

## SOME HYPERSTHENES FROM SOUTHEASTERN PENNSYLVANIA AND DELAWARE\*

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### ABSTRACT

A new procedure is proposed for the chemical analysis of minerals high in iron, magnesium and silica; and low in calcium, aluminum and the alkalis. Chemical, optical, *x*-ray and spectrographic analyses of 8 high iron hypersthene from Southeastern Pennsylvania and Delaware are presented. Curves are given showing the variation of the optical properties with chemical composition.

### INTRODUCTION

In general, although optical properties of minerals depend on their chemical composition, mineralogists have usually been content to put all their effort into obtaining the optical properties while letting someone else make a mediocre chemical analysis of the mineral. While it is true that the mineralogist, in most cases, cannot be expected to do the analysis himself, he should see that it is done well.

A recent investigation (1, 2), as to the reliability of results of analysis of silicate rocks, has shown that even when performed by experts, the results obtained varied widely. This is no reflection on the analysts, but only proves that silicate analyses are difficult.

In the case of the analysis of a mineral, another complication arises. Most laboratories simply follow Washington's method of analysis (3). Yet the composition of the mineral being analyzed is usually completely different from that of a rock for which the method was designed. For instances, in a high iron hypersthene, very little calcium, aluminum, sodium and potassium are present, while rocks contain relatively large amounts of all four. Magnesium and ferrous iron amount to about 50 per cent of the total in a hypersthene, whereas these same elements are generally contained in very small amounts in a rock. Consequently, the usual method of rock analysis should not be used and a new method devised.

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This paper proposes a method for the analysis of high iron hypersthene and any similar material. In the same category might be included the entire enstatite-hypersthene series, the anthophyllites and the olivines; that is, any mineral containing high iron or magnesium, high silica, and very low calcium, aluminum and alkalis.

Previous work on the analysis of minerals has usually been done on crystals which were large enough to be hand picked and separated. Except for the work of Hess and Phillips (4) and Hess (5), very little attention has been given to minerals from fine grained rocks. This has been due to the difficulty of separating the minerals from the rock. However, with the advent of the Franz isodynamic magnetic separator, fine grained rocks can be separated into their components fairly easily. These fine grained minerals give a truer picture of the relation of the mineral to the rock than large size crystals which had to form under special conditions. At the same time, a much larger amount of pure mineral can be obtained, allowing the analyst much more freedom in his work.

Another fault of many analyses of minerals has been the lack of purity of the material analyzed. While optical data can be obtained on impure material simply by picking out the correct grains to measure, the chemical analysis should be made on a sample having a purity of over 99 per cent. Otherwise, the chemical analysis can have little meaning, since the effect of the impurities on the analysis can not be determined. Yet many curves have been drawn using analyses of impure minerals. It is paradoxical that mineralogists will refuse to accept a chemical analysis that is over one half per cent in error and yet submit for the analysis a mineral with 5 per cent impurity. If the mineralogist demands a 99 per cent accurate analysis, he should submit a 99 per cent pure sample.

This study has been concerned with the separation and analysis of high iron hypersthene from a series of rocks found in southeastern Pennsylvania and Delaware. The rock sites concerned are the Wilmington gabbro massif, the Baltimore gneiss complex and ultrabasic rocks associated with them and with the Wissahickon schist. This is part of a major study of the rocks and their relationship throughout this region, being carried out by the Geology Department of Bryn Mawr College. At the time of the separation of the hypersthene a series of hornblendes from the same and associated rocks were separated and analyzed. This work will be reported in a separate paper by Abraham Rosenzweig of Bryn Mawr College.

In every case, the material taken for analysis was obtained in a purity of over 99.5 per cent. No correction was made for the impurity, since this degree of purity is as good as the accuracy of the chemical analysis. The purification was accomplished by means of a Franz isodynamic magnetic

separator and heavy liquids. Enough material was obtained in pure form to carry out complete chemical and optical analyses on each sample. In no case was a single crystal analyzed, but the material was always obtained from relatively fine grained rocks.

Much work has been done previously on this series of minerals, notably by Winchell, Walls, Henry and Poldervaart; relating the chemical composition to the optical properties. However, there are questionable points in all of this work. The first curves drawn for the hypersthene were by Winchell in 1923 (6), and were used as standards for years when published in his text (7). However, because of the lack of complete data and the possible inaccuracy of some analyses, the curves were not too reliable. He made the statement that "the curves were not final and infallible but the best from all available data" (6). The data used was collected from the literature. Practically all the analyses had been made on single crystals, hand picked by the mineralogists.

The next major attempt at drawing curves for this series was done by Walls (8) and Henry (9), working about the same time. They did some original work on material purified by them but the majority of the points on which they based their curves were the same as those used by Winchell and suffered from the same faults. Even the material analyzed by them was not of the highest purity. For this reason Walls states ". . . it is clear that no high degree of accuracy can be claimed for these curves."

