Change in Chemical Composition of Rock Samples during Preparation for Analysis

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It has been shown that up to 3% of the volume of rock samples may escape as dust during their preparation (crushing and grinding) for chemical analysis. The mineral and chemical composition of the escaping rock dust may frequently differ from that of the main sample, as demonstrated by analytical examples. The effect of such selective separation can, in most cases, be regarded as negligible, though in some instances it may be of considerable importance in an evaluation and classification of the sample.

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Introduction

The preparation of rock samples for chemical analysis is a very important process. This first stage as a rule includes the crushing, splitting, grinding, mixing and, if necessary, the drying of the sample (Kolthoff & Sandell 1964). The equipment generally used is a jaw crusher, a splitter and an agate mortar or mill with agate inset. Jaw crushers are widely employed in laboratories to reduce the large fragments of silicates to pea size or smaller. This step always leads to the formation of rock dust, some of which escapes from the main sample. The amount of rock dust formed depends on several factors, the most important apparently being the brittleness, the fissility and the hardness of the mineral (Taggart 1950). Jaw crushers are frequently equipped with a suction device to eliminate the dust nuisance. Clearly, the greater the efficiency of the suction, the larger will be the amount of the rock dust removed. In the present study, the amount of rock dust escaping during the preparation of samples was estimated to be about 3 per cent. A comparison of the chemical composition of the main sample and that of the derived dust is therefore of interest.

In the present investigation dust and main samples of rock types with lithologies varying in chemical composition from acid to ultrabasic were analysed by normal wet chemical methods. In addition, X-ray diffractometer determinations were carried out on the same samples to establish the distribution of minerals in the main powder and in the corresponding dust fraction.

Crushing and pulverizing. Analytical methods

In the present investigation a jaw crusher from Morgaardshamar Mek. Verksted AB. has been used. The jaw crusher was connected to a cyclon with a hose, and a special filter (100 mesh) for collecting the rock dust was placed

inside the hose. The desired reduction of the fragments was achieved by narrowing the gap between the two metal plates of the jaw crusher. Each sample was treated twice in the crusher, first with a wide gap and secondly with a narrow gap. After this step, the sample consists of rock fragments ranging in size from about one mm to the finest rock dust. The main samples are then repeatedly halved until the weight of the powder is about 30 g. This is then treated in a mill with agate inset for about 1½ minutes, after which all the fragments are smaller than 100 mesh. The pulverized rock sample is then transferred to a sample box.

The technique employed in the determination of the major components is based partly on gravimetric, spectrophotometric and flamephotometric methods. The determination of silica, alumina, calcium, magnesium and total iron has been carried out after the conventional classical method. The determination of titanium, manganese, sodium, potassium and ferrous iron has been based on a method described by Langmyhr & Graff (1965). For the determination of water the method of Penfield was employed, while the carbon dioxide determinations were carried out by the direct method described by Kolthoff & Sandell (1959). Further, the determination of principal minerals both in the main sample and in the dust fraction was made by X-ray diffractometry (Table 1).

Results

As can be seen from Table 1, there is some change in the proportion of the main minerals in the two fractions. According to E. Evensen (personal communication 1972), these changes can be quite large for some trace elements. In specimens I–V the minerals somewhat concentrated in the rock dust are those of microcline, mica, chlorite and calcite, as well as rutile and sphene. The last two minerals are accessories and are omitted from Table 1. Acid plagioclase is found to be relatively concentrated in the main fraction, while quartz is either weakly concentrated in the main sample or shows an equal distribution. Equal distributions seem to be the rule for the minerals pyroxene and epidote.

As a consequence of these changes in the mineral concentrations, some differences will also occur in the chemical compositions of the two fractions. Table 2 shows the average chemical compositions of 38 rock samples and the average differences in the corresponding dust fractions. The mineral composition of the rock samples is shown in Table 1, columns I–V. Possible accessory minerals have not been considered in this example.

The differences between the chemical composition of the main fraction and that of the dust fraction can be completely explained by the change in mineral content in these two fractions, with the exception of the higher content of divalent iron in the rock dust. This iron increase would probably be partly due to contamination from the crusher. Minute fragments ($<$ 150 mesh) of iron would tend to tear from the jaws and mix with the rock dust.

Table 1. Comparison of mineral contents in the main samples and the corresponding dust fractions

Sample number	I	II	III	IV	V	VI	VII	VIII
Acid plagioclase	D	D	D	D	D	D		
Microcline	I			I		I		
Quartz	E	D	E	D				
Mica	I	I		I	I			I
Pyroxene				E				
Hornblende			I		D			
Epidote	E	E	E					
Chlorite		I	I		I			E
Dolomite								D
Magnesite								D
Calcite	I	I						
Serpentine							I	
Talc								I
Olivine							D	

D   Mineral proportion decreased in the rock dust relative to that in the main fraction.
I   Mineral proportion increased in the rock dust relative to that in the main fraction.
E   Equal distribution of the minerals in the two fractions.

