Crystal structure of vanadyl monohydrogen phosphate bis(2,2'-dipyridine) monohydrate, $C_{20}H_{19}N_4O_9PV_2$

O. M. Yaghi, H. Li and T. L. Groy

Arizona State University, Department of Chemistry and Biochemistry, Goldwater Center for Science and Engineering, Tempe, AZ 85287-1604, USA

Received December 4, 1996, CSD-No. 402742

Source of material: A mixture of 500 mg of vanadium(V) oxide, 2190 mg of phosphoric acid (85%) and 1540 mg of 2,2'-bipyridine was dissolved in 13 ml of water and stirred for 10 minutes at room temperature, then transferred to a stainless steel vessel which was sealed and placed in a programmable furnace. The temperature was raised to 443 K at a rate of 5 K/min and held at that temperature for four days. The temperature was lowered to ambient at a rate of 0.1 K/min. The resulting olive green parallelepipedal crystals of the title compound were mechanically separated from a mixture containing black and green solid, then washed with distilled water (3 x 15 ml) and ethanol (3 x 10 ml) and air dried to give 500 mg (30% based on V_2O_5). Elemental microanalysis calculated for $C_{20}H_{19}N_4O_9PV_2$: C, 40.56; H, 3.23; N, 9.46; P, 5.23; V, 17.20. Found: C, 39.99; H, 3.18; N, 9.38; P, 5.40; V, 17.27.

$C_{20}H_{19}N_4O_9PV_2$, monoclinic, $P12_1/c1$ (No. 14), $a = 9.256(2)$ Å, $b = 16.254(5)$ Å, $c = 15.383(5)$ Å, $\beta = 99.28(2)^\circ$, $V = 2284.0$ Å³, $Z = 4$, $R(F) = 0.044$, $R_w(F) = 0.100$.

Table 1. Parameters used for the X-ray data collection

Crystal:	olive green parallelepiped, size 0.25 x 0.28 x 0.80 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	9.49 cm ⁻¹
Diffractometer:	Siemens P3
Scan mode:	ω
$T_{\text{measurement}}$:	293 K
$2\theta_{\text{max}}$:	51°
$N(hkl)_{\text{unique}}$:	4179
Criterion for I_o :	$I_o > 2 \sigma(I_o)$
$N(\text{param})_{\text{refined}}$:	332
Program:	SHELXTL-PC

Table 2. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U_{iso}
H(40)	4e	-0.095(2)	0.947(3)	0.519(3)	0.053
H(3)	4e	0.6267(4)	0.5919(3)	0.4224(3)	0.053
H(4)	4e	0.7755(4)	0.7040(3)	0.4736(3)	0.058
H(5)	4e	0.6674(4)	0.8259(3)	0.5149(3)	0.050
H(6)	4e	0.4104(4)	0.8310(2)	0.5060(2)	0.042
H(3')	4e	0.4680(5)	0.4910(3)	0.3767(3)	0.058
H(4')	4e	0.2872(5)	0.3896(3)	0.3440(3)	0.066
H(5')	4e	0.0464(5)	0.4186(3)	0.3634(3)	0.059
H(6')	4e	-0.0119(4)	0.5515(2)	0.4036(3)	0.048
H(13)	4e	-0.4747(4)	0.6100(3)	0.1631(3)	0.055
H(14)	4e	-0.6402(4)	0.7071(3)	0.2042(3)	0.060
H(15)	4e	-0.5520(4)	0.8254(3)	0.2789(3)	0.049
H(16)	4e	-0.2989(4)	0.8514(2)	0.3040(2)	0.041
H(13')	4e	-0.3052(5)	0.5307(2)	0.1243(3)	0.049
H(14')	4e	-0.1129(5)	0.4536(2)	0.0845(3)	0.057
H(15')	4e	0.1335(5)	0.4976(2)	0.1319(3)	0.051
H(16')	4e	0.1720(4)	0.6208(2)	0.2096(3)	0.047
H(1W)	4e	0.255(7)	0.587(2)	0.625(3)	0.115
H(2W)	4e	0.340(6)	0.556(3)	0.696(3)	0.115

Table 3. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
V(1)	4e	0.13204(6)	0.71766(4)	0.45418(4)	0.0212(3)	0.0283(3)	0.0312(3)	0.0042(2)	0.0066(2)	-0.0039(3)
V(2)	4e	0.00026(6)	0.77043(4)	0.26426(4)	0.0236(3)	0.0278(3)	0.0296(3)	-0.0045(2)	0.0073(2)	-0.0074(3)
P(1)	4e	0.03232(9)	0.89897(5)	0.42032(6)	0.0232(4)	0.0221(4)	0.0348(5)	0.0013(3)	0.0042(4)	-0.0087(4)
O(1)	4e	0.1453(3)	0.8349(2)	0.4639(2)	0.029(1)	0.029(1)	0.040(1)	0.005(1)	-0.002(1)	-0.009(1)
O(2)	4e	-0.0333(3)	0.8699(2)	0.3266(2)	0.035(1)	0.029(1)	0.037(1)	0.004(1)	0.001(1)	-0.007(1)
O(3)	4e	0.1044(3)	0.9822(2)	0.4164(2)	0.040(2)	0.031(1)	0.042(2)	-0.009(1)	0.014(1)	-0.007(1)
O(4)	4e	-0.0968(3)	0.9039(2)	0.4730(2)	0.029(1)	0.032(1)	0.047(2)	0.001(1)	0.013(1)	-0.008(1)

Table 3. (Continued)

