WRI 09-R024

MERCURY CONTINUOUS EMMISSION MONITOR CALIBRATION

TOPICAL REPORT

Start Date: 2005
End Date: 2009

By
John F. Schabron
Eric W. Kalberer
Ryan B. Boysen
William C. Schuster
Joseph F. Rovani

April 30, 2009

Work Performed Under Cooperative Agreement
Task 62 under DE-FC26-98FT40323

For
Electric Power Research Institute
Palo Alto, California

And
U.S. Department of Energy
Office of Fossil Energy
National Energy Technology Laboratory
Morgantown, West Virginia

By
Western Research Institute
Laramie, Wyoming

Kamalandu Das
Task 62

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ACKNOWLEDGMENTS

Funding for this study was provided by the U.S. Department of Energy under Cooperative Agreement DE-FC26-98FT40323, and by the Electric Power Research Institute.

DISCLAIMER

ABSTRACT

Mercury continuous emissions monitoring systems (CEMs) are being implemented in over 800 coal-fired power plant stacks throughput the U.S. Western Research Institute (WRI) is working closely with the Electric Power Research Institute (EPRI), the National Institute of Standards and Technology (NIST), and the Environmental Protection Agency (EPA) to facilitate the development of the experimental criteria for a NIST traceability protocol for dynamic elemental mercury vapor calibrators / generators. These devices are used to calibrate mercury CEMs at power plant sites. The Clean Air Mercury Rule (CAMR) which was published in the Federal Register on May 18, 2005 and vacated by a Federal appeals court in early 2008 required that calibration be performed with NIST-traceable standards. Despite the vacature, mercury emissions regulations in the future will require NIST traceable calibration standards, and EPA does not want to interrupt the effort towards developing NIST traceability protocols. The traceability procedures will be defined by EPA. An initial draft traceability protocol was issued by EPA in May 2007 for comment. In August 2007, EPA issued a conceptual interim traceability protocol for elemental mercury calibrators. The protocol is based on the actual analysis of the output of each calibration unit at several concentration levels ranging initially from about 2-40 $\mu g/m^3$ elemental mercury, and in the future down to 0.2 $\mu g/m^3$, and this analysis will be directly traceable to analyses by NIST. The EPA traceability protocol document is divided into two separate sections. The first deals with the qualification of calibrator models by the vendors for use in mercury CEM calibration. The second describes the procedure that the vendors must use to certify the calibrators that meet the qualification specifications. The NIST traceable certification is performance based, traceable to analysis using isotope dilution inductively coupled plasma / mass spectrometry (ID/ICP/MS) performed by NIST in Gaithersburg, MD. The outputs of mercury calibrators are compared to one another using a nesting procedure which allows direct comparison of one calibrator with another at specific concentrations and eliminates analyzer variability effects.

The qualification portion of the EPA interim traceability protocol requires the vendors to define calibrator performance as affected by variables such as pressure, temperature, line voltage, and shipping. In 2007 WRI developed and conducted a series of simplified qualification experiments to determine actual calibrator performance related to the variables defined in the qualification portion of the interim protocol.

EXECUTIVE SUMMARY

Mercury continuous emissions monitoring systems (CEMs) are being implemented in over 800 coal-fired power plant stacks throughput the U.S. Western Research Institute (WRI) is working closely with the Electric Power Research Institute (EPRI), the National Institute of Standards and Technology (NIST), and the Environmental Protection Agency (EPA) to facilitate the development of the experimental criteria for a NIST traceability protocol for dynamic elemental mercury vapor calibrators / generators. These devices are used to calibrate mercury CEMs at power plant sites. The Clean Air Mercury Rule (CAMR) which was published in the Federal Register on May 18, 2005 and vacated by a Federal appeals court in early 2008 required that calibration be performed with NIST-traceable standards. Despite the vacature, mercury emissions regulations in the future will require NIST traceable calibration standards, and EPA does not want to interrupt the effort towards developing NIST traceability protocols. The traceability procedures will be defined by EPA. An initial draft traceability protocol was issued by EPA in May 2007 for comment. In August 2007, EPA issued a conceptual interim traceability protocol for elemental mercury calibrators. The protocol is based on the actual analysis of the output of each calibration unit at several concentration levels ranging initially from about 2-40 $\mu g/m^3$ elemental mercury, and in the future down to 0.2 $\mu g/m^3$, and this analysis will be directly traceable to analyses by NIST. The EPA traceability protocol document is divided into two separate sections. The first deals with the qualification of calibrator models by the vendors for use in mercury CEM calibration. The second describes the procedure that the vendors must use to certify the calibrators that meet the qualification specifications. The NIST traceable certification is performance based, traceable to analysis using isotope dilution inductively coupled plasma / mass spectrometry (ID/ICP/MS) performed by NIST in Gaithersburg, MD. The outputs of mercury calibrators are compared to one another using a nesting procedure which allows direct comparison of one calibrator with another at specific concentrations and eliminates analyzer variability effects.

The qualification portion of the EPA interim traceability protocol requires the vendors to define calibrator performance as affected by variables such as pressure, temperature, line voltage, and shipping. In 2007 WRI developed and conducted a series of simplified qualification experiments to determine actual calibrator performance related to the variables defined in the qualification portion of the interim protocol.

In December 2007, EPA held a meeting at Research Triangle Park (RTP), NC to initiate a plan for 2008 activities for the newly formed "Mercury Standards Working Committee" project. Participants include EPA, EPRI, NIST, WRI, power plants, calibrator and gas cylinder vendors, and consultants. The ultimate goal of the effort is to provide experimental data to help develop reasonable and technically sound protocols for certification and verification of calibrator output, the use of gas cylinders, and oxidized mercury vapor generators to "inform the process"

of preparing the next iteration of the EPA NIST traceability protocol. The committee's activities have been divided into six tasks. These are summarized below:

Task 1. Evaluation of calibrator field certification procedures. This is being coordinated by RMB consultants and it involves the participation of approximately 25 stack sites at power plants. RMB is also maintaining a portion of their web site devoted to uploading and downloading protocols and experimental data for the various tasks in the overall effort.

Task 2. Intra and interlaboratory evaluations of calibrator output comparison procedures. This is expected to provide best case data for comparison with the Task 1 field data. The experimental evaluations are to be performed primarily by EPA (RTP) and WRI.

Task 3. Evaluate and recommend procedures and uses for elemental mercury vapor calibration gas cylinders.

Task 4. Develop the statistical procedures for presenting and evaluation data.

Task 5. Evaluate various approaches for field verification of calibrator output and acceptable performance.

Task 6. Develop procedures and acceptability criteria for the use and verification of oxidized mercury vapor generators.

The current report describes the work performed to date by WRI on intralaboratory comparison of calibrators under Task 2 to develop "best case" data using 3x3 nesting analysis comparisons (three concentrations, three measurements at each concentration for a particular concentration span). Some work with gas cylinders under Task 3 is reported also.

Transfer of certification traceability from NIST involves comparing the output of the NIST Prime calibrator at NIST at specific concentration values for a Vendor Prime calibrator using the nesting procedures, and then comparing the Vendor Prime calibrator output to a Candidate / User calibrator output at the same concentration settings using the nesting procedures. Output of the NIST Prime calibrator at the specific concentrations has been established by NIST analysis using ID/ICP/MS. Once the concentration values at the specific NIST set point concentrations are established for a Candidate / User calibrator, it can be used to calibrate a spectrometer analyzer.

