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EDITORIALS.

INDUSTRIAL INCOMPATIBILITIES.

THE physician and the pharmacist are vitally concerned with the subject of incompatibility as applied to substances in the materia medica, and they must be on guard against therapeutical, pharmaceutical and chemical incompatibilities if their prescribing and dispensing are to be productive of good. A thorough knowledge of the properties of their medicines and the proper application of chemical and physical principles have led them to the refined pharmaceutical products of to-day products that contrast strongly with the crude medicaments of a century ago.

Is there not something of the same sort in the industrial world? One does not have to go very far back into the history of manufacturing processes to discover marked "incompatibilities" among industries. The introduction of the Le Blanc soda process resulted in the formation of large quantities of hydrogen chloride which simply escaped through the chimneys and caused great damage to the surrounding country; the ammonia liquor and tar from gas works found their way into drains and rivers; refuse from slaughterhouses constituted no mean nuisance; and even the offal and waste from the houses of a mediaeval city became a source of infection. Thus air, water and the very earth itself were rendered unfit for many industries and offensive to the senses of man.

But with the pressure of stern necessity for improved conditions laid upon the manufacturer, and with the growth of new industries, there were developed ways for utilizing the hydrochloric acid, ammonia, tar, packing-house refuse, household garbage and even sewage. The chemical fertilizer, made from factory wastes that had been a menace to agriculture, proved to be the salvation of exhausted farm land; and purified ammonia and coal-tar colors advanced the art of the bleacher and dyer who had complained about the pollution of his water supplies by gas works refuse. Industrial incompatibility gave way before technical progress.

Similar results can be expected in the cases of the industrial incompatibilities that remain. The elimination of the coal smoke nuisance, the conversion of smelter gases into valuable products, the proper treatment of mine waters, the cure of the dust evil at manufacturing plants of various kinds, are problems by no means incapable of solution in the near future. As heretofore industrial incompatibility will be replaced by industrial interdependence, but meanwhile the principle of "live and let live" must be exercised or the law of nuisance invoked.

W. C. EBAUGH.

COMMITTEES.

THE man who remarked very thoughtfully that a committee was a body which took a week to do something which could be done by a good man in an hour, was nearly correct. And yet the necessity for committee work has increased and is sure to increase in the future with a civilization which is developing along coöperative lines. Committee work is in its nature coöperative even when directed or dominated by an individual.

For the chemist and the engineer the greatest need for committee work has been for the purpose of standardizing and developing methods of testing, sampling and analysis. Various chemical and engineering societies have appointed such committees from time to time as occasion seemed to require. Some of these committees reported in due time; others, either from the complexity of the problem laid before them or because of lack of enthusiasm for the work, more than lived up to the pessimistic definition given above and never reported at all. On the whole, the most systematic work of this sort which has heretofore been performed, must be credited to the Association of Official Agricultural Chemists. Beyond question this organization, through its referees, has developed and systematized more methods (and these in a difficult field of analysis) than any other single chemical society in this country or in the world. In a different field the American Society for Testing Materials and the International Society are doing excellent work. The American Chemical Society has done much in a few limited fields to further the work. The Geological Survey and the Bureau of Standards have contributed. So have the American Foundrymen's Association and the American Brass Founders' Association. The National Fertilizer Association has had a committee at work upon methods of fertilizer analysis, the American Leather Chemists' Association has worked on methods of interest to its members, the United States Steel Corporation has a committee on steel and ore analysis. The latest development is the coöperative committee, consisting of committees appointed by two or more societies, working together on the same subject.

The necessity for committee work being granted and the desirability of perfected and codified methods of testing, sampling and analysis being manifest, nevertheless is there not a certain amount of danger here in having so many committees working on similar subjects? To be sure, up to the present time there has not been much duplication of work or overlapping of territory but there has been some. The fact that so many organizations of diverse character feel at liberty to appoint committees on the subject carries with it the certainty that duplication or conflict will ensue as time goes on. There is also the danger of having too many chemical cooks in the kitchen. It would

be unfortunate if any perfunctory or ill-considered work were to be done in the building up of standard methods. There have been indications during recent months that certain referees and committees were working more in the direction of issuing reports than of performing necessary and useful. work, and of adding to the present difficulties by the addition of new methods no better than the old, rather than by the elimination of all but the best. The official recognition of a method no better than the current one or only "just as good" is hardly justifiable. It adds to the confusion. Improvement, simplification, and elimination are the things to be desired. Also the centralization of the work in the hands of a few recognized and representative organizations.

ORIGINAL PAPERS

THE ASH OF COAL AND ITS RELATION TO ACTUAL OR UNIT COAL VALUES.

By S. W. PARR AND W. F. WHEELER. Received July 1, 1909.

In the report of the committee of the American Chemical Society on coal analysis,¹ occurs this question: "Are carbonates likely to be present in the ash in such amounts that heating over a blast lamp would lessen the weight appreciably?"

In studying the properties of the organic constituents or actual coal substance as distinct from the inorganic material, it was found necessary to scrutinize more carefully the methods for obtaining these inorganic values. In directing our attention to the ash itself, considerable evidence accumulated to the effect that carbonate of lime was a constituent part of the coal. In taking up this matter, therefore, with a view to determining the extent and quantity of this material, fifty samples of Illinois coal were selected covering all the important districts of the state. An analysis was made of the carbonate content of each and a further analysis was made of the mineral constituents of the ash. Test was also made as to the presence of chlorine and this element was determined in addition. The methods employed were those already well known and familiar to all chemists. In the case of chlorine, a five-gram sample of coal, ground to pass a 100mesh sieve, was digested with water and filtered and this filtrate taken for the determination of

¹ Jour. Am. Chem. Soc., 20, 284 (1898).

chlorine by the usual volumetric process by means of a standard solution of silver nitrate. It was only necessary to guard against acidity and in some instances the iron was present to an extent requiring its removal. In the case of the carbonates, a determination was made of the amount of carbon dioxide present by liberating the same with acid and determining the amount volumetrically. For this purpose the apparatus shown herewith (Fig. 1) was used.



Fig. 1.

This is a modification of the apparatus already described in the determination of total carbon in coal, soils, etc.¹ By this apparatus the carbon dioxide liberated is absorbed by the large pipette, P (Fig. 1) and the residual gas returned to the burette for reading. The contraction in volume thus indicates the amount of CO_2 present, and this volume is transferred to gravimetric terms by reference to the table recently published in this Journal² The entire list of values for the

¹ Jour. Am. Chem. Soc., **26**, 294 (1904). ² Ibid., **31**, 237 (1909). fifty samples is published in *Bulletin*, —— of the Engineering Experiment Station of the University of Illinois under the title, "Unit Coal and the Composition of Ash."¹

Only fifteen of the samples are selected for reproduction here. These consist of numbers giving the higher percentages of carbon dioxide and of chlorine. It should be said, however, that while the remaining samples fall below one per cent. of carbon dioxide, the average for the other samples would be considerably over 0.5 per cent. If we consider all the carbon dioxide present as being combined in the form of calcium carbonate it is readily seen that in not a few instances, coal, at least from the Illinois field, may have an amount of this constituent ranging from three to five per cent.

TABLE I-ANALYSIS OF COAL ASH. Description. Dry coal basis. usual . Per cent. fused ash. cent Per cent. by thod. Per Seam No. No. No. Ash metl cent. CaO. in fu Tab. CO2. L,ab. ci. Location. 1* 734 Coal City, Grundy Co..... 2 5.82 0.88 None 34.0 2 1411 Hollis, Peoria Co..... 2 10.90 0.98 0.27 18.6 896 Spillerton, Williamson Co..... 5 10.68 0.70 0.31 3 3.9 4* 1085 Eldorado, Saline Co..... 5 11.49 1.22 0.04 25.3 5 1116 Ledford, Saline Co..... 5 9.89 1.51 0.05 26.5 6 720 Lincoln, Sangamon Co..... 5 13.81 0.54 0.32 30.8 54 5 22.4 1407 Edwards Sta., Peoria Co..... 5 16.25 2.15 0.03 9 31.1 10 1410 Maxwell, Peoria Co..... 5 14.78 1.57 None 31.3 1413 Pekin, Tazewell Co..... 5 ... 1.36 None 11 31.6 557 Westville, Vermillion Co..... 5 9.15 1.08 0.15 12 5.6 13* 1178 Trenton, Clinton Co..... 6 15.56 2.48 0.10 31.7 420 Zeigler, Franklin Co..... 14 7 7.53 0.21 0.55 6.3 ... 0.28 0.47 422 DuQuoin, Perry Co..... 7 15 6.7 16 1120 Gelatia, Saline Co..... 7 ... 1.20 0.07 11.6

In the further prosecution of our studies, it was desired that an accentuation of these carbonate conditions be obtained which was done in the following manner: Four samples were selected from among the lot of fifty and these are indicated by stars in Table I. These samples in buckwheat size were subjected to the floating process wherein a solution of calcium chloride, 1.35 specific gravity, was used. By this means those portions of coal low in ash and high in organic constituents would rise to the surface. Each sample of coal was thus made to yield two subdivisions, one very low and the other very high in ash. Table II will illustrate this fact. By subjecting these artifically prepared samples to the same process of analysis the chlorine and carbon dioxide values are at once shown. It

¹ Bull. -, E. E. Sta., S. W. Parr and W. F. Wheeler.

is evident from an examination of the column for chlorine that this material was not completely washed out after application of the floating process, since the percentage amount is practically the same in both divisions and altogether higher in amount than the normal quantity in the coal, as is evident by reference to the values in Table I.

	TA	BLE II-ASH CONSTITUENTS OF F	LOAT A	ND SIN	к Солі	
			Dry o	coal ba	sis.	
ab. No.	ab. No.		sh by usual method. Per cent.	02. Per cent.	l. Per cent.]	aO. MgO in fused ash, per cent.
H	H	Description.	•	U	0	U
1	734	Float, Grundy Co., Ill	4.57	0.10	1.00	0.85
2	734	Sink, Grundy Co., Ill	21.99	3.68	1.15	6.50
3	1095	Float, Saline Co., Ill	6.42	0.13	0.17	0.40
4	1095	Sink, Saline Co., Ill	19.94	2.38	0.31	3.88
5	1178	Float, Clinton Co., Ill	8.54	0.21	1.41	0.88
6	1178	Sink, Clinton Co., Ill	31.90	5.27	1.20	8.36
7	1403	Float, Peoria Co., Ill	10.37	0.37	1.07	1.29
8	1403	Sink, Peoria Co., Ill	34.24	7.35	1.17	10.20

It is evident that in these samples we have an excellent opportunity for testing the effect of a high content of calcium carbonate upon the ash as determined by a use of higher or lower temperature. Determinations were therefore made in the usual manner and also by carrying the heat by means of a blast lamp to the fusion point and repeated until a constant weight was obtained. The results are given in Table III.

TABLE III-VARIATIONS IN ASH VALUES WHERE CALCIUM CARBONATE IS A CONSTITUENT OF THE COAL.

ľab. No.	,ab. No.	Description	Ash as deter- nined by usual nethod. Per p	Ash as deter- nined by blast- ng to constant or ng to constant or u eight and usion. Per cent.	Difference in non- percent of dry noal. Per cent.	Ash corrected or CO2 and Cl. p. Per cent.
	734	Float Grundy Co. Ill	4 57	3 54	1 03	1 64
2	734	Sink Grundy Co. Ill	21 00	16.85	5 14	21 68
-	754	blik, orandy co., m	-1.75	10.00	3.14	21.00
3	1095	Float, Saline Co., Ill	6.42	5.96	0.46	6.26
4	1095	Sink, Saline Co., Ill	19.94	18.59	1.35	21.28
5	1178	Float, Clinton Co., Ill	8.54	7.23	1.31	8.85
6	1178	Sink, Clinton Co., Ill	31.90	26.88	5.02	33.35
7	1403	Float, Peoria Co., Ill	10.37	9.08	1.29	10.52
8	1403	Sink, Peoria Co., Ill	34.24	22.93	11.31	31.45

In this table there is presented the differences which result from these two methods. It becomes, therefore, an answer to the question raised by the committee as to the effect of heating over a blast lamp and the consequent lessening of weight. It is true these samples are exaggerated cases, but it is none the less true that for the purposes for which these samples were being used, namely, a study of the actual coal constituents, it was necessary that a correct determination of the ash be secured. The first evidence of difficulty occurred in all cases where the high ash content was present, or in the "sink" sample, by reason of the uncertainty involved in securing concordant weights in the duplicate determinations. Evidently the application of a little more or a little less heat results in the decomposition of more or less of the calcium carbonate. However, under column (a) the ash as determined by the usual method was obtained with as good an agreement as possible, although a variation of two per cent. in results was frequently met with by this method. In column (b) the heat was carried to the point of fusion and the application of the blast was repeated until a constant weight was secured. It is certainly a striking illustration of the possible difference in results when we examine, for example, samples No. 8 of Table I and see a variation in values from 34.24 per cent. to 22.93 per cent., or a difference of over 11 per cent. in the value for ash as obtained by the two processes. Similarly unallowable, though not such extreme differences are found in the other high carbonate samples of the table. It must be obvious from such values that some other method must be resorted to for securing the correct ash values if we are to arrive at any correct conclusions in our study of the actual or organic part of the coal. The method followed in these samples of Table III was to carry the high temperature in a platinum crucible to the fusion point or to the point of constant weight. By then adding to the weight of ash thus obtained the percentage of carbon dioxide as obtained by the process already outlined and also the weight of chlorine, a percentage of ash was secured quite different from any of the values indicated in Table II or III. These values are shown in column (d) and consist of the values in column (b) plus the carbon dioxide and the chlorine indicated in Table II.

The results of the facts thus far set forth are somewhat disconcerting to some of our established methods, notably the values obtained by ultimate analysis. In the case of a coal with one or two per cent. of carbon dioxide in the form of calcium carbonate the total carbon as obtained by means of the combustion furnace would be high and the error thus involved would extend throughout all the determinations, such as the amount of oxygen, available hydrogen and the calculation of the heat values by DuLong's formula. Moreover, the error involved in the ash determination itself would affect the calculations to the "ash and water-free" basis or other methods for arriving at so-called "pure coal" values.

ACTUAL OR UNIT COAL.

As has already been indicated the initial necessity for determining the correct value for ash and inorganic constituents resulted from our studies upon the character and constancy of the composition of the organic or actual coal constituents. If, for example, it is conceded that in a given sample of coal which had been subjected to the floating out process the organic or actual coal of the "float" part is substantially the same and would have the same heat value as the organic constituents of "sink" coal, then when reduced to this "pure coal" basis the heat values should closely correspond. If there is wide disagreement, then we must conclude that our values for ash are in error, and that a correct ash or inorganic factor would bring these values when calculated to the "pure coal" basis in substantial agreement. As illustrating this point, Table IV is presented which takes the heat values as indicated on the "dry coal" basis and calculates them to the "ash free" condition, taking the ash value as obtained by blasting to a constant weight as already indicated in Table III. The heat values thus obtained are so widely apart as to indicate at once a wide error in our ash values.

TABLE IV "UNIT	COAL"	IN	SAMPLES	WITH	HIGH	CALCIUM	CARBON-	
			The second s					

ATE. In per cent, of dry coal

			A CONTRACTOR			STATE STATE		
Tab. No.	I,ab. No.	Description.	Ash as deter- mined by high fusion to con- stant weight.	co ₂ .	ci.	B. T. U.	Pure coa B. T. U I.00 — as	r.] Diff.
1	734	Float	3.54	0.10	1.00	13475	13969	
2	734	Sink	16.85	3.68	1.15	10733	12908	1061
3	1095	Float	5.96	0.13	0.17	13663	14528	
4	1095	Sink	18.59	2.38	0.31	11122	13662	866
5	1178	Float	7.23	0.21	1.41	12634	13618	
6	1178	Sink	26.88	5.27	1.20	8856	12111	1507
7	1403	Float	9.08	0.37	1.07	12796	14073	
8	1403	Sink	22.93	7.35	1.17	9216	11941	2132

It is to be noted that these differences range from over 800 to over 2000 B. T. U., which is a difference of from 6 to 15 per cent. Aside from its bearing upon the method of ash determination, the subject of the heat values to be attributed to the values of actual or unit coal is one of great importance, not only in the scientific study of this subject, but in its practical bearing in connection with coal contracts and coal inspection work.

That the importance of this matter has long been recognized may be seen from reference to the work of Lord and Haas,1 who were the first to call attention to the matter and gave extended analytical data to prove the constancy of heat values over large areas when referred to the actual or unit coal. W. A. Noves² called attention to similar conclusions concerning the Indiana field. The practical value of this fact as substantiated, especially in the matter of coal inspection, has been urged by Mr. Bement.3 A true appreciation of the practical importance of the subject seems. therefore, to have been revived by him. A point at issue, however, has been raised as to the method of arriving at these so-called "pure coal" values. A graphical method has been suggested by Taylor and Brinsmaid.⁴ Although indirect and resting entirely on empirical data for a given region. it is no doubt of much practical value. It has been the effort of the authors of this paper, in so far as it is possible, to arrive by direct analysis at the inorganic constituents and, as contributing to this end, the determination of the carbon dioxide and the chlorine content are in evidence. In combating the method ordinarily employed for arriving at the heat values on the "pure coal" basis, it was argued by Parr⁵ that not only was the sulphur a disturbing element in the case, but certain volatile elements were present which escaped determination as part of the ash and were therefore included in the actual coal, thus introducing a variable which prevented the obtaining of accurate values for that substance. For example, if the shaley constituent of the ash is hydrated, or if other volatile material is chemically combined with the mineral or ash substance in such a manner that it would be driven off only at red heat, then an error in calculation would result equivalent to the amount of such hydration. Analytical data in support of this contention has been furnished by Mr. Wheeler,6 and it is the purpose of this part of the discussion to carry forward the evidence along this line. As the most suitable medium for arriving at the point in question the samples already referred to as being separated into "float" and "sink" divisions or subdivisions of the same coal into samples of low and high ash

- ⁵ Jour. Western Soc. of Eng., 2, 1762 (1906).
- ⁶ Trans. Am. Inst. Mining Engs., 38, 621 (1908).

¹ Trans. Am. Inst. Mining Engs., 27, 259 (1898).

² Jour. Am. Chem. Soc., 20, 285 (1898).

³ Ibid., 26, 636 (1906).

⁴ THIS JOURNAL, 1, 65 (1909).

content, a number of coals have been so treated and results are given in Table V.

Now, upon the hypothesis that the actual or unit coal in these two divisions of the same sample should have the same heat value, we have listed under columns (a), (b), (c) and (d) the values obtained by different methods of calculation, on the theory that those which most closely agree in the two divisions of the same sample have arrived the most closely in the percentage values to be attributed to the ash or inorganic substance.

If we credit, for example, to the actual coal everything excepting the ash as weighed, and the moisture, we shall have unit values as shown under column (a). This is the "pure coal" of Bement.

If we take out the heat due to sulphur and correct the remaining value for ash as determined plus the moisture plus all of the sulphur, we shall have the value as derived for column (b), which would be the results obtained by means of the method suggested by Lord and Haas.

Under column (c) we have the method suggested by Dr. Noyes, the results of which are obtained by calculating the indicated values over to the material as free from ash and moisture and correcting the ash further for one-half of the sulphur.

In column (d) there is introduced as a natural component of the ash an amount of hydration equal to 8 per cent. of the ash. There is a further correction for sulphur which in the original coal is considered as in the form of iron pyrites. A tabulation of these several methods of calculation, therefore, would be as follows:

(a) According to Bement,

B. T. U. as indicated 1.00 (Moisture + ash as weighed)

(b) According to Lord and Haas,

B. T. U. - 4050 S.

1.00 — (Moisture + ash as weighed + sulphur)

(c) According to Noyes,

B. T. U. as indicated

1.00 - (Moisture + ash as weighed + 1/2 sulphur)

(d) According to Parr and Wheeler,

B. T. U. - 5000 S

1.00 - (Moisture + ash + 5/8 S + 0.08(ash - 10/8 S)

The method of deriving a formula embodying the conditions prescribed under (d) would be as follows:

First, with reference to the subtraction of the

heat due to the sulphur. It should be borne in mind that the purpose of this study is, first to arrive at the actual weight of unit coal as represented by the expression 1.00-(all non-coal constituents); and second, to derive the actual heat per unit weight to be credited to this material, by dividing the indicated heat for this substance by the weight which produces it. Hence, for this particular purpose the sulphur must be eliminated, both as to its heat value and as to its weight in the material whose value is sought for. This procedure may not suit the purpose of the engineer who has in mind only the available heat without reference to its source, but that is a matter quite apart from the facts which it is the purpose of this discussion to establish.

Second, the expression 5000 S has been used as indicating the heat due to the combustion of the sulphur for the reason that the value 4050 S as used in formula (b) represents the heat of combustion for pure sulphur, while the heat of combustion of sulphur in the form of iron pyrites, FeS_2 , combines also the heat of formation of iron oxide, Fe_2O_3 . It is the resultant value, therefore, of the several reactions involved that is desired.

According to direct tests by Somermeier,1 in the combustion of coal with known weight of iron pyrites, the indicated heat per gram of sulphur so combined is 4957 calories. In calculating heat values the correction introduced for the combinations resulting from calorimeter reactions as compared with open-air combustion is 2042 calories per gram of pyrite sulphur, hence 4957-2042 or 2915 calories (5247 B. T. U.) represent the heat due to the burning of one gram of sulphur in pyritic form instead of 2250 calories (4050 B. T. U.), the amount which would be credited to sulphur in the free condition. A strict application of these values, therefore, would call for a correction of 5247 S as representing the heat to be subtracted for the sulphur. This, however, would imply that all of the sulphur is in the pyritic form. Since a certain portion of the sulphur is always present in the organic or other form of less heat-producing capacity, it is deemed more nearly correct to use an even factor of 5000 as representing the heat to be credited to unit amounts of the total sulphur present.

The factors for the divisor in the formula under (d) are derived as follows:

¹ Jour. Am. Chem. Soc., 26, 566.

The atomic ratio of iron to sulphur in iron pyrites (FeS_2) is 56:64.

That is, 7/8 of the total sulphur is the equivalent of the iron present as Fe.

The atomic ratio of the oxygen of the ash combined as Fe_2O_3 to the total sulphur which it replaces is 48: 128.

That is, 3/8 of the total sulphur is the equivalent of the oxygen present in the ash combined as Fe_2O_3 , hence the ash as weighed may be corrected for the iron pyrites FeS_2 burned to Fe_2O_3 by subtracting from the ash 10/8 of the weight of the sulphur as determined. This remainder, therefore, is considered as the shaley and carbonate constituent upon which the 8 per cent. of water of hydration, carbon dioxide, etc., is calculated. The expression for the total non-coal substance then becomes

Non-coal = moisture + ash as weighed + 5/8 S + 0.08 (ash - 10/8 S).

This expression can be simplified as follows:

Clearing of fractions and combining we have

Non-coal = moisture + 1.08 ash + 21/40 S.

In this expression the factor 21/40 S can not be further simplified by making it 1/2 S for the reason that our correction for sulphur is already too small by that part of the organic sulphur not covered by the addition to the ash value of 3/8 of the total sulphur indicated in the original formula. On the contrary, we will be approaching nearer the truth by increasing slightly the sulphur correction which may be done with convenience in calculating by making this factor read 22/40 S or 1/2 S + 1/20 S.

Hence the simplication of the entire formula under (d) would be

B. T. U. or unit
$$coal =$$

Indicated B. T. U. -5000 S.

1.00 - (Moisture + 1.08 ash + 1/2 S + 1/20 S).

Since the analytical values given in the table are based upon the coal as oven-dry, of course the moisture factors in the above formulae drop out and would not enter into the calculations. In the table, for example, sample No. 1 has an indicated B. T. U. for the dry coal of 12356. The calculations, therefore, for each column are:

(a)	$=\frac{12350}{1.00-0.1166}$	1	•		 	 	 •		•••	•	 日本・	· · · ·	••••		•	I	3,98	87	

$$(b) = \frac{12350 - 4050 (0.0599)}{1.00 - (0.1166 + 0.0599)} = \dots \dots 14,709$$

$$(c) = \frac{12356}{1.00 - (0.1166 + 0.0299)} = \dots 14,477$$

12356 - 5000 (0.0590)

$$d) = \frac{1000 - (1.08(0.1166) + 0.02995 + 0.00299)}{14,331} = 14,331$$

In Table V, which follows, the actual or unit coal values for the two subdivisions of each sample are in such close agreement under the last formula the values for which are given in column (d) that it would seem fair to conclude that a correct factor for the inorganic constituents is represented by the factors in that formula. This is true at least for the samples chosen.

