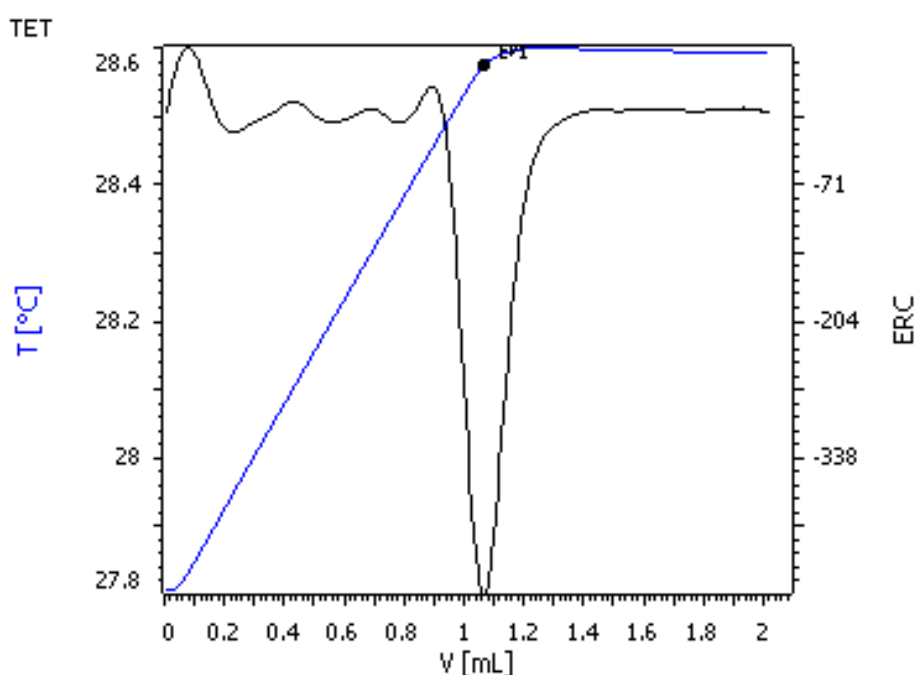


# Determination of hydrochloric acid in acidic solutions containing iron and aluminum



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

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
Auxiliary solution	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.

## Analysis

10 mL aliquots of 1:4 diluted «sample solution» is pipetted into titration vessels, and 15 mL dist. water and 5 mL 620 g/L KF solution are added to complex Fe and Al present. The solutions is titrated with  $c(\text{NaOH}) = 2 \text{ mol/L}$  to a single exothermic endpoint.

## Parameters

Stirring speed	14
Dosing rate	4 mL/min
Filter factor	50
Damping until	0.5 mL
Evaluation start	0.5 mL
End points	exothermic
EP criterion	-50

## Results

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