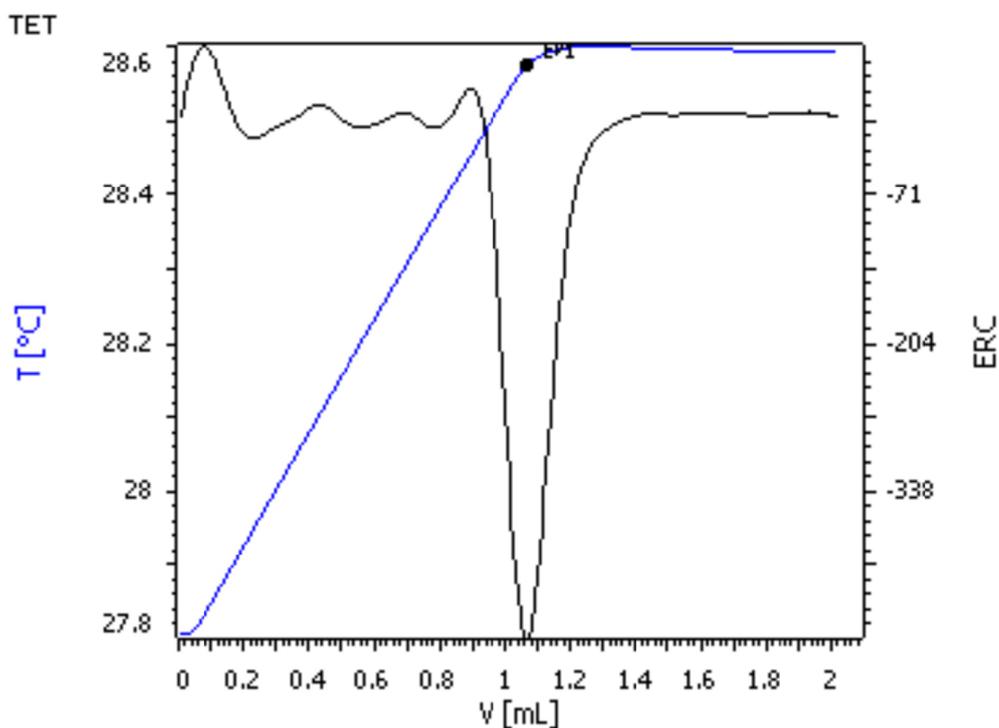


# Determination of hydrochloric acid in acidic solutions containing iron and aluminum



The presence of the hydrated ion  $[\text{Fe}(\text{H}_2\text{O})_6]^{3+}$  can interfere with the determination of «free acid» due to the low  $\text{pK}_a$  ( $\sim 2.2$ ) of this ion. Ions of metals such as Fe, Cu, and Al can be masked effectively with fluoride, and permit the determination of the acid content by thermometric alkalimetric titration with good accuracy and precision.

# Method description

## Samples

«Sample solutions» were prepared from reagent grade HCl,  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ , and  $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  to approximate those that a customer desired to have analyzed. Due to the highly concentrated nature of the customer's own solutions, it was necessary to prepare them in such a manner that they represented a 1:4 dilution.

Nominal concentration of the "sample solutions":

	HCl g/L	Fe <sup>3+</sup> g/L	Al <sup>3+</sup> g/L
Sample A	*	76.0**	56.9***
Sample B	*	114.9**	0.5***

\* Results reported above

\*\* Experimental details reported in **AN-H-119**

\*\*\* Experimental details reported in **AN-H-120**

## Sample preparation

1:4 diluted «sample solution»

## Configuration

Basic equipment list for automated titration

814 USB Sample Processor	2.814.0030
859 Titrotherm	2.859.0010
Sample rack 24 x 75 mL	6.2041.340
Thermoprobe, fluoride resistant	6.9011.040
Sample beaker 75 mL	6.1459.400
802 Rod Stirrer	2.802.0010
Stirring propeller 104 mm	6.1909.020
1 x 800 Dosino	2.800.0010
1 x Dosing unit 10 mL	6.3032.210

## Solutions

Titrant	2 mol/L NaOH, standardized against potassium hydrogen phthalate
	620 g potassium fluoride (near saturated) prepared in dist. water (Note: lower concentrations of KF can be employed, so long as a considerable excess of reagent can be added, over and above the stoichiometric requirements of the

	Fe and Al contents of the sample aliquot).
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## Analysis

10 mL aliquots of 1:4 diluted «sample solution» were pipetted into titration vessels, and 15 mL dist. water and 5 mL 620 g/L KF solution (~55 mmol/L KF) added to complex Fe and Al present. The solutions were titrated with 2 mol/L NaOH to a single exothermic endpoint.

## Parameters

Basic experimental parameters

Titrant dose rate (mL/min)	4
ERC EP1 (exothermic)	-50
Data smooting ("filter factor")	50
Stirring speed (802 Rod Stirrer)	14
Evaluation start (mL)	0.5
Damping until (mL)	0.5

## Calculations

$$\text{g/L HCl} = ((\text{EP1} - \text{blank}) \times \text{C01} \times \text{C02})/\text{C00}$$

EP1 = endpoint in mL

C00 = sample weight in mL

C01 = concentration of titrant in mol/L

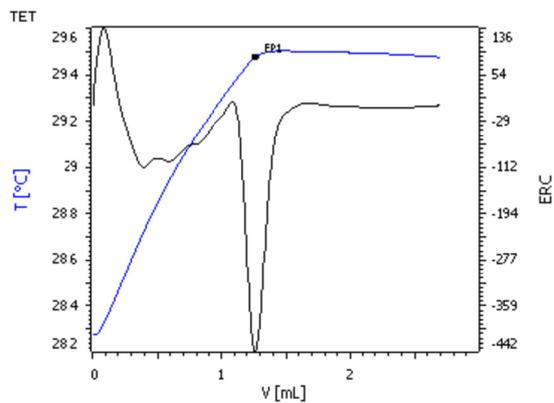
C02 = molecular weight of HCl (36.46 g/mol)

## Results

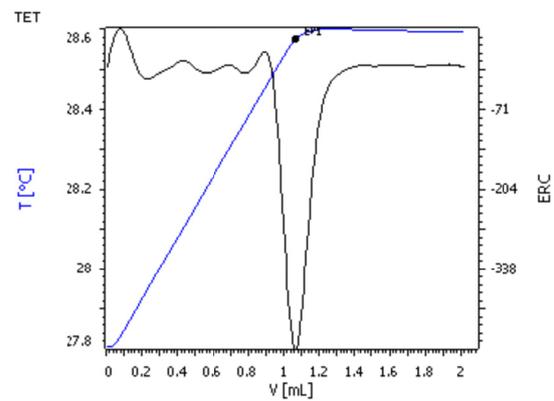
HCl in g/L	
Sample A	36.35 ± 0.00
Sample B	30.62 ± 0.11

# Method description

## Titration Plots



Titration of HCl in Sample A



Titration of HCl in Sample B