

Determination of trace metallic impurities in high-purity quartz by ion chromatography

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Abstract

A method has been developed for the determination of relevant trace impurities (alkali, alkaline and transition metals) in high purity quartz by ion-chromatography. In situ reagent (HF) purification and simultaneous sample dissolution was achieved in a multichannel vapour phase digestion assembly. Twenty-one samples can be digested at a time in this vapour phase system. Significant decrease in the process blank levels for all the analytes was observed. Drastic reduction (250 times) of NH_4^+ blank was achieved in the described vapour phase digestion, which enables the determination of trace concentration of sodium in high purity quartz. After volatilisation of the matrix and unreacted HF, the clear water leached solutions were injected into an ion-chromatograph equipped with conductivity detector for the determination of alkali and alkaline earth metals. In the case of transition metals, the trace residues were leached with 10 mM HCl and after separation on a mixed bed analytical column (IonPac CS5) were detected by spectrophotometry after post column derivatisation using 4-(2-pyridylazo)resorcinol (PAR). The accuracy of the result was checked by their comparison with those obtained by independent methods like inductively coupled plasma (ICP) MS and ICP atomic emission spectrometry. The achievable detection limits are between 0.4 ng/g (Li) and 22 ng/g (Mn). The application of the method to the determination of the above trace metals in two high-purity-grade quartz samples is demonstrated.

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1. Introduction

High-purity quartz is widely used to produce optical wave-guide, high performance lens, prisms, photovoltaic materials and for frequency control applications. Any trace impurities (Fe, Cu, Ni, etc.) in this material strongly influences the quality of the final products [1]. Impurities like alkali/alkaline earth metals degrade the ultraviolet performance, shifting the UV cut off (153 nm) to longer wavelength [1]. In addition to the stringent purity requirement, Fe^{3+} causes absorption and results in network defects under reducing conditions [1]. It is therefore desirable to determine accurately the contents of trace impurities in high purity quartz for quality control.

High-performance ion chromatography (IC) has been well established as an analytical technique for determining a wide range of trace metals in a variety of complex

matrices [2–4]. To our knowledge, no report concerning ion-chromatographic analysis of high purity quartz has been made until now, despite its capability in providing sensitive, fast and multielement capability in a single analysis.

The predominant techniques that have been used to determine trace metal impurities in high-purity quartz include graphite furnace atomic absorption spectrometry [5], inductively coupled plasma (ICP) atomic emission spectrometry (AES)/flame atomic absorption spectrometry (FAAS) [6] and ICP MS [7]. Prior to analysis by these techniques [5–7], sulfuric acid is heated with the final digests as post decomposition procedure to evaporate silicon related matrix components such as hydrofluorosilicic acid (H_2SiF_6) and unreacted HF. However, the use of sulfuric acid (b.p. 340 °C) in sample digestion results in solution matrices that present compatibility problems for separation of Groups I and II cations by IC. Samples of very low pH protonate some of the cation exchange sites (carboxylate functional groups) on the stationary phase, lowering charge density and capacity. Hydronium ion in the low pH samples, which acts as eluent, de-focusses the analyte band, resulting in low peak

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efficiency. Poor peak resolution and irregular base line can result in cation analysis when an acidic solution is injected onto a weak acid type cation exchange (IonPac CS12) column [8]. Even a recently introduced high-capacity column, the IonPac CS16 [9], requires a dilution factor of 36 to maintain acidity below 100 mM H^+ , when only 100 μ l of H_2SO_4 is used in digestion. Some trace elements could then be below quantification limit. Similarly, acidic solutions are not compatible with the eluent system of the analytical column (IonPac CS5) commonly used for separation of transition metals [10]. Alternatively, acidity of the sample solution can be reduced in a high capacity anion exchange resin in the OH^- form [11], but such additional pretreatment step might contaminate the sample solution for such ubiquitous trace elements (Na, Ca, Fe, etc.). Additionally, higher impurity levels in sulfuric acid results in an increase in the process blank.

Thus, main practical problems in sample treatment [12] prior to IC are related with sample dissolution and matrix elimination, which must be carried out as per clean chemistry protocol [13] in order to avoid contamination and to obtain low process blanks. Hydrofluoric acid (GR/AR grade) is often contaminated with traces of foreign elements, and dissolution procedure requires quantities of acid in excess of 10 times the sample (quartz) mass [7,13], which results in unacceptable reagent blank values. Exploiting vapour phase digestion (VPD) can circumvent the reagent blank problem. The vapour phase reaction principle has been utilised successfully for quartz [5,6], biological matrices [14], and recently in our laboratory for arsenic metal [15], arsenious oxide [16] and boric acid [17] samples.

This paper evaluates the feasibility of a dissolution method without sulfuric acid, so that the resulting solution (after matrix volatilisation) is amenable to ion-chromatographic analysis for desired trace elements. Matrix dissolution effected in a multichannel vapour phase digestion (MCVPD) assembly, significantly reduces the reagent blanks, where GR/AR grade acid is used instead of the costly high purity grade HF. In the described VPD system, 21 samples can be digested in a single batch, thus greatly improving the sample throughput. The results obtained by ion chromatography have been compared with those obtained by other established techniques.

2. Experimental

2.1. Vapour phase digestion apparatus

The high purity quartz samples were digested using a vapour phase digestion assembly as shown in Fig. 1. The polypropylene vessel is partitioned into reagent compartment (lower portion) and sample compartment (upper portion) by placing a perforated sample rack, made out of polypropylene (PP) sheet. Twenty-one grooves (31 mm \times 8 mm) were machined on the sample rack in which sample containers were kept. Fifty-seven holes (10 mm) were

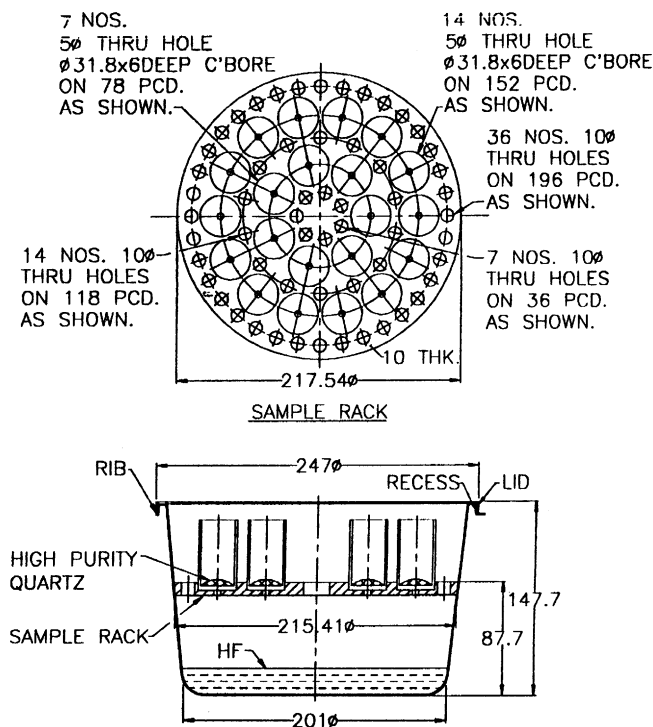


Fig. 1. MCVPD assembly.

drilled to channelise the generated acid vapours into the sample compartment. The polypropylene vessel and lid have air locking arrangement through a rib in the lid and corresponding recess in the vessel. Perfluoroalkoxyethylene (PFA) vials (Cole Parmer, USA) were used as sample containers. To clean the VPD assembly, a mixture of HF– HNO_3 (1:1) was placed in the reagent compartment, capped and kept in a water bath ($\sim 90^\circ C$) for 2 h. After cooling, it was thoroughly rinsed with deionised (DI) water and always kept in closed condition when not in use. Normal precautions for trace analysis were observed throughout. PFA containers were soaked for 24 h in 2% HNO_3 and rinsed several times before use.

