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Standard Reference Materials:

The NIST Traceable Reference Material Program for Gas Standards

Franklin R. Guenther, William D. Dorko,
Walter R. Miller, and George C. Rhoderick

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FOREWARD

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CONTENTS

1. INTRODUCTION

A NIST Traceable Reference Material (NTRM) is a reference material produced by a commercial supplier with a well-defined ***traceability*** (the definition of emphasized words can be found in Appendix 1, "Definition of Terms") to the National Institute of Standards and Technology (NIST). This traceability is established via criteria and protocols defined by NIST that are tailored to meet the needs of the metrological community to be served. The NTRM concept was established to allow NIST to respond to the increasing needs for high quality reference materials with constant human and financial resources. Reference material producers adhering to these requirements are allowed to use the "NTRM" trademark to identify their product.

The gas NTRM program was established in 1992 in partnership with U.S. Environmental Protection Agency (EPA) and specialty gas companies as a means for providing end-users with the wide variety of certified gas standards needed to implement the "Emissions Trading" provision of the 1990 Clean Air Act. Gas NTRMs are produced and distributed by specialty gas companies with NIST oversight of the production and maintenance, and direct involvement in the analysis. NTRMs can be developed for any pollutant, concentration and balance gas combination for which a NIST primary standard exists. The gas standards prepared according to this program are related, within known limits of ***uncertainty*** [Ref. 1]), to specific gaseous primary standards maintained by NIST.

This program is jointly administered by the NIST Analytical Chemistry Division and the NIST Standard Reference Materials Program. The purpose of the program is to produce gas mixtures (NTRMs) to supplement the supply of existing gaseous ***Standard Reference Materials***™ (SRMs) and to be used where SRMs have been used in the past. The program is also designed to replace the EPA Certified Reference Material (CRM) program. The EPA has agreed to accept an NTRM in place of a CRM.

The procedures described herein are based on NIST experience relative to the production and certification of gaseous SRMs, and are intended to assure the development of reliable NTRMs. The integrity of the gas NTRMs produced through this program is assured by NIST through active quality assurance measures. These measures include; NIST oversight of the production and analysis of the NTRM by the producer; NIST analysis of cylinders representative of the NTRM; NIST assignment of the NTRM certified value and associated uncertainty; documentation supplied by NIST, including details of the NTRM certification, a Certificate of Traceability, and cylinder labels; and, NIST oversight of the long-term maintenance of the NTRM batch by the producer.

2. PROGRAM OVERVIEW

2.1 Description

This section gives an overview of the entire program (see Appendix 2 for flow chart). Where needed, subsequent sections explain various parts of the program in greater detail.

A NIST Traceable Reference Material (NTRM) is a reference material produced by a commercial supplier with a well-defined traceability to the National Institute of Standards and Technology (NIST). This traceability is established via criteria and protocols defined by NIST that are tailored to meet the needs of the metrological community to be served. The NTRM concept was established to allow NIST to respond to the increasing needs for high quality reference materials with constant human and financial resources. Reference material producers adhering to these requirements are allowed to use the "NTRM" trademark to identify their product.

A gas NTRM consists of a multi-cylinder ***batch*** (the definition of emphasized words can be found in Appendix 1, "Definition of Terms") prepared in such a manner that all the cylinders contain a gas mixture identical in composition and ***concentration***. The cylinders, cylinder treatment, reagent gases, and gas blending procedure must assure mixture stability for a minimum period of time. The concentration of the NTRM must be within the bounds of a suite of gaseous ***NIST primary standards*** (Appendix 3). The concentration of an NTRM is determined by analysis with specific NIST standards. The uncertainty assigned to the concentration depends on the combined uncertainty in the NIST standards, the producer's ***homogeneity*** analysis, and the analysis performed at NIST. A single certified concentration and uncertainty value are assigned to the batch. The certified concentration and uncertainty, the period of time during which the concentration of the NTRM is certified, the presence of any ***impurities*** of consequence, and a list of all the cylinders included in the batch is given in a NIST Report of Analysis. A Certificate of Traceability is issued for each successful NTRM, along with labels to be affixed to each cylinder.

2.2 Qualification of Producer

In order to qualify for participation in the NTRM program, the producer must meet certain minimum requirements. These requirements are intended to assure the high quality of NTRMs and their availability to U.S. laboratories. There are three requirements;

1) The producer must have in place a documented ***quality system.*** This should follow international or national guidelines (*ISO Guide 25, ANSI/NCSL Z540-1-1994* or equivalent).

2) The producer must have the necessary facilities, equipment, and personnel to produce and analyze the gas mixture in the manner described in this document. NIST may require a meeting with key personnel and/or a site visit to the producer's facility.

3) The producer must agree to one or more of the following; a) make the NTRMs available for sale to U.S. customers; b) demonstrate that traceable standards produced from NTRMs (as described in section 6.2) will be made available to U.S. customers; or, c) will be used to produce gas standards according to the EPA protocol standards document [Ref. 2].

2.3 Batch Planning and Scheduling

Before an NTRM batch is prepared, the producer must contact NIST for discussions, including the establishment of a production/certification schedule agreeable to both parties. At this time any questions can be addressed so that there is a mutual understanding of procedures. The producer must submit a plan describing the analytical procedures to be used, including the identification of the ***batch standard*** and calibration standards to be used. NIST will supply the producer with technical specifications the NTRM is expected to meet or exceed in order to be certified. After concurrence on pertinent points the batch preparation can proceed.

NIST will provide to all NTRM producers a certification schedule that will detail the optimum dates for submission of NTRM batches for certification analyses. The schedule will detail, by chemical component, when NIST will best be able to deliver certification results within a minimum time period. NTRM batches submitted within the schedule will be given higher priority over those submitted outside the optimum submission period. NIST will complete it's certification measurements within 3 months of receipt of the NTRM cylinders for analysis. NIST may need additional time to certify NTRMs submitted out of sequence.

2.4 Batch Production

Each NTRM must be prepared as a multi-cylinder ***homogeneous*** batch (minimum 10 cylinders). The cylinders must be clean, of standard size and construction, and of known compatibility and history with the composition of the proposed NTRM. All NTRM cylinders within the batch must be identical with respect to size, construction, valve, and cylinder treatment. The cylinders must be equipped with valves of the appropriate material that conform to Compressed Gas Association (CGA) recommendations for the particular gas mixture. The reagent gases used must be of high purity and quality to ensure that impurity levels and mixture stability meets NIST technical specifications.

Care must be exercised when filling the cylinders in order for the batch to meet NIST homogeneity specifications. Any one of several fill methods can be used, including; 1) from a bulk mixture prepared in a single container; 2) from several containers connected together in such a manner that delivery occurs simultaneously from each container; 3) by use of a dynamic blending system; or, 4) other fill method shown to meet NIST batch homogeneity specifications. A diagram of the filling manifold, which includes the position of each of the cylinders, will be maintained as part of the NTRM batch records. All cylinders will be reported to NIST referencing the cylinder number stamped permanently on the cylinders, along with a sequential ***sample number*** assigned to each cylinder. Further details on batch production can be found in Section 3.

2.5 Producer Analysis

The responsibility of the producer is threefold; 1) to determine the concentration relationship among all of the cylinders in the batch (batch homogeneity); 2) to establish the average concentration of the batch; and, 3) to establish a baseline from which to compare future analyses performed to evaluate batch stability. To accomplish this, one or more ***NIST certified standards*** (see Definition of Terms) are to be used. The producer's data are used to assess batch homogeneity and are included in the overall expanded uncertainty in

the concentration of the batch. Thus it is imperative that the producer generates high quality data in order for the expanded uncertainty to be minimized. Section 3 provides more details on the analysis phase of NTRM batch production.

2.6 NIST Data Review

The producer must submit to NIST all the data generated from activities described in sections 2.4 and 2.5. The producer should review these data prior to submission to determine if the batch meets NIST specifications. Once the producer is confident that the batch meets specifications, the producer should submit the data using a NIST approved data format. NIST will review the data to determine if the batch meets NIST specifications and to select representative cylinders from the batch for NIST evaluation. Upon request, the producer will provide NIST with any additional data and/or information relating to the production and analysis of the NTRM batch. Proprietary information is generally not needed for this review, however NIST can enter into nondisclosure agreements if necessary.

Data that must be sent to NIST include; 1) cylinder fill date and method; 2) manifold fill position with cylinders identified by cylinder and sample number; 3) identification of batch standard; 4) analytical data including cylinder concentration, calibration standards, and order of analysis; and, 5) impurity data for specified species. The data will be submitted to NIST using the data submission form in Appendix 4, or by electronic means, such as a computer floppy disk, in a comma-separated variable (CSV) file format.

NIST will review the data to determine if; 1) the analytical method yields reliable results; 2) the batch homogeneity meets NIST specifications; and, 3) the batch concentration meets target values. If the data supports it, NIST will proceed to the certification phase by selecting a minimum of 2 cylinders for NIST evaluation. The batch standard will most likely be included in the ***selected cylinders***. The producer will then send to NIST these cylinders plainly labeled with sample number, producer, specie, and concentration. If the batch is rejected at this point due to evidence of inhomogeneity in the submitted data, NIST may ask the producer to perform additional analytical work and resubmit the batch. *However, if the inhomogeneity is due to the fill method, the batch can not be certified.*

2.7 Batch Certification

After NIST reviews the data the producer must send the selected cylinders to NIST for evaluation. NIST will evaluate the cylinders for concentration and may analyze for impurities. The NIST analysis will involve comparisons of the NTRM cylinders against NIST standards. NIST standards that may be used in these analyses include primary standards, SRMs, and ***SRM lot standards***. In general the analyses that NIST performs to value assign NTRM batches will be identical to those used in the certification of SRMs. The NTRM will be certified for a specific period of time dated from the NIST Report of Analysis. At the end of this period the producer must submit ***recertification*** data to NIST in order for the certification period to be extended. As long as the producer maintains the batch as described in Section 5, a customer purchasing an NTRM cylinder will have a certified period extending from the date of sale. More details on the NIST certification process can be found in Section 4.

2.8 Maintenance of NTRM Batches

It is the responsibility of the producer to maintain the NTRM batch over the potential lifetime of the individual cylinders. In general the batch standard is used to measure the stability of the entire batch. Periodic analysis of the batch standard against a NIST certified standard will allow a determination of the batch stability over time. NIST must be notified if the batch standard shows a change in concentration greater than half of the stated uncertainty for the batch. NIST will recommend a course of action to further assess batch stability. Actions that may be recommended include; 1) NIST reanalysis of the batch standard; 2) analysis of some or all remaining batch cylinders by the producer; or, 3) recall of selected cylinders for analysis by the producer. More details on the maintenance of NTRM batches can be found in Section 5.

2.9 Batch Certification Cost

NIST is compensated directly by the producer for the certification of each candidate NTRM batch. Fees are established on a yearly basis and are effective on October 1 of each year. Fees are based on the number of cylinders in the batch with an additional surcharge if the certification requires interpolation between primary standards. Also included in the fee is a charge for preparation of the Certificates of Traceability and the cylinder labels. A purchase order must be submitted to NIST before any work can proceed.

3. BATCH PRODUCTION AND ANALYSIS

3.1 Overview of Producer Responsibilities

The NTRM batch must be blended in such a way as to assure gas mixture homogeneity and stability. After blending, the contents of each cylinder in the batch must be analyzed by the producer. The analytical procedure recommended here is considered to be the minimum effort necessary to adequately evaluate each batch for homogeneity and stability. All NTRMs must be stable, and each batch should be homogeneous in composition and concentration. An unstable batch must be rejected, while a batch exhibiting a small inhomogeneity in concentration may be acceptable, providing that the resulting overall expanded uncertainty in the concentration does not exceed the desired level. The NTRM program is based on a batch certification; all cylinders comprising the batch are assigned the same absolute concentration. Thus, any batch inhomogeneity will increase the uncertainty assigned to the certified value of the batch.

The objective of the producer's analysis is to compare each cylinder mixture (sample) in the batch to one or more NIST certified standards. The simplest procedure would involve direct analysis of each NTRM sample with a NIST certified standard, but with most analytical methods this approach would use a large proportion of the available gas. Consequently, it is recommended that the concentration be determined on a relative basis by selecting one sample from the batch, designated the batch standard, to which all of the other samples are compared. The batch standard is then analyzed against one or more NIST certified standards. The concentration of all the other samples can be determined from the measured ratio to the batch standard.

Analyses for impurities which might compromise the use and stability of the NTRM will be performed using procedures determined by the producer. Maximum impurity levels are given in Appendix 3.