The curves obtained by Hess and Phillips (4) were the first based on analyses which had been done with great care, on pure material, and on material from fine grained rocks. Where the material analyzed was impure, the impurity was analyzed and corrections made for it. From this careful work very good curves were obtained. However, no hypersthene containing over 25 per cent ferrous silicate was included.

In 1947 Poldervaart (10) described the analyses of two hypersthene and drew curves based on most of the preceding work. Winchell, in the latest edition of his text (11), has published a new series of curves, for which he gives no reference but which resemble those of Poldervaart. Still, these curves are based on analyses which are not of the highest reliability. Therefore, it was proposed to analyze the pure high iron hypersthene obtained from the fine grained rocks in this region and use this data as a basis for a group of new curves.

#### LOCATION AND DESCRIPTION OF THE HYPERSTHENE-BEARING ROCKS

*Sample 35-5.* This specimen was found 0.1 mile north of the crossroads of U. S. Route 202 at Talleyville, Delaware, 300 yards west of the road (West Chester 92756).\* It is a

\* Note: This system of location is based upon the standard 15-minute quadrangles of the United States Geological Survey, which are subdivided into nine 5-minute sub-quadrangles.

noritic eucrite of medium fine grain. By volume analysis on a thin section it contains: 10 per cent hypersthene, 6 per cent augite, 23 per cent hornblende, 2 per cent magnetite and 59 per cent plagioclase of composition anorthite 72 per cent. There is a trace of apatite.

*Sample 35-6.* This specimen came from an outcrop in the park at Franklin and Sycamore Streets, Wilmington, Delaware (Wilmington 31268). It is a quartz norite: dark, massive and fine-grained. By volume analysis it contains: 11 per cent hypersthene, 7 per cent augite, 14 per cent hornblende, 5 per cent magnetite, 13 per cent quartz and 50 per cent plagioclase of composition anorthite 58 per cent. There is a trace of apatite.

*Sample 35-8.* This specimen was found in an abandoned quarry on the east bank of Brandywine Creek in the city of Wilmington, Delaware (Wilmington 32237). It is a quartz norite, dark and of fine grain and is the prevailing type in the quarry. By volume analysis it contains: 5 per cent hypersthene, 1 per cent augite, 3 per cent magnetite, 27 per cent quartz and 64 per cent plagioclase of composition anorthite 46 per cent. There is a trace of apatite and biotite.

*Sample 35-9.* This specimen was found in the same quarry as sample 35-8 but cross-cutting the prevailing rock as dikes. It is a norite of fine grain, slightly darker than 35-8. By volume analysis it contains: 18 per cent hypersthene, 19 per cent augite, 3 per cent magnetite and 60 per cent plagioclase of composition anorthite 52 per cent. There is a trace of apatite and epidote.

*Sample 35-13.* This sample was found in Radnor quarry, in the lower quarry wall on the west side (Norristown 85826). It is an altered hypersthene gabbro of medium grain. By volume analysis it contains: 14 per cent hypersthene, 14 per cent augite, 4 per cent magnetite, 6 per cent potash feldspar, 7 per cent garnet and 55 per cent plagioclase of anorthite content 41 per cent. There is some anti-perthite and sericite with a little apatite and epidote.

*Sample 35-14.* This specimen was also found in Radnor Quarry (Norristown 85826), but in the northwest corner. It is a hybridized norite, of coarse grain. By volume analysis it contains: 10 per cent hypersthene, 3 per cent magnetite, 26 per cent quartz, 4 per cent potash feldspar and 57 per cent plagioclase of composition anorthite 36 per cent. There is some anti-perthite and sericite and traces of garnet, biotite and apatite.

*Sample 35-19.* This specimen was found in a boulder fence on the south side of Faulk Road, 1.5 miles northeast of the intersection of U. S. Route 202 (West Chester 96413). It is a norite, medium grained and fairly light in color. By volume analysis it contains: 21 per cent hypersthene, 9 per cent augite, 13 per cent hornblende and 57 per cent plagioclase of composition anorthite 69 per cent. There is a trace of magnetite.

*Sample 35-32.* This specimen was found in boulders in a field 800 feet south of the West Chester Pike at the Dinwoody Home (Chester 22476). It is a eucritic norite of coarse grain. By volume analysis it contains: 55 per cent hypersthene, 10 per cent augite, 1 per cent hornblende and 34 per cent plagioclase of composition anorthite 83 per cent. There is a trace of magnetite.

#### PREPARATION AND PURIFICATION OF SAMPLES

The rock was crushed by means of jaw crushers and steel rolls, so that grinding action was avoided as much as possible to prevent oxidation of

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ranges. These sub-quadrangles are number 1 to 9 from left to right. The first number in the designation denotes this 5-minute sub-quadrangle. Each sub-quadrangle is then subdivided into 9 parts similarly, which are denoted by the second number in the designation. These are similarly subdivided and designated, so that with five numbers a location is very accurately placed on the map.

the ferrous iron. The crushed material was screened, and that portion passing through the 100 mesh screen and remaining on the 150 mesh screen was taken for the separation. This material was washed with water to remove adhering fines and dried.

