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Superseding ARP1320A

**Determination of Chlorine in Oxygen from Solid Chemical Oxygen Generators**

**RATIONALE**

The A-10 Committee concluded that this analytical method will not be updated in the future. There is no plan to develop a replacement document at this time. Therefore, the Committee tasked the sponsor to ballot to stabilize this document at the current revision level.

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## 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.

## 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.