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# Tetrasodium $P^1,P^4$-Bis(5′-adenosyl)-tetraphosphate Dodecahydrate

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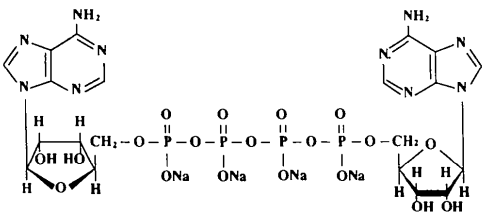
## Abstract

The title compound, $4Na^+.C_{20}H_{24}N_{10}O_{19}P_4^-.12H_2O$, was found to be the dodecahydrate. The two ribose rings adopt the 3′-*endo* envelope conformation, with the glycosyl linkage *anti*. The exocyclic side chains have the *g* conformation. The crystal structure is stabilized by interactions involving the $Na^+$ ions and hydrogen bonds.

## Comment

$P^1,P^4$-Bis(5′-adenosyl)tetraphosphate ($Ap_4A$) has various biological activities (Lüthje, 1989). We established the practical enzymatic synthesis of tetrasodium $Ap_4A$ (Nakajima, Tomioka, Kitabatake, Dombou & Tomita, 1989) and have developed it for a new medicine. It was very hygroscopic, but we obtained the nonhygroscopic crystal by crystallization with water. Structural analysis showed that it crystallized with 12 water molecules per formula unit.



$Na_4Ap_4A$

The two glycosyl torsion angles [O1—C6—N4—C2 −150.1 (4) and O17—C16—N9—C12 −152.1 (4)°] have the *anti* conformation. The two ribose rings adopt the 3′-*endo* envelope conformation. The pseudorotational phase angles *P* and puckering amplitudes $\tau_m$ were calculated from the ribose torsion angles (Altona & Sundaralingam, 1972). The values for the C6—C7—C8—C9—O1 ring are $P$ = 17.3° and $\tau_m$ = 37.0°. The values for the C16—C17—C18—C19—O17 ring are $P$ = 17.3° and $\tau_m$ = 38.0°. The exocyclic side-chain torsion angles [O4—C10—C9—C8 47.3 (5) and O14—C20—C19—C18 47.1 (5)°] have the *g* conformation.

Each $Na^+$ ion is surrounded by six atoms. Na1 is coordinated by two hydroxy O atoms (O2, O3) and four water molecules (O20, O21, O22, O23). Na2 is

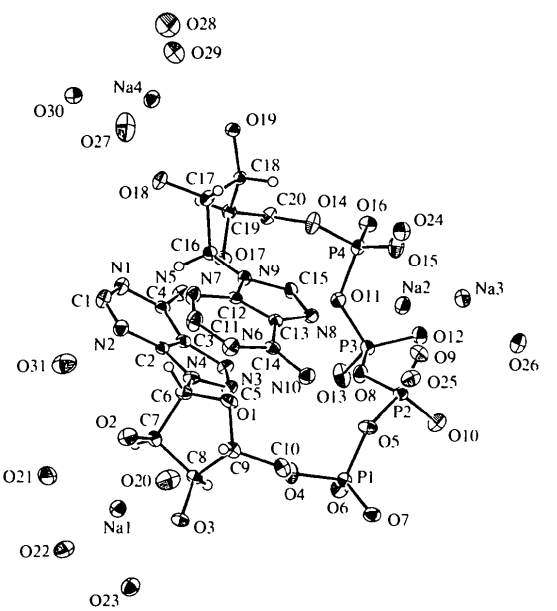


Fig. 1. Molecular structure showing 30% probability displacement ellipsoids. Bonds involving $Na^+$ ions are omitted for clarity.

coordinated by two phosphate O atoms [O9, O13$^{i}$; symmetry code: (i) $x$, $y$, $z + 1$], two water molecules (O24, O25) and two N atoms of adenine rings (N3$^{i}$, N8). The Na3 ion is coordinated by three phosphate O atoms (O9, O12, O15) and three water molecules [O20$^{ii}$, O23$^{ii}$, O26; symmetry code: (ii) $-x$, $\frac{1}{2} + y$, $1 - z$], and Na4 is coordinated by two hydroxy O atoms (O18, O19) and four water molecules (O27, O28, O29, O30). All water molecules form intermolecular hydrogen bonds. The molecules are connected by the coordinate bonds of the Na ions and the hydrogen bonds.

## Experimental

Synthesis was carried out by the reaction of ATP in aqueous solution for 6 h at 313 K in the presence of leucyl *t*-RNA synthetase (Nakajima *et al.*, 1989). Recrystallization was from water. The density $D_m$ was measured by flotation in methyl iodide/carbon tetrachloride.

*Crystal data*

$4Na^+.C_{20}H_{24}N_{10}O_{19}P_4^-.12H_2O$
$M_r = 1140.50$
Monoclinic
$P2_1$
$a = 12.748\ (2)$ Å
$b = 20.265\ (3)$ Å
$c = 8.562\ (2)$ Å
$\beta = 90.38\ (1)°$
$V = 2211.7\ (6)$ Å$^3$
$Z = 2$
$D_x = 1.712$ Mg m$^{-3}$
$D_m = 1.710$ Mg m$^{-3}$

Cu $K\alpha$ radiation
$\lambda = 1.54184$ Å
Cell parameters from 25 reflections
$\theta = 28.2$–$28.4°$
$\mu = 2.993$ mm$^{-1}$
$T = 293$ K
Prism
0.50 × 0.25 × 0.10 mm
Colourless

*Data collection*

Rigaku AFC-7*R* diffractometer
$\omega/2\theta$ scans
Absorption correction: $\psi$ scans (North, Phillips & Mathews, 1968)
$T_{min} = 0.47$, $T_{max} = 0.70$
3667 measured reflections
3416 independent reflections
3361 observed reflections [$I > 3\sigma(I)$]

$R_{int} = 0.023$
$\theta_{max} = 60.1°$
$h = -14 \rightarrow 14$
$k = -22 \rightarrow 0$
$l = -9 \rightarrow 0$
3 standard reflections monitored every 150 reflections
intensity decay: 0.6%

*Refinement*

Refinement on $F$
$R = 0.036$
$wR = 0.054$
$S = 1.28$
3361 reflections
622 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o) + 0.001936F_o^2]$

$(\Delta/\sigma)_{max} = 0.02$
$\Delta\rho_{max} = 0.28$ e Å$^{-3}$
$\Delta\rho_{min} = -0.37$ e Å$^{-3}$
Extinction correction: secondary, isotropic
Extinction coefficient: $5.8\ (5) \times 10^{-6}$
Atomic scattering factors from Cromer & Waber (1974)

