WESTINGHOUSE
ELECTRIC CORPORATION

Material Test Specification 80166

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TENTATIVE SPECIFICATION

TESTING INERTEEN 7336-9

GENERAL:

1. This specification covers the methods of testing for fixed chlorine, electrical resistivity, electrical stability and phenoxypropene oxide content of Inerteen 7336-9.

OPERATIONS:

2. FIXED CHLORINE DETERMINATION:
   (a) METHOD: The method consists of heating a weighed amount of the material to be analyzed in a sealed glass tube with silver nitrate and fuming nitric acid and weighing the resulting silver halide.
   (b) REQUISITES FOR ANALYSIS:
      (1) A pyrex combustion tube 50 to 90 cm long and approximately 2 cm in diameter.
      (2) A funnel approximately 40 cm long for transferring the silver nitrate and nitric acid to the combustion tube.
      (3) A weighing tube 7 to 8 cm long and 6 to 8 mm in diameter.
      (4) Solid silver nitrate and fuming nitric acid free from chlorides.
   (c) METHOD OF ANALYSIS: Put approximately 0.2 gram of the material for analysis into the weighing tube. With the long funnel, transfer 1 gram of finely powdered silver nitrate and 5.0 ml of fuming nitric acid to the combustion tube. Remove the funnel and insert the weighing tube in the combustion tube, taking care to prevent any acid from coming in contact with the sample. Keeping the tube inclined, seal it in the flame of a blast lamp. After cooling (still inclined), transfer the tube to an iron protecting tube and place it in a horizontal position in a furnace designed for work of this type. Allow the end of the glass tube to project 2 to 3 cm from the furnace. Raise the temperature gradually to 280-300 C (536-572 F), consuming 3 to 4 hr to reach this temperature. Hold at this temperature for approximately 20 hr. Allow the furnace to cool to room temperature and, wearing goggles and protecting the hand with a glove, open the safety door of the furnace. Carefully heat the protecting end of the glass combustion tube with a small blast lamp flame until the glass is softened and the internal pressure blows a hole in the glass. Do Not Remove The Tube Until All Internal Pressure Has Been Relieved. Remove the combustion tube from the iron tube and examine. If no oily drops are present and oxidation seems to be complete, cut off the end of the tube with a heated nichrome wire. Thoroughly wash out the contents with water, filter off the silver chloride, and weigh in a Gooch crucible.

   If oxidation is not complete after 24 hr heating, the tube shall be resealed and the heating cycle repeated.

   Calculation:

$$\text{Percent chlorine} = \frac{\text{Weight of silver chloride}}{\text{Weight of sample}} \times \frac{35.46}{143.34} \times 100$$

DSW 550797

3. DETERMINATION OF ELECTRICAL RESISTIVITY:

(a) APPARATUS:

(1) Electrodes, General Electric Co. Dwg H-3962689, with center electrode open
(2) 800 ml pyrex beaker
(3) 0-150 C range calibrated thermometer, 12 in. long
(4) Grooved pyrex plate for spacing electrodes
(5) 500 volt d-c source

(b) CLEANING:

NOTE: Clean the electrodes, beaker, grooved pyrex plate and thermometer before <u>each</u> sample is tested.

(1) ELECTRODES: Wash with benzene or acetone until all visible contamination is removed. Wash in hot water, using compound 2654-9 and a brush. Rinse thoroughly in distilled water. Dry in an oven at 100 C (212 F) for 1 to 2 hr.

(2) GLASSWARE: Wash with acetone until all visible contamination is removed. Wash in hot water, using compound 2654-9 and a brush. Rinse several times in hot water. Fill with or dip into chromic acid. Rinse in cold water and then in distilled water until all traces of acid are removed. Dry in an oven at 100 C (212 F) for 1 to 2 hr with the beaker in a nearly horizontal position, the bottom end being slightly elevated, and the open end resting on a clean filter paper.

(c) PROCEDURE: Fill the beaker containing the electrodes with the Inerteen to be tested immediately upon removal from the drying oven. Raise the temperature of the sample to 100 C (212 F) by heating in an electrically heated box designed to hold an 800 ml beaker. Apply the heat from the sides only. The beaker may be supported with a wire holder so designed that it may also be used as a means of carrying the beaker and electrodes. Stir the sample, while heating, to insure an even distribution of heat. Remove the beaker from the heating element when the temperature reaches 98 or 99 C (208 or 210 F) and place in the testing position upon a piece of cork or asbestos. Continue stirring. The temperature should coast up to about 100 C (212 F) while stirring, but not higher than 101 C (213.8 F) and will then fall through 100 C (212 F) during the test. Adjust the electrodes on the grooved, pyrex plate so the cylinders are concentric. (Preferably adjusted before final temperature has been reached and simply checked immediately before testing.) Connect leads from the resistivity set to cell, connecting the center electrode to the positive side. Close the resistivity set switch and adjust the shunt to give a suitable deflection on the scale. Read the deflection 1 minute after closing the resistivity set switch.

