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The Major-, Minor-, and Trace-Element Analysis of Silicate Rocks and Minerals from a Single Sample Solution

By *B. Ayranci*, Zürich *)

Abstract

The determinations of the major-, minor-, and trace-elements in a silicate rock and mineral are carried out from a single sample solution, produced in a non-oxidizing environment by means of $\text{HF} + \text{H}_2\text{SO}_4 + \text{H}_3\text{BO}_3$ acid disintegration.

This new procedure allows the determination of each component several times, and enables the analyst to check the analytical results for each component by means of different methods from a single solution.

Introduction

Most laboratories, working with wet chemical analytical methods, have given up using 3-sample solution methods according to the "wet-chemical rapid analytical procedures" (e.g. 41, 42, 45, 51) for the analysis of silicates. More usually analysis are performed with two sample solutions:

The first solution (identical to the metal-oxide solution c of the "wet chemical rapid analytical procedures") is used for the determination of the major-, minor-, and sometimes also trace-elements (e.g. 8, 13, 40).

The second solution is prepared by dissolution of the sample in $\text{HF} + \text{H}_2\text{SO}_4$ and is used for the determination of the ferrous iron (e.g. 7, 9, 23, 28, 42, 54). Generally, two different methods are used to prepare the first solution:

1. The fusion technique

The silicate sample is fused by means of a flux such as: LiBO_2 , $\text{Li}_2\text{B}_2\text{O}_7$, $\text{LiF} + \text{H}_3\text{BO}_3$, $\text{LiF} + \text{B}_2\text{O}_3$, $\text{LiF} + \text{Li}_2\text{CO}_3$, $\text{Na}_2\text{B}_2\text{O}_7$, Na_2CO_3 , NaOH etc. in a crucible of graphite, platinum, gold-platinum, zirconium, nickel, silver etc.,

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and the fusion product (cake) is dissolved in diluted acid such as HCl, HNO₃ etc. (e.g. 1, 11, 27, 35, 46).

2. The acid-disintegration method

The silicate sample is dissolved by means of HF + one more different acid such as HCl, HNO₃, HClO₄, H₂SO₄ etc. in an autoclave (e.g. 8, 13, 25, 32); boric acid is added after the decomposition of the sample. The quantitative analysis of many components (e.g. Si, Al, Ca, Mg, Ti). From the first solution (by either method 1 or 2) by means of atomic absorption spectrophotometric (AAS), colorimetric, complexometric etc. methods is complicated by the presence of silica and/or fluoroboric acid in the matrix (e.g. 1, 8, 20, 22, 34). The interference of these in the determination of the other components by AAS are commonly minimised by means of extreme dilution of the sample solutions and/or adding sufficient alkalis (e.g. Sr, Li, La, Cs, K) to the sample and standard solutions before measuring (e.g. 1, 8, 16, 20).

A further way to eliminate such interference is to use natural rock standards of similar composition to the sample solution instead of calibrating with pure metal salt solutions (7, 44).

The following discussion deals with a new analytical procedure:

1. The major-, (including ferrous iron), minor-, and trace-elements are determined from a single sample solution.
2. The interference of silica and/or fluoroboric acid matrix in the determination of other components are completely eliminated by a simple procedure (see below) so that the single solution can be used not only for AAS, but also for other methods of analyzing components.
3. The method allows an internal check of analytical reproducibility in several different steps for the same components by different methods.

The method has been tested with success by analyzing some international reference rocks and synthetic zeolites.

A. Analytical procedure

The single silicate solution is produced in a non-oxidizing environment by means of HF + H₂SO₄ + H₃BO₃ acid disintegration method (7). The SiO₂, ferrous iron, total iron are determined immediately by means of AAS and spectrophotometer from the aliquots of the silicate solution.

The interference of Si, B, F and/or H₂SO₄ on the analysis of other components are completely eliminated from the remainder of the solution by supplementary evaporation. The residue is dissolved with diluted HCl (1 + 9).

The analysis of components with this method is carried out by means of an AAS (Perkin-Elmer 420, combined with a printer and recorder) a flame-photometer (EEL 100), a spectrophotometer (Zeiss PM-6) and EDTA titrations. The other analytical methods can also be applied for the determination of components (e.g. volumetric, potentiometric, polarographic, ion selective electrodes methods).

