C11*A*—C12*A*	1.498 (5)	C11*B*—C12*B*	1.503 (5)
C11*A*—C14*A*	1.496 (4)	C11*B*—C14*B*	1.495 (5)
O1*A*—Pd1*A*—N1*A*	81.6 (1)	O1*B*—Pd1*B*—N1*B*	82.2 (1)
O1*A*—Pd1*A*—N2*A*	95.9 (1)	O1*B*—Pd1*B*—N2*B*	93.4 (1)
O1*A*—Pd1*A*—C11*A*	160.44 (9)	O1*B*—Pd1*B*—C11*B*	161.82 (9)
N1*A*—Pd1*A*—N2*A*	175.3 (1)	N1*B*—Pd1*B*—N2*B*	174.9 (1)
N1*A*—Pd1*A*—C11*A*	80.8 (1)	N1*B*—Pd1*B*—C11*B*	81.0 (1)
N2*A*—Pd1*A*—C11*A*	102.2 (1)	N2*B*—Pd1*B*—C11*B*	103.6 (1)

Pd1*A*—N1*A*—C6*A*—C1*A*	−5.7 (4)
Pd1*A*—N1*A*—C9*A*—C10*A*	4.8 (4)
Pd1*A*—O1*A*—C1*A*—C6*A*	6.3 (4)
N1*A*—Pd1*A*—C11*A*—C10*A*	−25.3 (2)
N1*A*—Pd1*A*—O1*A*—C1*A*	−7.1 (2)
O1*A*—Pd1*A*—N1*A*—C6*A*	7.0 (2)
C11*A*—Pd1*A*—N1*A*—C9*A*	13.2 (3)
C9*A*—C10*A*—C11*A*—Pd1*A*	34.6 (3)
N1*A*—C9*A*—C10*A*—C11*A*	−27.4 (4)
C10*A*—C11*A*—C14*A*—O4*A*	138.1 (4)
O1*A*—C1*A*—C6*A*—N1*A*	−0.9 (5)
Pd1*B*—N1*B*—C6*B*—C1*B*	−4.2 (4)
Pd1*B*—N1*B*—C9*B*—C10*B*	5.9 (4)
Pd1*B*—O1*B*—C1*B*—C6*B*	6.3 (4)
N1*B*—Pd1*B*—C11*B*—C10*B*	−22.3 (2)
N1*B*—Pd1*B*—O1*B*—C1*B*	−6.6 (3)
O1*B*—Pd1*B*—N1*B*—C6*B*	5.9 (2)
C11*B*—Pd1*B*—N1*B*—C9*B*	10.6 (3)
C9*B*—C10*B*—C11*B*—Pd1*B*	31.6 (3)
N1*B*—C9*B*—C10*B*—C11*B*	−26.1 (4)
C10*B*—C11*B*—C14*B*—O4*B*	−28.9 (5)
O1*B*—C1*B*—C6*B*—N1*B*	−1.8 (5)
C22*B*—O2*B*—C10*B*—C11*B*	52.3 (4)

The H atoms were placed in calculated positions with C—H 0.95 Å and isotropic displacement parameters set at 1.3 times those of the bonded C atoms. Methyl H atoms at C13 were calculated in conformations guided by difference maps. Attempts at refining the H atoms in least squares proved unsuccessful and they were thus left in calculated unrefined positions. The approximate pseudo-center caused no refinement problems. The highest residual density in the difference map is located 0.988 Å from Pd1*A*.

Data collection: *CAD*-4 *Operations Manual* (Enraf–Nonius, 1977). Cell refinement: *CAD*-4 *Operations Manual.* Data reduction: *PROCESS* in *MolEN* (Fair, 1990). Program(s) used to solve structure: Patterson and Fourier methods in *MolEN.* Program(s) used to refine structure: *LSFM* in *MolEN.* Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *CIF IN* in *MolEN.*

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1380). Services for accessing these data are described at the back of the journal.