Impure hypersthene was separated from the other minerals in the rock, such as magnetite, augite, hornblende, quartz, and feldspar, etc. by means of a Franz isodynamic magnetic separator. Product purity by this method was between 95 and 99 per cent. The sample was washed with water to remove any fines formed during the separation and then dried. Hypersthene was separated as the heavy fraction with methylene iodide having a specific gravity of 3.3. After separation the grains were washed with acetone. This gave products of over 99.5 per cent purity in all but a few cases where there was garnet present, which had a specific gravity above that of the hypersthene, or augite and/or hornblende, which had specific gravities below that of the hypersthene but above 3.3. In those cases, a rough specific gravity of the material was determined and two separations were made using Clerici solution. The first, with liquids of specific gravity just above that of the hypersthene, in which case the impurities were separated as the heavy fractions. The second, with liquids having a specific gravity just below that of the hypersthene, in which case the impurities were found in the light fractions. After separation, the grains were washed well with hot water to remove the thalious salts and dried at 110° C. In all samples, product purity was above 99.5 per cent.

At all stages during the purification process materials were examined with a binocular microscope. This allowed the examination of a large number of grains at one time. Impurities were seen quite easily because of the definite difference in color, luster and general appearance. The final product was also examined petrographically by making a balsam mount of a random sample.

#### PRINCIPLES OF THE CHEMICAL ANALYSIS

While none of the individual determinations are original, the method used is a combination of known methods best adapted for use with hypersthene and related minerals. Some original innovations have been added. The analysis was carried out on a solution containing known amounts of the elements analyzed for, in the proportion in which they might be encountered. In all cases, satisfactory results were obtained.

One of the major changes in the analysis is the decomposition of the sample with hydrofluoric and perchloric acids, thereby introducing no extraneous sodium salts. At the same time, separate aliquots can be taken of optimum size for each determination. Silica is determined on a small separate sample by dehydration with perchloric acid and the resulting filtrate discarded because of the high concentration of sodium salts introduced by fusion. No attempt is made to separate calcium from the magnesium when analyzing for the latter, because of the high magnesium and low calcium content. Aluminum is determined directly because of its low percentage, and corrected only for titanium, present in even smaller

amounts. The alkalis are determined after a hydrofluoric and perchloric acid decomposition. Before analyzing for chromium colorimetrically, hydrochloric acid is used instead of alcohol to remove manganate interference. Water is determined by volatilization and absorption. The other elements are determined in the usual way.

#### PROCEDURE FOR THE CHEMICAL ANALYSIS

1. *Specific Gravity.* This determination is made first so that the mineral can be recovered and used for subsequent analyses. Dry and weigh a 25 ml. pycnometer containing a thermometer, using a similar bottle as a tare. Fill the pycnometer with water and reweigh, noting the temperature of the water. From this weight and from the density of water at this temperature calculate the volume of the pycnometer. Check this result by weighing the pycnometer filled with water at different temperatures. This gives the weight of water contained in the pycnometer at any temperature.

Weigh 5 grams of the sample into the pycnometer by means of a powder funnel. Cover with about a quarter of an inch of water containing a wetting agent. Place the pycnometer in a desiccator and evacuate carefully with water suction so that none of the sample is lost by violent bubbling. Leave the pycnometer in the desiccator for 15 minutes, then remove, fill with water and allow to come to equilibrium in the balance case. Insert the thermometer, dry the pycnometer with a clean cloth, weigh quickly and note the temperature.

The amount of wetting agent added to the water is about 0.01 per cent, which has no appreciable effect on the density of the water. It aids the determination by preventing the entrapment of air by the sample. Fairly large fragments (about 100 mesh) are preferred for this determination. After use, the sample is washed well, dried and used for the chemical analysis.

2. *Water.* Weigh a 0.5 gram sample into a porcelain boat, place in a combustion tube and heat for 30 minutes at 1000° C., while passing dry air through the tube. Dry the air by passing it through anhydrous calcium chloride and anhydrous magnesium perchlorate. Collect the water in a weighed absorption bulb containing anhydrous magnesium perchlorate. Remove the bulb, place in the balance and reweigh after it reaches equilibrium. Run a blank determination under the same conditions of time and temperature.

3. *Silica.* The method is essentially the same as that of Willard and Cake (12). Fuse a 0.20 to 0.25 gram sample with sodium carbonate in a platinum crucible for 30 minutes. Leach the melt with dilute hydrochloric acid, add perchloric acid and evaporate to fumes. Continue fuming for 30 minutes, cool, dilute with water, filter and wash alternately with hot dilute hydrochloric acid and water until the paper is free of perchloric acid. Evaporate the filtrate to fumes and repeat the process. Combine the filter papers and ignite in a weighed platinum crucible. Cool and reweigh the crucible. Treat the residue in the crucible with a few drops of sulfuric acid and about 10 ml. of hydrofluoric acid, evaporate to dryness, ignite for 15 minutes, cool and reweigh. The loss in weight is equal to the weight of silica in the sample.

