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METHOD OF DETERMINING NICKEL CONTENT OF STEEL

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5 Claims. (Cl. 23—230)

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This application is a continuation-in-part of application Serial No. 601,292, filed by this inventor, Frederick B. Clardy, on June 23, 1945, for Method of and materials for determining nickel content of steel, now abandoned.

This inventor has found that, while the process and material used as defined in his application, Serial No. 601,292 produced a nickel precipitate, the intensity of the red color of which is proportional to the quantity of nickel present in the alloy, the color so liberted is not permanent. It fades and determinations of the percentage of nickel present based upon relatively old stains are likely to be erroneous.

So much of application 601,292 as covers the basic process and the same reaction components as used in this present application are adopted herein.

Therefore, this invention relates to an improved nickel spot test material and its method of preparation and use.

More particularly, this invention is directed to a method of testing for nickel content in steel and other metal alloys which is non-hazardous. It involves the use of only a weak acid solution and especially prepared absorbent test sheets. The test can be performed by an inexperienced operator and is reliable. According to these improvements, any metal which has been dissolved and whose pH is approximately 7, such as from 6–8, will give a positive test for nickel if nickel is present above 0.10% or 0.20%.

Prior to this invention, spot tests for the presence of nickel were used throughout the industry to identify nickel bearing alloys to determine the type of alloy. It is exceedingly important that a convenient test be available as it is estimated, for example, that 75% of the steel alloys in use today contain nickel in varying amounts. The prior spot tests were usually made with the aid of filter paper and from 2 to 4 solutions. The tests required the use of trained personnel to add the solutions in the proper order and amounts as well as to interpret the results.

Prior efforts to overcome some of the problems include using:

(*a*) Mixed acids and buffered dimethylglyoxime solution.

(*b*) Mixed acids, an impregnated paper containing citric acid and dimethylglyoxime and a strong caustic solution.

The latter process proved very unreliable and both methods give a brown iron hydroxide stain, making it difficult for even a trained operator to decide the nickel content if it is under 1%.

I have found that all of the disadvantages and difficulties can be overcome by effecting solution of the alloy metals as by a dilute acid solution and absorbing the reaction products on my specially developed filter paper or other absorbent materials which have been treated with four chemical compounds (1) to decolorize the basic metal salt, (2) to buffer the excess acid, (3) to fix and render permanent the color of the precipitate and (4) dimethylglyoxime to react with the nickel to give its characteristic red action.

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Any salt which will form colorless iron salt as for example, the alkali metal phosphates and fluorides, may be used as a decolorizing material for low alloy ferrous metals (steel) in which iron is, of course, the basic metal. Suitable buffers include sodium acetate, ammonium acetate, potassium acetate. The most efficient color fixing component has been found to be sodium pyrophosphate.

Thus the preparation of the various reagents comprises impregnating the filter paper with sodium pyrophosphate, sodium fluoride, sodium acetate, and dimethylgloxime. Also, the acid solution is prepared by adding sodium phosphate, sodium pyrophosphate to 10% solution of nitric acid. In creasing amounts of phosphate, pyro or ortho, tend to diminish the color of the nickel precipitate. Decreasing the amounts of these two ions increases the brown color due to iron hydroxide.

The treated paper readily gives a positive clear red color in the presence of 0.0% nickel; a faint red color in the presence of less than 0.5% nickel, and an increasing amount of color for percentage above 0.5%.

It is therefore an object of the invention to provide a method of spot testing for nickel content of alloys by the use of a dilute acid solution and test sheets.

Another object is to provide a test filter paper or other absorbent material treated with a decolorizing agent, a buffer, a color fixing agent, and an alcoholic solution of dimethylglyoxime.

Another object is to provide a test filter paper treated with a salt that will form substantially colorless iron salts with low alloy ferrous metal in acid solution, a small quantity of pH control substance and dimethylglyoxime.

These and other objects of invention will be manifest from the description and claims.

According to a preferred embodiment, sheets of filter paper 18 by 22 inches are soaked in a solution containing 10 parts C. P. sodium acetate ($NaC_2H_3O_2.3H_2O$), 3 parts of sodium fluoride, (NaF), 5 parts of C. P. sodium pyrophosphate ($Na_4P_2O_7.10H_2O$) and 80 parts of distilled water. The paper is dried flat and then soaked in a 1% alcoholic solution of dimethylglyoxime. The paper is again dried and cut into strips or circles as desired. The acid solution is made by dissolving 13 grams of sodium phosphate (Na_3) $PO_4.10H_2O$), 1 gram of sodium pyrophosphate ($Na_4P_2O_7.10H_2O$) is distilled water, adding 10 ml. of concentrated nitric acid (e. g. 1.42)

and diluting to 100 ml. with distilled water. In general, the phosphates are preferred because they are inexpensive and non-hazardous.

