SAE Aerospace
An SAE International Group

AEROSPACE RECOMMENDED PRACTICE

SAE ARP1320 REV. A

Issued 1976-01
Revised 1991-01
Reaffirmed 2011-06

Superseding ARP1320

Determination of Chlorine in Oxygen from Solid Chemical Oxygen Generators

FOREWORD

Two factors are unique to the determination of chlorine in oxygen from chlorate candles. First, chlorine is checked during the initiation phase lasting a minute or less. In some systems this means small gas samples and hence small amounts of chlorine. Detector tubes are not sufficiently sensitive or mechanically capable of drawing in the required volume of gas. Second, the oxygen atmosphere is different from air in terms of using chlorine-in-air determination.

A number of methods are available for the determination of chlorine and/or chlorine dioxide in air. All involve a fritted element to disperse the sample stream into fine bubbles for better reaction with the absorbing medium. Determination of the resulting chloride ion in solution can then be done in various ways. There are not ASTM, EPA, or ACGIH methods that deal with chlorine in pure oxygen.

Since sampling and analysis had been done with air as the carrier gas, and recognizing that pure oxygen may affect the analysis, work was done at Wright Patterson Air Force Base on a colorimetric procedure for chlorine in oxygen (see Reference 2.1). This included studies of trapping the chlorine and a comparison of the methyl orange (see Reference 2.2) and O-tolidine (see Reference 2.3) analytical methods. The methyl orange method was found to be most applicable and not subject to interference by the pure oxygen stream. Accordingly, methyl orange is given as the method of choice, with the possibility of using a method in which the sorbed chlorine reacts with KI in solution to release iodine (see Reference 2.4). The latter can be determined by continuous electrolytic titration, making it a more nearly real time method.

The method should be adaptable to low levels of chlorine. Again, the methyl orange method was found to be sufficiently sensitive.

A major point in any of the methods is determination of efficiency of the fritted filter. This should be checked against a known chlorine concentration in a challenge gas stream.

TO PLACE A DOCUMENT ORDER: Tel: 877-606-7323 (inside USA and Canada)
Tel: 724-776-4970 (outside USA)
Fax: 724-776-0790
Email: CustomerService@sae.org
SAE WEB ADDRESS: http://www.sae.org

1. SCOPE:

This ARP covers a procedure to be used in the determination of 0.05 to 0.3 ppm of chlorine in oxygen from any type of generator used for emergency or other life-support systems. The methyl orange method described can be considered as a referee technique. Instrumental analysis is also given in Section 8.

1.1 Purpose:

This ARP describes a method for rapid and reliable measurement of chlorine in oxygen from 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 0.3 ppm limit set for chlorine in oxygen. The test is sufficiently flexible to make the determination during a part of or for the first 60 s after activation, since this is the usual time during which chlorine may be found in the oxygen being generated.

2. REFERENCES:

2.1 Gisclard, J. B. and Hinman, P. V., A Colorimetric Procedure for the Determination of Chlorine in Oxygen, presented at SAE A-10 Committee Meeting, May 11-12, 1972.

2.2 Tentative Method of Analysis for Free Chlorine Content of the Atmosphere (Methyl Orange Method), Subcommittee 2, ASTM, Health Laboratory Science, 8(1) 53 (1971).

2.3 Porter, L. E., Free Chlorine in Air, a Colorimetric Method for Its Estimation, Ind. Eng. Chem. 18, 731 (1926).

2.4 Saltzman, B. E., Preparation and Analysis of Calibrated Low Concentrations of Sixteen Toxic Gases, Anal. Chem., 33, 1100 (1961).

3. PROCEDURE SYNOPSIS:

A bubbler with methyl orange indicator in dilute HCl solution is set up to draw off 2 liters per minute of oxygen from the oxygen generator delivery tube. After sampling for 1 min, the bubbler solution is transferred to a spectrometer to determine the change in transmittancy caused by chlorine reaction. Chlorine concentration is determined from a curve prepared from calibrations under the same sampling conditions (flow and time) using a chlorine permeation tube.

