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THE MEASUREMENT OF MINERAL ELECTRODE POTENTIALS.

by

John Charavelle Ingram.

A

T H E S I S

submitted to the faculty of the
SCHOOL OF MINES AND METALLURGY OF THE UNIVERSITY OF MISSOURI
in partial fulfillment of the work required for the

D E G R E E O F
BACHELOR OF SCIENCE IN GENERAL SCIENCE

Rolla, Mo.

1913

Approved by *V. Gottschalk*
Professor of Chemistry.

15678

---Introduction---

The first mention in scientific literature of potential difference between minerals seems to be in the work of Robert Were Fox published in Phil. Trans., 1830, pp. 399-414. Being a geologist and mining engineer, Fox was principally interested in the applications of the phenomena to practical geology. As there was, at that time, no means of measuring the small difference of potential developed he was able to detect the direction only of current and in this way arranged the minerals in a series as has recently been mentioned by Gottschalk and Buehler*. He refers to many other experiments which do not exactly pertain to the subject in hand but which point out the state of development some eighty years ago and the amount of work that was done on it. Of the fifty-two available articles by Fox, twenty-seven deal with the subject of magnetism and electricity as applied in some way to geology. Mention is also made in some of his articles of other men who were carrying on investigations along the same line at the time** Thus it is very probable that a rather

** Sir Andrew Crosse, mentioned in American Electr. I, 1836-37, p.135.

**Prof. Reich, mentioned in Edinburgh New Phil. Journal, number unknown.

*Gottschalk and Buehler: Oxidation of sulphides, Economic Geology, Vol. VII, 1912.

extensive literature grew up around this subject even at such an early date.

It is interesting to note, historically, that the work of Fox on "Production of metallic veins by electric action" was directly opposed by the disciples of both Hutton and Werner. The great controversy had not at this time abated entirely and it might seem as if the opposition he would naturally meet and the promise of a more fruitful field of endeavor caused him to abandon this work. His last article dealing with this subject appeared in 1842, after which date he devoted himself to the measurement of temperatures in the earth. It has been suggested that a review of the work of Fox and others along these lines would be of considerable interest to the geological world and might well be made the subject of another thesis.

In 1870, we find W. Skey, an Australian geologist, publishing several articles on electrode potentials but he, like Fox, was able to determine only the relative positions of the minerals with respect to their potentials. It was through these articles by Skey that the reference to the Fox works was discovered by V. H. Gottschalk and H. A. Buehler. The latter, in 1912, independent of the previous work of Fox or Skey, advanced an electrolytic theory in explanation of certain abnormal

rates of oxidation in their experiments*. They then set about to measure the actual electromotive force developed on the surface of minerals. Their measurements are not absolute but show great variation with different specimens and on different parts of the same specimen. In the same year R. E. Dye**, in a thesis under Prof. V.H. Gottschalk, encountered the same difficulty in trying to determine the electrode potential of galena. Within the last year R. C. Wells*° has done some work along this line and in a preliminary paper gives the results of a few measurements but does not mention the accuracy of nor the variations that occur in these measurements.

The present investigation was undertaken with a view to consider the variables which tend to cause inaccuracy in the above mentioned measurements and as preliminary to a further study of the subject by the Missouri

*Gottschalk, V.H. and Buehler, H.A.: Economic Geology, Vol.VII, No.1, p.15 (1912).

**R.E.Dye: Thesis, Missouri School of Mines and Metallurgy, "Electrode potential of galena."

*°R. C. Wells: Mineral electrodes; Original Communications, Eighth International Congress of Applied Chemistry, Vol. 21, pp.149-154.

(4)

Bureau of Geology and Mines. The subject is one which, judging from the work of others and from my own experience, will require much more experimentation before results are obtained which are sufficiently reliable for the use of the economic geologist.

I.

PROBABLE CAUSES OF SOME VARIATIONS.

It might be well here to mention, in tabulated form, the causes that were investigated; these causes will afterwards be taken up in detail and the experimental data given in their proper places. Two general classifications may be made of these causes and may be called, for the sake of convenience, fundamental and superficial causes. The former might be termed those conditioned by the physical peculiarities of the specimen while the latter depends on conditions and methods of measurement.

A. - Superficial causes.

(1) Method of measurement.

(a) Errors in measuring instrument.

(a-1) Condenser effect.

(a-2) Change in working cell.

(b) Effect of time of measurement.

(c) Effect of size of drop.

(a-1) Surface tension.

(a-2) Surface covered.

(2) Surficial effects.

(a) Grease from handling.

(b) Oxide covering.

(c) Irregularities of surface.

(3) Changes in the solution during measurement.

(6)

B. - Fundamental causes.

- (1) Physical condition of surface.
 - (a) Crystal or fractured face.
 - (b) Twinning.
- (2) Difference on different crystal faces.

