UNITED STATES PATENT OFFICE.

ROBERT G. MEWBORNE AND HENRY K. McCONNELL, OF LOUISVILLE, KENTUCKY, ASSIGNORS TO THE KENTUCKY TOBACCO PRODUCT COMPANY, OF LOUISVILLE, KENTUCKY, A CORPORATION OF NEW JERSEY.

METHOD OF TESTING NICOTIN SOLUTIONS.

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To all whom it may concern:

Be it known that we, ROBERT G. MEWBORNE and HENRY K. McCONNELL, citizens of the United States, residing at Louisville, county of Jefferson, State of Kentucky, have invented certain new and useful Improvements in Methods of Testing Nicotin Solutions, fully described in the following specification.

This invention relates to an improved method for determining the nicotin content of solutions, and especially for determining such content in solutions in which matter other than nicotin may be present and which would combine with or be precipitated by the reagent to be used for the precipitation of nicotin.

The invention has for its especial object the provision of a method for making a quick and accurate test of the nicotin content of any solution containing nicotin, without the delay and use of fine instruments which a laboratory test involves, the novel test being such that it may be successfully performed in the field and the services of a chemist not being required.

Nicotin solutions are now widely used in large baths for dipping cattle, sheep and other animals, for the purpose of freeing them of various forms of vermin, as ticks, lice, etc. The nicotin content of these dipping baths must be of a certain strength to be efficient, and under some conditions the bath is prescribed by the Department of Agriculture of the United States and by State regulations, to be of and maintained at a certain definite nicotin content. To fulfil these requirements, it is necessary to make frequent tests at the place where the baths are being used. Heretofore, in order to ascertain the content of such baths, it has been necessary to take a sample of the bath and send it to a laboratory and there test it by certain methods which require a considerable amount of time in carrying them out, and fine instruments and accurate calculations. There is, as far as we know, no test which could be carried on in the field by persons not trained chemists by which an accurate determination of the content of these nicotin baths could be made, and there was no test for nicotin prior to that invented by us which could be carried out, even by trained chemists, with sufficient rapidity to enable a determination to be made quickly enough for practical use, because of the change which took place in the meanwhile in the nicotin content of the bath. These nicotin baths after use contain, through the washing off therein of excrement, dirt, etc., certain extraneous matters, the presence of which complicates the proper determination of the nicotin content of the bath, no matter how carried out. While we have produced, broadly, a novel method for determining the nicotin content of solutions in which such extraneous matter is not so present, our complete method also provides for the elimination of such matter other than nicotin which would combine with the reagent thereafter used for precipitating or combining with the nicotin.

Broadly speaking, our method for the determination of the nicotin content of a solution consists in taking a known quantity of the solution and acidifying the solution with a mineral acid, preferably hydrochloric acid which we have found to produce the best results, until the solution, when tested by Kongo paper or otherwise, shows the presence of free mineral acid. This assumes that the solution is alkaline. If the solution should already have the desired small excess of mineral acid, of course this step would not be required. To obtain the slight excess of acid desired, the acid is added in small quantities, a test being made after each addition until the reaction indicates that only a small excess of mineral acid is present. By these steps it becomes possible to insure the desired slight acidity of the solution, thereby avoiding, on the one hand, alkalinity, and, on the other hand, such a degree of acidity as would make the process unreliable, these results being obtained even when the process is carried out by ordinary workers not having the skill and training of expert chemists. A suitable nicotin precipitating or combining reagent is then added, this reagent, previously having been standardized of a definite strength such that a known definite amount of the reagent will precipitate or combine with a known definite weight of nicotin, the reagent being added in a quantity sufficient to precipitate or combine with substantially all of the expected

