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Thermo. Titr. Application Note No. H-081

Title:	Determination of Phosphoric and Nitric Acid in Nitrophos Liquors

Scope:	Determination of phosphoric and nitric acids in liquors from the Nitrophos fertilizer manufacturing process.

Principle:	Titration in a standardized solution of 2 mol/L NaOH in a sodium chloride solution to sharpen the third endpoint.

Reagents:	Titrant: 2 mol/L NaOH, standardized against potassium hydrogen phthalate Conditioning solution: 180g/L NaCl

Method:	Basic Experimental Parameters:
	Titrant delivery rate (mL/min.) 5
	Delay before titration commences (sec) 5
	No. of exothermic endpoints 3
	Data smoothing factor (DSF) 93
	Stirring speed (802 stirrer) 15
	Titration: Weigh accurately approximately 0.7mL (approximately 1g) of Nitrophos liquor into a clean, dry titration vessel. Add 30mL 180g/L NaCl solution, and allow to stand for approximately 30 minutes before titrating in order that any contained hexafluorosilicic acid is fully hydrolyzed.
	Blank determinations: Prepare titration solutions as above, weighing masses of Nitrophos liquor encompassing a range from approximately 1.5 to 0.6g. Titrate, and prepare two graphs. In the first graph, plot sample mass (x-axis) against titration volume to the first endpoint, EP1. Determine the y-intercept from regression analysis. This will be the blank (B1) for the nitric acid determination. In the second graph, plot sample mass (x-axis) against the difference between the third and second endpoints, EP3-EP2. Once again, determine the y-intercept from regression analysis. This will be the blank (B2) for the phosphoric acid determination

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Examples:	Nitrophos process liquor	
H_3PO_4% w/w	24.1±0.25 (n=11)	
HNO_3% w/w	7.8±0.17 (n=11)	

Calculations:

$$H_3PO_4 \% = \frac{((EP3 - EP2 - B2) \times NaOH\ mol/L \times 97.9769 \times 100)}{(sample\ mass,\ g \times 1000)}$$

$$HNO_3 \% = \frac{(((EP1 - B1) - (EP3 - EP2 - B2)) \times NaOH\ mol/L \times 62.99564 \times 100)}{(sample\ mass,\ g \times 1000)}$$

Thermometric Titration Plot:

Legend:
Red = solution temperature curve
Black =second derivative curve (for endpoints)

H3PO4 in Nitrophos

EP2: 4.117ml
IP1,D2 Min -H

EP3: 5.507ml
IP1,D2 Min -I

EP1: 2.052ml
IP1,D2 Min -G