EPA has authority and discretion to specify how NIST traceability is defined for a Candidate / User calibrator in the traceability protocol. An analyzer which is calibrated can be used to determine the mercury concentration at any concentration within the NIST Traceable certified calibration range. Since it has been established that both calibrator output and spectrometer responses are highly linear, the spectrometer is typically calibrated or spanned between zero and a calibration point(s) required by the user. WRI is suggesting that a technically sound approach for certifying a User Prime Candidate calibrator would be to transfer certification at specific concentration points directly traceable to NIST analyses, and then to define a line equation for the User unit in the full concentration span range of interest. In this manner the spectrometer can be calibrated using the linear equation at any desired point(s) within the calibration range.

Data from duplicate 3x3 nesting sequences between two calibrators show minimal output ratio variations for measurements made over several days. Using the highest quality nitrogen or air possible is absolutely necessary for operating these calibrators to minimize elemental mercury source passivation. One issue of concern is to define a recommendation for the required frequency of recertification of the calibrators. It seems that an arbitrary recertification time interval could result in unnecessary efforts and associated costs. An arbitrary time interval also may miss units that are not operating properly between certifications. Instead, the frequent application of a simple, quick and reliable quality control procedure that can identify deterioration of performance could be used as the criterion that triggers a recertification event. In the current work, a draft simple and rapid quantitative method was developed to verify that calibrator output has not changed over time. When a calibrator is new, it provides a highly linear output through the origin. Changes deviating from the original linearity profile indicate that that then unit needs attention and recertification. The proposed procedure involves measuring the calibrator output at two concentrations and calculating an output ratio, which is compared to the theoretical concentration output ratio. This can be performed with a single calibrator unit, and does not require bracketing against another unit. This can be the basis for a QC chart to monitor the ratio over tine, or it can be used in cases where nesting ratios change >5 % over time to determine which calibrator has changed. We suggested that this or a similar procedure be implemented as part of the traceability protocol. In early 2009 this QC check procedure was incorporated into the draft 2009 interim traceability protocol as section 7.2.2 by EPA.

Results from nesting studies with elemental mercury calibration gas cylinders against a Reference elemental calibrator unit were consistent with results from the work done previously by RMB in which they also observed that the actual output of the mercury gas cylinders does not appear to be stable. Passivated regulators required more time initially to provide a level output than when they were used in subsequent experiments.

OBJECTIVES

A main objective of the current work is to provide data on the performance and capabilities of elemental mercury calibrators for the development of realistic NIST traceability protocols for mercury vapor standards for CEM calibration, and to generate example best case laboratory data to demonstrate the practical utility and limitation of the nesting procedures used for calibrator certification. This work is providing a direct contribution to the enablement of continuous emissions monitoring at coal-fired power plants. The purpose of the current phase of this project is to generate data to "inform the process" of preparation of the next version of the interim NIST traceability protocol by EPA to be issued in 2009.

INTRODUCTION

WRI is working closely with NIST and EPA to facilitate the development of technically sound, yet practical experimental procedures for EPA to incorporate into a new traceability protocol. The WRI analytical group has unique, specialized capabilities in analytical method development and validation that are being applied to this effort. The purpose of the current phase of this project is to evaluate issues related to the use of dynamic elemental mercury calibrators that are based on mercury vapor headspace above elemental mercury.

CEM Calibration and NIST Traceability

EPA Specification 12 was issued on May 18, 2005 as part of the Clean Air Mercury Rule (CAMR), which was vacated in 2008. It states that mercury CEMs must be calibrated with NIST-traceable standards (Federal Register 2005). To work towards meeting this requirement, an initial draft of an elemental mercury generator traceability protocol was circulated by EPA in May 2007 for comment, and a broad-based conceptual interim protocol was issued in August 2007 (EPA 2007). Initially it was assumed that the calibration and implementation of mercury CEMs would be relatively simple, and implementation would follow the implementation of the Clean Air Interstate Rule (CAIR) for SO_2 and NO_X monitoring, and sulfur emissions cap and trade. However, mercury emissions have proven to be significantly more difficult to accurately determine than was originally thought. Although both CAMR and CAIR were vacated by court rulings in 2008, EPA has indicated that since the requirement for NIST traceable standards will be a part of any new rule, the development of a NIST traceability protocol for mercury CEMs should continue uninterrupted during this interim period.

Calibration techniques for CEMs that have been proposed include calibration gas cylinders, mercury vapor permeation tubes, mercury diffusion tubes, and elemental mercury vapor pressure generators. The latter technology has now been accepted as the most viable approach for power plant deployment for long term use. This technology uses saturated headspace devices that contain a small amount of liquid elemental mercury in a temperature-controlled vessel, or equilibrium chamber. This technology has the possibility of generating large quantities of calibration gas over extended periods of time for CEM calibration. By precisely and accurately controlling and or measuring the equilibrium chamber temperature, a controlled concentration of mercury in nitrogen or air can in theory be generated. Each vendor has developed a unique design for their elemental mercury calibrator unit. Typically, a low flow mass flow controller (cc/min) is used to sweep an equilibrium chamber with air or nitrogen. This is diluted further using a relatively high flow mass flow controller (L/min) to provide a diluted vapor calibration stream of known concentration. In early 2008, there were two primary vendors in the market place for mercury CEMs for coal-fired power plants in the U.S.: Tekran and Thermo Fisher. The focus of the current work was with calibrators from these two vendors. Other vendors offering calibration devices based on this principle include Cemtrex / Mercury-Instruments, Nippon, and PSA.

Prior Work

Prior to this project, NIST did not provide a recommended mercury vapor pressure equation or list mercury vapor pressure in its vapor pressure database. The NIST Physical and Chemical Properties Division in Boulder, Colorado was subcontracted under this project in 2005 to study the issue in detail and to recommend a mercury vapor pressure equation that the vendors of mercury vapor pressure calibration units can use to calculate the theoretical elemental mercury vapor concentration in an equilibrium chamber at a particular temperature. A NIST recommended equation was developed and published in the peer-reviewed literature as a result of this work (Huber et al. 2006a, 2006b). This equation is used to calculate the vapor pressure of liquid elemental mercury from the triple point to the boiling point, and provides a vapor pressure that is consistent with the Clausius/Clapeyron thermodynamic equation. The new NIST equation is different from some other equations in use, and it provides a vapor pressure at 20 ºC that is about 7% higher than the mercury vapor pressure listed in the 1928 International Critical Tables. The new NIST equation is cited as the basis for the mercury vapor pressure table in the 2007-2008 CRC Handbook of Chemistry and Physics (CRC 2007).

There continues to be disagreement among the vendors on the correct vapor pressure equation for use in the calibration units. In addition, identical units from the same vendor do not necessarily provide identical concentrations at the same settings (Schabron et al. 2007, Schabron and Rovani 2008). Because of this, it was agreed in a special meeting held in Orlando, FL in March 16, 2006 that the NIST traceability of calibration units would be performance based. Each NIST certified concentration will be a value directly traceable to an analysis by NIST using ID/ICP/MS. The chain of traceable analyses must be unbroken. To be legally defined as traceable for mercury monitoring, the procedure used to produce data for the unbroken chain by the vendors in their facilities at power plant sites must be provided by EPA in the form of a NIST Traceability Protocol document. This approach assumes that the calibrator output remains stable over time. In December 2007, EPA held a meeting at Research Triangle Park, NC to initiate a plan for 2008 activities for the Mercury Standards Working Committee project. Participants include EPA, EPRI, NIST, WRI, power plants, analyzer/calibrator and gas cylinder vendors, and consultants. The ultimate goal of the effort is to provide experimental data to help develop reasonable and technically sound protocols for certification and verification of calibrator output, the use of gas cylinders, and oxidized mercury vapor generators for the next iteration of the EPA NIST traceability protocol. The committee's activities have been divided into six tasks. These are summarized below:

Task 1. Evaluation of calibrator field certification and procedures. This is being coordinated by RMB consultants and it involves the participation of approximately 25 stack sites at power plants. RMB is also maintaining a portion of their web site devoted to uploading and downloading protocols and experimental data for the various tasks in the overall effort.

