Designation: D4051 – 10

StandardPractice for
Preparation of Low-Pressure Gas Blends[1]

This standard is issued under the fixed designation D4051; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This practice covers a laboratory procedure for the preparation of low-pressure multicomponent gas blends. The technique is applicable to the blending of components at percent levels and can be extended to lower concentrations by performing dilutions of a previously prepared base blend. The maximum blend pressure obtainable is dependent upon the range of the manometer used, but ordinarily is about 101 kPa (760 mm Hg). Components must not be condensable at the maximum blend pressure.

1.2 The possible presence of small leaks in the manifold blending system will preclude applicability of the method to blends containing part per million concentrations of oxygen or nitrogen.

1.3 This practice is restricted to those compounds that do not react with each other, the manifold, or the blend cylinder.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Summary of Practice

2.1 Through the use of a blending manifold, the blend components are combined based upon partial pressure. Components are added in order of ascending vapor pressure; that is, components of lowest vapor pressure are added first, with the exception that components at concentrations of 5 % or less would usually be added first. Compressibility factors are applied to the component partial pressures to convert them from ideal to real gas. The real partial pressures, which are proportional to gas volumes, are normalized to give mol percent composition of the blend.

3. Significance and Use

3.1 The laboratory preparation of gas blends of known composition is required to provide primary standards for the calibration of chromatographic and other types of analytical instrumentation.

4. Apparatus

4.1 *Blending Manifold*—Construct manifold as shown in Fig. 1. Details of construction are not critical; that is, glass, pipe, or tubing with welded or compression fittings may be used. The manifold must be leak free and arranged for convenience of operation. More than one feedstock connection point may be included if desired. Connections to the pump and manometer shall follow accepted vacuum practice. Valves shall have large enough apertures to permit adequate pumping in a reasonable length of time.

4.1.1 The finished manifold shall have a leak rate no greater than 1 mm Hg/h (0.133 kPa/h).

4.2 *Gauge,* a well-type manometer such as the Meriam Model 30EB25 (see Note 1).

NOTE 1—A high-vacuum gauge of the McLeod Manostat type pressure transducer or a 0 to 2 bar (absolute) gauge may be included in the manifold system to determine how well the system has been evacuated.

4.2.1 Alternatively, an electronic pressure gauge may be used in place of a manometer.

4.3 *Pump,* high-vacuum, two-stage, capable of pumping down to a pressure of 1.33×10^{-4} kPa (0.1 µm).

5. Reagents and Materials

5.1 *Blend Components,* high-purity, as required depending on the composition of the proposed blend.

5.2 *Nitrogen,* high purity, as required, for purging and for balance gas, where applicable.

6. Procedure

6.1 Connect the blend cylinder to the manifold at position *A* (see Fig. 1 for valve and position designations). Open valves *1*, *2*, *3*, and *6* and evacuate the manifold system thoroughly. Valves *4* and *5* are closed.

NOTE 2—A McLeod Manostat type gauge may be used at various times during the procedure to determine how well the system has been evacuated and to indicate if there are leaks present. Otherwise, a steady

[1] This practice is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.04.0A on Preparation of Standard Hydrocarbon Blends.

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*A Summary of Changes section appears at the end of this standard

