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6-Amino-1,3-dimethyl-5-(2-ethylphenylazonio)uracil Bromide Dihydrate

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Abstract. $C_{14}H_{18}N_5O_2^+.Br^-.2H_2O$, $M_r = 404.3$, triclinic, $P\bar{1}$, $a = 7.1738$ (8), $b = 10.110$ (2), $c = 13.394$ (2) Å, $\alpha = 70.34$ (1), $\beta = 75.61$ (1), $\gamma = 79.34$ (1)°, $V = 880.6$ (2) Å^3, $Z = 2$, $D_x = 1.52$ g cm^{-3}, graphite-monochromatized Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å, $\mu = 27.3$ cm^{-1}, $F(000) = 416$, $T = 298$ K, $R = 0.052$ for 2640 observed reflections. The bulky organic cation is essentially coplanar and the protonation takes place at the azo nitrogen N(8). The additional proton participates in

a short intramolecular bond between N(8) and O(4) [N(8)···O(4) = 2·578 (4) Å]. The cations, bromide ions and crystal water molecules are bonded together by hydrogen bonds.

Introduction. Over recent years we have concentrated our interest on various modified 6-amino-5-phenylazo uracil derivatives and their metal complexes (Kivekäs, Colacio, Ruiz, Lopez-Gonzalez & Leon, 1989; Suarez-Varela, Legros, Galy, Colacio, Ruiz & Lopez-Gonzalez, 1989; Ruiz, 1988; Colacio, Costes, Kivekäs, Laurent & Ruiz, 1990; Ruiz, Colacio, Lopez-Gonzalez, Sundberg & Kivekäs, 1990; Colacio, Ruiz, Lopez-Gonzalez, Salas, Olivier, Quiros & Beauchamp, 1990). We have previously reported that some gold complexes of the type $(H_2L)(AuX_2)$ (where H_2L is the protonated form of 6-amino-5-phenylazouracil derivatives and $X = Cl$, Br) present a high degree of growth inhibition of tumour cells *in vitro*, indicating that these compounds would be active *in vivo*. To discover whether either the gold complex anion or the uracil cation is the active part of the compound we have prepared the salts H_2LBr. This paper is devoted to a description of the crystal structure of one of these compounds.

Experimental. The 6-amino-1,3-dimethyl-5-(2-ethyl-phenylazonio)uracil (HL) was synthesized by coupling 2-ethylaniline and 6-amino-1,3-dimethyl-uracil as previously described (Ruiz, 1988). The compound was recrystallized from hot ethanol with a yield of 75% (m.p. 537–538 K). The hydrobromide $H_2LBr.2H_2O$ was prepared by solving HL in ethanol containing HBr in molecular ratio 1:1. By slow evaporation at room temperature orange needles were obtained.

Crystal of size 0·18 × 0·15 × 0·32 mm was measured with a Nicolet P3F diffractometer. Unit-cell parameters were determined from 25 well centred reflections in the range $16 < 2\theta < 26°$. Intensity data were collected in the ω-scan mode, with scan speed 1·5–20° min^{-1} in the range $3 < 2\theta < 55°$. Three strong reflections monitored periodically exhibited variation of relative intensity in the range of 1·000–0·962. Intensities were collected up to $\sin\theta/\lambda = 0·65$ Å^{-1}. Intensities were corrected for Lorentz and polarization effects and for absorption (through the ψ-scan technique) with maximum transmission factor 1·000 and minimum 0·772. 4048 unique reflections ($0 < h < 9$, $-12 < k < 13$, $-16 < l < 17$) were measured, giving 2640 observed reflections according to the criterion $F_o > 4\sigma(F_o)$. The structure was solved by direct methods using the *SHELXS*86 program (Sheldrick, 1990). Full-matrix least-squares refinement was performed using the *XTAL*2.2 program system (Hall & Stewart, 1987) which minimized

the function $\sum w(|F_o| - |F_c|)^2$, where $w = 1/\sigma^2(F_o)$. A $\Delta\rho$ map calculated after anisotropic refinement of all non-H atoms revealed approximate positions of all H atoms, except those of the methyl carbon C(1). The strongest maximum near C(1) was considered as an H atom and the other two were placed at their calculated positions (C—H = 1·0 Å). Refinement (based on F) of all atoms, except the H atoms of C(1) and those of the crystal water molecules, with 278 parameters gave $R = 0·052$, $wR = 0·040$, $S = 2·35$, $(\Delta/\sigma)_{max} = 0·31$, $(\Delta/\sigma)_{av} = 0·037$, $\Delta\rho_{max} = 0·95$, $\Delta\rho_{min} = -0·88$ e Å^{-3}. Neutral atomic scattering factors were those included in the program systems and the corrections for anomalous dispersion for all atoms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).* The ^{1}H NMR spectrum of the compound was recorded on a Bruker AM300 spectrometer, using $(CD_3)_2SO$ as solvent.