2.2. Reagents and standards

High-purity DI water with a specific resistance of 18 $M\Omega$ cm or greater was obtained through a Millipore water purification system. Methanesulfonic acid (Loba Chemie, Bombay, India), hydrofluoric acid (AR grade, SD fine, India) and Lithium hydroxide monohydrate (AR grade, Thomas Baker (Chemicals) Bombay, India) were of analytical grade. 4-(2-Pyridylazo)resorcinol (PAR) sodium salt metal indicator (GR grade, Loba Chemie) and pyridine-2,6-dicarboxylic acid (PDCA, HPLC grade, EMerck) were used. Highly pure sulfuric and hydrofluoric acid (both suprapur grade) were from Merck, Germany. Laboratory-prepared sub-boiled HCl was used. Stock standards (1000 mg/l) were prepared from reagent grade salts. Working standards were prepared by further diluting the 1000 mg/l standards to the range expected for the trace metals of interest.

2.3. Instrumentation and chromatographic conditions

Chromatographic analyses were performed on a metal free Dionex DX-300 ion chromatograph (Dionex, Sunnyvale, CA, USA). The system consisted of an advanced gradient pump (AGP-1), a liquid chromatographic module (LCM-3) and a conductivity detector (CDM-3). For cations (alkali/alkaline earth metal), an IonPac CS12 (250 mm × 4 mm) analytical column and CG12 guard column (50 mm × 4 mm) was utilised which provides fast separation in isocratic elution. A 25 µl sample injection loop was used. Methane sulfonic acid (25 mM) was used as eluent at a flow rate of 1 ml/min. Cation self-regenerating suppressor (CSRS-ULTRA, 4 mm) from Dionex was used for suppressing the conductivity due to the eluent.

An IonPac CS5 (250 mm × 4 mm) analytical column and the corresponding guard column, IonPac CG5 (50 mm × 4 mm), were used for the separation of transition metals. A pressurised reagent reservoir for post column reagent (PAR) addition and a VDM-2 variable wavelength absorbance detector were used. The analysis of the transition metals was carried out with the following optimised mobile phase—a mixed eluent of PDCA (6×10^{-3} M) and LiOH (8.6×10^{-3} M), pH 4.8, was pumped isocratically at a flow rate of 1 ml/min. The post column reagent (4×10^{-4} M) was prepared in 3 M ammonia and 1 M acetic acid mixture. The reagent was delivered at a flow rate of 0.4 ml/min and mixed (knitted reaction coil, 375 µl) with the column effluent. The absorbance was monitored at 520 nm. A 100 µl sample injection loop was used. For sample loading, a Rheodyne (six port, Model 9126) injection valve was fitted with sample loops made from polyether ether ketone (PEEK). The instrument control, data acquisition and processing was performed with Indtech chromatographic software (Indtech, Mumbai, India) via an ACI-2 advanced computer interface.

ICP QMS (VG Elemental, UK) and ICP AES (JY-2000, France) were utilised as techniques to cross validate the results.

2.4. Vapour phase digestion of quartz powder

Quartz samples (0.5 g, size –50 to +100 mesh) were weighed into the PFA vial in a class 1000 clean room. The PFA vials were placed in the grooves (31 mm diameter) of the sample rack. Hydrofluoric acid (130 ml, AR grade) was carefully poured into the reagent reservoir through a polypropylene funnel. The vessel was capped and the assembly was placed on a single hole (diameter 180 mm) water bath (90 °C) for digestion. An exposure time of 7 h was required for complete digestion of 15 quartz samples (15×0.5 g, six blanks). When the dissolution was complete, the MCVPD assembly was allowed to cool to room temperature. PFA vials were removed from the sample rack with a plastic forcep, capped and dried externally. PFA vials containing ~1.5 ml of residual liquid were evaporated to complete dryness on a IR hot plate (PTFE coated, surface

temperature = 100 °C) inside a laminar flow clean bench. After complete dryness, the trace residues were leached with DI water (2 ml) for analysis of alkali/alkaline earth metal cations. Similarly in another set of digested samples, the residue after evaporation was extracted with 2 ml of 10 mM HCL prior to separation on IonPac CS5 column. A four-fold dilution factor is thus used. In all cases, injection of the sample was done in triplicate.

