# Thermo. Titr. Application Note No. H-055

| **Title:** | **Analysis of sodium and phosphorus in sodium tripolyphosphate pre-cursor solutions** |
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| ***Scope*:** | Determination of Na and P and [Na]/[P] in precursor solutions and solids in the manufacture of sodium tripolyphosphate. |
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| ***Principle*:** | Ionisable protons in samples containing dihydrogen monophosphate and monohydrogen monophosphate anions are titrated with standard NaOH in a saline environment. |
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| ***Reagents:*** | 2M NaOH solution (standardized).<br>Saturated NaCl solution (approximately 350g/L) prepared from A.R. NaCl. |
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***Method:***

Basic Experimental Parameters:

| | |
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| Titrant delivery rate (mL/min.) | 2 |
| No. of exothermic endpoints | 75 |
| Data smoothing factor | 50 |
| Stirrer speed | 6 |

Procedure:

An aliquot of sample solution (between 5 and 10 mL) is pipetted into a titration vessel. 15 mL saturated NaCl solution is added. DI water is added to bring the total volume in the vessel to approximately 30 mL. The solution is titrated to the second exothermic endpoint with 2 M NaOH.

In the case of solid samples, these are weighed accurately into a clean, dry titration vessel, and the solids dissolved in 15 mL deionized water. 15 mL saturated NaCl solution is added to bring the total volume to approximately 30 mL.

The aliquot or sample size should be adjusted to give a total titre of approximately 7-8 mL.

If only the [Na]/[P] molar ratio is desired, the titrant need not be standardized.

***Results (example):***

| | [Na]/[P] | $NaH_2PO_4$ g/L | $Na_2HPO_4$ g/L |
|---|---|---|---|
| | 1.651±0.0001 (n=5) | 23.8±0.02 (n=5) | 52.4±0.03 (n=5) |

***Calculations:***

| Item | Symbol | Item | Symbol |
|---|---|---|---|
| M NaOH | M | Blank, mL | B |
| FW $NaH_2PO_4$ | $FW_1$ | Titre to endpoint 1, mL | $EP_1$ |
| FW $Na_2HPO_4$ | $FW_2$ | Titre to endpoint 2, mL | $EP_2$ |
| Sample vol. mL | $S_V$ | Sample mass, g | $S_M$ |

$$\frac{[Na]}{[P]} = 2 - \frac{(EP_1 - B)}{(EP_2 - (EP_1 - B))}$$

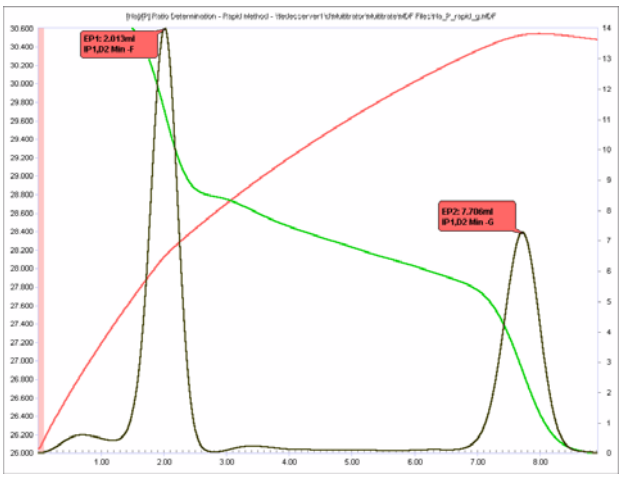
$$NaH_2PO_4\ g/L = \frac{((EP_1 - B) \times M \times FW_1)}{S_V}$$

$$NaH_2PO_4\ \%w/w = \frac{((EP_1 - B) \times M \times FW_1 \times 100)}{(S_M \times 1000)}$$

$$Na_2HPO_4\ g/L = \frac{(((EP_2 - EP_1) - (EP_1 - B)) \times M \times FW_2)}{S_v}$$

$$Na_2HPO_4\ \%w/w = \frac{(((EP_2 - EP_1) - (EP_1 - B)) \times M \times FW_2 \times 100)}{(S_M \times 1000)}$$

***Thermometric Titration Plot:***




Legend:

Red = solution temperature curve

Green = first derivative curve

Black = second derivative curve