TABLE V-COMPARATIVE VALUES OF "ACTUAL COAL." Heat values of "actual coal" as calculated by different methods.

						and the second second second	Poner States	Charles and the second	ATHICS PART	The second second second	Charles of Chapters	ALL BELLEVILLE	State (Bull screek)
the No.	b. No.		Ove	en-dry c	oal.	(a Non-ce ash only corrected to indica T. U.	z) pal as v (un- l) Ref. ted B.	(b) Non-c ash (unce ed) + su ref. to B. -4050 S.	oal as orrect- ulphur T. U.	(c) Non-co ash + 1/2 phur ref. T. U. as cated.) sul- to B. indi-	(d Non-co 1.08 × a 22/40 S. 1 B.T.U) sh + Ref. to 5000 S.
T.8	L,a	Description of sample.	Ash. S	ulphur.	B.T.U.		Diff.		Diff.		Diff.		Diff.
1	6130	Sangamon County, Ill. Lump coal:											
		(a) Untreated	11.66	5.99	12356	13987		14709		14477		14331	
	6131	(b) Floated, sp. gr. less than 1.35	6.12	3.20	13300	14164	+177	14523		14412	65	14340	+9
2	6129	LaSalle County, Ill. Washed screenings:											
		(a) Untreated	10.05	3.43	12885	14316		14616		14602		14566	
		(b) Floated, sp. gr. less than 1.35	3.94	2.33	13922	14487	+171	14754	+138	14680	+78	14615	+49
3	6132	Virgo County, Indiana. Nut:											
		(a) Untreated	16.84	7.62	11790	14170		15230		14858		14698	
	6133	(b) Floated, sp. gr. less than 1.35	4.27	3.08	13870	14478	+308	14836	-394	14725	-133	14638	60
4	6135	Sullivan County, Indiana. Lump:											
		(a) Untreated	6.11	3.37	13664	14551		14944		14819		14741	
	6134	(b) Floated, sp. gr. less than 1.35	2.53	1.29	14259	14624	+73	14771	-173	14820	+1	14700	-41
5	471	Franklin County, Ill. Face sample:											
		(a) Sp. gr. greater than 1.35	18.00	0.57	11639	14194		14236		14244		14467	
	463	(b) Sp. gr. less than 1.35	4.64	0.54	13765	14435	+241	14492	+229	14476	+232	14513	+46
6	472	Perry County, Ill. Face sample:											
		(a) Sp. gr. greater than 1.35	22.17	1.15	10922	14033		14183		14136		14405	
	465	(b) Sp. gr. less than 1.35	4.22	0.86	13763	14369	+336	14464	+281	14434	+298	14446	+41
7	473	Williamson County, Ill. Face sample:											
		(a) Sp. gr. greater than 1.35	17.75	1.15	11766	14306		14451		14405		14599	
	467	(b) Sp. gr. less than 1.35	4.08	0.99	13942	14535	+229	14644	+193	14617	+212	14615	+16

An extended compilation¹ has been made covering results from this laboratory and from the United States Geological Survey and also from the Ohio State Survey. The values derived for a given locality or geological seam are in such good agreement as to still further substantiate this method for arriving at the true ash values. There is thus afforded, moreover, a method for arriving directly at the amount, and consequently the heat value, of that portion of the coal substance which we have designated as actual or unit coal.

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THE TECHNICAL APPLICATION OF TITANIUM.

By ISADOR LADOFF. Received July 17, 1909.

Titanium so far has found only one application; namely, coloring porcelain, artificial teeth, leather and textiles.

Almost ten years ago an English firm made the first attempt to utilize titanium compounds for coloring purposes. The process used by that firm was as follows: Twelve pounds of caustic soda were melted in an iron kettle of three and onehalf gallons, over a coke fire. The molten mass was left for a time at dark red heat and three pounds of ground rutile containing 80-90 per cent. titanic acid (TiO₂) were added in small portions. The mass was mixed with an iron rod frequently and kept at a high temperature during an hour. The fused conglomerate was poured on an iron slab and allowed to cool. The brittle mass was then pulverized and digested with cold water during 48 hours and stirred at intervals. The insoluble portion was freed by frequent change of water and filtration. The superfluous caustic was removed, while the hydrated oxide of titanium remained. The washed and filtered oxide was digested in 2 3/4 gallons of cold 30 per cent. hydrochloric acid during one to two days, and frequently stirred. During this process the temperature was not allowed to exceed 38° C. The hydrochloric acid solution was then diluted with 2-3 times its quantity of water and poured into a saturated solution of caustic soda, ammonia or other alkali, so as to show an alkaline reaction. The titanium was this way precipitated as a hydroxide contaminated with some iron. In order to purify it from iron, two pounds of sulphate of sodium were added in

crystals. The hydroxide of iron was this way converted into a sulphide and the color of the precipitate turned into a dark gray. The precipitate was thrown on the filter, washed twice with water to remove the superfluous sulphate of sodium; sulphurous acid was then applied for the purpose of decomposing the iron. The remaining hydroxide of titanium was then dried and ignited.

*

To the titanic acid was added four times its weight of crystalline oxalic acid and the mixture dissolved in as little hot water as practicable. A clear solution followed. The solution was evaporated in earthenware dishes or metallic dishes covered with lead to crystallization and cooled. The crystals were then drained from the mother liquor. To the titanium oxalate remaining in the motherliquor a one and a half times larger quantity of acid oxalate of ammonium dissolved in hot water was added and the fluid concentrated to a syrupy condition. This then was allowed to solidify on cooling into a crystalline mass, containing besides titanium ammonium oxalate an acid titanium oxalate and some free oxalic acid.

In order to get 120 pounds of this crystalline mass the following ingredients were used:

30 lbs. rutile (80-90% TiO2).

120 lbs. caustic sodium.

- 20 lbs. crystalline sulphate of sodium.
- 27 gals. hydrochloric acid of 30%.
- 96 lbs. crystalline oxalic acid.
- 38 fbs. crystalline ammonium oxalate.

The wash-water from the rutile flux contained enough alkali to precipitate the hydroxide from the hydrochloric acid solution. The oxalic acid regained by partial evaporization and crystallization amounted to about 25–26 lbs. This was only about 70 lbs. that had to be used in the continuous process of production.

The price of titanium in the ore was about the same as of solid tin metal, *i. e.*, too high if we take into consideration the complicated process of manufacture and purification.

H. W. Kearns and Dr. Barness applied on Dec. 5, 1896, for letters patent on titanium compounds, tannic compounds and similar compounds for coloring vegetable fibers (cotton, linen, jute, etc.) in raw or manufactured state during any stage of manufacture. Experiments made by Dr. Franz Erben¹ proved that a fiber treated with a tanning compound as a mordant and then subjected to the action of ¹ Chem. Z., No. 14, 1905.

¹ Bull. 37, University of Illinois, Eng. Exp. Sta.

titanium ammonium oxalate furnishes a saturated golden yellow color of great durability withstanding the action of boiling and washing. This coloring is preferable to that produced by chlorinated primalin and even the best sulphur colorings on the market. Dr. C. Dreher¹ is less enthusiastic concerning this tannin-titanium yellow in application to leather. However, Dr. Erben claims a favorable experience during nine years.

By the addition of small quantities of auranein G or O, acid orange or similar products the shades of yellow or orange can be modified at will.

The tannin-titanium base allows the improvements of many other colorings as for instance, red, green, blue, etc.

The man who did most for the introduction of titanium into metallurgical processes is Dr. Auguste J. Rossi.

In his contribution to the *Iron Age*, February 6 and 20, 1896, he summarizes his conclusions concerning titaniferous iron ores as follows:

1. These ores have been certainly smelted in Sweden for years without any difficulty.

2. Their metallurgical treatment for a certain number of years in England by Dr. Forbes, in a large furnace, has proved a perfect success.

3. Furnaces were run for years in the Adirondacks with these ores with excellent results.

4. The metal they yield, either as pig metal, iron or steel, possesses special valuable qualities.

5. These ores, which are found in large masses in many States of the Union, are almost invariably "Bessemer ores," and as such it is asserted have been used in Pennsylvania furnaces with great advantage.

6. When containing very large percentages of titanic acid (as much as 38 to 40 per cent. and even 48.60 per cent., like the ilmenite of Canada), and consequently a very small amount of iron (32 to 35 per cent. or less), their treatment though perfectly successful, metallurgically speaking, has not proved economical as to fuel. However, it would be more proper indeed to call an ore 'like the St. Urbain ore (Canada), which contains 48.6 per cent. of titanic acid, corresponding to 29.1 per cent. titanium and only 28.49 per cent. iron, a titanium ore than to call it an iron ore.

Dr. Forbes' treatment consists in adding to the titaniferous ores as fluxes, limestone and quartz or silica-bearing materials in such quantities as to form, with the titanic acid, a compound reproducing approximately a natural mineral of titanium, known to be fusible at a moderate temperature (3 of scale of Dana), the sphene or titanite, a silico-titanate of lime, containing about 35 per cent. of TiO₂,

25 per cent. to 33 per cent. of lime and 28-35 per cent. of silica. Dr. Rossi's method obviates the addition of silica, taxing the capacity of the furnace to excess. He introduces magnesia to a good amount into the slag by using a magnesia limestone, a dolomite. The alumina from the stone and ash of fuel and that very generally present as principal basic constituent of these ores furnishes all the amount which is required to form the tribasic compound with the magnesia and lime of the stone and the titanic acid of the ore. In the same manner that magnesia introduced in certain proportions into an alumina lime silicate renders the latter more fluid and fusible. the addition of magnesia to a titano-silicate of lime and alumina considerably increases its fusibility and especially its fluidity.

Dr. Rossi ran in a very small furnace, with a blast at a temperature not over 250 or 300° F. at the most, without any difficulty, slags of the following composition: SiO_2 16.63, TiO_2 34.66, CaO 26.03, Al_2O_3 7.26, MgO 10.27, FeO 7.12. Oxygen ratio 4.3 practically; actually 4.31. The ore smelted in that furnace contained only 1.5 to 2 per cent. silica or 20 per cent. titanic acid.

The conclusions arrived at by Dr. Rossi are as follows:

1. Titanic acid can form definite compounds, perfectly fusible, if properly fluxed, containing as much as 35 to 50 per cent. of titanic acid, with alumina, lime and magnesia as bases, and admissible as slags, in blast-furnace work. Larger percentages still, such as 65 per cent., can enter into a compound, and it remains fusible.

2. In running a furnace under special conditions of temperature and pressure of blast, no troubles have been experienced from titanium deposits.

That the smelting of titaniferous ores containing less than 20 per cent. titanic acid by means of an electric furnace is easier, than in an ordinary furnace, was proven by recent investigations, accounts of which were published in the technical literature.

We will refer here to a case not published yet. On December, 1906, Professor W. Borchers, of the Technische Hochschule at Aix La Chapelle, Belgium, prepared a mixture of titaniferous iron, sand of Java with lime and carbon in proportions calculated to produce a scorification of the gangue during the reduction to iron. A resistance furnace was used and a direct current of about 500 amperes with a pressure of 50 to 60 volts was applied. The fusion of the mineral proceeded with the greatest ease. At the expiration of an hour and a half after the operation was started the temperature of the fused mass in the furnace was taken with the aid of Vanner's pyrometer. This temperature was found to be somewhat higher than 1900° C.

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After this operation the slag was transferred to one crucible, while the metal was tapped into another crucible. Both crucibles were of course preheated. The slag was very fluid. The same day about three o'clock P.M. the contents of the cooled-down crucibles were examined.

A solid homogeneous ingot of metal weighing 1 kilogram, 620 grams, was found. The slag was black and had a vitreous fracture.

The analysis of the mineral was as follows:

Silicon	1.6	
Aluminum	traces	
Protoxide of iron 28.5 Fe =	22.18	57 15
Peroxide of iron 49.95 Fe =	34.97	57.15
Oxide of manganese	0.98	
Lime	0.37	
Magnesia	2.35	
Sulphuric acid	0.69	S = 0.027
Phosphoric acid	0.096	P = 0.042
Copper	0.012	
Arsenic	0.005	
Titanic acid	16.00	
Loss on ignition	0.068	
	20- State Barriel	

100.00

The analysis of the metal was as follows:

Carbon, total	3.05
Manganese	1.52
Sulphur	0.01
Phosphor	0.11
Silicon	0.37
ron	94.94
	and the second second

100.00

There is, therefore, little doubt that titaniferous ores of less than 20 per cent. of titanic acid can be profitably smelted. All metallurgists agree that the qualities of the iron made from titaniferous ore and steel obtained from titanic pig metal are excellent. This fact gave Dr. Rossi the idea to use an alloy of iron and titanium made in the electric furnace as an improver of steel.

The addition of titanium to steel increases the transversal strength from 17 to 23 per cent. (average results given in pounds per square inch). Bars of one inch square, loaded at center, with 12 inches between support, were used for transverse strength tests. For tensile tests round bars 1 1/8 inches in diameter and from 18 to 24 inches long were used. The ferro-titanium added to the steel contained about 12 per cent. of titanium and from

6 to 8 per cent. carbon. About one pound to every 100 pounds were added in small portions. There seems to be no advantage in using a ferrotitanium containing more than 25 per cent. titanium. Titanium also increases remarkably the elasticity of steel.¹

The function of titanium in steel consists in its high affinity for oxygen and especially nitrogen.

VACUUM EVAPORATION.

By P. B. SADTLER.

Received July 1, 1909.

It is a notable fact that, in this country at least, the knowledge of construction and operation of vacuum evaporating apparatus is very limited. There are a few industries, among which are the glucose, cane and beet sugar industries, where the engineers are in general pretty thoroughly familiar with this subject. Outside of these industries ignorance of the basic principles of evaporation actually frequently prevents engineers and superintendents from obtaining the best results. Those operating vacuum apparatus in their plants are prone to drop the subject completely as soon as their apparatus has been "broken in," only to take up the subject again when something goes wrong or when increased capacity must be installed to meet the demands of a growing business. Besides this class there is a long, though gradually diminishing, list of factory owners and others who either have entirely neglected to consider this important subject, or who, on doing so, have been frightened by some obstacle that seemed unsurmountable.

In this article will be discussed only the form of evaporator in which saturated steam is the heating medium as no evaporation can at present be done by direct fire so well as in the approved types of boiler, where the steam for the evaporator is made. The reasons why direct fire evaporation does not as a rule pay will be discussed on a later page.

A closed evaporator heated by steam coils has the following advantages:

I. Less steam is used because the apparatus is smaller and there is no exposed surface of boiling liquid, hence the amount of radiation and consequent loss of heat is reduced.

2. When evaporating under vacuum the temperature of the system is so lowered as to be nearly

¹ Dr. Rossi's contribution to Cassier's Magazine, Sept., 1905.

down to that of the atmosphere, with the result that the radiation loss is decreased. In other words, if a liquid is boiled in an open pan under atmospheric conditions the boiling point may be 220° F., or 150° above average atmospheric temperature. In a single-effect vacuum evaporator the average temperature may be 138° or 68° above atmospheric temperature. The radiation from the open pan would compare with that of the vacuum pan as 150° to 68° roughly.

3. The above considerations, however, are of little importance compared with the fact that a vacuum evaporator, either single- or multiple-effect, may be run by using, as a heating substance, the *exhaust steam* from engines, pumps, etc., thus enabling certain establishments to run an evaporator at practically no cost for steam.

4. There are many liquids which should be kept below the temperature 280° to 300° of high pressure steam coils, or even below atmospheric boiling point 212° F. Among these are such as sugar, glucose, sugar of milk, milk, glue, glycerine, etc.

5. Greater temperature differences may be attained in single effects, hence greater capacity for a given size apparatus.

6. When multiple-effect evaporators are used, the steam economy is increased to such an extent as to make the steam consumption run as low as 1/5, in some cases, of the actual amount of water evaporated from the liquid. There are some industrial concerns at present which could not operate and pay dividends without multiple-effect evaporators in constant use.

MULTIPLE-EFFECT PRINCIPLE.

The most primitive illustration of a multipleeffect evaporator is seen in an organic laboratory every day. Frequently a so-called volatile substance is put into a flask connected to a condenser and heated by a steam bath in which water is boiled. The vessel containing the boiling water is the first effect of a double-effect evaporator and the flask containing the low-boiling substance is the second effect. In the case of large scale work instead of boiling off a naturally volatile substance in the second effect, we boil off water whose vapor above it is kept at reduced pressure, thus making the water a relatively low-boiling substance. Thus in practice we have a succession of pans or effects, the coils of each one of which gets its heating vapor from the liquid of the preceding pan. The first pan of the series takes its

heating vapor from the original source, the boiler or exhaust pipe of an engine; the last effect gives up its vapor to a condenser in which the highest vacuum is maintained. An important point to be borne in mind is, that every effect is the condenser that brings about the pressure reduction necessary for the operation of the preceding pan.

The most popular and universally serviceable evaporator is the triple effect, although they are in daily operation on a large scale up to sextuple effect. In fact, multiple-effect water stills have been operated in twelve successive effects. A definite determination of the number of effects to use to obtain the best economy when working on any given solution, is difficult to make. The best tests, of course, are those made on the large evaporators and as some expense and annovance to the owner may result, the proper scientifically conducted tests are not carried out. One can, therefore, only go by the judgment of the designers and experts in this field in determining the best layout for economical multiple-effect evaporation.

STEAM CONSUMPTION.

Before discussing at length the factors governing the steam consumption in an evaporator, it is well to state several fundamental facts to be proven later:

1. The number of effects used in an evaporative system does not necessarily bear any relation to the evaporative capacity of the system.

2. A given amount of water at the boiling point to be evaporated requires a corresponding amount of steam to evaporate it irrespective of the form or size of the vessel in which the evaporation is accomplished. Speaking broadly, a pound of water evaporated requires that a pound of steam be condensed in a single effect.

3. The capacity of a heating surface is dependent on the temperature of the steam and not the pressure resulting therefrom.

4. An evaporator should not be considered as a heat engine, as it does no external work.

To get the best idea of the heat transactions in the evaporator, we should consider one effect at a time. Also, for simplicity, assume that the effects are of the same construction and bear in mind that each one is the condenser to produce the vacuum in the preceding effect. For every pound of dry steam entering the coils of the first effect, there is one pound of vapor produced by

the evaporation of the solution which we assume enters at the boiling temperature. As only a fraction of the total amount of water is to be evaporated in the first effect, the solution passes partly concentrated into the next effect. But as a lower pressure, and consequently lower temperature, is being maintained in the second effect, the solution passes in a superheated condition to the next effect, where a certain amount of evaporation takes place, due to the lowering of the pressure. Therefore in the second effect we have to expect the evaporation of another pound, plus increment due to the changing part of the heat of the liquid into heat of vaporization. Likewise in the third effect there is produced evaporation to the extent of a pound, plus two increments. In other words, the latent heat of a pound of steam is transferred through the heating surfaces of one effect after another, the amount of steam being, however, gradually increased by the spontaneous evaporation of the solution passing from one effect to another successively in a superheated condition. Finally this heat passes into the cooling water of the condenser and is lost. If we were to assume that there was no radiation of heat from the system and that pure water were being boiled, the above conditions would be always true and, in a sextuple effect, a single pound of initial heating steam would accomplish the evaporation of between six and seven pounds of water.

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In actual fact, however, we have to contend with the consumption of heat in two other directions:

I. In concentrating any solution we have to accomplish the partial separation of a liquid from a solid. There is, of course, a certain solution pressure against which we must work, and the actual amount of energy expended in this way is measured by the free energy of the chemical reaction of solution. This is not considerable when we concentrate to ordinary density. Hence it may be said that the total reversible energy expended in evaporation is equal to the free energy of reaction.

2. There is a radiation loss from each effect depending on the number of effects, their temperature with respect to atmospheric temperature, the amount and quality of the insulating covering used and other conditions such as degree of exposure of the apparatus to the weather.

These losses are cumulative from one effect to another so that where a solution has boiling points appreciably above that of water, it is not economical to attempt the operation of more than a limited number of effects.

DIAGRAMS.

To illustrate several cases of heat transactions obtaining in an evaporator:



Figure I shows a multiple effect heat diagram similar to that of Hausbrand for pure water in a quadruple-effect evaporator, where it is assumed that the difference in temperature from one effect to another is infinitesimal and where water enters at boiling temperature and the vessels do not radiate heat.



Figure 2 shows a heat diagram for a triple effect where the assumptions are made that the solution enters at atmospheric temperature, the solution has an appreciable excess in boiling temperature over that of water, and there is an appreciable temperature difference from one effect to another. The white areas represent the steam condensing and causing evaporation; the shaded areas represent the heat and energy losses as expressed in steam. The areas shown are not definitely measurable or calculable, but merely in the diagram show the tendencies existing in an evaporation system.

The heat transferences taking place in a multiple effect are altered in character to a great extent by the method of feeding the solution to be concentrated and the method of preheating if such is necessary. There are four ways of feeding the solution to the evaporator:

1. The dilute solution is fed entirely into the first effect and passes successively from one effect to another at higher and higher concentrations.

2. The dilute solution is fed in equal amounts separately into each effect, and boiled in each at the same concentration.

3. The dilute solution is fed entirely into the last effect and passes back to the first effect, a method known as running backward.

4. The dilute solution is pumped into the whole evaporator before starting and boiled in batches.

While inquiring into the relation, merits and advantages of these methods, let us consider at the same time the methods of bringing the dilute liquor to the boiling point and the bearing of these methods upon the heat transactions.

While in many cases a recently completed chemical reaction or industrial process leaves the dilute solution at a temperature approaching the boiling point, such as we find to be the case in certain mercerizing work, in electrolytic soda work or packing house rendering, there are other cases such as brine evaporation or sugar evaporation where the most efficient process necessarily includes the preheating of dilute solutions. In general, it may be said that running a comparitively cool solution into an evaporating apparatus decreases its efficiency and interferes with proper operation.

The usual method of preheating in running a multiple-effect concentrating evaporator as mentioned, in case I, above, is to employ a steam heater to preheat the solution entering the first effect by the use of live or exhaust steam. Frequently it is assumed that it is quite sufficient to turn on a little extra steam and run the solution cold into the evaporator. When this is done a portion of the heating surface of the effect, into which the cold solution is run, plays the part of a preheating apparatus. An evaporator is an expensive article to use as a preheater.

Where evaporating at equal densities, as in the second case above, there may be attached to each

effect a countercurrent heater in which the water of condensation, flowing from an effect, partly preheats the liquor being pumped into the effect. It can readily be perceived that the amount of heat that can be transferred to the solution from the water of condensation is insufficient to bring the former up to the required boiling point. This method is only applicable to precipitation work, as in multiple-effect salt evaporators.

When "running backwards" preheating is not often the practice as the boiling point of the last pan, the one which the solution enters, is not very high and the step up in temperature from one pan to another is not very great. This method has the advantage, other things being equal, that in the pan with the densest solution, there is the highest temperature, which tends towards better circulation and consequently higher efficiency. In using this method it is necessary to pump the liquid out of one effect into the next. The fact that steam is used to do the pumping need not have much bearing on the question, as it must be borne in mind that the exhaust steam from pumps is available for evaporation purposes.

The total consumption of heat in a multiple effect is composed of the energy consumed in actual separation of the liquid and the solid in solution; the radiation of heat from the walls of the vessel; the heat passing away in the circulating water of the condenser; and the heat passing away in the condensation water. In a test on an evaporator these can all be determined by actual measurement except the energy of evaporation and the radiation loss. There is always considerable doubt as to just what the latter amounts to in various types of evaporator and with various coverings. It is possible to measure it by special test, though this is rarely done. The energy of evaporation may be calculated for certain solutions and simply depends on a formula which may be derived from the gas laws.

It is shown by Nernst in the last edition of his "Theoretical Chemistry" that the energy expended in evaporation is

$$K \times R \times T \times (C - C'),$$

where R = the gas constant, T = the absolute temperature, C' = initial concentration, and C =the final concentration both expressed in mols. of salt per mol. of water. K is a constant quantity for any given substance in solution. For certain inorganic salts these constants have been determined, being derivable from the vapor pressure curves.

EFFICIENCY AND EVAPORATIVE CAPACITY.

A heat engine is an apparatus that transforms a portion of the heat received into dynamic energy. An evaporator is not a heat engine, therefore, but if the assumption be correct that solution is a chemical combination of solvent and solute, evaporator transforms heat into chemical an energy. When following this analogy between a heat engine and an evaporating system, we find that as a matter of fact the efficiency or the quantity $\frac{Q-Q'}{Q}$, where Q is the heat entering and Q - Q' is the heat transformed in chemical energy, is very low. The heat disposed of through other channels is very great. For instance, the latent heat of the entering steam eventually finds its way in large part into the circulating water of the condenser. Therefore, it will readily be seen that if, as we have shown above, this energy demanded for evaporation is entirely dependent upon absolute temperature and degree of concentration, no one type of evaporator has more efficiency than another, but the efficiency is entirely dependent upon the solution and its attendant physical conditions.

