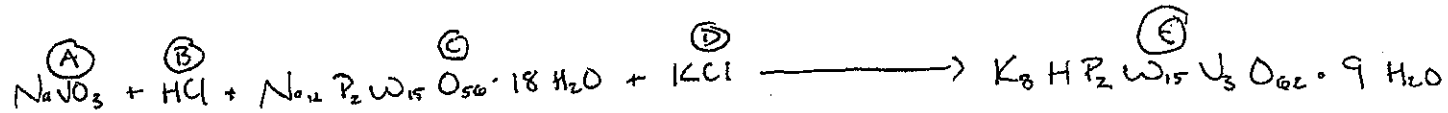

(1) Dissolve (A) in 700 mL HOT H_2O, cool, then add (B)
(2) Stir, then add (C) Slowly
(3) Continue stirring for 10 min, then add (D). filter + crystallize overnight
(4) Dry to yield (E) at 60°C

Compound	FW	d	Quantity	mmoles	equiv	Flamable category from NFPA 704 (red)	Health Category From NFPA 704 (blue)	Instability/Reactivity from NFPA 704 (yellow)	Other from NFPA 704 (white) specify	What is the most common route of exposure: 1. Inhalation; 2. Skin/Eye; 3. Swallowing	Carcinogen, teratogen, mutagen (Y/N)	Oral Toxicity category	Dermal toxicity category	Skin Irritation Category	Eye irritation category	Lachrymator (Y/N)	Explosive (Y/N)	Pyrophoric (Y/N)	Oxidizer (Y/N)	Corrosive (Y/N)
						Big numbers = more hazard						Lower numbers = more hazard								
A	121.9295		4g	32.8		0	2	0	—	1,2,3	N	1	1	1	1	N	N	N	N	Y
B	36.46	1.49	16mL	96		0	3	1	—	2,3	Y	1	1	1	1	N	N	N	N	Y
C	4315.788			10.7																
D	74.5513		100	1350		0	1	0	—	1,3	N	3	3	3	3	N	N	N	N	N
E	4440.204		38g																	

(C) - See sheet 2

(E) - Similar properties to (C) - see Sheets 1 & 2

Is this a repeat experiment or a new experiment? If so, what is the Reaction Number?

Is a standard protocol followed? (Give reference) Finke et al, ~~Tungsten Heteropolytungstate Dioxygen Chem. Chem. 1987, 26, 3058-3064~~ Trisubstituted Heteropolytungstates JACS 1986 2947-2960

What are the reaction conditions and associated processes (heating, cooling, pressure, vacuum, reflux, etc?)

Hot H_2O, cooling, stirring, filtration, drying

What precautions must be made for with respect to the reaction conditions and associated processes?

6M acid used, ~~Tungsten~~ Vanadium present,

Are there specific emergency procedures necessary for this process: Yes/No. If yes, give details:

Acid spill kit, Yes - Skin contact, remove clothing + shower immediately, eyes - wash immediately

Reaction and/or reagent quenching procedures. (How is this reaction stopped? What hazards are involved?)

Strong acid, Tungsten Vanadium

What control measures will be taken during the course of this reaction? (Check boxes)	Safety Glasses	X	Lab Coat	X	Fume Hood	✓	Safety Screen	✓			
	Gloves (type)	Nitrile ✓			Marigolds		Other				
	Scrubbing train (type):				Other:						
Waste Disposal	Chlorinated		Hydrocarbon		Silica		Solids		Heavy Metal ✓		Other
What supervision category does it fall under? (Consult Hazard Rubric)	A		B		C		D				

