	Chestnut-oak bark. No. 1.	Chestnut-oak bark. No. 2.	Hemlock bark. No. 3.	Hemlock bark. No. 4.	Quebracho wood. No. 5.	Oak-bark extract. No. 6.	Leached oak bark. No. 1.	Leached oak bark. No. 2.	Leached hemlock bark. No. 3.
Ash	6.02	8.05	1.65	1.45	0.88	1.35	6.35	6.96	1.55
Carbon	0.77	0.11	0.23	0.59	0.11	1.57	0.15	0.15	....
Sand and silicic acid	2.34	3.33	....	....	0.88	1.88	....	....	7.15
Sand	....	....	1.12	3.53	....	....	1.52	1.46	....
SiO_2	....	...	0.78	2.84	....	....	1.40	0.77	....
$Al_2O_3+Fe_2O_3$	0.08	0.86	3.07	3.86	0.64	5.59	0.71	0.55	2.05
MnO	1.26	0.33	2.26	4.09	0.10	2.21	0.58	0.28	2.41
CaO	52.63	58.24	52.52	49.35	61.27	19.15	57.42	60.50	53.97
MgO	1.18	1.77	1.98	2.92	4.56	2.38	0.56	1.13	1.68
K_2O	2.95	2.62	6.02	7.64	0.24	29.44	1.77	0.84	2.35
Na_2O	0.63	0.40	0.27	0.95	1.22	1.01	0.44	0.38	0.66
Cl	0.44	0.10	0.20	0.24	not det	not det	0.10	0.19	not det
SO_3	0.17	0.12	0.82	1.88	1.11	2.86	0.17	0.12	1.71
P_2O_5	0.64	1.13	2.62	2.94	0.50	3.72	0.51	0.15	1.98
CO_2	36.51	31.47	27.66	19.10	28.30	not det	33.88	33.15	25.50
	99.60	100.49	99.55	99.93	98.93		99.21	99.67	99.46

[CONTRIBUTIONS FROM THE CHEMICAL DIVISION OF THE U. S. DEPARTMENT OF AGRICULTURE, NO. 33.]

ON THE DETERMINATION OF POTASH WITHOUT THE PREVIOUS REMOVAL OF IRON, CALCIUM, ETC.

BY C. C. MOORE.[1]

Received March 18, 1898.

I HAVE found it practicable to wash out ferric, aluminic and other chlorides, and the excess of platinic chloride which is present by means of an acidulated alcoholic wash. Thus it is possible to avoid the precipitation and filtering of ferric hydroxide, calcium oxalate, etc., and the subsequent evaporation and ignition for the removal of ammoniacal salts.

The substance under examination is brought into solution by the usual acid digestion, and the excess of acid removed by evaporation, the residue and insoluble matters separated by filtration. The filtrate is now ready for the potash estimation. To it is added the usual platinic chloride solution, only enough

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[1] Communicated by Dr. H. W. Wiley.

to combine with the potash being necessary. The mixture is evaporated, on the steam-bath, nearly to dryness, so that it solidifies on cooling, just as in the ordinary determination. From fifteen to twenty-five cc. of the acidulated alcohol are added, and allowed to stand two or three minutes with an occasional stirring. The ferric, aluminic, etc., and excess of platinic chlorides readily go into solution, leaving the crystallized potassium chlorplatinic salt and part of the neutral sulphates, sodium chloride, etc. These residues are brought onto a filter, either paper or Gooch, and after one or two washings by decantation, the potassium chlorplatinate and the other residue are washed onto the filter, and washing continued with the same acidulated alcohol until all excess of platinic chloride is washed out. Now wash with the Lindo ammonium chloride solution (200 grams ammonium chloride to one liter of water) until sulphates, sodium chloride, etc., are washed out. Then wash out the ammonium chloride with eighty-five per cent. alcohol, after which a pure potassium chlorplatinate salt is left, which is dried and weighed as usual.

The acidulated alcohol is prepared by passing dry hydrochloric acid gas into cool ninety per cent. alcohol. This is easily done by gently heating strong aqueous hydrochloric acid in a flask. The liberated gas is passed through sulphuric acid, and into the alcohol which is kept in a cooled vessel. Some ethyl chloride will be formed, but most of the gas will be held in solution mechanically by the alcohol. The absorption is continued until one cc. of the alcohol neutralizes about two and one-third cc. of a normal potassium hydroxide solution, using phenolphthalein or any other suitable indicator.

