A PLEA FOR GREATER COMPLETENESS IN CHEMICAL ROCK ANALYSIS.[1]

BY W. F. HILLEBRAND.

THE valuelessness to the mineralogist and geologist of many of the analyses of mineral substances made in earlier times is a fact too well-known to need substantiation. Defective methods of analysis, the difficulty of procuring pure reagents, and want of time for exhaustive examination have been largely responsible for this condition, but lack of appreciation of the fact, now so well established, that substances present in small amount may have an important bearing on the discussion of results, has no doubt contributed in no small measure to it.

Whatever the causes, the result has been the necessity for a vast amount of repetition in analytical work, and it behooves the present generation of chemists to heed well the warning and to work with a two-fold purpose in view, that of lightening the labors of those who come after us by enabling them to use our work with less supplementary examination, and of thereby enhancing our own reputations by meriting encomiums on work that has stood the test of time.

This monition applies to the lithologist as well as to the chemist, and the former should seek to make or have his analyses made as complete as possible, and not, as is so often the case, be content with determinations of silica, alumina, the oxides of iron, lime, magnesia, the alkalies, and water; even going so far sometimes as to ask the chemist to omit tests for other constituents that may be present in small quantity for the sake of getting a greater number of more or less incomplete analyses accomplished. The latter, it is true, may serve the immediate purpose for which they were intended, but their incompleteness may on the other hand not only conceal points fruitful of suggestion to the attentive mind, but, what is of still greater importance, they may be actually misleading. Enough instances of totally inaccurate conclusions to be drawn from them have fallen under my own observation to fully justify this plea in favor of greater completeness in rock and mineral analyses made for purely scientific purposes.

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[1] Read at the Baltimore Meeting December 27, 1893.

This is shown by the difference between the following analyses. The specimens were taken and analyzed at widely separated times and by different persons, it is true, but they were unquestionably from the same rock mass in which, however much the relative proportions of the different mineral constituents might vary within certain limits, there can be no reason to doubt the general distribution of all the elements shown by the second analysis.

	Earlier Analysis	Later Analysis
SiO_2	54.42	53.70
TiO_2	....	1.92
Al_2O_3	13.37	11.16
Cr_2O_3	....	0.04
Fe_2O_3	0.61*	3.10*
FeO	3.52*	1.21*
MnO	....	0.04
CaO	4.38	3.46
SrO	....	0.19
BaO	....	0.62
MgO	6.37	6.44
K_2O	10.73	11.16
Na_2O	1.60	1.67
Li_2O	trace	trace
H_2O below 110°C	....	0.80
H_2O above 110°C	2.76**	2.61
CO_2	1.82	....
P_2O_5	....	1.75
SO_3	....	0.06
F	....	0.44
Cl	....	0.03
	99.58	100.40
Less O for Fl		.19
		100.21

Another instance of a similar kind is given below. Here again certain differences are explainable by natural variations

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* From the fact that repeated determinations of the iron oxides in this and related rocks from the same region show always a great preponderance of ferric oxide, it is not improbable that the figures given for the two oxides in the first analysis were accidentally transposed.

** In the published analysis it does not appear whether this is total water, or, as seems probable, only that remaining above 100°C.

in the proportions of the constituent minerals, but it can hardly be doubted that TiO_2, BaO, SrO, P_2O_5, and SO_3, were present in both specimens in approximately the same amounts. In the earlier analysis in this case determinations of some supposed unimportant constituents were purposely omitted or only made qualitatively, with results which cannot be otherwise than fatal to a full comprehension of the mineralogical nature of the rock.

	Earlier Analysis	Later Analysis
SiO_2	44.31	44.65
TiO_2	not est.	0.95
Al_2O_3	17.20	13.87
Fe_2O_3	4.64	6.06
FeO	3.73	2.94
MnO	0.10	0.17
CaO	10.40	9.57
SrO	....	0.37*
BaO	....	0.76
MgO	6.57	5.15
K_2O	3.64	4.40
Na_2O	4.45	5.67
Li_2O	....	trace
H_2O below 110° C	0.77	0.05
H_2O above 110° C	....	2.10
H_2O by ignition	3.30	....
CO_2	....	0.11
P_2O_5	....	1.50
Cl	....	trace
SO_3	....	0.61
	99.11	99.92

While strongly upholding the necessity for more thorough work, necessarily somewhat at the expense of quantity, it is far from my intention to demand that an amount of time altogether disproportionate to the immediate objects to be sought should be expended on every analysis. But I do maintain that in general the constituents which are likely to be present in sufficient amount to admit of determination in the weight of sample usually taken for analysis—say 1 gram for SiO_2, Al_2O_3, etc., to 2 grams for certain other constituents—should be sought for, qualitatively at least, in the ordinary course of quantitative

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* Not entirely free from CaO.

work, and their presence or absence noted among the results. If present in little more than traces that knowledge alone may suffice, for it is often more important to know whether or not an element is present than to be able to say that it is there in amount of exactly 0.02 or 0.06 per cent. In the tabulation of analyses a special note should be made in case of intentional or accidental neglect to look for substances which it is known are likely to be present. Failure to do this may subject the analyst to unfavorable criticism when at some future time his work is reviewed and the omissions are discovered by new analyses.

Finally, whenever possible, a thorough microscopical examination of the rock in thin section should precede the chemical analysis. This may be of the greatest aid to the chemist in indicating the presence of unusual constituents or of more than customary amounts of certain constituents, whereby, possibly, necessary modifications in the analytical procedure may be employed without waste of time or labor.

ELECTROLYTIC SEPARATIONS.

BY EDGAR F. SMITH.

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FREUDENBERG published an article recently entitled "Über die Bedeutung der elektromotorische Kraft für elektrolytische Metalltrennungen" (*Zeit. für phys. Chemie.*, **12**, 97), in which are facts of great importance to all interested in the determination and separation of metals in the electro-chemical way. Some statements, however, have been made by Freudenberg to which I feel it my duty to reply. I discover upon p. 116, for example, these lines: "Auch gelingt eine Trennung (Kupfer) von Kadmium, welche bisher nur bei Gegenwart von Saltpetersäure möglich schien, vortrefflich, wenn man die Lösung mit 10–20 cc. verdünnter Schwefelsäure versetzt und mit .einer Spannung von 2 Volt elektrolysiert. Das Kupfer wird rasch und völlig kadmium-freigefällt." This very separation was carried out successfully three years ago by Smith and Frankel (*Am. Chem. J.*, **12**, 104–112 and *Ber. d. Chem. Ges.*, **23**, *Ref.* 413) as will be observed from the literature references and the examples that follow: