readily calculated as in the well-known methods for the volumetric determination of this element. When chromium has been determined, the solution is again brought to the boiling point, and the chromium again oxidized to chromic acid by the addition of a 2 per cent. solution of potassium permanganate until a slight permanent precipitate of manganese dioxide is obtained. The solution is again cooled until cold and the manganese dioxide filtered out and the method continued as though the chromium had not been determined. If more than one gram of ferrous ammonium sulphate has been used, 1 or 2 g. additional sodium pyrophosphate should be added.

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THE INFLUENCE OF FINE GRINDING ON THE WATER AND FERROUS-IRON CONTENT OF MINERALS AND ROCKS.

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It has been the general practice, following the directions of numerous writers, to reduce most rock and mineral powders to a very fine state of division preliminary to their analysis. This seemed called for on several grounds—to secure a proper sample of an inhomogeneous mixture, the need of extreme fineness for the ferrous iron determination, for that of the alkalies by the J. Lawrence Smith method, for all decompositions that involve the use of boric oxide or lead oxide, and in general in order to insure complete and speedy decomposition by fusion with an alkali carbonate. A very fine powder is, however, far less needed for the last-named purpose than has been commonly supposed. The writer's recommendation has been to prepare a main sample of moderate fineness and to grind separate small portions of this finer when needed.

Now comes the Swedish chemist, Robert Mauzelius, and shows[1] that fine grinding is highly detrimental in the case of minerals containing ferrous iron because of its resulting oxidation. Led by Mauzelius's work, Nicholas Knight has shown that siderite oxidizes when finely ground.[2] A. L. Day and E. T. Allen had shown how very great is the effect of fine grinding on the moisture content of minerals[3] and Mauzelius in the paper cited adds to our knowledge on this point by quantitative data with respect to rock powders. These writers clearly show that coarse powders entirely free from hydroscopic water, or containing but a few hundredths of one per cent. of it, take up moisture progressively as the grinding

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[1] *Sveriges Geol. Undersökning Arsbok*, **1** (1907), No. 3.

[2] *Chem. News*, **97**, 122.

[3] *Pub. Carnegie Inst.*, No. 31, pp. 56–7; *Am. J. Sci.*, **19**, 93; *Bull. U. S. Geol. Survey*, **305**, p. 55.

continues, and that the amount so taken up can readily be brought to exceed 1 per cent. of the weight of the sample. This is, so far as amorphous minerals are concerned, entirely in harmony with what Bunsen observed for spun glass,[1] a condition in which the surface exposure is very great. Since many minerals and nearly all rocks contain ferrous iron, Mauzelius rightly insists that the determination of this component shall be made on the coarsest material that can be completely decomposed by the acid that is employed. Further, in view of the great absorption of atmospheric moisture, and in such varying degree according to the fineness of the sample that no two analysts can be expected to prepare samples of the same mineral that will contain like amounts of moisture, he argues that the old practice of stating the analysis in terms of dried material should be followed in preference to the later practice of reporting the moisture given off below 100–110° as well as that firmly held at that point. To this last recommendation, objection may be made, as I shall show later.

The oxidation of ferrous iron was such an important observation in view of the extent to which it may go (up to about 40 per cent. of the FeO present in one of Mauzelius's experiments) that the subject seemed to demand further investigation. It was evident that acceptance of the direction to use only relatively coarse powders would introduce serious complications and uncertainties into the execution of an analysis, for there are many minerals that can not be readily decomposed by boiling hydrofluoric acid even when reduced to pass a sieve of 150 meshes to the linear inch (nearly 60 to the centimeter, or approximately 0.08 mm. width of apertures between wires). It was therefore necessary to devise, if possible, some means of fine grinding that would not involve oxidation of iron.

Much of the tabulated matter of this paper is but a confirmation and extension of what Allen, Day and Mauzelius have shown, but its presentation in detail seems justified on account of the intimate bearing of the subject on mineral analysis and its consequent importance to all mineral analysts, mineralogists and petrologists. Often, valuable observations have passed unheeded or insufficiently heeded and been eventually forgotten. Frequent reiteration is called for if they are to become generally known and appreciated.

In the course of the experiments that were made on powders ground in air, no explanation that occurred seemed adequate to fully account for the oxidation of ferrous iron except that of strong local heating of the grains at the moment of fracture or attrition under the pestle. The effect should be especially marked with pyritic minerals. The idea of intense local heating, though at first thought improbable, becomes

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[1] *Wied. Ann.*, 24, 327 (1885).

less so when it is remembered that steel strikes sparks from flint. If the oxidation is due simply to greater surface exposure it ought seemingly to continue at a lessened rate after cessation of the grinding, but powders finely ground in past years, show to-day the same FeO content as when originally analyzed. Of course, there is no general heating to account for the phenomenon, as when a rock is pulverized in a closed ball mill, where the temperature may rise to such a degree as to dehydrate the gypsum that is added to Portland cement clinker.[1]

Led by the thought of local heating as the chief cause of oxidation of the ferrous iron, it occurred to me to test the effect of grinding on the content of water that is given off above 100–110° and is supposed to be chemically held in large part, for it seemed possible that this might be reduced by grinding. If so, however, the loss is more than offset by a very large gain due to increased surface exposure, as the results tabulated below plainly show. This gain in firmly held water, quite in accord again with what might be expected in view of the firmness with which Bunsen found (*l. c.*) the adsorbed water to be held by glass, has an important bearing on the question of the permissible size of grain of a sample that is to be analyzed, and strengthens in the strongest manner the argument of Mauzelius for a coarse sample. For the rock powders tested by myself, the increase in this firmly held water seems to be as a rule of nearly the same general order of magnitude as that of the loosely held moisture, but it is very much greater with the meerschaum tested (16 below).

The following remarks explain certain features of the table. The determinations of the first three numbers were made simply to test the claims of Mauzelius, those that follow relate also to increase of water above 105° under different conditions of grinding. The designations < 100-mesh, < 150-mesh,[2] etc., mean that all the material was simply

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[1] R. K. Meade, *Portland Cement*, p. 307. In connection with the dehydration of gypsum, an interesting experiment made by Dr. E. C. Sullivan and Mr. Geo. Steiger in this laboratory, may well receive mention. They were able by grinding several hours daily for several days with a mechanical grinder, to reduce the water of crystallization of gypsum from 20 per cent. to about 5 per cent., without evidence that a limit had been reached. It is regrettable that no effort was made to ascertain if this lost water could be regained by exposure to the air after grinding.

[2] The following are the approximate diameters of the apertures in the sieves used.

Mesh. mm.
80 = 0.20
100 = 0.18
120 = 0.11
150 = 0.08
200 = 0.065

These sieves were of metal, the use of which, in preparing samples for full analysis, is forbidden if traces of metals like those of which the sieve is made are to be looked for. In such cases, sieves of the best silk bolting-cloth shaken in a very gentle manner should be used.

crushed to pass the respective sieves with as little production of fine powder as possible and without grinding. This was effected by brief crushing in the steel mortar figured on p. 46 of *Bulletin* 305 *of the U. S. Geological Survey*, then sifting, transferring the coarser matter again to the mortar and repeating these operations till all had passed the sieve. Nevertheless, there was unavoidably much powder formed that passed 150 meshes and even 200 meshes to the inch. Material thus prepared and analyzed was then ground in air and also under water, absolute alcohol, or carbon tetrachloride, sometimes all three, in order to test the effect of these liquids, both on oxidation of iron and absorption of water. The 20-minute and 30-minute grindings were all done by hand, those of longer duration by the McKenna mechanical grinder. Grinding by hand is less effective in producing fine powder than by a mechanical grinder for the same length of time, and different grinders may be very unequally effective (6, *c*, *d*). In comparing figures in the table it should be remembered that equal periods of grinding do not necessarily result in the production of powders of the same fineness, especially as between dry and wet grinding. Where more than one determination of water or iron was made on a given sample all are reported in order to show, more particularly, the generally excellent agreement of the iron values on a given sample when the rocks are readily decomposable and the variations to be expected when they are not. In the latter cases, finer grinding would have led to closer agreement but lower values.