Task 2. Intra and interlaboratory evaluations of calibrator output comparison procedures. This is expected to provide best case data for comparison with the Task 1 field data. The bulk of the experimental evaluations are to be performed by EPA (RTP) and WRI.

Task 3. Evaluate and recommend procedures and uses for elemental mercury vapor calibration gas cylinders.

Task 4. Develop the statistical procedures for presenting and evaluation data.

Task 5. Evaluate various approaches for field verification of calibrator output.

Task 6. Develop procedures and acceptability criteria for the use and verification of oxidized mercury vapor generators.

The main thrust of the current effort, is to provide EPA with extensive data to inform the process of developing a technically sound and viable traceability protocol document. The current report describes the work performed to date by WRI in Task 2 to conduct intralaboratory and intralaboratory calibrator comparisons to develop best case laboratory data on the generators using nesting analyses. In addition, WRI conducted experiments under Task 3 to evaluate the possible use of elemental mercury calibration gas cylinders for field verification of elemental mercury calibrator output.

EXPERIMENTAL

Calibrators

For the Task 2 effort at WRI, two calibrators from Tekran and Three calibrators from Thermo Fisher were used. The WRI Thermo model 81i mercury calibrators are named Thermo 02 (serial # 0613917136), Thermo 03 (serial # 0636220561), and Thermo 04 (serial # 0618117699). The WRI Tekran model 3310 calibrators are designated Tekran 03 (serial #

70810307), and Tekran 04 (serial # 70827161). Manufacturer-prescribed operating instructions were followed for the use of each of these devices.

Tekran 03, Thermo 02, and Thermo 04 were designated as Candidate generators that were used in the initial bracketing comparisons with Thermo 03 as a Reference generator in August - September 2008. Thermo 03 was a Reference unit shipped to NIST in early 2008 for certification as a Vendor Prime. Due to technical difficulties, NIST was unable to provide certified values for the three analysis points they used: 2.7, 5.7, and 8.1 µg/m^3. Therefore, for purpose of the comparison work in this study, the assumption was made that the "true" values were equivalent to the Thermo 03 Reference calibrator set points of 2.7, 5.7, and 8.1 µg/m^3. Thermo 02 and Thermo 04 calibrators both required the addition of 3-4 g (~ 0.3 mL) of elemental mercury to the inlet flow path in the equilibrium chamber before they worked properly. The Tekran calibrators could not be set at 8.1 µg/m^3 for the high value concentration for 10 µg/m^3 span calibration, so the pre-set high value of 9.6 µg/m^3 was used.

Following the September 2008 bracketing study, Thermo 03 and Thermo 04 calibrator units, which had been used extensively in prior work, required elemental mercury source replacement. It was determined that these two units were not operating the same as before repair, so they were designated Thermo 03a and Thermo 04a for the December 2008 bracketing study. The Tekran 04 unit was received in November 2008 so it was added to the December 2008 bracketing study, during which. Thermo 03a was used as the Reference unit. Additional bracketing sequence studies were conducted in January and February 2009. For the 2009 work, Tekran 04 was used as the Reference generator since Tekran provided it with a certificate of traceability against a Vendor Prime calibrator.

In February 2009 EPA RTP provided two of their calibrator units for evaluation and cross comparison against the WRI Tekran 04 Reference unit. The two EPA units were designated EPA Thermo 02 (serial # 0630018816) and EPA Tekran 05 (serial # 3016). Following the conclusion of the bracketing experiments, these two units were returned to EPA. Photographs of the WRI calibrator evaluation laboratory work station are provided in Figure 1.

Figure 1. WRI Calibrator Evaluation Laboratory

Gas Cylinders

Two mercury-in-air calibration gas cylinders were used in the study. One cylinder was from Airgas (ND26350) with a certified label concentration of 8.670 µg/m^3 +/- 5%. This cylinder was used with an Airgas coated/passivated two-stage mercury gas regulator. The other cylinder was from Spectra Gases (CC266095) with a label concentration value of 9.5 µg/m^3. This cylinder was used with a Spectra Gases coated/passivated two-stage mercury gas regulator. A photograph of the gas cylinders and flow rotameter is provided in Figure 2.

Figure 2. Mercury Calibration Gas Cylinders with Passivated Regulators

Analyzer

A Thermo Fisher Model 80i continuous emission atomic fluorescence elemental mercury analyzer (serial # 0636220551) was used as the primary analyzer for this study. The analyzer was updated in April 2007 with the new Thermo iPort32 software with lamp heater upgrade kit provided by Thermo Fisher. All calibrator and gas cylinder nesting comparison experiments in this study were conducted using air as the source gas.

Air Source

Two Thomas T-2820ST portable oil-free air compressors were used as the air source for the calibrators described in this report. The compressors replaced the use of breathing quality compressed air cylinders used in the past. Each compressor's air outlet was fitted with a regulator, an indicating desiccant chamber which functions as an air dryer, and a 5 micron particulate filter. The effect of the two sources, cylinder vs. compressor on manifold pressure and concentration using a Thermo Fisher 81i calibrator has been evaluated experimentally and found to be insignificant. Fittings were PFA from Swagelok. PFA tubing was PFA –T6-062 3/8-inch i.d. and PFA-T4-062 ¼ - inch i.d. from Swagelok. Following the September 2008 study, additional carbon cleanup cartridges provided by Thermo Fisher were added to the compressed air lines in the laboratory near to the inlets of the Reference and Candidate calibrators as a further precaution against any potential contamination of air from the room air used by the compressors or the compressed air exiting the compressors. When the Tekran units were received, Tekran air dryer and iodated carbon cleanup cartridge manifold trains were installed at each air compressor. For future work, refrigerated air dryers will be installed also to provide the highest possible quality air. Photographs of the two air compressors and the carbon cartridges near the calibrator inlets are provided in Figures 3and 4, respectively.

Figure 3. Oil-free Air Compressors with Tekran Air Purification Manifolds

Figure 4. Thermo Activated Carbon Cartridges Near Calibrator Inlet Fittings

Calibrator Bracketing Comparison Tests

The bracketing sequence at each concentration is shown in Table 1. The calibrator comparison tests were conducted using a bracketing procedure employed by the National Institute of Science and Technology (NIST) whereby traceability is transferred from the NIST Prime calibrator to the Vendor Prime at specific concentration. In all cases, there was no significant drift of zero noted during any of these experiments. A reading for "zero air" was obtained at the beginning and end of each nesting sequence.

