Preparation of Primary Standards by Gravimetric Methods

GML Technical Procedure

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1. Purpose

This document provides the technical procedures for the preparation of primary standards (as compressed gas mixtures) by gravimetric methods. These procedures are valid for the following mixtures:

- Carbon monoxide in air
- Methane in air
- Nitrous oxide in air
- Sulfur hexafluoride in air
- Combinations of the above

Mixtures prepared by gravimetric methods are traceable to the SI quantity mass.

2. Scope

This document describes procedures for the preparation of primary standards by gravimetric methods.

3. References

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4. Terms and Definitions

- **Analyte**: One or more compounds of interest. For example, in an N_2O-in-air standard, N_2O is the analyte.

- **Control Volume**: An object of similar mass and volume to that for which the mass is to be determined. The control volume is used to track relative changes in the mass of the primary object to be weighted (cylinder, transfer tube, capillary, etc.) and also to track drift in the balance.

- **Diluent**: The gas, usually ultra-pure or synthetic air, which is used to dilute the mole fraction(s) of the analyte(s) to the desired level.

- **Mass Piece:** Weight (stainless steel or brass) from a calibration mass set, traceable to national standards (NIST).

- **Mole Fraction**: The ratio of the number of moles of a particular compound to the total number of moles present. Commonly used abbreviations include: ppm = μmol mol^{-1}, ppb = nmol mol^{-1}, and ppt = pmol mol^{-1}.

- **Primary Standard**: A measurement standard established using a primary reference measurement procedure, or created as an artifact, chosen by convention.

- **Receptacle**: Volume to which gas will be added (included cylinders, transfer tubes, and other devices used to store gas samples).

- **Reference Cylinder**: Cylinder of dry air designated for the calibration of other standards for quantities of the same kind. The instrument response function is often determined from instrument response relative to the reference cylinder. The reference cylinder is also used to track instrument drift on short time scales.

- **Scrubbing System**: Various absorbents, packed in stainless steel volumes, used for removing impurities from diluent gas.

- **Secondary Standard**: A standard whose value is determined through analysis relative to primary standards, or through comparison to other secondary standards with verification by comparison to primary standards, for a quantity of the same kind. These standards are used to calibrate the instrument response. Use of secondary standards for routine calibration prolongs the life of primary standards.

- **Tertiary Standard:** A standard whose value is determined through analysis relative to secondary standards, for a quantity of the same kind.

- **Transfer Tube or Transfer Volume**: A singled-ended or double-ended tube (usually stainless steel) with valves on one or both ends, used for transferring an aliquot of gas to a cylinder.

5.0 Procedures

5.1 Gas Blending

Gas blending is performed using a gas manifold, which consists of a vacuum pump, a series of valves, and an optional gas scrubbing system. Valves and other materials should be chosen to minimize “memory effects” when gases of different mole fractions are introduced. The gas scrubbing system consists of different absorbents or catalysts to remove trace contaminants from the diluent gas. The selection of absorbents/catalysts depends on the analyte of interest (e.g. Section 5.5.2).

5.2 Cylinder Selection and Preparation

Primary standards are prepared in various types of cylinders; the choice of which depends on its ability to maintain the integrity of a particular gas mixture at a particular mole fraction, during

long-term storage. Cylinder size, materials, valves, and surface treatments vary depending on the stability characteristics.

For example, N_2O and SF_6 standards are typically prepared in 5.9 L aluminum cylinders with brass pack-less valves, while carbon monoxide standards are prepared in 29.5 L cylinders owing to potential out-gassing of CO that may occur over time. For standards prepared at mole fractions near those found in the remote troposphere, cylinders are typically conditioned to a gas mixture of similar mole fraction for several days or weeks prior to use. Cylinders can be reused when contamination or surface losses are expected to be negligible (i.e., when cylinders will be exposed to similar mole fractions and gas mixtures).

5.3 Balance Selection and Use

Weighing can be performed on a number of electronic and analytical balances. Balances should be selected to maximize weighing precision, unless other factors are deemed more important. Weighing is performed relative to a control object (cylinder, capillary, or transfer tube) of similar mass and volume. This allows for the tracking of drift and also eliminates the need for buoyancy corrections in most cases. Objects should be weighed multiple times relative to control objects. Linear drift correction is generally sufficient. Efforts to achieve optimal weighing precision should be pursued, including minimizing air drafts, floor vibrations, and rapid changes in room temperature.