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

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

| | $x$ | $y$ | $z$ | $U_{eq}$ |
|---|---|---|---|---|
| P1 | 0.14716 (8) | 0.00000 | 0.2277 (1) | 0.0281 (3) |
| P2 | 0.23224 (8) | 0.12288 (7) | 0.3482 (1) | 0.0289 (3) |
| P3 | 0.21986 (8) | 0.22628 (8) | 0.1196 (1) | 0.0296 (3) |
| P4 | 0.13228 (8) | 0.34216 (8) | 0.2673 (1) | 0.0292 (3) |
| Na1 | −0.3951 (1) | −0.1270 (1) | 0.2046 (2) | 0.0392 (5) |
| Na2 | 0.0961 (1) | 0.1739 (1) | 0.7392 (2) | 0.0411 (5) |
| Na3 | 0.3221 (1) | 0.2603 (1) | 0.4837 (2) | 0.0411 (5) |
| Na4 | −0.4032 (1) | 0.4729 (1) | 0.3022 (2) | 0.0412 (5) |
| O1 | −0.1661 (3) | 0.0571 (2) | 0.2922 (4) | 0.0374 (8) |
| O2 | −0.3697 (3) | −0.0111 (2) | 0.1708 (4) | 0.0469 (9) |
| O3 | −0.2146 (2) | −0.1021 (2) | 0.1227 (3) | 0.0341 (8) |
| O4 | 0.0242 (2) | −0.0067 (2) | 0.2069 (3) | 0.0399 (9) |
| O5 | 0.1575 (3) | 0.0604 (2) | 0.3489 (4) | 0.0362 (8) |
| O6 | 0.1868 (3) | 0.0184 (2) | 0.0722 (4) | 0.0425 (9) |
| O7 | 0.1918 (3) | −0.0583 (2) | 0.3095 (4) | 0.0385 (8) |
| O8 | 0.1756 (2) | 0.1647 (2) | 0.2142 (4) | 0.0377 (8) |
| O9 | 0.2215 (3) | 0.1575 (2) | 0.4998 (4) | 0.0422 (9) |
| O10 | 0.3391 (3) | 0.1040 (2) | 0.2997 (4) | 0.0450 (9) |
| O11 | 0.1300 (3) | 0.2794 (2) | 0.1529 (5) | 0.050 (1) |
| O12 | 0.3192 (2) | 0.2493 (2) | 0.1926 (4) | 0.0385 (8) |
| O13 | 0.2167 (3) | 0.2104 (2) | −0.0485 (4) | 0.051 (1) |
| O14 | 0.0108 (2) | 0.3547 (2) | 0.2912 (4) | 0.0436 (9) |
| O15 | 0.1736 (3) | 0.3250 (2) | 0.4231 (4) | 0.047 (1) |
| O16 | 0.1808 (3) | 0.3976 (2) | 0.1815 (4) | 0.0374 (8) |
| O17 | −0.1769 (3) | 0.2855 (2) | 0.2081 (3) | 0.0353 (8) |
| O18 | −0.3826 (2) | 0.3484 (2) | 0.3328 (4) | 0.0415 (9) |
| O19 | −0.2278 (2) | 0.4425 (2) | 0.3847 (3) | 0.0346 (8) |
| O20 | −0.3660 (4) | −0.1218 (2) | 0.4760 (5) | 0.063 (1) |
| O21 | −0.5747 (3) | −0.1095 (2) | 0.2186 (6) | 0.062 (1) |
| O22 | −0.4336 (3) | −0.1542 (2) | −0.0596 (4) | 0.050 (1) |
| O23 | −0.3668 (3) | −0.2477 (2) | 0.2463 (4) | 0.047 (1) |
| O24 | 0.0933 (3) | 0.2909 (2) | 0.7033 (4) | 0.0429 (9) |
| O25 | 0.1121 (3) | 0.0573 (2) | 0.7855 (4) | 0.046 (1) |