This matter of efficiency is noted here as there is an erroneous conception among some evaporator engineers as to the meaning of the word efficiency. It has been the practice among some to speak of the relative amount of evaporation per unit area of heating surface as the efficiency of an evaporator. The difficulty with using the term efficiency where the term capacity or duty would be more applicable, is that it leads those unacquainted with the principles to the idea that greater or less consumption of heat obtains in different types of evaporators. In different types of evaporators the evaporative capacity does vary considerably, but it is not the purpose of this article to discuss this side of the question, as the relative merits and demerits of different makes of machinery demand a separate article.

Before discussing the subject of capacity and the calculation of heating surface, it is well to approach the subject from the point of view of transmission of heat through the heating surface.

HEAT TRANSMISSION.

Heat transmission is measured by four fundamental factors, namely, temperature, weight, space,

and time. To be more specific, the common combination of units, in which we express heat transmission, is British thermal units, per square foot, per hour. Therefore, the coefficient of the heat transmission of a medium would be the figure which gives the number of British thermal units passing per hour from a warmer to a colder substance through one square foot of the medium for which we desire the coefficient.

It will readily be seen that the ability of the medium to transmit heat in a greater or less degree is governed by the following factors, namely, nature of the material, thickness of the medium, difference in temperature of the warmer and the colder substance. Hence, we have

$$h = k \frac{(t-t')}{d} m,$$

where h is heat transmitted, m is the time during which transmission takes place, d the thickness of the transmitting medium, t - t' difference in temperature, and k a constant dependent on the nature of the material.

From the above we see that

$$k = \frac{d \times h}{(t - t')m},$$

whence we are able to determine a constant k for any material by determining d, h, (t - t'), and mfor one square foot of heating surface. For practical purposes, where for a given material the limits of the factor d are quite narrow, this is omitted and the constant becomes

$$k = \frac{h}{(t - t')m}.$$

In the case of the transference of heat from one substance to another, for evaporative purposes, we have several possibilities: that of heat passing *from* a liquid, *from* steam, or *from* other gases; also heat passing to a body of liquid or to a film of liquid.

In evaporation work the case of heat passing from a liquid is not of very wide importance. Other things being equal k would be high in this case, but the difficulties involved in this method of heating necessarily outweigh those of steam heating. A high-boiling liquid may be heated and made to pass through heating tubes, giving a high transference constant, but the velocity necessary for its passage through the tubes is great enough to make the idea unworthy of consideration.

In the case of saturated steam there is not the

same necessity for an appreciable velocity directly in relation with the walls of the heating tubes, though this is very desirable. Unlike the case of direct heating, the effect is obtained by the latent heat of vaporization. Although the heating of the liquid is not due to difference in temperature between the steam and the liquid being heated, there must be a quite appreciable difference in temperature to make effective use of the latent heat. The larger the difference in temperature the greater is the heat transmission of the metal walls.

As water dissolves air to a small extent the steam from the boiler always contains air in greater or less amount. This air tenus to lessen the heat transmission so that it will be found in practice that better boiling takes place in the heating coil, where the velocity is considerable.

It can readily be seen, therefore, that the presence of *air in the heating steam is detrimental*. Just as air reduces the heat transmission so also does the water produced by condensation of the steam. The heat conductivity of water is far below that of a metal, so that a *steam coil containing a layer* of water is not fully effective.

The usual case of heating by other gases than steam is direct firing of boilers or evaporators, or the utilization of waste heat from furnaces, incinerators, etc. Here the transmission of heat is due to the difference in temperature and the velocity of the gas. It will readily be seen that if a body of cooled inert, non-condensable gas stays in contact with the wall of the heating surface the efficiency of the locality where that takes place is greatly reduced. On the other hand, in the case of steam the pressure back of the heating steam is cause for constant supply of the steam, which gives up its latent heat to the wall of the heating surface. A non-condensable gas, to be equivalent in effect to a condensable gas, like steam, must be led against the heat-transferring wall with considerable velocity to cause the rapid removal of cooled gas and prevent lodging of cooled gas in any small corners or devious places.

Very little can be determined as to the theoretical side of the transmission of heat *to* different solutions and solids. In general, it may be said that the completeness of the circulation of the liquid, the consequent rapidity of the disposal of the steam formed at the contact between the liquid and the heating surface, and the degree of mobility of the liquor are the chief factors for consideration here. More can be said later of interest and of value from a practical standpoint.

The heat transmission may be said to be greater when the liquid is in contact with the heating surface in as thin layers as possible. This is accomplished by allowing the liquor to spray or trickle over the heating surface or by bringing the liquor in contact with the surface in the form of a foam. When a film is in contact with the heating surface the evaporation may be said to be local; hence there is a local cooling tendency and consequent greater temperature difference between the liquor heated and the heating gas. This it will be seen will increase the heat transmission.

There has been an erroneous notion prevalent among some engineers that the evaporative capacity of a heating surface of any given form and area is inversely proportional to the thickness of the wall of the heating tube and proportional to the heat conductivity of the particular metal used. This idea is quite wide of the mark, as decided by results. If two otherwise equal evaporators be installed in which one has heating tubes of 20gauge and the other 15-gauge, such a theory would require that the latter would have half the evaporative capacity of the former, 15-gauge being twice as thick as 20-gauge. As a matter of fact the difference due to this variation in thickness is negligible. However, the difference between the evaporative capacities of two heating surfaces of different metals is measurable if two metals of widely different conductivities be chosen, but even then the difference is not great as might be supposed.

Kent gives, among others, the following conductivities:

Silver	1000
Copper (rolled)	845
Iron (wrought)	436
Steel	397

In spite of these figures the evaporative factor of copper tubes is only 10 per cent. to 15 per cent. greater than that of steel tubes.

To explain these facts we must consider that the resistances of the metallic wall to the passage of heat are threefold, namely, (1) resistance of entry of heat into the metallic wall, (2) resistance to passage through the wall, (3) resistance to the exit from the metal to the boiling solution. Let these resistances produce conductivities a, b, and d, respectively. Let C represent the evaporative factor of the metal by experiment.

 $\frac{\mathbf{I}}{\mathbf{C}} = \text{total resistance} = \mathbf{K} \left[\frac{\mathbf{I}}{a} + \frac{\mathbf{I}}{b} + \frac{\mathbf{I}}{d} \right] \text{ or the sum}$ of the three resistances.

$$C = \frac{I}{K\left[\frac{I}{a} + \frac{I}{b} + \frac{I}{d}\right]}$$
 where K is an arbitrary

constant. Conductivity a is effected by the ease with which the steam for heating is carried off and also the velocity of the steam; conductivity b is constant for a given metal; conductivity dis dependent upon the solution being handled, its boiling point, mobility, scaling properties, etc.

CALCULATION OF HEATING SURFACE.

The amount of heating surface necessary in an evaporator to be used for any given class of work depends on the following factors: 1. The amount of water to be evaporated, (2) the number of effects desired, (3) the initial steam pressure, (4) the boiling points of the solution to be evaporated, (5) the evaporative factor for a given type of machine, and for a given solution to be evaporated,

In calculating the amount of water to be evaporated, it is merely necessary to obtain from tables or analysis, the per cent. of solids in the dilute and the concentrated liquor. From this we can obtain the amount of water to be evaporated. When the number of effects in which the evaporation is to be done is determined, a calculation should be made of the water evaporated in each effect and from that, what the concentration should be maintained at, in each effect. When these concentrations are known the corresponding boiling points should be found from a good table of boiling points or by experiment, or a curve such as shown in Fig. 3 for certain grades of caustic



soda washings may be used. The *degrees of* excess boiling temperature is the total of each of these boiling points less 212° F. In other words, if the boiling points in three effects of a triple effect are respectively 214° , 220° , and 236° , the

excess boiling temperature is $(214 - 212) + (220 - 212) + (236 - 212) = 34^{\circ}$.

From the steam tables of pressure and temperature the *total temperature range* should be found. Or it will be found very useful to use a curve as shown in Fig. 4 where the pressures and



temperatures are plotted. Readings from such a plot compare favorably in accuracy with actual steam gauge readings on the evaporator. This temperature range then equals the temperature corresponding to initial steam pressure minus the temperature of steam pressure corresponding to vacuum maintained in the last effect.

The evaporative factor is a constant quantity. The total temperature range less the excess boiling is the effective range. This divided by the number of effects equals the average effect difference. The evaporative factor is derived from experience and from operative tests on the type of apparatus best adapted and on the given class of liquor to be evaporated. Probably the same factor would hold, for instance, on all submerged tube evaporators of the general type of those under discussion and would hold for either caustic soda solutions or carbonate of soda solutions, scaling conditions, etc., being otherwise the same. In fact the same constant quantity could be used, and has been used, in designing evaporators for a large range of inorganic chemical solutions, such as sodium chloride, sodium hydrate, sodium carbonate, sodium sulphate, sodium phosphate, sodium acid phosphate, sodium thiosulphate, potassium chloride, potassium hydrate, ferrous sulphate, calcium acetate, etc. However, in cases of tannic acid, sugar, glucose, glycerine, black liquor, or resinate of soda, packing house tank water, garbage

tank water, etc., somewhat different conditions obtain and the factor varies. The factor also differs slightly for different materials for the heating surface, that for copper or aluminum being distinctly higher than that for iron.

To specify the units on which this factor is generally based we might say that it is expressed as gallons of water evaporated, per square foot of heating surface per degree difference in temperature per hour.

- If C = evaporative factor
 - E = average effect difference
 - W = gallons evaporated
 - T == time in hours during which evaporation proceeds
 - S = square feet of heating surface

$$C = \frac{W}{E \times T \times S}.$$

Hence, if we find from experience or trial tests the value of C and we calculate E and W, as explained above, it is easy to obtain the required heating surface.

The successful designing of an evaporator is dependent first on the recognition of certain chemical and physical facts relating to the substance to be evaporated; second, on whether or not due regard is paid to certain thermal and thermodynamic principles involved; and third, on certain mechanical features that arise in the construction. It is not to be supposed that either an engineer, familiar with the best boiler practice, or a chemist, familiar with laboratory or open-pan evaporation in the works, could successfully design a multipleeffect evaporator. But certain facts in regard to design should certainly aid either a chemist or engineer in procuring the proper design or making a purchase.

As the principal purpose of an evaporator is to concentrate a solution of some sort, it will be readily seen that the chemical and physical laws governing concentrated solutions will be of importance. These are only known qualitatively and the quantitative laws which govern the chemistry of dilute solutions are inapplicable.

As most measurements of the concentration of solutions in the works are made with a hydrometer we generally speak of concentrating between certain limits in degrees, Baumé, Twaddell, Brix, salinometer, or specific gravity. It is, therefore, necessary to obtain or devise tables or curves showing the relation of these units of concentration with the percentage of solid matter in solution. The purpose of this is to obtain accurate information as to the actual amount of water that is going to be evaporated in a unit of time. This is especially important in cases where the vapor arising from the solution is the source of heat in the succeeding effect of the evaporator.

Of equal or greater importance, is a table or curve for a given solution which shall accurately show the relation between the boiling point of the solution and its concentration. It has been shown above, that the boiling point of a solution at any period of its progress through the evaporator has a direct bearing upon the size of the evaporator. A boiling point curve is generally obtained by taking simultaneous readings upon a hydrometer and thermometer, while boiling a solution actively under atmospheric conditions. This may best be accomplished in a laboratory where accurate means of determination are provided. After this curve is obtained a correction should be made for temperature at boiling to bring the hydrometer readings to the same temperature standard.

In cases where a substance is intended to precipitate from solution, during the process of evaporation, there should be used in conjunction with the above table also a table of solubilities of the substance at different temperatures.

The thermodynamic data necessary consist of the regular steam engineer's tables, showing the relations between temperature, pressure, heat of liquid, heat of vaporization, etc.

There are other chemical and physical data of which account must be taken in designing. The more information obtainable in regard to possible incrustation and scaling of heating surface, the clearer will be the idea as to what mechanical features to introduce for the removal of scale and as to what excess capacity to allow for this contingency. As in boiler practice, one of the greatest sources of annoyance is the deposition of gypsum on the tubes. This annoyance is encountered in the salt industry especially; on account of the peculiar solubility relation of gypsum, it is found more expedient to evaporate at low temperature with large heating surface, thus minimizing the scale. Another similar case is that of soda washings in pulp mills, where cooking liquor is evaporated for soda recovery. If the lime used in the course of the process is high in silica and alumina, these find their way to the evaporator and deposit in a dense fibrous scale. If such possibilities as

these are to arise, it is found expedient to have the heating surface in excess of the calculated area and easily removable from the evaporator for cleaning purposes. Moreover, it should be removable where corrosion is expected. There are some chemical solutions which corrode iron at the higher temperatures but are inactive at lower temperatures. In these cases the heating surface bears the brunt of the destruction in the evaporator.

Another important question is, as to the nature of a solution's activity under boiling conditions. In other words, does the boiling solution tend towards entrainment or foaming, or spattering? These points must be known beforehand, as they decidedly influence the proportioning of the evaporator.

PROPERTIES OF LIQUIDS AS AFFECTING CONSTRUC-TION.

In some industries the multiple-effect evaporator is among the largest and most expensive of the installations in the factory. Where a large evaporator is to be built for constant use, great attention should be paid to details of its design and construction. The liquids commonly evaporated in any quantity can be divided into two classes: those that foam on boiling and those that do not. It may be said in general that a liquor that entrains (or sprays and passes over with its vapor) does not foam, and a foaming liquor does not entrain. Also it is noticeable that solutions of alkaline reaction tend to foam while those of acid reaction tend to entrain. Most sugar juices and glucose come under the latter class. To provide against losses due to entrainment in an evaporator there are to be mentioned such methods as the use of baffle plates, catchalls, and high-vapor dome. The so-called film evaporators are subject to the difficulty in this respect that the vapor space allowed is invariably small and from the time a portion of the vapor leaves the boiling solution it carries with it a large amount of spray in suspension in its rapid passage through tortuous channels of small cross-section. The simpler types of evaporator whose construction is more like that of a tank are open to the objection often, that the vapor is drawn off from a dome directly over the boiling surface. In these simpler types of evaporators it can be arranged easily to have the sectional area large enough to make the rising of the vapor slow and the height of the dome sufficient to cause practically all the entrainment to drop back. In order not to cause any inversion or possible discoloration in the sugar juices it has been found advisable to have as little as possible of the juice in actual transit through the apparatus. This is accomplished by two principal methods: one by having a rather shallow bank of tubes in which the tubes are packed in the tube sheets as closely together as possible; another by spraying the juice over the heating surface, collecting it underneath and pumping it back again to the spray. This latter method rather subjects the juice to loss from entrainment, unless the vapor dome is extremely high.

In dealing with soda solutions the problems arising are of a very different nature from those in sugar work. Under the head of soda solutions we may include, among other important cases, the evaporation of sodium carbonate, sodium phosphate, pure caustic soda and caustic soda containing brine (electrolytic), black liquor (pulp-mill), mercerizing soda, etc. Except in the case of the more concentrated liquor of the last effect these are all more or less foamy when boiling. The use of baffle plates alone does not accomplish the prevention of loss when foaming takes place. It has been found that the only sure and efficient way to deal with foam, that is to cause it to subside, is to break the individual bubbles by application of heat. Hence the evaporator should be built in such a way that the heating surface is in a high bank above the level of the liquor evaporating. On operating the apparatus it may easily be found by trial what is the proper level of liquid shown in the gauge glass to prevent the foam from rising much above the top of the tubes and yet keep the tubes covered. About the most difficult liquors to handle in this respect are those in pulpmills or mercerizing plants. In these cases it is necessary to have the heating tube bank almost half the height of the evaporator.

In salt industries there are two main difficulties which have to be met by the designer of an evaporator: first, the salt has to be removed from the apparatus constantly as it precipitates from solution; second, scale that deposits upon the heating surface has to be removed periodically. In order to render the removal of precipitated salt simple and rapid it has been found best to construct the evaporator with a hopper-shaped bottom having

either one or two hoppers leading through a valve at their lowest point into a receiver where the salt is separated by draining through a false bottom. Another method consists in precipitating the salt continuously down a barometric leg under the evaporator, draining it and conveying it from the bottom by a closed elevator. This latter method is generally most efficient when working on a very large scale. In salt evaporation work, shut-downs are frequently necessary every day because of the constant deposit of gypsum upon the heating surface. This happens to such an extent that the evaporative capacity of the apparatus is reduced to a point where it no longer pays to operate. Ready means of access should be provided so that the heating surface may be thoroughly cleaned, or arrangements should be made for rapid pumping and boiling of suitable chemicals for the disintegration of scale.

There are certain acid salts which require special construction in order to line the evaporator with lead. This lead lining has never been done on a very large scale. In principle it consists in first pickling in sulphuric acid the plates of the machine, these being built with flanges that can be bolted together. The pickled surface is then given a tin coating. A sheet of lead of the proper thickness and size is coated with tin. The two tinned surfaces are laid together and the lead hammered to fit. Then the lead is "sweated" on by heating the iron from the outside to the melting point of the tin plating, which is lower than that of the lead. In this way the lead lining becomes attached to the steel by a homogeneous weld. Where the flanges are bolted together the contact of the two lead surfaces generally prevents leakage, but can be made more sure by the process of lead-burning.

It is generally most expedient to work out the size of the heating bank from the amount of heating surface determined upon before deciding upon the general dimensions of the shell of the evaporator. The first thing to consider is the bore of tubing to be used. If we are dealing with a vertical type of evaporator where the tubes are expanded into two horizontal tube sheets, the bore of the tubing is determined by the nature of the liquid which is to be evaporated in them and the magnitude of the work to be accomplished. In the case of the horizontal type of evaporator where the steam is in the inside of the tubes and the liquor

on the outside, there has been found a ratio of length to diameter for which the steam that condenses within gives a maximum evaporative capacity. If the tubes are too long and constricted, the steam will condense before it can pass to the far end of them, and a certain amount of heating surface is thereby wasted. It is usual to decide upon a certain length and diameter of tubing, however, arbitrarily from experience. It is then easy to decide, from the nature of the solution and taking into account the temperature differences and rate of evaporation, the proportioning of the heating bank. In a vertical effect when the length, diameter and centre line distances are decided it is merely a question of filling a certain number of tubes in the minimum sized circle.

Tubes may be attached to the tube heads or tube sheets by one of two methods, that of packing them in or expanding them in. To pack the tube in the tube sheet well, the hole in the sheet must be countersunk conically to admit of the forcing in of the rubber packing. This rubber ring is pressed in by a metallic place so that it is forced against the hole in the tube sheet and against the tube. This plate is held to the sheet by a stud bolt, and is arranged to hold down four, six or eight packing rings simultaneously.

When tubes are expanded into the tube sheet, it is expected that they are to be removed at no time during their life, whereas packed tubes can be easily removed, cleaned and repacked. Tubes expanded into the tube sheets properly make a perfectly steam-tight joint for almost any pressure or vacuum. The tubes are extended through the sheets a slight distance and are beaded over and are expanded by a special tool for this purpose. The Prosser expander rolls a corrugation in the tube, just inside of the sheet and beads it outside the sheet. In this way the sheet is braced against pressure from within or without. The Dudgeon expander rolls the tube flat against the side of the hole and beads it over on the outside. This method is not as effective in results, but is less of a strain on the metal of the tube.

I hope at a later date it may be possible to go more deeply into the practical side of this subject, presenting as far as possible the best solutions to problems arising from day to day.

SWENSON EVAPORATOR CO., CHICAGO. THE JOURNAL OF INDUSTRIAL AND ENGINEERING CHEMISTRY. Sept., 1909

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RECOVERY OF GLYCERINE BY THE TWITCH-ELL PROCESS COMPARED WITH THE RECOVERY OF GLYCERINE BY WASTE SOAP LYE METHODS.

By O. T. JOSLIN. Received July 17, 1909.

As the Twitchell process has now been used on a manufacturing scale for ten years in one factory, for eight years in a half dozen or more factories, and in many others for a lesser period, there are ample figures to show very closely what advantages this process possesses over waste soap lye methods.

I will give a schedule of the operations involved and the costs and credits of the two methods.

Cost of Twitchell process plant to work 8000 lbs.	
fat per day including glycerine evaporating plant	\$2800.00
Cost of waste soap lye glycerine plant for 8000	
lbs. fat per day	3900.00
T OF WORKING 8000 LBS. FATS OR OILS IN THE KI	STTLE BY TI
OLD PROCESS.	
Cost of boiling kettle through six changes, killing	
change and one settle; steam, labor, etc., at \$2.00	
per change	\$12.00
Cost of salt for three changes at 1/4 c. a lb., 1920 lbs.	
salt equals \$4.80 four-fifths of salt recovered.	

gives salt cost Cost of saponifying with caustic 18.58 per cent. of	0.96
60 per cent. caustic at \$2.10	31.20
	\$44.16

All other costs will remain same in both instances.

COST OF WORKING 8000 LBS. FATS OR OILS BY THE TWITCHELL PROCESS.

Steam (coal at \$3.00 per ton)	\$1.00
Labor	1.00
Sulphuric acid (at 80 c. per 100 lbs.)	0.80
Barium carbonate	0.75
Reagent (at 15 c. per pound)	7.20
	\$10.75
Expense (insurance, repairs, deterioration) 20 per cent, of cost of Twitchell plant, \$1450.00 equals	
\$290.00 for the year, divided by 250 days, equals	1.16
	\$11.91
Cost of working 100 lbs. of stock	\$0.1488
Entire Cost of Working 8000 Lbs. Fats or Oil by Process and through the Kettle.	Y TWITCHELL
Twitchell process cost	\$11.91
Cost of boiling kettle through four changes, killing change, one salt change, one strengthening change	
and one settle; steam, labor, etc., at \$2.00 per	
change	8.00
Cost of 640 lbs. salt at 1/4 c. a lb. (no salt recovered)	1.60
95 per cent soda ash at 1 c for 48 per cent	17 65
5 per cent, soda caustic at \$2,10 for 60 per cent	1.56
o per cent, soua causile at parto for 60 per cent	
	\$40.72
Cost of saponifying with 95 per cent. ash and 5 per cent. caustic, \$19.21, or \$31.20 - \$19.21 = \$11.99	

or 15 c. per hundred lbs. stock saved by soda a saponification.

RECOVERY OF GLYCERINE BY THE TWITCH- COST OF WORKING WASTE SOAP LYES FROM 8000 LBS. STOCK PER DAY, 18,000 LBS. LYE.

Acid	\$.50	
Chemicals	1.00	
Labor, two men all day	3.50	
Expense (insurance, repairs, deterioration):		
20 per cent. of \$3900.00 equals \$780.00 per year		
of 250 days, equals \$3.12 a day	3.12	
Steam for heating and handling lyes aside from evap-		
orating	1.50	
Steam for evaporating equivalent to 1500 lbs. coal at		
\$3.00 a ton	2.25	
Water, 26,000 gallons per day at 10 c. a 1000 gallons	2.60	
	\$14.47	

Maximum amount of glycerine obtainable by waste soap lye methods is 8 lbs. absolute glycerine per 100 lb. neutral fat.

On stock containing 5 per cent. free fatty acids the amount of glycerine would be reduced to 7.5 lbs.

On 8000 lbs. this gives 600 lbs. at 13 c. a lb., \$78.00.

COST OF WORKING TWITCHELL PROCESS GLYCERIN	IE.
8000 lbs. stock per day 5200 lbs. glycerine water	
Lime	\$ 0.05
No other expenses of treatment.	
Labor, one man a day	1.75
Expense (insurance, repairs, deterioration):	
20 per cent. of 1350 equals \$270.00 per year of	
250 days.—	1.08
Steam for evaporating, 433 lbs. coal at \$3.00 a ton	0.65
Water, 12,500 gallons per day at 10 c. a 1000 gallons	1.25
	\$4 78

Maximum amount of glycerine obtainable, 95 per cent. of the glycerine in the fat, at 5 per cent. free fatty acids in the fat, would give 9.5 lbs. glycerine in the water per 100 lbs. stock and allowing 5 per cent. loss of glycerine in process of working up the glycerine water, would give 9 lbs. of absolute glycerine on the fat. On 8000 lbs. this gives 720 lbs. glycerine at 14 c., a lb. equals \$100.80.

Old method.	Cost.	Twitchell process.	Cost.
Making soap	\$44.16	Making soap, includ-	
Glycerine recovery	14.47	ing cost of Twitchell process	\$40 72
	\$58.63	Glycerine recovery .	4.78
	45.50	Stand State State States	
			\$45.50
Profit of	.\$13.13	due to manufacturing.	
Value of glycerine reco	vered by	Twitchell process	\$100.80
Value of glycerine rec	overed	by waste soan lye	a contraction
process			78.00
Profit due to glycerine			\$ 22.80
Profit due to manufa	cturing .		\$13.13
Profit due to glycerine			22.80
Profit on 8000 lbs, stor			\$35.93
Profit on 100 lbs. stock			0.45
Additional profit per da	ay due to	the Twitchell process	\$35.93

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The advantages mentioned above are due to the following differences between the two processes:

First, the fact that almost all the glycerine is separated from the stock before it goes to the kettle avoids the loss due to glycerine remaining in the soap, and the loss due to the complicated and lengthy process necessary in recovering glycerine from waste soap lyes.

