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Sodium Cytosine-5-methylenesulfonate Trihydrate*

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Abstract. $C_5H_6N_3O_4S^-.Na^+.3H_2O$, $P2_1/c$, $a = 5.235$ (1), $b = 22.843$ (5), $c = 9.141$ (2) Å, $\beta = 90.93$ (1)°, $Z = 4$, $D_c = 1.709$, $D_o = 1.70$ Mg m^{-3}, $V = 1092.8$ Å^3, $M_r = 281.2$, $\lambda(\text{Mo } K\alpha_1) = 0.70926$ Å, $T = 299 \pm 1$ K, $F(000) = 584$. The intensity data were collected on an automated diffractometer and the structure was solved by direct methods. Full-matrix least-squares refinement, with the temperature factors of the non-H atoms allowed to refine anisotropically, yielded an R of 0.038 for the 2160 significant data. The anions are packed as pairs of columns linked by N–N hydrogen bonds and bridged by solvent interactions.

Introduction. Bisulfite has been shown to be mutagenic in a number of microbial systems (see, for example, Hayatsu, 1976; Shapiro, 1977). Because bisulfite catalyzes the deamination of cytosine by reversibly adding across the C(5)–C(6) double bond, and because most mutations attributed to bisulfite have been characterized as guanine–cytosine (G–C) to adenine–thymine (A–T) transitions, cytosine deamina-

tion has been proposed as the mechanism of bisulfite mutagenesis.

One system in which bisulfite-mediated mutations have been observed, and characterized as G–C to A–T transitions, is the *r*II system of T4 bacteriophage (Summer & Drake, 1971). However, bacteriophage T4, as do other T-even bacteriophages, contains hydroxymethylcytosine in place of cytosine in its genome. A deaminated product of the reaction of bisulfite with hydroxymethylcytosine has been suggested as the ultimate mutagen in the T4 system (Shapiro, 1977).

It has recently been shown that the major product of the reaction of bisulfite with hydroxymethylcytosine under conditions similar to those employed for the modification of cytosine, namely acid pH and high bisulfite concentration, is cytosine-5-methylenesulfonate
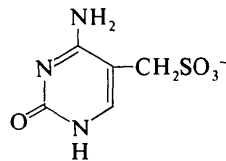
rather than a C(6) substituted dihydropyrimidine sulfonate (Hayatsu & Shiragami, 1979).

Cytosine-5-methylenesulfonate was synthesized according to Hayatsu & Shiragami (1979), and recrystallized as the sodium salt from aqueous ethanol. A crystal $0.32 \times 0.35 \times 0.30$ mm was selected for the diffraction study. The space group and approximate cell dimensions were determined from precession and Weissenberg photographs. More-accurate cell dimensions were obtained by least-squares refinement with the use of the observed setting angles for 12 Mo $K\alpha_1$ reflections, in the range $40 < 2\theta < 50°$, measured with an Oak Ridge computer-controlled diffractometer (Busing, Ellison, Levy, King & Roseberry, 1968). The density of the crystals was determined by flotation in mixtures of chloroform and dibromomethane. Intensity data were collected on the diffractometer with Nb-filtered Mo $K\bar{a}$ radiation using the $\theta-2\theta$ scan technique. Of 2515 reflections with $2\theta < 55°$, 2160 were considered observed $[|F_o| > 3\sigma(F_o)]$. Each intensity was assigned a variance $\sigma^2(I)$ based on counting statistics plus a term $(0.04I)^2$ empirically derived during refinement. No absorption correction was applied ($\mu = 0.37$ mm^{-1}). No significant changes were observed in two standard reflections during the course of the data collection.

