

# Certificate of Analysis

## STANDARD REFERENCE MATERIAL 1612

### Certified Gas Standard

### Hydrocarbon in Air

J. M. Ives and E. E. Hughes

Hydrocarbon concentration  $0.00117 \pm 0.00001$  mole percent (calculated as methane)

The concentration of hydrocarbon is relative to the concentration of all other constituents of the gas. The limit of uncertainty shown is a composite estimate of the error based on uncertainties in the preparation and the final method of analysis. The reported value represents the total hydrocarbon content of the sample expressed as the effective methane concentration.

The hydrocarbon concentration obtained by the analytical method described on the reverse page for one determination of each of 20 samples in the lot is  $0.001173 \pm 0.000001$  mole percent, where the limit is the 95 percent confidence interval of the mean. The concentration calculated from the mixing data is  $0.00118 \pm 0.00001$  mole percent. This limit includes possible errors in measurement of the pressures involved and uncertainties in the hydrocarbon content of the diluent air. The value certified is that obtained from the analytical data and is chosen over the calculated concentration because of the inherently higher degree of accuracy involved in the analytical measurement. The limit on the certified value takes into account uncertainties in the composition of the methane, uncertainties in the composition of the primary laboratory standards, and the precision of the analytical measurement.

Preparation and chemical analyses leading to certification of this Standard Reference Material were performed by J. M. Ives and E. E. Hughes.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of J. K. Taylor.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. W. Mears.

Washington, D. C. 20234  
October 31, 1969

J. Paul Cali, Acting Chief  
Office of Standard Reference Materials

(over)

**PREPARATION AND ANALYSIS:** Standard Reference Material 1612 was prepared in bulk by stepwise dilution of hydrocarbon (99.65 mole percent methane) with air of measured low hydrocarbon content. The pressure of each constituent was determined at each step in the dilution and the final concentration of hydrocarbon was estimated from these data. The final value of the hydrocarbon concentration as well as the concentration of hydrocarbon in each intermediate mixture and in the diluent gases was also determined analytically by a flame ionization method. The validity of the results obtained by this method was assured by comparison with a set of primary laboratory standards prepared by weighing the individual components on a precision analytical balance. Two reference points were used in this technique. An upper point was fixed by a primary standard of  $0.0009301 \pm 0.0000038$  mole percent while a lower point was established with a secondary standard of  $0.000212 \pm 0.000002$  mole percent hydrocarbon used as a convenient "zero" because of the difficulty of obtaining hydrocarbon free air. The composition of this latter standard was verified by comparison with a primary standard and with air containing less than  $2 \times 10^{-6}$  mole percent hydrocarbon, prepared by passage over copper oxide at 1050 °C.

The hydrocarbon from which this Standard Reference Material and the primary standards were prepared had the following composition.

CH <sub>4</sub>	-	99.65 mole percent
C <sub>2</sub> H <sub>6</sub>	-	0.19 mole percent
C <sub>3</sub> H <sub>8</sub>	-	.11 mole percent
C <sub>4</sub> H <sub>10</sub>	-	.05 mole percent

The diluent air contained less than  $2 \times 10^{-5}$  mole percent hydrocarbon, assumed to be methane.

Conversion of the hydrocarbon content of the primary standards to a concentration as methane is accomplished by multiplying the molar concentration of each constituent of the hydrocarbon fraction by the appropriate number of carbon atoms.

**STABILITY:** No change in hydrocarbon content was observed after transfer from the bulk to the sample containers, nor after storage at room temperature for a period of nine months.

The certified hydrocarbon concentration is believed to be reliable for the gas as contained in an unopened cylinder for a period of at least five years. Periodic analyses of samples from the lot will be made and users will be notified if any significant deviation from the above value occurs.

**USE AND STORAGE:** The sample is contained in a disposable cylinder and will not be refilled.

Each cylinder has been leak-tested at the joint between valve and cylinder. Valves are hand-tightened and were determined to be leak-free before filling. The valves are equipped to accept CGA-580 fittings. To avoid contamination of the contents all connections should be evacuated, or flushed with the sample, before use.

Cylinders should be stored at room temperature. Cylinder valves are equipped with rupture-disk relief valves set to rupture at 1200 lb/in<sup>2</sup> ( $8.3 \times 10^6$  N/m<sup>2</sup>).

Each cylinder is filled to about 500 lb/in<sup>2</sup> ( $3.4 \times 10^6$  N/m<sup>2</sup>). The total volume of gas is approximately 68 liters measured under standard conditions.

DOT special permit number 5075.