Second, the fact that Twitchell process liquors contain from 15 per cent. to 20 per cent. glycerine, while waste soap lyes run between 3 per cent. and 5 per cent, results in a saving of glycerine on account of the very much less evaporation required in the case of Twitchell liquors.

Third, the greater strength in glycerine of the liquors reduces the amount of water to be removed by evaporation to 1/4 or 1/6 of what is necessary to be removed from waste soap lyes. This of course makes a commensurate reduction in the amount of steam, condensation water, labor and time, and consequently expense.

Fourth, the absence of salt from the glycerine liquors is a most important advantage, which, while readily recognized by all who have used the waste lye methods, is not fully appreciated until both methods have been used and compared.

The presence of salt in glycerine lyes makes a larger, more complicated and more expensive evaporator necessary. It also causes the evaporator and all other parts of the plant to corrode and wear out much more rapidly. It necessitates salt strainers and an extra pump, and a considerable extra amount of labor, while some glycerine always remains in the salt, and sometimes much. This glycerine salt should, of course, go back to a kettle which is having the glycerine extracted from it, but it does not always go this way, and even when it does there is some loss of glycerine anyway, as mentioned above.

Fifth, the much greater purity of Twitchell liquors over waste soap lyes makes several additional savings. The treatment necessary is simple and cheap in the extreme, both in regard to materials and labor required. The waste material separated as a result of this treatment is so much smaller in amount that there is a saving of glycerine here, as such residues can never be completely freed of glycerine.

The absence of salts and other impurities causes the glycerine liquors to evaporate much more easily, with a consequent saving similar to that accruing from the less amount of water to be removed as above mentioned.

Sixth, a crude glycerine containing 90 per cent. of glycerine can be obtained with much more ease and certainty than an 80 per cent. crude can be obtained from waste soap lyes. This makes an appreciable saving in drums, labor of handling and freight.

Seventh, the greater strength and purity of the crude glycerine puts it in a special class known as saponification crude. Saponification crude should have 88 per cent. of glycerine or more and 5/10 per cent. or less of ash. This standard is easily reached by Twitchell liquors. Saponification crude usually brings one cent a pound or more on the absolute glycerine contained than soap lye crude. This advantage accrues to the producer when the glycerine is sold as crude. If a plant distils its own crude it derives the advantage in the form of greatly increased capacity of a still of a given size for this crude over waste soap lye crude, less steam and labor and a greater yield, and a purer product with one distillation, as C. P. glycerine can be made with one distillation. Finally, I never heard of any one finding glycols in Twitchell glycerine.

Another important advantage is the saponification with sodium carbonate instead of sodium hydroxide. This makes a saving of 14 or 15 c. on every hundred pounds of stock saponified in this way. For a good many years there was a strong, prejudice against the sodium carbonate saponification. This prejudice was without reason and during the last few years it has almost entirely disappeared. It is a fact that the soap made by carbonate saponification is the same in every way as soap made from the same stock by caustic saponification. There is also no difficulty in making the carbonate saponification in the kettle, except the slight difficulty which always accompanies a new method of procedure. This fact was first demonstrated in Europe, but is now accepted by all the experienced American soapmakers.

There are in addition several lesser advantages which the Twitchell process possesses. One is the decrease in the odor which the process occasions in the case of many strong smelling stocks. In some cases this becomes a matter of great importance. Corn oil for instance was formerly of little value for soap and was but little used for this purpose, but since the introduction of the Twitchell process its use has been greatly increased, as the process diminishes the odor so greatly, that corn oil becomes available for soap and soap powder, when worked by the Twitchell process, while it is entirely out of the question when not so worked. The process removes the heavy disagreeable odor of this oil to such an extent that it is not noticeable in the finished soap product, even when unperfumed. The odor of animal fats is also greatly diminished and this is a great advantage in the manufacture of toilet soaps.

There is a class of soaps, known as cold-made or half-boiled, in the manufacture of which the glycerine has heretofore been lost, as it remained in the soap. There are several special reasons why soaps of this nature should be made, and they are made in large quantities. These soaps can be made from Twitchell fatty acids, and large quantities are now being so made, the glycerine being previously removed and saved. As glycerine has no apparent detergent qualities and as it sometimes comes out on the soap, to its detriment, the soap is really better without the glycerine than with it, aside from the great money-saving.

Although many persons claimed that the glycerine made some difference in the appearance of the soap, it has been demonstrated to the satisfaction of a great many soap men, including the writer, that this is not so. The writer remembers one instance in particular where four soaps were prepared, one without glycerine and the other three with 5, 10 and 15 per cent. of glycerine respectively, and it was impossible to tell them apart by appearance, feeling or trial washing.

Also in the case of soft soaps there is no other way of saving the glycerine than by making them of fatty acids. Large quantities of soft soaps have been made in Europe by the Twitchell process for several years past. There has been ample demonstration, both in this country and Europe, that the removal of the glycerine from soft soaps makes no apparent change in the appearance or other properties of these soaps, nor in the washing properties. In fact it requires a careful chemical analysis to distinguish between the soaps with glycerine and those without. While some soft soaps are being made by the Twitchell process in this country, the matter has not been taken up in this respect to the extent that it should be, although it looks as if there would be an advance in this respect in the near future, especially since the price of glycerine has become so high.

Finally, this process is rapidly increasing in use in this country, Europe and in other parts of the world, and there is no doubt but what the next few years will see the process in such general use as to be almost universal.

[CONTRIBUTION FROM THE LABORATORY OF ARTHUR D. LITTLE.] HYGIENIC SIGNIFICANCE OF SULPHUR IN GAS.

By F. E. GALLAGHER. Received July 17, 1909.

Several states—namely, Connecticut, District of Columbia, Massachusetts, Wisconsin, and New York—have statutory limitations relating to the purity of illuminating gas, particularly in regard to sulphur. In addition to the state restrictions, there are local restrictions in a number of cities. In general these restrictions specify that the sulphur compounds shall not exceed 20 to 30 grains per 100 cu. ft. These laws can all be traced to arbitrary English legislation, and cannot be said to be based upon a rational experimental foundation.

Sulphur exists in illuminating gas mainly as carbon bisulphide, which upon burning goes to sulphur dioxide. Sulphuric acid may be formed under certain conditions by subsequent oxidation.

The experiments here described were made near a gas works (Malden, Mass.) from which gases containing different amounts of sulphur were obtained. A small room of 600 cu. ft. capacity was fitted up as a gas-burning room, and equipped with a fan for stirring the air and an exhaust fan for controlling the ventilation. Air samples were drawn from the room through Emmeling absorption towers containing hydrogen peroxide solution, which served to oxidize and absorb the sulphur dioxide, which was finally determined as BaSO₄. All results were corrected to standard pressure and temperature. There were determined in each experiment:

The amount of sulphur entering the room.
 The sulphur dioxide content present in the

air of the room. 3. Humidity.

4. Carbon dioxide content in the air of the room. This was used as a measure of the ventilation.

The ventilation was considered to be good when CO_2 equaled 6 to 12 parts per 10,000 and very restricted and unfavorable when CO_2 equaled 30 to 40 parts per 10,000.

Each individual experiment was run for 6 to 8 hours. Gas was always burned at the rate of 5 cu. ft. per hour.

The following are illustrative of the experiments made:

Expt. 1.—With one man in the room the ventilation was regulated so that the CO_2 equaled 7 parts per 10,000. The gas was then lighted and the average increase in SO_2 over several hours determined. With the sulphur in gas equal to 21.6 grains per 100 cu. ft., the average SO_2 content in the air equaled 0.28 part per million. With the sulphur in gas equal to 27.5 grains per 100 cu. ft., the average SO_2 content of air equaled 0.34 part per million.

Expt. 2.—The exhaust was regulated so that good ventilation was maintained with the gas continuously burning. With 18.6 grains gas, the SO₂ in the air under equilibrium conditions equaled 0.28 parts per million. With 31.6 grains gas, the SO₂ in the air equaled 0.49 part per million.

Expt. 3.—The ventilation was checked as much as possible, window and door closed and sealed, and exhaust fan stopped. The CO_2 content then equaled 38 parts per 10,000. With 23.3 grains gas the SO_2 value under equilibrium conditions equaled 0.53 part per million. With 35.8 grains gas the SO_2 value under equilibrium conditions equaled 0.77 part per million.

These experiments are but a few of many made. They illustrate, however, the general experimental scheme and indicate the extreme values encountered for SO_2 content in gas-lighted rooms; namely, 0.2 to 0.8 part per million. For conditions of good ventilation and with practically any commercial gas the limiting concentrations for sulphur dioxide in the air of rooms would be 0.1 to 0.4 part per million. For conditions of very restricted ventilation these limits would be 0.4 to 0.8 part per million.

As a result of extensive experiments, Lehmann showed that SO_2 in the air could not be detected below 6 parts per million; between 6 and 11 parts per million it would be just noticeable, while 14 to 20 parts would be somewhat disagreeable for people not accustomed to sulphur dioxide. He furthermore established¹ the fact that sulphur dioxide, when present in the air in such amounts as to be irritating to the nasal passages, produced no lasting physiological injury. In connection with this data our experimental results indicate that the SO_2 content of gas-lighted rooms is wholly negligible as regards comfort or health.

Further experiments bearing upon this problem gave the following results:

1. In no case, even under the most restricted conditions of ventilation, did the burning of gas give rise to noticeable odors of sulphur gases.

2. With the doors and windows sealed and the ventilation restricted as much as possible, the air of the room changed 1.8 times per hour. When the ventilation was rendered favorable for regular living conditions, the rate of change of air was several times greater than the figure just given.

3. As regards the vitiation of air by carbon dioxide, it was found that one man actively exercising had about the same effect as gas burning at the rate of 5 ft. per hour.

4. Paper on the walls and ceiling only moderately reduced the effectiveness of the plaster in the absorption of sulphur gases.

5. Sulphur gases formed on the combustion of illuminating gas are removed from the air of rooms in three ways: by the changing air in the ordinary course of ventilation, by condensation along with water vapors on the cold walls and windows, and through absorption by the alkaline constituents of the walls and ceilings.

6. As the sulphur content of a room increases markedly, the rate of reaction between the plaster and the sulphur gases becomes greater, thereby effecting a proportionally more rapid removal of the sulphur gases.

7. Calculations of the sulphur dioxide content of the air of rooms from the sulphur introduced by the inflowing gas are wholly unreliable if they fail to take account of the different ways in which sulphur dioxide escapes from a room.

The considerations and fears that originally prompted stringent sulphur-in-gas limitations were for the most part without justification and the liability of danger from sulphur has been greatly exaggerated and misunderstood.

MOISTURE DISCREPANCIES IN PHOSPHATE ROCK OF THE PACIFIC.

By CARLTON C. JAMES. Received July 17, 1909.

The Committee on the Analysis of Phosphate Rock of the National Fertilizer Association presented a report some time ago which showed the results obtained by some thirty chemists upon

¹ K. B. Lehmann: Arch. für Hygiene, 18, 180 (1893).

four samples of phosphate rock from the Southern States of South Carolina, Tennessee and Florida. These samples were prepared as uniformly as it was possible to make them, yet in the moisture determination the difference between the maxima and minima of the four samples ranged from 0.48 to 0.81 per cent. If the samples had been taken from the cars or cargo by thirty chemists individually in quantities that each thought sufficient, reduced to the form they considered proper, and in the way they deemed best, quartered and reduced by each in his own way to the desired size sample and placed in containers that each thought proper, it would not be unreasonable to suppose that the differences would show still more astonishing discrepancies. We have had occasion to study these differences in the phosphate of the Pacific with the idea of determining the causes and to reduce them as much as possible.

The material with which we have to deal comes from Ocean and Pleasant Islands, two small isles west of the Gilbert group and nearly on the equator. The material ranges in size from fine dust to rocks six inches in diameter. It is of coral origin and some of the larger pieces show distinctly the coral formation. Much of the material is in the form of spherical pieces resembling bird shot. Analysis of this material shows about 83 per cent. tricalcium phosphate, 3.5 per cent. to 4 per cent. calcium carbonate and usually less than I per cent. iron and aluminium oxides. This rock is received in cargoes of from 2000 to 4000 tons so that it is of considerable importance that representative samples be taken, not only for quality but for moisture as well. A difference of five- to eighttenths per cent. in the moisture determinations would mean a financial consideration of \$250 to \$500, an amount about which probably no one would care to have any doubt as to whether it were his own or his neighbor's.

The sampling is done in a manner agreed upon by buyer and seller. As the rock comes out of the steamer practically one per cent. of the cargo is taken as sample: for a 2000-ton cargo the sample weighs about 25 tons. Every hundredth bucket- or basketful as it comes from the hold is set aside in closely woven sacks and held on board in a dry place until the cargo is discharged. The moisture sample is taken twice a day in glass jars holding at least two pounds. Usually Mason jars holding two quarts are used and any pieces too large to enter are broken with a hammer to a convenient size. When discharging is completed the moisture sample, weighing from 100 to 200 pounds, is combined in the presence of agents of buyer and seller and three samples of about five pounds each are taken. If there are many large rock pieces, these are again broken by hammer until they are about one-half to threefourths of an inch in diameter. When both agents signify that they are satisfied the samples are sealed and each agent takes one for analysis; the third is retained for a referee in case the analyses should fall outside the limits set in the contract.

The quality sample of one per cent. of the cargo is sent in sealed bags to the fertilizer works where it is crushed and ground in the presence of the seller's agent to about 50 mesh. A small sample is taken from each 125 pounds. These are combined, quartered to about 25 pounds and three samples drawn as was done with the moisture sample.

The results of the analysis of several such samples are here given:

Moisture sample.	Ground quality sample.	Difference.
Per cent.	Per cent.	Per cent.
3.11	2.58	0.53
4.96	3.21	1.75
4.55	3.80	0.75
3.82	3.26	0.56
5.16	3.41	1.75

Each result given is the average of some six separate samples, all of which did not vary more than two-tenths per cent. It will be seen that the third column shows some remarkable differences between the moisture in the moisture sample and that in the ground sample.

By grinding the moisture sample in a small mill to about eight mesh it had been discovered some time ago that not only the size of the rock was reduced but the moisture content as well. Consequently, it was desirable that grinding and handling of the moisture sample should be practised as little as possible.

When the last above-mentioned result was obtained showing a difference of 1.75 per cent. between the two samples it was believed that a mistake had been made somewhere in the process of taking the sample or in its analysis, and suggestions were made that the jars in which the rock was placed were not entirely dry, that the drying temperature had been too high and that the samples had taken up moisture in cooling.

In order to shed some light upon the subject, during each day of discharging, tests were made of the rock taken from the steamer and of the same rock from the mill after grinding. The results are as follows:

Unground. Per cent.	Ground to 50 mesh. Per cent.	Difference. Per cent.
3.72	3.48	0.24
3.75	3.58	0.17
3.51	3.34	0.17
4.01	3.88	0.13
3.43	3.16	0.27
4.58	4.28	0.30

The results given are averages of six determinations. Samples of 20 grams each were weighed into glass' stoppered weighing bottles and dried in an air bath at 100° C. for five hours, both ground and unground samples being in the same air bath at the same time and cooled in the same desiccator so that the conditions should be as nearly identical as possible.

The results show that some moisture is lost in grinding but do not account for such a difference as 1.75 per cent. It had been noticed in weighing out the moisture sample that a representative sample depended to a considerable extent upon the operator. In a sample consisting mostly of fine and shotty particles there were embedded small pieces of rock from one to two and occasionally three centimeters in diameter. If the sample were shaken the fine particles would sift to the bottom and the larger particles would come to the surface, a point which had to be guarded against. Therefore, it was decided to make other experiments to determine if the size of the rock particles would have sufficient influence to make such a difference.

Three samples of 20 grams each were weighed into weighing bottles, duplicates being run at the same time. The samples were neither crushed nor ground and were subjected to no treatment other than that they were separated on screens according to size.

The first sample consisted of large rock pieces 15 mm. in diameter; the second, small pebbles 2 to 5 mm. in diameter; the third, fine particles through a 30-mesh screen. The samples were all placed in the same air bath and subjected to a temperature of $100-105^{\circ}$ C. to constant weight which took about five or six hours. When all the samples showed constant weight, that weight was taken as the loss at $100-105^{\circ}$ C. The temperature was then raised to $120-125^{\circ}$ C. and was maintained thereat until constant weight was again established. Results:

	Diam.	Per cent.	loss at	Per cent. loss be- tween 100		
	in mm.	100-105°.	120-125°.	and 125°C.		
Rock pieces	15	3.361	3.519	0.158		
Small pebbles	2-5	3.970	4.205	0.235		
Through 30 mesh, Difference in loss be- tween rock pieces	0-0,8	4.858	5.062	0.204		
and fine particles		1.497	1.543			

The loss at 120-125° C. is shown to be greater than that at 100-105° C. by about 0.2 per cent., but this may or may not represent water. In the case under consideration the loss may be due to organic matter: roots and stems which have become too small to be removed. Probably such a high temperature would never be used for obtaining the moisture in phosphate rock, and discrepancies due to this cause would be nil. The results also show that moisture is retained unequally in the sample and varies with the size of the particles. This no doubt is due to the greater surface exposed by the finer material, but whether the moisture varies in proportion with the fineness has not been determined. It is desirable to get a representative sample containing proportional parts of the fine and coarse material so that the correct amount of water may be determined, but as has been pointed out above, two difficulties are presented. If the sample is to be ground to particles of about the same size it would have to be reduced to 40 or 50 mesh with a loss of moisture, whereas, if it is left unground there must be left to the discretion and integrity of the chemist the procuring of correct results.

Other conditions and other material might not give rise to such discrepancies: we simply present these two points as they have occurred here, but from the results obtained here and those published by the National Fertilizer Association it would seem that the determination of water is not as simple as at first might be supposed and deserves more consideration than is usually given it.

LABORATORY OF THE PACIFIC GUANO & FERTILIZER CO., HONOLULU, HAWAII.

[CONTRIBUTION FROM THE LABORATORY OF ARTHUR D. LITTLE, INC.] THE ANALYSIS OF LEAD ARSENATE FOR WATER-SOLUBLE IMPURITIES.

> By Roger C. Griffin. Received July 17, 1909.

Where large lots of any material are being held pending the result of the analysis of the sample, time becomes an important factor. The determination of water-soluble impurities in arsenate of lead according to the method of Haywood¹ requires at least eleven days and generally twelve. Our experience in the analysis of a considerable number of samples of this substance has led us to believe that the necessary time can be very materially shortened without sacrificing accuracy of results. In the first place, to determine whether it is necessary to shake the dried sample with water for as long a period as ten days, the following experiment was carried out:

Portions of two grams of the dried sample were weighed into large glass-stoppered bottles containing two liters of distilled water free from CO_2 . Each bottle was thoroughly shaken about eight times each day. At the end of two days the contents of the first bottle (A) were filtered through a dry filter, 200 cc. of the filtrate evaporated to dryness in a weighed platinum dish on the water bath and the total soluble impurities dried at 100° in an oven for two hours, then cooled in a desiccator and weighed. At intervals of about two days the contents of one of the remaining bottles were treated in exactly the same manner, the contents of the last bottle being analyzed on the tenth day. The results obtained were as follows:

Bottle.	А.	В.	C.	D.
Days	2	4	6	10
Wt. of sol. matter (gram)	0.0075	0.0060	0.0065	0.0064
Per cent. of sol. matter (dry				
basis)	3.75	3.00	3.25	3.20

The increase after four days is seen to be insignificant since it is hardly possible to weigh closer than to 0.0005 gram under these conditions. On this account it also seems advisable to take 500 cc. for evaporation instead of 200 cc. On another sample tests were made after shaking for three days and for ten days, respectively, evaporating 500 cc. of the filtrate. The results were as follows:

Bottle.	F.	G.
Days	3	10
Wt. of sol. matter (gram)	0.0253	0.0250
Per cent. of sol. matter (dry basis)	5.08	5.00

The difference between the results obtained from three days' shaking with water and those obtained from ten days' shaking is well within the limits of experimental error.

Considerable difficulty was often experienced in obtaining a clear filtrate, and the precipitate has considerable tendency to clog the pores of the filter paper and make the filtration proceed very slowly. We found, however, that with a Gooch

¹ Dept. of Agriculture, Bureau of Chemistry, Bull. 105.

crucible a perfectly clear filtrate could be obtained, and at the rate of 500 cc. in from 5 to 15 minutes. The asbestos mat must be thoroughly washed, of course, to remove any soluble matter or loose shreds. The filtration is also more rapid if the crucible and mat are ignited before use. The method of procedure which we finally found to be the most satisfactory and rapid is as follows:

The mat should be thick enough so that when held up to the light the holes in the crucible are not quite visible. After it has been prepared in the usual manner about 250 cc. of distilled water are poured through the crucible, followed by 5 cc. concentrated HCl and this in turn by 250 cc. more of water. The crucible is then strongly ignited for two or three minutes. After it is cool, 5 cc. of concentrated HCl are drawn through it and then 200 cc. of water. The suction flask is thoroughly rinsed out with water and as much as possible poured out. It is not necessary to dry either the flask or the crucible. About 25 cc. of the liquid to be filtered are sucked through the crucible, and the flask is rinsed out with this liquid and emptied. This is repeated twice and then the filtration is proceeded with until about 600 cc. of filtrate have been obtained. This is then poured into a graduated 500 cc. flask, after rinsing out the latter several times with small portions of the liquid, and made up to the mark.

After having weighed the total soluble matter, time may be saved by using this material for the determination of water-soluble As₂O₅. (It may be said here that in only two of the samples we have analyzed have we found any water-soluble PbO, one of these contained only a trace and the other 0.30 per cent. In case soluble PbO is present, however, it is necessary to evaporate another portion of the water extract for its determination.) The residue is taken up in a little water and evaporated with a few cc. of HNO₃. If it is evaporated directly with H2SO4, according to the method of Haywood, the H2SO4 chars the acetates which are always present (unless the arsenate was made from Pb(NO₃)₂) and the separated carbon causes considerable inconvenience. Evaporation to dryness with HNO₃ destroys these acetates. This dry residue is then taken up with a very little water and after adding 0.5 cc. of concentrated H₂SO₄, evaporated to a syrup on the steam bath, and then on the hot plate to fumes, to remove the HNO3. From this point the method of Haywood as given in Bulletin 105 is followed.

Briefly then, the method for the determination of water-soluble impurities in arsenate of lead, which we have found to give satisfactory results with a minimum expenditure of time, is as follows:

Two grams of the dried material are weighed into a large glass stoppered bottle containing 2000 cc. of distilled CO,-free water, and allowed to stand three days, shaking frequently. The solution is then filtered through a Gooch crucible and 500 cc. of the filtrate are evaporated to dryness on the steam bath in a weighed platinum dish, dried to constant weight (two hours) in a water oven and weighed for total soluble matter. The latter is taken up with very little water and a few cc. of HNO3, evaporated to dryness on the steam bath, washed into a small beaker with a little water and after adding 0.5 cc. of concentrated H₂SO₄ evaporated to a syrup on the steam bath and then to fumes on the hot plate. 10 cc. of water are added and the beaker gently swirled to throw any PbSO4 into the centre. If any is present it is filtered out through a tiny filter paper, washing with 5 per cent. H₂SO₄. The As₂O₅ is determined in the filtrate according to the method of Haywood. If soluble PbO is present it is determined in a separate portion of the original filtered water extract by Haywood's method. The complete analysis can easily be carried out in five days.

The samples of arsenate of lead which have come under our observation have been very variable in composition. The total PbO has ranged from 64.32per cent. to 73.28 per cent.; total As_2O_5 from 32.48 per cent. to 23.73 per cent.; total water-soluble matter from 1.96 per cent. to 5.08 per cent.; and soluble As_2O_5 from 0.12 per cent. to 1.95 per cent. These percentages are based on the dry material. The moisture in the original paste has varied from 43 per cent. to 68 per cent.

Haywood¹ gives the soluble As_2O_5 obtained from C. P. arsenate of lead as 0.63 per cent. to 0.85 per cent.

The water-soluble As_2O_5 in the commercial samples of lead arsenate has two possible sources: (1) dissolved arsenate of lead, and (2) disodium arsenate incompletely washed out of the material. The fact, however, that a sample may contain as little as 0.12 per cent. of water-soluble As_2O_5 , whereas chemically pure lead arsenate has been 'ound to dissolve sufficiently in water to give 0.85 per cent. of water-soluble As_2O_5 , seems to indicate that lead arsenate is practically insoluble in water which contains even a very small amount of disodium arsenate. 0.12 per cent. of water-soluble As_2O_5 corresponds to 0.0019 gram of Na_2HAsO_4 per liter of water, or only about 2 parts per million. The water-soluble As_2O_5 in commercial lead arsenate therefore is due to the Na_2HAsO_4 present as impurity; and as this substance is comparatively easily soluble in water, it must all be dissolved out of 2 grams of lead arsenate by 2 liters of water in much less time than ten days.