The solvent action of this acidulated alcohol on potassium chlorplatinate is about equal to that of the Lindo ammonium chloride solution, and about one-third that of ordinary eighty per cent. alcohol. Or, if expressed in figures, one gram of pulverized potassium chlorplatinate was digested for two hours in 500 cc. of acidulated alcohol, at a room temperature of 80°, the flask being shaken continuously. The solvent action under these conditions was found to be one part in 60,000.

In experimenting, the ash sample used by the "Association of Official Agricultural Chemists" in 1897 was used. The reporter found this ash to contain about 11.05 per cent. of potash. The sample was digested in hydrochloric acid, evaporated to dryness and taken up with hydrochloric acid and water, so that the portion of the filtrate used corresponded to one-half gram of ash, and contained less than one-half cc. of hydrochloric acid. Four portions were taken from each of two samples, and only sufficient platinic chloride was added to combine with the potassium chloride present. The variation of the maximum and minimum results in each sample was 0.03 per cent., and the average of the eight determinations varied 0.01 per cent. from the result found by the Association.

It was desired to see under what extreme conditions the potash could be determined. An aliquot portion corresponding to one-half gram was used. To this were added 2.0 grams ferric chloride, 0.30 gram aluminic chloride, 0.20 gram sodium chloride, and 0.03 gram calcium sulphate, and only sufficient platinic chloride to combine with the potassium chloride. The result was less than 0.40 per cent. higher than the known percentage, and it is possible that potash was in the chemicals added.

Again, a solution was made of 0.30 gram potassium chloride, 0.56 gram calcium sulphate, 1.00 gram sodium chloride, and 7.0 grams ferric chloride. Only five cc. of a standard solution of platinic chloride were added, which is but a trifle above the requisite amount for the potassium chloride. Duplicate determinations and a blank were run. The evaporation was continued until a solution of the consistency of molasses was formed amounting to about five cc. To wash each sample in a Gooch, 190 cc. of the acidulated alcohol, and 250 cc. of ammonium chloride solution were used, and thirty-five minutes required for complete washing.

While one sample recovered 110 per cent. of the original potassium chloride, the second result was 100.50 per cent., and the blank was not stained. The potassium chlorplatinate crystals were below medium size, very uniform and of splendid color.

It was observed that the presence of other salts was beneficial, as it made the crystallization of the potassium chlorplatinate more perfect, and prevented any subsequent reduction.

Should the original substance contain ammoniacal salts, they must be removed, else ammonium chlorplatinate will be formed.

The decomposition and removal of ammoniacal salts by nitric acid is most applicable here. The process is described in "Crookes' Select Methods," page 32. If hydrochloric acid is present, it is beneficial, as the chlorine which would be liberated also decomposes ammoniacal salts.

THE DETERMINATION OF METHANE, CARBON MONOXIDE, AND HYDROGEN BY EXPLOSION IN TECHNICAL GAS ANALYSIS.

BY W. A. NOYES AND J. W. SHEPHERD.

Received March 7, 1898.

THE determination of carbon monoxide in technical gas analysis with the Orsat apparatus is not very satisfactory, partly because the gas is absorbed so slowly by the cuprous chloride, and partly because the cuprous chloride must be very frequently changed unless two absorption bulbs filled with the reagent are used. These difficulties may be avoided and the carbon monoxide, hydrogen, methane, and nitrogen may be determined by explosion, if the determination is accompanied by a determination of the oxygen consumed in the combustion, by explosion, of a known volume of the gas. For this purpose we have introduced in the Orsat apparatus in place of the fourth bulb an explosion pipette of the form shown in Fig. 1. The pipette has the advantage over that usually supplied with apparatus for gas analysis, that during the explosion the gas is confined by glass stop-cocks on both sides, insuring positively against loss during the moment of high pressure in the apparatus. A little sulphuric acid is added both to the water in the explosion pipette and to that in the measuring burette. This greatly decreases the ionization of the carbonic acid and so lessens the solubility of the carbon dioxide in the water. The results given below demonstrate that the error arising from the solubility of carbon dioxide in dilute acid in the case of mixtures containing six to eight per cent. of the gas must be very small.

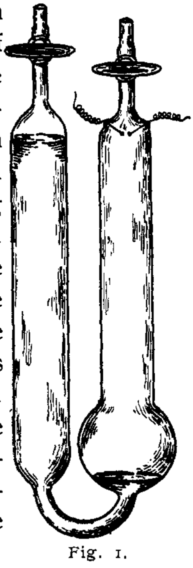

Fig. 1.