

criteria for a recommended standard....

**OCCUPATIONAL EXPOSURE
TO
ACETYLENE**



U.S. DEPARTMENT OF HEALTH, EDUCATION, AND WELFARE

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IX. APPENDIX I

SAMPLING-MONITORING METHOD USING A COMBUSTIBLE GAS METER AND SAMPLING METHOD FOR USE WITH GAS CHROMATOGRAPHIC ANALYSIS

The method presented in Appendix I, Section A, is a modification of that published by the Mine Safety Appliances Company. [67] The sampling and analytical methods presented in Appendix I, Section B, and in Appendix II are based on those described by the Intersociety Committee in Methods of Air Sampling and Analysis. [68]

Section A - Combustible Gas Meter Method

(a) Atmospheric Sampling

A combustible gas meter shall be used to determine acetylene concentrations in areas where exposure to acetylene is suspected. Only instruments designed for use with acetylene shall be used because other combustible gas meters may contain copper or silver wires or filaments which can form dangerous acetylides on contact with acetylene.

(b) Sampling Procedure

Follow the instructions given in the manual for each combustible gas meter. Typically, the sampling procedure will require the following steps:

(1) Sweep the combustion chamber free of combustible gases and fill it with fresh air.

(2) Turn on the batteries and apply the proper voltage to the bridge.

(3) Balance the bridge to zero deflection on the meter while the fresh air is in the open chamber.

(4) Draw the air sample into the meter and record the meter reading. Repeat this at least three times; calculate and record the average of the readings.

(5) Determine the concentration of acetylene in the air samples from the calibration curve provided with each properly calibrated meter.

(6) Record a description of sampling location and conditions, equipment used, time, and any other pertinent information.

(7) If the concentration of acetylene as determined by this procedure is 1,500 ppm or greater, take an atmospheric sample for gas chromatography as described in Section B of this Appendix.

Section B - Sampling Method for Use with Gas Chromatographic Analysis

Samples shall be collected in areas where acetylene is in use and where acetylene concentrations might be expected to exceed 1,500 ppm. A description of sampling location and conditions, equipment used, time and rate of sampling, and any other pertinent information shall be recorded.

(a) Equipment

(1) Rigid-walled gas sample container made of glass and fitted with stopcocks.

(2) One atomizer rubber bulb set or automatic buret bulb.

(b) Sampling Procedure

(1) Flush the container out three times using the rubber

buret bulb attached to the tube stopcock.

(2) Collect the sample, close the stopcocks tightly, and remove the atomizer bulb.

(3) Give the container an identifying number and record appropriate field information.

(4) Send the samples to the laboratory for analysis as soon as possible.

X. APPENDIX II
ANALYTICAL METHOD FOR ACETYLENE

Principle of the Method

An aliquot of the atmospheric sample contained in the sampling container is injected into a gas chromatograph. The area of the resulting peak for acetylene is determined and compared with areas obtained from injection of a pure acetylene standard.

Range and Sensitivity

The lower limit of detection of this analytical procedure is 0.01 ppm/sample by volume.

Interferences

Any compound which has about the same retention time as acetylene under the gas chromatographic conditions described in this method, eg, methane, will interfere with the analysis. This type of interference can be alleviated by changing the operating conditions of the instrument, usually the column or the column temperature.

Precision and Accuracy

Replicate analyses of aliquots of uniform air samples and standards should not differ by more than 10% of the standard deviation.

Advantages of the Method

The method is rapid and especially applicable to routine collection and analysis of grab samples. Elution of acetylene from the gas chromatograph is effected in 16 minutes or less.

Apparatus

(a) Gas chromatograph equipped with a flame ionization detector and suitable sampling valve.

(b) Column (2.4 m x 3 mm) packed with activated alumina coated with 17% (by weight) B,B'-oxydipropionitrile. Other columns which achieve the desired separation may be used.

(c) Mechanical or electronic integrator, or a recorder and some method for determining peak area.

(d) Syringes: 1-ml and other convenient sizes for preparation of standards.

Reagents

(a) Acetylene with a guaranteed minimum purity of 99 mole%.

(b) Bureau of Mines Grade A helium.

(c) Hydrogen: 98.9% purity.

(d) Pure grade nitrogen.

(e) Pure grade air.

Analysis of Samples

(a) Procedure: Air samples collected in the field and returned to

the laboratory are analyzed according to the following procedure:

- (1) Turn on recorder.
- (2) Set the electrometer attenuation.
- (3) Connect the sample tube to the inlet of the sampling valve on the gas chromatograph and flush 20 ml of the sample through the loop.
- (4) Inject a 1-ml aliquot of the sample.
- (5) Elute the sample from the column.

(b) Make duplicate injections of each sample and of each standard dilution used in obtaining the standard curve. No more than a 10% difference should result in the peak areas recorded for each sample.

(c) Measure the areas of the sample peaks with an electronic integrator or by some other suitable method for area measurement.

(d) Calculate the concentration of acetylene present in the sample directly from the standard curve. No corrections are necessary for the injected volume since it is the same for both the sample determinations and the standard curve.