3.2 Batch Preparation

Before an NTRM batch is prepared, the producer must contact NIST for discussions, including the establishment of a production/certification schedule agreeable to both parties. At this time any questions can be addressed so that there is a mutual understanding of procedures. The producer must submit a plan describing the analytical procedures to be used, including the identification of the batch standard and calibration standards to be used. NIST will supply to the producer with technical specifications the NTRM is expected to meet or exceed in order to be certified. After concurrence on pertinent points the batch preparation can proceed.

Each NTRM must be prepared as a multi-cylinder homogeneous batch (minimum 10 cylinders). The cylinders which will contain the NTRM mixture must be clean and of known compatibility and history with the composition of the proposed NTRM. All cylinders must be identical with respect to size, construction, valve, and cylinder treatment. The cylinders must be equipped with valves of the appropriate material that conform to Compressed Gas Association (CGA) recommendations for the particular gas mixture.

Care must be exercised when filling the cylinders in order for the batch to meet the NIST homogeneity specifications. Any one of several fill methods can be used including; 1) from a bulk mixture prepared in a single container; 2) from several containers connected together in such a manner that delivery occurs simultaneously from each container; 3) by use of a dynamic blending system; or, 4) other fill methods shown to be effective by the producer. The reagent gases must be of high purity and quality to ensure that impurity levels and mixture stability meets NIST specifications. The concentration of the component of

interest (analyte) must be at, or bracketed by, NIST primary standards (Appendix 3). A diagram of the filling manifold, including the position of each of the cylinders, must be maintained as part of the NTRM batch records. The identities of all cylinders will be reported to NIST referencing the cylinder number stamped permanently on the cylinders, along with a sequential sample number assigned to each cylinder. The sample numbers will span from one through the number of cylinders in the batch and will be permanently assigned to the cylinders throughout the valid certification periods of the NTRM.

Multicomponent NTRM mixtures may be produced as long as every component which is to be certified has a concentration at, or bracketed by, NIST primary standards. Chemical interactions between components of the NTRM must not occur, or must have completed by the time the producer initiates the first analysis. The balance gas, cylinder walls, and valve must be chemically inert with respect to the certified components. Certification of each component is performed independently of all other components.

3.3 First Analysis

An initial analysis of the NTRM batch should be performed two or more days after preparation (see Appendix 3 for ***incubation period***). One analytical protocol that may be used is to select one cylinder from the batch, designated the batch standard, to which all other samples from the batch are compared. The batch standard must be analyzed against appropriate SRM(s) or other NIST certified standards to determine concentration and to establish a reference for later analyses intended to confirm the stability of the batch. The batch standard should be analyzed at least six times using a NIST certified standard to calibrate the instrument. This analysis should consist of at least six alternations of the batch standard and the NIST standard. All of the remaining samples are then analyzed in duplicate, using the batch standard as a reference.

During the first analysis the producer must analyze for impurities using procedures determined by the producer. NIST reserves the right to identify cylinders to be analyzed. For nitric oxide mixtures, all cylinders must be analyzed for nitrogen dioxide.

3.3.1 Instrument Performance

The general characteristics of the analyzer response relative to concentration must be known and demonstrated. This is accomplished by constructing a calibration curve using a minimum of three gas mixtures. The gas mixture nearest in concentration to the nominal batch concentration must be a NIST certified standard. If the analyzer has a significantly nonlinear function then a minimum of four calibration standards are required. If a model using a straight line instead of a quadratic fit results in a predicted concentration different by 0.5 % or more, then four or more standards and a quadratic fit must be used. The standards must include a NIST certified standard above and below the concentration of the NTRM (if such standards are available). Obviously this is not possible when the NTRM is being compared to either the lowest or highest concentration NIST certified standard available in a particular analyte series; these situations will be addressed on a case-by-case basis considering the limits of NIST analytical capabilities.

Extrapolation beyond the calibration curve is not recommended without technical justification. Thus, the NTRM concentration should be adjusted to lie within the concentration bounds of the available calibration gases.

The principal elements that contribute to the imprecision of measurement include instrument ***sensitivity*** and drift. The sensitivity of the instrument should be high enough so that differences in concentration of 0.2 % relative can be easily measured. Instruments are available that are capable of measuring all current SRMs with this sensitivity. However, most instruments exhibit a certain amount of signal drift. The drift has two components, short term drift (noise), and a long term drift characterized by a slowly changing signal under constant operating conditions. The effect of short term drift can be minimized by a number of techniques including multiple analysis of each sample, and signal averaging. The effect of long term drift can be minimized by frequent recalibration of the instrument. The characteristics of each analytical system should be considered individually to determine the optimum frequency of calibration and the approach taken to compensate for or control the effect of noise. It should be noted that the effort expended in minimizing the effects of drift will improve the precision of the analyses relating the NTRM to the NIST certified standard. In general, if duplicate analyses of single samples consistently agree within 0.2 % relative, then the effect of noise is at an acceptable level, and if repeated analysis of the same sample performed at intervals during the analysis of a batch agree within the same limits, then the long term drift is at an acceptable level.

3.3.2 Preliminary Data Evaluation

Some preliminary conclusions regarding the homogeneity and stability of a batch may be drawn from the results of the first analysis. If the batch is homogeneous when filled, and if no subsequent reactions have occurred randomly in the cylinders, the concentration of all samples should be the same within the limits of precision of the analytical method. If inhomogeneity among the samples is observed, it will not be possible at this time to determine whether such inhomogeneity is due to improper preparation or to reactions in the cylinders.

If the first analysis indicates that no serious problem exists, the entire batch is set aside for 30 days. If the data indicate that the batch may be bad, NIST should be notified.

3.4 Second Analysis

After the required minimum 30-day hold period has passed, all samples in the batch must be analyzed in the same manner as the first analysis using the same NIST certified standard(s). The first analysis served to define the concentration of the NTRM and to test for batch homogeneity. Decisions regarding stability depend on the second analysis. Sufficient time will have elapsed so that reactions in individual cylinders, which were of too low a rate to be recognized by the distribution of concentrations obtained in the first analysis, can be measured through this subsequent analysis.

3.5 Batch Stability and Homogeneity

The stability and homogeneity of an NTRM batch must be carefully evaluated. The batch is considered stable if the concentration in each NTRM cylinder has not changed appreciably (less than the measurement uncertainty) between the first and second analyses. If no change is detected, the batch is most likely stable and the producer may proceed. However, if an inspection of the data from the first and second analyses reveals gross instability the batch must be discarded and remade. NIST will assist the producer in analyzing the data if there are questions concerning significance of the differences found.

