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AEROSPACE RECOMMENDED PRACTICE

ARP 1320

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Revised

DETERMINATION OF CHLORINE IN OXYGEN FROM SOLID CHEMICAL OXYGEN GENERATORS

1. PURPOSE

This ARP establishes a method for a rapid and reliable measurement of chlorine in oxygen released during the use of a solid chemical oxygen generator. The method of sampling and analysis used in this procedure is adequate to encompass the range of interest relevant to the limit set for chlorine in oxygen which is 0.2 PPM. The test will be sufficiently flexible to achieve the determination during a part of or for the entire 60 seconds after activation since this is the usual time during which chlorine may be found in the oxygen being generated.

2. BACKGROUND

Methods employing detector tubes are not sufficiently sensitive or mechanically capable of drawing in the required volume of gas during the very short period of time when the chlorine is present. Therefore, a procedure was developed that is not only sensitive for low concentrations but is also capable of taking a representative volume of gas to provide the greatest degree of accuracy and reliability. This includes the selection of reagents that remain stable and a simple colorimetric procedure that can be easily performed by any laboratory with a minimum of equipment.

3. SCOPE

This ARP covers a procedure to be used in the determination of 0.05 to 0.3 PPM of chlorine in oxygen generated from any type of generator used for emergency or other life support systems.

4. REQUIREMENTS FOR LABORATORY EQUIPMENT AND SUPPLIES

- 4.1 Spectrophotometer: The spectrophotometer used in this procedure shall be capable of making measurements in the range of 505 nanometers, such as a Spectronic 20 complete with twelve 1-inch (25.4-mm) glass tubes or equivalent.
- 4.2 Fritted Glass Scrubber: The glass scrubber for absorbing chlorine shall consist of a gas dispersion tube, coarse frit, Corning No. 39533, inserted in a 200-mm (8-inch) test tube with side arm, Corning No. 9840, as shown in Fig. 1. This simple arrangement employs conventional glassware in a manner that has been used for many years in gas scrubbing procedures. Since collection efficiency is a measure of the ability of a scrubbing device to remove the gas from the mainstream, the fritted insert provides greater surface area by breaking the stream into very small bubbles. The gas dispersion tube described above has been used to obtain the standard curve shown in Fig. 2. The curve was prepared from known concentrations of chlorine, and is accurate and realistic since it duplicates actual sampling conditions. It also shows the maximum deviation expected from all factors that went into the preparation of the curve such as weighing and readings of the rotameter and spectrophotometer. While several glass dispersion tubes from the same source have been found to be similar in collection efficiency, the analyst may find it desirable to have several tested during the preparation of his own standard curve with a chlorine permeation tube. Once performed, it is not necessary to repeat the procedure.

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- 4.3 Rotameter: The rotameter shall consist of any rotameter with a glass float capable of measuring a flow rate of 2 liters per minute with an accuracy of $\pm 2\%$ when connected in series with the glass scrubber as shown in Fig. 1. Dwyer Visi-Float, Model 23 BV, (0-5 LPM) or equivalent.
- 4.4 Suction Pump: Small diaphragm type used for drawing the oxygen at a rate of 2 liters per minute through 10 ml of reagent in the scrubber, or any convenient source of vacuum that can be regulated to produce the desired flow rate.
- 4.5 Miscellaneous Supplies: A supply of glassware shall be made available consisting of a 1 liter and a 200 ml volumetric flask, two 10 ml volumetric pipettes, and two 1 ml and 5 ml Mohr pipettes.
- 4.6 Reagents: 10 grams of methyl orange, 1 liter of .1 Normal HCl and a supply of distilled water.

5. CALIBRATION

- 5.1 Flowrate: The system shall be calibrated in advance of any determination by setting up the apparatus as shown in Fig. 1 and adjusting the flow rate as required to obtain the desired flow of 2 liters per minute through the solution in the scrubber.

5.2 Preparation of Reagents:

- 5.2.1 Methyl Orange Stock Solution (.05%): Weigh out 0.10 gram of methyl orange, dissolve it in about 100 ml of distilled water in a 200 ml volumetric flask, warm slightly to hasten solution, cool and dilute to 200 ml.

