Thermo. Titr. Application Note No. H-012

Title:	**Determination of Ferrous Ion Content of Heat Exchanger Wash Solutions**

***Scope*:**	Determination of ferrous ion in heat exchanger and vessel acid wash solutions, to measure the effectiveness of acid inhibiters used in the solutions. Depending on the condition of the sample, the lower practical limit for the determination will vary from approximately 20-100mg/Kg Fe^{2+}. Samples with high silicic acid contents require relatively large amounts of dilution water to render them mobile, and this limits the aliquot size and hence the amount of Fe^{2+} which can be analyzed.

***Principle*:**	An aliquot of acid wash solution is further acidified and titrated to a single thermometric endpoint with standard potassium dichromate solution. $Cr_2O_7^{2-} + 14H^+ + 6e \leftrightarrow 2Cr^{3+} + 7H_2O$ $[Fe^{2+} \leftrightarrow Fe^{3+} + e] \times 6$ $Cr_2O_7^{2-} + 14H^+ + 6Fe^{2+} \leftrightarrow 6Fe^{3+} + 2Cr^{3+} + 7H_2O$ *ie.*, $0.1N\ K_2Cr_2O_7 = 0.1/6\ mole/L$

Reagents:	Standard 0.1N $K_2Cr_2O_7$ 10% w/v H_2SO_4

Method:	Basic Experimental Parameters: Data rate (per second) 10 Titrant delivery rate (mL/min.) 1 No. of exothermic endpoints 1 Data smoothing factor 35 Procedure: Weigh accurately approximately 20mL of acid wash solution into a titration vessel. Add 10mL 10% w/v H_2SO_4, and titrate to a single exothermic thermometric endpoint. If the sample is mobile, an aliquot of up to 50mL may be used.

Results:

Analysis of acid wash solutions from “acid shot” of alumina refinery heat exchanger

Sample I.D.	Aliquot, mL	Titre, mL	Fe^{2+} mg/Kg
1	21.1869	1.081	290
	20.6534	1.073	296
2	19.9588	0.986	281
	20.0909	0.979	277
3	19.4922	0.824	240
	20.3586	0.859	240
4	24.9266	0.852	194
	22.4261	0.838	212
5	21.5989	1.347	355
	21.4737	1.341	356
6	22.7956	1.095	273
	21.5213	1.037	274
7	22.8675	0.931	231
	22.8750	0.933	232
8	22.5952	0.443	110
	21.7592	0.417	107

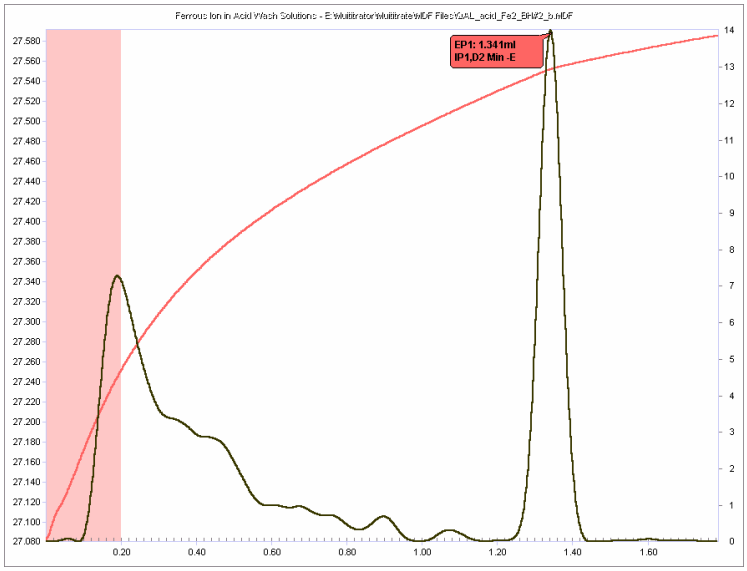
Calculation:

$$mg\ Fe^{2+}/Kg = \frac{((titre, mL - offset, mL) \times N\ K_2Cr_2O_7 \times FW\ Fe \times 1000)}{sample\ mass,\ g}$$

Example:

$$\frac{((1.347 - 0.010) \times 0.1028 \times 55.845 \times 1000)}{21.5989} = 355 mg\ Fe^{2+}/Kg\ sample$$

Thermometric Titration Plot:



Legend:

Red = solution temperature curve

Green = first derivative curve

Black = second derivative curve