All water percentages not otherwise indicated are based on direct weighings. If it were important to show the actual amount of water taken up by a given weight of unground material, the mode of statement would have been otherwise than by percentages of those weights. To give the percentages of ferrous oxide actually found would convey an incorrect impression. They have therefore been corrected by raising them in proportion to the amount of water absorbed during grinding, so as to be properly comparable with the percentages in the unground material and give a true picture of the extent of oxidation suffered by the latter. These corrections range from 0 to 0.13 per cent. The same result would have been reached by reducing all FeO percentages to an anhydrous basis. In 1, 2, and 3 the FeO percentages in ground material are still too low, the full correction not being applicable for lack of data as to how much absorbed water was held at 110°.

Effect of Grinding in Air.

The table shows in the first place with respect to the moisture, that while some rocks and minerals contain in the lump, and even in material as fine as 100-, or even 120- and sometimes 150-, mesh size, little moisture (2*a*, 5*a*, 12*a*, 13*a*) or none at all (14*a*, 15*a*), many, and particularly those that have undergone more or less alteration into hydrous minerals,

No.	Material.	Maximum grain of bulk sample.	Exp. No.	Conditions of grinding.			H_2O %.		FeO % corrected for H_2O taken up.			
				Grams of bulk sample.	Enveloping medium.	Time, minutes.	105°—	105°+	Pratt method.		Cooke method.	
1	Basalt[1]	0.08 mm. < 150-mesh	*a*	unground	....	....	0.24	..	7.03	7.07	......	
									7.09	7.11	......	
			b	4	abs. alc.	30	0.34	..	7.09	7.12	......	
									7.14	..	......	
			c	4	air	30	0.32	..	7.01	7.07	......	
									7.08	7.16	......	
									7.18	7.23	......	
			d[2]	20?	air	?	0.45	..	7.12	..	......	
			e	4	air	120	0.83	..	6.87	6.93	......	
2	Diorite[3]	0.08 mm. < 150-mesh	*a*	unground	....	..	0.07	..	3.65	3.65	3.55	3.52
			b	4	abs. alc.	120	0.32	..	3.48	3.50	3.41	3.50
			c	4	CCl_4	120	0.48	..	3.30	3.37	3.26	3.31
			d	4	H_2O	140	0.53	..	3.41	3.44	3.37	3.39
			e	4	air	120	0.54	..	3.29	3.37	3.30	3.39
									3.41	..	......	
			f[4]	20?	air	?	0.28	..	3.52	..	3.37	..
3	Andesite	0.08 mm. < 150-mesh	*a*	unground	....	...	1.14	..	2.57	2.58	......	
			b	4	abs. alc.	15	1.21	..	2.58	..	......	
			c	4	H_2O	15	1.39	..	2.53	..	......	
			d	4	air	15	1.46	..	2.53	..	......	
4	Andesite	0.08 mm. < 150-mesh	*a*	unground	....	....	1.01	3.40	3.32	3.34	......	
			b	4	air	30	1.18	3.54	3.09	..	......	

[1] The rather wide variations for FeO in the same series, particularly *c* are ascribed to the refractoriness of an iron-bearing constituent, which necessitated several retreatments.

[2] Sample originally analyzed.

[3] Difficult to decompose by HF in powder coarser than 150-mesh.

[4] Sample originally analyzed.