4. EQUIPMENT AND SUPPLIES:

4.1 Spectrophotometer:

The spectrophotometer (Spectronic 20 or equivalent, complete with 12 1-in [25-4-mm] glass tubes) used in this procedure shall be capable of making measurements in the range of 505 nm.

4.2 Fritted Glass Scrubber:

The glass scrubber for absorbing chlorine shall consist of a gas dispersion tube, coarse frit, inserted in a 200-mm (8-in) test tube with side arm, as shown in Figure 1. This arrangement employs conventional glassware in a manner that has been used for many years in gas scrubbing procedures. The fritted insert provides greater surface area by breaking the stream into very small bubbles.

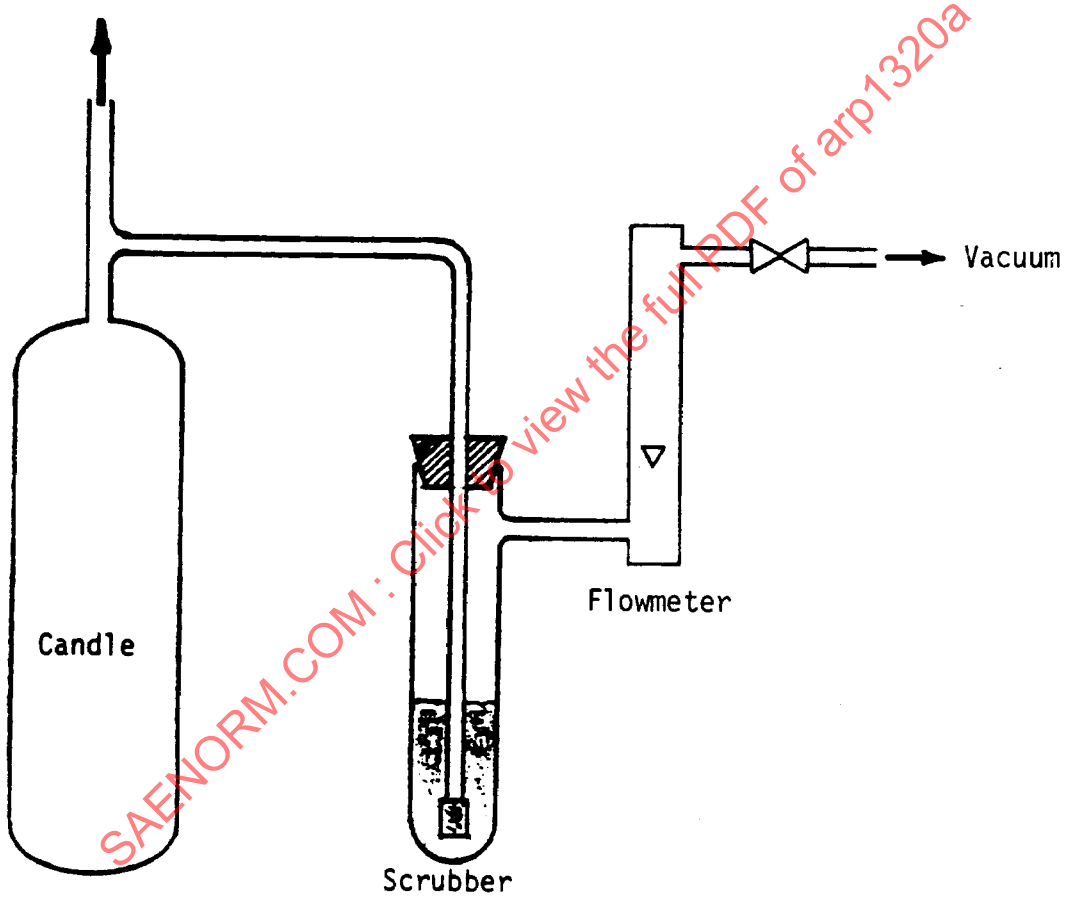

FIGURE 1 - Sample Train for Determination of Chlorine in Chlorate Candle Oxygen

4.3 Flowmeter:

The flowmeter shall be capable of measuring 2 L/min with an accuracy of ±2% when connected in series with the glass scrubber as shown in Figure 1.

