To apply the method to lead sulphide ores it will be necessary to convert into the carbonate,[1] dissolve in dilute nitric acid and peroxidize as directed above.

To our chief chemist, Mr. William Brady, the writer is indebted for valuable suggestions and for permission to publish this method.

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THE PHOTOMETRIC DETERMINATION OF SULPHUR IN COAL.

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THE use of sodium peroxide as an oxidizing agent for the sulphur of coal has received considerable attention, but the violence of the reaction has brought disfavor upon the method. However, by means of a closed bomb, as in the Parr calorimeter, there has been fully demonstrated the practicability of using sodium peroxide for this purpose. Indeed, over two years ago Mr. Milton Hersey, of Montreal, Canada, in a communication to the author, reported the very satisfactory use of the residues from the calorimetric process for gravimetrically determining the sulphur. Later articles by Sundstrom[2] and by von Konek[3] have advocated the same method.

Coupling the sodium peroxide method of arriving at a combustion with the photometric method proposed by Mr. Hinds,[4] there seem to be possibilities well worth investigating.

The work with the photometer, however, either as outlined by Mr. Hinds, or as elaborated by Mr. Jackson,[5] was not found satisfactory. A careful study was made of the variable elements that entered into the method. The method prescribed a candle of standard power, maintained at a definite distance from the bottom of the graduated tube in which was read the depth of liquid through which the outline of the candle flame could be seen. It was soon found that the strength of the light had little to do with the matter. A stronger light would illuminate the

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[1] Furman's "Manual of Practical Assaying," page 139.

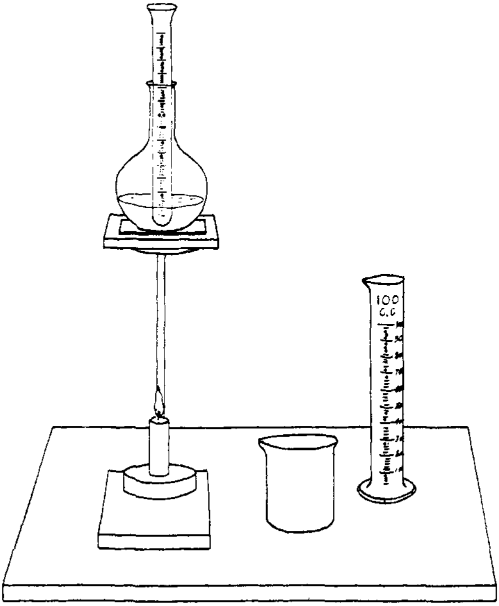
[2] This Journal, **25**, 184.

[3] *Ztschr. angew. Chem.*, 1903, p. 517.

[4] This Journal, **23**, 269.

[5] *Ibid.*, **23**, 799.

liquid to a corresponding degree and cause the outline of the candle flame to disappear at about the same depth as a lesser light with a less illumination of the liquid. As between a common candle and a forty candle-power acetylene light there was no marked difference. Indeed, the greatest difference was noted when the diffused light was cut out by diaphragms, in which case the light could be seen through a greater depth. An extreme illustration of this fact was afforded by completely blackening an incandescent light bulb and then cleaning a small spot to show a short length of the glowing filament. This bit of filament, which afforded no illumination to the liquid, could be seen through a very much greater depth than was the case with an ordinary candle, though its power was far below the standard. Other disturbing conditions related to the method of precipitation, whether hot or cold, whether the barium salt was added in the solid or the liquid state, whether readings were made at once, or on standing, or whether precipitations made in the cold were subsequently heated or not. The control of the conditions regarding the light has been accomplished with a greatly modified apparatus in the following manner, as shown in Fig. 1.



PHOTOMETER FOR SULPHUR DETERMINATIONS.

The tube, graduated in millimeters, is placed in a common flask having a little clear water in the bottom. The flask is placed on a square of glass resting on a carbon plate about ⅝ of an inch thick and having a ¼ inch hole in the center. The glass prevents the condensation of moisture on the bottom of the flask. The carbon plate is adjusted about 10 inches above the flame of a common candle. It will be noticed that the reading tube has a round bottom. This is carefully blown, of clear glass without flaw, and ground on the outer surface, the whole, when immersed, playing the part of a lens. By this arrangement, together with the diaphragm effect of the hole in the carbon plate, a pencil of light is secured with the minimum amount of illumination being imparted to the liquid. Moreover, instead of the varying and indefinite outline of a candle flame there appears a steady compact point of light. The end reading is thereby rendered sharp and definite. It is interesting to note that precipitations made with the barium salt in solution, or with the sulphate solution hot, transmit the rays from the candle as white light, while in the case of precipitations made with the crystals of the salt, the red rays only are transmitted, the illumination of the liquid is in this way still further reduced, and the sharpness of the end reading is thereby promoted. To secure concordant results, definiteness of precipitation must be obtained. This is accomplished by adding the barium salt to 100 cc. of the slightly acid solution at room temperature, and after dissolving completely, heating on the water-bath to about 70°. Allow to stand for half an hour and bring to the room temperature, when it is ready to transfer to the photometric tube for reading. It is not necessary to detail the results of the large number of experiments for testing the variations due to temperature and time of standing before reading. The above conditions give the most constant results.