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The Inserting Host–Guest System in $[N(CH_3)_4]_8[(CH_3COO)V_{22}O_{54}].4.25H_2O$

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Abstract

The title compound, octakis(tetramethylammonium) docosavanadate acetate 4.25-hydrate, was synthesized and found to crystallize in the tetragonal space group $P4_2/n$. 22 vanadium square pyramids form a barrel-shaped cluster which hosts the acetate ion; this is the first case where an organic molecule is hosted by a vanadium oxide cluster. The clusters are linked into a three-dimensional net by hydrogen bonds with water molecules. Tetramethylammonium ions fill the space between the clusters.

Comment

Recently, it was found that mild hydrothermal synthesis in the presence of the tetramethylammonium template, $[N(CH_3)_4]^+$ (TMA), leads to the formation of metastable vanadium oxide structures with open frameworks: $(TMA)V_4O_{10}$ (Zavalij *et al.*, 1996), $Li_xV_2O_4.H_2O$ (Chirayil *et al.*, 1996), $(TMA)V_3O_7$ (Zavalij *et al.*, 1997*a*) and $(TMA)V_8O_{20}$ (Chirayil *et al.*, 1997). Nevertheless, polyvanadate clusters can often be formed as well. We previously reported the decavanadate compounds with mixed cations $[Li(H_2O)]_2(TMA)_4[V_{10}O_{28}].4H_2O$ (Zavalij *et al.*, 1997) and $Na_4(TMA)_2[V_{10}O_{28}].20H_2O$

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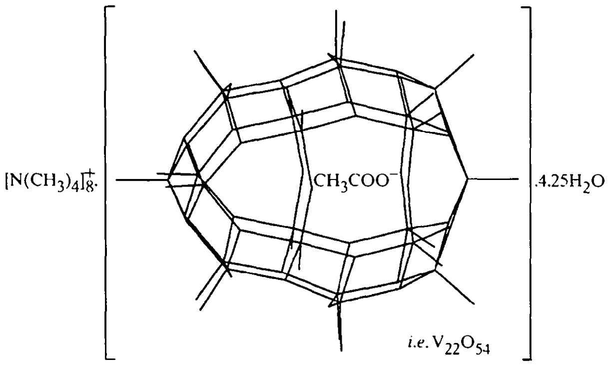
(Zavalij *et al.*, 1997*b*), and also $(TMA)_4[H_2V_{10}O_{28}]$-$(CH_3COOH).2.7H_2O$ (Pecquenard *et al.*, 1998).

This work presents a new compound, (I), formed by a docosavanadate cluster, *i.e.* $V_{22}O_{54}$, tetramethylammonium cations, and water of crystallization. An acetate ion forms an inserting guest–host system with the cluster. This type of cluster can also host such ions as perchlorate in $[N(CH_2CH_3)_4]_6[(ClO_4)HV_{22}O_{54}]$ (Müller *et al.*, 1991) and molybdate in $(TMA)_8[(MoO_4)V_{22}O_{54}]Cl$ (Nazar, 1996). The latter would be isostructural with the title compound but for the guest anion and solvent (Cl^- instead of water of crystallization). The size of this ellipsoidal cave can accommodate an anion even bigger than acetate since the distances from its center to the surrounding O atoms vary from 3.8 to 4.4 Å.



(I)

The coordination polyhedra of the V atoms are square pyramids (SPs) that share edges and corners and form a barrel-shaped docosavanadate cluster (Fig. 1). A double-bonded O atom at the apex of the SP is directed outside the cluster. Two SPs form the top and bottom of the barrel-shaped cluster and are connected together by four chains. Each of these chains consists of five SPs that share the neighboring edges of the base. The local or non-crystallographic symmetry of the docosavanadate cluster, $\bar{4}m2$, is higher than crystallographic symmetry $\bar{4}$, which is probably due to the packing effect since the local symmetry of the cluster does not match the crystallographic axes (Fig. 2). The CH_3COO^- ion hosted in the cluster is disordered in two opposite directions due to coincidence of the $\bar{4}$ axis and the center of the C—C bond, so that the carboxyl groups are directed in opposite directions. Therefore, the occupation factor is 1 for the C1*a* and 0.5 for the O1*a* atom. The guest ions are situated so that the O atoms are directed toward the bases of 20 SPs [V···O1*a* 2.8 (1)–3.2 (1) Å and O═V···O1*a* 161 (1)–175 (1)°]. It is very common for a vanadium(+4,+5) oxide framework to complete square-pyramidal coordination to a distorted octahedron by an additional weakly bonded O atom or other donor of electrons in a position across from the double-bonded O atom.