ON THE VOLUMETRIC ESTIMATION OF URANIUM AND VANADIUM.

By Edward DE MILLE CAMPBELL AND CHAS. E. GRIFF Received June 16, 1909.

Up to this time there have been three volumetric methods proposed for the quantitative estimation of uranium and vanadium when occurring together. These methods have all been put forth because of the need for a rapid and accurate method for the analysis of carnotite ores.

The first of these is that of Friedel and Cumenge.¹ In this method the ore is dissolved in nitric acid and the vanadium, iron and aluminum are rendered insoluble by evaporating to complete dryness. Uranium and the alkalies are extracted by water containing a little ammonium nitrate. The vanadium, in acid solution, is reduced with sulphur dioxide and the uranium with zinc and sulphuric acid, after which each is titrated with standard permanganate.

A second method is that of A. N. Finn.² The ore is dissolved in dilute sulphuric acid (1:5), evaporated to fumes, cooled and diluted. An excess of sodium carbonate is added, the solution boiled and filtered and the precipitate washed with hot water. The precipitate is redissolved in the smallest possible amount of sulphuric acid and reprecipitated with an excess of sodium carbonate in order to insure complete extraction of the uranium and vanadium. The combined filtrates and wash waters are slightly acidified with sulphuric acid, 0.5 g. of ammonium phosphate added and the uranium precipitated as phosphate by rendering the solution alkaline with ammonia. The solution is filtered and the uranium estimated by dissolving the precipitate in sulphuric acid and reducing with zinc. The filtrate from the uranium phosphate is acidified with sulphuric acid, the vanadium re-

¹ Amer. Jour. Sci., 10, 135 (1900).

² Jour. Amer. Chem. Soc., Oct., 1906.

duced with sulphur dioxide, and titrated, like the uranium, with a standard solution of potassium permanganate.

A third method is that of Fritchle.1 The ore is dissolved in nitric acid, diluted with water, sodium carbonate added in excess followed by a large excess of sodium hydroxide. The sodium hydroxide retains the vanadium in solution but leaves the iron and uranium undissolved. This precipitate, after washing, is dissolved in hot, dilute nitric acid and the iron precipitated with ammonium hydroxide, the uranium being kept in solution by the addition of a large excess of ammonium carbonate. The uranium is reduced with strip aluminum and titrated with permanganate. The precipitate of ferric hydroxide is dissolved in dilute sulphuric acid, the iron reduced with aluminum and determined with permanganate. In a separate sample the uranium, vanadium and iron are reduced together with sheet aluminum and titrated with permanganate, the vanadium being calculated from the total amount of permanganate less that required for the uranium and iron. The assumption is made that the amount of permanganate required to oxidize vanadium reduced by sheet aluminum is approximately twice that which would be required if the vanadium had been reduced with sulphur dioxide.

The object of the present research was to devise a volumetric method for the determination of uranium and vanadium in the presence of each other, the method not involving a gravimetric separation of these two elements.

G. Edgar has proposed a method for the differential reduction of iron and vanadium² and another for the differential reduction of molybdenum and vanadium.³ The iron is reduced to the ferrous condition and the vanadium to the V_2O_2 condition by the use of zine and sulphuric acid in a Jones reductor. The reduced solution is caught in a titration flask containing a ferric salt. The solution of highly reduced vanadium reduces the ferric iron to the ferrous condition and the amount of reduced iron present registers the reduction of the vanadium. The solution is then titrated as if a solution of ferrous iron alone were to be reoxidized with permanganate.

The reduction of a vanadium solution to the condition of V_2O_2 and oxidation in the ordinary

3 Ibid., 25, 332 (1908).

way with permanganate always fails to give accurate results for the reason that the solution of vanadium in this reduced condition has such a strong affinity for oxygen that it becomes partially reoxidized before the solution can be titrated. The use of the ferric alum as suggested by Edgar prevents this oxidation.

In the experiments tried in this laboratory slightly acid solutions of pure vanadyl sulphate and pure uranyl sulphate were used. The uranium solution was standardized by the usual method of precipitation and weighing as U_3O_8 ; also volumetrically by reduction with zinc and sulphuric acid and titration with standard twentieth-normal permanganate. The vanadium solution was standardized by reduction with sulphur dioxide, the excess of which was removed by the passage of a current of carbon dioxide through the boiling solution, followed by titration with permanganate.

Preliminary experiments were made to demonstrate that sulphur dioxide has no reducing action on uranyl solutions and that the titration of vanadium solutions when reduced with zinc and sulphuric acid to the V_2O_2 condition always requires less than three times the number of cubic centimeters of permanganate necessary to reoxidize when reduced with sulphur dioxide. Numerous experiments were made to determine the best conditions for the reduction and titration of uranium solutions.

Belouhoubeck in 1867 proposed the method for the reduction of uranyl solutions by zinc and sulphuric acid and titration with permanganate. Investigators since that time are much divided in opinion concerning the accuracy of results thus obtained. Some claim that the reduction proceeds further than the UO, stage and that the reduced solution needs exposure to the air in order to reoxidize to the UO, condition before the titration with permanganate is made. Among those supporting this view are Pullman,1 Goettsch,2 and McCov and Bunzel.3 Kern made extensive researches on uranium in 19004 and shows that the reduction does not proceed below the UO2 stage when sulphuric acid is used, even upon five hours' boiling. All of the above used not less than fifty grams of zinc, and the ratio of concentrated acid to water varied from 1:6 to 1:4.

Kern used sodium carbonate in the titration

³ Ibid., **31**, 367 (1909).

¹ Eng. and Min. Jour., 70, 548 (1900).

² Amer. Jour. Sci., 26, 79 (1908).

¹ Amer. Jour. Sci., 16, 229 (1903).

² Jour. Amer. Chem. Soc., 28, 1541 (1906).

⁴ Ibid., 23, 685 (1901).

flask to create an atmosphere of CO_2 in order to prevent the oxidation of the uranous solution by air, but Pullman is of the opinion that this means was ineffective and that the reduced solution was really reoxidized to the UO_2 condition before the titration was made.

In numerous reductions which were made in this laboratory as Kern directs, except that not more than 15 grams of granulated zinc were used in any one reduction, it was found that results were not very concordant. It was thought that less violent reducing conditions would more easily effect the reduction of uranyl compounds in solutions not so strongly acid.

Reductions were tried on several solutions of uranvl sulphate containing 0.1023 g. of elemental uranium, gravimetrically standardized. In some cases 5 g. of zinc were used with 5 cc. of concentrated sulphuric acid and 95 cc. of water. The reduction was carried on at a slow-boiling temperature in an Erlenmeyer flask, the mouth of which was closed with a cork through which a small funnel passed. In some cases 2 cc. of free acid were added to 95 cc. of water, and in other cases one cubic centimeter was sufficient to effect the reduction. About 5 cc. of concentrated sulphuric acid were added when the reduction was thought to be complete; the solution was cooled somewhat and was then rapidly filtered through glass wool to remove undissolved zinc. After dilution to 150 to 175 cc. the solution was titrated with twentieth-normal permanganate. Results obtained in this way showed but small variation among themselves. The average of a large number gave a uranium content of 0.1036 g., or a positive error of 0.0013 g. from gravimetric results. The average of a large number of reductions carried on exactly in the way that Kern directs gave a larger positive error than this. Kern states that at least 45 minutes are needed for the reduction of 0.1 g. of uranium solution, but the reduction in the presence of relatively small amounts of free acid was always complete in a half hour, and more often in 15 minutes on this quantity of uranium, 0.2 g, was easily reduced in this manner in 45 minutes. The conclusion is that a solution more nearly neutral is more desirable for the reason that it is more rapid and the results show less variation among themselves. Pullman and others speak of the appearance of brownish colors when the reduction is effected with such large quantities of zinc and acid. This indicates reduction

below the UO_2 condition. Working in the presence of relatively small amounts of free acid such color changes have not been noticed, and this may account for the uniformity in results obtained here, since there is no necessity for the reoxidation by atmospheric oxygen to the UO_2 stage.

Numerous experiments were made with other elements than zinc as reducing agents on mixed uranyl and vanadyl solutions. Among those tried were silver, lead, copper, and aluminum. Kern used aluminum¹ as a reducing agent for uranium and Fritchle used it for both uranium and vanadium. Silver, lead and copper offered no advantages which would suggest their use for this purpose. It was thought that some agent might be found which would reduce uranium to the UO, condition but at the same time would reduce the vanadium only to the V₂O₄ or V₂O₃ condition. The solution could then be tritated with permanganate in the ordinary way. Since uranium is more difficult to reduce than iron, and since vanadium is much more easily reduced than iron, any agent tried which completely reduced the uranium was found to reduce the vanadium to such a low state of oxidation that reoxidation by the air took place before the solution could be titrated. It was thought that possibly the oxygen of the air could be utilized in oxidizing the reduced solution to some definite point before titration with permanganate. Experiments were made to test the effect of bubbling air through the reduced solutions but it was found that conditions of acidity. concentration and temperature of the solution so influenced the results as to make them unreliable as a basis for a quantitative method.

Aluminum offered some advantages over zinc as a reducing agent for the reason that it left no residue in the solution and after reduction is completed the aluminum can be washed free of all adhering liquid very easily. It was effective in the reduction of both uranyl and vanadyl solutions. When strip aluminum was used in an open vessel with mixed solutions of these two elements, 2 or 3 cc. of sulphuric acid being present, reduction proceeded rather slowly. To hasten this process a spiral of heavy aluminum wire, wound to fit a ten-inch test-tube, was used. A mixed solution of uranyl sulphate and vanadyl sulphate was placed in the test tube and 50 cc. of water and 5 cc. concentrated sulphuric acid were added. The spiral was dropped in, the solution heated to

1 Jour. Amer. Chem. Soc., 23, 685 (1901).

boiling over the naked flame and then immersed in a bath of boiling water. The tube was covered with a watch-glass. Reduction began at once and about five minutes after the lavender color characteristic of V_2O_2 had appeared the reduction was found to be complete. The testtube and contents were cooled somewhat by immersion in running water, then the spiral was withdrawn by means of an aluminum hook fastened to a glass rod. A cubic centimeter or two of concentrated sulphuric acid was added and titration made with permanganate.

Using this manipulation with uranium solutions alone, the following results were obtained:

10 cc. uranyl sulphate solution required the following number of cc. of twentieth-normal permanganate:

1	1.	54
1	1.	34
1	1.	34
1	1.	40

15 cc. uranyl sulphate required

664

16.71

The calculated number of cubic centimeters of permanganate needed for 15 cc. of the above solution was 17.08. This figure was calculated from gravimetric results, weighing as U_3O_8 and assuming oxidation to take place on titration from UO_2 to UO_3 .

When vanadium solutions alone were reduced and titrated in the same way less permanganate than the calculated amount was always needed, and this was the case with mixed solutions of uranium and vanadium.

As the vanadium content was lessened in proportion to the uranium content the results came nearer the calculated amount and became more uniform. This would be expected on account of the strong tendency of V_2O_2 to reoxidize in contact with air. To prevent this reoxidation 50 cc. of a solution of ferric alum, made by dissolving 26 grams of the crystallized salt in a liter of water slightly acidulated with sulphuric acid, were used.

The manipulation was as follows: The mixed solutions of uranium and vanadium were placed in a ten-inch test tube, 2 to 5 cc. of concentrated sulphuric acid added with 50 cc. of water, the tube heated on the open flame to boiling, then covered with a watch-glass and immersed in a boiling water bath until reduction was complete as indicated by the lavender or grayish color of the solution. The tube is cooled in running water until hydrogen bubbles just cease to rise from the surface of the spiral, the spiral drawn to the top of the test-tube with the aluminum hook, 50 cc. of the cold ferric alum solution are poured over the spiral and allowed to mix with the reduced solution, the spiral is drawn out and the solution titrated with twentieth-normal permanganate in the test-tube until nearly finished; a little free sulphuric acid is added, if necessary, and the solution finished in an Erlenmeyer flask at a temperature of 80°.

Operating in this way the following results were obtained, using 5 cc. uranyl sulphate solution and 5 cc. vanadyl sulphate solution:

Cc. twentieth-norm	nal permanganate.
18.39	18.49
18.49	18.74
18.34	18.7
18.59	18.65
10 60	

The calculated amount of permanagnate needed is 18.6 cc. This calculation is based on the assumption that the vanadium is reduced to the V_2O_2 condition and requires three times as much permanganate as the same quantity reduced with sulphur dioxide, while the calculation for uranium is the same as in uranium alone above.

Satisfactory results were obtained in this way when the uranium and vanadium were present in equal quantities; when the vanadium is greatly in excess the tendency to reoxidize is harder to overcome and the results show more variation.

To apply this method to a carnotite ore the manipulation would be as follows:

0.3 to 0.5 g. of the ore is dissolved in an Erlenmeyer flask in 40 cc. of 1:5 sulphuric acid or a mixture of nitric and sulphuric acids, if desired, care being taken to expel all the nitric acid by evaporation. The solution is evaporated until the greater part of the acid is driven off. It is cooled, diluted, an excess of sodium carbonate added and, while boiling, hydrogen peroxide is added drop by drop until the color of the precipitated iron shows it to be in the ferric condition. The solution is filtered and the precipitate washed with hot water. The precipitate is dissolved in the smallest possible amount of I: I sulphuric acid, an excess of sodium carbonate added, after which it is boiled, filtered and washed. The combined filtrates and wash waters are acidified with sulphuric acid and an excess of the acid added amounting to 2 to 5 cc. of the concentrated acid. Sulphur dioxide is passed in and when the color

indicates that the vanadium is reduced the solution is boiled and a rapid current of carbon dioxide passed through the boiling solution to expel the last traces of the sulphur dioxide. The titration is made on the hot solution with permanganate of a strength not to exceed twentieth-normal. The iron precipitate can be dissolved and the iron estimated in the usual way if desired.

The volume at this point can not conveniently be more than 75 cc. if the reduction with the spiral is to be carried on in one tube.

If the ore is quite rich it is well to divide the solution here and carry the reduction on in two test-tubes. The two solutions may be combined by pouring into the Erlenmeyer flask near the end of the titration and the titration completed as if the reduction had been carried on in one tube. From this point on the procedure is the same as in the reduction of the mixtures of the pure salts described above. The number of cubic centimeters of standard permanganate used in the titration of the reduced solutions in the test-tube minus three times the number of cubic centimeters used in the titration after reduction with sulphur dioxide gives the number of cc. needed to reoxidize the uranium alone from UO_2 to UO_3 .

Four samples of carnotite thus treated gave for uranium, 15.92, 15.66, 15.26, 15.46 per cent.; for vanadium, 8.47, 8.57, 8.44, 8.44 per cent.

Edgar used a Jones reductor filled with zinc in the differential reduction of iron and vanadium and of molybdenum and vanadium. In a number of experiments in this laboratory, where a zinc reductor was used for the reduction of the uranyl and vanadyl solutions, the results obtained were not so satisfactory as those obtained with the apparatus above described. If it be true that the uranium is reduced below the UO2 state, as Pullman and others suggest, then to pass it into a solution of ferric alum at once would give too high results, as far as uranium alone is concerned, but since some provision of this kind is necessary to prevent the reoxidation of the highly reduced vanadium it appears that an error is likely to occur in one direction or the other whether the ferric alum be used or whether it be omitted.

The method above described furnishes a convenient means of separating uranium and vanadium without the necessity of gravimetrically separating these two elements and of determining each with a degree of accuracy fully equal to that which can be obtained by any of the methods heretofore published.

UNIVERSITY OF MICHIGAN, ANN ARBOR, June 14, 1909.

ON THE INFLUENCE OF THE TEMPERATURE OF BURNING ON THE RATE OF HYDRA-TION OF MAGNESIUM OXIDE.

By Edward De Mille Campbell. Received July 1, 1909.

In 1885 a court-house in Kassel, Germany, was seriously injured by expansion of the cement used in its construction. An investigation of the cause of this expansion was made by a government commission, of which Rudolph Dykerhoff, Dr. Ing. h. c., was Chairman. The cement used in the construction of the court-house was found to contain from 18-20 per cent. magnesium oxide. This led to a series of experiments to determine the influence of magnesia on Portland cement, and particularly the maximum amount which could be present without causing dangerous expansion. After more than twenty years of experimental work in Germany, France, England and the United States, different investigators have failed to come to complete unanimity regarding the maximum allowable per cent. of magnesia in Portland cement.

In an article entitled "Some Conditions Influencing Constancy of Volume in Portland Cements"1 which appeared in 1906, the author, in conjunction with A. H. White, gave the results of a series of experiments extending over a period of four years, which showed that the expansion of Portland cement caused by magnesia was due to the slow hydration of free magnesium oxide, and that combined magnesia is without effect so far as expansion is concerned. The fact that the amount of expansion caused by the presence of magnesia is due more to the form in which the magnesia exists than to the total amount present offers a simple explanation for the differences in opinion of different investigators concerning the maximum allowable per cent. The expansion bars made from neat Portland cement containing free magnesia show that when kept in water the bar containing I per cent. of free magnesia has ceased to expand at the end of three years, that with 2 per cent. at the end of six years, while those with 3 per cent. and 4 per cent. have apparently not ceased expanding after eight years' immersion.

1 J. Am. Chem. Soc., 28, 1273, Oct., 1906.

For many years it has been well known that certain natural cements, which are burned at a comparatively low temperature, but which often contain as much as 28 per cent. of magnesia, will give mortar or concrete as constant in volume as that made with good Portland cement. At the same time such natural cements cannot be depended upon, as not infrequently concrete made from such material will slowly expand and occasionally completely disintegrate from excessive expansion. Since these natural cements, high in free magnesia and yet giving satisfactory results, are burned at a much lower temperature than Portland cements, it would seem natural to expect that since the expansion is due to the hydration of magnesium oxide there must be some close relation between the temperature of burning and the rate of hydration.

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In a special publication by Rudolph Dykerhoff, Dr. Ing. h. c., entitled "Ueber die Wirkung der Magnesia in gebranntem Zement," published 44.40 per cent., loss on ignition 51.06 per cent., total 99.95 per cent. The composition of the burnt magnesite from such materials would be by calculation: silica 2.53 per cent., alumina and ferric oxide 2.70 per cent., calcium oxide 3.96 per cent., magnesium oxide 90.78 per cent. If there was no combination of magnesium oxide after dissociation of the carbonate with silica, alumina or ferric oxide, the amount of water required to completely hydrate the magnesia according to the equation

$$MgO + H_2O = Mg(OH)_2$$

would be 40.51 per cent. of the weight of the burnt magnesite. Under the same conditions, the amount of water which would be required to satisfy the equation

$$CaO + H_2O = Ca(OH)_2$$

would be 1.27 per cent. of the weight of the burnt magnesite.

In burning the various samples the method of procedure was substantially the same in all the

				T.	ABLE I.						
Length Hydr.	500°.	600 ⁰	700 ⁰ .	800°.	900 ⁰ .	1000 ⁰ .	1100 ⁰ .	12000.	13000	1400 ⁰ .	1450°.
1 day	24.83	36.61	36.51	36.52	23.40	18.00	7.61	0.43	0.46	0.58	0.34
3 days	25.45	37.63	37.92	38.65	41.00	41.93	22.84	2.05	1.25	1.24	0.93
7 days	25.64	37.93	38.30	38.51	42.26	42.38	39.22	4.85	2.17	1.85	1.55
14 days	25.89	38.21	38.55	38.72	42.59	42.73	41.65	15.28	3.32	2.74	2.43
21 days	26.09	38.38	38.69	38.89	42.75	43.06	42.34	32.23	4.43	3.56	3.29
28 days	25.48	38.03	38.45	38.64	42.05	42.73	42.13	36.15	5.75	4.11	3.35
2 months	30.58	40.63	40.61	40.41	44.46	44.39	43.97	38.92	17.36	13.24	10.64
3 months	30.51	40.38	40.47	40.33	44.51	44.57	44.69	39.12	20.29	17.55	14.81
4 months	30.66	40.38	40.47	40.29	44.58	44.57	44.69	40.02	22.55	20.48	17.55
6 months	30.59	40.35	40.45	40.29	44.64	44.70	44.86	40.48	24.54	24.12	20.45
9 months	30.39	40.35	40.39	39.94	44.72	44.85	45.18	41.19	27.48	27.60	22.59
12 months	30.34	40.34	40.53	40.32	44.88	44.92	45.33	41.34	28.99	28.12	23.31
18 months	29.99	40.03	40.14	40.03	44.63	44.54	45.34	41.74	31.22	30.31	24.85

in 1908, the author summarizes the results of his own experiments extending over a period of more than twenty years, and assumes that magnesium oxide burned at a low temperature hydrates readily and completely when immersed in water, but shows that the same oxide, when burned in a wind furnace at a temperature such as is employed for sintering Portland cement, combined with only about 3.5 per cent. of water during each of two evaporations to dryness.

The object of the experiments here described was to determine the quantitative relation existing between the temperature at which magnesium oxide from magnesite was burned and the rate at which such magnesium oxide would combine with water at ordinary temperature to form the hydroxide. The magnesite used for the work had the following composition: Silica 1.24 per cent., alumina and ferric oxide 1.32 per cent., calcium oxide 1.94 per cent., magnesium oxide

cases. For temperatures up to 1000° C. 16-18 grams of the finely ground magnesite, contained in a magnesium oxide crucible, were placed in a Heraeus platinum resistance furnace. Two standardized platinum-rhodium thermocouples were used for measuring the temperature. One couple was placed in the annular space between the crucible and the side of the furnace, while the other was embedded in the center of the finely ground magnesite. The temperature of the furnace was gradually raised until the thermocouple inside the crucible containing the magnesite had reached the desired temperature, and after this had been reached the temperature was maintained for one hour. The furnace was then allowed to cool and the burnt magnesite removed, ground to pass a 100-mesh sieve, and quickly stoppered. For samples burned above 1000° C. the magnesite was first subjected to burning at a low red heat in order to completely remove carbonaceous matter and so prevent de-



terioration of the thermocouple embedded in the magnesite when a high temperature was reached. These samples were burned in a specially designed platinum resistance furnace which was capable of maintaining a temperature of 1450° C. for a good many hours without volatilization of the platinum and consequent burning out. The control of the temperature was such that in no case did the thermocouple inside the crucible containing the magnesite vary more than four degrees in the hour during which the material was being held at constant temperature.

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Samples of one gram each of the burnt magnesite, ground to pass a 100-mesh sieve, were placed in a series of weighed porcelain crucibles. Sufficient distilled water was then placed in each crucible to completely cover the sample. As soon as the samples were covered the crucibles were placed in a vacuum desiccator containing concentrated sulphuric acid, and the water was allowed to evaporate. Samples which were covered with water one day and placed in a vacuum desiccator over sulphuric acid appeared dry the following day. The rack containing the set of crucibles was then placed in a drying-oven at from 102°-105° C. and allowed to remain over night. After cooling, the crucibles were weighed, and the gain in weight taken as the one-day hydration. After the one-day hydration water was again added, and the samples allowed to stand in a closed desiccator containing some water for two days, after which they were again dried in vacuo over sulphuric acid for one day, and again in a steam dryingoven over night. This second gain in weight was taken as the hydration after a total of three days. A similar method of hydrating and dryingfirst in vacuo, followed by steam drying-ovenwas used for the hydrations up to and including the twenty-eight day test. Through an oversight the samples in this last case were left in the drying-oven two days before weighing. The fact that the samples dried in the drying-oven lost appreciably after prolonged drying led to a change in the method of drying after the twenty-eight day test. Beginning with the two-month test the samples, after allowing to stand covered with water at room temperature, were placed in a vacuum desiccator over sulphuric acid and allowed to remain until the weight had become practically constant. This usually required from seven to nine days. The percentage gain in weight of the various samples, after the different periods of hydration, is given in Table I.

The table shows clearly that at 500° C. the magnesite was not completely dissociated in one hour. Experiments made in this laboratory upon calcite showed very slight dissociation at 600° C. after an hour's heating. Since magnesium oxide alone probably exists as such in the sample burned at 600° C., and since such magnesium oxide, as stated in the earlier part of this paper, would require 40.51 per cent. of the weight of the burnt magnesite for complete hydration, this amount has been taken as the basis for computing the curves given in 'Table II, which shows graphically the progress of the hydration.

A study of the curves, considered in connection with the fact that samples after hydration lose some of their water when dried in a steam-oven, seems to indicate the following conclusions:

r. That magnesite is not completely dissociated at 500° C. in one hour under the conditions used in the experiments.