The structure was solved with the aid of the direct-methods program *MULTAN* 78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). The H atoms were located from a difference electron-density map. The atomic parameters were refined by full-matrix least-squares methods, with anisotropic thermal parameters for the non-H atoms and isotropic thermal parameters for the H atoms. The final values of R, the weighted $R\{[\sum w(F_o - F_c)^2/\sum wF_o^2]^{1/2}\}$, and σ, the goodness of fit $\{[\sum w(F_o - F_c)^2/(n - p)]^{1/2}$, where $n = 2160$ reflections and $p = 203$ variables$\}$, were 0.038, 0.043 and 1.28, respectively. The average shift on the final cycle of refinement was 2% of the e.s.d. All refinements were performed with the XRAY system (Stewart, Kruger, Ammon, Dickinson & Hall, 1972). The final atomic parameters are given in Table 1.*

Discussion. A drawing of the anion, including bond distances and angles, is shown in Fig. 1. The bond distances and angles about the six-membered ring and substituents O(2) and N(4) are consistent with the average values reported in a review of several cytosine structures (Voet & Rich, 1970). The C(7)–S bond length, and the bond lengths and angles about the sulfonate group are comparable to those reported for other sulfonate structures: C–S 1.774 (3) and 1.818 (2) Å; average S–O 1.455 and 1.457 Å; average C–S–O 105.2 and 105.8°; average O–S–O 113.3 and 112.9° for, respectively, orthanilic acid (Hall & Maslen, 1967) and sodium 5,6-dihydro-2-thiouracil-6-sulfonate (Jain, Lee, Mertes & Pitman, 1978).

Atoms O(2), N(4), C(7) and the ring atoms are nearly coplanar; the r.m.s. deviation of these atoms from their least-squares displacement plane is 0.027 Å. The sulfonate group is rotated out of the plane. The torsion angles C(4)–C(5)–C(7)–S and C(6)–C(5)–C(7)–S are 90.2 (2) and −91.9 (2)°, respectively. The sulfonate O atoms are nearly staggered with respect to the substituents on C(7); the angles C(5)–C(7)–S–O are −51.5 (2) for O(11), 68.1 (2) for O(12) and −172.7 (1)° for O(13).

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

The isotropic temperature factor is $\exp[-8\pi^2 U(\sin \theta/\lambda)^2]$; the values given are the arithmetic averages of the principal components of the anisotropic temperature factors. Standard deviations in units of the last significant digits are given in parentheses.

	x ($\times 10^4$)	y ($\times 10^5$)	z ($\times 10^4$)	U (Å$^2 \times 10^4$)
N(1)	9099 (4)	47105 (8)	1830 (2)	271 (9)
C(2)	11176 (4)	44691 (9)	2552 (2)	221 (9)
O(2)	11737 (3)	39455 (7)	2291 (2)	301 (8)
N(3)	12506 (3)	48044 (7)	3511 (2)	223 (8)
C(4)	11739 (4)	53537 (8)	3787 (2)	200 (9)
N(4)	13075 (4)	56508 (8)	4180 (2)	269 (9)
C(5)	9528 (4)	56114 (8)	3073 (2)	203 (9)
C(6)	8287 (4)	52606 (9)	2108 (2)	253 (10)
C(7)	8610 (4)	62187 (8)	3408 (2)	218 (9)
S	100048 (9)*	67668 (2)	22738 (5)*	201 (2)
O(11)	12763 (3)	66822 (7)	2355 (2)	295 (8)
O(12)	8980 (3)	66643 (7)	799 (2)	300 (7)
O(13)	9172 (3)	73238 (7)	2873 (2)	328 (8)
Na	87514 (16)*	31790 (4)	14157 (9)*	280 (4)
O(W1)	5203 (4)	24002 (8)	5331 (2)	357 (9)
O(W2)	6135 (4)	33289 (8)	3438 (2)	353 (9)
O(W3)	4121 (3)	60074 (8)	9868 (2)	346 (9)

* Coordinate $\times 10^5$.

* Lists of structure factors, anisotropic thermal parameters and H-atom positional parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36554 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
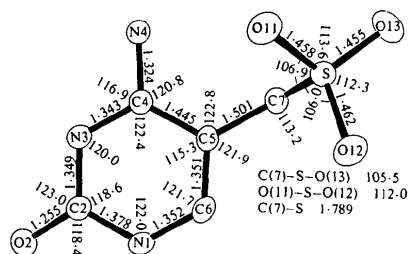

C(7)–S–O(13) 105.5
O(11)–S–O(12) 112.0
C(7)–S 1.789

Fig. 1. Bond distances (Å) and angles (°). The boundary thermal ellipsoids are drawn at the 50% probability level. The average standard deviations in the bond lengths and angles are 0.003 Å and 0.2° respectively. Illustrations were prepared with the aid of the computer program *ORTEP* (Johnson, 1965).