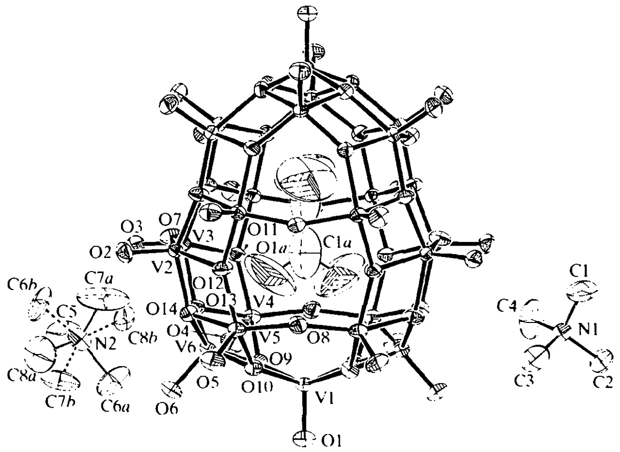


Fig. 1. Displacement ellipsoid plots of the $[(CH_3COO)V_{22}O_{54}]^{8-}$ and $[N(CH_3)_4]^+$ ions shown at 50% probability levels.

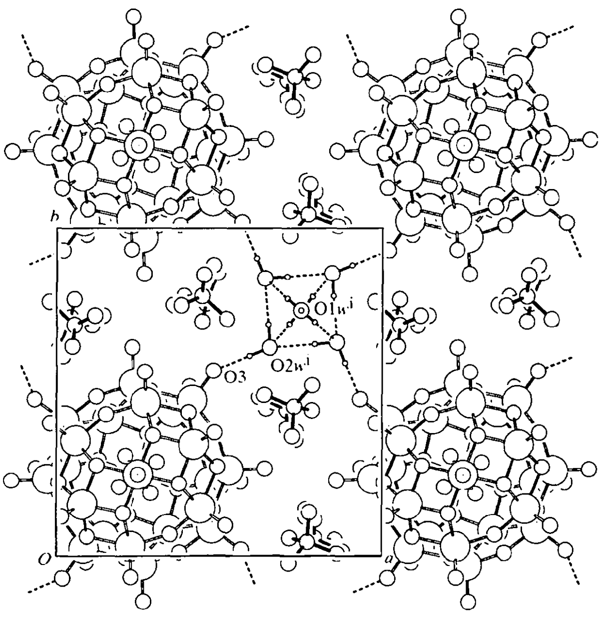


Fig. 2. The packing of the $[(CH_3COO)V_{22}O_{54}]^{8-}$ anions, the $[N(CH_3)_4]^+$ cations and the disordered water molecules viewed along the *c* axis. Hydrogen bonds are shown as dashed lines. [Symmetry code: (i) $\frac{1}{2}+x$, $\frac{1}{2}+y$, $1-z$.]

The particularly disordered water molecules, whose occupation factors are 0.87 (4) and 0.63 (2) for O1*w* and O2*w*, respectively, form an unusual octahedral association (Fig. 2). These water molecules are linked to each other and to the vanadate cluster by hydrogen bonds so that they form a layer in the *ab* plane. Two other hydrogen bonds, O1*w*···O1, link the layers together. They are parallel to the *c* axis and are therefore not shown in Fig. 2. The TMA cations work as separators between each pair of clusters in the *ab* plane. One of the TMA ions (N2) is disordered, so that its two orientations, with probabilities 75 and 25%, differ from each other by an ~60° rotation about the C5—N2 bond.