For balances that are auto-calibrating, a calibration sequence should be performed on the day of use. For balances that are not auto-calibrating, the calibration should be checked and recorded prior to use. This can be done by either weighing a series of mass pieces from a mass set, or by adding the receptacle to be weighed to the balance, and then adding a mass piece approximately equal to the expected change in mass. This method ensures that the mass to be determined has been referenced to at least one standard mass piece at the appropriate span.

Standard mass pieces and control objects should be stored in a dust-free environment, and retained for future comparison to provide a history of relative calibrations. Prior to weighing receptacles, dust and other loosely attached objects such as labels or paper tags should be removed if these are likely to come off during preparation of the gravimetric standard. Objects should be at room temperature for weighing.

When using the mass comparator, users should be aware that the comparator is only accurate when comparing equal masses. A correction factor may be required when comparing unequal masses, and can be determined by using a gas cylinder as a tare weight, and adding 1-kg and 5-kg stainless steel weights to the tare. A custom-made aluminum cylinder cap is used to mount the 1-kg and 5-kg weights vertically on the tare cylinder.

5.4 Traceability

Balances should be serviced and calibrated periodically by an accredited organization to ensure NIST traceability. Experience has shown that services provided by Mettler Toledo are satisfactory. The period of service depends on the level of precision and accuracy required, and performance history of the balance. The following balances should be serviced annually: Sartorius CCE40K3, Mettler Toledo XP6, Mettler Toledo XPR205, and Mettler Toledo XSR1203S. In the event that a balance cannot be certified, weighing should be referenced to standard mass pieces.

Mass pieces should be calibrated regularly, with the schedule determined by considering the likely change in mass over a period of time and the effect of these changes on uncertainties associated with preparing standards. Records of mass piece calibration or comparison should be kept for reference.

Measurement of the oxygen mole fraction in dilution air is traceable to NIST SRM 2659A ($20.863 \pm 0.021\%$ O_2).

5.5 Purity Assessment

5.5.1 Reagents

High-purity reagent gases should be obtained whenever possible. An assay of reagent gases used to prepare primary standards should be performed to the extent possible. The purity of the reagent (determined either from in-house assay, or manufacturer's report) should be recorded along with any purity corrections applied. If a purity assay is available, the impact of the uncertainty in the purity assessment should be considered, and additional purity assessment performed, if warranted.

If a purity assay of the reagent substance is not available or direct assessment cannot be performed due to technical limitations, a secondary purity assessment can be performed by analyzing a diluted sample of the reagent and comparing impurities in the diluted sample to those in the dilution gas. Methods available for purity assessment include GC-MS, GC-ECD, various spectroscopic methods, and trace water vapor analysis. Methods to purify reagents may also be employed, such as freezing samples using liquid nitrogen and pumping off non-condensable gases.

5.5.2 Diluent Gas and Minor Components

The quality and composition of the dilution gas and minor components should be considered with respect to the component of interest. Experience has shown that impurities in the dilution gas are often critical and significant (ISO 19229, 5.2). The presence or absence of other trace

gases could also impact the measurement if significant measurement artifacts exist. For example, argon could be important if standards are to be analyzed using single-line spectroscopic instruments.

The choice to use synthetic or purified whole air for dilution gas depends on the analytical technique and the relative purity of the dilution air. In general, purified whole air has been used for CH_4 and CO, and synthetic air has been used for N_2O and SF_6.

The choice to use dilution air as received from the vendor (without additional purification) or after additional purification should be considered with respect to the gas of interest.

For example, N_2O standards made in the early 2000s were made using synthetic air with additional purification to remove ~1-5 nmol mol^{-1} N_2O present in the dilution air. This introduced ~0.5 nmol mol^{-1} uncertainty due to variable purification efficiency, which was difficult to control. Consequently, more recent N_2O standards were prepared without additional purification, as better-quality dilution air became available (N_2O approx. 0.1 nmol mol^{-1}).

If additional purification is desired, the following materials should be considered:

a) Molecular sieve 13x to remove N_2O (at -78 °C)
b) Activated carbon to remove SF_6 (at -78 °C)
c) Sofnocat 420 or 514 to remove CO

Quantifying the mole fractions of impurities in dilution gas can be difficult. Amount fractions of the major component in synthetic air and purified whole air are typically very low (~0.1% of the prepared mole fraction), and can be difficult to quantify in a traceable manner.

For example, it can be difficult to quantify 2 nmol mol^{-1} CH_4 in a sample of purified air, and establishing traceability at that level is often not possible. Even when traceable standards are available, they are typically not available at comparable amount fractions, and extrapolation to the mole fraction needed can be problematic. Therefore, uncertainty budgets should include a realistic assessment of uncertainties associated with purity assessment of the dilution gas.