3. Result and discussion

3.1. Assessment of MCVPD system performance

On heating the MCVPD assembly, HF vapours or distilled HF reacts with SiO₂ forming volatile silicon tetrafluoride. The SiF₄ vapours comes in contact with the solution in the lower reagent compartment, dissolves in the HF to form H₂SiF₆ and remains in the solution. Thus large part (80%) of quartz matrix from the sample is eliminated during the sample digestion. The residual HF after VPD along with the remaining silicon related matrix was volatilised off during heating on IR hot plate to dryness. The trace impurities in the liquid reagent (HF) are not added to the sample and this allows for in situ reagent purification. As expected, the application of vapour phase digestion instead of direct reaction is to decrease the reagent blank. To confirm this, several blank measurements were carried out both in presence and absence of sample inside the VPD chamber and the mean blank values were determined. Fig. 2a shows the chromatogram of the process blank (alkali/alkaline earth) when AR grade HF is used for direct dissolution of 0.5 g quartz. Fig. 2b is the chromatogram of MCVPD process blank obtained using the same AR grade HF. A comparison of chromatograms demonstrates the in situ purification efficiency of the described vapour phase digestion system. Another interesting observation is the presence of large ammonium peak in chromatogram (Fig. 2a) obtained by direct dissolution. Initially it was suspected to be due to contamination of ammonia from the laboratory air. Such large contamination of NH₄⁺ in clean bench conditions seemed unlikely. On further experiments it was observed that NH₄⁺ peak height was linearly proportional to the volume of AR grade HF volatilised. Thus, the contamination of NH₄⁺ is believed to be due to the presence of ammonium hydrogen fluoride (NH₄)HF₂ in the HF whose decomposition temperature is 240 °C and hence is not lost, when the sample digests are evaporated to dryness at 100 °C. Some batches of AR grade HF contain very high amount of (NH₄)HF₂ and during the volatilisation process it gets concentrated. When trace levels of analyte ions are eluted adjacent to a high concentration ion (NH₄⁺), the peak integration of low concentration ions becomes difficult.

Though NH₄⁺ is not a required analyte in quartz, a large concentration of it in HF might create difficulty to quantify trace level of sodium/potassium, since these ions have

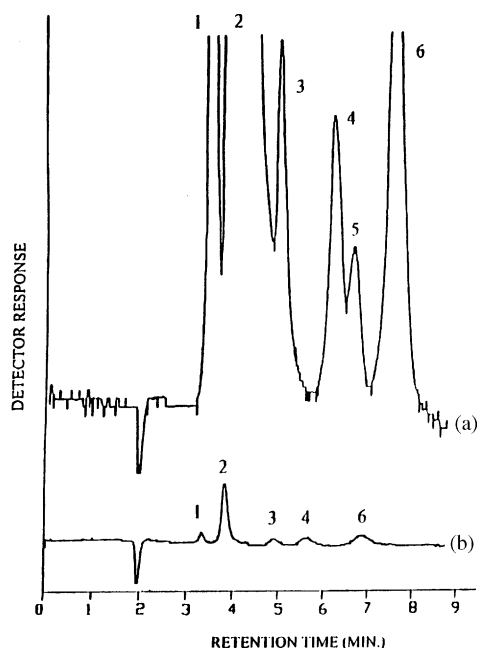


Fig. 2. (a) Chromatogram of alkali/alkaline earth metal cations obtained as reagent blanks, when AR grade HF (5 ml) was used in direct dissolution of 0.5 g of quartz. Peaks: (1) Na^+ (170 ng/ml); (2) NH_4^+ (4520 ng/ml); (3) K^+ (90 ng/ml); (4) Mg^{2+} (75 ng/ml); (5) unidentified; (6) Ca^{2+} (240 ng/ml). (b) Chromatogram showing reagent blanks when same AR grade acid is used in the vapour phase digestion. Peaks: (1) Na^+ (3.5 ng/ml); (2) NH_4^+ (18 ng/ml); (3) K^+ (4 ng/ml); (4) Mg^{2+} (2.5 ng/ml); (6) Ca^{2+} (5 ng/ml). Chromatographic conditions are described in the text.

similar selectivities for the carboxylic acid functional groups of the CS12 column [18]. As can be seen in Fig. 2b, the NH_4^+ contamination is significantly reduced in the reagent blank in vapour digestion when the same acid is used.