Description of crystals.

The work was carried on almost wholly upon pyrite. It was the original intention to work with galena and sphalerite as well but the limited time and the difficulty of obtaining results made it necessary to confine the research to pyrite.

Several types of pyrite and galena crystals were used. A general description of them is given below.

Pyrite.

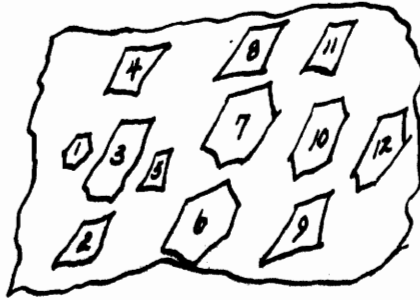
- No.1. Portion of a large pyritohedron exhibiting three faces. Fractured part shows concentric structure. The three faces are much pitted and the surfaces have a flaky appearance. This is a Western pyrite, the locality being unknown.
- No.2. Large, almost perfect pyritohedron from West, showing curved faces. The entire surface of the crystal is covered with a film of oxide.
- No.3. Dark, colored, dull, distorted octahedron. Appeared to be fine-grained.
- No.4. Large pyritohedron from Bingham, Utah. Bright faces and very little twinning.
- No.5. Large, cubic crystal with curved faces show-

ing striations which are twinning planes.

Locality unknown.

Galena.

No.1. A large, hand specimen of Missouri galena showing twelve distinct, modified octahedral crystals embedded in one face. The numbering of these crystals is shown in the following diagram.



