

it would be well to confirm its presence by any of the usual tests on a fresh sample of the milk.¹

If the milk has been colored with aniline orange, the colored curd, on applying the strong hydrochloric acid in the test-tube, will *immediately* turn pink. In the case of the caramel, the color of the curd itself remains unchanged, the solution only turning blue and that gradually; on the contrary with the aniline orange the curd itself takes the pink color the moment the acid touches it. If a large amount of the aniline orange has been used in the milk, the curd will sometimes show the pink coloration when hydrochloric acid is applied directly to it, before treatment with ether, but the color reaction with the fat-free curd is very delicate and unmistakable.

SUMMARY OF SCHEME FOR COLOR ANALYSIS.

Curdle 150 cc. milk in casserole with heat and acetic acid. Gather curd in one mass. Pour off whey, or strain if curd is finely divided. Macerate curd with ether in corked flask. Pour off ether.

Ether Extract.

Evaporate off ether, treat residue with NaOH and pour on wetted filter. After the solution has passed through, wash off fat and dry filter, which if colored orange, indicates presence of annatto.

(Confirm by SnCl_2).

Extracted Curd.

(1) *If Colorless.*—Indicates presence of no foreign color other than in ether extract.

(2) *If Orange or Brownish.*—Indicates presence of aniline orange or caramel. Shake curd in test-tube with concentrated hydrochloric acid.

If solution *gradually* turns blue; indicative of caramel.

(Confirm by testing for caramel in whey of original milk.)

If orange curd *immediately* turns pink; indicative of aniline orange.

THE REPEATED USE OF THE DOUBLE CHLORIDE OF COPPER AND POTASSIUM FOR THE SOLUTION OF STEEL OR IRON IN ESTIMATING CARBON.²

By GEO. WM. SARGENT.

Received March 20, 1900.

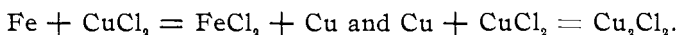
IN the *Chemical News*, Vol. 79, p. 169, which appeared April 14 of last year, there is an article headed: "The Estimation of

¹ See Nineteenth Annual Report of the Mass. State Board of Health (1887), p. 183.

² Read at the March meeting of the Philadelphia Section of the American Chemical Society.

Carbon in Steel; Apparatus and Materials," signed J. T., in the course of which, it is stated, that the copper ammonium chloride may be reused as many as eight times, provided it is oxidized by drawing air through it after each solution, and provided the practice of allowing the sample to dissolve overnight, is adopted. After the operation had been repeated four or five times, there was a decided decrease in the rate of solution and the speed of filtering, therefore it was practicable to use it successively but three or four times, unless the solution of the drillings was made in neutral copper ammonium chloride by shaking in a stoppered flask, when upon removing the carbon and oxidizing, the clear green liquid could be filtered from the iron oxide and reused *ad infinitum*.

Copper potassium chloride, owing to its freedom from organic matter, has supplanted the ammonium salt. Most chemists use the former in a hydrochloric acid solution,¹ when by the aid of a stirring machine, the drillings are dissolved in less than an hour. The reaction is expressed in the following equation:



If now the cuprous chloride be oxidized to cupric, there is no reason why the double chloride solution should not be used repeatedly until the accumulated iron salts become too great. As stated above, this has been done, but the time consumed in oxidizing by a current of air was too long,—several days of an almost continuous current of air through a liter of the acid double chloride failed to completely oxidize the copper salt. The source of my air current was a filter pump, making the waste of water very considerable.

The oxidation was then attempted by the electric current using a cell such as is used for electrolytic oxidations. The result was fairly satisfactory, 3 quarts being oxidized in two days' time; but both these methods yield the cupric chloride at the expense of the hydrochloric acid:

$\text{O} + 2\text{HCl} + \text{Cu}_2\text{Cl}_2 = \text{H}_2\text{O} + 2\text{CuCl}_2$, and $2\text{HCl} + \text{Cu}_2\text{Cl}_2 = \text{H}_2$,
at the cathode and cupric chloride at the anode. Not knowing the quantity of acid consumed in the oxidation, without keeping careful account of the drillings and solution used, it would be

¹ See "The Chemical Analysis of Iron," by Blair.

difficult to tell just how much acid should be added after each oxidation in order to have the double chloride of the proper strength for the next solution. Again the chlorine formed at the anode would attack it. Therefore, I decided to try the effect of chlorinating the solution direct.

The result has been most agreeable. At the present time we have three bottles of 3 quarts' capacity; in the one, the filtered double chloride ready for the solution of the drillings is kept; the second receives the undiluted filtrate from the carbon, while the third, filled with the filtrates, stands in the corner of the hood with a current of chlorine gas passing through it. It requires just a day to chlorinate 3 quarts, and that is about the amount consumed each day. When chlorinated the solution, instead of being of a dirty brown, has almost the original color of copper potassium chloride. After standing over night in the hood and being filtered, the objectionable odor of chlorine is gone and the solution is again ready for use. The chlorinated double chloride is more energetic in its solvent action. In some instances the solution of the drillings has been accomplished in fifteen minutes.

As many as eleven solutions have been made with one quantity of the double chloride, and the time required for the eleventh solution and filtration was very little longer than for the first.

The following results were obtained by the successive use of the same solution :

Number of sample.	Carbon. Per cent.	Number of times the double chloride solution had been previously used.
17	0.864	None
"	0.864	1
1922	0.605	None
"	0.614	2
1930	0.755	None
"	0.752	3
1942	0.410	None
"	0.411	4
1944CX	1.608	None
"	1.614	6
1991	0.385	None
"	0.384	7
1969	0.613	None
"	0.615	11

The saving effected by reusing the copper potassium chloride solution is no inconsiderable amount, especially where much of the salt is used. Roughly estimating, 1 pound of black oxide of manganese, and 1.5 pounds of hydrochloric acid, are sufficient to reconvert 3 quarts of the double chloride, worth about \$1.00.

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[CONTRIBUTIONS FROM THE CHEMICAL LABORATORY OF CASE SCHOOL OF APPLIED SCIENCE, NO. 33.]

ON THE DETERMINATION OF CARBON AND HYDROGEN BY COMBUSTION IN OXYGEN USING COPPER OXIDE.¹

BY CHARLES F. MABERY AND WILLIAM R. CLYMER.

Received March 15, 1900.

IN a former paper² on combustion with copper oxide it was stated that some experiments were in progress to determine the conditions of efficiency, and limits of accuracy when applied to the analysis of oils containing only carbon and hydrogen.

In the long and tedious experience in the analysis of the great number of hydrocarbons that have been under examination in this laboratory, there have been ample opportunities to observe the working details in the hands of a large number of operators who have served at different times as assistants.

The difficulties in the way of securing results with oils containing only carbon and hydrogen, that will calculate closely to 100 per cent., are much greater than with substances containing smaller proportions of these elements, and require great skill and patience on the part of the operator.

Our experience shows that copper oxide is reliable after it has been brought to a constant condition by blank trials. The gases that are evolved in the combustion of some of the hydrocarbons require the highest temperature in presence of oxygen that can be brought to bear, without too great strain on the combustion tube.

The first essential is a drying apparatus that will remove water and carbon dioxide from air and oxygen, and remain in good condition without too frequent renewal. The apparatus now in

¹ The work described in this paper formed the subject of a thesis by Mr. Clymer for the Degree of Bachelor of Science.

² This Journal, 20, 510.