nicotin content in the solution, care being taken, however, that the reagent is not added in such quantities as to be in excess, that is, in such quantities as to precipitate or combine with all of the nicotin in the solution on account of the danger of adding an excess of the reagent and spoiling the test. This solution is now shaken or otherwise agitated, and a small portion filtered off. To this filtered solution is added a known quantity, as, for instance, a drop, of a reagent that will precipitate nicotin. The formation of a precipitate in this filtered portion on the addition of this reagent indicates that there has not been added a sufficent amount of the reagent to precipitate or combine with all the nicotin present. The precipitating or combining reagent is then added to the original solution in small quantities, the filtration of a small portion of the original solution being repeated and a test made of the filtrate by the addition of a drop of the reagent between each addition of the reagent to the original solution. This test is repeated until the filtrate shows no reaction or precipitation by the addition of the drop of the reagent. When this point is reached, all the nicotin in the original solution taken for test will have been precipitated or combined with the reagent, and the volume of the original solution being known, and the amount of the added reagent being known, the nicotin content is readily calculated. These steps constitute a method of adding the standardized solution of a reagent for nicotin in the absence of an indicator, that is, without the use of any indicator. It is known that although some indicators will give a sharp enough change of color when used with solutions of chemically pure nicotin, none so far known are sufficiently satisfactory for use in the titration of solutions made from commercial preparations of nicotin, and are entirely unavailable with dipping baths. Furthermore, all danger of a reaction of the indicator with either the nicotin or the reagent for determining it is avoided by the omission of such an indicator as would injuriously affect the result. It will be noted that the steps of filtering off and testing a portion of the solution, as hereinbefore described, will avoid the necessity for the use of the usual indicators, and yet give the desired determination of the point where the addition of the nicotin-determining reagent is to cease.

The reagents employed for precipitating or combining with the nicotin may be any substance that will precipitate or combine with nicotin quantitatively, so that the nicotin content of the solution can be calculated from the amount of reagent added. We preferably employ as such a precipitating or combining reagent, silico-tungstic acid, some advantages of this reagent being its delicate reaction with nicotin and the insolubility of the salt which it forms with nicotin.

The process thus far described may be and is intended to be used for determining the nicotin content of a solution in which matters other than nicotin are not present which would combine with the reagent employed. This is not usually the case, however, and, as before stated, in testing dipping baths for nicotin there is present much extraneous matter which would combine with the reagent employed for preciptating or combining with the nicotin, and thus complicate and render difficult the proper determination of the nicotin by the test. We have discovered that these complications and difficulties can be overcome by preliminarily adding to the test portion of the bath a reagent which will combine with those matters other than nicotin which would combine with the reagent to be employed if then present. While various reagents may be used for this purpose, we have found that in actual practice the basic acetates of lead will precipitate those substances other than nicotin in the bath which would combine with the silico-tungstic acid. After the addition of such reagent, the test portion of the bath may be filtered and tested for the nicotin content as above described.

While we specify the basic acetates of lead as a suitable reagent for precipitating this extraneous matter, particularly in field baths, we having found the use of such reagent important for the field baths actually tested by us, we do not restrict ourselves to such reagent, but will use a precipitating or combining reagent which will take out of the solution such matter, the reagent used for effecting this varying with the particular character of the solution being tested.

A specific example for carrying out our method for testing a nicotin dipping bath such as is ordinarily used for dipping cattle, sheep, etc., is as follows: A 100 c. c. sample or test portion of the bath liquor is measured into a suitable receptacle, such as a bottle. To this is added 100 c. c. of water; 30 grams of a basic lead acetate is now added, and the whole shaken for a sufficient length of time to permit precipitation of the matter which would be precipitated by the silico-tungstic acid afterward used. This solution is now filtered, and an aliquot portion (usually one-half, which will be about 105 c. c.) of the filtrate is taken and acidified with a mineral acid, preferably hydrochloric acid. This acid is added in small quantities, as, for instance, 2 c. c. at a time. After the introduction of each addition of hydrochloric acid, the solution is tested with Kongo paper and the acid is added until the solution shows just acid with the paper. The nicotin precipitating reagent, preferably silico-tungstic acid of a

standardized definite strength, is now added to the solution in the bottle, the amount of the reagent added being practically the expected nicotin content of the solution, care being taken, however, that the amount of silico-tungstic acid added is not sufficient to precipitate all the nicotin present. The solution is now shaken, and a small portion filtered off. To this filtrate is added a very small quantity of silico-tungstic acid, such as a drop. If a precipitate is formed by the addition of this drop of silico-tungstic acid, more of the silico-tungstic acid is added to the original solution, the amount added being a small quantity, the mixture shaken, and another portion of the original solution is filtered off, and to this filtrate is added a small quantity, as a drop, of the silico-tungstic acid. This process is repeated until the filtrates show no precipitate. This indicates that all the nicotin in the original solution has been precipitated. The volume of the original solution being known, from this and the known amount of silico-tungstic acid added the nicotin content of the solution is readily determined, as above explained.