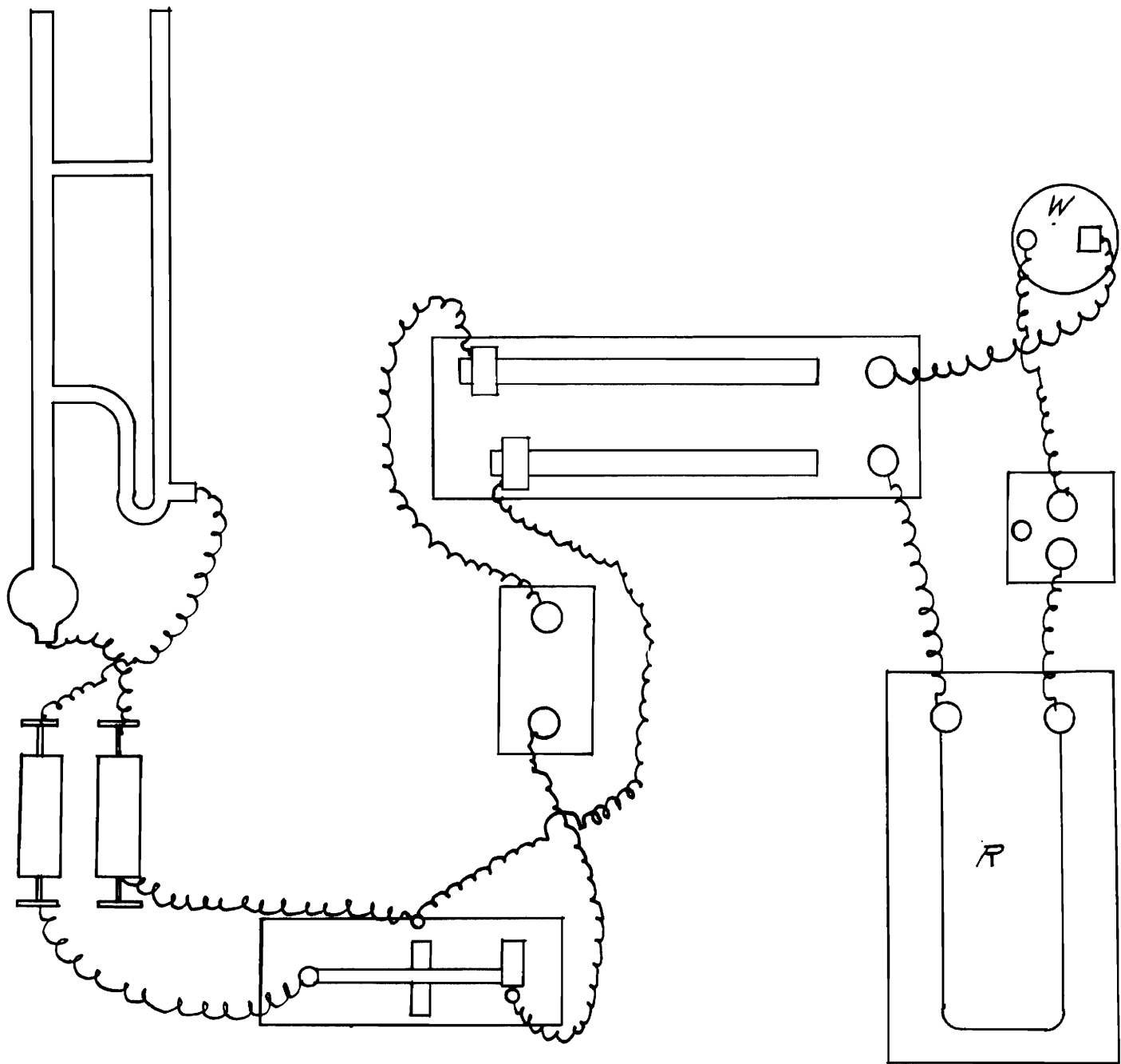


Figure I

Method of Measurement.

The measurements were carried out as in most of the former work along this line by compensating the electromotive force by the Poggendorff method, with a Lippmann capillary electrometer as zero instrument. The several parts of the apparatus were connected up as shown in the drawing. All measurements are only relative and were made against copper. For ease in reading and to avoid calculation the apparatus was made direct reading by compensating the E.M.F. of the working cell by connecting in a resistance box and plugging a sufficient number of ohms to balance this E.M.F. With the other box ~~was~~ set at 1.019, the voltage of the standard cell used, a reading on box "B" gave volts direct.

The final method of measurement used, and the one which seems reasonably the best, is as follows: with the crystal in the holder, turn the two-way switch so as to connect the standard cell and the box "B"; if the mercury in the capillary of the electrometer moves, set box "A" until this shows no deviation. Now place the drop on the crystal and adjust the copper rod which completes the contact; then set the box "B" at the reading expected, throw the two-way switch to make the circuit through the crystal and observe the behavior of the capillary electrometer; if the mercury is not stable plug box "B" until there can be observed no more movement. The reading of box "B" is then the desired E.M.F. in hundredths of volts.

Superficial causes.

I. Method of Measurement.

(A) Errors in measuring instrument.

There are two general sources of error noted, due to the apparatus itself. The first of these, called the "condenser" effect, is little understood and nothing was done with it in this investigation. It was observed by Prof. Gottschalk that at times there was a variation in the electrometer itself and it was observed at times during the following experiments, especially after a rather large charge had been measured. It was named "condenser" effect from its supposed cause, the accumulation of small charges on the surface of the glass capillary. It was first thought to be due to thermal currents developed at the brass junctions but this was soon disproved by eliminating these junctions. At times, when this effect was most troublesome, the electrometer could be disconnected from the rest of the apparatus and a movement of the mercury bubble observed by simply touching together the two wires. This "effect" is not very detrimental to the measurement as it is intermittent and readily noticeable by the bubble not coming immediately to rest but drifting so that no reading can be taken. If the electrometer is short circuited and left so, for a time, the "effect" will disappear.

The other source of variation is one which did not seem to trouble the previous writers and may be due here entirely to the apparatus used. After quite a few observations had been taken, it was noted that there appeared a certain variation in the readings due to an apparent drift in the working cell. In other words, the number of ohms necessary to compensate the E.M.F. of the working cell was variable. Previous writers have found that the apparatus once set at zero remained practically constant through a large number of readings. Several new Columbia and Wesco cells delivering approximately 1.5 volts were tried but the same variation was observed.

An idea of the amount of this variation and its relation to the time of reading may be gained from the following table which gives the time and the setting necessary to bring the apparatus to zero reading.

Table I.

Time in Min.	1st trial ohms	2nd trial ohms	3rd trial ohms
0	326.0	329.5	330.0
10	326.2	329.0	330.0
20	326.7	330.0	330.5
30	328.0	331.5	331.0
40	327.7	332.7	330.5
50	327.0	327.0	330.0

It was for this reason that it was found necessary to set the instrument at zero before each reading and check this zero reading afterwards. To this end the two-way switch was inserted as in this manner the time was cut down as well as tedious manipulation avoided.

(B) Time factor in measurement.

Eliminating the possibility of variation in the measuring instrument, it is still possible to get different measurements with the same drop at different intervals. The following readings, to illustrate this, were taken at random from ten distinct sets of trials.

Table II.

Time	Pyrite 1 Volts	Pyrite 1 Angle 2	Face 1 Volts	Pyrite 4, Face 1 Angle 1	Galena Crystal 2, Total face
0	0.224	0.215		0.230	0.145
10	0.22	0.215		0.227	0.145
20	0.22	0.21		0.225	0.140
30	0.215	0.21		0.225	0.140
40	0.215	0.207		0.227	0.142
50	0.215	0.205		0.230	0.140

The above readings were taken in the usual manner except, instead of removing the drop after each trial, it was left undisturbed. The copper rod was allowed to remain in the drop but the circuit was broken that the current might not aid in dissolving traces of the mineral which would change

the concentration of the solution.