The preparation of sample solution

Weigh accurately 0.200 g sample (dried at 105°C) in a 15 ml PTFE-cylinder (7), add 1–2 ml previously boiled cold H₂O, mix well; add 2.5 ml H₂SO₄ (96%), 5 ml H₂O, 1 ml HF (40%), fill up with H₂O (all H₂O used for the sample solution is boiled deionised H₂O) and close the cylinder well to prevent vaporization of Si, F, B and oxidation of Fe (II); shake it for 1–2 minutes, set in a water bath (80°C) to $\frac{1}{3}$ of its length. Occasionally remove the cylinder from the bath and shake it a short time. Generally the aluminosilicates dissolve within 2–3 hours, but the samples rich with resistant minerals such as chromite, magnetite, spinel etc. need a very long time. An alternative fusion method is being tested for these kinds of samples (AYRANCI, in preparation). After complete dissolution of the sample (which appears as a milky solution in the PTFE-cylinder), remove the cylinder and cool it quickly in H₂O. Open the cylinder and quantitatively wash the dissolved sample solution with warm H₂O and 20 ml 2.5% (W/V) boric acid into a 250 ml conical PVC-flask. Add a further 40–50 ml warm H₂O and a magnet bar. Close the flask well and mix the solution vigorously by means of a magnetic stirrer. Remove the magnet bar and wash. Fill up with H₂O to 250 ml (*main solution*). The main solution must be very clear. Treat in the same way and at the same time 3–5 rock standards and a blank. Transfer the main solution into a 250 ml PVC-flask and close well with a screwed lid.

The main solution is separated into two portions: Aliquot *a* and aliquot *b*.

Aliquot a (800 ppm), transfer accurately 50 ml of the sample, standard, and blank from the main solutions into a 50–100 ml PVC-flasks; screw well and reserve for the determination of the SiO₂, FeO, and/or total iron.

Aliquot b (3200 ppm), is a silica-fluoroboric-, H₂SO₄-acid free solution and it is prepared from the main solution as follows: Quantitatively wash the rest of the main solutions of the sample, standard, blank into a platinum, or PTFE-dish or vitreous carbon-dish and evaporate slowly on a water or sand bath. Remove the probable residues of silica by adding 0.5–1.0 ml HF (40%) and 1.5–2.5 ml H₂SO₄(2+1) or HNO₃ to the dishes. Evaporate again by means of a quartz lamp and/or an infrared lamp or a sandbath. Dissolve the still-

moist residue in 5–10 ml HCl (1 + 9) and 20–25 ml H₂O by means of a heated magnetic stirrer. Quantitatively transfer the solution into a 50–100 ml conical flask and make up with H₂O to 50–100 ml and mix well. Transfer the aliquot *b* in a PVC-flask; screw and mix well. The aliquot *b* is used for several methods (e.g. colorimetric AAS, potentiometric, polarographic, ion selective electrodes, complexometric methods) for the recovery of individual components except ferrous iron, silica, B, F.

B. Determination of components

The colorimetric methods are used for the determination of the ferrous iron, total iron, sometimes SiO₂; P₂O₅, TiO₂ from the aliquot *a* and *b*.

The AAS methods are applied for the analysis of SiO₂, total iron, from the aliquot *a* and for the determination of many major-, minor-, and trace-elements from the aliquot *b*.

The flamephotometer is used for the determination of Na₂O, K₂O, and Li₂O from the aliquot *b*.

The determination of CaO and MgO is also carried out by EDTA titration from the aliquot *b*.

I. DETERMINATION BY THE SPECTROPHOTOMETER (sf)

1. The determination of ferrous iron

The ferrous iron is determined as α - α' -red dipyridyl ferrous iron complex from the aliquot *a*. The optical density is measured at 522 nm in a 1 cm cell. For the calibration of the ferrous iron three natural silicate standards are used which are prepared together with the sample solution. The colorimetric determination is selective and simple.

Reagents: a) α - α' -Dipyridyl solution: Weigh 0.200 g of α - α' -dipyridyl salt and dissolve it in 100 ml HCl (1.5 + 98.5), filter if necessary.

b) Sodium-acetate (2 M) solution: Weigh 136 g sodiumacetate (anhydrous) and dissolve it in ca 250–300 ml and dilute to 500 ml.

Procedure: Transfer accurately 2.5–5 ml (= 2–4 mg) of the aliquot *a* in a 100 ml conical flask and add 20–30 ml H₂O, 1 ml reagent *a* and 5 ml reagent *b* fill up to 100 and mix well.