No.	Material.	Maximum grain of bulk sample.	Exp. No.	Conditions of grinding.			H_2O %.		FeO % corrected for H_2O taken up.			
				Grams of bulk sample.	Enveloping medium.	Time, minutes.	105°—	105° +	Pratt method.		Cooke method.	
5	Diabasic norite or gabbro[1]	0.08 mm. < 150-mesh	*a*	unground	....	....	0.03	0.66	13.55	13.35	13.54	13.24
			b	4	abs. alc.	20	0.10	0.86	13.04	13.14	13.11	..
									13.15	13.20	......	
									13.23	..	......	
			c	4	CCl_4	60	0.34	0.94	12.95	13.00	......	
			d	4	air	30	0.10	0.66	12.91	12.93	13.06	13.07
									13.06	13.07	......	
			e[2]	20?	air	?	0.10	0.65	..	..	......	
			f[3]	4	air dried by H_2SO_4	120	0.74	1.00	12.72	12.79	......	
6	Diorite-porphyry	0.18 mm. < 100-mesh	*a*	unground	....	....	0.29	1.97	2.71	2.72	......	
			b	20?	air	?	0.42	2.07	..	..	2.38	..
			c	4	air, machine A	120	0.91	2.26	2.30	2.31	......	
			d	4	air, machine B	120	1.90	2.73	1.54	1.54	......	
			e[4]	4	air dried by H_2SO_4, machine B	120	1.36	2.38	1.80	..	......	
7	Altered basaltic greenstone	0.18 mm. < 100-mesh	*a*	unground	....	....	0.22	3.13	6.68	..	......	
			b	4	air	120 or more	1.70	4.19	3.74	..	......	
8	Quartz-diorite	0.11 mm. < 120-mesh	*a*	unground	....	....	0.37	2.03	2.58	..	......	
			b	4	air	120 or more	1.08	2.33	1.69	..	......	
9	Quartz-diorite	0.11 mm. < 120-mesh	*a*	unground	....	....	0.35	1.60 1.64	2.08	2.09	......	
			b	4	abs. alc.	120	0.54	1.80	2.02	2.02	......	
			c	4	air	30	0.40	1.56 1.64	2.04	2.07	......	
			d	4	air	120	1.01	1.86 1.95	1.48	1.48	......	

[5] An iron-bearing garnet in this rock was extremely refractory unless very finely ground.

[5] Sample originally analyzed.

[5] Exposed to air of room after grinding.

[6] Exposed to air of room after grinding.

No.	Material.	Maximum grain of bulk sample.	Exp. No.	Conditions of grinding. Grams of bulk sample.	Enveloping medium.	Time, minutes.	H_2O %. 105°—	105°+	FeO % corrected for H_2O taken up. Pratt method.		Cooke method.
10	Quartz-diorite	0.11 mm. < 120-mesh	a	unground	....	....	0.21	0.65	2.55	2.58	......
							0.22	0.65			
			b	4	abs. alc.	120	0.39	0.88	2.58	2.58	......
			c	4	CCl_4	120	0.41	0.80	2.47	2.51	......
			d	4	air	120	0.69	1.05	2.33	2.34	......
							0.70				
11	Granite	0.11 mm. < 120-mesh	a	unground	....	....	0.12	0.46	2.28,	2.28	......
			b	20?	air	120 or more	0.21	0.53	1.85	..	......
12	Granite	0.11 mm. < 120-mesh	a	unground	....	....	0.03	0.24	1.93	2.01	......
									2.08	..	
			b	4	abs. alc.	120	0.32	0.64	2.01	2.03	......
									2.06	..	
			c	4	air	120 or more	0.67	0.72	1.86	1.86	......
13	Altered basaltic rock	0.18 mm. < 100-mesh	a	unground	....	....	0.03	2.00	8.33	8.34	......
								2.12			
								2.15			
			b	4	abs. alc.	120	0.54	2.42	8.18	8.21	......
			c	4	air	30	0.15	2.19	8.24	..	......
			d	20?	air	not known but very long	1.16	3.34	5.86	..	......
							1.21	3.36			
14	Quartz	mostly finer than 0.08 mm. < 150 mesh	a	unground	....	....	none	0.06[1]	..	..	......
			b	4	air	120	0.35	0.45	..	..	......
15	Berlin porcelain unglazed[2]	?	a	uncrushed	....	....	none	none	..	..	......
			b	?	air	?	0.62	0.18	..	..	......
16	Meerschaum	coarse	a	unground	....	....	5.76	6.79	..	..	......
			b	4	air	600	6.33	9.81	..	..	......