From the above and similar evidence it was thought best, in following measurements, to adopt a standard method of reading. The method adopted, to eliminate this variation as much as possible, was as follows: the drop was placed on the crystal and the electrometer set at zero; then box "B" was set at the reading expected and all connections made; next the rod was placed in the drop and the reading taken in the shortest possible time.

(C) Influence of size of drop.

The size of the drop might possible effect the reading in two ways: namely, first by the variation in surface tension in different size drops and secondly as it is this which determines the area on the crystal over which the E.M.F. is measured. In Dye's*work it is stated that "the smaller drop gives the higher reading" and that "this greater E.M.F. may be the result of an accelerating action due to surface tension." This was investigated in the following manner; the copper rod was moistened and inserted into the smallest drop that could well contain it, the reading was then taken, a small drop from a capillary tube was then added to the one already on the crystal and the next reading taken. A series of twelve

R. E. Dye, The electrode potential of galena: Thesis at Missouri School of Mines, 1912, page 5.

such were measured, each time increasing the size of drop. The experiment was carried out in the manner outlined above in order that there might not enter any surface variation other than that due to the increasing area covered by the drop.

Readings taken with boiled, distilled water using copper rod:

Pyrite V Face I middle of Face

Table III.

No.	1st trial	2nd trial	Remarks
	volts	volts	
1	0.14	0.155	
2	0.15	0.155	
3	0.12	0.17	
4	0.165	0.175	
5	0.18	0.17	
6	0.18	0.175	
7	0.19+	0.185	
8	0.20	0.21	Same drop as No.7 spread to include more surface.
9	0.20	0.215	
10	0.245	0.22	
11	0.25	0.23	
12	0.25	0.22	

It will be noted that while the results in the above experiment are exactly opposite to those obtained by Dye,

the methods used in the two experiments were very different. In the former work different points on the crystal were used with the various sized drops which, as will be shown later, may have some influence on the variation.

It would be impossible here to distinguish between the effect of surface tension and any variation caused by a difference in potential due to inequalities in the surface of the crystal, as the two effects are simultaneous. Although no data can be given to support it, it seems most probable that the maximum effect is the latter. It would appear then that the effect of surface tension in these kind of measurements would be the last to be eliminated.

2. Surficial effects.

(A) Grease from handling.

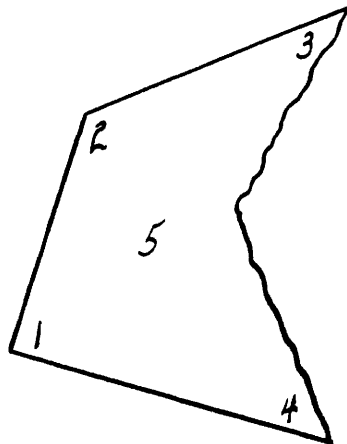
It was suggested by Szivessy* that in handling the specimens used in previous work no particular care was taken to avoid touching the surface upon which the measurements were to be taken. It was thought that any grease which might be left upon the surface in this way might interfere with the measurement should the drop happen to be placed wholly or partly on a point of the crystal so covered. To determine the amount of this effect measurements were made in the usual way on a typical crystal before and after attempting to remove the grease. The grease was removed by boiling the crystal in absolute alcohol for a few

*Szivessy, G., Ann.Phys.Band 36, Heft 1, No.11, p.183, 1911.

minutes and then drying in air and using immediately. Different points on the crystal, all the angles and the center, were measured for E.M.F.; these readings were averaged and checked against average reading taken by covering the whole fact with water and reading.

Results given below were obtained on Pyrite No.1, Face I.

Sampling the face: Figure 1 represents Face I of pyrite No.1, and shows method of what might be called "sampling the face." It may be thought that more reading would give a more correct "sample" but this method was found to yield as accurate results as could be expected.



Readings given in the following tables were taken at the points indicated on the sketch:

Face as originally used; grease not removed.

Table IV

Point	1st trial volts	2nd trial volts	3rd trial volts	4th trial volts
1	0.198	0.230	0.200	0.190
2	0.175	0.175	0.180	0.165
3	0.168	0.190	0.180	0.185
4	0.132(?)	0.185	0.175	0.155
5	<u>0.194</u>	<u>0.120(?)</u>	<u>0.140</u>	<u>0.190</u>
Average	0.173	0.180	0.175	0.177
Observed average)	0.172	0.185	0.180	0.172

With grease removed.

Table V.