Table 1 – Measurement Sequence for Each Concentration

Reference Calibrator	Response 1
Candidate Calibrator	Response 2
Reference Calibrator	Response 3
Candidate Calibrator	Response 4
Reference Calibrator	Response 5
Candidate Calibrator	Response 6
Reference Calibrator	Response 7

This procedure is called a 3x3 nesting process where the Candidate calibrator is bracketed by the Reference calibrator at each of three elemental mercury concentrations with three nested readings at each concentration. The three concentrations used were: 2.7, 5.7, and 8.1 $\mu g/m^3$ when a Thermo calibrator was used as the reference and at 2.7, 5.7, and 9.6 $\mu g/m^3$ when a Tekran unit was used as reference. The nesting ratio between the Reference calibrator and Candidate calibrator is a useful value when tracking relative stability over time. This ratio can be calculated as:

$$R_{Can1} = \text{Response 2} / (\text{Avg Response 1,3})$$
$$R_{Can2} = \text{Response 4} / (\text{Avg Response 3,5})$$
$$R_{Can3} = \text{Response 6} / (\text{Avg Response 5,7})$$

The mean ratio is:

$$R_M = \text{Avg}\,(R_{Can1}, R_{Can2}, R_{Can3})$$

The procedures used are described in detail in Appendix A for using Thermo Fisher equipment and in Appendix B for using Tekran equipment. When a Thermo 81i calibrator is compared with a Tekran 3310 calibrator using a Thermo 80i continuous atomic emission spectrometer, the Thermo procedure described in Appendix A is used for the Thermo calibrator and analyzer, and the Tekran procedure in Appendix B is followed for operation of the Tekran calibrator. For operating a free standing Tekran 3310 calibrator the system is to be run in manual mode, however, one important modification is made to the procedure. The Tekran 3310 does not electronically record output the same way that the Thermo 81i does. That being the case, it is not possible to transfer a comprehensive bulk data set from the Tekran 3310 calibrator to a PC spreadsheet. Therefore, the user is required to record the Calibrator set point and actual output as displayed on the Tekran PC. This information should be included in a chart to be submitted along with the bulk data files of the other instruments involved in the bracketing study.

RESULTS AND DISCUSSION

Initial Calibrator Bracketing Experiments

The results from an initial series of experiments conducted in August – September 2008 using the bracketing procedure are provided below. The response values for each of the data sets are five minute average responses of Candidate and Reference calibrators compared to each other at three concentrations within the 10 $\mu g/m^3$ span range. The calculated nesting ratios of the responses are listed below (Tables 2-4). The responses and nesting ratios are diagnostic of analyzer drift and/or calibrator drift. A changing ratio indicates relative output differences between the calibrators. Analyzer drift would be reflected as absolute response values that change while the ratios remain essentially constant.

The initial bracketing experiments involved comparison of the Reference calibrator Thermo 03 and the Candidate calibrator Thermo 04. These sequences were carried out after leaving both of the calibrators and the analyzer with power on overnight without airflow. Airflow was initiated in the morning of the same day in which the experiments were conducted, between one and six hours prior to collecting data. As can be seen in Table 2 below, the average nesting ratios for all three concentrations indicate the average relative output differences inherent in the two Thermo 81i calibrators.

Variations in relative outputs between the five experiments as reflected by the nesting ratios were observed. The relative standard deviations for the average nested ratios at each concentration below range from 1.17% at low mercury concentration to 2.09% at high concentration. These values are statistically high and they call the precision of the data into question. The data below do not exhibit any specific trend for the drift of the average nested ratio. However, the primary cause for the high relative standard deviation is most likely the amount of time the instruments were provided with air flow prior to measurements of output.

Table 2: Nesting Comparison of Thermo 03 and Thermo 04 (< 8 hours of air flow)

Test Number	Concentration	Avg Nested Ratio	Concentration	Avg Nested Ratio	Concentration	Avg Nested Ratio
118724	8.1 µg/m3	1.01	5.7 µg/m3	0.99	2.7 µg/m3	0.99
118725	8.1µg/m3	1.00	5.7µg/m3	0.99	2.7µg/m3	0.97
118726	8.1µg/m3	1.04	5.7µg/m3	1.02	2.7µg/m3	1.00
118729	8.1µg/m3	1.03	5.7µg/m3	1.01	2.7µg/m3	1.00
118730	8.1µg/m3	1.05	5.7µg/m3	1.02	2.7µg/m3	1.00
Average:		1.03		1.01		0.99
Std. Deviation:		0.0214		0.0151		0.0116
% RSD		2.09		1.50		1.17

The results shown in Table 3 for three sets of comparisons of the calibrators Thermo 03 and Thermo 04 were obtained in a manner similar to those in Table 2. As before, the units were powered up over night. The primary difference in procedure is that experiments conducted for Table 3 included overnight air flow to the instrument which was set at zero concentration for at least 12 hours prior to obtaining any measurements. The data indicate improved stability of the 81i units after extended exposure to air flow. The average nested ratio data in Table 3 all have relative standard deviations of 0.55 - 0.72% at all three concentrations measured; much lower than that found in the first data set above. Not only does this indicate improved performance of the calibrator units, but also a more representative optimal data set for comparison of the two instruments. In this case, it is still apparent from the average nested ratios that there are differences in relative outputs between calibrator units which can be quantified in terms of the average nesting ratios. It is recommended, based on the results presented here, that all calibrator units be powered up and exposed to zero air flow for at least 12 hours prior to use.

Twelve sets of bracketing data obtained in the study with the units powered for a minimum of 12 hours and a minimum 12-hour air flow before measurements are provided in Appendix C. Results are provided for Thermo 04, Thermo 02, and Tekran 03 Candidate calibrators in comparison with the Reference calibrator, Thermo 03. As mentioned previously, for purposes of this study, since NIST certified values were not provided by NIST, the set point concentrations of Thermo 03 were considered to be the actual "certified" values.

Table 3: Nesting Comparison of Thermo 03 and Thermo 04 (> 12 hours of air flow)

Test Number	Concentration	Avg Nested Ratio	Concentration	Avg Nested Ratio	Concentration	Avg Nested Ratio
118737	8.1µg/m3	1.05	5.7µg/m3	1.03	2.7µg/m3	1.02
118738	8.1µg/m3	1.04	5.7µg/m3	1.02	2.7µg/m3	1.01
118739	8.1µg/m3	1.04	5.7µg/m3	1.02	2.7µg/m3	1.00
Average:		1.05		1.02		1.01
Std. Deviation:		0.00689		0.00560		0.00725
% RSD		0.66		0.55		0.72

Additional bracketing studies were conducted using Thermo 02, an additional 81i Candidate calibrator. The units were powered up with zero air flow for a minimum of 12 hours before data were obtained As can be seen in Table 4, the average nesting ratios provide quantitative measurements of the relative difference in output between Thermo 02 and the Thermo 03 reference unit as reflected by the average nesting ratios. The relative standard deviations ranged from 0.65 – 0.94%.

Table 4: Nesting Comparison of Thermo 03 and Thermo 02 (> 12 hours of air flow)

Test Number	Concentration	Avg Nested Ratio	Concentration	Avg Nested Ratio	Concentration	Avg Nested Ratio
118731	8.1µg/m3	0.96	5.7µg/m3	0.97	2.7µg/m3	0.99
118732	8.1µg/m3	0.95	5.7µg/m3	0.97	2.7µg/m3	1.00
118734	8.1µg/m3	0.95	5.7µg/m3	0.96	2.7µg/m3	0.99
118735	8.1µg/m3	0.95	5.7µg/m3	0.96	2.7µg/m3	1.00
118736	8.1µg/m3	0.93	5.7µg/m3	0.95	2.7µg/m3	0.98
Average:		0.95		0.96		0.99
Std. Deviation:		0.00894		0.00641		0.00647
RSD (%)		0.94		0.67		0.65

The final set of bracketing experiments was conducted using the Thermo 03 and Tekran 03 calibrators. The instruments were left with the power on in static mode continuously throughout evaluation. Results from this comparison are shown in Table 5. The Tekran calibration unit was also evaluated at a high concentration of 9.6 µg/m^3 rather than at 8.1 µg/m^3 because the unit had been pre-set at the factory to run at the three concentrations listed in Table 5 for a nominal 10 µg/m^3 analyzer span calibration set. Therefore, the Thermo 80i analyzer was spanned using the Thermo 03 Reference unit at 8.1 µg/m^3 and then the Thermo 03 unit was set at 9.6 µg/m^3 for the initial bracketing comparison. For purposes of this study, the outputs at the 9.6, 8.1, 5.7, and 2.7 µg/m^3 Thermo 03 set points were treated as the actual certified set point concentration values. The nested ratios provide a quantitative measurement of the differences in output between the two calibrators. The Tekran unit stated a higher nominal output from the software for the calculated, desired output. Interestingly, the average nested ratios indicate very good stability at the high and mid concentrations, with rsd values of 0.79% and 0.81%, respectively. An unexpected relative instability was observed at the low concentration, with an rsd of 1.36%. There is no obvious explanation for this deviation at low concentrations from the bulk data. The 5-minute averages for each data point indicate stability in the instruments within each experiment. From the nested ratio data below no trend is observed from a time standpoint.

