

NOTES ON THE ANALYSIS OF "IRON-STONE" BY HUBERT  
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Iron-stone is the name which is applied in popular usage to the rock which is at present finding considerable application as a building material in and about Halifax. For this purpose it has several advantages, and some disadvantages. On account of its structure and the presence of well-developed joint-planes, it is quarried into rectangular blocks with a fair degree of ease. The flat surfaces of the planes allow of its being built into a smooth wall, and the familiar iron-rust stains, where the rock has been exposed to the weather allow of artistic effects being produced by placing the colored blocks in symmetrical positions.

A decided disadvantage, however, lies in the difficulty which has been experienced in finding a cement which sticks closely enough to the stone to prevent seepage of water through the masonry, thus producing unsightly stains on the interior wall. This difficulty is being obviated, it is hoped completely, in the case of the new Science Building at Studley by building the wall double, using iron-stone outside and granite as a lining, for ordinary cement forms with granite a weather-proof wall.

From the geological standpoint, iron-stone is metamorphosed shale of great age, belonging as it does to the Pre-Cambrian period. In the course of geological time it has become greatly changed so that the original shale has now become a hard slate. Several influences have combined to produce this result. It has been very severely folded, in some places even crumpled, so that the originally flat-lying beds are now found in all attitudes, even approaching the per-

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pendicular, and the great folds may be traced all over the country wherever exposures occur.

Another important metamorphosing influence was the intrusion of great masses of granite in Devonian time. The effect of this was to harden the slate by baking it and to produce many minerals which give a characteristic spotted appearance as the contact with the granite is approached.

It is this hardened slate from near the contact which has received the name of iron-stone, and which is used for building purposes. Microscopic examinations of a series of sections of the slate taken from the proximity of the granite contact made last year by Prof. D. S. MacIntosh, gave evidence that the folding took place prior to the intrusion and also showed the development of a series of minerals, such as cordierite, in small ovoid patches found even at considerable distance from the contact, slender crystals of andalusite found nearer and also several other less prominent minerals, such as biotite, sericite and muscovite. The small amount of carbonaceous material in the original shale was changed to graphite and feldspar and quartz particles are found. Up to the present no fossils have been discovered, a circumstance which points alike to its great age and the severe metamorphosis that it has undergone.

As found in most localities, it is a hard dark grey rock, characterized by red stains of ferric oxide where it has been exposed to the action of the weather, of a rather homogeneous structure and stony appearance. Bands are found which contain so much silica and are so hard that they exhibit the phenomenon of conchoidal fracture. As the granite contact is approached the slate changes its appearance slightly and becomes what is known as spotted slate due to the development of minerals. Patches and small crystals of pyrites are common and under the influence of the weather, some of the minerals have dissolved out, giving a pitted surface.

The specimen used in the following analysis was procured from the grounds at Studley. It was quarried from across the North West Arm, near Halifax, in the old King's Quarry, a few hundred yards from the contact with the granite. It was slightly mineralised but care was taken to select a specimen which had not been weathered to a sufficient extent as to become stained. By the courtesy of the Technical College it was reduced to a very fine flour in their machines, and was then very carefully mixed. The actual specimen analyzed was obtained by selecting small portions from all parts of the mass, and these portions were again ground in an agate mortar until no grit was perceptible. A weighed-out portion of this was taken and thoroughly mixed with about eight parts of sodium carbonate, placed in a platinum crucible, and two more parts of sodium carbonate placed on top.

The crucible was heated gently at first and then more violently until a clear fusion took place, the crucible being meanwhile loosely covered. On cooling, the crucible and its cover were placed in a solution of 50° hydrochloric acid in 100<sup>cc</sup> of water and allowed to thoroughly disintegrate. The residue of silica was filtered off and washed, and the filtrate evaporated to dryness and dehydrated on the water-bath for two hours, the residue moistened with concentrated hydrochloric acid, and dissolved in about 150<sup>cc</sup> water, filtered and washed.

A second evaporation and dehydration was found necessary in only a few cases. The silica was carefully ignited to constant weight. It was then treated with a few drops of concentrated sulphuric acid and hydrofluoric acid and warmed to expel the silicon tetrafluoride and finally the sulphuric acid and the crucible again weighed. The difference of these two weighings gave the silica contents of the iron-stone which was found to be 58.05%. The residue in the crucible, which was chiefly iron and aluminum oxide, amounted to .0140 g. and as an even gram portion had been taken

this reads 1.40% which will be added to the determination of iron and aluminium oxides.

The filtrates from the silica were combined and the iron and aluminium contained determined in the following way:—

A few drops of bromine water were added and the solution boiled to oxidize any ferrous iron present, and a considerable volume of ammonium chloride added and the solution made slightly alkaline with ammonium hydroxide. The precipitated hydroxides were filtered off and washed carefully, dried in the oven and ignited to constant weight. This with the weight of the iron and alumina carried down by the silica gave a weight of .3225 g. or 32.25% of combined iron and aluminium oxides, assuming the iron to be all in the ferric condition.

The iron content was determined by preparing another sample as before, removing the silica and making the solution up to standard volume. Two accurate fractions were then taken and one titrated direct with standard permanganate solution to obtain the proportion of ferrous iron. This process is open to some objection as it is very difficult to ensure that no oxidation of the ferrous iron should take place during the processes of grinding the sample and of fusion, and the proportion calculated from it can only be approximately correct. The other fraction was evaporated with sulphuric acid and the hydrochloric acid expelled. It was then reduced by means of a Jones reductor and titrated with standard permanganate. Thus the ratio of the ferrous iron to total iron was established and from it the proportions of the two oxides calculated, proving to be ferrous oxide 1.72%, ferric oxide 4.51%—total iron 6.23%.

The calcium and magnesium were determined by the Richards method. This method is based upon the fact that the amount of magnesium occluded by the calcium oxalate precipitate. depends upon the concentration of magnesium

molecules present. Hence the ionization of the magnesium is favoured in every way possible.

In detail the procedure consists in diluting the filtrate from the iron and aluminium to about 500<sup>cc</sup> and adding ammonium chloride in some quantity, and to precipitate the calcium a boiling solution of oxalic acid is added, which contains three or four equivalents of mineral acid. A few drops of methyl orange are added and the solution neutralized very slowly (at least half an hour for complete neutralization). Considerable excess of hot ammonium oxalate is added and the solution allowed to stand some hours and the calcium oxalate filtered off and washed chloride free with a one per cent. solution of ammonium oxalate. The precipitate on careful ignition yielded decimal 67% of calcium oxide.

The magnesium was determined by evaporating the filtrate until salts began to crystalize out and then diluting until these just dissolved. Then exactly one-third volume of strong ammonia was added and an excess of sodium hydrogen phosphate. The precipitate formed is magnesium ammonium phosphate and this on ignition yields magnesium pyrophosphate from which the magnesium oxide content was calculated, proving to be 1.18%.

Sulphur was determined by taking fresh samples of iron-stone and fusing with ten parts of a mixture of four parts sodium carbonate with one of potassium nitrate. The samples were very carefully mixed and fused by heating the crucible placed in a hole in an asbestos board to deflect any gases from the flame which might contain sulphur. When all action had apparently ceased the residue was cooled and repeatedly extracted with boiling water and finally with boiling dilute sodium carbonate solution. The solution thus obtained was made acid, evaporated and the silica dehydrated and the nitric acid expelled by moistening with hydrochloric acid, and again evaporating. The silica was then filtered off and

the filtrate diluted to 400<sup>cc</sup> brought to boiling and hot barium chloride solution added very slowly in slight excess and the solution allowed to digest for some hours. From the precipitate of barium sulphate the sulphur content was calculated and found to be 1.67%.

The water contained in combination was estimated by igniting a fresh sample for some hours in a covered crucible to constant weight. This was found to give a proportion of volatile matter of 6.49%. As this, however, probably includes the sulphur a determination of the sulphur contained in the residue was made showing that over two-thirds of the total sulphur had been driven off. This weight subtracted from the total loss of weight on ignition gave the loss due to the volatilization of the water which was found to amount to 5.23%.

These results may be summarized as follows:—

“IRON-STONE.”

Silica.....	58.05	
Combined Oxides: Alumina	26.02	
Ferrous Oxide	1.72	
Ferric Oxide	4.51	32.25
Calcium Oxide.....		.67
Magnesium Oxide.....		1.18
Sulphur.....		1.67
Water.....		5.23
		—99.05

A partial analysis of another sample of iron-stone was made, the specimen being taken from a rather silicious band very near the granite contact and with the minerals quite highly developed. The results were:—

Silica.....	64.48
Combined Oxides.....	26.06
Sulphur.....	.32
Magnesium Oxide.....	1.56
Volatile matter, including water and probably some sulphur.....	2.68

These figures show that considerable variation in composition may occur and the low proportion of volatile matter and sulphur and the high silica are significant of the effect of the granite intrusion upon the slate in its immediate vicinity.

Although the microscope reveals the presence of feldspar, it is in such small quantities that no effort was made to estimate the alkalis.

The following tables give the analyses of typical slates.

A black roofing slate from Vermont gave:\*

Silica.....	59.7
Combined Oxides: Alumina	17.0
Ferrous Oxide	4.9
Ferric Oxide..	.5... 22.4
Magnesium Oxide.....	3.2
Calcium Oxide.....	1.3
Alkalis.....	5.2
Water.....	4.1
Other Oxides and Carbon.....	4.3
	—100.3

A slate from Wales gave:

Silica.....	60.5
Combined Oxides: Alumina	19.7
Ferrous Oxide.	7.8... 27.5
Magnesium Oxide.....	2.2
Calcium Oxide.....	1.1
Akalis.....	5.4
Water.....	3.5
	—100.2

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\* Pirsson's Rocks and Rock-Minerals, page —