If cylinder-to-cylinder differences within the batch are observed, then the producer and NIST must decide if this inhomogeneity will result in an unacceptably large expanded uncertainty. An NTRM is useful only if the expanded uncertainty in the concentration is at or below a certain level. In general the expanded uncertainty should be less than two times the maximum stated uncertainty of the NIST certified standards used for calibration. If the inhomogeneity is small enough then this level will not be exceeded. If it is large and the acceptable expanded uncertainty level will be exceeded, then the batch should be remade.

The concentration of the analyte(s) in all of the cylinders of an NTRM batch should be identical. Differences in concentration observed among cylinders should reflect only the combined uncertainty in the measurement process [Ref. 1]. Differences greater than this combined uncertainty may arise from several sources. There can be differences that result from faulty mixing, or from dilution by residual gases left in the cylinder prior to transfer of the bulk mixture. A more critical source arises from reactions, chemical or physical, that occur in the cylinder after transfer. Because these reactions are largely due to wall effects, and because no two cylinder walls are alike, the reactions generally proceed at different rates in different cylinders. The two effects may be differentiated by sets of measurements made over a period of time sufficiently long so that the extent of reaction is greater than the measurement uncertainty in the analysis. If a batch is analyzed immediately after preparation and individual samples are found to have concentrations that vary more than the measurement uncertainty, then the mixture is either inhomogeneous, or some or all of the samples in the batch are unstable. If the batch is reanalyzed after some time and each sample has essentially retained its previous value, then the samples are probably stable, but the batch is inhomogeneous. If the concentration of one or more of the samples has changed significantly, then it is plausible to conclude that the batch is unstable.

It may not be necessary to discard an inhomogeneous batch, but an unstable batch must be discarded. An inhomogeneous but stable batch may be used if the inhomogeneity is sufficiently small that the expanded uncertainty does not exceed the acceptable level. If during blending, several large cylinders are connected together to allow simultaneous delivery, then some inhomogeneity in the batch may result if the concentrations in the source cylinders differ slightly. In the special case of large scale dynamic dilution systems where two components are mixed under constant flow conditions and are then compressed into the cylinders in the batch, some inhomogeneity may result, depending on the design of the delivery system. If variations in concentration are observed in a batch prepared by this method, the variation may be related to the position of individual cylinders on the manifold, and it is therefore important to record the manifold position of each cylinder.

The nature of the concentration distribution of samples in a batch may give significant information concerning both the homogeneity and stability of the batch. A batch which has concentrations that vary no more than can be accounted for by the measurement uncertainty of the analytical method, is probably

homogeneous and stable. A batch in which the range of values exceeds significantly what might be expected on the basis of measurement uncertainty, and in which several samples are considerably below the average, is probably unstable. A batch in which the range of values is large but all concentrations are essentially symmetrically distributed around the average, was probably not prepared or transferred in such a way that homogeneity was assured. Occasionally, one or more samples in a batch will be found with concentrations appreciably different from the average for the batch. These may represent faults in the preparation or filling of the cylinder. These cylinders may be removed from the batch, provided the batch is otherwise shown to be stable and homogeneous.

All of the data at this stage of NTRM production must support the assumption that the batch is stable, homogeneous, and meets all other specifications for an NTRM. If there is any suspicion that the batch may be unstable or inhomogeneous, then further analytical work should be conducted before a final decision is made to proceed.

At this point there should be sufficient data for the producer to decide whether or not the batch will qualify as an NTRM. This decision must be based on the measured concentration, stability and homogeneity. Ultimately the decision will be subject to an independent examination of the data and to a NIST analysis of selected samples from the batch for concentration value assignment.

4. BATCH CERTIFICATION

4.1 Overview

After the producer submits the required data to NIST (see sec. 2.6), the data will be reviewed and a minimum of two cylinders from the batch will be selected to be sent to NIST for analysis. Procedures that are similar to those used for SRM certification will be used to analyze the selected NTRM cylinders. The analyses will be used to determine the concentration of the analyte(s) and any impurities that may compromise the use of the NTRM. This process will normally be completed within three months of sample submission. A Report of Analysis, Certificates of Traceability, and cylinder labels will be prepared by NIST and issued to the producer for all approved NTRMs. A Report of Analysis will also be issued to the producer for rejected batches detailing the reasons the NTRM failed certification.

4.2 NIST Analytical Protocol

NIST analysis of the selected cylinders from the NTRM batch is modeled on the procedures used by NIST to analyze SRMs. In a typical NTRM certification procedure, the producer's batch standard is analyzed using a NIST reference method. Typically this method is an instrumental procedure calibrated with a gas reference material, such as gravimetrically prepared NIST primary standards, SRMs, or SRM lot standards. Then the same instrumental procedure is used to compare the remaining selected cylinders to the NTRM batch standard. The instrumentation used in these analyses is chosen based on; 1) a stable and noise free signal to ensure a high degree of precision in the intercomparisons; and, 2) an adequate signal ***resolution*** to detect small (0.25 %) concentration differences. The same instrumentation used for the certification of an SRM is used for the analysis of the corresponding NTRM.

Since the concentration of an NTRM batch can be at or between NIST primary standard concentrations, two possible cases exist for the analysis of the selected cylinders. Where the NTRM and NIST primary standard concentration are close, an SRM, SRM lot standard, or primary standard will be used to analyze the producer's batch standard. Where the concentration of the NTRM falls between NIST primary standard concentrations, more standards will be used. The number and range of standards used to analyze this type of NTRM will depend on instrument function and the concentration of the NTRM.

In both cases, the producer's batch standard is analyzed by comparing it to a NIST standard(s) a minimum of five times on each of two different days. Potential instrument drift is corrected by alternately analyzing the NIST standard and the producer's batch standard. The NTRM response data is then correlated to the NIST standard response data using a linear or a quadratic instrument response function. Most of the instruments used by NIST have a linear response function.

After the producer's batch standard is analyzed, the instrumental procedure is used to compare the other selected cylinders to the batch standard. The same basic analytical sequence is followed, analyzing the producer's batch standard frequently to correct for instrument drift. A minimum of five comparisons of each sample to the producer's batch standard is made. The goal of the certification model is to minimize the measurement uncertainty.