Point	1st trial volts	2nd trial volts	3rd trial volts	4th trial volts
1	0.255	0.265	0.260	0.260
2	0.220	0.220	0.180	0.130(?)
3	0.200	0.220	0.210	0.210
4	0.200	0.210	0.200	0.200
5	<u>0.250</u>	<u>0.250</u>	<u>0.240</u>	<u>0.270</u>
Average	0.225	0.233	0.238	0.214
Observed Average)	0.225	0.230	0.230	0.210
Observed after 10 minutes standing)	0.227	0.230	0.235	0.205

Comparison.

Table VI.

	By original method		With grease removed	
	Calculated	Observed	Calculated	Observed
1st	0.173	0.172	0.225	0.225
2nd	0.180	0.185	0.233	0.230
3rd	0.175	0.180	0.238	0.230
4th	<u>0.177</u>	<u>0.172</u>	<u>0.214</u>	<u>0.210</u>
Average	0.176	0.177	0.227	0.223

Galena No. 1.

Table VII.

Crystal No.	Without removing grease	1.	2.
		With grease removed	With grease removed
1	0.08	0.14	0.14
2	0.075	0.14	0.135
3	0.073	0.14	0.12
4	0.085	0.17	0.10
5	0.085	0.145	0.12
6	0.080	0.14	0.14
7	-----	0.17	0.14
8	0.100	0.165	----
9	0.060	0.15	0.15
10	0.095	0.17	----
11	-----	0.17	0.16
12	-----	0.16	0.155

From tables IV and V it appears, as might be expected, that the average of a number of "drop" readings checks up fairly well with the measurement obtained when the entire face is covered. Disregarding such measurements as are marked by a question mark, the maximum variation between any two trials on the same point is, in table IV, 0.05 volts while in table V it is 0.03 volts.

When we bring together the results of these two tables to construct table VI a distinct and fairly constant difference is noted in the readings before and after removing the supposed grease coating. The maximum difference is about 0.05 volts. The fact that a treatment with hot alcohol, which would tend to dissolve any fatty compounds on the surfaces, gives a fairly constant E.M.F. which is higher than that previously assigned to pyrite, is in agreement with the supposed effect of any coating. The coating, when present at the point of measurement, would naturally give a low reading. The data given so far will be borne out by certain measurements made in connection with succeeding experiments; that is, the average E.M.F. for pyrite, as measured against copper in all the determinations made in connection with this work, will be very near 0.225 volts.

The measurements obtained for galena, as set down in table VII, are more variable than those for pyrite but show very well the effect of removing the grease coating. It was

found impossible to secure measurements for 7, 11, and 12 on account of the difficulty of getting the water to stick on the faces of these crystals. This may have been due to an excessive amount of grease coating on these surfaces which prevents them from being wetted by the water.

(B) Oxide covering.

The development of oxide on the surface of crystals worked with was noticeable only on the surface of the pyrite specimens. Several fairly perfect pyritohedrons considered for use were found to be covered with a thin layer of oxide and gave extremely variable E.M.F.'s. It was found impossible to remove this layer without rendering the surface unfit for use. The effect of such oxidized spots would be analogous to that of the grease covering except that the limonite or hematite has an E.M.F. of its own. Gottschalk and Buehler* give 0.08 to 0.26 for the E.M.F. of hematite; no previous measurement of limonite could be found, but, from preliminary experiments made on a few nodules there can be no doubt that a potential is developed.

It has been found very difficult to obtain a working specimen which is entirely free from minute spots of oxide. On observing the specimens with the naked eye they appear bright and free from oxidation but, upon viewing their sur-

*Gottschalk and Buehler; Econ. Geol., Vol.VII, 1912, p. 15.

face by means of the metallographic microscope there appears to be a film of oxide over the entire surface. In some spots the oxidation appears to have been more pronounced than in others. The following photographs will give some idea of the general appearance of the surfaces.