2. The determination of total iron (as Fe₂O₃)

If desired, the total iron can be determined as a ferrous iron-dipyridyl complex either from the aliquot *a* or from the aliquot *by* means of spectrophotometer (see analytical scheme).

Reagents: Dissolve 0.400 g α - α' -dipyridyl in ca. 200 ml H₂O by adding 3.5 ml HCl, dissolve 10 g hydroxylammonium hydrochloride in ca. 200 ml H₂O; dissolve 34 g sodium acetate (anhydrous) in ca. 300 ml H₂O. Mix all together and transfer into a 1000 ml conical flask; fill up with H₂O to 1000 ml.

Procedure: Transfer 1–5 ml from the aliquot *a* or 0.5–1.0 ml from the aliquot *b* in to a 100 ml conical flask and add ca. 20–30 ml H₂O and 10 ml reagent; fill up with H₂O to 100 ml and mix well.

The optical density is measured at 522 nm in a 1 cm cell and the calibration of total iron is carried out with 3 rock standards.

3. The determination of TiO₂

The TiO₂ is determined as a yellow hydrogen peroxide complex from the aliquot *b*. The aliquot *b* can also be used directly for the determination of TiO₂ by means of AAS. The determination of TiO₂ (by colorimetric methods) and Al₂O₃ (by colorimetric and/or complexometric methods) is best done using aliquot *b*. This is because this aliquot contains only a minimum amount of F, compared to the decomposition methods, solutions produced by HF + HClO₄ or HF + HNO₃.

Reagents: Transfer 5 ml H₂SO₄ (96%), 10 ml H₃PO₄ (85%) and 10 ml H₂O₂ (30%) into a 100 ml conical flask and mix, dilute to 100 ml.

This solution must always be freshly prepared.

Procedure: Pipette 5 ml (= 16 mg) solution from aliquot *b* in a 20 ml beaker and add 5 ml reagents.

The optical density is measured at 430 nm with a 1 cm cell. The calibration of TiO₂ is carried out with 3 rock standards. If the concentration of TiO₂ is over 2.5%, 2.5 ml (= 8 mg) of solution must be used.

4. The determination of P₂O₅

The P₂O₅ is determined as a yellow vanadomolybdate complex from the aliquot *b* at 430 nm. The optical density is measured in a 1 cm cell. The calibration curve is linear up to 0.5% P₂O₅.

Reagent: Dissolve 0.5 g of NH₄VO₃ in ca. 200 ml H₂O and add 66 ml HNO₃ (65%). After cooling mix with 10 g (NH₄)₆Mo₇O₂₄·4H₂O dissolved in ca. 300 ml H₂O. Mix all together and transfer into a 1000 ml conical flask and fill up with H₂O.

Procedure: Transfer 5 ml (= 16 mg sample) from the aliquot *b* in a 20 ml beaker; add 5 ml of the reagent and mix well.

The optical density is measured after 5 minutes. The calibration of P₂O₅ curve is carried out with 3 rock standards.

II. DETERMINATION BY THE ATOMIC ABSORPTION-SPECTROPHOTOMETER (AAS)

The AAS-methods are used more frequently for the determination of many major-, minor-, and trace-element analysis: SiO_2 and/or total iron are analyzed from the aliquot *a* (800 ppm), but the other components are determined from the aliquot *b* (3200 ppm solution).

The determination of components from the aliquot *a*

1. The determination of SiO_2

The SiO_2 concentration (about to 250 ppm Si) is determined directly from the aliquot *a* and a $\text{C}_2\text{H}_2\text{-N}_2\text{O}$ flame is used at 251.9 nm (slit 0.2) during the analysis. The absorption signals on the recorder are expanded 2–3 times. The calibration of SiO_2 is carried out with the use of reference rock standards (e.g. U.S.G.S. Standard Set). In our experience the alternative method using a series of a stock SiO_2 solutions (prepared from SiO_2 or sodium metasilicate) is not sufficiently accurate, although this method is used by several analysts.

2. Total iron

The total iron may be determined in small concentration (about 10%) directly from the aliquot *a* by means of a $\text{C}_2\text{H}_2\text{-N}_2\text{O}$ flame. 1 ml La 4% may be added to overcome the interferences due to the presence of silica on the determination of total iron in the sample and standard solutions. It is, however, more useful to use natural rock standards for the calibration.

The determination of total iron can be carried out more easily from the aliquot *b* by adding 0.5–1 ml La 4% and by means of an air-acetylene flame at 248.1 nm (slit 0.2). It is however simpler to determine the total iron either from the aliquot *a* or from the aliquot *b* (see analytical complete scheme) by means of colorimetric methods.

Determination of components from the aliquot *b*

The fluoroboric acid, silica free solution (aliquot *b*) allows the determination of all other components by means of AAS, spectrophotometer, complexometric titrations, volumetric determinations, flamephotometric methods etc.

Three different dilution from the aliquot *b*, by adding 0.5–1.0 ml La 4% or Cs – will be sufficient for the determination of Al, total iron, Ca, Mg, Mn, Na, K etc. by means of AAS.

Solution I:

Transfer accurately 5 ml (= 16 mg sample) of aliquot *b* into a 50–100 ml conical flask, add 20–30 ml H_2O + 1 ml La 4% and fill up to 50–100 ml with H_2O and mix well.

- Solution II:* Transfer accurately 2 ml (= 6.4 mg sample) of aliquot *b* into a 100 ml conical flask and add ca. 20–30 ml H₂O + 1 ml La 4% and fill up with H₂O to 100 ml and mix well.
- Solution III:* Transfer accurately 1 ml (= 3.2 mg sample) of aliquot *b* into a 100 ml conical flask and add ca. 20–30 ml H₂O + 1 ml La 4% and fill up with H₂O to 100 ml, mix well.
- La-solution 4% (w/v):* Dissolve by heating 47 g La₂O₃ (99.99%) in 200 ml HCl (25%) and dilute with H₂O to 1000 ml (1 ml = 4% La).

1. The determination of Al₂O₃

(and eventually MnO total iron, CaO, MgO, K₂O, Na₂O, etc.)

The Al₂O₃ is determined with a C₂H₂-N₂O flame at 309.3 nm (slit 0.7) from the solution (I) by means of AAS. The calibration curve is linear up to about 50 ppm Al, and therefore two or three natural silicate standards are sufficient to calculate a factor for the determination of Al₂O₃ concentration, if the absorptions are measured.

The determination of CaO, total iron, MnO and in some cases MgO (up to the concentration of 7, 5, 3, 0.5 ppm) can be carried out from the same solution by means of an air-C₂-H₂-flame.

The K₂O and Na₂O, Li₂O is determined in small concentrations from the same solution. If the K₂O and Na₂O concentrations lie between 2–6%, the determination can also be made by means of a flamephotometer.

2. The determination of CaO

(and eventually Al₂O₃, total iron, MgO, MnO, K₂O, Na₂O etc.)

In most cases the CaO is determined from the solution II with an air-acetylene-flame at 422.8 nm (slit 0.7) by AAS. The same solution is also used for the determination of alkalis and MgO, MnO, total iron, Al₂O₃ and Li₂O etc., if the concentration of elements lies within the detection limit but not over the linear range of components (i.e. Al₂O₃ ~ 10 ppm, MgO < 0.8 ppm). The determination of K₂O, Na₂O, Li₂O is sometimes carried out from solution II by means of flamephotometry.

The calibration curve is made with natural silicate rock standards.

3. The determination of MgO

(and eventually CaO, Na₂O, K₂O, Fe₂O₃, MnO, Al₂O₃ etc.)

Solution III is prepared for the determination of MgO, at 285 nm (slit 0.7) with the air-acetylene flame by AAS. This solution is mostly suitable for the determination of alkali components but less useful for the determination of other components such as Al₂O₃, Fe₂O₃ by means of AAS. In some cases however it is possible to determine total iron and CaO, Li₂O from this solution.

The calibration curve for the determination of MgO is constructed with natural rock standards.

Determination of the trace elements from the aliquot *b*

For the determination of trace elements from the aliquot *b* two dilutions are prepared:

Solution IV: Transfer 5 ml (= 16 mg sample) of aliquot *b* into a 25 ml conical flask, and 0.5 ml La 4% and fill up with H₂O to 25 ml; mix well.

Solution V: Transfer 10 ml (= 32 mg sample) of aliquot *b* into a 25 ml conical flask and add 0.5 ml 4% La and fill up with H₂O to 25 ml; mix well.

The solution IV allows the determination of several trace elements such as Ag, Au, Be, Co, Cu, Cr, Cs, Li, Ni, Pb, Sr, Zn, Ti, etc.