Experimental

The reaction mixture consisted of V_2O_5, (TMA)OH and LiOH in a 1:2:1 molar ratio acidified with 3 *M* acetic acid in the pH range 5.2–6.2. Hydrothermal synthesis was conducted at 458 K for 3 d. The cluster compound is often encountered with the known phases (TMA)V_3O_7 (Zavalij *et al.*, 1997*a*), (TMA)V_4O_{10} (Zavalij *et al.*, 1996) and $Li_xV_2O_4.H_2O$ (Chirayil *et al.*, 1996). Black crystals of (I) were manually separated for single-crystal X-ray diffraction analysis.

Crystal data

$(C_4H_{12}N)_8[V_{22}O_{54}]$-$(C_2H_3O_2).4.25H_2O$
$M_r = 2713.4$
Tetragonal
$P4_2/n$
$a = 14.4890\,(2)$ Å
$c = 21.5432\,(3)$ Å
$V = 4522.6\,(2)$ Å^3
$Z = 2$
$D_x = 1.993$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 512 reflections
$\theta = 3.17$–$27.58°$
$\mu = 2.25$ mm^{-1}
$T = 295$ K
Prism
0.24 × 0.24 × 0.18 mm
Black

Data collection

Siemens SMART CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\rm min} = 0.57$, $T_{\rm max} = 0.67$
27 168 measured reflections
5459 independent reflections

4991 reflections with $F_o > 4\sigma(F_o)$
$R_{\rm int} = 0.051$
$\theta_{\rm max} = 28.2°$
$h = -18 \rightarrow 19$
$k = -19 \rightarrow 15$
$l = -19 \rightarrow 28$
Intensity decay: 0.6%

Refinement

Refinement on F
$R = 0.052$
$wR = 0.076$
$S = 1.53$
4991 reflections
321 parameters
H atoms not refined
$w = 1/[\sigma(F_o)^2 + 0.004F_o^2]$

$(\Delta/\sigma)_{\rm max} = 0.01$
$\Delta\rho_{\rm max} = 0.70$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.73$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

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

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U^{ij}a^ia^j\mathbf{a}_i.\mathbf{a}_j.$$