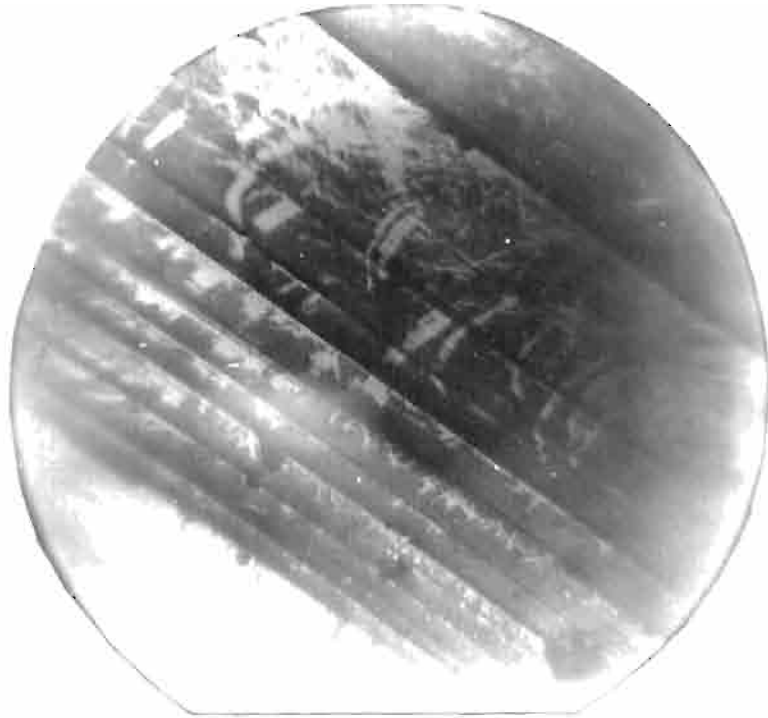


Figure III
Plate IV

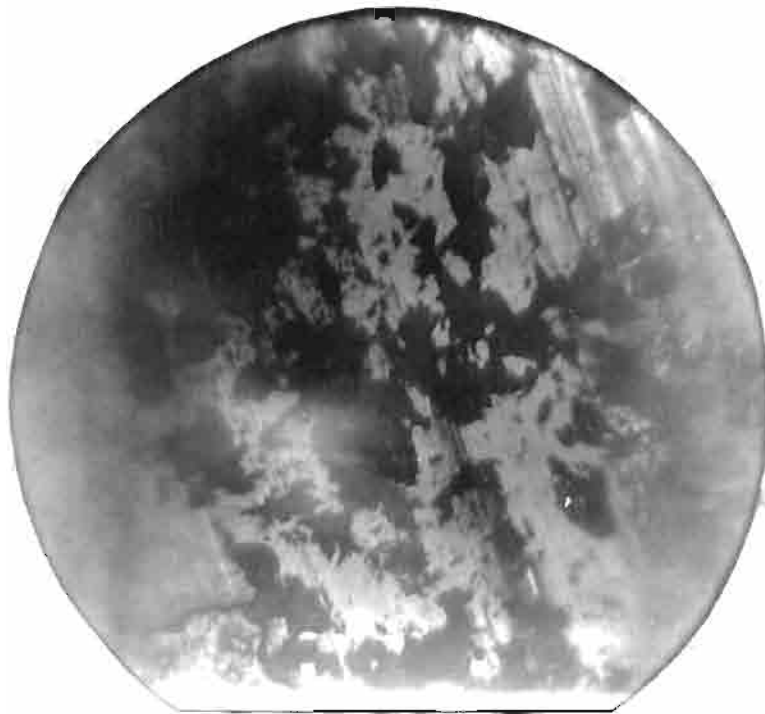


Figure IV
Plate I

These photographs were taken on Face I of pyrite 1 and 4, using a magnification of 120 diameters. The light portions show the unoxidized pyrite while the dark spots indicate the oxide film. From figure III it appears that the total face was at one time covered with such a film and parts of that film removed mechanically by rubbing or by contact of some sort. The physical condition of the surface of pyrite 1, as it appears in figure IV, seems to be due to differential oxidation rather than to a rubbing off of the oxide.

From a number of observations similar to those shown by figures III and IV, and from the fact that the crystals invariably show oxidation when observed through the microscope, it is thought that this may have an important bearing on the accuracy of mineral potential measurements, especially of such minerals as are readily oxidized. If limonite or hematite and pyrite have distinct E.M.F.'s., as noted previously, and no attempt has been made to remove the former from the surface of the latter, what we have been measuring and calling the potential is really the combined effect of both. The more one observes the surfaces of pyrite crystals, under the microscope, the more one is impressed with the likelihood of this being an important source of variation in the measurements.

The supposed interference of this oxide film could be overcome in the measurement of fractured faces by tak-

ing the readings on freshly fractured surfaced. The present investigation was confined almost entirely to the potential developed on crystal faces so that, in carrying on this investigation, other means will have to be resorted to. It seems very probable that the film could be removed without affecting the mineral by such mechanical means as are used in polishing steel specimens for use in metallography. The lack of time prevents the carrying out of this phase of the work at the present writing, but it will be taken up in the near future in connection with the researches carried on by the Missouri Bureau of Geology and Mines.

(C) Irregularities of surface.

As will be brought out under the head of "fundamental causes", some of the previous writers have been led by certain of their results to believe that the potential developed on crystal faces is essentially different from that on fractured surfaces. Another interesting fact, brought out by the microphotographs, is the great irregularity of supposedly plane surfaces. Some of the best illustrations of this are shown in figures V and VI.