2. That dissociation of the magnesium carbonate is complete at 600° , while that of calcium carbonate is not.

3. That the hydration of magneisum oxide burned at 600° , 700° or 800° C. is practically complete in three days.

4. That between 800° and 900° C. the calcium carbonate is dissociated, and that combination takes place between basic and acidic oxides, resulting in the formation of silicates or aluminates. The silicates or aluminates so formed combined with more water than would be required for the complete hydration of the basic oxides alone.

5. That a change in the constitution of the magnesium oxide sets in between 1000° and 1100° C. resulting in a marked decrease in the rate of hydration, and that this change becomes more marked with rise of temperature of burning, until at 1450° C., or nearly the temperature required for burning Portland cement, the magnesium oxide after eighteen months' immersion in water has combined with only 61.4 per cent. of the water required for complete hydration.

CHEMICAL LABORATORY, UNIVERSITY OF MICHIGAN, ANN ARBOR, June 18, 1909.

FREE LIME IN PORTLAND CEMENT. A MILL STUDY.

By ARTHUR G. SMITH. Received July 17, 1909.

The purpose of this paper is to present actual results obtained by testing for free lime both the night and day runs of portland cement as manufactured at the mill according to the microscopic method of detecting free lime, as published by Alfred H. White.¹

A preliminary statement as to how samples are taken and observed is necessary here to properly interpret the results following. From four tube mills grinding finished cement a sample was taken each hour in the twenty-four, upon which tests for fineness, setting time and soundness were made. This gave a very accurate set of results showing the nature of the product leaving the mills for each hour. The pats so made from these separate samples, were, after 24 hours, kept in boiling water seven hours and a careful record made as to their condition. It will be noted that the results are reported as the number of perfect pats, number of fair pats and number of bad pats. By perfect pats are meant those which were strong, neither checked nor warped, and free from cracks; fair pats, those slightly checked or warped; bad pats, those badly checked, cracked or disintegrated. In addition to the hourly samples taken, a general average sample was taken on each shift. To get this sample as nearly an average of the output of all four mills as possible it was decided to take it from the screw conveyor carrying the cement to the stockhouse. The method devised is as follows: In the bottom of the screw conveyor housing a hole about 1/8 inch in diameter was bored and so placed that it was directly under the flight of the conveyor. From this it will be seen that upon each revolution of the screw a small portion of the cement was forced through the opening and collected. It was upon this general average sample from each twelve-hour run that the microscopic tests were made. The results covering a twoweek run are herewith tabulated:

DAY RUN.

	Pr	ediction from	Boiling test.				
1909. Date.		of average sample.		General average.	Per- fect.	Fair.	Bad.
June	3	Perfect (Trace)	Perfect Total pats	47	45		2
	4	Perfect (Trace)	Perfect Total pats	48	41	4	3
u	5	Perfect (Trace)	Perfect Total pats	48	45	1	2
"	6	Perfect (Trace)	Perfect Total pats	48	47		1
u	7	Perfect 1/8 bad	Perfect Total pats	48	39		9
e	8	Perfect (Trace)	Perfect Total pats	46	44		2
u	9	Perfect (Trace)	Perfect Total pats	46	44		2

¹ THIS JOURNAL, 1, 5, January, 1909.

			DAY RUN (Cont.).			
]	Prediction from	Boiling	test.		
000	1	of average	Ceneral	Dor		10
Date		sample.	average.	fect.	Fair.	Bad.
une	10	Perfect	Perfect			
		(Trace)	Total pats 25	25		
"	11	Perfect	Perfect			
		About 1/6 bad	Total pats 48	40	1	7
"	12	Perfect	Perfect			
		1/4 bad	Total pats 48	38		10
u	13	Perfect	Perfect			
		(Trace)	Total pats 48	48		
"	14	Perfect	Perfect			
		(Trace)	Total pats 48	48		
	15	Perfect	Perfect	4.5		
"	14	(Irace)	Total pats 46	45		1
	10	Perfect	Total pate 43	36	2	5
		I/I Data	10tal pats 40	50	-	3
			NIGHT RUN.			
une	3	Perfect	Perfect	25		
"		About 1/4 bad	Total pats 44	35		9
	4	About 1/4 had	Periect 48	40	6	2
u	5	Perfect	Perfect	40	0	2
	3	About 1/4 bad	Total pats 48	39	4	5
u	6	Perfect	Perfect			
		(Trace)	Total pats 48	45	1	2
u	7	Perfect	Perfect			
		1/3 bad	Total pats 48	36	8	4
u	8	Perfect	Perfect			
		1/4 bad	Total pats 48	40	2	6
u	9	Perfect	Perfect			
		(Trace)	Total pats 37	36		1
"	10	Perfect	Perfect	S		
		(Irace)	Total pats 46	44		2
	11	Perfect	Total pats 48	32	5	11
"	12	Perfect	Porfact	02		**
	12	(Trace)	Total pats 48	45 .		3
u	13	Perfect	Perfect			
		(Trace)	Total pats 48	48		
u	4	Perfect	Perfect			
		(Trace)	Total pats 41	41		
и	15	Perfect	Perfect			
		1/4 bad	Total pats 38	32	4	2
"	16	Perfect	Perfect			
		1/8 bad	Total pats 43	37	3	3 *

In making our prediction with the microscope we endeavored to estimate the number of the hourly pats which we thought would be bad, judging from the amount of free lime present in the average sample. It will be noted that in most cases the prediction with the microscope checked very accurately with the boiling tests.

In predicting results by the microscopic method, we practically assumed that no other agencies besides free lime were causing unsoundness in the cement. We think we were justified in this, because the routine operation at this plant is very favorable for elimination of the other factors which might cause unsoundness except free lime, it being the standard practice to grind the raw materials, consisting of limestone and clay, to a fineness of not less than 97 per cent. through a 100-mesh screen and the finished cement to an average fineness of 98 per cent. through a 100-mesh sceen and 83 per cent. through a 200-mesh screen. Further, the raw materials contain less than the average proportion of ingredients which might cause unsoundness.

It will be noted that on each shift there were some few hourly pats which did not perfectly pass the boiling test. We attribute this to two causes: first, an occasional non-uniform mix of the raw materials, and second, to the formation of "rings" in the kiln. It is the nature of the class of raw materials used to cause "ringing" in the kilns, and when these so-termed "rings" have to be barred out, some underburned clinker is very likely to go through the kiln. This underburned clinker has no chance to age as the clinker passes direct from the kilns through a cooler and is ground to finished cement in from six to eight hours after leaving the kilns.

CONCLUSION.

It is the writer's belief that the microscopic test for free lime will be a great aid to the manufacturer. If the results obtained can be made quantitative, and an accurate method for sampling devised, good results must be obtained.

COPPER-CLAD STEEL—A NEW METALLURGI-CAL PRODUCT.

By WIRT TASSIN. Received July 1, 1909.

For many years past attempts have been made to cover steel with a copper coat which could be of any desired thickness, and to so firmly weld the two metals that the combined product could be submitted to any of the usual methods for working metals without destroying the integrity of this weld.

The many methods tried in the past have been more or less successful failures, either from a metallurgical or a commercial standpoint, and it is only recently that a process has been developed for the successful welding of copper to steel in such a manner that it will stand the many methods used for working metals, and be at the same time a commercial metallurgical product. The weld between the copper and the steel is perfect within the limitations of a metallurgical process. The copper can be separated from the steel only by melting it off. The weld will resist sudden temperature changes such as heating the combined metals red hot and then quenching them in icewater. It will resist both stress and shock. In the accompanying illustration (Fig. 2) a section of a round is shown that has had its copper cut through to the steel, the section placed in a vise and repeatedly struck with a hammer.

The process by which this result is obtained may be described as follows: Steel of any desired composition and shape, which, for the purpose of this description will be regarded as being a mild basic open-hearth steel, rolled into rounds and cut into 26" billets, to make wire rods, is sandblasted and pickled to remove scale. The billet, which has previously been drilled and tapped at each end, is hung by means of a rod and bushing screwed into one end, in a pre-heater and is brought to a red heat. When the desired temperature has been reached, the billet is then drawn into a tube (also previously heated) by means of a rod which is screwed into the top of the bushing. This rod slides up and down in the center hole of a threejawed chuck, which holds the tube and centers the billet in it. A steel flange is then screwed on the bottom of the billet, thus forming with the tube a mold in which the billet is the core.

The mold and billet are now carried to a pot of specially prepared copper which is in a supermolten condition. The billet with its attached flange is now lowered out of the tube and into this copper, and kept there for a length of time sufficient to wet the surface of the steel and to form an alloy film. The billet is then drawn from the copper up into the tube, and the billet and its mold is carried to a second pot containing commercially pure copper, in which the final coat of copper is applied. The mold with its billet as a core is lowered into this pot and the molten copper rushes in through two openings in the top of the tube until the mold is filled. The mold is then withdrawn from the second crucible, and when the copper has frozen, the chuck, rod and flange are unscrewed. The tube and its contents are next placed in a ram and the copper-clad steel billet is pushed out of its mold. The billet is now given a washing heat and is then rolled to the desired size.

The rolling is, in general, similar to that of steel or copper, and it is interesting to note that in spite of the great differences between the physical properties of copper and steel, the two metals when so welded have, under proper conditions, practically the same rate of flow. The proportional areas

of the copper and the steel remains practically a constant from the larger to the smaller sizes.

The tensile strength of copper-clad is also remarkable. Regarding the metal as having 40 per cent. of its sectional area made up of copper, its tensile strength after the proper treatment is equal to, and may be greater than that of a steel having a composition similar to that of the steel in the copper-clad, but whose sectional area is equal to that of the clad metal. An illustration of this may be seen in the following table of commercial wires, in which due allowance must be made for the difference between hard and soft drawing:

Name.	Diam. in inches.	Breaking weight in lbs.
Galvanized steel	0.162	1406
B. B	0.162	1250
E. B. B	0.162	1140
Copper	0.162	1237
Copper-clad	0.162	1874

having a relatively low elastic limit, gives a figure directly comparable with that of a steel treated under like conditions whose sectional area is equal to that of copper-clad, and whose composition is similar to that of the steel composing its core.

Copper and steel in the presence of moisture form a galvanic couple, and the corrosion of the steel proceeds with great rapidity. The resistance to corrosion of copper-clad so far as the copper coating is concerned is, of course, the same as that of copper. But, in view of the marked galvanic action set up between copper and steel, it would be assumed that corrosion would quickly occur at the exposed ends, and be a constant factor so long as an electrolyte was present. This supposition is not so, at least in the presence of fresh or salt water. Test samples placed in water, through which a current of air has been allowed to bubble con-



Fig. 1.

This table is an illustration of the fact that the tensile strength of copper-clad is not the mean of the strength of copper and steel, for the composition of the steel in the galvanized wire quoted is practically that of the steel in copper-clad, and, making liberal allowance for the difference in the heat treatment of the two, the breaking weight of the copper-clad is equal to, if not greater than, that of the galvanized, and but six-tenths of it is steel.

The above statement is also true of its elastic limit, which will average 90 per cent. of its tensile strength. The elastic limit of copper under the best conditions is 60 per cent. of its ultimate strength. That of steel is a variable, but for comparison take it at 90 per cent. Copper-clad steel, four-tenths of whose sectional area is a metal tinuously for three months, demonstrated that after a certain period of time, somewhere between 15 and 40 days, corrosion practically ceases, as shown by taking the loss in weight of the tests at varying periods. It is believed that this stopping of corrosion is a result of the following conditions:

After a certain amount of rust has been formed, it appears that a thin film of copper mixed with some copper oxide is plated out or deposited between the iron oxide and the unattacked steel, and that this film will act as a preservative coat as long as it remains intact. If broken, further oxidation sets up and the process simply repeats itself. While it is true that the corrosion on the end of a wire is not a factor in its life, yet the corrosion of the end of a relatively large diameter may become very serious. If the above observations hold true on larger sizes (so far it has not been tried on sizes above 3/8''), it will have quite a bearing on material suitable for marine work. Tests along this line are now being carried out, and the evidence to date points to a confirmation of the observations made on the smaller sizes.

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The uses to which copper-clad steel may be put are many. The first and most obvious is a wire to be used for electrical and mechanical purposes. The conductivity requirements for electrical use depends directly upon the amount and kind of copper used in the coat. Thus a copper-clad wire, four-tenths of whose sectional area is copper, 320 lbs. The one weighs 166 pounds per mile and the other 61. Comparing copper-clad with galvanized iron telephone wire, a much smaller size of copper-clad may be used for the same ohmic resistance, thus:

Wire.	Diam. in inches.	Ohms per mile at 68° F.	Weight per mile. Lbs.
E. B. B	0.134	18.83	250
B. B	0.134	22.04	250
Copper-clad	0.134	7.50	266

Where a high tensile strength and resistance to corrosion is an essential and an increased conductivity is desirable, as for example in power transmission requiring long spans and in catenaries, its value is apparent.



Fig. 2.

will have a conductivity of 40, since both copper and steel wire possess certain disadvantages for electrical work: the one having a low tensile strength and under given conditions a lack of toughness, the other having a low conductivity and is subject to a more or less rapid corrosion. Copper-clad wire has a strength and toughness equal to that of steel, a conductivity far greater than that of steel, and it will not corrode. For example, in telephone work the life of a line is dependent upon its breaking weight and elastic limit. The breaking weight and elastic limit of a No. 10 copper wire are respectively 530 and 293 lbs., while that of a No. 14 copper-clad is 760 and For mechanical purposes, such as bridge work, derrick guys, rigging, springs, rounds of all sizes suitable for anchor bolts, pump rods, etc., where resistance to corrosion combined with a high tensile strength is an essential, copper-clad is admirably adapted since steel having any desired physical qualities may be used as the core.

For large structural shapes it is questionable whether or not this material will ever have a commercial use, but for light shapes suitable for skylight and similar work, its value is obvious. It is non-corrodible and possesses the strength of steel. Copper is weak, galvanized iron corrodes.

To sum up, the material has a greater strength

than copper, and, under given conditions, is equal to, if not greater, than that of steel. It has a greater conductivity than steel and less than that of copper. Its resistance to corrosion is equal to that of copper and immeasurably greater than that of steel.

A SERIES OF PARALLEL DETERMINATIONS WITH THE MAHLER AND PARR CALORIMETERS.

By S. W. PARR AND W. F. WHEELER. Received July 10, 1909.

The samples used in this series of parallel tests were kindly furnished by fuel engineering companies and inspection chemists in the course of regular inspection work from the larger commercial centers, such as New York, Pittsburg, Chicago, Birmingham, Louisville, Topeka, etc. An examination of the record shows the samples to have come from the following states: fourteen from Pennsylvania, eight from West Virginia, seven from Illinois, six from Kansas, three from Kentucky, two from Alabama, and one each from Maryland and Indiana.

It was sought also to cover a wide range in composition, and in consequence the *c* h constituent is seen to vary from three per cent. to twentytwo per cent. and the sulphur from one-half to five and one-half per cent.

For the bomb calorimeter the Mahler-Atwater type was employed, being platinum-lined and using oxygen from the Lindé Air Products Company. The bomb was standardized by means of pure naphthalene, using 9692 calories as the value of that substance. A Beckmann thermometer, graduated to hundredths of a degree, corrected by the Bureau of Standards at Washington, was used in both series of tests.

In the Parr calorimeter the procedure was simplified to the extent that all coals were air-dried to a content of moisture not exceeding 3 per cent. and, in consequence, no further drying in the oven was needed. In addition to the sodium peroxide, the uniform amount of one gram of pure potassium chlorate as accelerator was used in the charge, with the usual one-half gram sample of coal. The rise in temperature was corrected by a factor made up of the following components:

Each per cent. of ash was multiplied by	0.00275°	· C.
Each per cent. of sulphur was multiplied by	0.005°	C.
The correction for heat reaction of the accelerator was	0.130°	C.
The heat of combustion for the fuse wire was	0.008°	C.

This correction factor was uniform for all of the coals excepting those of the Illinois-Indiana type. which had an additional correction of 0.024° C. This additional factor is made necessary by reason of the high percentage of hydration or water of composition which characterizes the coals of this type. They are readily distinguished by the fact that their normal content of water is high. exceeding 5 per cent. Another characteristic is the high content of volatile matter which results in a low ratio of fixed carbon, the latter constituent being almost without exception below 50 per cent. of the coal. The deriving of the correction factors in this manner greatly simplifies this phase of the process, and as is evident from an examination of the table, it is applicable to all the wide variations. not only in type of coal, but in ash and sulphur content. In the table of results which follows, the variations + are calculated to percentages of the Mahler values. It will be seen that the average variation of the entire forty-two coals is 0.03 per cent., the highest variation being 0.7 per cent. There are only three cases where this amount of variation occurs. The results are, therefore, in remarkably close agreement, indeed approximating the duplication of results that is ordinarily possible with good manipulation by means of the same instrument.

Attention should be called to the anthracites, Nos. 28 and 29. These have had a correction made for the unburned carbon. After dissolving the fusion and acidifying, the unburned material was caught upon a filter, dried and swept into a counterpoised capsule for weighing. Sample 28, for example, has as an average of three determinations, 2.82 per cent. of unburned carbon. The indicated heat value, therefore, applying the usual correction for ash, sulphur, etc., was 12775, and this multiplied by 1.0282, gave as a final value 13135 B. T. U. In sample No. 29 the indicated heat value was 12595 and the unburned coal was 2.867, hence the final value was 12595 × 1.0286 or 12955 B. T. U.

Concerning this additional correction, it may be well to note the necessity of making a similar correction for anthracites in using the bomb calorimeter. No determination on such material is reliable which does not take account of the unburned substance remaining in the bomb. This is especially notable in anthracites with an ash content above 6 or 7 per cent. For example, with the anthracite sample No. 28, the washings THE PARR CALORIMETER IN COMPARISON WITH THE MAHLER-ATWATER BOMB.

from the bomb were filtered through a Gooch crucible, weighed and ignited as for an ash determination. The loss of weight was 0.010. This loss represents pure carbon, hence it should have added to it the proportionate amount of ash and moisture in order to bring the loss to the same basis as the initial coal for correcting the weight taken or actually burned. The combined weight of ash and moisture is 0.1241 gram, hence the above loss of 0.010 is divided by (1.00 — amounts to 7.86 B. T. U. for each per cent. of calcium carbonate present and, as coals of the central-western type are not infrequent with four or five per cent. of this material present, it constitutes a variable that may properly receive attention where that type of apparatus is employed.

In the case of sample No. 45, use was made of this coal to test the effect of abnormally high ash upon the heat of fusion with sodium peroxide and thus verify the correction factor made use of for

Tab, No.	Lab. No.	Locality.	Moisture. Per cent.	Ash. Per cent.	Sulphur. Per cent.	Calories by Mahler- Atwater bomb.	Calories by Parr calorim- eter.	Diff. in calories.	Diff. in per cent. of bomb value. Per cent.
1	2090A	Williamson Co., Ill	. 2.32	11.60	2.67	6922	6907		-0.2
2	2111	Kanawha, W. Va	. 0.88	10.70	1.82	7571	7587	+16	-0.2
3	2112	Pittsburg seam	. 1.07	10.41	1.26	7496	7527	+31	+0.4
4	2113	Kanawha splint	. 1.12	14.94	0.92	6982	6970	-12	-0.2
5	2114	Fox Ridge, E. Ky.	. 1.31	8.63	4.41	7559	7540	-19	-0.2
6	2115	Pittsburg seam	. 1.00	11.35	1.79	7446	7449	+3	0.0
7	2116	Waverly, W. Ky.	1.88	11.92	3.83	6926	6929	+3	0.0
8	2119	Frontenac, Kan	1.16	12.14	4.80	7200	7159	-41	-0.6
9	2120	Frontenac, Kan,	1.79	8.08	3.20	7621	7618	-3	0.0
10	2121	Frontenac Kan	1 12	20.46	5 58	6405	6415	+10	+0.2
11	2122	Frontenac, Kan,	1.80	6.29	3.00	7786	7814	+28	+0.4
12	2123	Frontenac, Kan	1.43	13.76	3.44	7066	7084	+18	+0.3
13	2124	Frontenac, Kan	1.90	5.98	3.09	7783	7798	+15	+0.2
14	2125	Black Creek Ala	1 92	12 63	0.93	7434	7450	+16	+0.2
15	2126	Little Warrior Ala	0.88	15 53	0.82	7100	7153	+ 53	+0.7
15	2120	Pa Cas Wash Co Pa	1 53	8 32	0.02	7624	7650	+ 35	+0.5
17	2127	Ceorge's Creek Md	0.78	8 51	1 12	7004	7805	-9	-0.1
10	2128	Somerset Co. Pa. Knickerbocker	0.79	9.10	0.96	7954	7895		-0.1
10	2129	Somerset Co. Pa. Quemahonig	0.66	6.73	1.00	2000	2066	-20	-0.4
19	2130	Cambria Co. Pa. Hanriatta	0.70	4 80	0.72	8212	8201	-22	-0.3
20	2131	Cambria Co., Pa., Henriette	. 0.19	4.09	0.72	8313	8291	-22	-0.2
21	2132	Cambria Co., Pa., Heinfette	. 0.65	4.12	0.70	8400	8360	-40	-0.5
22	2132	Cambria Co., Pa., Naut. Y Gio	. 0.04	0.10	1.41	8179	8148	31	-0.4
23	2134	Cambria Co., Pa. Sterning	. 0.57	4.99	1.26	8285	8225		-0.7
24	2135	Cambria Co., Pa., Pardee	. 0.89	1.55	1.63	1951	7991	+34	+0.4
25	2136	Cambria Co., Pa., Miner's vein	. 0.73	4.39	0.52	8552	8313	-19	-0.2
26	2137	Cambria Co., Pa., Sugar Loai	. 0.66	5.89	1.31	8163	8176	+13	+0.1
27	2138	Cambria Co., Pa., Sugar Loar	. 0.51	6.06	2.64	8201	8148		-0.6
28	2139	Anthracite	. 1.19	11.22	0.80	7284	7296	+12	+0.1
29	2140	Anthracite	. 1.04	13.13	1.20	7153	7197	+44	+0.6
30	2141	Pocahontas	. 0.73	5.05	0.64	8294	8241	53	-0.6
31	2142	Pocahontas	0.91	16.51	1.25	7097	7109	+12	+0.2
32	2144	Floyd City	. 1.71	4.89	0.99	7808	7845	+37	+0.5
33	2180	Vermilion Co., Ill	. 2.28	22.40	5.07	5931	5920	-11	-0.2
34	2193	Indiana Block	. 2.39	8.14	2.54	7278	7309	+31	+0.4
35	1003	O'Fallon, Illinois	. 2.31	12.64	4.79	6605	6652	+47	+0.7
36	1114	Saline Co., Illinois	. 2.55	8.81	2.41	7194	7200	+6	+0.1
37	1543	Pocahontas	. 0.85	3:23	0.71	8447	8425	-22	-0.3
38	1545	Williamson Co., Ill	. 4.33	9.02	1.89	6966	6982	+16	+0.2
39	1763	Thayer, Ill	. 2.90	10.66	4.82	6755	6789	+34	+0.5
40	1878	Murphysboro, Illinois	. 2.41	4.64	0.69	7631	7581	50	-0.6
41	1884	Westmoreland, Pa	. 1.18	5.29	0.98	7998	8029	+31	+0.4
42	1999	W. Va. gas coal	. 0.94	3.66	0.70	8051	8073	+22	+0.3
43	2000	Pocahontas	. 0.52	5.26	0.56	8288	8247	41	-0.5
44	2005	Vermilion Co., Ill	. 2.23	17.48	4.32	6412	6393	-19	-0.3
45	2392	Bone coal	. 6.02	38.71	5.85	7240	7194	-46	-0.6

0.1241) or 0.8759, giving 0.0115 as the amount of unburned coal. This figure, subtracted from the coal as weighed out, gives the amount of coal actually involved in the combustion and which must be used in calculating the Mahler value.

Occasionally coals of the bituminous type are met with, sufficiently high in carbonate of lime to require a correction for the Mahler process, due to the heat required to dissociate the $CaCO_3$. This the ash. This is a bone coal and the correction factor amounts to a total of 0.270°. The results are in sufficiently close agreement to establish the correction factor as applicable to even such a high ash as is here shown. While the factors as herein made use of are slightly altered from those formerly published,¹ the variations are chiefly in the distribution of the correction factors. For ex-

1 Jour. Am. Chem. Soc., 29, 1606.

ample, the hydration of the ash is found to require a larger factor for that constituent with a correspondingly smaller one for that form of hydration which has been designated as water of composition.

With any method of heat determination too much emphasis cannot be placed upon the need of care in manipulation. It must be remembered that small errors in reading and a disregard for radiation and thermometer corrections are multiplied by two or three thousand in the final results.

UNIVERSITY OF ILLINOIS, URBANA, ILL.

