

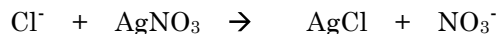
AQUACOUNTER Application Sheet	COM series	DATA No. F2	1st edition
Electronics	Quantification of trace chlorine ion in SiO <sub>2</sub> (silicon dioxide)		

## 1. Measurement outline

SiO<sub>2</sub> is used widely in various fields as the raw material for IC wafer abrasive and silicone resin. Trace chlorine ion contained in SiO<sub>2</sub> may often cause deterioration in product performance. Therefore, analysis on chlorine ion contained in material is an important item on which the product performance depends.

The method for quantifying chlorine ion in SiO<sub>2</sub> applies potentiometric titration with silver nitrate titrant by dispersing the sample in solvent (acetone, alcohol, etc.) which is acidified with acetic acid or nitric acid.

This section introduces an example in which approximately 20g of SiO<sub>2</sub> powder was weighed precisely to be dispersed in 50mL and acidified with nitric acid for potentiometric titration with silver nitrate titrant.



## 2. Reagents and Electrodes

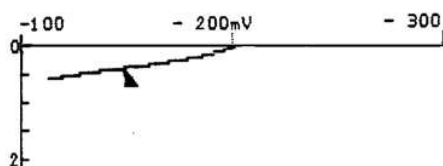
(1) Reagents	Titrant	0.002mol/L AgNO <sub>3</sub> titrant
	Titration solvent	50mL acetone for one measurement
	Loading buffer	0.1mL 1+1 nitric acid
(2) Electrodes	Indicator electrode	Silver indicator electrode AG-312 to IE jack (P/N D231259-A)
	Reference electrode	Silver reference electrode MS-231 to RE jack (P/N D231243-A)

### 3. Measurement conditions example (for COM-1600S)

Master File 1							
Condition 1 (for BLANK)				Condition 2 (for measurement)			
Method	AUTO			Method	AUTO		
Amp No.	2	Mode No.	20	Amp No.	2	Mode No.	21
Buret No.	1	Pre Int	0 sec	BURET No.	1	Pre Int	0 sec
Meas Unit	mV	Del K	0	Meas Unit	mV	Del K	2
S-Timer	0 sec	Del Sens	0 mV	S-Timer	0 sec	Del Sens	0 mV
CP mL	0 mL	Int Time	5 sec	CP mL	0 mL	Int Time	5 sec
Direction	N/A	Int Sens	0 mV	Direction	N/A	Int Sens	3 mV
DP mL	0 mL	Brt Speed	2	DP mL	0.4 mL	Brt Speed	2
End Sens	70	Pulse	40	End Sens	100	Pulse	40
Over mL	0 mL			Over mL	0 mL		
Max volume	5 mL			Max Volume	20 mL		
Unit	mL			Unit	ppm		
Blank	0 mL			Blank	BLANK result value		
Factor	Titer of the titrant			Factor	Titer of the titrant		
Molarity	0.002			Molarity	0.002 mol/L		
K	35.453			K	35.453		
L	0			L	0		
Formula	D			Formula	$(D-B) \times K \times F \times M \times 1000 / S$		

### 4. Measurement example

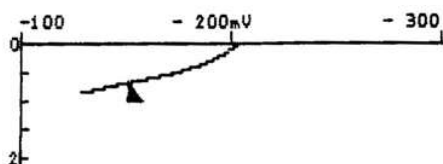
#### (1) Blank measurement example



#### Blank measurement results on solvent

Sample No.	Solvent volume (mL)	Titration value (mL)
1	50	0.35
2	50	0.35
Avg.		0.35mL

#### (2) Sample measurement example



#### Measurement results on trace chlorine ion in SiO<sub>2</sub>

Sample No.	Sample volume (g)	Titration value (mL)	Concentration (ppm)
1	20.209	0.71667	1.2941
2	20.228	0.70000	1.2341
3	19.735	0.70000	1.2649
Avg.			1.204 ppm
Std. Dev.			0.03 ppm
C.V.			1.5 %

## 5. Outline

### (1) About blank test on titration solvent

When quantifying trace chlorine in resin, the trace chlorine contained in the solvent will cause measurement error. Thus it is necessary that the blank test is always conducted on the solvent to make correction.

### (2) About indicator electrode

Since this measurement is a non-aqueous titration which uses organic solvent, the indicator electrode needs to be selected with consideration of its chemical resistance. AG-312 in which silver electrode is fixed to Teflon is recommended as the indicator electrode for this measurement (AG-311 uses epoxy resin and may become dissolved).

### (3) About acidic loading buffer

The acidic loading buffer used in this measurement is acetic acid or nitric acid. Caution is required when nitric acid is used in measurement of trace chlorine ion since the silver electrode may be dissolved and cause error if the nitric acid concentration becomes too high.

### (4) About reference electrode

Since this measurement is a non-aqueous titration, the liquid junction block for the reference electrode may be dehydrated and increase electrical resistance. Thus it is necessary that the crystal of inner solution be removed regularly by soaking it in purified water or by loosening the sleeve for the liquid junction block.

### (5) About standard addition method

The inflection point for titration curve may become unclear when chlorine ion is quantified in the trace amount of several ppm. Favorable results may be obtained when the standard addition method is used as the titration method in such cases. In this method, solution containing chlorine ion (about several tens of ppm) is prepared in advance to titrate a certain volume of this reagent and calculate the blank test value. Then a certain volume of the sample and this reagent is titrated. The measurement result is calculated by subtracting the blank test value from the measurement value.

## Key words

SiO<sub>2</sub>, silicon dioxide, abrasive, chlorine ion, precipitation titration, standard addition method

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