DSW 550798

Calculations:

K_1 CELL CONSTANT: Measure the capacity of the dry cell in air (no liquid) on 1000 cycle bridge.

$$K_1 = 11.3\ C$$

Where:

C = Capacity of the cell in micro-microfarads

K INSTRUMENT CONSTANT: The resistivity test set has a 1 megohm resistor in series with the test circuit. Therefore, if the test leads are shorted and a measurement taken, the deflection for a 1 megohm resistance measurement is obtained and K is calculated from the formula.

$$K = \frac{D}{ES}$$

Where:

D = Deflection obtained with the test leads shorted
E = Voltage of the supply batteries
S = Shunt reading

RESISTIVITY:

$$\text{Resistivity (ohm cm)} = K_1 \frac{ESxK}{Dx} \times 10^6$$

Where:

Dx = Galvanometer deflection with unknown sample
E = Voltage
Sx = Shunt used
K = Constant

In use, it is convenient to combine the equations for resistivity and instrument constant.

$$\text{Resistivity} = K_1 \frac{ESx}{Dx} \frac{D}{ES} = K_1 \frac{DSx}{DxS}$$

Check the value of K at least twice a day and the 500 volt supply once a day. Standard size radio "B" batteries may be used as a cource of supply.

4. TEST FOR ELECTRICAL STABILITY:

PROCEDURE: Determine the resistivity of a sample of the Inerteen at 100 C (212 F). The sample used for the determination of the electrical resistivity in Section 5, for which the resistivity is already known, may be used. Pour the sample into a 1 pt, glass jar with a clamp-on glass cover, both of which have been cleaned according to Section 5(b)(2). Fill the jar to 85% of its volume, leaving an air space of 15% of the volume at the top of the jar. Stretch a piece of aluminum foil over the mouth of the jar and place an untreated paper gasket over the rim of the jar and on top of the edges of the foil so that the foil is held securely in place. Place the glass cover in position and clamp down, taking care not to break the foil membrane. Place the sample in an oven at 100 C $\pm$ 1 (212 F $\pm$ 1.8) for 96 hr. Remove from the oven and determine the electrical resistivity immediately while the temperature of the sample is still at 100 C (212 F). Report any marked change in the color of the sample.

DSW 550799

5. DETERMINATION OF PHENOXYPROPENE OXIDE:

(a) METHOD: The phenoxypropene oxide is reacted with excess pyridinium chloride in the presence of pyridine, and the excess pyridinium chloride titrated with standard sodium hydroxide to a cresol red-thymol blue end point.

(b) APPARATUS:

(1) 500 ml Erlenmeyer flasks
(2) Pyrex Soxhlet extraction apparatus with 125 ml capacity flask
(3) PIPETTE: 20 ml transfer type pipettes
(4) SEPARATORY FUNNELS: 250 ml Pyrex Squibb, pear-shaped funnels

(c) REAGENTS:

(1) INDICATOR: Mix 1 part 0.1% cresol red in water and 3 parts of 0.1% thymol blue in water.
(2) PYRIDINIUM CHLORIDE REAGENT: Dissolve 16 ml of concentrated hydrochloric acid (sp gr 1.16) in 1 liter of CP pyridine.
(3) SODIUM HYDROXIDE 0.1 N: Dissolve 4 grams of CP sodium hydroxide in 1 liter of distilled water. Standardize against potassium acid phthalate.

(d) PROCEDURE:

(1) Weigh into a 500 ml flask to the nearest 0.1 gram approximately 100 grams of Inerteen. Add, by pipette, 20 ml of the pyridinium chloride reagent and attach to a reflux condenser and reflux 30 minutes. Measure reflux time from the start of boiling. At end of reflux period, turn off heat and pour into the top of the condenser 50 ml of distilled water. Detach flask from the condenser and, with a small stream of water from a wash bottle, wash the end of the condenser with a few milliliters of distilled water. Let the washings drain into the flask. Cool the flask and contents to room temperature with ice or tap water. Add 1 ml of the indicator and titrate with 0.1 N sodium hydroxide to a distinct purple color. The flask should be swirled rapidly so that there is thorough mixing of the contents.
(2) Repeat the above steps substituting 70 ml of Inerteen known to contain no phenoxypropene oxide to give blank titration.

(e) CALCULATION:

$$\frac{(\text{ml blank} - \text{ml sample})\ (\text{normality of NaOH})\ (15.0)}{\text{Grams sample}} = \%\ \text{phenoxypropene oxide}$$

(f) USED INERTEEN: The procedure is the same as for new Inerteen through the cooling of the flask after refluxing; then pour the contents of the flask into a separatory flask. Shake thoroughly and permit the Inerteen and water layers to separate. Remove funnel stopper and drain the Inerteen into the original flask. Drain the water into a separate flask. Return the Inerteen to the separatory funnel and wash the reflux flask 3 times with 10 ml portions of distilled water. Pour the washings from the reflux flask into the separatory funnel after each washing. Thoroughly shake the water and Inerteen in the funnel. As soon as they have separated, draw off the Inerteen and add the water portion to the original extraction water. Add 0.5 ml of indicator to the combined water extracts and titrate with 0.1 N NaOH to a distinct purple color. Follow the same procedure using an Inerteen known to contain no phenoxypropene oxide to determine blank. The calculations are the same as for new Inerteen.

APPROVED:

ACCEPTED:

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