KIMA CHEMICAL CO.LTD
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METHOD OF ANALYSIS

(HPMC)

DESCRIPTION: -

White to slightly off-white fibrous or granular powder, almost odourless, hygroscopic after drying.

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IDENTIFICATION :-

With constant stirring add a quantity equivalent to1.0 gm of the dried substance into 50 ml of carbon-dioxide-free water previously heated to 90°. Allow to cool, stirring until solution is complete(solution A).

A) Heat 10 ml of above solution in a water bath with stirring. At temperatures above 50° the solution becomes cloudy or a flocculent precipitate is formed. On cooling , the solution becomes clear or slightly opalescent.

B) To 10 ml of solution A add 10 ml of 1M sodium hydroxide or 1M hydrochloric acid. In either case the mixture remains stable.

C) After evaporation of the water from solution of test B a thin film is produced.

D) To 10 ml of solution A add 0.3ml of 2M acetic acid & 2.5 ml of a 10% w/v solution of tannic acid ; a yellowish white, flocculent precipitate is produced.

E) To solution add 8 ml of 70% w/w solution of sulphuric acid. Heat in a water –bath for exactly for exactly 3 minutes & cool immediately in ice. When the mixture is cool, carefully add 0.6ml of a solution containing 3 gm of ninhydrin in 100 ml of a 4.55% w/v solution of sodium metabisulphite, mix well & allow to stand at 25°; a pink color is produced immediately which becomes violet within 100 minutes

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ASSAY :- HP CONTENT & MP CONTENT

Hydriodic acid – Use a reagent having a specific gravity of at least 1.69, equivalent to 55% HI.

Internal Standard Solution :-
Transfer about 2.5 gm of toluene, accurately weighed, to a 100-ml volumetric flask containing 10 ml of o-xylene, to volume & mix.

Standard preparation :-
Into a suitable serum vial weigh about 135 mg of adipic acid & 4.0 ml of Hydroiodic acid, pipet 4ml of Internal standard solution into the vial, and close the vial securely with a suitable seprum stopper. Weigh the vial and contents accurately, add 30 microlitre of isopropyl iodide through septum with a syringe, again weigh, and calculate the weight of isopropyl iodide added, by difference. Add 90 microlitre of methyl iodide similarly, again weigh, and calculate the weight of methyl iodide added, by difference. Shake, and allow the layer to separate.

Assay preparation :-
Transfer about 0.065 gm of dried Hypromellose, accurately weighed, to a 5 ml thick-walled reaction vial equipped with a pressure-tight septum-type closure, add an amount of adipic acid equal to the weight of the test specimen, and pipet 2 ml of Internal standard solution into the vial. Cautiously pipet 2 ml of Hydroiodic acid into the mixture, immediately cap the vial tightly, and weigh accurately. Mix the contents of the vial continuously while heating at 150° for 60 minutes. Allow the vial to cool for about 45 minutes , and again weigh. If the weight loss is graeter than 10 mg, discard the mixture, and prepare another Assay preparation.

Chromatographic system :-
To gas chromatograph is equipped with a thermal conductivity detector and a 4-mm X 1.8-m glass column packed with 20% liquid phase G28 on 100- to 120- mesh support SIC that is not silanized. Helium is used as tha carrier gas and the temperature of the column is maintained at 130°. Chromatograph the standard preparation, and record the peak responses as directed for Procedure: the relative retention times are about 1.0, 2.2, 3.6 and 8.0 for methyl iodide, isopropyl iodide, toluene, and o-xylene, respectively: and the resolution, R, between toluene and isopropyl iodide is not less than 2.0.

Calibration :-
Inject about 2 microlitre of the layer of the standard preparation into the gas chromatograph, and record the chromatogram. Calculate the relative response factor, Fm, of equal weighs of toluene and methyl iodide taken by the formula:

Qm / Rsm

Contd..

In which Qm is the quantity ratio of methyl iodide to toluene in the standard preparation, and Rsm is the peak area ratio of methyl iodide to toluene abtained from the standard preparation. Similarly, calculate the relative response factor, F1, of equal weighs of toluene and isopropyl iodide taken by the formula.

$$Q_1 / Rst$$

In which Q_1 is the quantity ratio of isopropyl iodide to toluene in the Standard preparation, and Rst is the peak area ratio of isopropyl iodide to toluene obtained from the Standard preparation.