Table 5: Nesting Comparison of Thermo 03 and Tekran 03 (> 12 hours of air flow)

Test Number	Concentration	Avg Nested Ratio	Concentration	Avg Nested Ratio	Concentration	Avg Nested Ratio
118741	9.6µg/m3	1.04	5.7µg/m3	1.05	2.7µg/m3	1.05
118742	9.6µg/m3	1.05	5.7µg/m3	1.05	2.7µg/m3	1.04
118743	9.6µg/m3	1.06	5.7µg/m3	1.07	2.7µg/m3	1.07
118744	9.6µg/m3	1.06	5.7µg/m3	1.06	2.7µg/m3	1.04
Average:		1.05		1.06		1.05
Std. Deviation:		0.00831		0.00856		0.0143
RSD (%)		0.79		0.81		1.36

Use of Line Equations for Analyzer Calibration

The transfer of traceability from the NIST Prime to a Vendor Prime unit must be point to point using the actual concentrations that were used to analyze output of the NIST Prime unit by ID-ICP/MS. The transfer of traceability from a Vendor Prime Reference calibrator to a Candidate User calibrator is also conducted at these same concentration points. However, the User unit is designed to be used at any concentration within the range of NIST traceable points. Therefore it seems that the optimal way to certify a User Candidate unit is to define a linear equation of actual concentration versus set point concentration, based on the three NIST traceable points for the concentration range of interest. From this equation the actual output of the Candidate calibrator can be calculated when it is set to any specific concentration within the certified range.

As part of the EPA Traceability Protocol, EPA could allow an analyzer to be calibrated using any desired set point concentration value(s) calculated from the line equation, within the range of –30 % from the low value NIST concentration point to +30 % of the high value certified NIST concentration point. This procedure is not unlike a colorimetric analysis, where three NIST traceable colorimetric standard solutions are used to obtain absorbance data to calibrate a spectrometer. The resulting line plot or linear equation is then used to determine the concentration of the analyte in sample materials.

For this approach to be useful, linearity plots of the Candidate concentration readings vs. the Reference concentration readings similar to that provided in Figure 5 need to be made first to ensure that a sufficient linear correlation exists between the Candidate and Reference calibrator. Data from the series of twelve nesting experiments are provided in Appendix C. The data are summarized in Tables 3-5 and the results are discussed below.

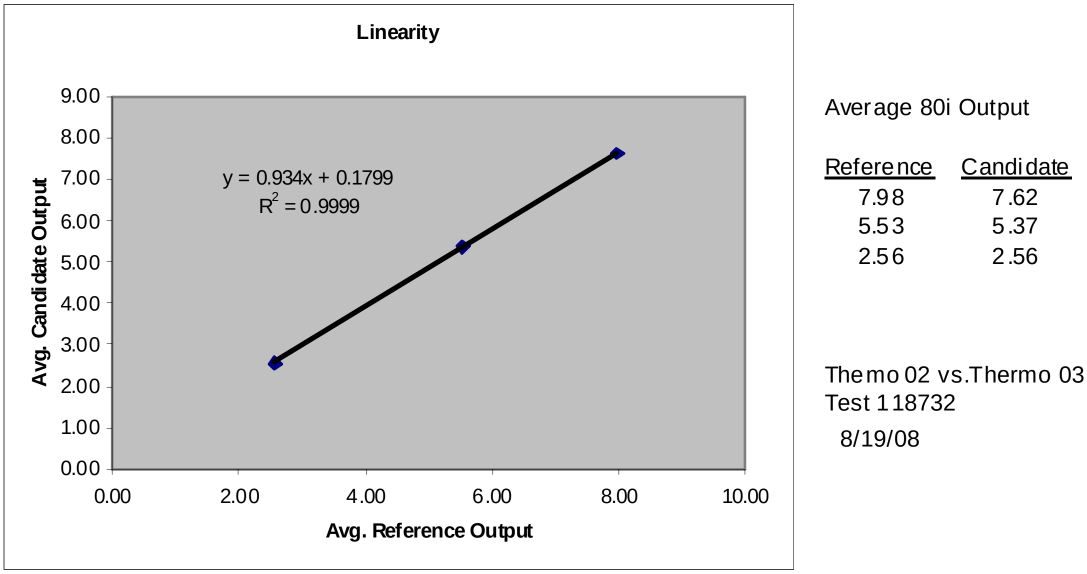

Figure 5: Linearity Plot of Analyzer Readings for Bracketing of Thermo 02 vs. Thermo 03.

Analysis of the slope and intercept data for the bracketing experiments above demonstrates the linearity of the relationship between the Candidate calibrator and the Reference calibrator, as discussed previously. The relationship between the two different calibrators remains stable as does the trend between the three concentrations measured. The large relative standard deviation for the intercept values is due to small differences in very small numbers. Table 6 contains data for comparison of Thermo 03 vs. Thermo 02 units in which the relative standard deviation of the slopes from the five comparisons is 1.14% with a small absolute variation in intercept. Table 7 contains data for comparison of Thermo 03 vs. Thermo 04 units in which the relative standard deviation of the slopes from the five comparisons is 0.54% with a small absolute variation in intercept.

Table 6: Linearity Comparison from Bracketing of Thermo 03 vs. Thermo 02

Test Number	Slope	Intercept
118731	0.940	0.138
118732	0.933	0.183
118734	0.929	0.178
118735	0.923	0.192
118736	0.912	0.199
Average:	0.927	0.178
Standard Deviation:	0.0106	0.0238
% RSD	1.14	13.4

Table 7: Linearity Comparison from Bracketing of Thermo 03 vs. Thermo 04

Test Number	Slope	Intercept
118737	1.07	0.158
118738	1.06	0.149
118739	1.06	0.172
Average:	1.06	0.160
Standard Deviation:	0.00577	0.0116
% RSD	0.54	7.26

The line equation data for the comparisons of Thermo 03 vs. Tekran 03 is provided in Table 8. Again, a good correlation is observed between experiments as indicated by the relative standard deviation of the slope of 1.28 % with a small absolute variation in intercept.

Table 8: Linearity Comparison from Bracketing of Thermo 03 vs. Tekran 03

Test Number	Slope	Intercept
118741	1.04	0.0593
118742	1.06	0.0509
118743	1.05	0.0521
118744	1.07	0.0774
Average:	1.06	0.0599
Standard Deviation:	0.0136	0.0122
% RSD	1.28	20.4

With good linearity between the Candidate and Reference units, the relationship between the Candidate setting and actual output could be established and used to transfer certification from the Reference to the Candidate calibrator. A complete summary data set presentation is provided in Figure 6. A plot of the actual Candidate outputs from a set of bracketing experiments versus the Candidate set point values at the three nominal NIST concentration values yields an actual concentration vs. set point line equation.