[Contribution from the Laboratory of Agricultural Chemistry of the University of Missouri.]

THE DETERMINATION OF PHOSPHORUS IN FLESH.

[FIRST PAPER.]

By P. F. TROWBRIDGE.

Received July 6, 1909.

During the winter of 1907–8 the author attempted to prepare the ash from a number of samples of meats for a further study of the mineral constituents of flesh.

A large platinum dish was used and the ignition of the dry sample was made in a muffle furnace at a very low heat. The platinum was attacked within a very few minutes and the analysis of a piece of the fused bottom of the dish showed the presence of phosphorus. This raised the question whether or not phosphorus was being volatilized from the regular meat samples that were being ashed prior to a determination of the phosphorus.

The method which we had adopted for the phosphorus determination in flesh consisted in baking in triplicate about 10 grams of the fresh meat in tared crucibles in an air oven at 110°–120° and then gently igniting in an open crucible over a Bunsen flame to a complete ash. With many of the samples it was necessary to continue the heating eight or ten hours to obtain complete combustion of the organic matter. In spite of the care exercised, some of the samples of ash would fuse and then the particles of unburned carbon could be removed only by heating in the muffle.

The phosphorus was determined on the ash by the usual gravimetric method after digestion with $HNO_3 + HCl$. Later the Neumann method of digestion with $H_2SO_4 + HNO_3$ was used. Considerable difficulty was experienced in removing all the ash (especially when fused) from the crucibles. Some of the samples were digested for an entire day.

In order to assure ourselves that the methods employed were reliable, we determined the phosphorus upon another triplicate set of fresh samples, by digesting about 10 grams of the fresh meat exactly as for the nitrogen determinations, neutralizing the excess of H2SO4 with strong NH4OH, and proceeding with the usual gravimetric determinations. These parallel determinations were made upon seventy-two samples from three animals, being the wholesale cuts of the meats (exclusive of bone), the skeleton, and the internal organs. We have averaged the triplicate determinations by the two methods and find that thirty-three of the samples gave higher results by the determination of the phosphorus from the ash, the average result for the thirty-three samples being 0.0133 per cent. above the determinations by digestion with H2SO4. Thirty-seven of the samples gave an average of 0.0064 per cent. phosphorus higher by the method of digestion with H₂SO₄ than by the method of ashing. With two of the samples the average of the triplicates was the same for each method.

For the seventy-two samples the method by ashing gives an average of 0.0028 per cent. phosphorus above that obtained by the other method. The greatest excess which the ashing method gives above the other method is 0.0452 per cent., while the greatest excess obtained by the method of digestion with H₂SO₄ is 0.0147 per cent.

		Phosph	orus after of sample	ashing	ning Phosphorus after diges tion with H ₂ SO ₄ , etc.					
Lab. No.	Description of sample.	g.	h.	i.	v.	zv.	x.	g, h, i.	Av. of <i>v</i> , <i>w</i> , <i>x</i> .	Tota Av.
08.2.10	Digestive and excretory organs	0.2266	0.2234	0.2285	0.2250	0.2233	0.2212	0.2262	0.2232	0.2247
08.2.63	Digestive and excretory organs	0.2043	0.2083	0.2117	0.2181	0.2162	0.2132	0.2081	0.2158	0.2120
08.2.41	Digestive and excretory organs	0.1937	0.1915	0.1929	0.1984	0.1931	0.1914	0.1927	0.1943	0.1935
08.2.18	Lean of round and rump	0.1996	0.1977	0.2019	0.1972	0.1808	0.1895	0.1897	0.1892	0.1945
08.2.71	Lean of round and rump	0.2040	0.2098	0.1994	0.2172	0.2040	0.2122	0.2044	0.2111	0.2078
08.2.90	Lean of round	0.2059	0.2055	0.2083	0.2097	0.2077	0.2111	0.2066	0.2095	0.2081
08.2.20	Lean of loin	0.1944	0.1962	0.1960	0.1932	0.1938	0.1655	0.1955	0.1842	0.1899
08.2.22	Lean and fat of flank and plate	0.1157	0.1172	0.1163	0.1162	0.1134	0.1078	0.1164	0.1125	0.1140
08.2.23	Lean of rib	0.1785	0.1717	0.1773	0.1661	0.1697	0.1665	0.1758	0.1677	0.1718
08.2.55	Liver	0.3331	0.3366	0.3325	0.3302	0.2728	0.2761	0.3341	0.2930	0.3136
08.2.80	Offal fat	0.0336	0.0360	0.0358	0.0353	0.0343	0.0357	0.0351	0.0351	0.0351

In most cases the whole six determinations agree so closely that all the results come easily within the limit of error, and the separation into the two sets of determinations is purely for the purpose of comparison. The preceding table shows a few of the results of the separate determinations.

We can only conclude that, while ignition of meats in a closed muffle causes a loss of phosphorus, the ignition in open crucibles with only a moderate heat will not cause the loss of an appreciable amount of phosphorus. However, as Leavitt and LeClerc¹ have shown, too much care cannot be exercised in the digestion of the ash so as to put all the phosphorus in a form that will be precipitated by the ammonium molybdate. This seems to be most surely accomplished by the Neumann² method of digestion with equal volume of sulphuric and nitric acids; or by long (8 to 10 hours) digestion with nitrohydrochloric acid.

Credit is here given Mr. Norman Hendrickson for his assistance in the laboratory work.

COLUMBIA, MISSOURI, June, 1909.

ANTI-PUTRESCENT EFFECTS OF COPPER.

By Alfred Springer, Ph.D., assisted by Alfred Springer, Jr Received May 14, 1909.

Following a discussion before the Cincinnati Section of the American Chemical Society as to whether the peculiar behavior of a certain Cincinnati certified milk, most largely used in that city. warranted the suspicion that it contained an antiseptic, I started a series of experiments both with certified milk and check ones with milk from one of my cows, which I watched carefully so it could not be tampered with. Under like conditions these milks behaved with marked difference. After making several incineration analyses of the certified milk, I found a trace of copper therein, but its presence was not sufficiently conclusive, because the copper chlorides are more or less volatile. I therefore digested a half liter of certified milk with 350 cc. of sulphuric acid in a Kjeldahl flask, heating the same without addition of any extraneous oxidizers twelve days until it had become practically colorless, then drove off almost all of the sulphuric acid. Placing the residue in a beaker, after diluting it with water, I electrolyzed it. The deposit on the cathode was then dissolved in hydrochloric acid, the excess of hydrochloric. acid driven off, and then dissolved in water. To

this was added potassium ferrocyanide and the iron and copper ferrocyanide separated by means of ammonia. I then filtered out the dissolved cupric ferrocyanide, allowed the excess ammonia to evaporate, and obtained a decided copper reaction with every bottle of certified milk I examined. although the amounts varied considerably. From a half liter of milk the copper and traces of iron which always deposited on the cathode hardly ever weighed more than two milligrams, but generally much less, therefore its exact weight could not be estimated. I then made check experiments with milk from my cow, but could find no copper. although the methods of analysis were exactly similar. In order to convince myself that only to the presence of this substance could be attributed the strange behavior of certified milk, I started a series of experiments first with copper salts, afterwards with copper spirals placed in the milk. While copper salts have been used for antiseptic purposes, their particular application to milk have not received proper attention. Provided these salts or metallic copper come in contact with milk, immediately after milking, most deep-seated reactions take place; however, if the milk is allowed to stand a very short time and become infected with numerous lactic bacteria the cupric salts cannot suppress these unless abnormally large amounts thereof are used.

I observed when ammonium hydrate, in quantities of 0.1 to 0.15 per cent., is added to milk treated with copper salts a reddish brown change of color gradually takes place therein and the underlying liquid becomes turbid. Where no copper is in the milk this change does not take place. This reaction is most pronounced and affords a good test for the detection of copper in milk. Should, however, ammonium hydrate to the extent of more than 0.2 per cent. be added, milks with and without copper gradually assume the reddish brown color, but only those containing copper become turbid. The most marked change produced by the presence of copper is that the milk retains its sweet odor even after the acidity contents are sufficiently high to curdle it. The milk then becomes covered with molds and the odor becomes decidedly nutty without trace of putrescence.

Having now satisfied myself that the certified milk contained copper and having employed methods which indubitably proved its presence, I next determined to find out where and how it was

¹ THIS JOURNAL, 30, 617 (1908).

² Ibid., 24, 1106 (1902).

added to the milk in a manner to escape detection by the Milk Commission; furthermore, whether it was added with the intention to deceive or whether the dairymen were themselves the innocent victims of a process they employed. My son, Alfred Springer, Jr., assisted me in most of the coming experiments and I herewith wish to acknowledge the services rendered by him.

Supplying ourselves with some absolutely clean broad-mouthed glass-stoppered bottles, we went to Lebanon. Aug. 10th, to inspect the dairy in question. We found it a model of cleanliness in every respect. Having the presence of copper uppermost in our minds, we examined the various possibilities of its addition. The Star cooler, made of tinned copper, was the first apparatus that suggested itself as capable of supplying some. An examination thereof showed that the tin had partly worn off at some few places and that the filling valves were made of brass. We then followed the steam pipes used for the cleaning of the bottles, the sterilization of the pails, the straining cloths and the cloths for wiping the udders of the animals. The pipes were iron but all of the gate valves brass.

The superintendent accompanied us through the dairy and willingly complied with all of our requests. We asked that several cows be milked direct into the bottles we had brought along, so that the milk would not come in contact with any utensil they had in the building. This was done and called raw milk. Then we asked for two bottles of milk which, in the usual mode of procedure, had only gone through their straining cloth into the pails. This we called strained milk. We then took two bottles of milk which had passed through the Star cooler. All these bottles were immediately placed in their ice room, then packed in ice and taken to my laboratory; we there subdivided them by running 25 cc. in two-ounce bottles, leaving some closed in the ice-chest and others at room temperature. I also made Kjeldahl digestions of the three kinds. The strained and the certified showed the presence of copper but not so the raw, thus proving that the copper had already entered through the straining cloths and the pails. The raw milk both in the ice-chest and at room temperature, as indicated by titrations with decinormal solutions of sodium hydrate, was much better than the strained or the certified. Both the strained and certified turned the second day at room temperature, the whey separating

completely, whereas the raw milk remained sweet three days and then turned normally. In the ice-chest the raw showed great superiority over the strained and certified. These experiments indicated a most undesirable state of affairs. the danger of which cannot be overestimated, namely, that even in a dairy, like that at Lebanon, where all precautions for absolute cleanliness are scrupulously followed, a milk taken directly from the cow, bottled and iced, should be so much superior to others set out from ten to fifteen minutes in the dairy atmosphere. It showed that the atmosphere of the dairy was charged with bacteria and spores of molds which quickly found their way into the milk. We then determined to make a still more crucial test and visited the dairy Aug. 18th, first having supplied ourselves with perfectly clean bottles, which we brought with us. We asked the superintendent to milk six cows, part milked direct into the bottles, part through the straining cloths into the pails, and the rest sent through the Star cooler before any other passed over it. Some of the bottles which had been directly filled were immediately closed and others left open in the dairy fifteen minutes. Furthermore, we took samples of the condensed steam at the washing and sterilizing rooms, also of such used to sterilize the cooler. The various milks, all from the same cows, were taken to my laboratory and subdivided into 152 two-ounce bottles with 25 cc. in each, some set out open, others closed in the ice-chest and at room temperature, some treated with ammonia, and others left for the growth of molds. The results on the charts indicate that an exceptionally good milk direct from the udders of the cow had greatly deteriorated during the short exposure before bottling in the usual mode of procedure.

We examined the samples of water and found that the first one, condensed at the washing room, used to sterilize the bottles, pails and cloths, contained part of the boiler compound mechanically carried over and copper. The condensed water used to sterilize the cooler also showed the presence of copper. We next examined the boiler compound used at this dairy and, strange to say, found that it contained copper salts and was really largely at the bottom of the whole trouble. The priming or passage of foam carried this copper over to the washing and sterilizing room, thus contaminating the cloths, bottles and pails used.

While no exhaustive experiments have been

made to determine whether copper is unsanitary in such small amounts as I found in the certified milk, the very fact that it changes the normal characteristics of the milk should be sufficient for its exclusion. In certified and inspected milk dairies the so-called sterilization processes are produced by means of live steam. In many of these dairies the feed water is hard and the use of boiler compounds becomes necessary; these frequently prime over even through pipes connected with the steam drum. Every ingredient of a boiler compound, whatever it is, ought be rigidly excluded from contaminating the sterilized cloths, pails or other apparatus. This can effectually be done by interposing a still, fed directly with water, and heating it with closed steam from the boiler. This still and all pipes leading to the sterilizing apparatus should be made of block-tin.

While examining the certified milk I made quite a number of experiments with copper salts. The one great fact which always loomed up was that they, even in quantities of one part in two millions, act as antiseptics to putrescent bacteria. Organic matter in solution seldom oxidizes directly to its final stage, but passes through intermediate conditions whereby the more complex forms are broken down. Putrefaction is practically limited to the fermentation of albuminoid substances, and is largely a hydrolytic change caused by the energy of anaerobic organisms, while decay results from aerobic microbial activity.

In order to test the efficiency of copper salts I made the following experiments, keeping check ones without the copper: egg albumin and water, blood albumin and water, egg albumin, pancreatin and water, blood albumin, pancreatin and water; chopped meat in water; eggs placed in cupric sulphate solution; egg and blood albumin with sewage; milk with sewage. In every case the copper salts displayed either greatly retardative or marked inhibitive properties towards the putrefactive bacteria, although in some cases the strength of the solution was insufficient to prevent putrefaction gradually setting in. It seems to me that this property possessed by cupric salts is of great therapeutic value and ought to be carefully studied by competent physiologists.

Diseases owing to the abnormally increased presence of putrefactive bacteria are not uncommon, and heretofore have not been successfully combated by the use of antiseptics. It strikes me that in copper salts we have the means of introducing a substance powerfully antiseptic towards putrefactive bacteria and probably otherwise harmless, which could be administered so as to dissolve in the alkaline mucus, secreted by the intestinal glands, while approaching the colon. Again the antiseptic properties of the copper salts toward the sewage bacteria may be of especial benefit to the dairymen, since so many dairies are located near running streams which, through proximity to large cities, are nothing but open sewers.

OCTOBER 4, '08.

Milk from Springer cow, udders carefully washed, milked in the open and direct in the bottles. First bottle raw, second bottle contained Miami boiler compound I: 2500, third bottle contained copper salt, one part copper to 250,000. The bottles were kept cold in ice-chest.

			Cold boiler com-	Copper,
		Raw.	pound, 1 : 2500. 1	: 250,000.
Oct.	5	5.45	5.45	5.45
u	6	5.55	5.55	5.55
u	7	5.6	5.6	5.6
- 4	8	5.7	5.8	5.8
u	9	5.5	6.15	5.7
"	10	5.85	7.4	5.95
"	12	15.7	8.35	9.00
"	13	23.8	13.1	18.3
"	14	26.5, all but 4	19.5	23.5
		turned		
"	15	27.0	21.5	25.0
"	16	28.0, all turned	23.0	26.0
u	17	28.5	24.0	27.5
u	19	33.5	29.5, 2 of 5 turned	29.0
"	20	33.5	33.5, all turned	30.2
u	21	34.0	33.5	31.2
44	23	35.0	35.0	32.0
4	24	35.8	35.7	32.5
4	26	37.1	37.0	33.0, 1 in 15 turned
"	27	37.8	37.7	34.0
u	28	38.5	38.4	35.7, half the bottles turned

NOTES AND CORRESPONDENCE.

[CONTRIBUTION FROM THE DEPARTMENT OF AGRICULTURAL CHEMISTRY, UNIVERSITY OF MISSOURI.]

LIMITATIONS OF STARCH AS AN ACCELERATOR.

By C. K. FRANCIS.

In the course of several analyses of soils in which the sample was mixed with starch and sodium peroxide and then fused, it was found that the blank gave practically the same percentage of phosphorus as several of the soils. This fact led to an investigation of the starch employed, and upon obtaining a positive test, several other samples of starches were examined. The method of analysis was to digest 5 grams in HNO₈-HCl (30 cc.-10 cc.) to a clear solution, cool, dilute with water, and then determine phosphorus by the gravimetric method.

The results, with name and source of the starch, are shown in the following table

NOTES AND CORRESPONDENCE.

			Ash,	P ₂ O ₅ ,
	Charles and Charles		per	per
No.	Starch.	Obtained from	cent.	cent.
96128a	Potato starch	Eimer & Amend	0.292	0.151
" Ь	u u	"	0.292	0.159
96129a	Corn starch	"	0.548	0.036
" Ь	· u u	u u	0.560	0.039
96130a	Bulk corn starch	Ridenour, Baker	0.116	0.043
" Ь	u u u	Grocery Co., K. C.	0.124	0.047
96131a	Table corn "	Early Breakfast	0.104	0.032
" b	u u u	Coffee Co., St. L.	0.116	0.035
96132a	Wheat starch	Eimer & Amend	0.112	0.089
" Ь	u u	и и	0.124	0.093
96133a	Soluble starch	Kahlbaum	0.392	0.167
" Ь	u u	4	0.410	0.169

While the amounts of phosphorus found are relatively small, they are thought sufficient to be considered where starch is employed as an accelerator for sodium peroxide fusions, especially when analyzing certain soils or other materials which contain but traces of phosphorus. As phosphorus was found in all samples of starches examined, sugar has been substituted in its place for the fusion work of this laboratory.

COLUMBIA, MO.

FIRE TEST AS GAUGE OF DANGER FOR KEROSENE.

To the Editor of "The Journal of Industrial and Engineering Chemistry:"

There is a paper on "The Relation between the Temperature of Kerosene and the Explosive Pressure of the Supernatant Mixture of Air and Vapor" by W. P. Bradley and C. F. Hare in THIS JOURNAL, Vol. I, No. 6, June, 1909, p. 345. I read it with great interest. It begins to put the matter on a definite scientific basis.

They criticize fire test as a gauge of the danger of oil. Also I saw in the newspapers that an international committee was to report on methods of oil testing, and among other things on the taking of the firing point. Surely the one duty of scientific men to all present methods of taking the fire test is to condemn them as unscientific and misleading. In a test to indicate the safety or danger of an oil we require one that warns us of the beginning of danger like the Elliot closed flash point and not the much higher figures got in the fire tests. Neither of the fire tests given, Elliot and Tagliabue, "guarantee against danger while the oil is in bulk and kept at a temperature below that of the test." These tests are worse than useless, for the seeming guarantee suggests security and hinders natural precautions and thereby creates or increases danger. For bulk quantities the temperature of the close Elliot flash point is also the point of permanent ignition.

I took a shallow tin dish, circular, 9 inches in diameter, and covered it with a lid having circular holes to test by. An oil that in Abel close cup flashed at 78° F. (25.6° C.), not only flashed but ignited permanently, or fired, at 76° F. or 2° below the closed flash point. Using the same dish without the cover as an open test, applying the flame every 2° it ignited explosively at 88° F. (31° C.) and continued to burn furiously. Tested every degree the firing point was 87° F.¹ The point for flash or fire alters with the amount of oil, and the danger we wish to gauge is not that of cupfuls

¹ Chemical News, June 3, 1893, and Journal of Society of Chemicas Industry, **15**, 173 (1896). but the largest quantities in household use, say, large lamps or gallon tins.

The fundamental fact, then, is that for bulk quantities in circumstances where the vapors are allowed to accumulate on the surface of the oil, the closed flash point, Elliot or Abel, is also the fire point.

If the fire point determined in any way is more than 10° F. above the closed flash point then in my opinion the method ought to be condemned as misleading.

In England, shops and cellars have often been burned in hot weather by the kerosene, which was being sold, accidentally catching fire and burning furiously, flash point between 73° F. (23° C.) and 80° F. (27° C.), and not infrequently the flame has run along the heavy vapor some distance to the oil.

If an international or other scientific committee takes up the matter of fire test, I hope they fix on one that aims at what professes to be *the minimum* temperature of permanent ignition; and that for at least the largest quantities in common household use. But I think the fire test should be done away with and only the flash point used and all dangers referred to that standard. D. R. STEUART.

BROXBURN, SCOTLAND, July 5, 1909.

MOISTURE CONTENT OF BUTTER.

Editor, Journal of Industrial and Engineering Chemistry:

I have read with interest the Note of Mr. F. W. Robison in the May number of THIS JOURNAL, on "Variation of Moisture Content in Butter, Etc.," and as I differ quite decidedly would like to note a few points.

Butter, as every one knows, is not a homogeneous mixture, the content of water, curd, salt and fat varies in every portion of a mass of butter from any other portion of that mass. The better the butter is made, the closer will the different constituents agree. Butter containing more than 15.99 per cent. of water is considered adulterated by the Federal Government, and it is my experience that all butter handlers consider this a fair limit.

Thousands upon thousands of tubs of butter are sampled each year by the Revenue Officers, and the question of sampling has been a very important one. In the only case under the law where a creamery has sued the Government to recover the tax assessed for manufacturing adulterated butter, the testimony shows that the Revenue Agent in taking the samples used a trier for the first tub in the presence of the President of the Creamery and the butter-maker. The trier had been thrust one or more times into the tub when the creamery people objected and asked the officer to take the samples with a ladle, and not use the trier as the tubs of butter after being punched five or six times with a trier would not have much of a market value. The officer complied, and the remaining samples were taken with a ladle furnished by the creamery, yet when they protested against the assessment and the case came into the Federal Court, the cry was raised that the samples were not taken with the trier, and were unfair. (The Government won the case.)

While assisting a Revenue Agent take some samples with a trier, I asked the Commission Merchant if he had trouble selling butter which had been sampled with a trier; he said: "Oh I will sell this one tub but will never sell another to the same man." The Revenue Office has received so many complaints about sampling with a trier that the method had to be abandoned in the interest of the entire butter industry, as it is the universal statement of the butter trade that every tub of butter sampled with a trier by a Revenue Officer for moisture is ruined as far as its sale as table butter is concerned.

The instructions now are to take a sample from the top of the tub and one from the bottom, combine the two into one sample, or to take two samples from opposite parts of the sides of the tub and combine them. Our experimental work proves this method to give as fair a result for the tub as any possible method. The only complaint I have ever heard against this method of sampling butter, came from the above-mentioned creamery people, who wanted this method used when the samples were being taken, but who cried "unfair" when the case came into Court.

> Respectfully, A. B. ADAMS.

CHIEF, DIVISION OF CHEMISTRY, BUREAU OF INTERNAL REVENUE.

BOOK REVIEWS AND NOTICES.

Sprechsaal Kalender for the Ceramic, Glass and Allied Industries. J. KOERNER. 115 pp. Mueller & Schmidt, Coburg.

This publication of 115 pages is one of the most unique attempts in chemical technology. It summarizes all of the recent work done in Germany in the field of silicates, paying particular attention to the accurate, scientific researches such as the work of Berdel on the vitrification of clay substance, quartz and feldspar, insoluble glazes, testing of glass, Rieke's researches on mica, lime, magnesia and titanic acid, Simonis' results on ternary mixtures of kaolin, quartz and feldspar, etc. In addition a good deal of general information is given regarding bodies, glazes and glass which is very useful. Dr. Koerner is to be congratulated in producing such a radical change in the make-up of such a publication, and it is to be hoped that this feature will be maintained in the future and include also the results of American research along these lines. A. V. BLEININGER.

The Testing and the Properties of Sand-Lime Bricks. H. BURCHARTZ. Royal Materials Testing Station, Gross Lichterfelde. 105 pp. Julius Springer, Berlin.

Exhaustive tests and statistics on the properties of these bricks. This work is probably the most thorough ever done in this field. The recommendations for specifications governing the purchase of sand-lime bricks are especially interesting and are as follows: The bricks must possess good structure and must have a good ring. The water absorption should not be more than 15 per cent. of the weight of the dry brick. When used as backing up bricks or as facing they should have a crushing strength of not less than 2100 pounds per sq. inch; the regular run of hard brick should show a crushing strength of not less than 2800 pounds per sq. inch, tested in the dry condition. The bricks must not soften in water and the crushing strength when tested wet should not be lower than when tested dry by more than 15 per cent. They must resist 25 successive freezings. The loss in crushing strength on freezing should not be more than 20 per cent. as compared with the dry strength. The crushing strength to be accepted should be the average of 10 tests. Variations amounting to 10 per cent. below the minimum requirements in strength are permissible.

A. V. BLEININGER.

NEW BOOKS.

This list was prepared by Mr. Paul Escher, 1319 Pearl Street, Alameda, Cal.

Food Chemistry.

