Thermo. Titr. Application Note No. H-079

Title:	**Determination of Free Acid in Heat Exchanger Cleaning Acid**

***Scope*:**	Determination of free acid in sulfuric acid („acid shot“) solutions employed in the removal of silicate scale in heat exchangers. This method is suitable for acid shot solutions where the silicic acid content is so high that the solutions have gelled.

***Principle*:**	Direct thermometric titration of a weighed amount of „acid shot“ solution with standard NaOH solution.

Reagents:	*Titrant:* 1mol/L NaOH solution. Standardize against potassium hydrogen phthalate.

Method:	Basic Experimental Parameters:	
	Titrant delivery rate (mL/min.)	4
	No. of exothermic endpoints	1
	Data smoothing factor (DSF)	50
	Stirring speed (802 stirrer)	15
	Titration: For fresh sulfuric acid solutions, weigh accurately into a titration vessel approximately 2mL of sample. Add 35mL DI water and titrate to a single exothermic endpoint. For used solutions, weigh in amounts of 2.5 – 5mL, depending on the stage of the acid shot cycle.	

Examples: *Acid shot samples*

Series A and B were samples of acid taken during two different acid shot cycles. Samples A1 and B1 represented freshly made up sulfuric acid solutions.

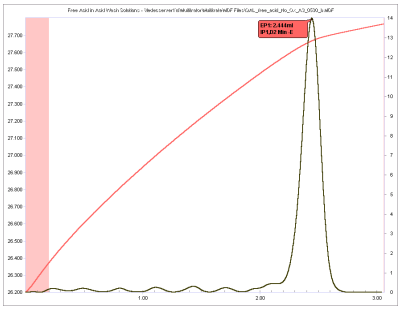
Sample ID	% free acid (as H_2SO_4)
A1	12.7. 12.7
A2	3.9. 4.0
A3	2.7. 2.7
A4	2.0, 2.0
B1	13.1, 13.1
B2	6.3, 6.3
B3	6.1, 6.1
B4	5.8, 5.8

Calculations:

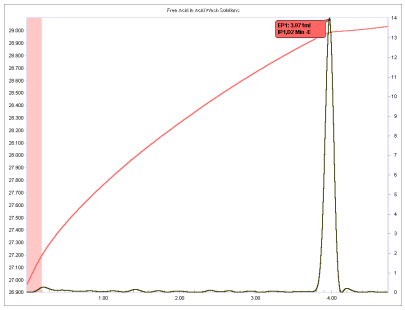
$$\%\,H_2SO_4 = \frac{((\text{Titre, mL} - \text{blank, mL}) \times \text{NaOH mol/L} \times 97.97 \times 100)}{(\text{sample mass, g} \times 1000)}$$

Thermometric Titration Plots:

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



Sample A4: Spent acid shot solution, very high gel content (high viscosity)



Sample B4: Spent acid shot solution, high gel content (high viscosity)