Thermo. Titr. Application Note No. H-051

Title:	**Determination of Sodium Hypophosphite**

***Scope*:**	Determination of sodium hypophosphite in electroless plating solutions.

***Principle*:**	Thermometric redox titration of hypophosphite with standard potassium permanganate.

$$[PO_2^{3-} + 2H_2O \leftrightarrow PO_4^{3-} + 4H^+ + 4e] \times 5$$

$$\underline{[MnO_4^- + 8H^+ + 5e \leftrightarrow Mn^{2+} + 4H_2O] \times 4}$$

$$5PO_2^{3-} + 4MnO_4^- + 12H^+ \leftrightarrow 5PO_4^{3-} + 4Mn^{2+} + 6H_2O$$

Thus, 4 mole MnO_4^- ≡ 5mole PO_2^{3-}

Reagents:	0.4 mol/L $KMnO_4$ solution, standardized 10% v/v H_2SO_4 solution

Method:

Basic Experimental Parameters:

Titrant delivery rate (mL/min.)	5
No. of exothermic endpoints	1
Data smoothing factor	90
Stirring speed (802 stirrer)	6
Delay before start (secs.)	5

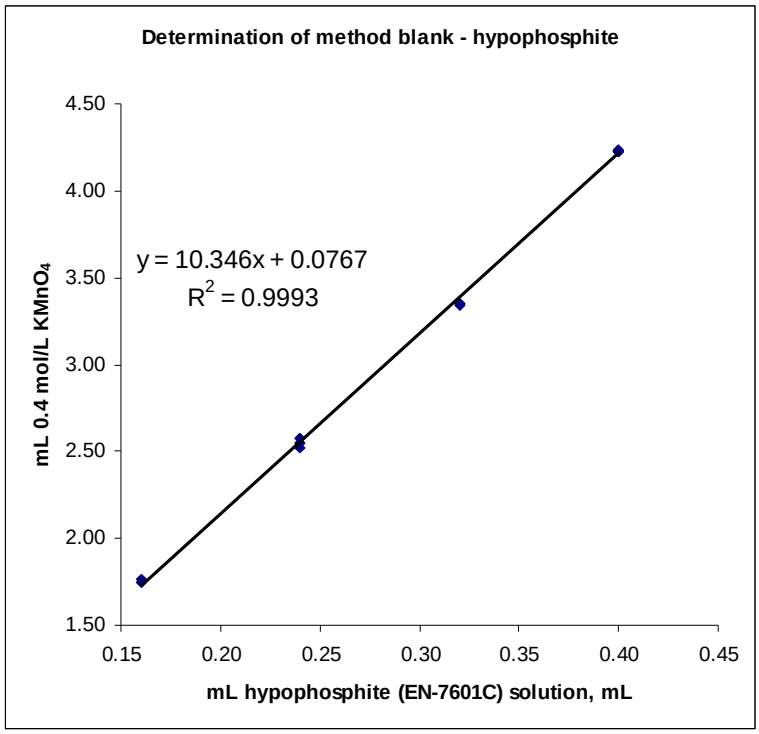
Prepare serial dilutions of commercial hypophosphite solutions, such that 10mL diluted aliquots yield between 0.4 and 1 mL of original solution, and give titres of 0.4 mol/L KMnO4 in the range 3 – 4mL. The following table can be used as a guide:

Solution	Aliquot of original solution, mL	Diluted in …mL volumetric flask with DI water	Aliquot taken for titration, mL	Equivalent volume of original aliquot, mL
Soln. 1 (~550g/L)	10	250	10	0.4
Soln. 2 (~400g/L)	5	100	10	0.5
Soln. 3 (~150g/L)	25	500	20	1
Soln. 4 (~250g/L)	10	250	20	0.8

Calculation:	Calculated as g/L sodium hypophosphite monohydrate, $H_2NaPO_2.H_2O$ (FW=105.993)
	$g/L = \frac{((titre - blank) \times M\,KMnO_4 \times FW \times 5)}{(sample\ vol.\ mL \times 4)}$

Results:	Commercial hypophosphite solutions for electroless plating	
	Sample	$H_2NaPO_2.H_2O$ g/L
	Solution 1	550.6±0.43 (n=5) %RSD = 0.08
	Solution 2	394.1±0.94 (n=5) %RSD = 0.24
	Solution 3	149.6±0.54 (n=6) %RSD = 0.36
	Solution 4	259.8±1.13 (n=6) %RSD = 0.44

Determination of method blank:	
	It is required that the method blank needs to be determined when setting up the method for the first time, or when substantial changes to the method such as Dosino delivery rate are made. This is done by titrating varying amounts of the same sample and plotting the response for both titrations. In the example given below, a dilute solution of the sample was prepared, and different aliquot volumes of this solution titrated. The calculated volume of sample in each aliquot is plotted on the x-axis, the corresponding titre of $KMnO_4$ on the y-axis.



Example of titration plot:

Legend:
Red curve=direct solution temperature plot
Black curve=second derivative curve