Comparison of WRI Thermo 03cal (REFERENCE) With WRI Thermo 02cal (CANDIDATE)
WRI Test # 118731, 8-19-2008

8.1µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Actual Cand.* Output
Zero Conce	0.01				
Thermo 03	8.02			8.10	7.75
Thermo 02	7.71	0.961			
Thermo 03	8.02				
Thermo 02	7.64	0.954	0.956		
Thermo 03	8.00				
Thermo 02	7.61	0.954			
Thermo 03	7.95				
5.7µg/m3 Nominal	**5-min avg** µg/m3	Nested Ratio	Avg Nested Ratio		
Thermo 03	5.57			5.70	5.50
Thermo 02	5.38	0.968			
Thermo 03	5.55				
Thermo 02	5.35	0.964	0.965		
Thermo 03	5.55				
Thermo 02	5.34	0.965			
Thermo 03	5.52				
2.7µg/m3 Nominal	**5-min avg** µg/m3	Nested Ratio	Avg Nested Ratio		
Thermo 03	2.57			2.70	2.68
Thermo 02	2.54	0.992			
Thermo 03	2.55				
Thermo 02	2.53	0.992	0.993		
Thermo 03	2.55				
Thermo 02	2.53	0.996			
Thermo 03	2.53				
Zero Conce	-0.05				

Cand. Output = Reference Conc. x Avg. Nested Ratio

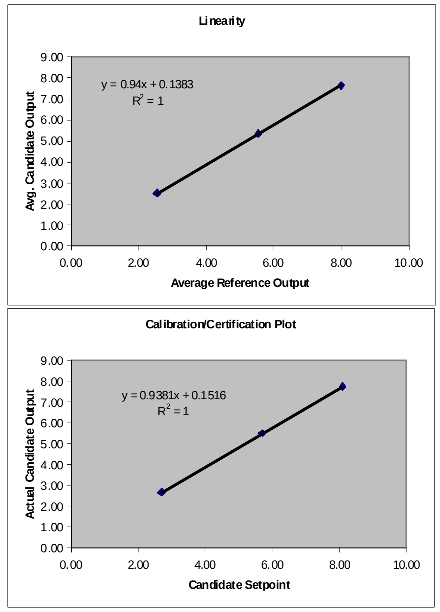

Figure 6: Example Data and Linearity and Calibration Certification Plots.

By plotting the actual certified Candidate concentration outputs (y) against the Candidate set points (x) of 8.1, 5.7, and 2.7 µg/m^3, for example, a line equation is defined. The line equation incorporates all of the experimental information and uncertainties at the three NIST traceable concentrations into a single line equation, which is a more accurate application of the data than if a single point value or point-to-point individual values, each with their own uncertainties, were to be used to calibrate an analyzer.

Table 9: Linear Equation Replicates for Certifying Thermo 02 from Thermo 03

	Actual Concentration, µg/m^3 at Set Points				
Test Number	2.7	5.7	8.1	Certification Line Equation	
118731	2.68	5.50	7.75	y = 0.938x + 0.152	
118732	2.70	5.53	7.73	y = 0.932x + 0.195	
118734	2.69	5.49	7.69	y = 0.927x + 0.190	
118735	2.69	5.48	7.67	y = 0.922x + 0.204	
118736	2.68	5.44	7.56	y = 0.910x + 0.211	
Mean	2.69	5.49	7.68	**y = 0.926x + 0.190**	
Standard Deviation	0.0084	0.0327	0.0742	0.0106	0.023
% RSD	0.31	0.60	0.97	1.15	12.1

This equation can then be used to calculate a "certified" concentration value from any set point within the certification range. Examples of this approach are provided in Tables 9 – 11 for the certification of Thermo 02, Thermo 04, and Tekran 03 units, respectively, against the Thermo 03 Reference unit. For purposes of this exercise, since NIST was unable to provide Vendor Prime certification for our Thermo 03 unit, we assumed that the set point values used for Thermo 03 (9.6, 8.1, 5.7, and 2.7 μg/m^3) were the "true" concentration values which would be identical to NIST certified values.

Table 10: Linear Equation Replicates for Certifying Thermo 04 from Thermo 03

	Actual Concentration, μg/m^3 at Set Points				
Test Number	2.7	5.7	8.1	Certification Line Equation	
118737	2.75	5.87	8.54	y = 1.071x - 0.169	
118738	2.72	5.82	8.43	y = 1.056x - 0.155	
118739	2.71	5.82	8.45	y = 1.062x - 0.182	
Mean	2.73	5.84	8.47	**y = 1.063x - 0.169**	
Standard Deviation	0.0208	0.0289	0.0586	0.0075	0.169
% RSD	0.76	0.49	0.69	0.71	8.13

Line slopes and intercepts are reported here to three decimals for subsequent calculations; they can be rounded down as appropriate. The origin point is not included in the line equations since the lines do not necessarily pass directly through the origin.

Table 11: Linear Equation Replicates for Certifying Tekran 03 from Thermo 03

	Actual Concentration, μg/m^3 at Set Points				
Test Number	2.7	5.7	9.6	Certification Line Equation	
118741	2.84	6.00	9.99	y = 1.036x + 0.060	
118742	2.80	5.98	10.09	y = 1.057x - 0.053	
118743	2.88	6.09	10.15	y = 1.053x + 0.053	
118744	2.80	6.02	10.17	y = 1.068x - 0.080	
Mean	2.83	6.02	10.10	**y = 1.054x - 0.051**	
Standard Deviation	0.0383	0.0479	0.0808	0.0133	-0.051
% RSD	1.35	0.79	0.80	1.26	1,410

The rsd values for the line slopes incorporate in a combined manner all of the individual uncertainties for each concentration, and these are comparable in magnitude to the individual rsd values for the individual points for a particular calibrator. The rsd values for the intercepts appear high since they represent small absolute value differences in very small numbers. Complete data sets for Tables 9, 10, and 11 are provided in Appendix C.

<u>December 2008 Calibrator Bracketing Experiments</u>

Following the September 2008 work, the concentration outputs of both the Thermo 03 and Thermo 04 units, which had been used extensively in the past, began to deteriorate. Thermo 03 was sent to Thermo Fisher for repair, and a new source provided by Thermo was installed in Thermo 04 by WRI. These calibrator units were designated as Thermo 03a and Thermo 04a following their repair. In December 2008, the nesting experiments were repeated in duplicate using the repaired Thermo 03a calibrator as the Reference unit with all of the other calibrator units in-house as Candidate units (Tekran 03, Tekran 04, Thermo 02, and Thermo 04a). For each Candidate unit, a set of readings was obtained once in the morning and once in the afternoon. The nesting summary data along with the linearity and calibration plots are provided in Appendix D. It is apparent from the data that both the Thermo 03a and Thermo 04a units are indeed basically different than the original Thermo 03 and Thermo 04 units prior to repair, so they should be considered to be different units. For the purposes of the study only, to demonstrate the procedure, it was assumed for the calibration line equations and plots in the appendices that the outputs for the particular Reference calibrator set points are the actual NIST traceable concentrations for the calibrator certification line plots. Overall, linearity appears quite good for all of the plots for all of the nesting studies performed throughout this study.

The comparison of the relative outputs of the calibrators was performed mathematically by dividing the average nesting ratios (Reference/Candidate) for a set of nesting experiments (Tables 3-5, Appendix D). Ratio values that were not directly measured were calculated from the nesting linearity equations and experimental nesting data (Tables 6-8, Appendix D). Relative output values for the September 2008 experiments are provided in Table 12.