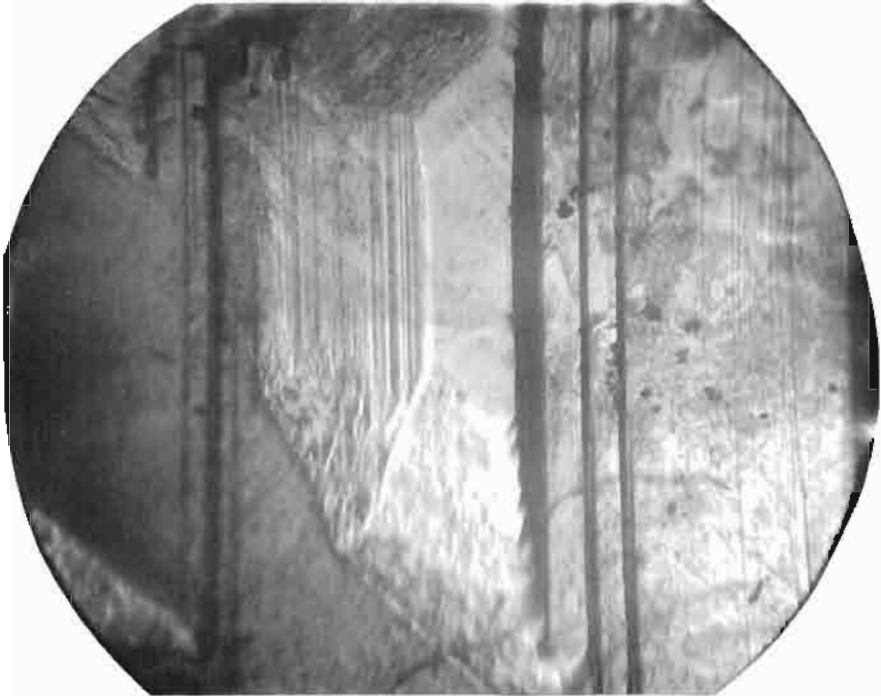


Figure V
Pyrite IV

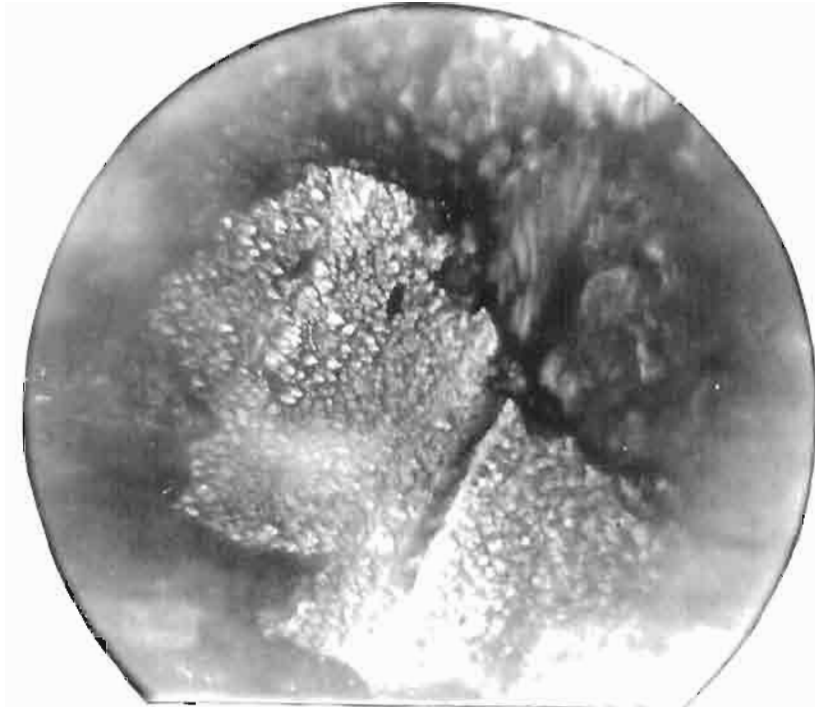


Figure VI
Pyrite I

Figure V, taken on pyrite No.4, shows pitting of the surface and a raised and fractured crystal; number 5 simply pittings which appear to the naked eye as flake pits. It seems reasonable to suppose that such irregularities of surface would not only have the effect of spreading out or including the drop as the case may be, but of bringing in the effect of a combined fracture and crystal face potential.

Very little work was done on this phase of the subject so that no data can be presented here. It is simply desired here to call the attention to the irregularities of surface as a possible cause of some of the variations observed.

III-Changes in the solution during measurement.

The variation of the potential measurement with the kind and concentration of solution used has been shown by R. E. Dye* R. C. Wells**and others. In the present investigation only distilled water was used for two reasons: as yet no other electrolyte has given more constant results and since this work was undertaken from a geological point of view it was desired to approximate conditions in the actual mineral veins.

