GENERAL ELECTRIC COMPANY, SCHENECTADY, N. Y.

Title QUANTITATIVE DETERMINATION OF CHLORIDES IN PYRANOLS

Scope

This method applies to the quantitative determination of chlorides in 1478, 1476, 1482, 1488, 1467 Pyranols and any mixture of these Pyranols.

Specification

The chloride content of any Pyranol listed above shall not exceed 0.10 parts per million by weight.

Standards

Using reagent grade sodium chloride, make up a 1 liter solution containing 100 parts per million by weight of chlorides as follows: weigh .1648 grams of reagent grade Na Cl and quantitatively transfer to a 1 liter volumetric flask. Dissolve in distilled water, dilute to mark and shake well. Place solution in 1 liter glass stoppered Pyrex bottle.

100 c.c. of the 100 ppm solution diluted to 1000 c.c. = 10 ppm
10 c.c. of the 100 ppm solution diluted to 1000 c.c. = 1 ppm
10 c.c. of the 10 ppm solution diluted to 1000 c.c. = 0.1 ppm
15 c.c. of the 10 ppm solution diluted to 1000 c.c. = 0.15 ppm
20 c.c. of the 10 ppm solution diluted to 1000 c.c. = 0.2 ppm
30 c.c. of the 10 ppm solution diluted to 1000 c.c. = 0.3 ppm

All solutions are stored in glass stoppered Pyrex bottles.
All solutions are freshly made every two weeks.
All bottles are shaken well and 10-25 c.c. discarded every time any solution is poured out of the bottle.
The basic standard is the 0.1 ppm solution. When examined in a 20x150 mm Pyrex test tube under a Tyndall beam after adding a few drops of acidified Ag NO_3 solution, the beam intensity within 30 seconds after the addition of the Ag NO_3 solution shall be just visible in a darkroom. If the beam intensity is not visible at all or is easily visible (i.e. too strong), the solutions shall be discarded and new standards made.

Apparatus

(1) Test tubes - Pyrex - 20x150 mm.
(2) Beakers, separatory funnels graduates, stirring rods
(3) A "Penlite" flashlight (2 batteries) having a 3 to 4 mm

(Cont. on H-6845480-1)

6/26/45 EL Raab 8/21/47 EL Raab

Date 5/29/40 **Prepared by** DW Caird

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DSW 330911

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diameter light aperture. New batteries must be used frequently in order to be able to perceive the .1 ppm standard.

Solutions

(1) Chloride free distilled water.
(2) Chloride standards as described above.
(3) A 10% solution of $AgNO_3$ containing approximately 10% by volume of reagent grade concentrated HNO_3.

Apparatus Cleanliness

(1) All glass apparatus such as beakers, graduates, stirring rods, separatory funnels, etc., shall be checked with chloride free distilled water just before using. A small amount of distilled water swirled in any piece of glass apparatus shall show no Tyndall cone when several drops of $AgNO_3$ solution are added to the water in a test tube and examined as previously described.

Extraction of Chlorides from Pyranols

(1) 50 c.c. of Pyranol are heated to 90-100°C, placed in a chemically clean 250 c.c. separatory funnel and extracted with two portions of hot (90-95°C) chloride free distilled water. The total weight of water shall be equal to that of the 50 c.c. of Pyranol tested. The first extraction shall be made by using approximately 2/3 of the water (50 c.c.), shaking violently for 30 seconds, 5 minutes separation and decantation of the bulk of the water layer into a beaker or small Erlenmeyer. The second extraction shall be made using the remaining 1/3 of the water (25 c.c.), shaking violently 30 seconds, 5 minutes separation, discarding the Pyranol layer, and combining the two water extracts in the beaker or Erlenmeyer. Mix both water extracts thoroughly.

Analysis of Water Extract

(1) 10 c.c. of the water extract is placed in a chloride free 20x150 min. test tube and is cleared by either of the alternative methods below.

(Cont. on H-6845480-2)

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(a) Centrifuge until the water shows a negative Tyndall beam effect.

(b) Shake in the test tube with 3-5 c.c. of chloride free ethyl ether (tested previously with water) until the water shows a nagative Tyndall beam effect. More than one ether treatment may be necessary to clear.

(2) Add 3-5 drops of the acidified silver nitrate solution. Do not shake.

(3) Test for chloride turbidity in a dark room using the spot beam as previously described with the beam directed across the diameter of the tube.

(4) The chloride content of the Pyranol is determined in parts per million by weight by comparing the chloride content of the extract with that of a standard chloride solution in the same manner. When comparing to standards, the $AgNO_3$ solution is added to the unknown and standards within several seconds and the Tyndall beam comparison made (~~within 30 seconds after the addition of the Ag NO_3.~~)

(5) The chloride content must not cause a greater Tyndall beam effect than that of a standard sodium chloride solution containing 0.10 parts per million by weight. This amount of chloride as previously mentioned, is the minimum that can be detected by this method as outlined.

15-45 sec after $AgNO_3$ (Memo Jenkins to Loewen 11/14/47)
Raab to Somogyi, Plant B

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