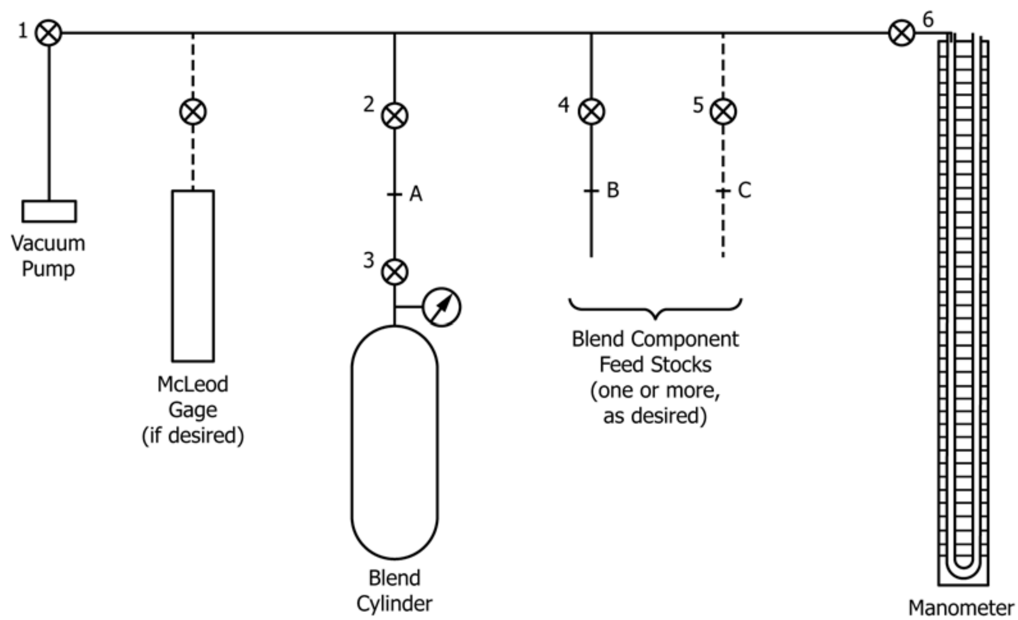

FIG. 1 Manifold System

state condition of the manometer or electronic pressure gauge reading can be taken as an indication that an acceptable vacuum has been attained.

6.1.1 When a good vacuum less than 0.01 kPa (0.1 mm Hg) is reached, connect one or more blend component cylinders to the manifold at positions *B* or *C*, or both. Close valve *2* and open valves *4* and *5*, thereby evacuating the connecting lines up to the blend component cylinder valves. When a good vacuum is again reached, close valves *4* and *5* and open the blend component cylinder valves. Ensure that the pressure of any blend component delivered to valves *4* and *5* does not exceed 200 kPa (1500 mm Hg). Record the initial pressure readings from both sides of the manometer.

6.1.2 The first component to be added will either have the lowest vapor pressure or will be present in the final blend at a concentration of 5 % or less. Assume that the first component feedstock is connected to manifold valve *4*. Close valve *1* and open valve *2* . While carefully watching the manometer or electronic pressure gauge reading, *slowly* open valve *4*. Allow the blend gas component to flow into the blend manifold until the desired precalculated manometer reading is reached (see 7.1). Close valve *4* and be sure that the pressure remains constant. If using a manometer, tap it lightly to be certain the correct reading is obtained. Record the reading of both sides of the manometer and then close valve *3*. Open valve *1* and wait until the manifold is thoroughly evacuated.

6.1.3 If the manifold includes only one feedstock connection point it will be necessary at this time to remove the first feedstock cylinder, connect the second, and evacuate the line back to the feedstock cylinder valve. Assume this to be the case; value *4* will, therefore, always be used as the feedstock control valve.

6.1.4 When manifold evacuation is complete, close valve *1* and *4*. Open the feedstock cylinder valve and then *slowly* open valve *4*, allowing the second blend gas to flow into the manifold. Carefully watch the manometer or electronic pressure gauge.

NOTE 3—All additions should be made slowly to avoid temperature changes.

6.1.5 When the pressure in the manifold is several pascals higher than the previous reading and is still slowly rising, slowly begin to open valve *3* so as to admit the component to the sample cylinder. Valve *4* will remain partially open. Continue to open valve *3* while controlling the flow through valve *4* until the next desired pressure level is reached, always maintaining a higher pressure in the manifold than that in the cylinder. Close valve *4*, allow the pressure to equilibrate, and record the manometer reading from both sides. Close valve *3*. When additional components are to be included in the blend, repeat the procedures outlined above for each component.

6.1.6 When all components have been added, and valve *3* is closed, evacuate the manifold, close valve *2* and disconnect the blend cylinder from the manifold at position *A*. To shut down the apparatus, close the feedstock cylinder valve and open valve *4* to evacuate the connection. Close valve *4*, remove the feedstock cylinder, close valve *1*, and by using valve *2* or *4*, *slowly* admit air into the system until it is at atmospheric pressure.

6.2 The blend *must* be mixed before it is used. This can be accomplished in several ways, one of which is to cause convection currents to occur within the cylinder. This may conveniently be done by heating one end of the cylinder with either a hot air gun or by running hot water over one end of it for about an hour. Never use a flame to heat the cylinder. Blends containing hydrogen or helium are very difficult to mix. Therefore, it is necessary to periodically alternate heating of first one end of the cylinder and then the other for several hours.

6.3 To prepare a blend containing components at the parts per million level, it is necessary to make an initial blend of those components at higher concentrations and then to make successive dilutions until the final desired concentration level