*R. E. Dye, Thesis, Missouri School of Mines, 1912.

**R. C. Wells, Mineral electrodes: Original Communications
Eighth International Congress of Applied Chemistry, Vol.21, pp.149-154

Since the measurements were taken very soon after the drop was placed on the mineral surface, very little time was given for the changing of the solution by the dissolving of any soluble salts formed. No attempt is here made to discuss at length the effect of different solutions; it is merely mentioned here to make the classification as complete as is possible at the present writing.

Fundamental Causes.

I-Physical condition of surface.

(A) Influence of crystal or fractured condition.

As mentioned on page 24, it was thought by some men who have considered the subject that the potential developed on a crystal face might be essentially different from that developed on a fracture face. A few measurements were undertaken on the faces and fractured parts of pyrites 1, and 4. Sufficient work has not been done in developing this part of the research to verify the following results: they are given merely as preliminary experiments to a more extended study of the subject.

Crystal number	Fractured part-volts	Crystal face volts
1	0.320	0.0
1	0.330	0.225
1	0.315	0.230
4	0.350	0.230
4	0.365	0.225
3	0.327	0.225
5	0.320	

The third column in the above table gives the average electromotive force on the crystal faces of the crystal. The first three measurements were taken on different portions of several fractured parts of crystal no.1.

These results seem to point to the probability of discovering, in the subsequent work, a distinct difference in potential due to the physical condition of the surface. It will be noted that all the measurements on fractured surfaces are much higher than those on the crystal surfaces, and approach 0.37 volts, the potential assigned by Prof. V. H. Gottschalk and H. A. Buehler to marcasite .

It would be very interesting, in this connection, to carry on this phase of the investigation using galena, on account of the possibility of obtaining such perfect fracture or cleavage faces.

(B) Effect of striations from twinning or other structural arrangements.

Again we resort to the microphotographs to bring out the general appearance of the crystal face to be studied. This summary of the possible causes of variation would not be complete were not some mention made of the probable effect of such crystallographic arrangements as twinning.

The following photograph was taken on face 1 of pyrite 5. On megascopic observation the crystal exhibits marked striations.



Figure III
Pyrite II

The surface of this crystal appears under the microscope to be made up of a series of ridges and valleys. The tendency of such a structure would be to concentrate the drop used in measurement in these valleys and spread it along the valley rather than allow it to include the ridges. In this way it influences the position of the drop and prevents the "sampling of the face". What would be the effect of such a structure on surface tension can only be conjectured. There can be no doubt that the drop is greatly distorted due to the attraction of the ridges and the tendency to elongate itself in a direction parallel to the general structure of surface. At this stage in the investigation it appears reasonable to suppose that further research will reveal the nature of this influence. At present there does not seem to be any distinct difference in the E.M.F. measured on such surfaces and those found on the faces of such crystals as Nos. 1 and 4, except that the measurements obtained on the former are more variable.

The following measurements were taken on pyrite No.5.

Position of Measurement	1st series	2nd series	3rd series
1	0.295	0.290	0.340
2	0.240	0.215	0.240
3	0.185	0.160	0.170
4	0.180	0.180	0.170
5	0.205	0.200	0.165
6	0.175	0.210	0.220
Portion of surface covered	0.235	0.230	0.230

(2) Difference on different crystal faces.

It has been the subject of much discussion during the progress of this thesis whether it would be reasonable to expect a fundamentally different potential on crystallographically different faces. As an illustration: would we find the same electromotive force developed on the faces of octahedral galena as we would find on cubic? No, such difference was noticed in the measurements on pyrite, the results obtained with crystals 1, 2, 3, and 4 being essentially the same as those with crystal 5. In this investigation the potentials measured on different faces of the same crystal were sufficiently similar to admit of the statement that no radical difference exists in these measurements.

Very little work has been done on crystallographically different specimens but, from the few observations

(34)

that have been made, this difference, if present, is very slight and will not be detected until more accurate methods of measurement are developed.

SUMMARY.

It is apparent, from the relative amount of space given to the various headings in this discussion, that the most important feature of the research at present is the detection and elimination of errors in the method of measurement and in the physical condition of the specimens. In this connection it was attempted, in the preceding pages, to bring out:

- (1) the importance of a standard method of measurement,
- (2) that the supposed grease covering can be removed by boiling the specimen in ethyl alcohol and that the potential measurement on a specimen so treated is about 0.05 volts higher for pyrite than that found in an untreated specimen,
- (3) that from microscopic observations made on the surface of some of the pyrite crystals it will be very necessary to remove the oxide coating before the true pyrite potential is obtained, and
- (4) that the investigation of those causes classed as fundamental must be left until the superficial effects are eliminated.