| O26 | 0.4933 (3) | 0.2332 (2) | 0.3956 (5) | 0.058 (1) |
| O27 | −0.3753 (3) | 0.4772 (3) | 0.0305 (5) | 0.079 (2) |
| O28 | −0.3678 (5) | 0.5942 (3) | 0.2768 (6) | 0.079 (2) |
| O29 | −0.4404 (3) | 0.4972 (2) | 0.5630 (4) | 0.056 (1) |
| O30 | −0.5804 (3) | 0.4556 (2) | 0.2609 (5) | 0.054 (1) |
| O31 | −0.5052 (3) | 0.0893 (2) | 0.0856 (6) | 0.069 (1) |
| N1 | −0.3006 (3) | 0.2794 (2) | −0.1592 (5) | 0.039 (1) |
| N2 | −0.3441 (3) | 0.1845 (2) | −0.0066 (5) | 0.038 (1) |
| N3 | −0.0654 (3) | 0.1794 (2) | −0.0587 (4) | 0.0341 (9) |
| N4 | −0.1894 (3) | 0.1226 (2) | 0.0712 (4) | 0.0319 (9) |
| N5 | −0.1320 (3) | 0.3077 (2) | −0.2279 (5) | 0.038 (1) |
| N6 | −0.2904 (3) | 0.0604 (2) | 0.6651 (5) | 0.042 (1) |
| N7 | −0.3419 (3) | 0.1549 (2) | 0.5117 (5) | 0.041 (1) |
| N8 | −0.0624 (3) | 0.1625 (2) | 0.5509 (4) | 0.0324 (9) |
| N9 | −0.1913 (3) | 0.2185 (2) | 0.4266 (4) | 0.0296 (9) |
| N10 | −0.1197 (3) | 0.0343 (2) | 0.7243 (5) | 0.039 (1) |
| C1 | −0.3651 (4) | 0.2387 (3) | −0.0848 (6) | 0.044 (1) |
| C2 | −0.2403 (3) | 0.1715 (2) | −0.0080 (5) | 0.028 (1) |
| C3 | −0.1645 (3) | 0.2066 (2) | −0.0855 (5) | 0.030 (1) |
| C4 | −0.1967 (3) | 0.2648 (2) | −0.1602 (5) | 0.030 (1) |
| C5 | −0.0832 (3) | 0.1302 (2) | 0.0364 (5) | 0.033 (1) |
| C6 | −0.2390 (4) | 0.0758 (2) | 0.1772 (5) | 0.036 (1) |
| C7 | −0.2787 (3) | 0.0130 (2) | 0.0959 (5) | 0.031 (1) |
| C8 | −0.1877 (3) | −0.0343 (2) | 0.1312 (5) | 0.030 (1) |
| C9 | −0.1516 (3) | −0.0138 (2) | 0.2942 (5) | 0.032 (1) |
| C10 | −0.0411 (4) | −0.0283 (2) | 0.3342 (5) | 0.038 (1) |
| C11 | −0.3602 (4) | 0.1000 (3) | 0.5922 (7) | 0.050 (1) |
| C12 | −0.2376 (3) | 0.1685 (2) | 0.5097 (5) | 0.027 (1) |
| C13 | −0.1589 (3) | 0.1342 (2) | 0.5838 (5) | 0.028 (1) |
| C14 | −0.1875 (3) | 0.0765 (2) | 0.6595 (5) | 0.031 (1) |
| C15 | −0.0851 (3) | 0.2119 (2) | 0.4572 (5) | 0.033 (1) |
| C16 | −0.2458 (3) | 0.2641 (2) | 0.3233 (5) | 0.031 (1) |
| C17 | −0.2879 (3) | 0.3258 (2) | 0.4069 (5) | 0.031 (1) |
| C18 | −0.1998 (3) | 0.3758 (2) | 0.3740 (5) | 0.028 (1) |
| C19 | −0.1677 (3) | 0.3562 (2) | 0.2083 (5) | 0.030 (1) |
| C20 | −0.0570 (3) | 0.3746 (3) | 0.1654 (5) | 0.037 (1) |