Likewise, chromatograms of reagent blanks for transition metals between direct dissolution and VPD are presented in Fig. 3. Overall reduction in reagent blanks for all the trace metals between direct dissolution and VPD are quantified and compared in Table 1. As can be seen from Table 1,

Table 1
Comparison of reagent/process blank (ng) between direct dissolution and VPD for 0.5 g of quartz ($n = 2$)

Analytes	Direct dissolution ^a	VPD
Li^+	n.d.	n.d.
Na^+	350	7
NH_4^+	9040	36
K^+	180	8
Mg^{2+}	155	5
Ca^{2+}	480	10
Fe^{3+}	360	10
Cu^{2+}	12	n.d.
Ni^{2+}	22	n.d.
Zn^{2+}	35	n.d.

n.d., not detected; VPD, vapour phase digestion.

^a HF (AR grade, 48%, v/v)—5 ml was taken in PFA vial and evaporated to dryness in clean bench. Trace metallic impurities were determined by IC as described in text.

Table 2
Recovery of analytes from quartz matrix and detection limits

Analytes	Added (ng)	Found (ng)	Recovery (%)	LODs (ng/g)
Li^+	25	25 (3)	100	0.4
Na^+	100	103 (4)	103	6.0
K^+	100	98 (6)	98	3.0
Mg^{2+}	100	96 (7)	96	3.1
Ca^{2+}	100	104 (5)	104	4.2
Fe^{3+}	50	52 (4)	104	10.3
Cu^{2+}	50	49 (5)	98	8.4
Ni^{2+}	100	95 (6)	95	10.8
Zn^{2+}	100	103 (4)	103	14.7
Co^{2+}	100	96 (5)	96	12.0
Mn^{2+}	200	198 (6)	99	22.0

LOD, limit of detection. Figures shown in the parentheses are relative standard deviations (%) for triplicate measurements.

reagent blanks of Na, K, Mg, Ca, Fe and Zn were significantly reduced. For instance, the reagent blank of critical elements like Na, Ca and Fe were reduced by 36–50 times. The total process blank values (normalized to 1 g sample) of these major elements (except NH_4^+) adds up to approximately to 80 ng. These values suggest that high-purity quartz in the purity range of 5–6N can be analysed using this procedure.

3.2. Recovery study and detection limits

In the VPD and subsequent volatilisation step, sulfuric acid was not used. As the recommended digestion procedure was modified, it was imperative to study the recovery of the trace analytes. High-purity quartz (0.5 g) was spiked with 25–200 ng of trace analytes, digested through VPD and analysed by IC. The recovery of spikes ranged from 95% for Ni^{2+} to 104% for Ca^{2+} (Table 2). The limit of detection (LODs) was defined by the protocol as the concentration of the analyte that gives a peak height response of three times the standard deviation of the VPD process blank ($n = 5$). The observed method detection limits are tabulated in Table 2. The method detection limits obtained are adequate for many end applications of quartz. However, in optical fibre extremely low levels of sodium, calcium and potential colouring elements (transition elements) are required to be determined which demand still lower detection limits. Further improvement in detection limits could be achieved by several means. These include large volume direct injection [19,20], a larger sample size (up to 1 g) and further minimisation of blanks (Fe, Ca, Na). The sources of these elements (Fe, Ca, Na) are most probably in the environment during the handling procedure after VPD in a normal laboratory.

3.3. Sample analysis, accuracy and precision

As no standard reference material for quartz was available in our laboratory, the accuracy of the proposed method was assessed by comparing data obtained by ICP AES/MS after volatilisation of the silica matrix using H_2SO_4 -HF