Discussion. The structure consists of approximately coplanar bulky 6-amino-1,3-dimethyl-5-(2-ethyl-phenylazonio)uracil cations (hereafter H_2L^+), bromide ions and crystal water molecules joined together by hydrogen bonds. The asymmetric unit of the structure with the atomic labelling is shown in Fig. 1. The positional and equivalent isotropic thermal parameters for the non-H atoms are given in Table 1, and selected bond distances and angles in Table 2.

Protonation of H_2L^+ takes place at the N atom N(8) of the azo group. Similar molecules have the same N atom as the protonation site (Kivekäs *et al.*,

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* Lists of structure factors, anisotropic thermal parameters, complete bond lengths, least-squares-planes data and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53769 (24 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
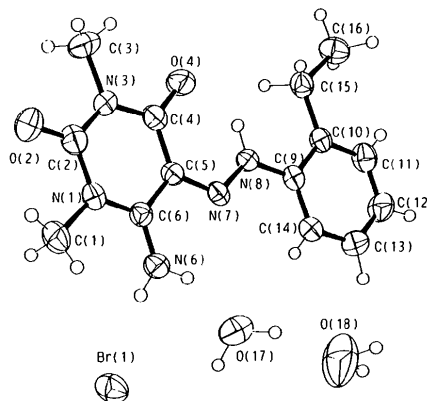

Fig. 1. Asymmetric unit of 6-amino-1,3-dimethyl-5-(2-ethyl-phenylazonio)uracil bromide dihydrate showing the atom-labelling scheme. The thermal ellipsoids for the non-H atoms are scaled to include 50% probability.

Table 1. *Positional and equivalent isotropic thermal parameters for the non-H atoms*

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

	x	y	z	$U_{eq}(\text{Å}^2)$
Br(1)	0·18467 (7)	0·27024 (5)	0·67235 (4)	0·0574 (4)
O(2)	0·2008 (4)	0·9210 (3)	0·3805 (2)	0·063 (3)
O(4)	0·5300 (4)	0·7986 (3)	0·0849 (2)	0·056 (3)
N(1)	0·2773 (4)	0·6875 (3)	0·4003 (2)	0·040 (3)
N(3)	0·3553 (5)	0·8618 (3)	0·2303 (2)	0·044 (3)
N(6)	0·3515 (5)	0·4501 (3)	0·4176 (2)	0·045 (3)
N(7)	0·5345 (4)	0·5070 (3)	0·2104 (2)	0·036 (3)
N(8)	0·6227 (4)	0·5323 (3)	0·1097 (2)	0·038 (3)
C(1)	0·1781 (6)	0·6577 (5)	0·5145 (3)	0·053 (4)
C(2)	0·2756 (5)	0·8300 (4)	0·3395 (3)	0·046 (4)
C(3)	0·3489 (7)	1·0122 (5)	0·1674 (4)	0·073 (5)
C(4)	0·4506 (5)	0·7614 (4)	0·1813 (3)	0·041 (4)
C(5)	0·4497 (5)	0·6171 (4)	0·2456 (3)	0·034 (3)
C(6)	0·3570 (5)	0·5802 (4)	0·3585 (3)	0·035 (3)
C(9)	0·7172 (5)	0·4211 (4)	0·0679 (3)	0·037 (4)
C(10)	0·8095 (5)	0·4590 (4)	−0·0422 (3)	0·035 (3)
C(11)	0·9044 (6)	0·3486 (5)	−0·0813 (3)	0·050 (4)
C(12)	0·9080 (6)	0·2104 (4)	−0·0172 (3)	0·056 (5)
C(13)	0·8176 (6)	0·1765 (4)	0·0897 (3)	0·054 (4)
C(14)	0·7207 (5)	0·2819 (4)	0·1320 (3)	0·043 (4)
C(15)	0·8034 (5)	0·6117 (4)	−0·1107 (3)	0·042 (4)
C(16)	0·9095 (6)	0·6375 (5)	−0·2286 (3)	0·055 (4)
O(17)	0·4506 (5)	0·1804 (3)	0·3918 (3)	0·097 (4)
O(18)	0·7839 (6)	−0·0001 (5)	0·4099 (4)	0·129 (6)