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

| | | | |
|---|---|---|---|
| P1—O4 | 1.582 (3) | P3—O8 | 1.593 (3) |
| P1—O5 | 1.609 (3) | P3—O11 | 1.599 (4) |
| P1—O6 | 1.475 (3) | P3—O12 | 1.484 (3) |
| P1—O7 | 1.484 (3) | P3—O13 | 1.475 (4) |
| P2—O5 | 1.585 (3) | P4—O11 | 1.606 (4) |
| P2—O8 | 1.595 (3) | P4—O14 | 1.584 (3) |
| P2—O9 | 1.483 (4) | P4—O15 | 1.473 (4) |
| P2—O10 | 1.478 (4) | P4—O16 | 1.480 (3) |
| O4—P1—O5 | 102.4 (2) | O11—P3—O12 | 108.9 (2) |
| O4—P1—O6 | 105.4 (2) | O11—P3—O13 | 107.8 (2) |
| O4—P1—O7 | 111.2 (2) | O12—P3—O13 | 119.8 (2) |
| O5—P1—O6 | 111.2 (2) | O11—P4—O14 | 101.1 (2) |
| O5—P1—O7 | 105.7 (2) | O11—P4—O15 | 111.7 (2) |
| O6—P1—O7 | 119.6 (2) | O11—P4—O16 | 107.8 (2) |
| O5—P2—O8 | 99.1 (2) | O14—P4—O15 | 105.4 (2) |
| O5—P2—O9 | 108.4 (2) | O14—P4—O16 | 110.8 (2) |
| O5—P2—O10 | 110.5 (2) | O15—P4—O16 | 118.7 (2) |
| O8—P2—O9 | 109.5 (2) | P1—O4—C10 | 121.1 (3) |
| O8—P2—O10 | 110.5 (2) | P1—O5—P2 | 130.7 (2) |
| O9—P2—O10 | 117.3 (2) | P2—O8—P3 | 128.4 (2) |
| O8—P3—O11 | 100.3 (2) | P3—O11—P4 | 129.2 (2) |
| O8—P3—O12 | 109.7 (2) | P4—O14—C20 | 122.2 (3) |
| O8—P3—O13 | 108.5 (2) | | |
| P1—O4—C10—C9 | 165.9 (3) | P3—O11—P4—O14 | −159.4 (3) |
| P1—O5—P2—O8 | 74.7 (3) | P4—O11—P3—O8 | 105.4 (3) |
| P2—O5—P1—O4 | −135.0 (3) | P4—O14—C20—C19 | 157.8 (3) |
| P2—O8—P3—O11 | −124.8 (3) | O5—P1—O4—C10 | −67.3 (4) |
| P3—O8—P2—O5 | −164.3 (3) | O11—P4—O14—C20 | −65.3 (4) |

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1993). Program(s) used to solve structure: *TEXSAN*; *SAPI*91 (Fan, 1991). Program(s) used to refine structure: *TEXSAN LS*. Molecular graphics: *TEXSAN*; *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *TEXSAN FINISH*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including contact distances, have been deposited with the IUCr (Reference: AS1187). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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# Trimethylammonium Trichlorostannate(II)

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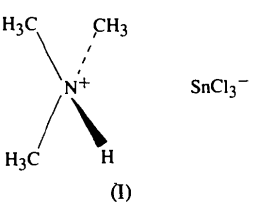
## Abstract

The $NHMe_3^+$ and $SnCl_3^-$ ions both have approximate threefold symmetry and lie on a mirror plane. The anion is pyramidal with Sn—Cl 2.515 (4) and 2.500 (3) Å and Cl—Sn—Cl 90.6 (2) and 89.1 (1)°. The pseudo-threefold axes of the ions are superimposed with close Cl···H contacts of 2.61 (7) and 2.71 (6) Å.

## Comment

The structure of the title compound, (I), is ionic with $SnCl_3^-$ and $NHMe_3^+$ lying on a mirror plane. The molecular dimensions (Table 2) show that the trimethylammonium ion has the usual tetrahedral shape and that the trichlorostannate ion is pyramidal. Both ions possess non-crystallographic threefold symmetry, and an unusual feature of the structure is the coincidence of their threefold axes (Fig. 1). The axial hydrogen of the trimethylammonium ion is aligned in the direction of the Cl atoms (Fig. 2).




(I)

The Cl···H distances (Table 3) are significantly less than the sum of the van der Waals distances, indicating weak bond formation. A search of the Cambridge Structural Database (CSD) (Allen *et al.*, 1991) revealed 11 other organometallic structures (CSD codenames ENCOSN, PECOTB20, PPECOT20, BITXUX, DOWCIJ, DUSWOD, GEHTUI, KAHWAR, SIGMEA, SIGNIF and VOGRAK) involving the $SnCl_3^-$ ion. In