) |
| C2—C1—C17 | 118.4 (2) | S—C10—C15 | 119.2 (2) |
| O3—C2—C1 | 116.3 (2) | C11—C10—C15 | 120.9 (2) |
| O3—C2—C3 | 121.2 (2) | C10—C11—C12 | 119.5 (3) |
| C1—C2—C3 | 122.5 (2) | C11—C12—C13 | 119.2 (2) |
| C2—C3—C4 | 120.8 (2) | Cl—C13—C12 | 119.4 (2) |
| C2—C3—C9 | 117.6 (2) | Cl—C13—C14 | 118.7 (2) |
| C4—C3—C9 | 121.6 (2) | C12—C13—C14 | 121.9 (2) |
| O4—C4—C3 | 122.0 (2) | C13—C14—C15 | 119.0 (3) |
| O4—C4—C5 | 123.4 (2) | C10—C15—C14 | 119.4 (2) |
| C3—C4—C5 | 114.5 (2) | C9—C16—C17 | 120.2 (2) |
| C4—C5—C6 | 119.2 (2) | C1—C17—C16 | 120.6 (2) |
| O3—C2—C3—C4 | −0.9 (4) | C2—C3—C4—O4 | −177.4 (2) |
| C1—C2—C3—C4 | 178.7 (2) | | |

The structure was solved using direct methods as incorporated in *MULTAN*80 (Main *et al.*, 1980). The remaining atoms were located in succeeding difference Fourier syntheses. The structure was refined by full-matrix least squares where the function minimized was $\Sigma w(|F_o|-|F_c|)^2$. The standard deviation on intensities, $\sigma(F_o^2)$, is defined as follows: $\sigma(F_o^2) = [S^2(C + R^2B)(pF_o^2)^2]/Lp^2$, where $S$ is the scan rate, $C$ is the total integrated peak count, $R$ is the ratio of scan time to background counting time, $B$ is the total background count, Lp is the Lorentz–polarization factor and the parameter $p$ is a factor introduced to down-weight intense reflections; in this case, $p$ was set to 0.040. Anomalous-dispersion effects were included in $F_c$; the values for $f'$ and $f''$ were those of Cromer (1974). The largest parameter shift was 0.01 times its e.s.d.

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# 5-(2,4-Dihydroxyphenyl)tetrazole Sesquihydrate

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## Abstract

The structure of the title compound [4-(5-tetrazolyl)-1,3-benzenediol sesquihydrate, $C_7H_6N_4O_2.3/2H_2O$] which crystallizes with two tetrazolylbenzenediol molecules per asymmetric unit together with three water molecules, has been determined by single-crystal X-ray diffraction at room temperature. The two molecules are linked through a hydrogen-bonded network to water molecules, forming layers extending along the *bc* face of the unit cell.

## Comment

The structure of the title compound, (I), has been determined as part of our studies of conventional liquid crystals and metalomesogens which contain heterocyclic rings (Gallardo & Merlo, 1993; Gallardo, Müller, Taylor, Bartulín & Martinez, 1993; Gallardo & Favarin, 1993; Gallardo & Begnini, 1995). The phenyl ring and the tetrazole ring of each molecule in the asymmetric unit are coplanar, which indicates an extension of the delocalized $\pi$-bonding system across the rings. A least-squares-plane calculation involving the atoms of both rings plus the atoms O1 and O2 in molecules *A* and *B* showed that the maximum deviation from planarity was less than 0.05 Å at O1 and O2'.