Table 2. The average of chemical analyses for 38 rock samples and the oxide variations in the corresponding dust fraction in absolute per cent

Average composition of 38 rock samples		Average deviation in the corresponding dust fraction
SiO_2	69.00	− 2.70
TiO_2	0.448	+ 0.063
Al_2O_3	12.07	+ 0.71
Fe_2O_3 (tot)	4.12	+ 0.83
FeO	2.62	+ 0.65
MnO	0.08	+ 0.02
MgO	3.40	+ 0.20
CaO	1.81	+ 0.22
Na_2O	3.51	− 0.21
K_2O	2.72	+ 0.15
H_2O-	0.31	+ 0.10
H_2O+	1.08	+ 0.41
CO_2	0.29	+ 0.12
P_2O_5	0.058	+ 0.020

The mineral combination shown in Table 1, column VI, is representative of feldspar. The average of the chemical composition of the main fraction of 10 feldspar samples and the differences in the corresponding dust fraction are presented in Table 3.

The results (Table 3) show smaller variations between the dust fraction and the main sample, but the tendencies are the same as those noted in Table 2.

Table 1, column VII, represents serpentine rocks. The results show a relative increase of serpentine concentration and a decrease of the olivine concentration in the rock dust. In Table 4 the average chemical composition of the main fraction of 6 serpentine rocks is presented together with the average deviation in the corresponding dust fraction.

Table 3. Average of 10 analyses of feldspar and the variation in the
corresponding dust fraction in absolute per cent

Average of 10 feldspar samples		Average of the deviation in the corresponding dust fraction
SiO_2	64.34	— 0.40
Al_2O_3	20.08	+ 0.24
Fe_2O_3 (tot)	0.07	+ 0.08
MgO	0.13	+ 0.01
CaO	0.46	+ 0.15
Na_2O	4.35	— 0.01
K_2O	9.91	+ 0.05
CO_2	0.14	+ 0.01

Table 4. Average chemical composition of serpentine rocks and the differences
in oxide contents in the corresponding dust fraction (absolute per cent)

Average of 6 serpentine rocks		Average of the deviation in the corresponding dust fraction
SiO_2	37.14	— 0.43
Fe_2O_3 (tot)	8.55	— 0.83
FeO	5.06	— 0.28
MnO	0.09	+ 0.04
MgO	44.10	+ 0.24
H_2O^-	0.01	+ 0.09
H_2O+	6.68	+ 1.22

Table 5. Average chemical composition of 6 steatite samples and the
deviation in corresponding dust fraction (absolute per cent)

Average of 6 steatite samples		Average of the deviation in the corresponding dust fraction
SiO_2	36.33	+ 3.30
TiO_2	0.38	— 0.07
Al_2O_3	6.42	— 1.27
Fe_2O_3 (tot)	9.71	— 0.38
FeO	7.63	— 0.04
MnO	0.15	+ 0.02
MgO	28.60	+ 0.56
CaO	3.07	— 0.62
Na_2O	0.04	— 0.04
K_2O	0.60	— 0.07
H_2O^-	0.12	+ 0.09
H_2O+	6.33	+ 0.65
CO_2	9.33	— 2.19
P_2O_5	0.04	+ 0.01

As a consequence of the mineral separation, the oxide percentages presented
in Table 4 show considerable deviations for iron and chemically bound water.
    Some experiments were also carried out on rock samples containing the
mineral combination shown in Table 1, column VIII, and the results of these
analyses, showing the average of 6 rock samples (Steatite), are presented in
Table 5.

From Table 1, column VIII, it can be seen that talc and mica are enriched in the dust fraction and that dolomite and magnesite are less dust-forming in this particular mineral assemblage. These differences are sufficient to explain the deviation in the chemical composition as shown in Table 5. In this case there is no contamination of iron in the dust fraction since we are dealing with a soft rock type.

Conclusions

It can be concluded from the present investigation that a certain measure of separation of minerals from rock samples occurs during the crushing process. Because the content and combination of minerals vary so much from sample to sample, it does not, however, appear feasible to advance any theory as to how much this separation will influence the ultimate analytical result. In most cases the oxide variations can be regarded as negligible, but in some instances these would have a bearing on the evaluation of the sample. The deviation and variation trends reported here should therefore be borne in mind when considering the results of chemical analyses, particularly those judged to be critical for interpretation or classification purposes.

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