Thermo. Titr. Application Note No. H-089

Title:	**Automated analysis of hexafluorosilicic acid**

***Scope*:**	Automated determination of the H_2SiF_6 and HF contents of industrial grade hexafluorosilicic acid

***Principle*:**	Titrate a sample of industrial grade hexafluorosilicic acid first with standard NaOH solution to a sharp endpoint, then continue the titration until species have reacted and an excess of NaOH exists. Back-titrate the excess NaOH with standard HCl solution. The first endpoint of the NaOH titration is used to calculate the "Total" H_2SiF_6 content, which is defined as the "Actual" H_2SiF_6 content plus any residual HF present. The excess of NaOH is used to force the hydrolysis of SiF_6^{2-} to $SiO_2(OH)_2^{2-}$ to completion. The back-titration of this excess NaOH with HCl then allows an accurate estimate of the "Actual" H_2SiF_6 content to be made.

Reagents:	Titrant 1: 2mol/L NaOH Titrant 2: 2mol/L HCl A.R. Potassium hydrogen phthalate

Method:	*Basic Experimental Parameters:*	
	NaOH titration:	
	Titrant delivery rate (mL/min.)	5
	No. of exothermic endpoints	1
	Data smoothing factor	75
	Maximum dose (mL)	10
	Stirring speed (802 stirrer)	8
	Delay before start (secs.)	5
	HCl titration:	
	Titrant delivery rate (mL/min.)	5
	No. of exothermic endpoints	1
	Data smoothing factor	60

	Maximum dose (mL)	10
	Stirring speed (802 stirrer)	10
	Delay before start (secs.)	60

Sample Preparation

Approximately 1mL of industrial grade hexafluorosilicic acid is weighed into a clean, dry titration vessel. 35mL of D.I. water is added.

Titration

A “chained” or “linked” titration program is used to automatically first titrate the acid with NaOH then allow an excess of NaOH to be added before starting a back-titration with HCl to quantify the excess NaOH which has been added. Prior to the commencement of the HCl back-titration, a 60 second delay is programmed to ensure that the hydrolysis reaction has come to completion.

Example:	*Fluka commercial hexafluorosilicic acid, nominal 25%w/w (n=12 replicate titrations)*
	“Total” H_2SiF_6 = 24.84±0.07% w/w “Actual” H_2SiF_6 = 19.22±0.09% w/w Residual HF = 0.78±0.02% w/w [F]/[Si] = 6.29±0.01

Calculations:

$$\text{"Total"}\ \%\,H_2SiF_6 = \frac{((V_1 - B_1) \times 144.0918 \times m_1 \times 100)}{(S \times 2 \times 1000)}$$

$$\text{"Actual"}\ \%\,H_2SiF_6 = \frac{(V_T - V_1 - (\frac{(V_2 - B_2) \times m_2}{m_1})) \times 144.0918 \times m_1 \times 100}{(S \times 2 \times 1000)}$$

$$\%\,HF = \frac{(\%\text{"Total"}\ H_2SiF_6 - \%\text{"Actual"}\ H_2SiF_6) \times 20.00634}{144.0918}$$

$$[F]/[Si] = \frac{((\frac{\%H_2SiF_6 \times 6}{144.092}) + (\frac{\%HF}{20.00634})) \times 144.092}{\%H_2SiF_6}$$

Legend:			
Molecular mass H_2SiF_6	144.0918	Endpoint vol., NaOH titration, mL	V_1
Molecular mass HF	20.00634	Blank vol. NaOH titration, mL	B_1
Molarity NaOH	m_1	Total vol. NaOH titrant delivered, mL	V_T
Molarity HCl	m_2	Endpoint vol. HCl titration, mL	V_2
Sample mass, g	S	Blank vol. HCl titration, mL	B_2

Titration desktop: