Department of Agriculture, at Madison, Wisconsin, for which acknowledgments are due.

The card of directions for making casein reprinted above may be revised as follows:

REVISED DIRECTIONS FOR MAKING CASEIN

A metal or wooden vat is needed to hold the skim milk which is heated by turning steam directly into it. This vat should have not less than $\frac{3}{4}$ in. steam pipe. Some arrangement for heating the water to be used in washing the casein should be provided. An ordinary wooden hay rake is used to stir the milk while curdling it.

For each 1000 lbs. of milk in the vat, measure out one pint of sulfuric acid. (Muriatic acid may be used with equal success.) Sulfuric acid when used must first be diluted by adding it to double the amount of water. (Always acid to water, never water to acid.)

Heat the skim milk to 95° F.

Add the acid in a small stream or streams to the milk while stirring the latter as vigorously as possible with one or more rakes. Use no more acid than necessary to obtain clear whey.

When fully curdled, let the curd settle for a few minutes, then push it slowly with the rake away from the gate toward the upper end of the vat. Put in the strainer and draw out the whey. Have ready in cans, water at 95° F. equal in weight to one-fourth of the milk used. Add this at once to the curd in the vat and stir up thoroughly with the rake, teeth down, so as to break up all lumps and wash every particle of curd.

Draw off the wash water, and press the curd as thoroughly as possible, if pressed curd is to be made for immediate drying.

If cooked curd is to be made, cover the washed and drained curd with hot water and run in steam to heat it to 180–190° F., until the curd unites into a single large mass which can be shoveled into barrels or boxes for shipment.

SUMMARY

In this paper, it is shown that by modification of the former factory process as to temperature, washing, etc., casein can be uniformly obtained which is suitable for making water-resistant glue.

MADISON, WISCONSIN

SOME NOTES ON PAINT ANALYSIS

By GEORGE J. HOUGH

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The methods described in this paper were devised by the writer to meet particular cases occurring in the course of regular work on the testing of paint supplies; and it is hoped that they may prove useful to those engaged in paint analysis who may have to deal with similar problems. Of course it is understood that with mixed paints, the paint vehicle must be extracted, and the pigment dried, before they can be analyzed.

DETERMINATION OF CUPROUS COPPER IN COPPER PAINTS

This method was devised for ship-bottom paints specified to contain a certain amount of copper in the form of cuprous oxide. The method is as follows:

To 0.5 g. of sample add 10 cc. of concentrated hydrochloric acid, stir well, heat gently for several minutes to dissolve all copper, dilute with cold water to about 200 cc., add 5 cc. of phosphoric acid, and titrate at once by potassium permanganate. The results are good except when the sample contains much unextracted matter, which causes a slight error in the titration.

EXAMPLES—(1) 0.5 g. of sample required 12.8 cc. of permanganate, equal to 28.8 per cent copper. (The same sample titrated one year later gave the same result.) Taking 0.5 g. of the same sample, separating the copper as copper sulfocyanate (CuCNS), and determining it by titration of the sulfocyanic acid gave 28.9 per cent copper.

(2) In another sample the cuprous copper titrated by permanganate was found to be 24.5 per cent. In the same sample, the copper separated by aluminum and determined by the iodide method was found to be 24.8 per cent.

To prove the accuracy of this method for commercial purposes, a sample of dry commercial cuprous oxide was taken, and 0.5 g. samples were dissolved and titrated as above; 0.5 g. of pigment used 27.3 cc. of permanganate with a value of 10 mg. iron per cc. equal to 11.66 mg. copper, showing the copper content to be 63.6 per cent; 2 more duplicate tests gave the same result. It will be noted that the factor 1.166 is used, instead of the theoretical factor 1.125, to change the iron value of the permanganate to the copper value; this factor was found by standardizing the permanganate against a carefully prepared sample of cuprous oxide.

Then samples of 0.2 g. were each treated with a solution of silver sulfate made by dissolving 1.3 g. of silver nitrate in 100 cc. of water and adding 10 cc. of concentrated sulfuric acid; the tests were made with cold solutions, and were allowed to stand for 1 hr. with occasional stirring; they were then filtered and washed, and the metallic silver that had been reduced by reaction with the cuprous copper was dissolved in nitric acid, and the silver titrated by Volhard's method, using a standard solution of ammonium sulfocyanide. In the reaction between cuprous copper and silver sulfate, one part of silver is reduced by one part of cuprous copper. The results of 4 determinations were, 63.85 per cent, 63.55 per cent, 63.85 per cent, 63.55 per cent of copper; this proves that the method is within allowable limits for commercial work. This test with a solution of silver sulfate cannot be made on an extracted pigment, as it is almost impossible to free the sample from oil and organic matter, which interferes seriously with the reaction between the copper and silver.

A RAPID METHOD FOR LIME IN WHITE LEAD

It is sometimes desirable to test white lead for lime, especially in putties, to ascertain how much lime has been added in the form of whiting. The following method depends on the solution of the white lead in a hot dilute solution of caustic soda, and it is removed by filtration from the insoluble whiting:

To 0.5–1.0 g. of sample add 50 cc. of 5 per cent caustic soda and heat to boiling for several minutes, decant on filter, add a little more caustic soda and then hot water, and decant; wash well by decantation with hot water and wash filter about four times; dissolve the residue in hot hydrochloric acid (1:2), dilute, neutralize with ammonia (filter off any lead precipitate), boil, and precipitate lime with a saturated

solution of ammonium oxalate; determine lime by titration with permanganate, or as desired.

When much lead is present (more than 50 per cent of the sample), boil a second time or even a third time with caustic soda. If the residue is well washed, no lead will remain with the lime.

EXAMPLES—(1) 0.5 g. of white lead and 0.5 g. of whiting (CaO present, 54.3 per cent) gave 54.2 per cent CaO.

(2) 0.1 g. of white lead and 0.5 g. of whiting (same amount of CaO as above) gave 54.2 per cent CaO.

(3) 0.8 g. of white lead and 0.2 g. of whiting (CaO present, 10.8 per cent) gave 10.3 per cent CaO.

(4) 0.9 g. of white lead and 0.1 g. of whiting (CaO present, 5.4 per cent) gave 5.6 per cent CaO.

CHROMIUM IN MIXED PIGMENTS

Much trouble is experienced in detecting small amounts of chromium in mixed pigments where chrome yellow has been used to slightly modify an olive or drab shade of color. The following test is quickly and easily made, and gives good results:

Treat the pigment with a few cubic centimeters of dilute nitric acid (1 : 5), stir well, let stand a minute or two, filter, and to the filtrate add a few cubic centimeters of hydrogen peroxide. A purple color indicates chromium, the chromic acid being oxidized to perchromic acid by the hydrogen peroxide. The color is not permanent and soon fades out, especially with small amounts of chromium. This test shows the presence of a very minute quantity of chromium, and it works equally well with chrome green pigments; but for the reaction to proceed successfully, the chromium must be present as chromic acid, and the solution must not be heated.

IMPROVED METHOD FOR CHROMIUM IN CHROME YELLOW

Dissolve 0.5 g. of sample in 15 cc. of 20 per cent caustic soda, warming until dissolved, dilute to about 200 cc. with cold water, add 2 g. of ferrous ammonium sulfate (previously dissolved in a little water), stir well, let stand a few minutes, acidulate strongly with dilute sulfuric acid, dilute cold to nearly 400 cc., and titrate by permanganate.

EXAMPLES—(1) 0.5 g. portions of chrome yellow used; titer of the permanganate was 10 mg. iron per cc.

Found 29.5 per cent, 29.6 per cent, 29.5 per cent, 29.6 per cent CrO_3 .

Same sample titrated by potassium dichromate gave 29.6 per cent, 29.5 per cent CrO_3 .

Same sample by the old standard method (treatment with ferrous ammonium sulfate) gave 28.9 per cent and 29.0 per cent CrO_3 .

EXAMPLE	NEW METHOD Per cent	OLD METHOD Per cent
2.....	26.8	26.3
3.....	19.0	18.8
4.....	28.8	28.4
5.....	17.4	16.3

This method is better than the old standard method using hydrochloric acid to dissolve the chrome, as some chromes are partly insoluble in acid, but readily decomposed by the caustic soda as in Test 5 above.

PREPARATION OF STARCH INDICATOR

Finally, I wish to call attention to a very convenient way of preparing starch indicator, which I devised several years ago. It depends on the well-known fact that starch is soluble in a dilute solution of salicylic acid. Indicator thus prepared keeps indefinitely; a solution put away in a well-corked bottle in a dark cupboard and kept for 2 yrs. was found at the end of that time to be unaltered and as good as when first made up.

METHOD—Dissolve 1 g. of salicylic acid in 100 cc. of distilled water, boil, pour into it 1 g. of potato starch mixed with a little water, boil gently till starch has dissolved, let cool somewhat, and then dilute with cold distilled water to 1 liter. This solution is clear and transparent, and gives a fine, deep blue color with iodine.

BUREAU OF SOILS
U. S. DEPARTMENT OF AGRICULTURE
WASHINGTON, D. C.

A NEEDLE VALVE WITH DELICATE ADJUSTMENT FOR HIGH PRESSURE GASES

By S. W. PARR

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Oxygen on tap is an essential feature of every present-day laboratory but the ordinary valve for opening and closing the steel cylinders is an unwieldy affair. Moreover, a standard connection interchangeable with all

cylinders by a ground metal joint was found to be a necessary feature in the designing of a calorimeter outfit of the oxygen bomb type. By slightly modifying the valve thus designed it has been made available for service under all conditions. The essential feature aside from the metal joint and lock-nut attachment is the possibility of definite control to the extent that by operation of the needle valve alone the gas may be taken directly from the cylinder to a com-