4.4 Suction Pump:

A small diaphragm pump is used for drawing the oxygen at 2 L/min through 10 ml of reagent in the scrubber. Any source of vacuum that can be regulated to produce the desired flow rate may be used.

4.5 Miscellaneous Supplies:

Additional equipment includes 1 liter and 200 ml volumetric flasks, two 10 ml volumetric pipettes, and two each 1 ml and 5 ml Mohr pipettes.

5. REAGENTS:

5.1 Reagents Required:

Ten grams of methyl orange, 1 liter of 0.1 N HCl and a supply of distilled water.

5.2 Methyl Orange Stock Solution (0.05%):

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.

5.3 Test Reagent (Bubbler Solution):

Transfer 1 ml of the methyl orange solution and 10 ml of the 0.1 N HCl to a 1 liter volumetric flask, then dilute to volume and mix.

5.4 Adjusting the Test Reagent:

5.4.1 Set the instrument for 100%T at 505 nm with a spectrophotometer tube filled with distilled water.

5.4.2 Transfer some of the prepared reagent to a tube, and obtain the reading. The transmittance should be 78%. The percent 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 greater than 78%, add increments of 1 or 2 drops of methyl orange stock solution to the flask and mix well or if less than 78% dilute with 0.001 N HCl as required until the test reagent shows 78% transmittance. Once prepared, the reagent is stable for six months in a clear Pyrex bottle but should be checked before use.

6. CALIBRATION:

6.1 Flowmeter:

The flowmeter may be checked by calibrating (under suction as for the gas sampling procedure) against a wet test meter or bubbler tube.

6.2 Fritted Gas Scrubber:

Fritted glass dispersion tubes vary, even within the same catalog number. The calibration curve of transmittancy versus parts per million chlorine (Figure 2) includes accumulative deviations for weighing and instrument readings, but applies only to a single frit. Collection efficiency of particular frits may be checked by using a chlorine permeation tube as a chlorine source, using diluting gas to reach the 0.05 to 0.3 ppm concentrations and operating the sampling system. A percent transmittance versus parts per million chlorine plot is then developed for each bubbler.

7. TESTING PROCEDURE:

7.1 Set up the apparatus as shown in Figure 1, making sure that all connections are leak free.

7.2 Transfer 10 ml (±0.05 ml) of the methyl orange test reagent to the scrubber, and adjust the flow rate to 2 SLPM.

7.3 Activate the generator and start the scrubbing at the time specified in the procurement specification. Scrub for 1 min.

7.4 Disconnect the tubing, wait 5 min, then fill a 1 in tube with the scrubber solution.

7.5 Adjust the spectrophotometer to 100%T with water as described in 5.4.1, then place the tube of scrubber solution in the instrument and obtain a %T reading.

7.6 Refer to the standard curve for the concentration of chlorine in the 2 liters of oxygen sampled. The parts per million concentration of chlorine in the oxygen stream is read directly from the transmittance versus parts per million chlorine curve prepared as a standard.

a. The calibration curve applies to a specific dispersion tube and assembly.

b. The flow for sample and chlorine-in-oxygen challenge mix used for calibration, must match. A 2 L/min flow is used here; appropriate pressure and temperature corrections must be made to produce a match.

7.7 The above method is designed for obtaining the average concentration over a 1 min sampling period, 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-s scrubbing periods for each. Any changes to the flow rate or scrubbing solution volume will affect the collection efficiency, requiring the preparation of a different calibration curve.