- 5.2.2 Test Reagent: Transfer 1 ml of the methyl orange solution and 10 ml of the .1N HCl to a 1 liter volumetric flask, then dilute to volume and mix.

5.3 Final Adjustment Procedure:

- 5.3.1 Transfer some distilled water to a tube of the spectrophotometer and set the instrument for 100%T at 505 nanometers.
- 5.3.2 Transfer some of the prepared reagent to a tube, and obtain the reading. The transmittance should be 78%. The % transmission is used instead of optical density because it is easier to make readings in %T on this section of the meter. If the transmittance is not 78%, add increments of 1 or 2 drops of methyl orange stock solution to the flask and mix well or dilute with .001 N HCl as required until the test reagent shows 78% transmittance. Once prepared, the reagent is stable for 6 months in a clear pyrex bottle but should be checked before use.

6. TYPICAL TESTING PROCEDURE

- 6.1 Set up the apparatus as shown in Fig. 1, making sure that all connections are leak proof.
- 6.2 Transfer 10 ml ($\pm .05$ ml) of the methyl orange test reagent to the scrubber, and adjust the flow rate to 2 LPM. Make an experimental run with laboratory air to make sure it is free from interferences.
- 6.3 Activate the generator and start the scrubbing at the time specified in the procurement specification. Scrub for one minute.
- 6.4 Disconnect the tubing, wait 5 minutes, then transfer the scrubbed solution to a 1 inch tube.
- 6.5 Adjust the spectrophotometer to 100% T with water as described, then place the tube of scrubbed solution in the instrument to obtain a reading.

- 6.6 Refer to the standard curve for the concentration of chlorine in the 2 liters of oxygen sampled.
- 6.7 The above method is designed for obtaining the average concentration of chlorine over a 1 minute period of time, but changes may be made in flow rate, scrubbing solution volume, or scrubbing time to adapt the procedure as needed. For example, if it is necessary to obtain data on peak concentrations, a manifolded sampling system can be employed. Such a system might consist of six scrubbers with a suitable arrangement for 10 second scrubbing periods for each. Any changes to the flow rate or scrubbing solution volume will affect the collection efficiency, thereby requiring the preparation of a different calibration curve.

PREPARED BY

SAE COMMITTEE A-10, AIRCRAFT OXYGEN EQUIPMENT

ASSEMBLY OF APPARATUS FOR DETERMINING
CHLORINE IN OXYGEN FROM CHLORATE CANDLE

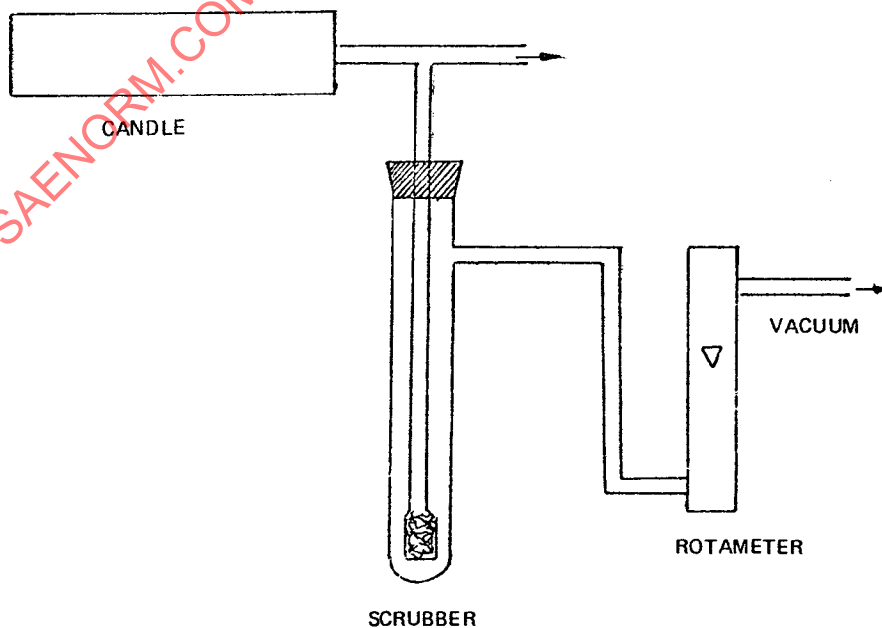


FIGURE 1