Ω Metrohm

Thermo. Titr. Application Note No. H-050

Title:	Determination of Sodium and Potassium Silicates

Scope:	Determination of sodium, potassium and silica values in sodium and potassium silicates.

Principle:	Acidimetric thermometric titration of sodium and potassium silicates in two contiguous steps. In the first titration, the caustic component (Na$_2$O or K$_2$O) is determined:

$$SiO_3^{2-} + 2H^+ \leftrightarrow H_2SiO_3 \qquad (1)$$

The titration is stopped automatically a little past the endpoint. Solid sodium fluoride is then introduced, and hydroxyl ions released by the reaction and equivalent to the SiO$_2$ content are then titrated in the second titration:

$$H_2SiO_3 + 6F^- + H_2O \leftrightarrow Na_2SiF_6 + 4OH^- \quad (2)$$
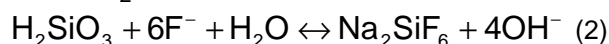
Results from both titrations are sent to the same dedicated spreadsheet linked to the titration methods, where all relevant analytical parameters are computed.

Reagents:	Titrant. 2 mol/L HCl, standardized
	Sodium fluoride, Merck "GR for analysis"

Method:	Basic Experimental Parameters:

Titrant delivery rate (mL/min.)	2
No. of exothermic endpoints	1
Data smoothing factor	55
Stirring speed (802 stirrer)	6 (1st titration)
	12 (2nd titration)
Delay before start (secs.)	5 (1st titration)
	30 (2nd titration)

Weigh accurately 2.2g sodium or potassium silicate into a clean dry titration vessel. Add 30mL DI water, and conduct the first titration for Na$_2$O or K$_2$O. After this titration stops automatically, add 5g NaF through a port in the titration vessel cap and start the second titration sequence. All titration data is sent to the linked spreadsheet for computation.

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| Calculations:	
	1. Na_2O (FW = 61.979) or K_2O (FW=94.196). $$\% Na_2O \text{ or } K_2O = \frac{((T_1 - B_1) \times FW \times M\,HCl \times 100)}{(1000 \times \text{sample mass, g} \times 2)}$$ 2. SiO_2 (FW=60.0843) $$\% SiO_2 = \frac{((T_2 - B_2 + PD + T_3) \times FW \times M\,HCl \times 100)}{(1000 \times \text{sample mass, g} \times 4)}$$ *Notes:* 1. *T_1=titre, B_1=blank in Na_2O or K_2O titration* 2. *T_2=titre, B_2=blank in SiO_2 titration* 3. *A pre-dose („PD") comprising about 80% of the estimated total titre is programmed into the method.* 4. *„T_3" refers to that excess volume in the Na_2O or K_2O titration between the endpoint and where the titration automatically stops.*
Legend to titration plots: *Red = solution temperature curve* *Black =second derivative curve*	 Na₂O, K₂O titration | SiO₂ titration
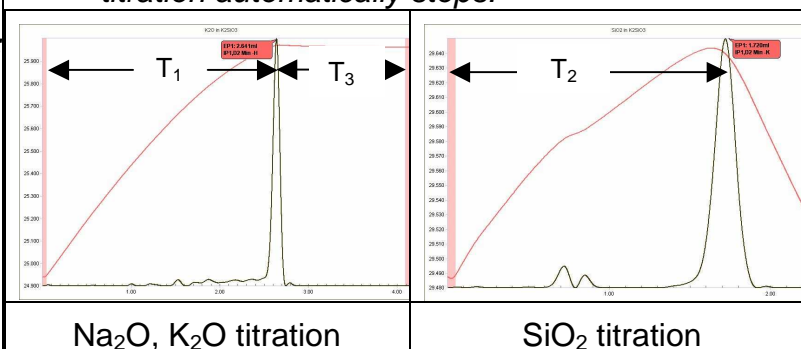
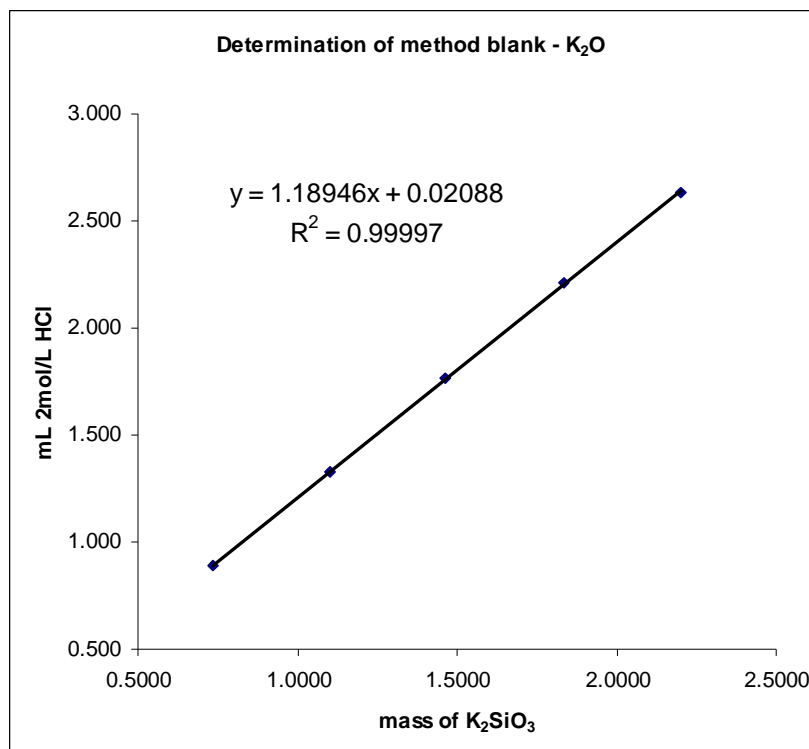| Results:	Production samples of sodium and potassium silicates. All determinations performed 10 times each. (Cited customer values in *shaded italics*)		
		Na₂SiO₃ #1	Na₂SiO₃ #2	K₂SiO₃
	Na₂O or K₂O%	φ=8.98±0.01 %RSD=0.11 *8.89*	φ=8.96±0.01 %RSD =0.12 *8.94*	φ=10.93±0.01 %RSD =0.05 *10.99*
	SiO₂%	φ=28.49±0.01 %RSD =0.04 *28.63*	φ=28.99±0.01 %RSD =0.02 *29.22*	φ=24.18±0.03 %RSD =0.13 *24.35*
	% solids	φ=37.47±0.02 %RSD =0.05 *37.63*	φ=37.95±0.02 %RSD =0.04 *38.15*	φ=35.11±0.03 %RSD =0.09 *35.34*
	SiO₂/Na₂O or K₂O	φ=3.17±0.003 %RSD =0.09 *3.19*	φ=3.23±0.003 %RSD =0.10 *3.27*	φ=2.21±0.003 %RSD =0.14 *2.22*