4. *Ferrous Iron.* The method of Soule (13) is used for this determination. Weigh a 0.5 gram sample into a round-bottomed flask containing a rubber stopper with an inlet and outlet tubes. Displace the air with a stream of carbon dioxide. Add 5 ml. of 1:1 sulfuric acid, 10 ml. of water and 15 ml. of hydrofluoric acid. Boil the solution gently with a continuous stream of carbon dioxide bubbling through the solution. Continue boiling until the mineral is dissolved completely. Allow to cool for 5 minutes under a stream of carbon dioxide. Add 100 ml. of a saturated solution of boric acid containing 2.5 per cent sulfuric acid. Cool the flask below room temperature and titrate with 0.1 N potassium permanganate solution.

5. *Sodium and Potassium.* The alkali metals are determined according to the method of Marvin and Woolaver (14). Weigh a 0.5 gram sample into a platinum dish and treat with 5 ml. of nitric acid, 10 ml. of perchloric acid and 10 ml. of hydrofluoric acid. Evaporate the solution to half of the volume and add 10 ml. of hydrofluoric acid. Evaporate to fumes of perchloric acid and continue heating for 15 minutes. Cool the solution, transfer with water to a pyrex beaker and evaporate to dryness. Bake the residue on a hot plate until no more fumes of perchloric acid are noted. Heat the sides of the beaker with a bunsen burner to remove the last traces of perchloric acid.

Cool, add 50 ml. of 10 per cent ammonium hydroxide and boil for 10 minutes. Filter and wash with 10 per cent ammonium hydroxide. Heat the filtrate to just below boiling and add 10 ml. of 5 per cent ammonium oxalate. Allow to stand one hour, filter and wash with water. Reheat the filtrate to 60° C. and add 5 drops of a 2 per cent solution of 8-hydroxyquinoline in 1:10 acetic acid. Allow to stand for one hour, filter and wash with water. Acidify the filtrate with hydrochloric acid and evaporate to dryness on the steam bath.

Treat the residue with aqua regia and evaporate to dryness with hydrochloric acid to insure the presence of only chlorides. Dissolve the residue in water and filter into a weighed platinum dish. Evaporate the filtrate to dryness on the steam bath, place in a cold muffle furnace and bring the temperature up to 550° C. slowly. Hold the furnace at that temperature for 30 minutes, remove the dish, cool and weigh. The increase in weight is mixed sodium and potassium chlorides.

The amount of sodium present in the mixed chlorides is determined by the method of Kolthoff and Sandell (15). Dissolve the chlorides in water and transfer to a beaker. Evaporate to dryness on the steam bath, add one ml. of water and 10 ml. of zinc uranyl acetate solution and stir well. Allow to stand one hour and then filter on a weighed porcelain crucible. Wash with small portions of the precipitating agent, five 2 ml. portions of ethyl alcohol saturated with the precipitate and finally with a small amount of acetone. Place the crucible in the balance case for 15 minutes and reweigh. The increase in weight is sodium zinc uranyl acetate with six molecules of water of crystallization and is calculated to an equivalent weight of sodium chloride which is subtracted from the weight of the mixed chlorides. The per cent of sodium and potassium oxides is then calculated. A blank determination is made using the same reagents.

6. *Chromium.* Mix a 0.1 gram sample with 3 grams of sodium carbonate in a platinum crucible and fuse for 30 minutes. Cool and leach with hot water. Filter the residue and wash with hot water containing a little sodium carbonate. Add 6 ml. of 1:1 sulfuric acid which will give the correct acidity (0.2N) on final dilution. Add 5 drops of concentrated hydrochloric acid and heat on the steam bath for an hour to reduce any manganate which is present. Cool and transfer to a 100 ml. volumetric flask. Add 2 ml. of a 0.25 per cent alcoholic diphenylcarbazide solution and dilute to the mark with water. Compare the intensity of the color obtained with a standard solution of potassium dichromate treated in the same way, using a colorimeter with a green filter.

7. *Decomposition of the Sample for the Determination of Aluminum, Ferric Iron, Titanium Manganese, Calcium, Magnesium and Nickel.*

Weigh a 2 gram sample into a platinum dish and add 10 ml. of perchloric acid, 5 ml. of nitric acid and 10 ml. of hydrofluoric acid. Heat the dish on the hot plate until the solution has decreased to half the volume, add 10 ml. of hydrofluoric acid and then evaporate almost to dryness. Cool, take up with water and transfer to a beaker. To assure recovery of all the salts, add 5 ml. of 1:1 hydrochloric acid to the dish, heat and transfer to the same beaker. Repeat this treatment 3 times or until all the salts are transferred to the beaker. Dilute to about 200 ml. and boil until all the salts are dissolved. Cool, add 20 ml. of hydrochloric acid, transfer with water to a 500 ml. volumetric flask and dilute to the mark.