Table 2. *Selected bond lengths (Å), angles (°) and hydrogen bonds (Å,°)*

O(2)—C(2)	1·203 (6)	N(6)—C(6)	1·289 (4)
O(4)—C(4)	1·232 (4)	N(7)—N(8)	1·300 (4)
N(1)—C(1)	1·472 (5)	N(7)—C(5)	1·338 (5)
N(1)—C(2)	1·396 (5)	N(8)—C(9)	1·411 (5)
N(1)—C(6)	1·355 (5)	C(4)—C(5)	1·424 (5)
N(3)—C(2)	1·380 (5)	C(5)—C(6)	1·442 (5)
N(3)—C(3)	1·468 (5)	C(10)—C(15)	1·506 (5)
N(3)—C(4)	1·378 (5)	C(15)—C(16)	1·530 (4)

C(1)—N(1)—C(2)	115·9 (3)	O(4)—C(4)—C(5)	122·9 (4)
C(2)—N(1)—C(6)	123·6 (3)	N(3)—C(4)—C(5)	117·4 (3)
C(2)—N(3)—C(3)	116·8 (4)	N(7)—C(5)—C(4)	124·9 (3)
C(2)—N(3)—C(4)	123·3 (3)	N(7)—C(5)—C(6)	114·7 (3)
N(8)—N(7)—C(5)	118·2 (3)	C(4)—C(5)—C(6)	120·3 (4)
N(7)—N(8)—C(9)	121·0 (3)	N(1)—C(6)—C(5)	117·5 (3)
N(1)—C(2)—N(3)	117·6 (4)	N(8)—C(9)—C(10)	116·9 (3)
O(4)—C(4)—N(3)	119·7 (3)	N(8)—C(9)—C(14)	121·5 (3)

A—H···B	A—B	A—H	H···B	A—H···B
N(6)—H(61)···O(17)	2·803 (5)	0·94 (4)	1·93 (4)	155 (3)
N(6)—H(62)···Br(1)	3·314 (3)	0·91 (4)	2·42 (3)	168 (4)
N(8)—H(81)···O(4)	2·578 (4)	0·90 (3)	1·84 (3)	138 (3)
O(17)—H(171)···O(18^i)	2·977 (5)	0·98	2·02	167
O(17)—H(172)···O(18)	2·735 (5)	1·05	1·74	156
O(18)—H(181)···Br(1^i)	3·225 (6)	1·06	2·19	165
O(18)—H(182)···O(2^{ii})	2·912 (5)	1·05	1·93	154

Symmetry code: (i) $1 - x$, $-y$, $1 - z$; (ii) $1 + x$, $-1 + y$, z.

1989; Suarez-Varela *et al.*, 1989; Ruiz, 1988). Bond parameters of the substituted pyrimidine moiety do not significantly differ from those reported for analogous compounds (Kivekäs *et al.*, 1989; Suarez-Varela *et al.*, 1989; Ruiz, 1988; Colacio, Costes *et al.*, 1990; Ruiz *et al.*, 1990; Colacio, Ruiz *et al.*, 1990). The six atoms of pyrimidine are coplanar to within 0·05 Å. Among the exocyclic non-H atoms, C(3) has the greatest deviation from this plane, −0·074 (6) Å. This high value might be due to the steric interactions between the methyl group at N(3) and the O(2)

and O(4) atoms as is indicated by the short C(3)···O(4) and C(3)···O(2) contact distances of 2·686 (5) and 2·724 (6) Å, respectively, which are 0·6 Å less than the sum of their van der Waals radii (Huheey, 1983). The C atoms of the ethyl group bonded to the phenyl ring are on the other hand essentially in the plane of the phenyl group. The deviations of the atoms C(15) and C(16) from the plane are 0·005 (7) and −0·001 (9) Å, respectively. Since the angle between the mean plane through the pyrimidine moiety and the phenyl ring is only 3·3 (1)° the whole H_2L^+ ion is approximately planar. The planarity of H_2L^+, arising from electron π delocalization, is probably further enhanced by the strong intramolecular hydrogen bond between the protonated N(8) atom of the azo group and the carbonyl O atom O(4) [N(8)···O(4) = 2·578 (4) Å]. The strength of this bond might be related to the position of the N(8)—H signal in the ^{1}H NMR spectrum of the compound. Thus, a higher downfield value might indicate a stronger hydrogen bond, since the C(4)=O(4) group has a deshielding effect. In good accord with the X-ray results, the spectrum of this compound shows the N(8)—H signal at 14·9 p.p.m., indicating a strong hydrogen bond. The same planarity has been observed for analogous protonated compounds assuming intramolecular hydrogen bonds between N(8) and O(4).