5.6 Transfer Procedures

Aliquots of liquid or gas are transferred to cylinders in a variety of ways. The four methods used in GML are described here.

5.6.1 Capillary Method

Note, the Capillary Method is not applicable to primary standards prepared in conjunction with GML's role as WMO/GAW CCL. It is included here for reference.

A liquid reagent is transferred to an evacuated gas cylinder using the glass capillary method. A glass capillary, sealed on one end, is first weighed relative to a control object. Then, a pure reagent or known liquid mixture is transferred to the capillary (either by cryogenic trapping on a vacuum manifold, or by inserting the capillary into a test tube with the open end submerged in liquid and drawing liquid into the capillary by pulling a slight vacuum on the test tube (Schmidt and Rook, 1983)). The condensed phase is moved to the sealed end by centrifuge or cryogenic cooling and the open end of the capillary is sealed in a flame. Liquid can also be drawn into the capillary using cryogens when the transfer is done on a vacuum manifold. If the procedure is performed on a vacuum manifold, sealing the capillary under vacuum cuts the capillary in two pieces (a sealed piece containing liquid, and a residual piece). After equilibrating to room temperature, the capillary piece(s) are weighed again relative to a control. The mass of liquid is determined from the difference between the relative weights, corrected for any additional changes in mass (buoyancy, density, mass added/lost upon sealing, etc). Typically, these corrections are very small (~0.03%).

The aliquot is transferred to an evacuated cylinder by inserting the capillary in a Teflon tube, and attaching the Teflon tube to the cylinder and to a supply of diluent gas. Room air is flushed out of the tube by flowing diluent gas to vent. Then, the cylinder valve is opened and a flow of diluent gas is established. The capillary is broken and gently heated to vaporize the aliquot and transfer all aliquot to the cylinder. Finally, the cylinder is pressurized with diluent gas.

5.6.2 Direct Transfer Method

This method involves the transfer of gas, either in pure or diluted form, directly to a cylinder followed by addition of dilution gas.

a) An evacuated cylinder is weighed relative to a control volume.
b) An aliquot of gas is transferred to the cylinder.
c) The cylinder is weighed relative to a control volume.
d) Dilution gas is added to the cylinder.
e) The cylinder is weighed a final time relative to a control volume.

5.6.3 Tube Expansion Method

This method involves the transfer of an aliquot of gas (pure or diluted) to a transfer volume (tube) and then to the cylinder. This method can be advantageous in that large dilution ratios (.e.g. 4000:1) can be achieved with relatively high precision, thus reducing the number of steps needed to reach ppb and ppt levels. Further, the cylinder is exposed to a high concentration of analyte for only a short time, since dilution gas can be added immediately to the cylinder without re-weighing.

a) An evacuated cylinder is weighed relative to a control volume.
b) An aliquot is transferred to an evacuated transfer tube on the vacuum manifold.
c) The transfer tube is weighed relative to a control volume.

d) The aliquot is allowed to expand into an evacuated cylinder.
e) The valve on the transfer tube is closed.
f) Residual aliquot in the transfer line is pushed into the cylinder using dilution gas.
g) The transfer tube is weighed again relative to a control volume.
h) Dilution gas is added to the cylinder.
i) The cylinder is weighed relative to a control volume.

5.6.4 Tube Flush Method

This method involves the transfer of an aliquot or gas (pure or diluted) to a transfer volume (tube) and then to the cylinder. Here, the aliquot is transferred to the cylinder not by simple expansion, but by repeated pressurization and expansion using dilution gas. This method has the advantage that the cylinder need not be under vacuum for analyte transfer.

a) A cylinder is weighed relative to a control volume.
b) An evacuated transfer tube is weighed relative to a control volume.
c) An aliquot is transferred to the transfer tube on the vacuum manifold.
d) The transfer tube is weighed relative to a control volume.
e) The aliquot is allowed to expand into the cylinder. It is helpful if the gas pressure in the transfer tube is higher than the initial pressure of the cylinder, but this is not strictly required. If $P_{tt} < P_{cyl}$, the aliquot can be expanded into an evacuated section of the manifold, pressurized to $P_{man} > P_{cyl}$, and then expanded into the cylinder.
f) The transfer tube is repeatedly pressurized with dilution gas ($P_{man} > P_{cyl}$) and expanded into the cylinder. The tube is typically flushed 10 or more times in this manner.
g) The valve on the transfer tube is closed.
h) Dilution gas is added to the cylinder.
i) The cylinder is weighed relative to a control volume.