	Occupancy	x	y	z	$U_{\rm eq}$
V1	1.0	1/4	1/4	0.03148 (4)	0.0220 (3)
V2	1.0	0.23621 (4)	0.52889 (4)	0.21924 (3)	0.0213 (2)
V3	1.0	0.41587 (4)	0.47764 (4)	0.27946 (3)	0.0213 (2)
V4	1.0	0.35498 (5)	0.44208 (5)	0.11479 (3)	0.0235 (2)
V5	1.0	0.02899 (4)	0.27279 (5)	0.11561 (3)	0.0232 (2)
V6	1.0	0.15704 (4)	0.42073 (4)	0.07909 (3)	0.0214 (2)
O1	1.0	1/4	1/4	−0.0435 (2)	0.0341 (13)
O2	1.0	0.2319 (2)	0.6396 (2)	0.21465 (1)	0.0351 (10)
O3	1.0	0.4809 (2)	0.5682 (2)	0.2807 (2)	0.0356 (10)
O4	1.0	0.4077 (2)	0.5201 (2)	0.0748 (2)	0.0377 (10)
O5	1.0	−0.0652 (2)	0.2705 (3)	0.0757 (2)	0.0387 (10)
O6	1.0	0.1208 (2)	0.4844 (2)	0.02232 (2)	0.0360 (10)
O7	1.0	0.1224 (2)	0.4895 (2)	0.23468 (1)	0.0307 (9)
O8	1.0	0.4344 (2)	0.3481 (2)	0.12371 (1)	0.0295 (9)
O9	1.0	0.2784 (2)	0.3760 (2)	0.05653 (1)	0.0244 (8)
O10	1.0	0.1297 (2)	0.2953 (2)	0.05835 (1)	0.0247 (8)
O11	1.0	0.3595 (2)	0.4799 (2)	0.19908 (1)	0.0285 (8)
O12	1.0	0.2864 (2)	0.5065 (2)	0.29973 (1)	0.0305 (9)
O13	1.0	0.2258 (2)	0.4941 (2)	0.13063 (1)	0.0277 (8)
O14	1.0	0.0591 (2)	0.4057 (2)	0.13231 (1)	0.0254 (8)
O1*w*	0.87 (4)	1/4	1/4	0.6694 (4)	0.158 (9)
O2*w*	0.63 (2)	0.1540 (7)	0.1421 (9)	0.7535 (5)	0.119 (6)
N1	1.0	0.9579 (3)	0.2705 (3)	0.8919 (2)	0.0326 (11)
C1	1.0	0.9464 (6)	0.2162 (5)	0.8328 (3)	0.070 (3)
C2	1.0	1.0237 (4)	0.2164 (5)	0.9319 (3)	0.052 (2)
C3	1.0	0.8676 (4)	0.2769 (6)	0.9237 (4)	0.070 (3)
C4	1.0	0.9934 (6)	0.3632 (5)	0.8776 (5)	0.087 (4)
N2	1.0	0.7933 (3)	0.0535 (3)	0.1100 (2)	0.0418 (13)
C5	1.0	0.7239 (6)	−0.0220 (7)	0.1117 (4)	0.080 (3)
C6a	0.75 (2)	0.7656 (8)	0.1177 (8)	0.0529 (5)	0.080 (4)
C7a	0.75 (2)	0.793 (2)	0.1088 (9)	0.1668 (5)	0.145 (8)
C8a	0.75 (2)	0.8862 (7)	0.0190 (11)	0.0917 (7)	0.098 (6)
C6*b*	0.25 (2)	0.879 (2)	0.013 (2)	0.161 (2)	0.072 (10)
C7*b*	0.25 (2)	0.8568 (13)	0.048 (2)	0.0555 (9)	0.057 (8)
C8*b*	0.25 (2)	0.766 (2)	0.142 (2)	0.1402 (14)	0.079 (11)
C1*a*	1.0	1/4	1/4	0.2131 (12)	0.144 (12)
O1*a*	0.5	0.289 (2)	0.192 (3)	0.1814 (12)	0.24 (2)

Table 2. *Selected bond lengths (Å)*

V1—O1	1.615 (5)	V4—O9	1.929 (3)
V1—O9	1.948 (3)	V4—O13	2.047 (3)
V1—O9$^{\rm i}$	1.948 (3)	V5—O5	1.613 (3)
V1—O10	1.950 (3)	V5—O8$^{\rm i}$	1.839 (3)
V1—O10$^{\rm i}$	1.950 (3)	V5—O12$^{\rm iii}$	1.905 (3)
V2—O2	1.609 (3)	V5—O10	1.938 (3)
V2—O7	1.776 (3)	V5—O14	2.007 (3)
V2—O12	1.908 (3)	V6—O6	1.620 (3)
V2—O11	1.971 (3)	V6—O13	1.831 (3)
V2—O13	1.980 (3)	V6—O14	1.837 (3)
V3—O3	1.616 (3)	V6—O10	1.913 (3)
V3—O7$^{\rm ii}$	1.826 (3)	V6—O9	1.936 (3)
V3—O11	1.915 (3)	O1—O1*w*$^{\rm ii}$	2.712 (10)
V3—O12	1.971 (3)	O3—O2*w*$^{\rm iv}$	2.825 (11)
V3—O14$^{\rm ii}$	1.979 (3)	O1*w*—O2*w*$^{\rm v}$	2.671 (13)
V4—O4	1.613 (4)	O1*w*—O2*w*	2.768 (13)
V4—O8	1.793 (3)	O2*w*—O2*w*$^{\rm v}$	2.96 (2)
V4—O11	1.898 (3)		

Symmetry codes: (i) $\frac{1}{2}-x, \frac{1}{2}-y, z$; (ii) $y, \frac{1}{2}-x, \frac{1}{2}-z$; (iii) $\frac{1}{2}-y, x, \frac{1}{2}-z$; (iv) $\frac{1}{2}+x, \frac{1}{2}+y, 1-z$; (v) $y, \frac{1}{2}-x, \frac{3}{2}-z$.