It will be seen that the herein described method involves determining nicotin by direct titration of a nicotin-containing solution with a standard solution of a reagent, most desirably a precipitant, for nicotin, silicotungstic acid being employed as the precipitant in the specific example given. The proper conditions for accurate work include substantial absence or prior removal of interfering matter, and slight acidity of the nicotin solution. It is usually desirable also to dispense with the use of an indicator, that is, of an indicator other than the reacting solutions themselves.

By our process we have produced a quick and sufficiently accurate test for the nicotin content of solutions, which can be carried out in the field, which can be performed by persons not trained chemists, and which avoids the necessity of laboratory tests and the consequent delay, fine instruments, and careful calculation necessitated by such laboratory tests.

While we have enumerated certain reagents for carrying out our method, as being preferable because found in actual practice to produce the best results so far as now known to us, we do not restrict ourselves to the use of such particular reagents in carrying out the novel steps of the method described, except as defined by the claims.

What we claim is:

1. A method of determining nicotin in solutions containing the same which comprises directly titrating a test portion of such a solution with a standard solution of silicotungstic acid.

2. A method of determining nicotin in solutions containing the same which comprises directly titrating a test portion of such a solution with a standard solution of silicotungstic acid in the presence of a suitable excess of free mineral acid.

3. A field test for determining the nicotin content of nicotin dipping baths which comprises directly titrating a purified test portion of such a bath with a standard solution of silicotungstic acid.

4. A field test for determining the nicotin content of nicotin dipping baths which comprises directly titrating a purified test portion of such a bath with a standard solution of silicotungstic acid, the titration being conducted in acid solution and in the absence of an indicator.

5. A field test for determining the nicotin content of nicotin dipping baths which comprises treating a test portion of such a bath to remove interfering matter, acidifying the test portion thus treated, and titrating the resultant solution directly with a standard solution of a reagent for nicotin.

6. A field test for determining the nicotin content of nicotin dipping baths which comprises acidifying a test portion of such a bath, and then titrating directly with a standard solution of silicotungstic acid.

7. A field test for determining the nicotin content of nicotin dipping baths which comprises treating a test portion of such a bath to remove interfering substances, acidifying the test portion thus treated, and then titrating directly with a standard solution of silicotungstic acid.

8. A field test for determining the nicotin content of nicotin dipping baths which comprises treating a test portion of such a bath with a basic lead acetate to remove interfering matter, and then titrating directly with a standard solution of a precipitant for nicotin.

9. A field test for determining the nicotin content of nicotin dipping baths, which consists in adding to a portion of the bath a basic lead acetate to precipitate those substances other than nicotin which would affect the silicotungstic acid to be used if such substances were present at the time of such use, filtering, acidifying the filtrate by hydrochloric acid added until the solution is slightly acid, precipitating the nicotin by adding to the acid solution silico-tungstic acid in quantity just sufficient to precipitate all the nicotin, and determining the nicotin content of the bath by the amount of silico-tungstic acid used and the strength of the silico-tungstic acid.

10. A field test for determining the nicotin content of a nicotin dipping bath, which consists in adding, to a sample portion of said bath, predetermined equal small amounts of a mineral acid, until the sample first indicates acidity on a suitable indi-

cator, then adding to the sample in successive stages known amounts of silico-tungstic acid, removing a specimen after each stage and testing it to determine whether or not it contains nicotin, ceasing the addition to the sample of silico-tungstic acid when such test of a specimen shows that all nicotin of the sample has been precipitated, and then determining the nicotin content of the bath by the amount and strength of silico-tungstic acid added to the sample.

In testimony whereof, we have hereunto set our hands in the presence of two subscribing witnesses.

ROBERT G. MEWBORNE.
HENRY K. McCONNELL.

Witnesses:
ORMSBY DeB. GRAY, JR.,
WILLIAM M. BERKELEY.