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
O(1A)	4e	0.1198(3)	0.6880(2)	0.5522(2)	0.049(2)	0.049(2)	0.035(2)	0.007(1)	0.012(1)	0.000(1)
O(2A)	4e	−0.0014(3)	0.8092(2)	0.1676(2)	0.052(2)	0.044(2)	0.033(1)	−0.015(1)	0.012(1)	−0.004(1)
O(1B)	4e	−0.0341(2)	0.7077(2)	0.3951(2)	0.022(1)	0.035(1)	0.037(1)	0.002(1)	0.006(1)	−0.006(1)
O(2B)	4e	0.1729(3)	0.7486(2)	0.3040(2)	0.024(1)	0.036(1)	0.047(2)	−0.003(1)	0.008(1)	−0.013(1)
N(1)	4e	0.3710(3)	0.7178(2)	0.4621(2)	0.022(1)	0.037(2)	0.029(2)	0.004(1)	0.004(1)	0.002(1)
C(2)	4e	0.4339(4)	0.6479(2)	0.4376(2)	0.028(2)	0.042(2)	0.032(2)	0.008(2)	0.007(2)	0.006(2)
C(3)	4e	0.5845(4)	0.6416(3)	0.4407(3)	0.029(2)	0.057(3)	0.048(2)	0.012(2)	0.014(2)	0.007(2)
C(4)	4e	0.6712(4)	0.7078(3)	0.4701(3)	0.027(2)	0.073(3)	0.047(2)	0.007(2)	0.014(2)	0.015(2)
C(5)	4e	0.6078(4)	0.7792(3)	0.4946(3)	0.031(2)	0.055(3)	0.039(2)	−0.007(2)	0.005(2)	0.012(2)
C(6)	4e	0.4557(4)	0.7816(2)	0.4893(2)	0.032(2)	0.042(2)	0.032(2)	0.000(2)	0.007(2)	0.003(2)
N(1')	4e	0.1893(3)	0.5978(2)	0.4160(2)	0.030(2)	0.027(2)	0.039(2)	0.008(1)	0.010(1)	0.000(1)
C(2')	4e	0.3299(4)	0.5799(2)	0.4099(2)	0.034(2)	0.035(2)	0.032(2)	0.010(2)	0.010(2)	0.004(2)
C(3')	4e	0.3683(5)	0.5026(3)	0.3825(3)	0.046(2)	0.041(2)	0.060(3)	0.023(2)	0.014(2)	−0.002(2)
C(4')	4e	0.2625(5)	0.4430(3)	0.3640(3)	0.066(3)	0.033(2)	0.066(3)	0.016(2)	0.008(2)	−0.007(2)
C(5')	4e	0.1208(5)	0.4602(3)	0.3739(3)	0.053(3)	0.031(2)	0.063(3)	0.001(2)	0.009(2)	−0.006(2)
C(6')	4e	0.0876(4)	0.5385(2)	0.3987(3)	0.037(2)	0.034(2)	0.050(2)	0.000(2)	0.013(2)	−0.008(2)
N(11)	4e	−0.2390(3)	0.7483(2)	0.2491(2)	0.025(2)	0.032(2)	0.027(1)	−0.002(1)	0.004(1)	0.000(1)
C(12)	4e	−0.2901(4)	0.6778(2)	0.2089(2)	0.029(2)	0.037(2)	0.028(2)	−0.006(2)	0.005(1)	−0.002(2)
C(13)	4e	−0.4398(4)	0.6602(3)	0.1921(3)	0.035(2)	0.057(3)	0.047(2)	−0.017(2)	0.007(2)	−0.008(2)
C(14)	4e	−0.5367(4)	0.7170(3)	0.2169(3)	0.025(2)	0.070(3)	0.057(3)	−0.006(2)	0.012(2)	0.000(2)
C(15)	4e	−0.4852(4)	0.7869(3)	0.2595(3)	0.030(2)	0.053(2)	0.040(2)	0.009(2)	0.012(2)	0.006(2)
C(16)	4e	−0.3351(4)	0.8016(2)	0.2746(2)	0.034(2)	0.034(2)	0.037(2)	0.003(2)	0.008(2)	−0.001(2)
N(11')	4e	−0.0368(3)	0.6488(2)	0.2110(2)	0.031(2)	0.030(2)	0.033(2)	−0.004(1)	0.010(1)	−0.006(1)
C(12')	4e	−0.1758(4)	0.6227(2)	0.1853(2)	0.033(2)	0.030(2)	0.027(2)	−0.009(2)	0.006(2)	−0.000(1)
C(13')	4e	−0.2060(5)	0.5493(2)	0.1400(3)	0.046(2)	0.031(2)	0.045(2)	−0.012(2)	0.005(2)	−0.008(2)
C(14')	4e	−0.0929(5)	0.5031(2)	0.1183(3)	0.067(3)	0.029(2)	0.048(2)	−0.008(2)	0.011(2)	−0.013(2)
C(15')	4e	0.0524(5)	0.5295(2)	0.1450(3)	0.054(3)	0.031(2)	0.046(2)	0.005(2)	0.020(2)	−0.007(2)
C(16')	4e	0.0737(4)	0.6020(2)	0.1906(3)	0.036(2)	0.041(2)	0.044(2)	0.001(2)	0.012(2)	−0.008(2)
O(1W)	4e	0.2774(4)	0.5416(2)	0.6520(3)	0.067(3)	0.052(2)	0.113(3)	0.003(2)	0.022(2)	−0.012(2)

Acknowledgment. The financial support of this work by the National Science Foundation of the United States (CHE-9522303) is gratefully acknowledged.

References

1. Siemens P3 Data Collection Software, Siemens Analytical X-Ray Instruments, Inc., Madison WI, USA 1993.
2. Siemens SHELXTL-PC. Siemens Analytical X-Ray Instruments Inc., Madison, Wisconsin, USA 1993.