4.3 Producer's Data Validation and Batch Value Assignment

The NIST concentration value for the producer's batch standard will be compared to the concentration determined by the producer. Since the producer is not responsible for final concentration value assignment, the producer's value need not be the same as NIST's, but it should be close. A factor (F) is determined using the equation;

$$F = C_{NIST} / C_{Producer}$$

where C_{NIST} is the concentration of the batch standard as determined by NIST, and $C_{Producer}$ is the concentration of the batch standard as determined by the producer. This factor is used to adjust the producer's analysis of the NTRM batch to the NIST analysis. If this factor is 1.000 ± 0.005, certification can proceed and the producer's data can be validated. If the factor deviates from unity more than 0.5 % the certification may proceed but NIST will work with the producer to identify the reason for the deviation. If, after reconciliation between NIST and the producer, the factor still deviates from unity more than 0.5 % the batch will be rejected.

NIST will also examine the data to determine if the ratio of the concentration values between the selected cylinders and the batch standard are the same as determined by both NIST and the producer. If the ratios determined by NIST are the same (within measurement uncertainty) as that determined by the producer, then the producer's data can be considered valid and certification can proceed. However, if the ratios do not match within measurement uncertainty the NTRM batch will be rejected.

Once the producer's data have been validated, NIST will examine the data for overall batch homogeneity. If the producer's data show that the batch is homogeneous and stable the batch can be certified. If the batch shows a percent relative standard deviation in the concentration of greater than 0.5 %, the batch will be rejected. A possible exception can be made where homogeneous batches are difficult to prepare due to the chemical nature of particular analytes.

If the batch is found to be homogeneous, and the producer's data have been validated, then NIST will proceed to assign a concentration value. NIST first adjusts the producer's homogeneity data using factor F so each cylinder's concentration value reflects NIST's value assignment of the batch standard. The final certified concentration value for the batch is the average of the producer's NIST adjusted homogeneity data.

4.4 Value Uncertainty

The uncertainty [Ref. 1] of the NTRM concentration value is estimated using a three-component additive analysis of variance model. The components are: 1) the assigned uncertainty of the standards used in the NIST analysis; 2) the measurement uncertainty of the NIST analysis of the NTRM batch standard; and, 3) the batch homogeneity measured by the producer.

A combined uncertainty for the NIST standard, u_{PS}, is derived from the assigned 95 % confidence interval of the standard (U_{PS}) using the appropriate coverage factor (example: an assigned 95 % confidence interval of 1.5 % would yield a combined uncertainty of 0.75 %, using a coverage factor of 2). A measurement combined uncertainty, u_A, is established from analytical experience and replicate measurements. A batch homogeneity combined uncertainty, u_H, is established from the producer's analysis of all the cylinders in the batch.

This model is used when an NTRM that is close in concentration to a NIST primary standard and the concentration value assignment can be accomplished by direct comparison. In the case where the NTRM concentration falls between that of two NIST primary standards and its concentration is determined by interpolation, u_{PS} is larger than just the combined uncertainty of one primary standard since more than one standard is used in the analyses.

The combined uncertainty of the NTRM batch is estimated from the experimental combined uncertainties and is calculated using the following equation;

$$u_c = (u_{PS}^2 + u_A^2 + u_H^2)^{½}$$

The NTRM expanded uncertainty U is calculated using the following equation;

$$U = ku_c$$

with the coverage factor k being equal to 2. The true concentration is asserted to lie within the interval defined by the batch certified value ± U with a level of confidence of approximately 95 percent [Ref. 1].

4.5 NTRM Documentation

After the certification process is completed, the NTRM cylinders are returned to the producer and NIST generates a Report of Analysis (ROA), which is issued to the producer. The ROA contains; 1) the NIST-assigned NTRM batch concentration value and it's uncertainty; 2) the certification period; 3) a description of the NIST value assignment procedure including the determination of the concentration factor (F); 4) a description of the NIST standard(s) used; 5) a listing of the NTRM samples analyzed by NIST along with the NIST and producer's concentration values; and, 6) a listing of all the certified cylinders in the NTRM batch and the concentration value for each as calculated by NIST.

Certificates of Traceability and cylinder labels are also prepared and sent to the producer. Each Certificate of Traceability displays the NTRM logo, the batch identification number, the concentration and uncertainty, and the certification period. The cylinder labels also display the NTRM logo, the NTRM batch identification number, and the batch concentration value and uncertainty.

4.6 NTRM Rejection Criteria

NIST will reject an NTRM for any one of the following reasons: 1) impurity concentrations are too high; 2) the concentration factor (F) diverges from unity more than 0.5 % after reconciliation between NIST and the producer; 3) the producer's and/or NIST's homogeneity data shows a concentration spread > 0.5 % relative; and, 4) NIST's concentration ratios to the batch standard do not agree within measurement uncertainty with the producer's concentration ratios. In all these cases NIST will work with the producer to resolve the problem for future NTRM work. NIST may reconsider the certification of a rejected batch if additional analytical work is performed by the producer and the batch resubmitted for certification.

5. MAINTENANCE OF NTRM BATCHES

5.1 Batch Recertification

An NTRM batch is certified from the date of the NIST Report of Analysis for a specified period of time, defined as the ***certification period***. When that time period expires, all cylinders comprising that NTRM batch are no longer certified. Customers who purchase an NTRM from a producer may receive a certification for the entire certification period commencing from the date of sale. However, if the NTRM batch expires and subsequently loses certification, the customer's NTRM cylinder likewise loses certification. Thus it is very important that the producer maintains the NTRM batch certification status for the projected lifetime of the individual cylinder mixtures.

Batch recertification is the process the producer must follow in order to maintain certification of the NTRM. The purpose of this process is to verify batch concentration and to assure continuing stability of the gas mixture. To accomplished this, a cylinder(s) that is representative of the batch must be analyzed using a NIST certified standard. Two methods to accomplish this are; 1) if the producer has retained the batch standard, and the batch standard was a member of the original batch production, then the batch standard may be analyzed as the representative of the batch; or, 2) a minimum of two cylinders from the batch must be analyzed. If the producer no longer has the two required cylinders, due to distribution to customers, the cylinders may have to be recalled for analysis. Also, it is recommended that the entire stock of cylinders from the batch that are still under the producer's control be analyzed.

Analysis of the batch standard, or the two representative cylinders, must be accomplished using the same procedure originally used by the producer. A NIST certified standard must be used to assign concentrations to these cylinders. The data generated by this analysis must then be sent to NIST. NIST will review the data and determine if the certification period should be extended. Usually if the data from the analysis match the original data within measurement uncertainty, or within half of the stated uncertainty from the original certified concentration, the certification period will be extended and a new certificate issued. NIST reserves the right to recall cylinders (usually the batch standard) from the producer for analysis by NIST. If the batch fails to be recertified, the producer must contact customers in possession of the NTRM cylinders. NIST reserves the right to request a listing of the disposition of all cylinders in the NTRM batch.