Procedure :-

Inject about 2 microlitre of the upper layre of the Assay preparation into the gas chromatograph, and record the chromatogram. Calculate the percentage of methoxy (-OCH_3) in the Hypromellose taken by the formula:

$$2 (31 / 142) FmRum (Wr/Wu)$$

in which 31/142 is the ratio of the formula weighs of methoxy and methyl iodide; Fm is defined under calibration; Rum is the ratio of the area of the methyl iodide peak to that of the toluene peak obtained from the Assay preparation; Wr is the weight , in gm, of toluene in the Internal standard solution; and Wu is the weight, in gm, of Hypromellose taken for the Assay. Similarly, calculate the percentage of hydroxypropoxy (-$OCH_2CHOHCH_3$) in te Hypromellose taken by the formula:

$$2(75 / 170)F_IR_{ul}(Wt / Wu)$$

in whìch 75 / 170 is the ratio of the formula weighs of hydroxypropoxy & isopropyl iodode; F_I is defined under Calibration; $R_{ul}$is the ratio of the area of the isopropyl iodide peak to that of the toluene peak obtained from the Assay preparation; Wr is the weght, in gm, of toluene in the Internal standard solution; and Wu is the weight, in gm, of Hypromellose taken for the Assay.

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INTRINSIC VISCOSITY (Apparent Viscosity) :-

1) Take 250 ml, tared wide mouth Bottle.
2) In a bottle take 98 gm dimineralised water at room temperature.
3) Add 2 gm of HPMC powder under magnetic stirring.
4) Stir with magnetic stirrer, until HPMC dissolve completely.
5) Place the bottle in an ice bath, and allow to remain in the ice bath for 2 hrs to ensure that solubilisation is completed and solution is clear.
6) Adjust the weight of the solution to 100 gm by adding dimineralised water.
7) Adjust the temperature of the solution to 20 +/- 0.1°c.
8) Determine the viscosity in a suitable viscometer of the ubbelohde type (911).

The Viscosity of the measured solution is calculated using the tube constant, k, and the measured efflux time, t, as follows:

2% Solution Viscosity (cP) = k X t (k = 0.02713)

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LOSS ON DRYING :- 14

Mix & accurately weight the substance to be tested , and, unless otherwise directed in the individual monograph, conduct the determination on 1 to 2 gm. If the test specimen is in the form of large crystals, reduce the particle size to about 2 mm by quickly crushing . Tare a glass-stoppered, shallow weighing bottle that has been dried for 30 minutes under the same conditions to be employed in the determination. Put the test specimen in the bottle , replace the cover , and accurately weigh the bottle and the contents. By gentle , practicable to a depth of about 5 mm generally, and not more than 10 mm in the case of bulky materials. Place the loaded bottle in the drying chamber, removing the stopper and leaving it also in the chamber. Drying the test specimen at the temperature and for the time specified in the monograph. Upon opening the chamber , close the bottle promptly, and allow it to come to room temperature in a desiccator before weighing

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pH of solution :-

With constant stirring add a quantity equivalent to 10 gm of the dried HPMC powder into 50 gm of dimineralised water at 80°c, stir for 15 minutes, allow to cool to 20°c in a ice bath, which will take 30 minutes. After the clear solution is produced, check the pH of the solution which should be between 6.5 to 6.8 .

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Heavy metals, Method II : (Limit NMT 10 ppm)

This test is provided to demonstrate that the content of metallic impurities that are colored by sulfide ion, under the specified test conditions, does not exceed the Heavy metals limit specified in the individual monograph in terms of the percentage (by weight) of lead in the test substance, as determined by concomitant visual comparison (see Visual Comparison in the section Procedure under Spectrophotometry and light-Scattering ⟨851⟩ with a control prepared from a Standard Lead Solution. [NOTE- Substances that typically will respond to this test are lead, mercury, bismuth, arsenic, antimony, tin, cadmium, silver, copper and molybdenum.]

Determine the amount of heavy metals by Method I, unless otherwise specified in the individual monograph. Method I is used for substances that yield clear, colorless preparation under the specified test conditions. Method II is used for substances that do not yield clear, colorless preparations under the test conditions specified for Method 1, or for substances that, by virtue of their complex nature, interfere with the precipitation of metals by sulfide ion, or for fixed and volatile oils. Method III, a wet digestion method, is used only in those cases where neither Method I nor Method II can be utilized.

Special Reagents

Lead Nitrate Stock Solution—Dissolve 159.8 mg of lead nitrate in 100 mL of water to which has been added 1 mL of nitric acid, then dilute with water to 1000 mL. Prepare and store this solution in glass containers free from soluble lead salts.

Standard Lead Solution—On the day of use, dilute 10.0 mL of Lead Nitrate Stock Solution with water to 100.0 mL. Each mL of Standard Lead Solution contains the equivalent of 10 μ g of lead. A comparison solution prepared on the basis of 100 μ L of Standard Lead Solution per g of substance being tested contains the equivalent of 1 part of lead per million parts of substance being tested.

pH 3.5 Acetate Buffer— Dissolve 25.0 g of ammonium acetate in 25 mL of water, and add 38.0 mL of 6 N hydrochloric acid. Adjust, if necessary with 6 N ammonium hydroxide or 6 N hydrochloric acid to a pH of 3.5 dilute with water to 100 ml, and mix.

Standard Preparation—Into a 50-mL color-comparison tube pipet 2 mL of Standard Lead Solution (20 μg of Pb), and dilute with water to 25 mL. Adjust with 1 N acetic acid or 6 N ammonium hydroxide to a pH between 3.0 and 4.0 using short-range pH indicator paper as external indicator, dilute with water to 40 mL, and mix.

Contd..

Test Preparation — Use a quantity, in g, of the substance to be tested as calculated by the formula:

2.0/ (1000L),

in which L is the Heavy metals limit, in percentage. Transfer the weighed quantity of the substance to a suitable crucible, add sufficient sulfuric acid to wet the substance, and carefully ignite at a low temperature until thoroughly charred. (The crucible may be loosely covered with a suitable lid during the charring.) Add to the carbonized mass 2 mL of nitric acid and 5 drops of sulfuric acid, and heat cautiously until white fumes no longer are evolved. Ignite, preferably in a muffle furnace at 500° to 600°, until the carbon is completely burned off. Cool, add 4 mL of 6 N hydrochloric acid, cover, digest on a steam bath for 15 minutes, uncover, and slowly evaporate on a steam bath to dryness. Moisten the residue with 1 drop of hydrochloric acid, add 10 mL of hot water, and digest for 2 minutes. Add 6 N ammonium hydroxide dropwise, until the solution is just alkaline to litmus paper, dilute with water to 25 mL, and adjust with 1 N acetic acid to a pH between 3.0 and 4.0, using short-range pH indicator paper as external indicator. Filter if necessary, rinse the crucible and the filter with 10 mL of water, combine, the filtrate and rinsing in a 50-mL color-comparison tube, dilute with water to 40 mL, and mix.

Procedure— To each of the tubes containing the Standard Preparation and the Test Preparation, add 2 mL of pH 3.5 Acetate Buffer, then add 1.2 mL of thioacetamide-glycerin base TS, dilute with water to 50 mL, mix allow to stand for 2 minutes, and view downward over a white surface : The color of the solution from the Test Preparation is not darker that that of the solution from the Standard Preparation.

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Microbial Limit Tests.

Total microbial count

Method of Analysis

Dissolve or suspend 10 g of the substance being examined in 90ml of Fluid Soybean Casein .Digest Medium containing 0.5% soyalecithin & 4% Polysorbate 20. Pipette 1 ml of the final dilution into three sterile petri dishes with the aid of micropipette.

To 2 of the plates add 15 to 20 ml of soybean-casein digest agar medium that has previously been melted and cooled to about 45 °C (Check the temperature with infra red thermometer) cover the petri dishes. Mix the sample with the agar by tilting or rotating the dishes, and allow the agar medium to solidify at room temperature. Invert the petri dishes, and incubate for five days at 34 ± 1°C for TBC.

To one of the plate add 15 to 20 ml of sterile Sabourauds Dextrose Agar at NMT 45°C for TFC and incubate the plates inverted at 22 ± 1°C for 120 hrs After incubation examine the plates in terms of number of microorganisms per g of the substance. If no colonies are recovered from the dishes representing the initial 1:10 dilution of the substance express the result as "less than 10 micro-organisms per g of substance "

Total Microbial count = Number of colonies observed in plate x dilution factor (10)

Release the sample as per the release specification.