The conditions are purely empirical, but not arbitrary within reasonable limits, except as to the size of the hole through the plate and the method of precipitation.

It has been necessary to work out a new table. This has been developed directly from a standard solution of potassium sulphate having 0.5438 gram dissolved in a liter, thus containing 0.0001 gram of sulphur by weight per cubic centimeter of solution. Since eyes differ in their sensitiveness to light, a standard solu-

TABLE I.

PHOTOMETRIC TABLE FOR SULPHUR

WEIGHT OF SULPHUR IN GRAMS PER MILLIMETER DEPTH

M.M.	SULPHUR IN GRAMS	M.M.	SULPHUR IN GRAMS	M.M.	SULPHUR IN GRAMS	M.M.	SULPHUR IN GRAMS
200	0.00138	151	0.00161	117	0.00204	83	0.00270
195	0.00140	150	0.00162	116	0.00206	82	0.00273
190	0.00142	149	0.00163	115	0.00208	81	0.00277
185	0.00145	148	0.00164	114	0.00210	80	0.00280
183	0.00147	147	0.00165	113	0.00212	79	0.00284
180	0.00150	146	0.00166	112	0.00214	78	0.00288
179	0.00150	145	0.00167	111	0.00216	77	0.00292
178	0.00151	144	0.00168	110	0.00218	76	0.00296
177	0.00151	143	0.00169	109	0.00220	75	0.00300
176	0.00151	142	0.00171	108	0.00222	74	0.00302
175	0.00151	141	0.00172	107	0.00224	73	0.00304
174	0.00152	140	0.00174	106	0.00226	72	0.00307
173	0.00152	139	0.00175	105	0.00228	71	0.00310
172	0.00152	138	0.00176	104	0.00230	70	0.00313
171	0.00152	137	0.00177	103	0.00231	69	0.00317
170	0.00153	136	0.00179	102	0.00233	68	0.00320
169	0.00153	135	0.00180	101	0.00234	67	0.00323
168	0.00153	134	0.00181	100	0.00236	66	0.00327
167	0.00154	133	0.00182	99	0.00238	65	0.00330
166	0.00154	132	0.00184	98	0.00239	64	0.00335
165	0.00154	131	0.00185	97	0.00240	63	0.00340
164	0.00155	130	0.00187	96	0.00241	62	0.00345
163	0.00155	129	0.00188	95	0.00243	61	0.00350
162	0.00155	128	0.00189	94	0.00244	60	0.00355
161	0.00156	127	0.00190	93	0.00246	59	0.00360
160	0.00156	126	0.00191	92	0.00248	58	0.00367
159	0.00156	125	0.00193	91	0.00250	57	0.00373
158	0.00157	124	0.00195	90	0.00252	56	0.00380
157	0.00157	123	0.00196	89	0.00255	55	0.00386
156	0.00158	122	0.00198	88	0.00257	54	0.00393
155	0.00158	121	0.00199	87	0.00260	53	0.00400
154	0.00159	120	0.00200	86	0.00262	52	0.00407
153	0.00159	119	0.00201	85	0.00265	51	0.00413
152	0.00160	118	0.00203	84	0.00267	50	0.00420

tion should be prepared by which to check the table for the individual using it.

TABLE II.—SHOWING PERCENTAGES OF SULPHUR.

	Illinois coal.	Washings from Mahler bomb. (Per cent.)	Residues from Parr calorimeter in photometer. (Per cent.)
1	Odin, pea	2.30	2.17
2	St. John's lump	1.55	1.65
3	Pana, slack	4.03	4.04
4	Danville, lump	2.16	2.31
5	Ridgely, pea	4.00	4.01
6	Bloomington, lump	2.57	2.68
7	Spring Valley, washed	3.04	3.20
8	Elmwood	1.53	1.61

Results from use of this method, as above outlined, in comparison with those obtained under standard conditions, are shown in Table II.

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CONTRIBUTIONS FROM THE CHEMICAL LABORATORY OF THE UNIVERSITY OF MICHIGAN.]

FURTHER EXPERIMENTS ON THE CLINKERING OF PORTLAND CEMENT AND ON THE TEMPERATURE OF FORMATION OF SOME OF THE CONSTITUENTS.[1]

BY EDWARD DEMILLE CAMPBELL.

THIS work is, in a way, a continuation of the results published by the author in this Journal (**24**, 248, 969; **25**, 1103), and the experiments have been largely suggested by the results of the work previously reported.

In a paper entitled "An Experiment upon the Influence of the Fineness of Grinding upon the Clinkering of Portland Cement" **25**, 1103), the author, with S. Ball, described the progress of linkering in a raw mixture used by a prominent eastern cement mill. This raw mixture was burned in the laboratory rotary cement kiln, employed for our previous experiments. Two experiments, 104 and 105, were made with this same raw maerial. In Experiment 104 the raw mixture was burned without

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[1] The laboratory work in the experiments to be described in this paper has been done during the present year by Messrs. E. E. Ware, D. H. Clary, and M. G. Doll, to whom I ish here to acknowledge my indebtedness for the care with which the work has been one.