The recommended method of handling recertification of the NTRM batch is to analyze, prior to shipment, a customer's NTRM cylinder and the batch standard against a NIST certified standard. If the data continues to show good stability for the batch, e.g., the concentration is within half the stated uncertainty of the original certified value, the cylinder can be shipped. When the batch certification period expires, these data can be supplied to NIST as evidence for recertification. This approach is best used when the entire batch is expected to be sold or used prior to the expiration date. NIST will use the last analysis date as the basis for the extended certification period.

5.2 Customer NTRM Cylinders

A customer who purchases an NTRM cylinder from a producer receives a certificate with a certification period commencing from the date of sale and extending over a set period of time. The length of this period of time is identical with the original batch certification period. As outlined in section 5.1, the certification period is linked with the batch certification, which is the responsibility of the producer to

maintain. Regardless of the batch certification period, the customer's certification expires at the end of the date of sale certification period. The customer must then contact the producer about recertification of their cylinder.

5.3 Customer NTRM Recertification

The producer is responsible for the recertification analyses of NTRM cylinders sold to its customers. The recertification analysis is accomplished in one of three ways; 1) against the original batch standard used to maintain the batch certification; 2) against a batch standard from a batch similar in composition, concentration and uncertainty that is currently still in certification; and, 3) against a NIST certified standard that is similar in composition and concentration. The analysis should provide at least three data points against the designated standard and demonstrate an instrumental variance well within the original stated uncertainty.

The data must be submitted to NIST which will give the final approval for recertification. NIST reserves the right to analyze the cylinder before approval is granted. If the submitted concentration is within the original certified concentration interval, recertification will usually be granted. If the submitted concentration is outside the original certified concentration uncertainty interval, recertification will be granted under the following conditions; 1) there is a known cause for the discrepancy that NIST acknowledges, and it does not impact on the usability of the standard; 2) impurities introduced into the cylinder by the customer has not compromised the standard; and, 3) the producer can demonstrate, through calibration data, that its analytical system is capable of reliably assigning a concentration distant from the original concentration.

Once notified that the recertification is granted, the producer can issue to the customer written notification of the certification status. Certification will be extended for the current certification period from the date of the recertification report.

6. TRACEABILITY TO NIST

6.1 NTRM Batch Traceability

The traceability of a gas NTRM batch relies on the unbroken chain of comparisons to NIST's primary gas standards. Through gravimetric preparation of the primary gas standards, NIST assures traceability to the mole, a basic unit of the International System of Units, known as SI. Since the beginning of the NIST gas standards program, the NIST primary gas standards have been the foundation of the gas SRM program. They are prepared and maintained by NIST, and regularly undergo stringent internal evaluation and intercomparisons with other international gas standards. In the national hierarchy of gas standards, they are at the pinnacle. The NIST certified standards, such as gas SRMs, are compared to these primary gas standards before certification. These NIST certified standards enable producers to prepare and maintain the NTRM batches that are provided to the public or used to produce other NIST traceable gas standards.

There is an unbroken series of analyses comparing NTRM gas mixtures to NIST certified standards, and a direct NIST involvement in the NTRM certification. NIST maintains records of certified NTRMs and certification periods. If an NTRM batch is still in use after the certified period expires, it must be reanalyzed and the data submitted to NIST for concurrence concerning stability. NIST reserves the right to request any NTRM cylinder be sent to NIST for analysis if there are any questions concerning stability or concentration. The direct involvement of NIST in the quality assurance of each NTRM batch should provide continued acceptability of the certified concentrations with time.

6.2 Mixtures Produced Referencing NTRMs and SRMs

Not all of the analytical activities of gas analysis laboratories require the stringent traceability of an SRM or NTRM. In these instances a gas mixture with a somewhat lower level of traceability may be all that is required. Gas mixtures prepared using a protocol referencing gas SRMs or NTRMs may be declared as NIST traceable if the general guidelines given below are followed. It is important to realize that these guidelines represent the minimum required in order for NIST to recognize a gas mixture as traceable. The minimum requirements for a NIST traceable gas mixture are;

1) The cylinders will be clean, of standard size and construction, and of known compatibility and history with the composition of the proposed standard. The cylinders will be equipped with valves of the appropriate material that conform to Compressed Gas Association (CGA) recommendations for the particular gas mixture. The reagent gases must be of high purity and quality to ensure that impurity levels and mixture stability meets NIST specifications.

2) The concentration of the mixture is within 5 % relative of the SRM or NTRM concentration to which it is related by analysis. The composition of the mixture should be identical to that of the SRM or NTRM. Multicomponent mixtures may be traceable to binary mixtures as long as there are no component interactions, and the balance gas is identical to the referenced mixtures (or chemically inert to the components). Stability must be assured through procedures similar to section 3.5 or demonstrated by data available to customers.

3) All sources of uncertainty in the concentration assignment are included in the final uncertainty statement. The model given in section 4.4 should be followed. The combined uncertainty of the referenced SRM or NTRM must be included in the mixtures expanded uncertainty.

4) An expiration date for the mixture must be given to the customer. The period of time the standard is valid will not exceed the original certification period of the corresponding SRM or NTRM. Example: If the original NTRM was certified for 2 years, the mixture can be given a maximum validation period of 2 years.

5) Documentation provided the customer will include concentration, expanded uncertainty, expiration date, significant impurities, and the specific SRM or NTRM referenced.

An excellent document describing a method to prepare a gas mixture referencing an SRM or NTRM is given in reference 3.

References

1) *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed. ISO, Geneva, Switzerland, 1993. See also *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*, B. N. Taylor and C. E Kuyatt, NIST Technical Note 1297, 1994 Edition

2) *EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards*, U.S. Environmental Protection Agency, EPA-600/R93/224, September 1993

3) *A Procedure for Establishing Traceability of Gas Mixtures to Certain National Institute of Standards and Technology SRM's*, E. E. Hughes and J. Mandel, NBSIR 81-2227 (revised 1989).

4) *International vocabulary of basic and general terms in metrology* (VIM), 1993, issued by BIPM, IEC, ISO, IUPAC, IUPAP and OIML.

