

# Ammonium Decavanadate–Synthesis and Analysis<sup>1</sup>

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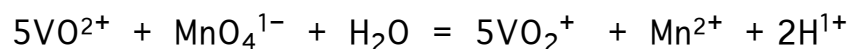
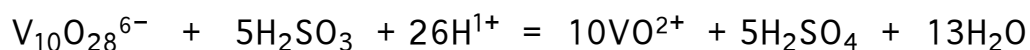
In this experiment, we will synthesize ammonium decavanadate,  $(\text{NH}_4)_6\text{V}_{10}\text{O}_{28}\cdot 6\text{H}_2\text{O}$ , and then confirm the vanadium content by titration with permanganate.

## Synthesis of Ammonium Decavanadate

Weigh 3.0 g of ammonium metavanadate ( $\text{NH}_4\text{VO}_3$ ) into a 250 mL Erlenmeyer flask. Add 100 mL of distilled water and a stir bar and heat to just below boiling, while stirring constantly, until most of the solid has dissolved. Filter the solution and add, with stirring, 4 mL of 50% aqueous acetic acid. Add 150 mL of 95% aq ethanol and cool in an ice bath for 15 minutes. Suction filter the orange product and wash twice with 15 mL portions of ice-cold ethanol. Continue suction for 5 minutes and then transfer the product to a tared beaker. Cover loosely and store for at least two days to allow the crystals to air dry. Weigh after drying to determine the yield.

## Vanadium Analysis

The V in the metavanadate will be reduced to  $\text{V}^{4+}$  and then titrated with  $\text{MnO}_4^{1-}$  according to the following equations:



Weigh two 0.3xxx g samples of your product into 250 mL flasks and dissolve each in 40 mL of 1.5 M  $\text{H}_2\text{SO}_4$  (warm if necessary). In the fume hood, add 50 mL of distilled water and 1 g of  $\text{NaHSO}_3$ . Swirl gently to dissolve and then allow to stand for 5 minutes. After standing, heat to a gentle boil and maintain for 15 minutes to remove excess  $\text{H}_2\text{SO}_3$ . Titrate the solutions with 0.02xxx M  $\text{MnO}_4^{1-}$  while still warm. The equivalence point is the first persistent darkening of the yellow solution.

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<sup>1</sup> adapted from D. C. Harris, *Exploring Chemical Analysis*, 2e, Freeman, 2001, pp 507–509.