[1] H_2O 105°+, by ignition.

[2] Also 0.80% H_2O as total ignition loss.

[3] Tests made in

contain in coarse condition a good deal of loosely held water (3*a*, 4*a*, 6*a*, 8*a*, 9*a*). Since this water is characteristic of certain minerals and affords a valuable index of the character and extent of alteration or of the presence of zeolitic minerals, it should not be excluded from the tabulated analysis, as Mauzelius recommends. On the other hand, the petrologist reports "kaolin" as the chief hydrous mineral in 13. The analysis shows absence of loosely held water, therefore, the mineral is probably in the crystallized form of kaolinite, which does not lose water at 100°. Since, further, such powders of grain as coarse as 100-, 120-, and even 150-mesh often show much less than 0.1 per cent. moisture, if hydrous alteration products are absent, it is reasonable to conclude that there has been occasioned relatively as little oxidation of iron in such powders as absorption of moisture, on the assumption that the small amount of water found results from the increased surface exposure due to crushing and is not inherent. Therefore, if the powder is completely decomposed by hydrofluoric acid it is safe to assume that a determination of ferrous oxide in it, carried out with proper precautions, will give a result close to the truth.

It is further evident that, whereas, long grinding in air (2 hours or more) by the mechanical grinder in most cases gives rise to marked oxidation of iron (6*d*, 7, 8, 9*d*, 11, 13*d*) and increase in the water content (6*d*, 7, 8, 9*d*, 10*d*, 13*d*, 14), these effects are usually not strongly marked when the grinding was by hand for only thirty minutes or less (5*d*, 9*c*, 13*c* and especially 1*c*, *d*) though almost invariably plainly enough revealed by the tests to forbid grinding when the highest accuracy is of importance. Occasionally, as in 3*d*, 15 minutes' grinding resulted in relatively appreciable oxidation of iron and absorption of moisture.

It is further very evident, as noticed by Mauzelius, that with different minerals very different degrees of oxidation result under like conditions, from a few per cent. (1*e*) up to nearly 50 per cent. (7) of the FeO after several hours' grinding, and it is not always the mineral that might *a priori* be regarded as most oxidizable that shows most oxidation. For instance, a magnesian siderite containing 46 per cent. FeO, tested by myself, suffered oxidation of only about 3 per cent. of its FeO when ground by itself for 15 minutes, and but little over 5 per cent. when ground for 2 hours with four times its weight of quartz powder. The nearly white color of the original powder had, however, become decidedly brownish in both cases. It is undoubtedly true that, as shown by Mauzelius for mica, a soft or tough mineral undergoes greater oxidation if ground together with a hard one, like quartz as in the above instance and with the mica tested by him, than by itself. This is due, of course, to the attrition effect of the harder mineral, hence, in different rocks the extent of oxidation of one and the same ferrous mineral may depend on the nature of its associates. Naturally the degree of water absorption will be less or greater for a like reason. Relatively slight oxidation of FeO after grinding in air (1, *c*, *d*) may sometimes be due to the hardness of the iron-bearing minerals or their toughness in absence of other hard ones, which would result in a far coarser powder than with some other rock or mineral ground for the same length of time, but in addition the relative stability of the chemical molecule is doubtless an important factor, perhaps the most important. Much remains to be done in accumulating experience with different minerals and their mixtures.