5) ISO Guide 30: 1981, *Terms and definitions used in connection with reference materials.* See also ISO 8402, *Quality - Vocabulary,* 1986.

Appendix 1: Definition of Terms

This section provides definitions of terms used in this document. The initial use of each of these terms within this document is signified by ***emphasized*** print. These terms may be defined differently in other documents released by NIST.

Batch: A group of gas mixtures, contained in gas cylinders, prepared by a producer in such a manner that all the gas mixtures are identical in composition and concentration. For the purposes of this program, each batch will contain a minimum of 10 cylinders.

Batch Standard: A gas mixture, similar in concentration and uncertainty to the NTRM batch, to which all cylinders of the batch are related through chemical analysis. The batch standard is usually a randomly chosen member of the NTRM batch. If the batch standard is not a member of the NTRM batch, it's concentration must be within 5 % of the batch nominal concentration.

Certification Period: The period of time a gas mixture is certified for component concentration.

Concentration: The amount-of-substance (SI unit: mole) of the subject compound within the cylinder. This is usually expressed as the amount-of-substance fraction (SI unit: mol/mol).

Homogeneity: A measure of the concentration spread exhibited by an NTRM batch. Usually measured as a percent relative standard deviation from the average concentration.

Homogeneous: Having a concentration spread of less than 0.5 % relative standard deviation.

Impurities: Unwanted chemical compounds within a gas mixture. Their concentration must not be large enough to jeopardize the intended use of the mixture.

Incubation Period: The period of time after production the gas mixtures are required to mature before analyses are performed. This period allows the gas mixture to stabilize to their final concentration, giving a better measure of batch homogeneity.

Inhomogeneous: Having a concentration spread in excess of 0.5 % relative standard deviation.

NIST Certified Standard: Gas mixtures that have been certified by NIST and which are within the valid certification period. Documentation in the form of a NIST Report of Analysis or Certificate must be available for inspection. An SRM is a valid certified standard, however ***Research Gas Mixtures*** (RGM) should be approved by NIST before use in NTRM production.

NIST Primary Standards: Gas mixtures maintained by NIST, which have been prepared gravimetrically at NIST, to which all measurements are ultimately compared.

NIST Traceable Reference Material (NTRM): A reference material produced by a commercial supplier with a well-defined traceability linkage to the National Institute of Standards and Technology (NIST). This linkage is established via criteria and protocols defined by NIST that are tailored to meet the needs of the

metrological community to be served. Reference material producers adhering to these requirements will be allowed the "NTRM" trademark.

Quality System: The organizational structure, responsibilities, procedures, processes and resources for implementing quality management.

Recertification: A process that must be completed in order for the gas mixture's certification period to be extended.

Research Gas Mixture (RGM): A gas mixture produced cooperatively by NIST and a producer. The gas mixture's concentration must be outside a current NIST Primary Standard suite or has a composition not currently supported by NIST.

Resolution: An instrument's ability to detect small changes in signal and report this change in the output data stream.

Sample Number: A sequential number assigned to the cylinders of an NTRM. The sample numbers span from one through the number of cylinders in the batch. They are permanently associated with the cylinders throughout the lifetime of the NTRM.

Selected Cylinders: Cylinders from the NTRM batch which are selected by NIST to be sent by the producer to NIST for value assignment.

Sensitivity: An instrument's ability to distinguish a true analytical signal from background noise.

SRM Lot Standard: The batch standard used by NIST to certify an SRM lot. The lot standard is also used to track the stability of the SRM lot.

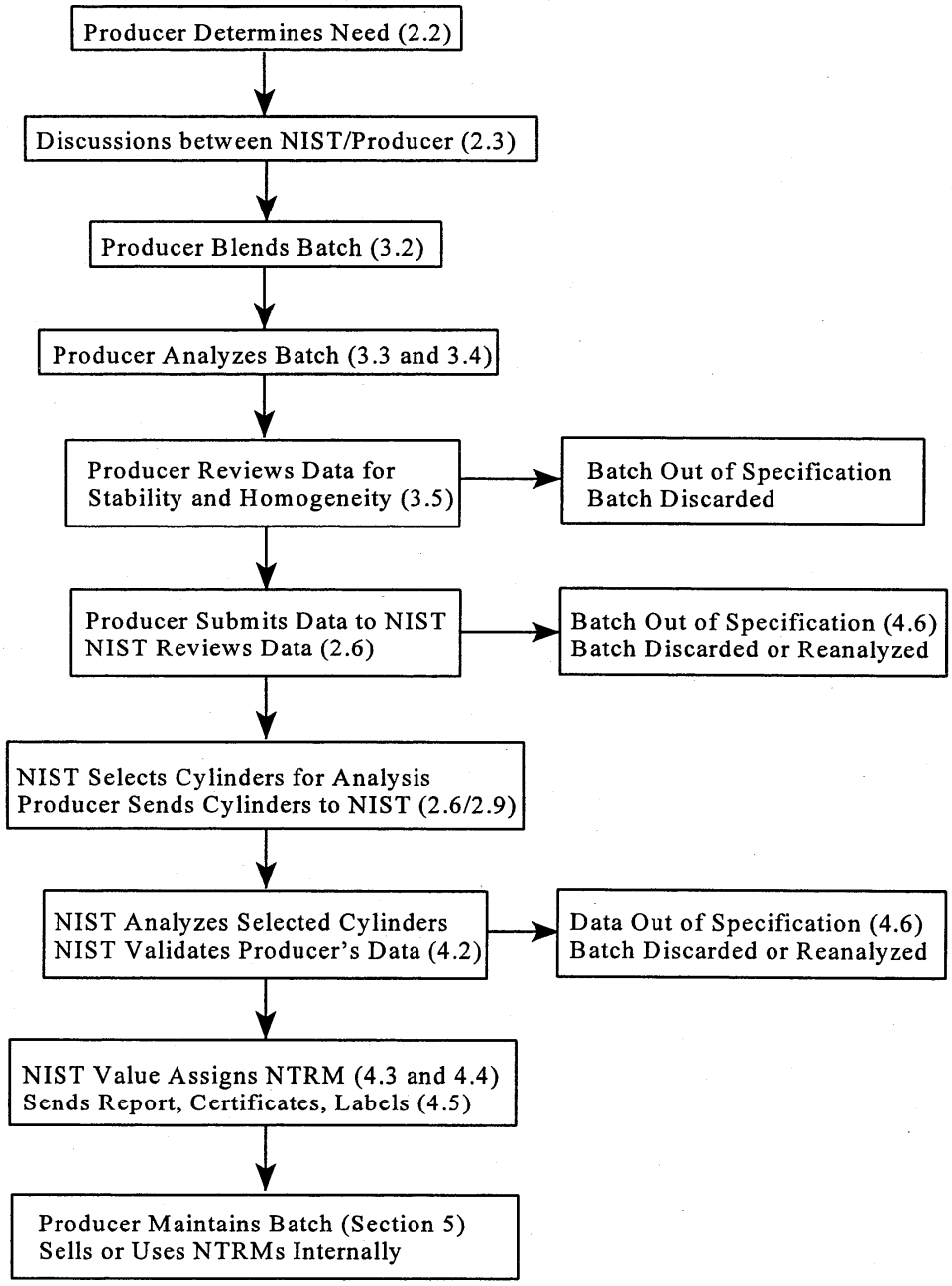
Stability: A measure of the concentration drift of a gas mixture over time. A gas mixture is stable if the concentration drift is small relative to the absolute concentration over time. For NTRMs the concentration drift must be within half of the stated uncertainty from the certified concentration over the entire certification period.