When low alloy ferrous steels, for example, are to be tested for nickel content, all that is required is to smear a drop (0.05 ml.) of the acid solution over an area the size of a dime of the cleaned and brightened metal and allow it to react for 60 seconds. Press the impregnated paper on the drop and observe the color developed. The nickel content can readily be determined by comparing the red color produced on the paper to standard stains.

When a solution containing dimethylglyoxime dissolved in alcohol is added to a solution containing nickel, if the solution is at a pH of from 6–8, a red color precipitate is formed. According to these improvements, this proper pH is obtained by permitting the acid to react to completion or by buffering the excess acid as above described.

In plant work, samples of steel filings, millings, or turnings are identified by first using nitric acid in the test so that the reaction subsides at about a pH of 7 and forms a slight brownish precipitate of ferric hydroxide. The ferric hydroxide may be dissolved in a weak acetic solution. The test in such cases will be as follows with the results similar to the above.

Approximately ½ gram of the metal is put into a 25 ml. beaker, to which is added ⅒ ml. of 1:1 nitric acid (accurately measured from a 5 ml. burette calibrated to ⅒ ml. division). When the action has subsided, 5 ml. of a 3% acetic acid is added, the solution stirred and the paper is then quickly dipped into the solution, removed and placed on a white surface, pressed free of excess solution with a blotter, and the color of the same compared with a set of standard stains. The total time required for the test is less than 30 seconds. In foundry control work, the nickel content of medium steel is usually under 0.50% which is easily determined as above. When the nickel content exceeds this figure it is redetermined colorimetrically.

The advantages of the process are that no prior training or instruction is necessary. The method is very reliable and will indicate the presence of nickel as low as 0.3%. No strong acids or bases are used and the acid solution is non-hazardous and safe. Only one solution is used which eliminates possible errors from the use of several solutions which could become dislabeled and used in incorrect order.

The invention described herein may be manufactured and used by or for the Government of the United States of America for governmental purposes without the payment of any royalties thereon or therefor.

What I claim is:

1. A dried absorbent material for use in determining the nickel content of metallic alloys in solution, said material impregnated with an aqueous solution of the dibasic salt

$$Na_2HPO_4.12H_2O$$

to form colorless salts of the basic metal of the alloy, an alkali metal acetate buffer to maintain the solution at a substantially neutral pH value, and an alcoholic solution of dimethylglyoxime to precipitate the nickel.

2. A dried absorbent sheet material for use in determining the nickel content of ferrous alloys in acid solution, consisting of a sheet of absorbent material impregnated with an aqueous solution consisting of 10 parts sodium acetate ($NaC_2H_3O_2.3H_2O$), 3 parts of sodium fluoride (NaF), 5 parts of C. P. sodium pyrophosphate ($Na_4P_2O_7.10H_2O$), and 80 parts of distilled water, the said sheet dried after impregnation with this solution and then saturated with a one percent alcoholic solution of dimethylglyoxime.

3. A dried absorbent sheet material for use in determining the nickel content of ferrous alloys in acid solution by absorption of said solution consisting of a sheet of absorbent paper impregnated with an aqueous solution consisting of 10 parts sodium acetate ($NaC_2H_3O_2.3H_2O$) for buffering said solution of alloy to a substantially constant neutral pH value, 3 parts soduim fluoride (NaF) to form a substantially colorless salt with the iron of the alloy, 5 parts C. P. sodium pyrophosphate ($Na_4P_2O_7.10H_2O$) to fix and render permanent the color of the nickel precipitate and 80 parts of distilled water, the said sheet dried after impregnation with said acqueous solution and then saturated with a one percent alcoholic solution of dimethylglyoxime for precipitating the nickel as a red crystalline precipitate.

4. An acid solution for dissolving ferrous alloys consisting of a solution of mixture of 13 grams of sodium phosphate ($Na_3PO_4.10H_2O$), 1 gram of sodium pyrophosphate

$$(Na_4P_2O_7.10H_2O)$$

in water, 10 ml. of concentrated nitric acid (S. G. 1.42), the solution diluted to a volume of 100 ml.

5. In a process of the colorimetric determination of the nickel content of ferrous alloys, the steps consisting of applying a small quantity of a dilute solution of nitric acid, sodium phosphate, and sodium pyrophosphate to the cleaned surface of a piece of the alloy and absorbing the resultant solution with a dried absorbent material impregnated with an aqueous solution of an alkali metal acetate for buffering the solution of the alloy to a substantially neutral pH value, a salt selected from the class consisting of sodium acid phosphate and an alkali metal fluoride for forming colorless salts with the iron of the alloy, an alkali metal pyrophosphate for fixing and rendering permanent the color of the nickel precipitate and an alcoholic solution of dimethylglyoxime for precipitation of the nickel in the alloy, whereby a red colored precipitate of nickelic dimethylglyoxime is formed, the intensity of the color of which is proportional to to the amount of nickel present and is fixed and permanent in color.

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REFERENCES CITED

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