Table 12. Average Relative Output Values for Calibrators from September 2008

September 2008	Reference Unit = Thermo 03			
Relatve Outputs	Concentration Set Point, ug/m3			
	9.6	8.1	5.7	2.7
	measured			
Thermo 02 / Thermo 03	0.946	**0.948**	**0.963**	**0.997**
Thermo 04 / Thermo 03	1.046	**1.042**	**1.032**	**1.020**
Tekran 03 / Thermo 03	**1.056**	1.063	**1.057**	**1.047**
	calculated			
Thermo 02 / Thermo 04	0.904	0.910	0.933	0.977
Thermo 02 / Tekran 03	0.896	0.900	0.911	0.952
Thermo 04 / Tekran 03	0.990	0.990	0.976	0.974

Relative output values for the December 2008 nesting experiments are provided in Table 13. The only direct comparison that can be made of relative generator stability between September 2008 and December 2008 is by using the Thermo 02 / Tekran 03 output ratio. These are the only units that had not been repaired between September and December. The results show that any changes in the relative outputs of these two units appeared to be minor: 1.6% at 9.6 µg/m^3, 1.1% at 8.1 µg/m^3, 2.6% at 5.7 µg/m^3, and 4.6% at 2.7 µg/m^3.

Table 13. Average Relative Output Values for Calibrators from December 2008

December 2008	Reference Unit = Thermo 03a			
Relatve Outputs	Concentration Set Point, ug/m3			
	9.6	8.1	5.7	2.7
	measured			
Thermo 02 / Thermo 03a	0.976	**0.984**	**1.046**	**1.162**
Thermo 04a / Thermo 03a	1.182	**1.193**	**1.226**	**1.356**
Tekran 03 / Thermo 03a	**1.071**	1.081	**1.119**	**1.164**
Tekran 04 / Thermo 03a	**1.130**	1.151	**1.220**	**1.386**
	calculated			
Thermo 02 / Thermo 04a	0.826	0.825	0.853	0.857
Thermo 02 / Tekran 03	0.911	0.910	0.935	0.998
Thermo 02 / Tekran 04	0.837	0.855	0.857	0.838
Thermo 04a / Tekran 03	1.104	1.104	1.096	1.165
Thermo 04a / Tekran 04	1.046	1.036	1.005	0.978
Tekran 03 / Tekran 04	0.948	0.939	0.917	0.840

Close scrutiny of the results of the December 2008 and January 2009 data suggests that the Thermo 03a Reference unit was still problematic following repair due to an unknown mechanical cause. The evidence is subtle, and this incident demonstrates a challenge in identifying improper performance of a Reference calibrator prior to conducting a nesting procedure. The reciprocals of the Tekran 04 / Thermo 03a nesting data for December in Table 13 are 0.884, 0.820, and 0.722 for 9.6, 5.7, and 2.7 μg/m^3, respectively. When compared with the corresponding values for January in Table 15, this represents a relative decrease in output of 5.5%, 7.3%, and 10.7% at 9.6, 5.7, and 2.7 μg/m^3, respectively. Values > 5 % indicate a change in one or both of the units. For the Thermo 04a / Tekran 04 nested ratios, however, the differences between December and January were 2.1%, 3.2%, and 4.9% at 9.6, 5.7, and 2.7 μg/m^3, respectively. These latter values fall within the proposed 5% acceptance criteria for changes over time.

Another consequence of the apparent mechanical problem with the Thermo 03a calibrator is demonstrated by comparison of Tekran 03 and Tekran 04 values calculated from December 2008 results was used as the Reference unit. The Tekran 03 and Tekran 04 units are both essentially equivalent to certified User calibrator units, and they both had been "certified" in the absence of an EPA protocol against a Vendor Prime unit by the manufacturer within three months of each other. The Tekran 03 unit was certified on July 11, 2008 and the Tekran 04 unit was certified on September 5, 2008. The certified values provided in Table 14 indicate that the outputs from these units are supposed to be essentially identical for each of the three concentration levels. However, the mathematical calculations of the expected ratios from the December 2008 results using Thermo 03a as the reference unit indicates that these two calibrators appear to differ by 5-16%, which is not actually the case (Table 13).

Table 14. Tekran User Prime Certification Sheet Summary Data, μg/m^3

Target / Set Concentration	Certified Concentration Tekran 03	Certified Concentration Tekran 04	Expanded Uncertainty Tekran 03	Expanded Uncertainty Tekran 04	Calculated Tekran 03/04 Ratio
0.00	0.000	0.000	na	na	na
2.70	2.781	2.779	0.046	0.046	1.0007
5.70	5.681	5.617	0.076	0.074	1.0114
9.50	9.477	9.467	0.122	0.118	1.0010

If it were not for the multiple comparisons in the above experiments it would be difficult to know that the Thermo 03a unit was not operating properly. For field certification against a certified Reference calibrator unit to be viable, a simple experimental procedure is needed to verify proper operation of the Reference unit prior to nesting.

January 2009 Calibrator Bracketing Experiments

For the January 2009 nesting study, WRI Tekran 04 was used as the Reference unit since it was provided by Tekran with certified concentration values against a Vendor Prime unit. Candidate units included WRI Thermo 02, Thermo 03a, Thermo 04a, and Tekran 03 units. The nesting data summaries, linearity plots, and calibration plots are provided in Appendix E. Since a Tekran reference unit was used, the concentration set points were 9.6, 5.7, and 2.7 μg/m^3, corresponding to the three User Prime certification points. The results are summarized in Table 15. As before, the comparison of relative outputs of the calibrators that were not measured experimentally against each other were calculated mathematically by dividing the average nesting ratios (Reference/Candidate) for the nesting experiments, which are provided in Appendix E.

As discussed above, both Tekran 03 and Tekran 04 are nominally certified User units, both having been certified against a Vendor Prime unit at the factory. The measured Tekran 03 / Tekran 04 nested ratios are very close to each other for these two units at 9.6 and 5.7 μg/m^3, which is consistent with the certification data provided in Table 14. However the difference at the 2.7 μg/m^3 setting is 5.1%. One of the two units is wrong at the low concentration, and there is no easy way of determining which one, unless additional comparisons are made with another certified Reference unit.

Table 15. Average Relative Output Values for Calibrators from January 2009 Data.

January 2009	Reference Unit = Tekran 04		
Relatve Outputs	Concentration Set Point, ug/m3		
	9.6	5.7	2.7
	measured		
Thermo 02 / Tekran 04	**0.852**	**0.893**	**0.906**
Thermo 03a / Tekran 04	**0.838**	**0.764**	**0.652**
Thermo 04a / Tekran 04	**1.024**	**0.979**	**0.932**
Tekran 03 / Tekran 04	**0.995**	**0.990**	**0.949**
	calculated		
Thermo 02 / Thermo 03a	1.016	1.169	1.390
Thermo 02 / Thermo 04a	0.832	0.912	0.972
Thermo 02 / Tekran 03	0.856	0.902	0.955
Thermo 03a / Tekran 03	0.842	0.772	0.687
Thermo 04a / Thermo 03a	1.222	1.281	1.429
Thermo 04a / Tekran 03	1.029	0.989	0.982

February 2009 Comparison of WRI Calibrators with EPA RTP Calibrators

In February 2009 we received two calibrators from EPA in Research Triangle Park for comparison with our Tekran 04 Reference unit. These units are designated EPA Tekran 05 and EPA Thermo 02. The nesting data are provided in Appendix F. A summary of the experimental data for the EPA Candidate units is provided in Table 16. Also provided are data for the WRI Tekran 03 unit from February 2 to the final measurement on February 20 following all of the nesting experiments with the EPA units. It appeared that the Tekran 03 / Tekran 04 nested ratios had not changed significantly between January 2009 and February 2 (Tables 15 - 16). However, a close examination of the data in Table 16 indicates a gradual creep upwards of the nested ratios between the WRI Tekran 04 Reference unit and the Candidate units. The final measurement of the EPA Tekran 05 Candidate unit against the WRI Tekran 04 Reference unit increased by 4.7 % from the first measurement on February 3 to the last measurement between the two units on February 12. These data indicate possible gradual decrease in concentration output of the WRI Tekran 04 Reference unit.