The solution V can be used for the determination of Ba, V, Ti, Sn, etc. in the detection limit of AAS.

The trace element analysis can also be carried out from the solution I–III. However, in many cases these solutions are too diluted for the determination of trace elements. The standard conditions for the determination of individual elements are given in detail by the manufacturers of AAS instruments and therefore are not discussed further.

III. DETERMINATION OF Na₂O, K₂O, Li₂O BY FLAMEPHOTOMETRY (FP)

The determination of K₂O, Na₂O, Li₂O is sometimes more successful by flamephotometry rather than by AAS methods, if the concentration of these components is over 3%.

Aliquot *b* is diluted and used for the determination of Na₂O, K₂O, Li₂O (solution VI). The solution VI may also be used for the AAS determination of other components.

Solution VI: Transfer 3 ml (= 9.6 mg sample) of aliquot *b* into a 100 ml conical flask and add 20–30 ml H₂O + 1 ml La 4% solution and make up with H₂O to 100 ml. The calibration of alkalis is nearly linear up to 3% using silicate rock standards. Above 3% it is necessary to use a calibration curve to 6%. The standard solution for the calibration curve may be produced from a stock solution.

Stock-solution (Na₂O, K₂O, Li₂O = 1000 ppm)

Weigh accurately 1.886 g NaCl (dried at 105° C), 1.583 g KCl (dried at 105° C), 2.472 g Li₂CO₃ (dried at 105° C) in 500 ml beaker, add 250–300 ml H₂O and 5 ml HCl and dissolve by warming.

After cooling, transfer the solution quantitatively into a 1000 ml conical flask and mix well. Fill up with H₂O to 1000 ml (1 ml solution = 1.0 mg Na₂O, 1.0 mg K₂O, 1.0 mg Li₂O).

The standard solutions for the calibration curve

Transfer accurately 0, 0.2, 0.3, 0.4, 0.5, 0.6 ml (respectively 0, 0.2, 0.3, 0.4, 0.5, 0.6 mg Na_2O , K_2O , Li_2O) of stock solution into six 100 ml conical flasks, add 20–30 ml H_2O and 1 ml La 4%, fill up with H_2O to 100 ml, mix well.

These solutions are used for the calibration curve of the Na_2O , K_2O , Li_2O and the determination of Na_2O , K_2O , Li_2O are carried out from the calibration curve after measurement with the flamephotometer.

The solutions VI can also be used for the determination of other components such as Al_2O_3 , MnO , MgO , total iron and CaO by means of AAS.

In most cases it is sufficient to use 3 diluted solutions from the aliquot *b* for the determination of at least six major and minor components by means of AAS and/or flamephotometer (see analysis scheme).

IV. DETERMINATION OF SOME COMPONENTS BY OTHER METHODS

If the samples contain higher concentration of MgO , CaO , Al_2O_3 total iron etc. (i.e. 30–40%) and only a few samples need to be analyzed, it is better to use classical rapid analytical methods for the determination of components: i.e. the CaO and MgO determination can be carried out very quickly from the 5–10 ml or more (16–32 mg sample or more) of aliquot *b* by means of complexometric titrations (6), or total iron can be determined volumetrically etc.

C. Conclusions

These new analytical procedures are more useful than the classical rapid wet chemical analytical methods because,

1. The determinations of the major-, minor-, and trace-elements including ferrous iron are carried from a single sample solution.
2. The inhomogeneity problem, which may result from 2–3 different weighings of the sample, is completely eliminated.
3. The method uses smaller amounts of sample for the analysis of components, but the determination of SiO_2 is carried out from the larger amount of sample.
4. The analytical cost in financial terms is small and the time of sample solution is relatively short. The method can be used easy for the rapid analysis of a series of samples.

5. The method allows the determination of components several times by different selected analytical instruments and methods; therefore it has many features allowing determination of internal consistency.

The samples are dissolved at relatively low temperatures (60–70°); because of this, it is possible that some undissolved minerals such as rutile, pyrite, chromite, zircon may remain. In this case it is necessary to use an autoclave. An alternative fusion method is now being tested in our laboratory and will be used for the analysis of eclogite rocks. The analytical results from some international rock standards determined using these procedures are tabulated below (see Table 1).

The amounts of the required sample weight and the volumes of the solution have been cited above in the analytical procedure. They may be changed without difficulty according to the specific requirements. For example:

100 mg sample may be used to produce 100 ml of main solution, and 20 ml (= 20 mg) of the main solution can be diluted after evaporation to give a 100 ml aliquot *b*.