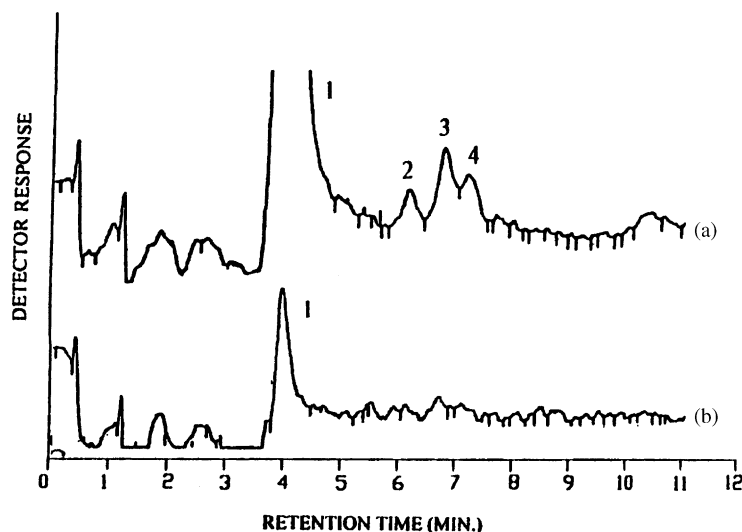


Fig. 3. (a) Chromatogram of transition metals obtained as reagent blank, when AR grade HF (5 ml) was used in the direct dissolution for 0.5 g of quartz sample. Peaks: (1) Fe^{3+} (180 ng/ml); (2) Cu^{2+} (6 ng/ml); (3) Ni^{2+} (11 ng/ml); (4) Zn^{2+} (17.5 ng/ml). (b) Chromatogram showing reagent blank when the same AR grade acid is used in the vapour phase digestion. Peak: (1) Fe^{3+} (5 ng/ml). Chromatographic conditions are described in the text.

acid (Suprapur grade) digestion [7]. Quantification of analytes was carried out from calibration graphs obtained from aqueous standards, which were linear ($r^2 > 0.991$) in concentrations ranging from 0.02 to 1 mg/l (transition metals) and between 0.01 (0.001, Li) and 1 mg/l for alkali/alkaline earth metal cations. Analysis results of two high purity quartz samples by IC (HF, vapour phase digestion) and those obtained by independent techniques (ICP AES/MS, HF– H_2SO_4 , direct dissolution) are presented in Table 3 and in chromatograms shown in Figs. 4 and 5. The results for most trace analytes show no significant differences indicating that both HF (AR grade, VPD) and HF– H_2SO_4 (both suprapur grades) digestion give fairly representative sample solutions. The *t*-test with multiple samples (paired by differences) was applied to examine whether the results by IC (HF, vapour phase digestion) and ICP AES/MS (HF– H_2SO_4 digestion) differed significantly at the 95% confidence level. As the calculated values of *t* were less than the critical *t* value of 2.571 (degrees of freedom = 5), it follows that there is no statistically significant difference between the IC and ICP AES/MS results. In the two analysed quartz samples, the concentration of Co was below the detection limit of the proposed IC method. It is remarkable that the extremely low concentration of Li in quartz-2 (7 ng/g, Table 3, Fig. 4b) is matching well with the ICP MS results. Determination of iron is normally difficult using a quadrupole ICP MS instrument because of the strong interference of $\text{Ar}^{40}\text{O}^{16+}$ at *m/z* 56, which is identical with the most abundant isotope of Fe [21]. As stated before, both $\text{Fe}^{3+}/\text{Fe}^{2+}$ are critical species in certain applications of quartz [1], and in this aspect ion-chromatography provides much better sensitivity than ICP AES. Unfortunately, the present method cannot be utilised for quantification of Fe^{2+} , as during matrix volatilisation approximately 95% of Fe^{2+} is oxidised to Fe^{3+} by oxygen in air. Precision of the measurements expressed as