The ethyl group lies at the same side of the molecule as the N(8)—H(8) bond as is normally observed for 2-substituted phenylazoniouracil cations (Kivekäs *et al.*, 1989; Suarez-Varela *et al.*, 1989). The orientation of the ethyl bond C(10)—C(15) is similar to that observed for the H_2L^+ ions in $[H_2L]_2[(NCS)_3Cu]$ (Sundberg, Kivekäs, Colacio & Ruiz, 1990). In the latter compound there are four non-equivalent H_2L^+ ions. In two of the ions the C(15)—C(16) bonds are directed away from the plane of the phenyl ring, while in the other two ions the bonds lie in the plane. This latter orientation is also found in the title compound $H_2LBr.2H_2O$.

Both the H atoms of the amino group and those of the water molecule participate in hydrogen-bond formation (Table 2), including the H(8) atom which participates in a strong intramolecular hydrogen bond, as mentioned previously.

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Structure of *N*-Methanesulfonyl-*N*-phenylhydroxylamine from Fenton's Reagent on Nitrosobenzene in DMSO

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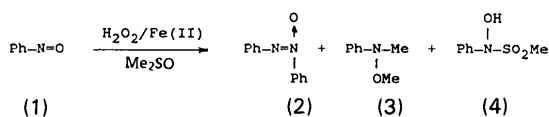
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Abstract. $C_7H_9NO_3S$, $M_r = 187 \cdot 2$, monoclinic, $P2_1/c$, $a = 9 \cdot 047$ (3), $b = 8 \cdot 774$ (3), $c = 10 \cdot 943$ (3) Å, $\beta = 97 \cdot 4$ (1)°, $V = 861 \cdot 4$ (5) Å^3, $Z = 4$, $D_x = 1 \cdot 44$ g cm^{-3}, λ(Cu $K\alpha$) = $1 \cdot 5418$ Å, $\mu = 30 \cdot 5$ cm^{-1}, $F(000) = 392$, $T = 293$ K, final $R = 0 \cdot 058$ for 1421 symmetry-independent observed reflections. The SO_2 group in the methanesulfonylhydroxylamine moiety adopts an orientation such that the N lone-pair bisects the O—S—O angle. Significant deviation from coplanarity is observed in the benzene ring.

Introduction. In our investigation of the reactivity of nitrosobenzenes with Fenton's reagent in dimethylsulfoxide (Me_2SO), we have isolated compounds (2), (3) and (4) (Cardellini, Greci, Stipa, Rizzoli, Sgarabotto & Ugozzoli, 1990).

The expected product from this reaction was (3), which was identified by elemental analysis and from spectroscopic data. Azoxybenzene (2) was most likely formed from nitrosobenzene (1) and phenylhydroxylamine which originated from (1) after re-

duction with methanesulfinic acid (Rudqvist & Torsell, 1971). (2) was identified by a comparison with an authentic sample (Vogel, 1984).

Though it has already been reported in the literature that methanesulfonic acid reacts with nitroso compounds leading to sulfonylated hydroxylamines of type (4) (Wayer, Geluk, Enbrts & Boer, 1970; Balyaev, Gornostaev & Suboch, 1975), it was difficult to foresee in the reaction of (1) with Fenton's reagent the formation of the *N*-methanesulfonyl-*N*-phenylhydroxylamine; in the present work the X-ray structural analysis of this product is reported.

Experimental. Dark, almost black prismatic crystals obtained by slow crystallization from benzene/ligroin. Siemens AED diffractometer on line to an IBM PS/2 M30 computer, Ni-filtered Cu $K\alpha$ radiation, θ–2θ scan, scan width from $(\theta$–$0 \cdot 60)°$ to $[\theta + 0 \cdot 60 + (\Delta\lambda/\lambda)\tan\theta]°$; scan speed $3 \cdot 0°$ min^{-1}. Crystal dimensions $0 \cdot 67 \times 0 \cdot 33 \times 0 \cdot 71$ mm. Cell dimensions based on 30 $(\theta,\chi,\varphi)_{hkl}$ ($20 \leq \theta \leq 30°$) measured intensities; 1640 ($-13 \leq h \leq 13$, $0 \leq k \leq 10$, $0 \leq l \leq 11$) symmetry-independent reflections ($3 \leq \theta \leq 70°$) collected at $T = 293$ K with a modified version (Belletti, Ugozzoli, Cantoni & Pasquinelli, 1979) of the Lehmann & Larsen (1974) procedure. One standard reflection every 50, no significant variation. Corrections for Lorentz and polarization effects, but not for absorption. Structure solved by direct methods

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