Supplementary Material

**Tris(2-Pyridylmethylamine)V(O)\textsubscript{2} complexes as counter ions of diprotonated decavanadate anion: Potential antineoplastic activity**

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Figure S1. FTIR spectrum of Compound 1.
Figure S2A and S2B. $^{51}$VNMR spectrum of Compound 1
Table S1. Bond distances for [VO$_2$(tpma)$_2$][H$_2$V$_{10}$O$_{28}$]$^-$•4H$_2$O (1).
Table S2. Bond valence calculations for oxygen atoms in [H$_2$V$_{10}$O$_{28}$]$^-$ for Compound 1.
Checkcif of the deposited structure.
Figure S1. FTIR Spectrum of Compound 1 from 1000-400 cm\(^{-1}\) in the region of decavanadate vibrations V=O, and V-O-V.
Figure S2A. $^{51}\text{V}$ NMR of Compound 1 dissolved in D$_2$O (pH 6.8) at 25°C. Typical signals of decavanadate and a peak at -509 that could be assigned to the [VO$_2$(tpma)] complex.

Figure S2B. After one week $^{51}\text{V}$ NMR of Compound 1 dissolved in D2O (pH 6.8) at 25°C. Typical signals of decavanadate have decreased, the peaks of V1 and V4 have increased and a peak at -509 that could be assigned to the [VO$_2$(tpma)] complex remained.
Table S1. Bond distances for [VO$_2$(tpma)]$_4$[H$_2$V$_{10}$O$_{28}$]·4H$_2$O (1)

| Bond          | Length (Å) | Bond          | Length (Å) |
|---------------|------------|---------------|------------|
| V1-V2         | 3.0983(6)  | N3A-C12A      | 1.342(4)   |
| V1-V3(i)      | 3.0171(6)  | N4A-C14A      | 1.335(4)   |
| V1-V4         | 3.1053(6)  | N4A-C18A      | 1.346(4)   |
| V2-V5         | 3.0586(6)  | C1A-C2A       | 1.487(5)   |
| V3-V4         | 3.0939(7)  | C2A-C3A       | 1.387(5)   |
| V3-V5         | 3.0988(6)  | C3A-C4A       | 1.372(6)   |
| V4-V5         | 3.0587(6)  | C4A-C5A       | 1.368(7)   |
| V1-O1         | 2.2268(17) | C5A-C6A       | 1.370(6)   |
| V1-O2         | 2.0670(17) | C7A-C8A       | 1.490(5)   |
| V1-O3         | 1.7576(18) | C8A-C9A       | 1.382(5)   |
| V1-O6         | 1.6111(18) | C9A-C10A      | 1.379(6)   |
| V1-O10        | 1.9463(18) | C10A-C11A     | 1.359(7)   |
| V1-O14        | 1.9203(17) | C11A-C12A     | 1.386(6)   |
| V2-O1(i)      | 2.1390(16) | C13A-C14A     | 1.493(4)   |
| V2-O1         | 2.1147(16) | C14A-C15A     | 1.389(4)   |
| V2-O2         | 1.9162(17) | C15A-C16A     | 1.367(5)   |
| V2-O5         | 1.6873(17) | C16A-C17A     | 1.370(6)   |
| V2-O9         | 1.6876(17) | C17A-C18A     | 1.371(5)   |
| V2-O14(i)     | 1.9465(17) | V1B-O1B       | 1.630(2)   |
| V3-O1         | 2.2884(17) | V1B-O2B       | 1.624(2)   |
| V3-O2(i)      | 1.9343(17) | V1B-N1B       | 2.258(2)   |
| V3-O4         | 1.7987(18) | V1B-N2B       | 2.111(3)   |
| V3-O8         | 1.6063(18) | V1B-N3B       | 2.277(2)   |
| V3-O12        | 1.9045(17) | V1B-N4B       | 2.123(2)   |
| V3-O14(i)     | 2.0186(17) | N1B-C1B       | 1.490(4)   |
| V4-O1         | 2.3271(16) | N1B-C7B       | 1.485(4)   |
| V4-O3         | 1.9436(18) | N1B-C13B      | 1.486(4)   |
| V4-O4         | 1.8657(18) | N2B-C2B       | 1.354(4)   |
| V4-O5(i)      | 2.0867(18) | N2B-C6B       | 1.342(4)   |
| V4-O7         | 1.6099(19) | N3B-C8B       | 1.340(3)   |
| V4-O11        | 1.7798(18) | N3B-C12B      | 1.341(4)   |
| V5-O1         | 2.3064(16) | N4B-C14B      | 1.351(3)   |
| V5-O9         | 2.0398(18) | N4B-C18B      | 1.339(4)   |
| V5-O10        | 1.9998(18) | C1B-C2B       | 1.495(4)   |
| V5-O11        | 1.8649(18) | C2B-C3A       | 1.383(4)   |
| V5-O12        | 1.7716(18) | C3B-C4B       | 1.375(5)   |
| V5-O13        | 1.6033(19) | C4B-C5B       | 1.379(5)   |
| V1A-O1A       | 1.631(2)   | C5B-C6B       | 1.377(5)   |
| V1A-O2A       | 1.624(2)   | C7B-C8B       | 1.491(4)   |
| V1A-N1A       | 2.250(2)   | C8B-C9B       | 1.382(4)   |
| V1A-N2A       | 2.090(3)   | C9B-C10B      | 1.373(5)   |
| V-O Bond | V-O length (R) | $s = (R/1.791)^{-0.1}$ | $\Sigma s$ |
|----------|----------------|-------------------------|-----------|
| V1-O1    | 2.2268         | 0.32932                 | 1.98708   |
| V2-O1(i) | 2.1390         | 0.40431                 |           |
| V2-O1    | 2.1147         | 0.42857                 |           |
| V3-O1    | 2.2884         | 0.28653                 |           |
| V4-O1    | 2.3271         | 0.26305                 |           |
| V5-O1    | 2.3064         | 0.27531                 |           |
| V1-O2    | 2.0670         | 0.48145                 |           |
| V2-O2    | 1.9162         | 0.70849                 | 1.86528   |
| V3-O2(i) | 1.9343         | 0.67533                 |           |
| V1-O3    | 1.7576         | 1.10077                 | 1.75978   |
| V4-O3    | 1.9436         | 0.65901                 |           |
| V3-O4    | 1.7987         | 0.97836                 | 1.79024   |
| V4-O4    | 1.8657         | 0.81188                 |           |
| V2-O5    | 1.6873         | 1.35552                 | 1.81424   |
| V4-O5(i) | 2.0867         | 0.45871                 |           |
| V1-O6    | 1.6111         | 1.71578                 | 1.71578   |
| V4-O7    | 1.6099         | 1.72232                 | 1.72232   |
| V3-O8    | 1.6063         | 1.74209                 | 1.74209   |
| V2-O9    | 1.6876         | 1.35429                 | 1.86939   |
| V5-O9    | 2.0398         | 0.51509                 |           |
| V1-O10   | 1.9463         | 0.65436                 | 1.22421   |
| V5-O10   | 1.9998         | 0.56984                 |           |
| V4-O11   | 1.7798         | 1.03251                 | 1.84617   |
| V5-O11   | 1.8649         | 0.81366                 |           |
| V3-O12   | 1.9045         | 0.73098                 | 1.78809   |
| V5-O12   | 1.7716         | 1.05712                 |           |
| V5-O13   | 1.6033         | 1.75878                 | 1.75878   |
| V1-O14   | 1.9203         | 0.70082                 | 1.89813   |
| V2-O14(i) | 1.9465       | 0.65402                 |           |
| V3-O14(i) | 2.0186       | 0.54329                 |           |