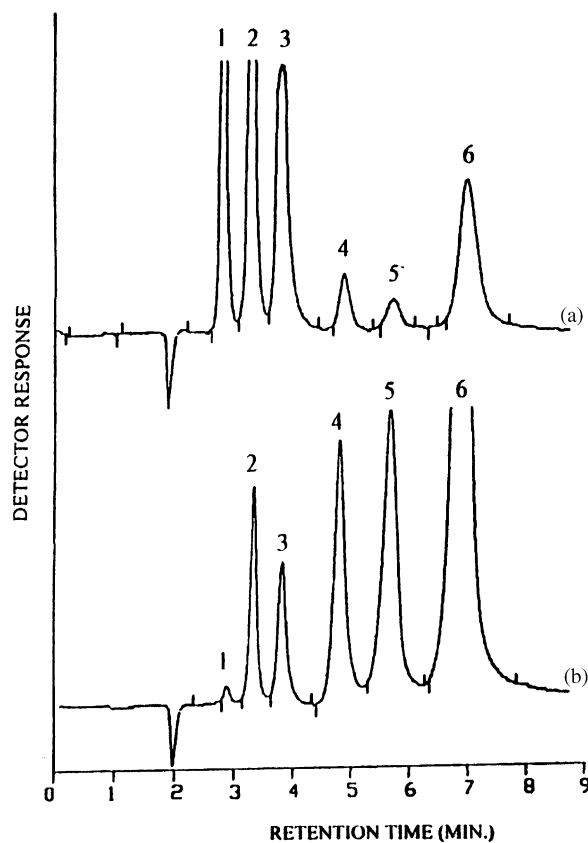


Fig. 4. (a) Chromatogram of trace level (alkali/alkaline earth) metals in high purity quartz-1. Peaks: (1) Li^+ (0.23 $\mu\text{g/g}$); (2) Na^+ (1.05 $\mu\text{g/g}$); (3) NH_4^+ (–); (4) K^+ (0.27 $\mu\text{g/g}$); (5) Mg^{2+} (0.07 $\mu\text{g/g}$); (6) Ca^{2+} (0.39 $\mu\text{g/g}$). (b) High purity quartz-2. Peaks: (1) Li^+ (0.007 $\mu\text{g/g}$); (2) Na^+ (0.41 $\mu\text{g/g}$); (3) NH_4^+ (–); (4) K^+ (0.68 $\mu\text{g/g}$); (5) Mg^{2+} (0.31 $\mu\text{g/g}$); (6) Ca^{2+} (2.3 $\mu\text{g/g}$). Chromatographic conditions are described in the text.

Table 3

Analyte concentrations ($\mu\text{g/g} \pm \sigma$) determined by ion-chromatography ($n = 6$) in quartz and comparison with results of independent methods ($n = 2$)

Analytes	Sample	IC	H_E	ICP MS ^a	ICP AES ^a
Li ⁺	qtz-1	0.23 ± 0.02	6	0.215	–
	qtz-2	0.007 ± 0.002	19	0.009	–
Na ⁺	qtz-1	1.05 ± 0.07	4	–	1.1
	qtz-2	0.41 ± 0.04	6	–	0.4
K ⁺	qtz-1	0.27 ± 0.02	5	–	0.25
	qtz-2	0.68 ± 0.03	4	–	0.72
Mg ²⁺	qtz-1	0.07 ± 0.01	10	0.10	–
	qtz-2	0.31 ± 0.04	8	0.34	–
Ca ²⁺	qtz-1	0.39 ± 0.06	7	–	0.43
	qtz-2	2.3 ± 0.2	6	–	2.4
Fe ³⁺	qtz-1	1.64 ± 0.12	3	–	1.5
	qtz-2	0.77 ± 0.08	4	–	0.85
Cu ²⁺	qtz-1	0.27 ± 0.02	4	0.29	–
	qtz-2	0.15 ± 0.01	4	0.16	–
Ni ²⁺	qtz-1	n.d.	–	<0.03	–
	qtz-2	0.075 ± 0.019	12	0.09	–
Zn ²⁺	qtz-1	0.085 ± 0.017	8	0.07	–
	qtz-2	0.065 ± 0.011	18	0.07	–
Co ²⁺	qtz-1	n.d.	–	<0.02	–
	qtz-2	n.d.	–	<0.02	–
Mn ²⁺	qtz-1	0.33 ± 0.02	6	0.35	–
	qtz-2	n.d.	–	<0.02	–

^a An amount of 0.5 g of quartz sample was dissolved in HF (5 ml, suprapur grade, Merck) and 0.25 ml H₂SO₄, after matrix and HF volatilisation (in clean bench) made up to 5 ml with 2% nitric acid. qtz, high purity quartz; n.d., not detected. H_E (homogeneity factor) = $(\text{R.S.D.})m^{1/2}$.

R.S.D. (%) was between 5 and 12% in most of the cases. Standard deviation includes inhomogeneity of the analysed material and measurement imprecision.