The transfer efficiency can be estimated by adding a known amount of additional diluent to the transfer tube after the analyte has been transferred to a cylinder, measuring the amount of analyte in the transfer tube, and comparing that to the amount of analyte initially added to the tube. Experiments with N_2O and CFC-12 indicate a transfer efficiency greater than 99.998%.

5.7 Data Collection and Storage

Data with respect to the preparation of the primary standards are kept in a notebook or in electronic form. Deviation from the procedures noted in 5.6.1-5.6.4 should be recorded and their potential effects explained. Values assigned to primary standards used to propagate the scale to secondary standards, are stored in a MySQL database.

5.8 Verification of Primary Standards

Verification is performed as a consistency check and as part of scale development. Verification of primary standards is accomplished by comparing a number of primary standards to each other using appropriate instruments. The relative responses of various primary standards should agree, to first order, with the expected response of the instrument. For example, standards should show a linear relationship on an instrument with an inherently linear response. Additional verification can be performed by preparing standards using a variety of techniques (5.6.1-5.6.4) and a variety of cylinder sizes or types. Note: Since individual primary standards are not distributed, we do not include verification uncertainty in the uncertainty estimate for an individual primary standard. Instead, we use multiple primary standards to define a response function (section 7.0), and derive an uncertainty for the response curve from uncertainties in the gravimetric standards and uncertainty associated with curve fitting.

Gas mixtures are allowed to "mix" for a minimum of one week prior to analysis to ensure homogeneity. Methods to induce mixing can also be employed, including "rolling," and differential heating/cooling (ISO 6142-1, 9.1). For mixtures that have endured long storage periods (years), the above methods are used to re-homogenize the mixture prior to use.

6. Calculations

6.1 Dry Air Mole Fraction

Primary standards are reported as dry air mole fraction. Molecular weights (MW) are calculated using recent values for atomic weights, including estimates for the natural abundance of various isotopologues in the source material (e.g. Meija et al., 2013). The molecular weight of synthetic air (O_2+N_2) or natural air (O_2+N_2 +Ar) is determined by measuring the oxygen content. Ranges for molecular weights were calculated from data in Wieser and Coplen (2011) and Meija *et al.* (2016), and included in the uncertainty analysis.

The mole fraction of a component (χ_i) is calculated as the number of moles of that component (m_i) divided by total number of moles (m).

$$\chi_i = \frac{m_i}{\sum m} \qquad (1)$$

For a single component standard in air,

$$\chi_i = \frac{\frac{M_i}{MW_i}}{\frac{M_i}{MW_i}+\frac{M_{air}}{MW_{air}}} + \chi_{i_air} \qquad (2)$$

where M is mass, MW is molecular weight, and χ_{i_air} is the mole fraction of component i present in the dilution air as impurity. The practical implication of using equation (2), as opposed to equation (3) in (ISO 6142-1:2015) is negligible (<0.01%). Further, a purity correction may be applied to individual components when known.

6.2 Uncertainties

Two types of uncertainties can be calculated for primary standards. The first is based only on weighing repeatability. Relative weight uncertainties are summed in quadrature, following procedures outlined in JCGM (2008). This uncertainty estimate includes only statistical uncertainties and is useful when comparing standards during verification.

A deviation from ISO 6142-1 is noted here. Currently, covariance between daughter standards and parents is not considered. Analysis of N_2O and SF_6 primary standards suggests that covariance is a minor contributor to uncertainty compared to (a), (b), (c), (i), and (j) below.

To estimate the total, expanded uncertainty, uncertainties are summed according to methods outlined in JCGM (2008).
Uncertainties may include:

a. Weighing repeatability
b. Reagent purity, including cross contamination from multiple reagents
c. Impurities in the dilution gas
d. Transfer efficiency
e. Loss of metal, paint, or labels from cylinders or receptacles
f. Leakage from transfer tubes
g. Memory effects in transfer lines
h. Physical or chemical interactions with cylinder surfaces
i. Composition of the dilution gas
j. Molecular weight
k. Scale transfer from primary-to-secondary and secondary-to-tertiary levels

Typically (a), (b), (c), (i), and (k) above are the most important.

7.0 Calibration Scales - (Excluding CO_2)

The preparation of primary standards ultimately leads to the establishment of a calibrations scale. Here, we define a calibration scale, and how it is updated. Within GML, an assignment of mole fraction by analysis is not typically made with reference to one primary standard. Instead, a suite of primary standards is prepared and related to a reference cylinder through analysis. The gravimetrically prepared values are used to establish a "response function," on a particular instrument.