Symmetry codes: (i) 1-x, -y, 1-z
### Supplementary Material

1. **checkCIF/PLATON report**

1.1.1 **Structure factors have been supplied for datablock(s) decatpa**

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors were found.

#### Datablock: decatpa

| Bond precision: | C-C = 0.0053 Å | Wavelength=0.71073 |
|-----------------|----------------|-------------------|

| Cell:           | a=11.6736(10)  | b=23.7172(17)     | c=16.5914(12) |
|-----------------|----------------|------------------|--------------|
| Temperature:    | 300 K          |                  |              |
| Volume          | Calculated     | 4570.1(6)        |               |
| Space group     | P 21/c         | P 1 21/c 1       |              |
| Hall group      | -P 2ybc        | -P 2ybc          |              |
| Moiety formula  | H2 O28 V10, 4(C18 H18 N4 O2 H2 O28 V10, 4(C18 H18 N4O2 | V), 4(H2 O) |
| Sum formula     | C72 H82 N16 O40 V14 | C72 H82 N16 O40 V14 | V), 4(H2 O) |
| Mr              | 2524.70        | 2524.69          |              |
| Dx, g cm⁻³      | 1.835          | 1.835            |              |
| Z                | 2              | 2                |              |
| Mu (mm⁻¹)       | 1.457          | 1.457            |              |
| F000            | 2536.0         | 2536.0           |              |
| F000’           | 2545.51        |                  |              |
| h,k,lmax        | 15,32,22       | 15,32,22         |              |
| Nref            | 11920          | 11854            |              |
| Tmin,Tmax       | 0.840, 0.864   | 0.660, 0.746     |              |
| Tmin’           | 0.840          |                  |              |

Correction method=# Reported T Limits: Tmin=0.660 Tmax=0.746 AbsCorr = NONE
Data completeness = 0.994

Theta(max) = 28.797

R(reflections) = 0.0393 (8572)

wR2(reflections) = 0.0917 (11854)

S = 1.049

Npar = 640
The following ALERTS were generated. Each ALERT has the format test-name_ALERT_alert-type_alert-level.