(I)
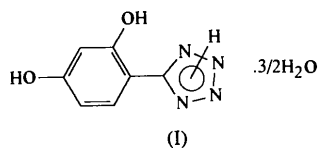
The structure consists of almost planar molecules joined by intermolecular hydrogen bonds forming layers parallel to the $bc$ plane. The phenolic OH group of molecule $A$ acts as an H-atom donor to water molecule 1 and as an H-atom acceptor from N4, which also forms an intramolecular hydrogen bond. On the other hand, the phenolic OH group of molecule $B$ acts as an H-atom donor to N4', forming an intramolecular hydrogen bond. The N4$\cdots$O2'$^v$ distance [symmetry code: (v) $1 - x$, $-y$, $2 - z$] is less than the sum of the van der Waals radii so that the H atom on N4 may be regarded as being involved in a bifurcated hydrogen bond.
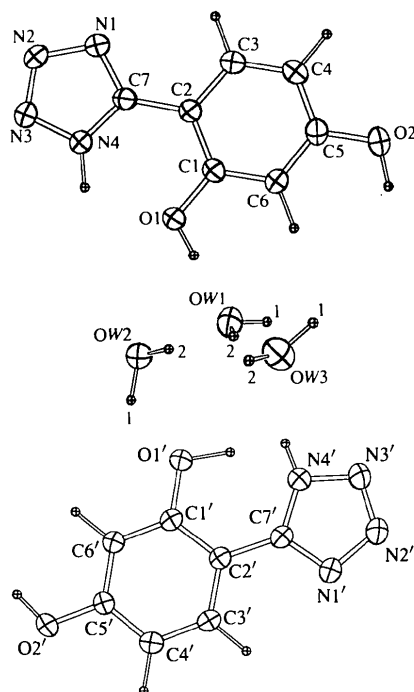


Fig. 1. Perspective view of the two unique molecules with atomic numbering scheme and with non-H atoms represented by 50% probability ellipsoids.
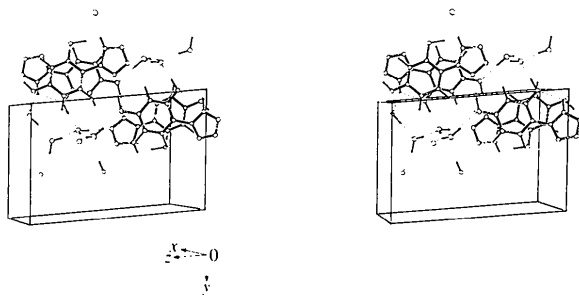


Fig. 2. Stereoscopic view of the unit cell illustrating the hydrogen bonds by dashed lines.

## Experimental

The title molecule was prepared by treating 2,4-dihydroxy-benzonitrile with $NaN_3$ in $NH_4Cl/DMF$. The reaction mixture was then heated at 343 K for 1 h, cooled and poured into 200 ml of water. The crude tetrazole was separated as solid, washed several times with water and recrystallized from water. Analysis: calculated for $C_7H_6N_4O_2$ C 47.19, H 3.37, N 31.46, O 17.98%; found C 47.20, H 3.20, N 31.92%.

*Crystal data*

| | |
|---|---|
| $2C_7H_6N_4O_2.3H_2O$ | Mo $K\alpha$ radiation |
| $M_r = 410.35$ | $\lambda = 0.7107$ Å |
| Triclinic | Cell parameters from 25 |
| $P\bar{1}$ | reflections |
| $a = 6.908\,(6)$ Å | $\theta = 9.80–13.51°$ |
| $b = 9.662\,(1)$ Å | $\mu = 0.116\,mm^{-1}$ |
| $c = 14.012\,(2)$ Å | $T = 295$ K |
| $\alpha = 84.53\,(1)°$ | Needle |
| $\beta = 74.50\,(3)°$ | $0.70 \times 0.40 \times 0.15$ mm |
| $\gamma = 86.98\,(3)°$ | Colourless |
| $V = 896.8\,(9)$ Å$^3$ | |
| $Z = 2$ | |
| $D_x = 1.519$ Mg m$^{-3}$ | |

*Data collection*

| | |
|---|---|
| Nonius CAD-4 diffractom- | $R_{int} = 0.0063$ |
| eter | $\theta_{max} = 24.97°$ |
| $\omega/2\theta$ scans | $h = -7 \rightarrow 8$ |
| Absorption correction: | $k = -11 \rightarrow 11$ |
| none | $l = 0 \rightarrow 16$ |
| 3443 measured reflections | 2 standard reflections |
| 2649 independent reflections | frequency: 30 min |
| 2351 observed reflections | intensity decay: 1.62% |
| [$F > 6\sigma(F)$] | |