8. *Ferric Iron.* Pipet a 200 ml. aliquot into a beaker and evaporate to 25 ml. Add 15 per cent stannous chloride dropwise to the hot solution until the yellow color of ferric chloride disappears and then add 2 drops in excess. Cool and add rapidly to the solution 10 ml. of a saturated mercuric chloride solution. Dilute to about 250 ml., add 10 ml. of phosphoric acid and titrate with 0.1 N potassium dichromate using diphenylamine sulfonic acid as indicator.

The total amount of iron present is calculated as ferric oxide. From this is subtracted the amount of iron known to be present in the ferrous state. This gives the amount of ferric oxide actually present in the sample.

9. *Aluminum and Titanium.* The method used for the determination of these elements is that given by Willard and Diehl (16). Pipet a 100 ml. aliquot from the original solution into a beaker, dilute to 400 ml. and add 3 grams of diammonium hydrogen phosphate. Neutralize most of the free acid with ammonium hydroxide and then add just enough to give a faint permanent precipitate. Add 2 ml. of concentrated hydrochloric acid and stir the solution until it becomes clear. Add 20 ml. of water containing 15 grams of sodium thio-sulfate and heat to boiling. Add 8 ml. of glacial acetic acid and 5 grams of ammonium acetate and boil vigorously for 30 minutes. Filter while the solution is still hot and wash with water.

Transfer the filter paper to the original beaker and add 10 ml. of hydrochloric acid and 50 ml. of water. Boil for a few minutes to dissolve the precipitate, filter and wash with 1:10 hydrochloric acid. Dilute the filtrate to 300 ml. and precipitate as before. Filter and wash this precipitate with water. Place the filter paper containing the precipitate in a weighed porcelain crucible, ignite and reweigh the crucible. The increase in weight is due to combined aluminum and titanium phosphates.

Add 3 to 4 grams of potassium pyrosulfate to the residue and fuse until it has dissolved completely. Cool and leach with 50 ml. of 1:1 sulfuric acid. Transfer with water to a 100 ml. volumetric flask, add 5 ml. of hydrogen peroxide (3 per cent) and dilute to the mark. Compare the intensity of the color formed with that of a standard titanium solution treated in the same way, using a colorimeter with a blue filter.

The amount of titanium oxide present in the sample is calculated, converted to titanium phosphate and subtracted from the weight of combined phosphates above. The remainder is aluminum phosphate, which is calculated to aluminum oxide.

10. *Calcium.* The method used for calcium is a modification of that given by Caley and Elving (17). Pipet a 100 ml. aliquot into a beaker, add 10 ml. of perchloric acid and 5 ml. of nitric acid and evaporate to dryness. Bake the residue on the hot plate and heat the beaker with the direct flame of a Bunsen burner to assure the removal of all the perchloric acid. Cool, add 200 ml. of water and boil for 15 minutes. Filter through a fine filter paper and evaporate the filtrate to dryness. Add 9 ml. of water, 10 drops of 1:1 sulfuric acid and 100 ml. of methyl alcohol. Allow to stand overnight and filter through a weighed fine porcelain filtering crucible. Wash with 90 per cent methyl alcohol, dry the precipitate for 30 minutes at 100° and ignite for 30 minutes above 450° C. Cool and weigh as calcium sulfate.

11. *Magnesium.* Pipet a 25 ml. aliquot into a beaker and dilute to 200 ml. Add 2 grams of ammonium chloride, heat the solution almost to boiling and add ammonium hydroxide until it is just alkaline to litmus. Add 5 ml. of saturated bromine water, test the solution to make sure it is still ammoniacal and then boil for 15 minutes. Filter the precipitate while the solution is still hot, wash well with water, dissolve it off the paper with hot dilute hydrochloric acid and reprecipitate as above. Combine the two filtrates, make just acid with hydrochloric acid and add 0.5 gram of diammonium hydrogen phosphate. Cool below room temperature, add ammonium hydroxide dropwise until the solution is just basic, allow the



calcium and magnesium to precipitate and then add an excess of 30 ml. of ammonium hydroxide. Stir the solution occasionally during the next 30 minutes and allow to stand overnight. Filter the precipitate through paper, wash with 1:20 ammonium hydroxide, dissolve off the paper with 1:10 hydrochloric acid and reprecipitate as above. Filter this precipitate through a weighed porcelain filtering crucible, wash with 1:20 ammonium hydroxide, dry and ignite for one hour. Cool and reweigh as combined magnesium pyrophosphate and calcium orthophosphate. The amount of calcium phosphate known to be present is subtracted and the percentage of magnesium oxide calculated.

12. *Manganese.* The method recommended by Willard and Greathouse (18) is used for the determination of manganese. Pipet a 25 ml. aliquot into a beaker, add 10 ml. of nitric acid and evaporate almost to dryness to expel the hydrochloric acid. Repeat until all the hydrochloric acid is removed. Add 10 ml. of nitric acid, 25 ml. of water and 5 ml. of phosphoric acid and heat the solution to boiling. Add 0.5 gram of potassium periodate, boil until the color of permanganate appears and continue boiling for a few minutes. Cool and dilute to the mark with water in a 100 ml. volumetric flask. Compare the intensity of the color with that of a standard manganese solution treated in the same way, using a colorimeter with a green filter.