Click on the hyperlinks for more details of the test.

**Alert level C**
- PLAT260_ALERT_2_C: Large Average Ueq of Residue Including O2W 0.170 Check
- PLAT355_ALERT_3_C: Long O-H (X0.82,N0.98A) O10 - H10 . 1.05 Ang.
- PLAT910_ALERT_3_C: Missing # of FCF Reflections Below Theta(Min). 5 Note
- PLAT911_ALERT_3_C: Missing FCF Refl Between Thmin & STh/L= 0.600 6 Report
- PLAT976_ALERT_2_C: Check Calcd Resid. Dens. O2W 0.57A From O2W -0.51 eA-3
- PLAT977_ALERT_2_C: Check Calcd Resid. Dens. O2W 0.83A From O2W -0.50 eA-3

**Alert level G**
- PLAT007_ALERT_5_G: Number of Unrefined Donor-H Atoms ................ 5 Report
- PLAT083_ALERT_2_G: SHEXL Second Parameter in WGHT Unusually Large 6.40 Why ?
- PLAT720_ALERT_4_G: Number of Unusual/Non-Standard Labels ........... 12 Note
- PLAT764_ALERT_4_G: Overcomplete CIF Bond List Detected (Rep/Expd) . 1.16 Ratio
- PLAT794_ALERT_5_G: Tentative Bond Valency for V1 (V) . 5.03 Info
- PLAT794_ALERT_5_G: Tentative Bond Valency for V1A (V) . 5.36 Info
- PLAT794_ALERT_5_G: Tentative Bond Valency for V1B (V) . 5.28 Info
- PLAT794_ALERT_5_G: Tentative Bond Valency for V2 (V) . 4.98 Info
- PLAT794_ALERT_5_G: Tentative Bond Valency for V3 (V) . 5.00 Info
- PLAT794_ALERT_5_G: Tentative Bond Valency for V4 (V) . 4.98 Info
- PLAT794_ALERT_5_G: Tentative Bond Valency for V5 (V) . 5.02 Info
- PLAT883_ALERT_1_G: No Info/Value for _atom_sites_solution_primary . Please Do !
- PLAT912_ALERT_4_G: Missing # of FCF Reflections Above STh/L= 0.600 56 Note
- PLAT933_ALERT_2_G: Number of OMIT Records in Embedded .res File ... 1 Note
- PLAT978_ALERT_2_G: Number C-C Bonds with Positive Residual Density. 1 Info

0 ALERT level A = Most likely a serious problem - resolve or explain
0 ALERT level B = A potentially serious problem, consider carefully
7 ALERT level C = Check. Ensure it is not caused by an omission or oversight
15 ALERT level G = General information/check it is not something unexpected

1 ALERT type 1: CIF construction/syntax error, inconsistent or missing data
7 ALERT type 2: Indicator that the structure model may be wrong or deficient
3 ALERT type 3: Indicator that the structure quality may be low
3 ALERT type 4: Improvement, methodology, query or suggestion
8 ALERT type 5: Informative message, check

Validation response form

1.1.2 Please find below a validation response form (VRF) that can be filled in and pasted into your CIF.

```
# start Validation Reply Form _vrf_PLAT260_decatpa
```
PROBLEM: Large Average Ueq of Residue Including O2W 0.170 Check
RESPONSE: ...
_vrf_PLAT355_decatpa

PROBLEM: Long O·H (X0.82, N0.98A) O10 - H10 . 1.05 Ang.
RESPONSE: ...

_vrf_PLAT910_decatpa

PROBLEM: Missing # of FCF Reflection(s) Below Theta(Min). 5 Note
RESPONSE: ...

_vrf_PLAT911_decatpa

PROBLEM: Missing FCF Refl Between Thmin & STh/L= 0.600 6 Report
RESPONSE: ...

_vrf_PLAT976_decatpa

PROBLEM: Check Calcd Resid. Dens. 0.57A From O2W -0.51 eA-3
RESPONSE: ...

_vrf_PLAT977_decatpa

PROBLEM: Check Negative Difference Density on H2WB -0.50 eA-3
RESPONSE: ...

# end Validation Reply Form
1.1.3 It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (Acta Crystallographica, Journal of Applied Crystallography, Journal of Synchrotron Radiation); however, if you intend to submit to Acta Crystallographica Section C or E or IUCrData, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

1.2 Publication of your CIF in other journals

1.2.1 Please refer to the Notes for Authors of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 13/07/2021; check.def file version of 13/07/2021
Datablock decatpo - ellipsoid plot

Prob = 50
Temp = 300

PLATON-Sep 28 02:19:12 2021 - (130721)

Z 162 decatpo P 1 21/c 1 R = 0.04 RES= 0 -37 X