Researcher Signature

Date 29 May 2013

Supervisor Signature

Date

Chemical Volumes	Micro < 0.5 L 1		0.5 L 2	Normal 3	2 L 4	Large > 2 L 5
Hazard Recognition USE HIGHEST SCORE ONLY	None			Routine		Extreme
Flamable	0		1	2	3	4 5
Health	0		1	2	3	4 5
Reactivity	0		1	2	3	4 5
Other: Specify	0		1	2	3	4 5
Process Conditions	N/A 0	Sub-ambient (P < 1 atm; T < 10°C) 3	Ambient (P = 1 atm; 10°C < T > 40°C) 1	2	3	Extreme 4 5
Explosive Hazard	N/A 0	No 0	Yes 5			
Radiation Hazard	N/A 0	Minimal 1 2	3	Normal 4		High 5
Other Hazard: Specify and Score	N/A 0	Minimal 1 2	3	Normal 4		High 5
Special Hazards	Inhalation Toxicity 0	5	Reactive	0		5
Procedure	Detailed & Written 0		Routine 1 2 3 4			Under Development 5
Personnel Preparedness and Training	Has run reaction at least 4 times 0		Routine (3 times=2, 2 times=3, 1 time=4) 2 3 4			Untrained 5
Ventilation Needed	Hood Used 0		General Lab Only 3			Unventilated room 5
Shielding Needed	Used 0					Not Used 5
Equipment Maintenance	Regularly Performed and Documented 0					Never Performed 5

Total Score 22

RECOMMENDED ACTIONS BASED ON SCORE		
LOW	< 15	Procedure can be performed unsupervised
MODERATE	15 - 25	Procedure can be performed with attention to specific hazards. Supervision by qualified undergraduate or qualified graduate student is recommended.
CAUTIOUS	26 - 30	Procedure may be perfromed with extreme caution. Supervision by faculty member is required
HIGH	30-35	Only faculty may perfrom this procedure
EXTREME	> 35	Procedure must be revised to lower the risk

EMERGENCY RESPONSE

Alarm Method	Evacuation Meeting Point	Phone Number

Preparation of $K_8HP_2W_{15}V_3O_{62}\cdot 9H_2O$. Sodium metavanadate, 4 g (32.8 mmol), was dissolved in 700 mL of hot water and cooled to room temperature followed by the addition of 16 mL of 6 M HCl (96 mmol). To this pale yellow, rapidly stirred solution, 46 g (10.7 mmol) of solid $Na_{12}P_2W_{15}O_{56}\cdot 18H_2O$ was added slowly. The solution becomes homogeneous within a few minutes becoming red-orange in color. Stirring was continued for an additional 10 min after the solution becomes homogeneous followed by addition of 100 g (1350 mmol) of solid KCl. The resulting precipitate was isolated by filtration and crystallized from hot pH 1.5 water overnight (the total solution volume for this crystallization being about 75 mL). The precipitate was isolated by filtration and dried in air in 60 °C to yield 38 g (80%) of a crystalline orange solid. ^{31}P NMR in pD 1.9 D_2O shows two lines at −6.7 and −14.3 ppm. Analysis for $K_8HP_2W_{15}V_3O_{62}\cdot 9H_2O$: K^+ calcd 7.31%, found (Galbraith) 7.34%; Na^+ calcd 0.0%, found (E + R) <0.04%. H_2O (by TGA weight loss at 200 °C) calcd for $9H_2O$, 3.71%; found, 3.68%. ^{51}V NMR (prepared by ion exchange to the Li^+ salt) shows a single resonance at −504 ppm ($\Delta\nu_{1/2}$ = 110 Hz) at 30 °C, pD 6.3. ^{183}W NMR (prepared by ion exchange to the Li^+ salt, undecoupled at 30 °C, pD 6.3): −158.2 (3 W), −178.7 (6 W, $\Delta\nu_{1/2}$ = 54 Hz), and −226.7 (6 W) (Figure 15B). Thirty eight grams of the potassium salt was dissolved in about 200 mL of water and was passed down 50 g of the Amberlyst 15 strongly acidic cation exchange resin charged in the H^+. No retention of the colored material is noted and the colored eluant was collected and the water removed by rotary evaporation of the solvent under water aspirator vacuum using a steam bath. About 32 g of the orange solid was recovered. ^{31}P NMR (2 g/mL of D_2O; pD about 1) shows two lines at −7.6 and −14.3 ppm. ^{51}V NMR (0.5 g/3 mL of D_2O, 21 °C) shows the major resonance at −576 ppm ($\Delta\nu_{1/2}$ = 468 ± 9 Hz) and a smaller (<5%) resonance at −558 ppm. IR (KBr pellet): 1075 (s), 1045 (m), 1010 (sh), 935 (m), 875 (w), 765 cm^{-1} (s, br).