Standard Reference Material™ (SRM): Certified reference materials issued by NIST. These are well-characterized materials produced in quantity to improve measurement science. SRMs are certified for specific chemical or physical properties, and are issued by NIST with certificates that report the results of the characterization and indicate the intended use of the material.

Traceability: The property of a result of a measurement whereby it can be related to appropriate standards, generally national or international standards, through an unbroken chain of comparisons [Ref. 4,5].

Uncertainty: A parameter, associated with concentration, that characterizes the dispersion of the values that attributed to the concentration assignment. A reported concentration value must have an associated uncertainty [Ref. 1,4,5].

Appendix 2: Production and Certification Flow Chart
(With Sections Referenced)



Appendix 3: NTRM Specifications

A partial listing of some primary gas standard suites which may bound NTRMs:

Mixture Components	Amount of Substance Range
Carbon Monoxide in Nitrogen	10 μmol/mol to 13 % mol/mol
Carbon Monoxide in Air	10 μmol/mol to 45 μmol/mol
Carbon Dioxide in Nitrogen	0.5 % mol/mol to 20 % mol/mol
Propane in Air	3 μmol/mol to 500 μmol/mol
Propane in Nitrogen	100 μmol/mol to 4 % mol/mol
Methane in Air	1 μmol/mol to 100 μmol/mol
Oxygen in Nitrogen	2 % mol/mol to 21 % mol/mol
Nitric Oxide in Nitrogen	5 μmol/mol to 3000 μmol/mol
Sulfur Dioxide in Nitrogen	50 μmol/mol to 3500 μmol/mol
Volatile Organics	Contact NIST

This list is not complete, any questions concerning other constituents and/or concentrations can be answered by contacting NIST.

Minimum incubation period before first analysis:

Two days for CO, CO_2, C_3H_8, CH_4 and O_2 then 30 days between the first and second analyses.
Four days for NO and SO_2 then 30 days between the first and second analyses.

Maximum allowable impurity levels:

Carbon Monoxide Mixtures	
Water vapor	5 μmol/mol
Carbon dioxide	1 μmol/mol or 0.1 % of the CO value whichever is greater
Methane	0.1 % of the CO value
Hydrocarbons	0.1 μmol/mol as methane
Carbon Dioxide Mixtures	
Water vapor	5 μmol/mol
Carbon monoxide	0.01 % of CO_2 value
Nitrous oxide	0.1 % of CO_2 value
Hydrocarbons	0.1 μmol/mol as methane
Propane Mixtures	
Water vapor	5 μmol/mol
Other hydrocarbons	0.4 % of the propane value expressed as propane
Methane Mixtures	
Water vapor	5 μmol/mol
Other hydrocarbons	0.5 % of methane value

Oxygen Mixtures

Water vapor	5 μmol/mol
Hydrocarbons as methane	3 μmol/mol
Argon in 2 % mol/mol oxygen	30 μmol/mol
Argon in 10 % mol/mol oxygen	50 μmol/mol
Argon in 21 % mol/mol oxygen	100 μmol/mol
Total other impurities	15 μmol/mol

Nitric Oxide Mixtures

Water vapor	5 μmol/mol
Nitrogen Dioxide	1 % of NO value (all batch cylinders analyzed)
Hydrocarbons	2 μmol/mol expressed as methane

Sulfur Dioxide Mixtures

Water vapor	5 μmol/mol
Hydrocarbons	2 μmol/mol expressed as methane

Appendix 4: Data Submission Form

The following form may be used to report the producer's NTRM information and analysis results to NIST. This form is available from NIST on a floppy disk.

FORM A - Producer's Report of Analysis of Candidate NTRM

Report Prepared by:

Date:
Company Name:
Address:

Phone and FAX #:
Individual preparing report:

Description:

NTRM Composition:

Corresponding SRM No.:

Date Blended:

Blending Location:
(Address, Phone, FAX and Contact Person)

Blending Method:

Manifold fill position diagram attached upon request:

Cylinders:

Size:

Material:

Manufacturer:

Treatment Process:

Previous Use:

Valve:

CGA #:

Straight or Tapered Threads:

Manufacturer:

Form A (continued)

NIST Certified Standards Used as Calibrants:

No.	Cylinder No.	(If SRM) Sample No.	Concentration and uncertainty	Certification Date
1.				
2.				
3.				
4.				

Brief Description of Analytical Method:

Form A (continued)

Instrument Calibration Procedure:

Calibration Date:

Analytical Sequence (order in which cylinders were analyzed):

No.	Calibrant	Concentration	Instrument Response
1.			
2.			
3.			
4.			
5.			
etc.			

Analysis of NTRM Batch Standard:

Analysis Date:

Batch Standard Description:

Analytical Sequence (order in which cylinders were analyzed):

No.	Cylinder or Calibrant	Instrument Response	Calculated Concentration
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
		Average =	_____
		Std. Dev. =	
		% Relative SD =	

Form A (continued)

Analysis of NTRM Cylinders against Batch Standard:

Date:

Analytical Sequence (order in which cylinders were analyzed)

Sample No.	Cylinder Number	Calculated Concentration
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		
18		
19		
20		
etc.		

Average =
Std. Dev. =
% Relative SD =

Impurity Analyses:

	Impurity	Concentration	Analytical Method
1			
2			
3			
4			