The response function can have different forms. Typically, the instrument response relative to a reference cylinder (*R*) is expressed as a linear or polynomial function of the mole fractions (*C*) of primary standards, $R = f(C)$. The best-fit function is then used to "assign" new values to each primary standard in the suite, as well as to the reference cylinder. The assignments are typically done over a period of time, such that multiple assignments can be averaged and statistics computed.

The primary standards and their assigned values constitute the calibration scale. Scales are named according to the year in which they are adopted. For example, the nitrous oxide scale consists of 13 primary standards analyzed on an Agilent 6890 gas chromatograph with electron capture detector, and is referred to as the WMO N_2O X2006A Scale. The scale is then transferred to secondary standards for routine use. If a primary standard is added or removed from the suite, this results in the formation of a new scale, or includes a statement of equivalency. A new scale can be related to an older scale via secondary or tertiary standards.

8.0 Safety

It is NOAA/GML policy to follow safe working practices when handling compressed gas cylinders and laboratory chemicals. Compressed gas cylinders should be secured (except when they are being weighed). Personal protective equipment (PPE) should be used when working with hazardous chemicals or in a high noise environment. Personnel working with radio nuclides (electron capture detectors) must be trained according to NOAA/GML policies.

9.0 Documentation

The calculated mole fractions and molecular weight of each primary standard is recorded in a database. A paper label is created and attached to the cylinder. The label identifies the mole fractions and their uncertainties for each component of interest, the creator, and the notebook page on which more information can be found. Calibration certificates are not prepared for primary standards.

10.0 Appendix

10.1 Uncertainties

Weighing Uncertainties

The uncertainties associated with mass determinations on analytical balances are determined from multiple weighing episodes, relative to a control object, and corrected for drift. Uncertainties depend both on the resolution of the balance and the weighing technique. Typical weighing sequences are ABABA …, ABBABBA…, and ABBBABBBA. Uncertainties, expressed as one standard deviation, are as follows:

Balance	**Capacity**	**Typical Repeatability**
Sauter E1216	60 kg	1 g
Sartorious CCE40K3	40 kg	2, 10 mg*
Mettler XSR1203	1200 g	0.7 mg
Mettler XPR205	200 g	0.03 mg
Mettler XP6	6 g	2 µg
Mettler UM3	3 g	1 µg

**for N30 (small) and N150 (large) aluminum cylinders, respectively*

Buoyancy Effects

Because all mass determinations are made relative to an object of similar density, buoyancy effects will only influence the standards when (a) the air density changes between weighing

episodes, or (b) the air density differs from the nominal 20°C and 1.20 kg m^{-3} (standard conditions to which weights are already referenced). The time scales for variations in humidity, room temperature, and pressure are typically long compared to the time scales of weighing. The contribution of buoyancy is approximately 0.015% of the weights used, and weights are normally already corrected for buoyancy at an air density of 1.2 kg m^{-3}. The error introduced by not correcting for air density in Boulder (1.0 kg/m^{-3}) is 0.0026%, and is ignored. Buoyancy corrections are applied to the capillary method because comparisons are made between objects of different density.

Uncertainty Example (N_2O)

The typical standard uncertainty for a single primary standard contributing to the WMO-N_2O-X2006A scale, at 325 nmol mol^{-1}, is 0.4 nmol mol^{-1}. Relative contributions of various uncertainty components are shown below.

Typical Primary Standard (N_2O)	Relative Contribution	Type	Distribution
Reagent purity	13%	B	rectangular
Mass determination (major component)	38%	A	normal
MW major component	< 1%	B	rectangular
Transfer efficiency	3%	B	normal
Oxygen/nitrogen content of the dilution gas	10%	B	rectangular
N_2O in dilution gas	35%	B	rectangular

Analysis of a suite of primary standards and subsequent determination of a response function by orthogonal distance regression leads to an uncertainty estimate for the response function, which can then be combined with uncertainties associated with scale transfer from primary to secondary standards, and then from secondary to tertiary standards. For current information on uncertainties, see https://gml.noaa.gov/ccl/ccl_uncertainties.html.

10.2 Equipment

The following equipment is critical to the functions described in this TP.

Item	Manufacturer	Model Number
Gas blending manifold (2)	Custom	
Analytical balance	Mettler Toledo	XP6
Analytical balance	Mettler Toledo	XPR205
Analytical balance	Mettler Toledo	XSR1203S
Analytical balance	Sauter	E1216
Mass comparator	Sartorious	CCE40K3
Mass pieces	Troemner	10 kg; 0.05-200 g set

Mass pieces	Mettler Toledo	1 kg, 5 kg
Oxygen analyzer	Systech Illinois	710
Transfer tubes (5, 10, 50 cm^3)	Custom	
Vacuum Pump	Varian	Tri-scroll