Table 2. *Hydrogen-bond parameters* (Å *and* deg)

Donor (D)	Acceptor (A)	D–A	H–A	D–H–A
N(1)	N(3^i)	2·959 (3)	2·12 (3)	170 (3)
N(1)	O(W3^{ii})	2·802 (3)	1·95 (3)	169 (3)
N(4)	O(W2^{iii})	2·870 (3)	2·13 (3)	140 (2)
O(W1)	O(13^{ii})	2·909 (3)	2·12 (4)	155 (3)
O(W1)	O(12^{iv})	2·935 (2)	2·11 (4)	165 (4)
O(W2)	O(2^v)	2·882 (2)	2·06 (4)	170 (4)
O(W2)	O(W1)	2·786 (3)	1·89 (5)	172 (3)
O(W3)	O(11^{vi})	2·846 (3)	2·04 (3)	171 (3)
O(W3)	O(2^{iii})	2·958 (3)	2·24 (4)	150 (4)

Symmetry code for superscripts: none x,y,z: (i) $3 - x, 1 - y, 1 - z$: (ii) $1 - x, 1 - y, 1 - z$: (iii) $2 - x, 1 - y, 1 - z$: (iv) $1 - x, -\frac{1}{2} + y, \frac{1}{2} - z$: (v) $-1 + x, y, z$: (vi) $-1 + x, y, 1 + z$.

Table 3. *Sodium-ion coordination* (Å)

Na–O(2)	2·471 (2)	Na–O(W1^{iii})	2·475 (2)
Na–O(12^i)	2·391 (2)	Na–O(W2)	2·344 (2)
Na–O(13^{ii})	2·323 (2)	Na–O(W3^{iv})	2·652 (2)

Symmetry code for superscripts: (i) $2 - x, 1 - y, -z$; (ii) $2 - x, -\frac{1}{2} + y, \frac{1}{2} - z$; (iii) $x, \frac{1}{2} - y, -\frac{1}{2} + z$; (iv) $1 - x, 1 - y, 1 - z$.
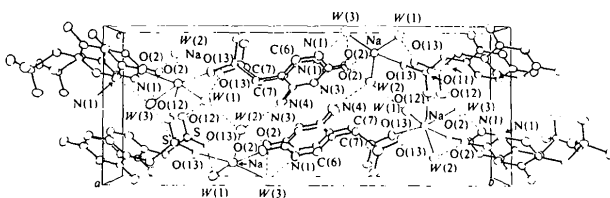

Fig. 2. Packing interactions. Two layers along the a direction are shown in perspective. The dashed lines represent hydrogen bonds. The fine solid lines represent sodium coordination. A hydrogen bond from N(4) to N(3) is not included in the drawing (Table 2).

Fig. 2 is a view of the packing interactions. The anions are packed with the aromatic rings stacked, forming columns along the a direction. The closest contacts are N(3)···C(6) 3·466 (3), N(4)···C(7) 3·431 (3), and O(11)···O(12) 3·573 (2) Å. The columns form pairs linked by hydrogen bonds from N(4) to N(3) and are further bridged by hydrogen bonds to water molecules (Table 2) and by coordination to the Na$^+$ ion (Table 3).

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Structure of lel lel lel Tris[(±)-2,3-butanediamine]cobalt(III) Chloride

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Abstract. [Co(C$_4$H$_{12}$N$_2$)$_3$]Cl$_3$, trigonal, space group $P\bar{3}1c$, $a = 12·275$ (4), $c = 7·874$ (1) Å, $Z = 2$. The structure was refined anisotropically to $R = 0·039$, $R_w = 0·045$ for 988 observed reflections. The cationic complexes have D_3 symmetry and are hydrogen bonded to chloride anions with N···Cl contacts of two