The crystal structure of the title compound was solved by direct methods. All V atoms and most O atoms were located from the *E* map. The remainder of the non-H atoms were found from the following difference Fourier syntheses. To locate the disordered acetate, the two-dimensional sections of the difference Fourier map were built. H atoms were located from difference Fourier synthesis, except for those of the disordered residues.

Data collection: *SMART* (Siemens, 1995). Cell refinement: *SAINT* (Siemens, 1995). Data reduction: *SAINT*. Program(s) used to solve structure: *CSD* (Akselrud *et al.*, 1993). Program(s) used to refine structure: *CSD*. Molecular graphics: *PLATON*97 and *PLUTON*97 (Spek, 1990). Software used to prepare material for publication: *CSD*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1386). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1998). C**54**, 1444–1446

Bis{[2,6-diacetylpyridine bis(2-furoylhydrazone)(1−)]dimethyltin(IV)} *trans*-Tetrachlorodimethylstannate(IV)

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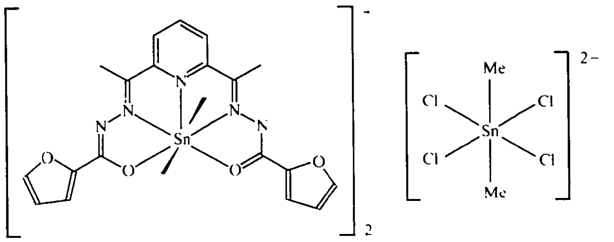
Abstract

The structure of the title compound, $[Sn(CH_3)_2(C_{19}H_{16}N_5O_4)]_2[SnCl_4(CH_3)_2]$, revealed the presence of two complexes. One is monocationic and consists of a heptacoordinated Sn^{IV} atom in a distorted pentagonal-bipyramidal geometry, with an aroylhydrazone ligand acting as a pentadentate species in the equatorial plane and two methyl groups in axial positions. The other complex is dianionic and has a hexacoordinated Sn^{IV} atom at the origin.

Comment

The chelating properties of 2,6-diacetylpyridine bis(aroylhydrazone)s (aroyl is benzoyl, picolinoyl, salicyloyl, thenoyl or aminobenzoyl) have been investigated and only one complexation mode has been found, *i.e.* with the aroylhydrazone ligands acting as pentadentate molecules and coordinating through the two enolate O, the two azomethine N and the pyridyl N atoms (Pelizzi *et al.*, 1984; Carini *et al.*, 1989).

The structure determination of bis{[2,6-diacetylpyridine bis(2-furoylhydrazone)(1−)]dimethyltin(IV)} *trans*-tetrachlorodimethylstannate(IV), (I), revealed the occurrence of two monocationic complexes surrounding a dianionic one, as shown in Fig. 1.



(I)

In the cation, $[Me_2Sn(Hdapf)]^+$, where Hdapf is 2,6-diacetylpyridine bis(2-furoylhydrazone), the ligand in the equatorial plane of the coordination polyhedron is planar, except for the furan groups, which have a dihedral angle of 24.7 (3)° between them. One furan ring (C15–C18, O3) makes a dihedral angle of 21.7 (3)° with the equatorial plane and an angle of 5.5 (3)° with the other furan ring (C19–C22, O4). Both furan rings have atoms with high displacement parameters, especially the C21, C22 and O4 atoms. The presence of a disorder effect in these atoms was investigated, but the calculations did not show any significant improvement of the model.

A selection of bond distances and angles are given in Table 1. Differences can be observed between the bond parameters in both arms of the ligand. The discrepancies in the distances were found in the pairs Sn2—O1/Sn2—O2 and Sn2—N2/Sn2—N4. The pairs of discrepant angles are O1—Sn2—N2/O2—Sn2—N4 and N2—Sn2—N3/N4—Sn2—N3. These differences occur because during the complexation process, the azomethine N1 atom loses its proton and the azomethine N5

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