*Refinement*

| | |
|---|---|
| Refinement on $F$ | $\Delta\rho_{max} = 0.11$ e Å$^{-3}$ |
| $R = 0.0468$ | $\Delta\rho_{min} = -0.24$ e Å$^{-3}$ |
| $wR = 0.0600$ | Extinction correction: |
| $S = 0.75$ | $F^* = F(1 - xF^2/\sin\theta)$ |
| 2351 reflections | Extinction coefficient: |
| 263 parameters | $x = 8.5\,(6) \times 10^{-8}$ |
| H-atom parameters not | Atomic scattering factors |
| refined | from *International Tables* |
| $w = 1.000/[\sigma^2(F)$ | *for X-ray Crystallography* |
| $+ 0.003087F^2]$ | (1974, Vol. IV) |
| $(\Delta/\sigma)_{max} = 0.65$ | |

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

$$B_{eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

| | $x$ | $y$ | $z$ | $B_{eq}$ |
|---|---|---|---|---|
| O1 | 0.2265 (3) | 0.0102 (2) | 0.7881 (1) | 3.30 (7) |
| O2 | 0.2743 (3) | −0.1202 (2) | 0.4627 (1) | 3.70 (7) |
| N1 | 0.2464 (3) | −0.3969 (2) | 0.9072 (1) | 2.87 (8) |
| N2 | 0.2330 (3) | −0.3826 (2) | 1.0043 (1) | 3.33 (8) |
| N3 | 0.2271 (3) | −0.2536 (2) | 1.0209 (1) | 3.29 (8) |
| N4 | 0.2344 (3) | −0.1809 (2) | 0.9340 (1) | 2.78 (7) |
| C1 | 0.2438 (3) | −0.0896 (2) | 0.7242 (1) | 2.38 (8) |
| C2 | 0.2548 (3) | −0.2283 (2) | 0.7601 (1) | 2.25 (8) |
| C3 | 0.2695 (3) | −0.3302 (2) | 0.6921 (2) | 2.66 (8) |

| C4 | 0.2744 (3) | −0.2949 (2) | 0.5939 (2) | 2.86 (9) |
| C5 | 0.2670 (3) | −0.1556 (2) | 0.5606 (2) | 2.63 (8) |
| C6 | 0.2526 (3) | −0.0529 (2) | 0.6249 (2) | 2.74 (9) |
| C7 | 0.2465 (3) | −0.2691 (2) | 0.8636 (2) | 2.31 (8) |
| O1′ | 0.7667 (3) | −0.4619 (2) | 0.8154 (1) | 3.67 (7) |
| O2′ | 0.7247 (3) | −0.0980 (2) | 1.0268 (1) | 3.58 (7) |
| N1′ | 0.7709 (3) | −0.1834 (2) | 0.5697 (1) | 2.71 (7) |
| N2′ | 0.7839 (3) | −0.2675 (2) | 0.4956 (1) | 3.25 (8) |
| N3′ | 0.7834 (3) | −0.3973 (2) | 0.5274 (1) | 3.28 (8) |
| N4′ | 0.7703 (3) | −0.3993 (2) | 0.6249 (1) | 2.80 (7) |
| C1′ | 0.7528 (3) | −0.3247 (2) | 0.8293 (1) | 2.38 (8) |
| C2′ | 0.7501 (3) | −0.2280 (2) | 0.7492 (1) | 2.16 (8) |
| C3′ | 0.7378 (3) | −0.0852 (2) | 0.7657 (2) | 2.56 (8) |
| C4′ | 0.7282 (3) | −0.0422 (2) | 0.8578 (2) | 2.81 (9) |
| C5′ | 0.7320 (3) | −0.1418 (2) | 0.9356 (1) | 2.46 (8) |
| C6′ | 0.7437 (3) | −0.2813 (2) | 0.9218 (1) | 2.63 (8) |
| C7′ | 0.7626 (3) | −0.2683 (2) | 0.6506 (1) | 2.30 (8) |
| OW1 | 0.0840 (3) | 0.2550 (2) | 0.7173 (1) | 3.77 (7) |
| OW2 | 0.3167 (3) | 0.3281 (2) | 0.8345 (1) | 3.69 (7) |
| OW3 | −0.3016 (3) | 0.3067 (2) | 0.6946 (1) | 4.02 (7) |