Furthermore it might be adequate to evaporate only a small portion of the main solution after SiO₂, FeO and/or total iron have been determined, if only few components have to be determined.

For example: To determine SiO₂, FeO, \sum Fe, Al₂O₃, CaO, Na₂O, K₂O, MnO, MgO in a granitic rock. First, SiO₂ is determined by AAS in few ml of the main solution. 5 ml of the main solution is used for the colorimetric determination of ferrous iron and a further 5 ml for total Fe (as Fe₂O₃) by means of spectrophotometer. Then, 25 ml of the main solution is transferred into a dish of platinum, PTFE or vitreous carbon and evaporated. The residue is dissolved with 10 ml HCl (1+9), 1 ml La 4% is added to the solution and diluted with H₂O to 100 ml (aliquot *b*).

Now Al₂O₃, \sum Fe (a second determination for checking), CaO, MnO, MgO are determined by AAS and Na₂O, K₂O, Li₂O are also determined by means of a flamephotometer from the same solution. If it is necessary the solution could be also diluted by 1/2 or 1/3 for the determination of alkalis.

This work deals only with the outline of the determination of components from a single silicate solution. It does not discuss in detail the difficulties, which depend upon the specific element and the analytical method used. Only the analyst's experience will tell how to avoid or overcome such difficulties.

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Table 1. The analytical results of the major-, minor-, and trace-elements from a single solution

Ref. Rocks	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	FeO	Weight %		TiO ₂	MnO	P ₂ O ₅	Cr	ppm			
					MgO	CaO					Cu	Ni	Sr	Ba
BR	38.25	10.22	5.62	6.55	13.32	13.76	2.63	0.2	1.05	420	70	270	1365	1075
BM	49.5	16.15	1.65	7.35	7.45	6.4	1.16	0.15	0.11	125	50	60	230	250
*BHVO-1	50.4	14.08	2.75	8.55	7.05	11.2	2.61	0.17	0.34	330	150	nd	320	nd
JB-1	51.95	14.58	2.26	6.0	7.68	9.3	1.37	0.17	0.25	nd	nd	nd	440	410
*STM-1	59.75	18.60	3.13	2.09	0.1	1.35	0.15	0.22	0.16	nd	nd	nd	nd	nd
ASK-1	59.45	18.72	1.7	2.6	0.98	3.15	1.15	0.13	0.01	nd	nd	125	700	1100
FK-N	65.05	18.65	0.04	0.07	0.01	0.13	0.03	0.004	0.02	nd	nd	nd	50	200
GS-N	65.85	14.71	1.89	1.62	2.28	2.55	0.65	0.05	0.28	nd	nd	nd	590	1400
*QLO-1	65.9	16.35	1.1	3.2	1.06	3.41	0.63	0.09	0.26	nd	nd	nd	380	1300
JG-1	72.3	14.25	0.38	1.65	0.75	2.15	0.27	0.05	0.09	60	nd	nd	180	nd
*RGM-1	73.3	13.9	0.56	1.3	0.3	1.32	0.3	0.04	0.06	nd	nd	nd	130	nd
GM	73.65	13.55	0.72	1.18	0.41	1.04	0.24	0.04	0.06	nd	nd	nd	135	330

Reference standards used for the calibration

DR-N	52.65	17.45	3.9	5.45	4.5	7.1	1.1	0.21	0.28	45	50	15	400	360
BCR-1	54.5	13.6	3.7	8.8	3.45	6.9	2.2	0.18	0.36	25	15	15	330	675
AGV-1	59.0	17.25	4.5	2.0	1.5	4.9	1.05	0.1	0.49	15	60	20	655	1210
GA	69.9	14.5	1.35	1.3	1.0	2.45	0.35	0.09	0.12	15	15	10	305	850

Abbreviations: nd = not determined

* Mean value from the 3 splits of sample

Synthetic Zeolite Na-A 32.68 27.72 16.85 22.75 Theoretical composition

(GS 38 Lü B) 32.15 27.66 17.06 (22.75) Analytical results (using a single solution, of ca. 150 mg sample. Analyst: B. Lütthi)

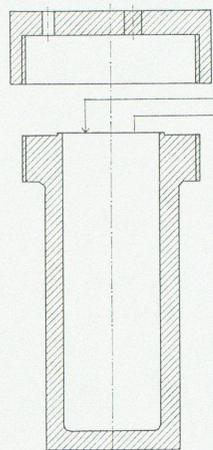
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The Analysis Scheme of the Major-, Minor- and Trace-Elements from a Single Silicate Solution

0.200 g sample + 1–2 ml H₂O + 2.5 ml H₂SO₄ (96%) + 1 ml HF (40%) + H₂O
+ 20 ml H₃BO₃ 2.5% (W/V)

MAIN SOLUTION
250 ml (= 200 mg)

Aliquot a

50 ml (= 40 mg) of the main solution

200 ml (= 160 mg)
Main Solution
– (Si, F, B, H₂SO₄)

SiO₂
> 250 ppm, Si nd
AAS C₂H₂-N₂O

Aliquot b
50 (or 100) ml/160 mg including
5 (or 10) ml HCl (1+9)

Σ Fe
> 5 ppm nd Fe + 1–2 ml
La 4%
AAS C₂H₂-N₂O

TiO₂
> 200 ppm Ti nd
AAS C₂H₂-N₂O

Complexometric Determination

p 5 ml (= 16 mg) + 50–100 ml
H₂O Titr. with ml EDTA
pH ~ 13 CaO

p 5 ml (= 16 mg) + ~100 ml H₂O
Titr. with EDTA
pH ~ 10.5 CaO + MgO

Abbreviations:

p = pipette
nd = not dilute
dw = dilute with H₂O
+R = add reagents
sf = Spectrophotometer
Titr. = titrate with
AAS = Atomicabsorption-Spectrophotometer
FP = Flamephotometer
SiO₂* = See for the preparation of the SiO₂ reagents
references (e.g. 51,53)

Atomic-Absorption-Spectrophotometric and Flame-Photometric Determination

Solution I
p 5 ml (= 16 mg) + H₂O + 1 ml La 4% dw 50 (100) ml.
Determ.: Al₂O₃, MnO, Σ Fe; CaO, MgO; Na₂O, K₂O, Li₂O etc.
AAS+FP

Solution II
p 2 ml (= 6.4 mg) + H₂O + 1 ml La 4% dw 100 ml.
Determ.: CaO, Σ Fe, MgO, MnO, Al₂O₃; K₂O, Na₂O, Li₂O etc.
AAS+FP

Solution III
p 1 ml (= 3.2 mg) + H₂O + 1 ml La 4% dw 100 ml.
Determ.: MgO, Na₂O, K₂O, Li₂O; CaO, MnO, Σ Fe, Al₂O₃ etc.
AAS

Solution IV
p 5 ml (= 16 mg) + 0.5 ml La 4% dw 25 ml.
Determ.: Ag, Au, Be, Co, Cu, Cr, Cs, Li, Ni, Pb, Sr, Zn, Ti, etc.
AAS

Solution V
p 10 ml (= 32 mg) + 0.5 ml La 4% dw 25 ml.
Determ.: Ba, V, Ti, Sn, etc.
AAS

Solution VI
p 3 ml (= 9.6 mg) + H₂O + 1 ml La 4% dw 50 (or 100) ml.
Determ.: K₂O, Na₂O, Li₂O; MnO, CaO, Σ Fe, MgO, Al₂O₃ etc.
FP+AAS

Spectrophotometric Volumetric Determination

SiO₂* > 75% SiO₂
p 1–2 ml (0.8–1.6 mg) + H₂O
+R dw 250 ml sf 640–750 nm

FeO > 10% FeO
p 2.5–5.0 ml (2–4 mg) + H₂O
+R dw 100 ml sf 522 nm

FeO
p 20 ml (= 16 mg) + ca. 50 ml H₂O
Titr. 0.01 N KMnO₄

Σ Fe (as Fe₂O₃) > 10% Fe₂O₃
p 2.5–5.0 ml (= 2–4 mg) + H₂O
+R dw 100 ml sf 522 nm

Σ Fe (as Fe₂O₃) > 10% Fe₂O₃
p 0.5–1.0 ml (= 1.6–3.2 mg) + H₂O
+R dw 100 ml sf 522 nm

TiO₂ > 2,5% nd TiO₂
p 2.5–5.0 ml (= 8–16 mg) + R nd
sf 430 nm

P₂O₅ > 0,5% nd P₂O₅
p 2.5–5.0 ml (8–16 mg) + R nd
sf 430 nm