- Kakao und Schokolade. By E. LUHMANN. 1909. kl. 8°. 210 pp., 68 Abb. Hannover. Mk. 4, Lubd. 4.40.
- Neue Arzneimittel u. Pharmazeutisch Spezialitäten. By G. ARENDS. 3 A. kl. 8°, VIII, 681 S. Berlin, 1909. Lnbd. M. 6.
- Volksernährungsfragen. By MAX RUBNER. Leipzig, Akademische Verlagsgesellschaft m. b. H., 1908. 143 Seiten.
- Uebersicht üb. die Jahresberichte der öffentl. Anstalten zur technischen Untersuchung v. Nahrungs- u. Genussmitteln im Deutschen Reich. 1905. Lex. 8°. VIII, 348 u. 45 S. Berlin, 1909. Mk. 7.60.
- The Canning of Peas, Based on Factory Inspection and Experimental Data. By A. W. BITTING. Food Inspector, Bureau of Chemistry. pp. 32, figs. 6. (Bulletin 125, Bureau of Chemistry.) Price, 10 cents.
- The Influence of Acidity of Cream on the Flavor of Butter. By L. A. ROGERS and C. E. GRAV. pp. 22. (Bulletin 114, Bureau of Animal Industry.) Price, 10 cents.
- The Influence of Sodium Benzoate on the Nutrition and Health of Man. By IRA REMSEN and ASSOCIATES. pp. 784, charts 4. Referee Board of Consulting Scientific Experts. (Report 88, Office of the Secretary.) Price, \$1.00.
- The Science and Practice of Cheese Making. By L. L. VAN SLYKE and C. A. PUBLOW. 12°. 16, 483 pp. New York, 1909. Cloth, d. 1.75.
- Beiträge zur Synthese der Fette. By P. SCHACHT. Symmetrische Glyceride. 8°. 60 S. Diss., Zurich, 1908.

Handbuch der Drogisten-Praxis. By G. A. BUCHHEISTER. 1 Teil. 9. Aufl. gr. 8°. I X, 1115 S. m. 352 fig. Berlin, 1909. Mk. 12, Lnbd. 13.40.

- Practical Physiological Chemistry. By P. B. HAWK. 2nd ed. 8°. London, 1909, 16 sh.
- American Medicinal Barks. By ALICE HENKEL, Assistant Drug-Plant Investigations. pp. 59, figs. 45. (Bulletin 139, Bureau of Plant Industry.) Price, 15 cents.

Fermentation Chemistry.

- Anleitung zur Biologischen Untersuchung u. Begutachtung Von Bierwürze, Bierhefe, Bier u. Brauwasser, zur Betriebskontrolle Sowie zur Hefereinzucht. By H. WILL. XVIII u. 482 S. m. 84 Abb. u. 3 Taf. Munchen, 1909. Lnbd. 12 Mk.
- Handbuch des Weinbaues u. der Kellerwirtschaft. By A. FRHR. v. BABO and E. MACK. 1. Bd. Weinbau. 3 A. Hrsg. v. K. Mader u. F. Zweifler. 1. Halbbd. gr. 8°. XX, 623 S. Berlin, 1909. Lnbd. 16 Mk.

Agricultural Chemistry.

- Die Destillation Industrieller und Forstwirtschaftlicher Holzabfälle. By W. B. HARPER. Deutsch von R. Linde. 1909. Berlin, J. Springer. Mk. 10, geb. Mk. 11.
- Landwirtschaftlich-Technische Nebengewerbe. By K. STEIN-BRUCK. Kl. 8°. VIII, 59, I X, 160, I X, 134, VI, 130, VIII, 89 u. VII, 59 S. m. Abb. u. 5 Taf. Hannover, 1909. Lnbd. 7 Mk.
- Fumigation Investigations in California. By R. S. WOGLUM. Special Field Agent. pp. 73, Figs. 28. (Bulletin 79, Bureau of Entomology.) Price, 15 cents.
- Proceedings of the Twenty-Fifth Annual Convention of the Association of Official Agricultural Chemists, Held at Washington, D. C., November 12-16, 1908. Edited by Harvey W. Wiley, Secretary of the Association. pp. 248, figs. 6. (Bulletin 122, Bureau of Chemistry.) Price, 40 cents.
- Die Fabrikation des Superphosphats M. Berücksichtigung der Anderen Gebraüchlichen Düngemittel. By L. SCHUCHT.
 3 A. gr. 8°. VIII, 460 S. m. 153 Abb. u. 4 Taf. Braunschweig, 1909. M. 18, gebd. 20.
- Die Vermehrung der Kaliwerke u. der Kaliabsatz. By H. MÜNSTER. Halle a/S, 1909. M. 3.60.

Electrochemistry.

- Die Elektrochemischen Verfahren der Chemischen Grob-Industrie. By J. BILLITER. Bd. I. Elektrometallurgie wässriger Lösungen. Halle a/S, 1909. 12.
- Elektrochemie Geschmolzener Salze. By R. LORENZ and E. KAUFLER. Lex. 8°. VI, 84 S. m. 17 Abb. Leipzig, 1909. 3.60.
- Calcium-Carbid und Seine Volkswirtschaftliche Bedeutung F. Deutschland. By W. LE COUTRE. Berlin, 1909. Mk. 3.
- Production Electrique de L'Ozone et Applications A L'Industrie, L'Hygiene, La Therapeutique. By E. DOUZAL. 8°. III. Paris, 1909. Fr. 15.

Analytical Chemistry.

- Ueber die Anwendung der Thermischen Analyse zum Nachweis Chemischer Verbindungen. By R. KREMANN, Lex. 8°. 76 pp. 43 Abb. Stuttgart, 1909. Mk. 2.40.
- Logarithmische Rechentafeln für Chemiker, Pharmazeuten, Mediziner und Physiker. By F. W. Küster. 9th Aufl. 12. Leipzig, 1909.
- Einrichtung von Laboratorien und Allgemeinen Operationen. By V. SAMTER, Halle a/S, 1909. Mk. 2.70.
- Laboratoriumsbuch F. die Industrie der Verflüssigten u. Komprimierten Gase. By K. URBAN. Gr. 8°. VIII, 40 S. m. 24 Abb. Halle a/S, 1908. 1.80

Coal, Fuel and Petroleum.

- Analyses of British Coals and Coke, and the Characteristics of the Chief Coal Seams Worked in the British Isles. By A. GREENWELL and J. V. ELSDEN. 3rd ed. 4°. London, 1909. sh. 10.
- Tests of Coal and Briquets as Fuel for House Heating Boilers. By D. T. RANDALL. 8°. 44 pp. London, 1909. 1 sh. 6 d.
- Das Erdöl. Hrsg. v. C. ENGLER u. H. HÖFER. 2 Bd. Die Geologie, die Gewinnung und der Transport des Erdöls. Lex. 3. 992 S. m. 307 Abb. u. 26 Taf. Leipzig, 1909. Mk. 46, Hlbfrz. 50.

Explosives.

- Explosivstoffe, auf Grund des in der Literatur Veröffentlichten Materials Bearbeitet. By H. BRUNSWIG. 1909. 177 pp. 45 Fig. 56 Tab. Leipzig. Mk. 8, geb. Mk. 9.
- Lustfeuerwerkerei für Berufsfeuerwerker und Leibhaber. By H. FALBESONER. Mit 391 verschiedenen Kompositionen u. Angabe der Bereitung aller im Handel schwer erhaltl. Präparate. gr. 3. 256 S. m. 100 Abb. Wien, 1909. 5, gebd. 6.

Photography.

Das Arbeiten Mit Farbenempfindlichen Platten. By E. König. (Photogr. Bibl. 25 Bd.) 8°. VII, 76 S. m. Fig. im Text u. 16 Taf. Berlin, 1909. 2.25, gebd. 2.85.

Water and Water Supply.

The Disinfection of Sewage and Sewage Filter-Effluents. By E. B. PHELPS. 1909. Water-Supply Paper No. 229, U. S. Geol. Surv., Washington.

Technical Chemistry.

- Wasch-, Bleich-, Blau-, Stärke- und Glanzmittel. By L. E. ANDES. 1909. 384 pp. 21 Abb. Wein, Mk. 5, geb. Mk. 5.80.
- Practical Electroplating: A Guide for the Electroplater. By W. L. D. BEDELL. 12. 244 pp. Newark, 1909. Cloth, \$2.00.
- Das Salz, Dessen Vorkommen und Verwertung in Samtlichen Staaten der Erde. By J. O. FRHR. v. BUSCHMANN. I. Bd. Europa. Lex. 8°. 784 S. Leipzig, 1909. 26, Hlbfrz. 30.
- Die Starkefabrikation. By J. SCHMIDT. (Bibl. d. ges. Landwirtschaft. 39 Bd.) I X, 134 S. m. 44 Abb. u. 5 Taf. Hannover, 1909. Mk. 2.20, Lnbd. 2.50.
- Betrachtungen ub. die Bewegende Kraft des Feuers u. die zur Entwicklung Dieser Kraft Geeigneten Maschinen. By S. CARNOT. (1824). Ubers. u. hrsg. v. W. Ostwald. (Ostwald's Klassiker d. exakt. Wissenschaften Nr. 37) 2. A. 8. 72 S. m. 5 Fig. Leipzig, 1909. 1.20.
- Air Liquide, Oxygene, Azote. 8°. 403 p. av. fig. Paris, 1909. Fr. 15.
- L'Industrie Des Parfums D'Apres Les Theories de la Chimie Moderne. By M. Orro. Notations et formules. Les parfums naturels. Les parfums artificiels. 8°. VIII, 545 pp. av. fig. et portr. Paris, 1909. Broche Fr. 22.50.
- Handbuch der Farbenlehre. By E. BERGER. 2 A. kl. 8. XIV, 304 S. m. 36 Abb. u. 8 Taf. Leipzig, 1909. Lubd. Mk. 4.50.

Metallurgy and Geology.

- Metallurgical Calculations. By J. W. RICHARDS. Part III. The Metals Other than Iron (Non-ferrous Metals.) 8°. New York, 1908 (1909).
- Welding and Cutting Metals by Aid of Gases or Electricity. By L. A. GROTH. 8. London, 1909. 19 sh. 6 d.
- The Preservation of Iron and Steel. By ALLERTON S. CUSH-MAN. Assistant Director, Office of Public Roads. pp. 40, pls. 4. (Bulletin 35, Office of Public Roads.) Price, 10 cents.
- Radioactivity and Geology. By J. JOLY. 8. London, 1909. 7 sh. 6 d.

Bibliography of North American Geology for 1906 and 1907. By J. M. NICKLES. With subject-index. 1909. Bull. 372, U. S. Geol. Surv., Washington.

Miscellaneous.

Kapillarchemie. By H. FREUNDLICH. Eine Darstellung der Chemie der Kolloide und verwandter Gebiete. Gr. 8°. 600 S. m. 93 Fig. u. Kurv. Leipzig, 1909. 16.30, gebd. 17.50.

SCIENTIFIC AND INDUSTRIAL SOCIETIES.

SEVENTH INTERNATIONAL CONGRESS OF APPLIED CHEMISTRY LONDON, MAY 27th TO JUNE

2nd, 1909.

REPORT OF SECTION VIII C.

Bromatology.

FRIDAY, MAY 28TH, PRESIDENT R. R. TATLOCK.

"The Systems in Force for the Control of the Food Supply." This subject was discussed by Dr. Wiley, America, Dr. Roun and Dr. Bordas, France, and Dr. Buchanan, Great Britain. Further discussion was postponed until additional papers were read on Monday the 31st at 10 A.M.

SATURDAY, MAY 29TH, PRESIDENT DR. BORDAS.

The morning was devoted to reports and discussion by the International Commission for the Unification of Analytical Results.

MONDAY, MAY 31ST, PRESIDENT DR. BORDAS.

The discussion of the subject "The Systems in Force for the Control of the Food Supply" was continued by Dr. Wauters, Dr. Kerp, Dr. McGill, Prof. Schaffer, Prof. Wysman and Dr. Van Rijn. Drs. Thorpe, McKerp and Blitz also discussed the subject. Papers on milk analysis were presented by Mr. Taffe, Messrs. Russell and Armaud and Dr. Bordas.

The International Commission on the Unification of Analytical Methods reported that several meetings had been held and a plan of organization adopted. Methods of analyses prepared by Messrs. André, von Buchka, Chapman, Cribb, Lavalls, Schoepp, Mastbaum, Pintti, Vandevelde, Wauters and Wiley were reported. Several new members were added to the commission and the following officers elected: M. von Buchka, *President;* Messrs. Gautier, Thorpe, Pintti, Schaffer, Wauters, Wiley, Wysman, *Vice-Presidents;* and Mr. Vandevelde, *General Secretary*.

P.M., DR. BORDAS, PRESIDENT.

Papers on the composition of brandy and other spirits were read and discussed. The following resolution was adopted "That brandy is a product of the distillation of wine, and the term is synonymous with eau de vie de vin."

TUESDAY, JUNE 1ST, 10-12.30 A.M., PRESIDENTS, PROF.

WYSMAN, M. WAUTERS, DR. BUCHKA.

F. W. Beck read a paper "On the Advantages and Disadvantages of Legally Binding Standards for Foods and Drugs." This was generally discussed and at 12.30 the Section adjourned to be photographed.

12.45-1.30, JOINT MEETING WITH SECTION I (CHOCOLATE). PRESIDENTS DR. KLOP, PROF. WYSMAN.

Several papers dealing with cocoa and chocolate were presented.

P.M., 4-6, Presidents Dr. McGill, Dr. Buchanan, Mr. Ferriers.

Papers on "The Use of Preservatives in Food" by Mr. Bevan, Dr. Thresh and Dr. Wiley were read and discussed. The Section adjourned at 5.30 *sine die.*

F. C. Соок.

RECENT PATENTS.

The following patents relating to industrial and engineering chemistry are reported by C. L. Parker, solicitor of chemical patents, McGill Building, 908 G Street, N. W., Washington, D. C.

922,494. Process of Making Fertilizing Material. JOHN W. LOWMAN, Nashville, Tenn. May 25, 1909.

This is a process of producing a phosphatic fertilizer, by pulverizing a predetermined amount of phosphate material, burning and pulverizing a predetermined amount of dolomite or magnesia limestone, burning and pulverizing a predetermined amount of fluor-spar, thoroughly mingling all of the said substances, adding a predetermined amount of potash thereto, reducing the product thus formed to a plastic mass by stirring water thereinto, and then heating the said mass.

922,523. Flux for the Autogenous Welding of Aluminum. MAX U. SCHOOP, Bois, Colombes, France. May 25, 1909.

The flux consists of chloride of potassium, chloride of sodium, bisulphate of potassium and chloride of lithium.

923,005. Process of Treating Nickle Ores. Anson G. BETTS, Troy, N. Y. May 25, 1909.

This is a process of treating an ore containing a metal of the iron group, by smelting to an alloy with another metal of the iron group, and electrolytically refining the alloy, using an electrolyte containing a soluble salt of each of such two metals, and producing a refined alloy containing the said two metals.

923,152. Silicon Alloy. JOHN T. H. DEMPSTER, Schenectady, N. Y. Assignor to General Electric Company, a corporation of New York. June 1, 1909.

The alloy consists of silicon and tellurium.

923,228. Method of Insulating Sheet Metal. WILLIS R. WHITNEY, Schenectady, N. Y. Assignor to General Electric Company, a corporation of New York. June 1, 1909. The process consists in coating a sheet-metal plate with a

colloidal suspension of gum copal and then heating said plate to produce a uniform adherent coating of gum copal.

923,232. Process for Making Alcohol. ADOLPH WOOLNER, JR., and ALADAR LASSLOFFY, Peoria, Ill. Assignors to Woolner Distilling Company, Peoria, Ill., a corporation of Illinois. June 1, 1909.

This is a process for the manufacture of alcohol consisting in growing in distillery shop fungi having diastatic properties, combining the same with a malt converted mash and distilling the mixture after fermentation.

923,358. Non-conducting Compound. FRANC J. JEWETT, New York, N. Y. Assignor to H. W. Johns-Manville Company, a corporation of New York. June 1, 1909. This is a composition of matter for use in heat insulation comprising short asbestos fiber, granulated cork, disintegrated sponge treated with a boron compound and infusorial earth.

923,411. Manufacture of Zinc Dust. SHERARD O. COWPER-COLES, London, England. June 1, 1909.

The process consists in electrodepositing zinc from a caustic soda solution upon a revolving disk upon which the disk is deposited in the form of a sponge and is continuously removed, and in then drying the spongy zinc so produced in a reducing atmosphere.

923,427. Process of Treating Petroleum Sludge to Produce Pitch, Asphalt, Etc. JOHN L. GRAY, Elizabeth, N. J. June 1, 1909.

The process consists in digesting the sludge until the major part of the acid has been removed, and then slowly heating.

923,428. Process of Treating Petroleum Sludge. JOHN L. GRAY, Elizabeth, N. J. June 1, 1909.

This is a process of separating the constituents of petroleum sludge into various grades by pumping off any mechanically entrained oil which may rise to the top, digesting the sludge until the light constituents including the unsaturated hydrocarbons rise to the top, withdrawing this by-product and the acid, then digesting again until the lighter grade rises, removing that grade and the acid, and so continuing until there remains a solid residuum.

923,429. Process of Separating Acid from Petroleum Sludge. JOHN L. GRAY, Elizabeth, N. J. June 1, 1909.

The process consists in washing or digesting the sludge with a weak acid solution.

923,581. Process for the Production of Carbon-Covered Metal Sheets for Dynamo-Brushes. JOHANNES F. P. RINGSDORFF, Essen-on-the-Ruhr, Germany. June 1, 1909.

The process consists in smearing a carbon paste on the surfaces of said metal sheets and subjecting them to hydraulic pressure.

923,846. Process of Extracting Nitrogen from Air. OLIVER P. HURFORD, Chicago, Ill. June 1, 1909.

This process is practiced by the use of the apparatus illustrated, in which carbonaceous fuel is burnt in the furnace shown at the left of the illustration, air being admitted in amounts sufficient for complete combustion. The gaseous products of combustion which are free from carbon monoxide



enter through a chamber where they are subjected to the action of lime water to absorb the carbon dioxide and the nitrogen being dried by the means of lime or other drying agent is drawn off for use.

923,864. Process for the Electric Dissociation of Metals by the Wet Method. June 1, 1909.

This is an electrolytic process for cleaning metal articles and subsequently coating them with metal which consists in placing the article to be coated in a bath consisting of a concentrated alkaline solution and an alkaline cyanide, placing in the solution a quantity of the metal of the kind of which the coating is to be formed, and connecting both the article and the coating metal to a suitable source of electricity, the former as a cathode and the latter as the anode.

923,891. Manufacture of Bituminous Roadways. CLIFFORD RICHARDSON, New York, N Y. Assignor to the Barber Asphalt Paving Co., Philadelphia, Pa., a corporation of West Virginia. June 8, 1909.

This is a process of preparing bituminous paving material which consists in employing a mineral aggregate of two sizes, the one consisting of No. 2 stone and the other screenings from three-eighths of an inch to dust, the latter being proportioned to fill the voids of the former; adding to the large sized material while cold a hot bituminous cement in amount more than sufficient to coat the stone; thoroughly mixing; and then adding the finer material.

923,916. Method of Thionous Precipitation. GEORGE C. WESTBY, Murray, Utah. June 8, 1909.

This is a method of precipitating copper or silver from their solution by means of the addition of a liquor containing thio salts of iron.

923,967. Process of Producing Camphene. CHARLES GLASER, Baltimore, Md. June 8, 1909.

The camphene is produced from bornyl chloride by the distillation of that substance at a temperature between 180° and 210° C. and for a time refluxing the distillate, then separating the distillate and collecting it.

923,076. Process for Obtaining Pure Copper. LUCIEN JUMAU, Paris, France. June 8, 1909.

The process consists in obtaining pure copper from a solution of a salt of copper, by subjecting said solution to the action of carbon monoxide in a closed receptacle, and heating the solution during such treatment.

923,077. Process for Obtaining Pure Metallic Copper from a Solution of Salt and Copper. LUCIEN JUMAU, Paris, France. June 8, 1909.

The process consists in obtaining copper from a solution containing salts of copper and subjecting said solution to heat and pressure; in the presence of a reducing agent containing hydrocarbons.

924,677. Process of Smelting Iron Oxides. HORACE W. LASH, Cleveland, Ohio. June 15, 1909.

The process consists in charging into a furnace a smelting mixture containing finely divided cast iron and carbonaceous material, and distributing in the mass of the smelting mixture chunks or masses of pig metal and applying a smelting heat to the charge. 924,770. Art of Treating Wood for Lumber. WILLIAM A. HALL, New York, N. Y. Assignor to American Mahogany Company, a corporation of Maine. June 15, 1909.

The wood is impregnated with an alkalized solution of the phlobaphenes of the hemlock bark, which solution contains an excess of alkali over that necessary to dissolve the phlobaphene extract.

924,818. Process of Producing High-grade Steel from Lowgrade Material. WILLIAM R. PALMER, Bridgeport, Conn. Assignor one-half to Frank A. Wilmot, Bridgeport, Conn. June 15, 1909.

The process consists in charging a primary furnace, with low-grade material of the character described, operating said furnace at a temperature below ordinary steel-making temperature, partially charging a secondary furnace with scrap and flux, operating said furnace at ordinary steelmaking temperatures, transferring a portion of the charge from the primary furnace to the secondary furnace for the completion of the refining operation, and returning a portion of the charge to the primary furnace to form a bath to receive the next charge of the low-grade material.

925,253. Process of Making Ammonia and Chlorin or Hydrochloric Acid. NORMAN L. WHITEHOUSE, Lewisham, London, England. June 15, 1909.

This is a process for obtaining chlorine from ammonium chloride consisting in heating at a low temperature ammonium chloride with an oxide of a rare earth metal, adapting to expel ammonia from ammonium chloride at that temperature and then heating the residue at a higher temperature in an atmosphere adapted to expel chlorine from a chlorine compound of a rare earth metal at the said higher temperature.

925,321. Process for Facing Stereotype Metal. WILLIAM R. FISHER, San Francisco, Cal. Assignor one-third to Joseph F. Moran, San Francisco, Cal. June 15, 1909.

The process consists in facing stereotype and like plates with a protecting metal by first washing the plate in caustic potash, then placing it in a bath of potassium cyanide, again washing the plate and placing it in a solution of platinum cyanide, and subsequently applying the facing metal by electro-deposition.

925,365. Metallization of Vitreous Ceramic Surfaces. QUENTIN MARINO, London, England. Assignor threefourths to Edwin Joseph Richardson, London, England. June 15, 1909.

The metallizing of the ceramic surface is accomplished by applying thereto a solution resulting from dissolving in hydrofluoric acid a gray substance consisting of filiform silver and basic nitrate of copper, and then, while the surface is wet with the solution, dusting said surface with a metal in a finely divided state and such as will reduce the silver and copper in the fluoride to a metallic state.

925,612. Refining of Steel in Electric Furnaces: OTTO THALLNER, Bismarckhutte, Germany. Assignor to The Firm of Bismarckhutte, Bismarckhutte, Germany. June 22, 1909.

This is a process for producing high-quality steel by preliminary refining of the steel in a fuel-heated furnace, then passing the steel so refined for treatment in a basic-lined electric furnace, after which the steel is passed to an acidlined electric furnace.

925,626. Apparatus for Recovering Metals from Ores and Other Substances. LEON DION, New York, N. Y. Assignor to the American-Hermatic Company, New York, N. Y., a corporation of Arizona. June 22, 1909.

In the treatment of ores to recover the metals contained in them, part of the metal passes off, in the form of fumes or vapors, when the ores or other substances containing them are subjected to the required heat to fuse them. The metal or metals thus carried off in the shape of fumes, gases or vapors may be concentrated or condensed, and thereby recovered or reclaimed, by subjecting these latter to the



action of an electric current supplied through appropriate electrodes, the result of which is to cause the particles of metal contained in or forming the fumes, or vapors of each to cohere and form masses of appreciable size, which are then thrown down or precipitated by the action of gravity or otherwise and may be recovered in that form. The apparatus illustrated is designed for use in carrying out this process, the metal laden fumes' being subjected to the action of a suitable electric current to precipitate the metal.

925,670. Composition for Oxidizing and Purifying Explosive Gases. HERBERT WALKER KNIGHTS, Transvaal. June 22, 1909.

This is a composition for destroying the noxious or poisonous fumes or gas arising during the blasitng in mines, consisting of permanganate of potassium, chloride of ammonium, and chlorate of potassium.

926,280. Process of Preparing Concentrated Hydrated Magnesia. WINFIELD S. MORRISON, Oakland, Cal. June 29, 1909.

The process consists in preparing a concentrated magma of magnesium hydroxide, by agitating and boiling the magma with a separate portion of water and settling each day for several days and then straining to remove the surplus water.

926;289. Process for Desulfurizing and Agglomerating Ores. CVRUS ROBINSON, Mount Vernon, N. Y. June 29, 1909.

The method consists in continuously desulfurizing ores, by continuously subjecting successive layers of moving ore to the action of heat in an oxidizing atmosphere without the use of extraneous fuel.