Naturally the idea of grinding in an inert atmosphere suggested itself, in order to prevent if possible the oxidation of FeO, if not the absorption of water. If the oxidation is due to local heating more than to surface exposure, this treatment should be effective in reducing or preventing it and it is hoped to put it to the test for the purpose of settling the point more than with any hope of providing a practical means of preparing fine powders that shall be chemically unaltered. One test has already been made of grinding the siderite already mentioned in an atmosphere of carbon dioxide, which is suggestive, though not conclusive, because it was found impossible under the conditions to secure complete exclusion of air from the box in which the grinding was done. After two hours' grinding and subsequent thorough exposure to the air, the powder was darker than the unground material or than that ground under alcohol, but decidedly lighter in color than that which had been ground for only one hour in full contact with air. The inference is that local heating plays an important part in promoting oxidation. That an inert atmosphere would not be effective in preventing absorption of water was apparent and was clearly shown by experiments 5*f* and 6, *d*, *e*, in which the grinding was done mechanically in an atmosphere dried by sulphuric acid, the mortar with its coarse powder having been placed in its housing together with vessels of sulphuric acid several hours before the grinding commenced and a current of dry air passing through during the whole of the operation. The powder (6*e*) was then, after exposure to the air of the room for several hours, found to contain nearly as much water, loosely and firmly held, as when ground by the same machine for the same length of time in undried air. That the amounts were not fully as great is of little importance in view of the varying effectiveness of the different grinders, and even of the same grinder on different days.

Effect of Grinding under Liquids.

The results of grinding under different liquids are of interest, though not conclusive by reason of the limited number of tests that have been made. The results referred to in the foregoing paragraph rendered it unlikely that with the same degree of comminution there would be any material decrease in the amount of absorbed water, and experiments served only to confirm this view. They did show, however, that the effect was to materially reduce the oxidation of the iron, water being in this respect apparently somewhat less effective than absolute alcohol or carbon tetrachloride. A striking effect of the carbon tetrachloride may be mentioned. The mineral powder in contact with it becomes highly flocculated and settles out at once when the grinding is interrupted, leaving a perfectly clear liquid above, in marked contrast to the lack of such effect with water and alcohol. As between the two named organic liquids, alcohol seems to be the more effective in preventing oxidation (2*b*, *c*, 5*b*, *c*, 10*b*, *c*) if we assume that the same degree of subdivision was reached with both in like periods of time, therefore the tests with carbon tetrachloride were not multiplied.

When alcohol was used, in most cases (1*b*, 3*b*, 10*b*, 12*b*) oxidation seems not to have taken place, even after two hours of continuous operation of the machine in the last two instances, at least not to a determinable extent. This again seems to be rather strong evidence that it is not increased surface exposure merely that gives rise to the marked oxidation of FeO when ground in air, but that local heating of the grains in contact with oxygen as they undergo crushing is the important factor. Were it not so, it would seem as if the iron should undergo oxidation after removal of the alcohol and exposure to the air, just as it then takes up water, but this it does not seem to do. In 2*b*, 5*b*, 9*b* for no evident reason the alcohol seems to have been less effective, taking always the FeO content of the crushed but unground bulk sample as the standard to go by. On the whole this method of securing a fine sample does not altogether commend itself, since, while eminently satisfactory sometimes, the error affecting it is appreciable at other times. Nevertheless it may give good service on occasion, hence the procedure to be followed in using it will be given. The coarse powder is covered in the mortar with absolute alcohol that leaves no residue whatsoever on evaporation. The amount used should not be more than enough to form a very liquid emulsion during the operation of grinding. One or two additions of alcohol may be needed if the grinding has to be of long duration. The grinding should be continued in any case till the mass becomes somewhat thick, then the mortar and pestle are left to dry by unaided evaporation of the excess of alcohol, a wide glass being supported above the mortar to keep out dust without too much impeding air circulation. When supposed to be thoroughly dry, the powder is transferred in its entirety to a watch glass and placed on the balance pan, where it is to be kept till it is quite evident that no further loss in weight takes place. It is then transferred to a sample tube.

Application of the Foregoing Results to Mineral Analysis.

It is plain from what has been said that nearly all mineral analyses that have been made in the past are affected by more or less serious errors in respect to water and the oxides of iron, the error being greater the farther the comminution of the sample was carried. Very many analyses show more ferric iron in minerals that should apparently contain none, and small amounts of water for which there seems to be no place in any probable formula. These are discordances for which a simple and in most cases probable explanation now offers. A revision of much past analytical work, particularly that relating to iron-bearing minerals, is called for, but before it can be properly done, adequate provision must be made to avoid the old errors. In the paragraphs following will be set forth the procedures that seem best adapted to secure this end, in the light of present experience. It is perhaps advisable to add here, in order to avoid misconception, that the errors in past work are not as a rule to be assumed equal to those in the above table that resulted from grinding for two hours or more, for few, if any, analysts have made it a rule to grind for such a length of time.