13. *Nickel.* The method used is that given by Sandell (19). Pipet a 25 ml. aliquot into a 100 ml. volumetric flask and add 5 ml. of 10 per cent citric acid and 2 ml. of saturated bromine water. Neutralize the solution with ammonium hydroxide and add 3 or 4 drops in excess. Add 1 ml. of a 1 per cent alcoholic dimethylglyoxime solution and dilute to the mark with water. Compare the intensity of the color formed with that of a standard nickel solution treated in the same way and containing an equivalent amount of iron as known to be present in the sample. The comparison is made by means of a colorimeter using a green filter.

## DETERMINATION OF OPTICAL PROPERTIES

### 1. *Volume Analysis of the Rock*

The volume analysis of the rock was made on a thin section using a Wentworth integrating stage. Five traverses were made perpendicular to any foliation, if present, and these results were averaged.

### 2. *Anorthite Content of the Plagioclase*

The anorthite content of the plagioclase in the rock was determined by measuring the indices of refraction on cleavage flakes by the immersion method and applying the Tsuboi curves (20).

### 3. *Optic Angle of the Hypersthene*

The optic angle of the hypersthene was determined on the universal stage in white light. The measurements were made on a balsam mount of the purified grains and on a thin section of the original rock. The average of 10 to 15 determinations was taken. A correction was applied for the difference between the beta index of the mineral and the index of refraction of the hemisphere used, which was 1.649.

#### 4. *Indices of Refraction of the Hypersthene*

The indices of refraction of the hypersthene were determined by the immersion method in sodium light. At least ten grains were checked for each index. The gamma index was measured on grains showing a centered acute bisectrix figure. The beta index was measured on both a centered acute bisectrix figure and an optic eye figure. The alpha index was measured on a centered obtuse bisectrix figure and a flash figure. Orientation of the grains was helped by the marked pleochroism of the hypersthenes.

The liquids used were measured on a Pulfrich refractometer, with corrections made for temperature differences. The liquids differed by 0.0020 to 0.0028 so that the indices of refraction of the minerals could be estimated to 0.001.

#### X-RAY AND SPECTROGRAPHIC DATA

The x-ray photographs were taken on finely ground powders using both chromium and copper targets. The photographs obtained were then measured and compared to data in the literature.

For the spectrographic determination, an ARL-Dietert 1.5 meter grating instrument was used. The samples were burned in an A.C. arc and the lines obtained compared to standard lines. No attempt at quantitative measurement was made.

#### DISCUSSION OF RESULTS

The analyses of the hypersthenes are listed in Table 1. All chemical analyses are averages of duplicate determinations with the greatest error of the duplicates being 0.5 per cent from the mean.

The limits of error in the specific gravity determinations are plus or minus 0.005. The optic angle determinations are correct to within 2 degrees and the index of refraction determinations to within 0.001.

The molecular percentage of ferrous metasilicate in each hypersthene was calculated by adding the number of moles of manganese and ferrous iron and dividing them by the total number of moles of magnesium, manganese and ferrous iron. No other elements were included. This method of calculating the percentage of ferrous silicate was decided upon as being the best and follows the method used by Henry (9) and by Hess and Phillips (4).

There are a few facts of interest which are not included in the tables. The strength of the pleochroism of the hypersthenes does not have any relation to the chemical composition. The pleochroism is red in the X direction, yellow-brown in the Y direction and green in the Z direction.

TABLE 1. ANALYTICAL DATA

|                                | 35-5  | 35-6  | 35-8   | 35-9   |
|--------------------------------|-------|-------|--------|--------|
| SiO <sub>2</sub>               | 50.70 | 50.60 | 51.43  | 52.00  |
| Al <sub>2</sub> O <sub>3</sub> | 0.05  | 0.04  | 0.06   | 0.57   |
| Fe <sub>2</sub> O <sub>3</sub> | 1.18  | 1.44  | 1.39   | 1.34   |
| FeO                            | 25.65 | 26.02 | 23.60  | 22.46  |
| MgO                            | 20.29 | 19.49 | 20.69  | 21.62  |
| CaO                            | 0.66  | 0.87  | 1.02   | 0.75   |
| Na <sub>2</sub> O              | 0.06  | 0.07  | 0.46   | 0.46   |
| K <sub>2</sub> O               | 0.05  | 0.06  | 0.06   | 0.07   |
| H <sub>2</sub> O               | 0.22  | 0.28  | 0.32   | 0.28   |
| TiO <sub>2</sub>               | 0.17  | 0.17  | 0.18   | 0.14   |
| Cr <sub>2</sub> O <sub>3</sub> | 0.00  | 0.004 | 0.00   | 0.003  |
| NiO                            | 0.00  | 0.00  | 0.00   | 0.00   |
| MnO                            | 0.75  | 0.95  | 0.73   | 0.57   |
| Total                          | 99.78 | 99.99 | 99.94  | 100.26 |
| % FeSiO <sub>3</sub>           | 42.2  | 43.7  | 39.8   | 37.5   |
| Sp. Gr.                        | 3.557 | 3.560 | 3.512  | 3.510  |
| 2V                             | -52   | -51   | -55    | -57    |
| alpha                          | 1.702 | 1.703 | 1.699  | 1.695  |
| beta                           | 1.711 | 1.713 | 1.708  | 1.705  |
| gamma                          | 1.716 | 1.718 | 1.713  | 1.710  |
|                                | 35-13 | 35-14 | 35-19  | 35-32  |
| SiO <sub>2</sub>               | 51.18 | 50.60 | 52.80  | 52.60  |
| Al <sub>2</sub> O <sub>3</sub> | 0.15  | 0.16  | 0.00   | 0.12   |
| Fe <sub>2</sub> O <sub>3</sub> | 1.85  | 0.97  | 1.67   | 0.95   |
| FeO                            | 25.16 | 25.71 | 18.65  | 17.14  |
| MgO                            | 18.00 | 18.96 | 24.95  | 25.65  |
| CaO                            | 1.87  | 1.65  | 1.05   | 2.17   |
| Na <sub>2</sub> O              | 0.42  | 0.07  | 0.44   | 0.88   |
| K <sub>2</sub> O               | 0.25  | 0.60  | 0.01   | 0.00   |
| H <sub>2</sub> O               | 0.28  | 0.44  | 0.14   | 0.14   |
| TiO <sub>2</sub>               | 0.25  | 0.19  | 0.09   | 0.15   |
| Cr <sub>2</sub> O <sub>3</sub> | 0.02  | 0.10  | 0.02   | 0.06   |
| NiO                            | 0.00  | 0.00  | 0.01   | 0.05   |
| MnO                            | 0.38  | 0.31  | 0.43   | 0.41   |
| Total                          | 99.81 | 99.76 | 100.26 | 100.32 |
| % FeSiO <sub>3</sub>           | 44.3  | 43.5  | 30.0   | 27.8   |
| Sp. Gr.                        | 3.536 | 3.530 | 3.463  | 3.431  |
| 2V                             | -51   | -53   | -64    | -69    |
| alpha                          | 1.703 | 1.703 | 1.685  | 1.685  |
| beta                           | 1.713 | 1.713 | 1.695  | 1.695  |
| gamma                          | 1.717 | 1.717 | 1.700  | 1.700  |

The most strongly pleochroic of all the hypersthene is sample 35-9, which has a lower ferrous silicate content than most of the others. Both samples 35-14 and 35-19 show weak pleochroism. This is surprising since sample 35-14 has the highest ferrous silicate content of all the samples. All the others show medium pleochroism.

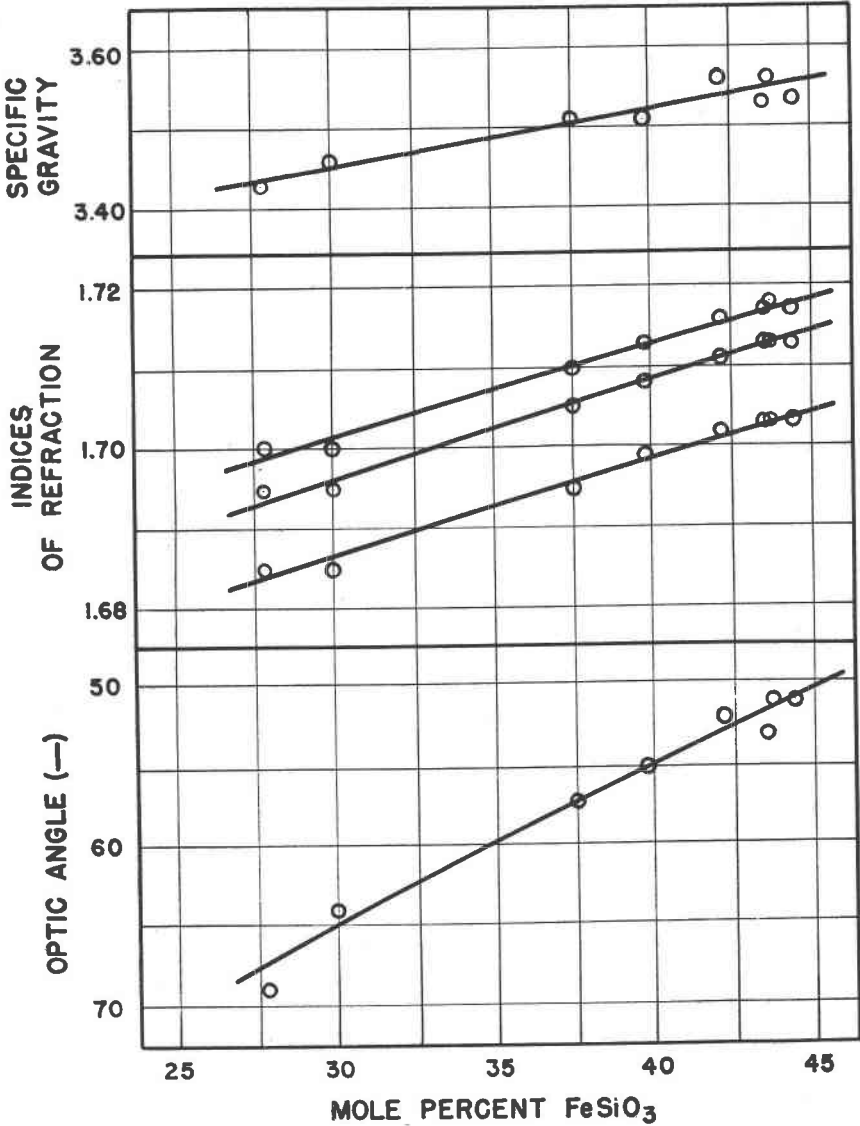


FIG. 1. Curves for optical properties and specific gravity of hypersthene.

The colors of the minerals by reflected light are as expected. That is, those with high iron content are dark brown and those with low iron content are light brown. There is one exception and that is sample 35-32. It has the lowest iron content of all but its color is darker than any except samples 35-13 and 35-14, which have a very high iron content.

The variation of the optic angle, indices of refraction and specific gravity with chemical composition of the hypersthene is plotted on Fig. 1. Within the limits of error, the points fall very well on the curves.

The curves do not agree exactly with any previous ones published for this series. However, the shapes of the curves are similar and it may be that the only difference is one of chemical composition. If the curves drawn by Hess and Phillips (4) were moved by an increase of three mole per cent ferrous silicate, they would correspond to the ones presented here. If those of Henry (9) were moved by a decrease of 6 mole per cent ferrous silicate, they would correspond. Except for the optic angle curve, those of Poldervaart (10) would correspond if moved by an increase of 3 per cent. So it seems that the controversy is concerned with the chemical analyses. It is felt that the previous analytical methods may have been in error enough to cause the curves to differ as they do.

The curves published in Winchell's latest text (11) are hard to interpret. They do not show the regularity in respect to the present curves, as do the other curves. The optic angle curve would have to be moved by a decrease of 9 mole per cent, the index of refraction curve by an increase of one mole per cent, and the specific gravity curve by a decrease of 4 mole per cent. However, since there is no reference as to the source of these curves, no further comment can be made.

In Table 2 are listed the interplanar distances (in Angstrom units) of the strongest reflections as obtained with these hypersthene and those listed in the American Society for Testing Materials card index, 1945 supplement, for hypersthene, II-1133, and for enstatite, II-1186.

TABLE 2. X-RAY DATA

| <i>Hypersthene</i><br>Collection 35 | <i>Hypersthene</i><br>II-1133 | <i>Enstatite</i><br>II-1186 |
|-------------------------------------|-------------------------------|-----------------------------|
| 3.14 (10)                           | 3.20 (10)                     | 3.16 (10)                   |
| 1.47 (8)                            | 1.49 (8)                      | 1.48 (6)                    |
| 2.86 (6)                            | 2.89 (8)                      | 2.85 (6)                    |
| 2.49 (6)                            | 2.48 (5)                      | 2.53 (6)                    |
| 2.07 (6)                            | 2.11 (5)                      | 2.11 (6)                    |
| 1.59 (3)                            | 1.60 (6)                      | 1.60 (5)                    |
| 1.38 (3)                            | 1.39 (6)                      | 1.39 (5)                    |
| 1.29 (3)                            | 1.30 (5)                      | 1.29 (3)                    |

In parentheses are the relative intensities of the reflections. It can be seen that the values obtained for these samples check very well with the accepted ones for hypersthene and enstatite.

Within the limits of error of measurement of the lines, there is no difference in the spacing between the hypersthene with the lowest iron content and that with the highest. There was a distinct decrease in the intensity of the reflections from the minerals with the most iron. This was not due to a change in the intensity of the reflections but to an increase in absorption of the  $x$ -ray by the iron content of the samples. With such a high iron content, practically all of the radiation from both the copper and chromium targets was absorbed.

Table 3 lists the results of the spectrographic analysis.

TABLE 3. SPECTROGRAPHIC DATA  
(Elements not chemically analyzed)

+ element present  
- element absent

| Sample | 5 | 6 | 8 | 9 | 13 | 14 | 19 | 32 |
|--------|---|---|---|---|----|----|----|----|
| Cu     | + | - | + | + | +  | +  | +  | +  |
| Co     | + | - | + | + | +  | +  | +  | +  |
| V      | + | + | + | + | +  | +  | +  | +  |
| Li     | - | - | - | - | -  | -  | -  | -  |
| Zn     | - | - | - | - | -  | -  | -  | -  |
| Pb     | - | - | - | - | +  | +  | -  | -  |
| Ag     | - | - | - | - | -  | +  | -  | +  |
| Sn     | - | - | - | - | -  | -  | -  | -  |
| Mo     | - | - | - | - | -  | -  | -  | -  |
| U      | - | - | - | - | -  | -  | -  | -  |

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