Determination of titration blanks:

It is required that titration blanks for both titration sequences be determined when setting up the method for the first time, or when substantial changes to the method such as Dosino delivery rate are made. This is done by titrating varying amounts of the same sample and plotting the response for both titrations. In the example given below, a dilute solution of the sample was prepared, and different aliquot volumes of this solution titrated. The calculated mass of sample in each aliquot is given.
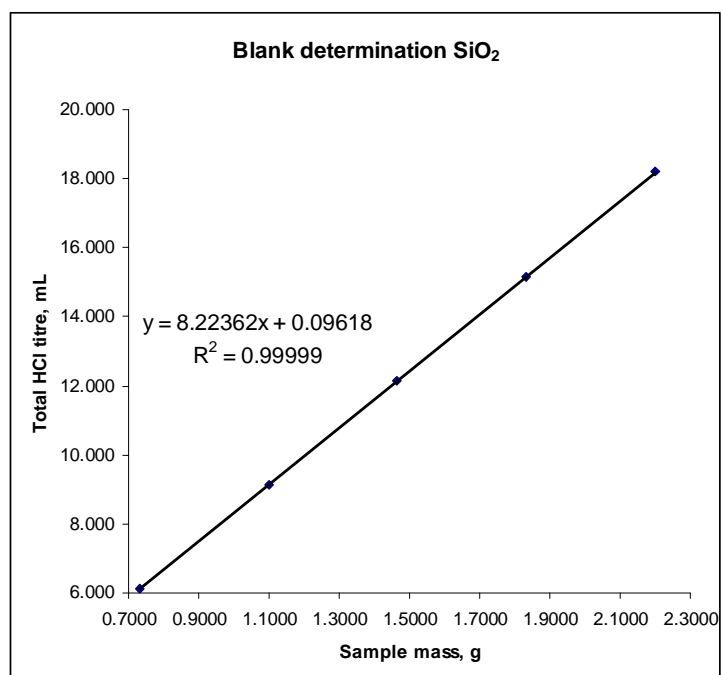
1. Blank estimation for Na_2O or K_2O titration.

Aliquot, mL	Calc. sample mass, g	T_1 mL HCl
30	2.1997	2.633
25	1.8331	2.207
20	1.4644	1.762
15	1.0998	1.328
10	0.7332	0.893

Determination of method blank - K_2O

$y = 1.18946x + 0.02088$
$R^2 = 0.99997$

Blank B_1 = y-intercept = 0.0209mL

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	2. Blank estimation for SiO$_2$ titration.				
Aliquot mL	Sample mass g	T$_3$ mL	PD mL	T$_2$ mL	Total titre (T$_3$+PD+T$_2$) mL
30	2.1997	1.345	15.00	1.849	18.193
25	1.8331	1.603	11.00	2.554	15.157
20	1.4644	1.577	8.00	2.573	12.150
15	1.0998	1.579	5.00	2.549	9.128
10	0.7332	1.613	2.50	2.020	6.133

Sample mass is plotted against total titre

Blank determination SiO$_2$

$y = 8.22362x + 0.09618$
$R^2 = 0.99999$

Blank B$_2$ = y-intercept = 0.0962mL

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