Treatment of Sample in First Stage of Analysis.—The mineral powder, prepared by careful crushing without grinding (p. 1123) serves as the starting point. If it is possible to effect complete or nearly complete decomposition within 30 minutes by dilute hydrofluoric acid with powder so coarse as just to pass a sieve of 80 meshes to the linear inch (about 0.20 mm. diameter of apertures), a powder of this degree of fineness should be used in preference to one finer. In general, such a powder can be used without grinding for all determinations that involve fusion with an alkali carbonate, but not always. If finer grinding, either in air or under alcohol (p. 1129) is imperative, it will be necessary with the ferrous iron portion, when there is enough of this constituent to make an appreciable difference, to make water determinations on the finely ground as well as on the coarse powder, in order to be able to reduce the results obtained for the ferrous oxide to the weight represented by the coarse powder. For other constituents, as the alkalies, this course will not generally have to be followed; it will be sufficient to take of the coarse sample a weight called for by the particular determination and to grind this in a large mortar with extreme care to avoid loss by dusting or flying of grains and then to cleanse the mortar by grinding in it a little of the flux that is to be subsequently employed or of some harmless material other than a flux, the rinsings being added of course to the main portion of the fine powder.

For the ferrous iron determination two courses are open: (*a*) to grind under alcohol until a degree of fineness is reached that will surely admit of complete decomposition by boiling dilute hydrofluoric acid within 15 or 20 minutes; (*b*) to treat the coarse powder directly with hydrofluoric acid. If a single treatment by (*b*) does not effect complete decomposition, and this will be very often the case, the procedure that has thus far commended itself is the following: Having titrated (according to the directions in *Bull.* **305**, *U. S. Geol. Survey*, p. 142) with permanganate to the first more or less transient pink blush throughout the whole body of liquid, let settle, decant and wash once by decantation with water, rinse the residue into a small agate mortar, decant most of the water after again allowing to settle, grind the residue for a short time, under what water remains, transfer it by a stream of hot water to the large crucible from which it was taken and repeat the treatment by hydrofluoric and sulphuric acids used in smaller amounts than the first time. The final titration may be made in the crucible itself if this is of at least 80 cc. capacity and the bulk of liquid in it is small, after quickly diluting with cold water till the crucible is nearly full.

This manner of treatment has afforded thus far satisfactory results and is in most cases to be preferred to that given as (*a*). Not only are the double determinations of water avoided, but also the error that often arises from oxidation by long grinding under alcohol. The very short grinding under water of the small fraction of the original iron-bearing mineral or minerals that remains after the first treatment with hydrofluoric acid can not conceivably lead to any detectable error.

In conclusion a few remarks are in place regarding the comparative merits of the Pratt and Cooke methods of decomposing minerals for the ferrous iron determination. In *Bull.* **305**, p. 138, it was said that indications seemed to slightly favor the former method, and this expression is strengthened by the comparative data contained in the foregoing table on pp. 1124–26. Although in some instances (2, *b*, *e*, 5, *a*, *b*, *d*) the two methods appear to be of equal merit, in others (2, *a*, *f*) they differ materially in favor of that of Pratt, and in the rest there is evident a general tendency toward lower results by that of Cooke.

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A NEW APPARATUS FOR THE QUANTITATIVE DISTILLATION OF AMMONIA.

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In *Z. physiol. Chem.*, **37**, 161, Folin describes a new method for the determination of free ammonia in physiological solutions, or solutions which decompose easily when subjected to any temperature above 50°. Essentially, his method consists of passing a current of air through the solution whose ammonia is to be determined and through a standard acid solution, which is then titrated back with a standard alkali solution.