3.4. Homogeneity study

From the standpoint of sample processing, it is desirable that the gross sample weighs no more than absolutely necessary, which is basically determined by the degree of heterogeneity of the sample. Hence an optimised sample mass should be taken for analysis in order to minimise the influence of inhomogeneity on analytical variance. Kurfurst et al. [22] proposed a homogeneity factor so that characterisation of solid material homogeneity was feasible. The homogeneity factor (H_E) is related to R.S.D. and sample mass (m) through the following equation

$$H_E = (\text{R.S.D.})m^{1/2}$$

Materials with homogeneity factors less than 10 are said to be very homogeneous. The quartz samples (sample mass 250 mg, size –50 to +150 mesh) reported in this work, have homogeneity factors between 4 and 12 for most of the elements except for Li ($H_E = 19$) and Zn ($H_E = 18$) in

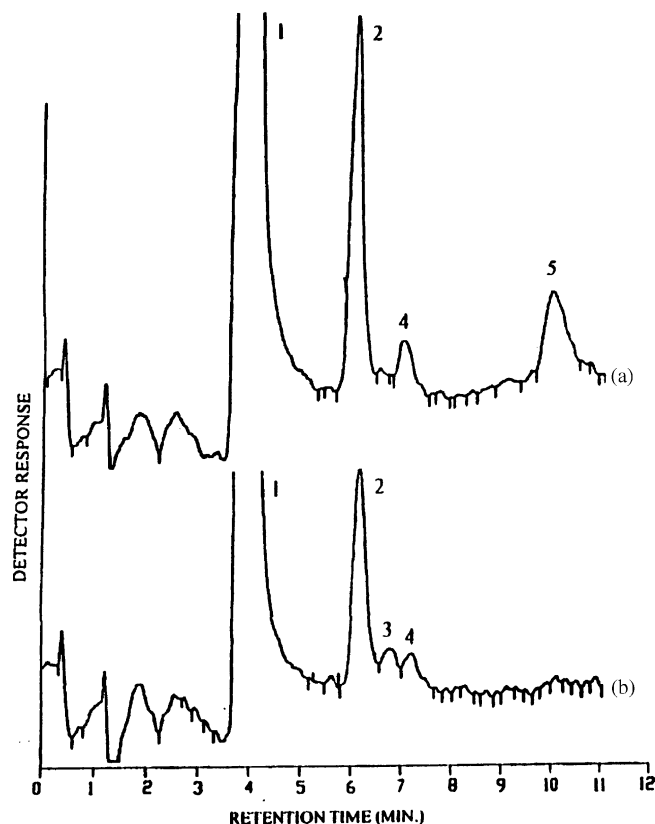


Fig. 5. (a) Chromatogram of trace level transition metals in high purity quartz-1. Peaks: (1) Fe³⁺ (1.64 $\mu\text{g/g}$); (2) Cu²⁺ (0.27 $\mu\text{g/g}$); (4) Zn²⁺ (0.08 $\mu\text{g/g}$); (5) Mn²⁺ (0.33 $\mu\text{g/g}$). (b) High purity quartz-2. Peaks: (1) Fe³⁺ (0.77 $\mu\text{g/g}$); (2) Cu²⁺ (0.15 $\mu\text{g/g}$); (3) Ni²⁺ (0.07 $\mu\text{g/g}$); (4) Zn²⁺ (0.06 $\mu\text{g/g}$). Chromatographic conditions are described in the text.

quartz-2 (Table 3). Considering the high sensitivity of IC, for quartz samples (–50 to +150 mesh) with higher impurity levels (>1 ppm), 100 mg sample is sufficient for analysis. But it was observed that the homogeneity factor for these elements was significantly high (between 15 and 30). It is to be expected that homogeneity factor for such samples would improve when working with smaller particle size (–150 mesh), which was not tried anticipating contamination during grinding.

4. Conclusion

The modified sample dissolution described in this work has led to the use of ion-chromatography for the analysis of high purity quartz. Dissolution carried out by vapour phase reaction controls the analytical blanks, which are often the limiting factors in accurate trace metal analysis. The multiplex digestion assembly used provides cost effective sample dissolution for trace analysis in high purity quartz. Further ion-chromatographic analysis provides high sensitivity, multi-element capability with low cost of analysis. All these cost savings do not compromise the quality of analytical results. In conclusion, high purity quartz represents a very suitable

matrix for ion-chromatography, when the matrix is removed by evaporation of the silicon tetrafluoride using HF alone. The low volume (μl) of sample required for quantification in IC, allows one to achieve only a limited dilution factor, so that the detection limits referred to the solid sample remain low.

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