

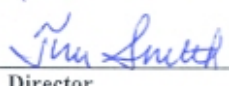


Recommend Approval:  9/27/12 Assistant Division Chief Date  10/5/12 Division Chief Date	Maryland Department of Transportation State Highway Administration Office of Materials Technology MARYLAND STANDARD METHOD OF TESTS	
Approved:  10-18-12 Director Date	COLORIMETRIC DETERMINATION OF SODIUM FERROCYANIDE IN SALT	MSMT 604

SCOPE:

This procedure is based on the conversion of sodium ferrocyanide (Na₄ Fe) (yellow prussiate of soda) (YPS) to ferrous ferrocyanide, FE [Fe (CN) 6] and the determination of its colorimetric concentration.

MATERIALS AND EQUIPMENT:

1. Concentrated sulfuric acid; H₂ SO₄.
2. Sodium ferrocyanide; Na₄ Fe (CN)₆ 10 H₂ O.
 - a. For a 0.1 percent solution – Weigh out 1.5927 g of reagent grade sodium ferrocyanide decahydrate. Then add this to a 1000 ml volumetric flask. Next add distilled water to the flask until the liquid comes to the 1000 ml mark. Each ml of this solution equals approximately to 0.001g of anhydrous YPS which, when added to a 25.0 g salt sample, is equivalent to 40.0 ppm.
 - b. For a 0.005 percent solution – Measure out 5.0 ml of the 0.1 percent solution into a 100 ml volumetric flask. Add distilled water to the flask until the liquid comes to the 100 ml mark. Each ml of this solution equals approximately 0.0005 g YPS which, when added to a 25.0 g salt sample, is equivalent to 2.0 ppm.
3. Ferrous sulfate; Fe SO₄ 7H₂ O.

For a 5.0 percent solution – Add 5.0 g of reagent grade sodium chloride to a 100 ml volumetric flask. Next add approximately 80 ml of distilled water and 2.0 ml of concentrated sulfuric acid. After the acid has been added, fill the flask to the 100 ml mark with distilled water and mix thoroughly.
4. A Colorimeter of suitable design to give satisfactory results over a range of 0 to 10 ppm YPS, when operated at a wavelength of 725 with the 1 in. (25 mm) diameter test cells.
5. Miscellaneous – 100 ml and 1,000 ml volumetric flasks, Whatman No. 5 filter paper or equivalent, graduated cylinders, etc.
6. Blank – Defined as a blank determination in which all steps of the analysis are performed in the absence of a sample.
7. A vial or container of Prussian blue and an eye dropper.

TEST PROCEDURE:

1. Precautions to be aware of when performing this procedure.
 - a. The “blank” and the ferrous sulfate solutions must be prepared fresh daily or as required.
 - b. Matched colorimeter test cells are used and are tested for uniformity with the blank solution daily or as required. Any deviation shown in transmittance values with the blank solution is generally traceable to improperly cleaned cells.
 - c. The test cell used for “unknowns” must be thoroughly swabbed and rinsed between the separate determinations to remove the accumulated film of Prussian blue which would lead to erroneous results.

2. Standard reference curve preparation.
 - a. Weigh six 25.0 g samples of reagent grade sodium chloride and transfer each to a 100 ml volumetric flask. Add precisely 80 ml of distilled water to each and agitate until dissolved. The total brine volume of each flask will be 88 ml. Number the flasks consecutively from 0 to 5 inclusive and add 0.005 percent sodium ferrocyanide (YPS) solution as indicated in Table 604-1.
 - b. Add 5 ml of the 5.0 percent ferrous sulfate solution to each flask and dilute to mark with distilled water. Agitate all flasks thoroughly and allow to age 15 minutes for maximum color development.
 - c. Using Sample No. 0 as a “blank” adjust the colorimeter to 100 percent transmission; then record the transmission values (as percent transmittance) against the known YPS concentrations (ppm) to establish the standard reference curve.

3. Sample preparation.
 - a. Add the sample and 640.0 ml of distilled water to a 1000 ml flask and mix until dissolved. Next filter the brine through a Whatman No. 5 paper or its equivalent. When the size of the bulk sample permits, use a sample splitter to get a 200.0 g sample. Also, any factor that adds turbidity to the test will influence accuracy of the results. Therefore, unknowns are prepared on the basis of some multiple of the ratio of 25 g salt per 80 ml of distilled water.

4. Place 88.0 ml of the filtered brine in a 100 ml volumetric flask, add 5.0 ml of the 5.0 percent ferrous sulfate solution and add some drops of Prussian blue and dilute to the mark with distilled water. Mix this solution thoroughly and let it age for 15 minutes for maximum color to development. Use the colorimeter to determine the percent transmittance.

5. Determine the YPS concentration by referencing the unknown sample to the standard curve.

REPORT:

1. Report YPS concentrations as ppm.

TABLE 604-1

Sample No.	ml 0.005% YPS to Add	Equivalent PPM (Parts YPS/Million Parts Salt)
0	None	None
1	1	2
2	2	4
3	3	6
4	4	8
5	5	10