### Table 2. Selected bond lengths (Å) and angles (°)

| N1—N2 | 1.359 (3) | N1′—N2′ | 1.360 (3) |
| N1—C7 | 1.324 (3) | N1′—C7′ | 1.324 (3) |
| N2—N3 | 1.286 (3) | N2′—N3′ | 1.290 (3) |
| N3—N4 | 1.337 (3) | N3′—N4′ | 1.343 (3) |
| N4—C7 | 1.347 (3) | N4′—C7′ | 1.343 (3) |
| O1—C1—C2 | 118.6 (2) | O1′—C1′—C2′ | 119.2 (2) |
| C1—C2—C7 | 122.1 (2) | C1′—C2′—C7′ | 122.6 (2) |
| N1—C7—C2 | 127.6 (2) | N1′—C7′—C2′ | 126.4 (2) |
| N4—C7—C2 | 125.3 (2) | N4′—C7′—C2′ | 125.8 (2) |

### Table 3. Hydrogen-bonding geometry (Å, °)

| D—H⋯A | D—H | H⋯A | D⋯A | D—H⋯A |
|---|---|---|---|---|
| OW1—H1W1⋯N2′i | 0.89 | 2.00 | 2.870 (3) | 166 |
| OW1—H2W1⋯OW3 | 0.94 | 1.90 | 2.786 (3) | 156 |
| OW2—H1W2⋯N1ii | 1.00 | 1.93 | 2.910 (3) | 166 |
| OW2—H2W2⋯OW1 | 0.99 | 1.77 | 2.747 (3) | 167 |
| OW3—H1W3⋯O2iii | 0.94 | 2.01 | 2.940 (3) | 170 |
| OW3—H2W3⋯OW2iv | 0.95 | 1.93 | 2.850 (3) | 163 |
| O1—HO1⋯OW1 | 0.95 | 1.78 | 2.717 (3) | 171 |
| O2—HO2⋯N1′i | 0.99 | 1.95 | 2.941 (3) | 177 |
| N4—HN4⋯O2′v | 0.99 | 1.92 | 2.842 (3) | 154 |
| N4—HN4⋯O1 | 0.99 | 2.10 | 2.631 (3) | 112 |
| O1′—HO1′⋯N4′ | 1.12 | 1.66 | 2.675 (3) | 147 |
| O2′—HO2′⋯OW2v | 0.96 | 1.82 | 2.784 (3) | 173 |
| N4′—HN4′⋯OW3vi | 0.86 | 2.08 | 2.943 (3) | 170 |

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

H atoms were located from a difference electron density map and were not refined. Most of the calculations were performed on a DEC 3000 AXP computer.

Data collection: *CAD-4-Express* (Enraf–Nonius, 1993). Cell refinement: *MolEN* (Fair, 1990). Data reduction: *MolEN*. Program(s) used to solve structure: *SHELX86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELX76* (Sheldrick, 1976). Molecular graphics: *ORTEP*II. Software used to prepare material for publication: *MolEN* (Fair, 1990).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NS1005). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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# n-Dodecylammonium Chloride

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### Abstract

The redetermination of the structure of $C_{12}H_{28}N^+.Cl^-$ corroborates and complements the limited data of a prior less accurate study. The structure consists of $C_{12}H_{28}N^+$ chains arranged head-to-tail in layers between layers of $Cl^-$ ions.

### Comment

The structure of dodecylammonium bromide has been reported previously along with a preliminary (projection) determination of dodecylammonium chloride (Gordon, Stenhagen & Vand, 1953). A more recent study of decylammonium chloride (DACl) and dodecylammonium chloride (DDACl) (Pinto, Vencato, Gallardo & Mascarenhas, 1987) resulted in the latter complex being solved from only 337 observed reflections using isotropic displacement factors. It was suggested (Pinto et al., 1987) that the finding that the reflection intensities were weak for both compounds was perhaps due to the closeness of a phase transition (Gault, Gallardo & Muller, 1985), which may provoke crystalline disor-