If we do not include the final measurement at 9.6 µg/m^3 in the nesting series for the WRI Tekran 03 unit, the percent relative standard deviation value for the Tekran 03 / Tekran 04 nesting data was 0.50 %. An apparent problem with the Reference unit at the last measurement increases the percent relative standard deviation to 4.24 %. Except for the final set of measurements for each of the units, these data can be used to cross correlate experimental results from nesting experiments performed at EPA RTP and at WRI. The EPA units were not provided with certified concentration values.

Table 16. Results of Nesting Experiments for EPA RTP Calibrators and WRI Tekran 03 against WRI Tekran 04 Reference Unit.

Nested Ratios of Candidate Calibrators with WRI Tekran 04 Reference Unit, February 2009									
	WRI Tekran 03			EPA Tekran 05			EPA Thermo 02		
ug/m^3	9.6	5.7	2.7	9.6	5.7	2.7	9.6	5.7	2.7
	1.002	0.993	0.953	1.063	1.017	0.917	1.041	1.002	0.948
	1.004	0.996	0.953	1.068	1.021	0.919	1.079	1.026	0.963
	1.002	0.996	0.956	1.080	1.017	0.899	1.088	1.024	0.959
	1.005	0.995	0.951	1.085	1.024	0.905	1.090	1.036	0.972
	1.014	0.999	0.951	1.115	1.027	0.898			
	1.111	1.030	0.955						
Mean	1.023	1.002	0.953	1.082	1.021	0.908	1.075	1.022	0.961
Std Dev	0.0433	0.0141	0.0020	0.0204	0.0044	0.0099	0.0228	0.0143	0.0099
% RSD	4.24	1.41	0.21	1.88	0.43	1.09	2.13	1.40	1.04

Calibration Gas Cylinder Bracketing Experiments

An alternative method for performing quality assurance checks on mercury calibrator systems that has been proposed is the use of mercury gas cylinders. Experiments discussed below were performed in an attempt to help determine the stability of the gas cylinders and provide a recommendation for their use in quality assurance for mercury calibrator systems. The current draft procedure requires verification of the candidate calibrator output by bracketing with a mercury gas cylinder at a single concentration. According to the draft traceability protocol, the candidate calibrator passes a quality assurance check when the calculated output from the bracketing comparison is within five percent of the certified output at the concentration tested. In this case, mercury gas cylinders were treated as Candidate calibrators in a bracketing comparison with a Reference elemental mercury calibrator.

Mercury gas cylinders and passivated / coated regulators were supplied by two vendors, Airgas and Spectra. Mercury concentrations of the two cylinders were labeled to be 8.6 and 9.5 $\mu g/m^3$ respectively. The mercury cylinders were balanced with compressed air only; no nitrogen testing was performed. All experiments followed the procedure outlined in RMB Consulting's December 2008 report titled "EPRI Hg Calibrator Traceability and QA/AC Procedures Project – Summary of Field Evaluation Test Program." In particular, no regulator passivation time or line purge was conducted prior to the bracketing experiments. The two mercury gas cylinders were bracketed with the WRI Tekran 4 Reference calibrator unit using a Thermo 80i analyzer. The WRI Tekran 4 Reference calibrator unit is certified as a User Prime unit to produce 9.467 $\mu g/m^3$ mercury when set to an output of 9.5 $\mu g/m^3$ mercury (Table 14). The candidate mercury gas cylinders were evaluated at three different flow rates (0.5, 1, and 2 L/min).

The results from the bracketing comparisons of WRI Tekran 4 (Reference) and the two mercury gas cylinders are provided in Table 17. Presentation of results in graphic form is provided in Appendix G. Both the Airgas and the Spectra Gases cylinders were bracketed four times with the Tekran 04 Reference calibrator unit. Bracketing was conducted twice at a flow of one liter per minute, once at a flow of two liters per minute, and once at a flow of 0.5 liters per minute. The draft protocol procedure calls for a flow of 1 L/min. The typical experiment lasted approximately 1 to 1.5 hours and used between 50 and 100 psi of gas from the mercury gas cylinders depending on the flow rate used.

The Airgas cylinder label indicated a concentration of 8.67 $\mu g/m^3$ of mercury with an initial certification date of 6/27/08. However, this is not in agreement with the bracketing comparison data in Table 17. Actual output from the cylinder ranged from 7.12 $\mu g/m^3$ to 8.02 $\mu g/m^3$. The average nested ratios for these comparisons were either 0.76 or 0.85 resulting in as much as an 11.8 % difference in average response ratios. This is well beyond the draft protocol's allowed 5.0 %. Each one of these experiments required approximately 60 minutes to achieve stable output from the gas cylinder. Flow rate did not appear to have a marked affect on either the time to reach stable output or the actual concentration. In fact one of the lowest of the four average outputs, 7.22 $\mu g/m^3$, was achieved at a flow rate of 2 L/min while one of the highest outputs, 7.95 $\mu g/m^3$, was obtained at a flow rate of 0.5 L/min. In retrospect it should be noted that the Tekran 04 Reference unit began to begin experiencing an apparent decrease in concentration output in late February. However, this would result in apparent higher Actual cylinder concentration readings, and not lower than the ones in Table 17. Therefore in a qualitative sense, the observations below are still valid.

Table 17. Summary of 2009 Mercury Gas Cylinder Bracketing Data.

Date	Reference Setpoint*	Label Tank Conc*	Flow Rate	Actual*	Average Response Ratios	% Difference from Initial	Time to Stability
Airgas Cylinder							
30-Jan	9.47	8.67	1L/min	7.12	0.76	Initial	60 min
11-Feb	9.47	8.67	2 L/min	7.22	0.76	0	60 min
11-Feb	9.47	8.67	0.5 L/min	7.95	0.85	11.8	60 min
16-Feb	9.47	8.67	1L/Min	8.02	0.85	11.8	10 min
Spectra Gases Cylinder							
Date	Reference Setpoint*	Label Tank Conc*	Flow Rate	Actual*	Average Response Ratios	% Difference from Initial	Time to Stability
30-Jan	9.47	9.5	1 L/min	8.42	0.89	Initial	136 min
11-Feb	9.47	9.5	2 L/min	9.26	0.98	10.1	40 min
13-Feb	9.47	9.5	0.5 L/min	8.9	0.94	5.6	40 min
16-Feb	9.47	9.5	1 L/min	9.49	0.99	11.2	72 min

* Concentrations in ug/m^3 at 1.0 atm. and 20 °C

The Spectra Gases cylinder label indicated a concentration of 9.5 µg/m^3 but only one bracketing comparison resulted in an average actual output that corresponded well to that value. Again, the average output of the gas cylinder was generally lower, ranging widely from 8.42 µg/m^3 to 9.49 µg/m^3. In this case the average response ratios for the last three experiments corresponded well and were within the 5.0 % maximum allowable difference from the protocol. However, the differences from the initial experiment were much greater than the allowable 5 %. With the exception of the first experiment, the average actual outputs are somewhat indicative of what should be expected. The values obtained at higher flow rates are closer to the indicated tank value while the lower flow rate results in a lower measured output. This could be indicative of insufficient flow. The time to reach a stable concentration varied greatly for the Spectra Gases cylinder and does not correspond well with any fluctuation in average actual output or average response ratios.

In comparing the behavior of the two gas cylinders several things are evident. First, it appears that the initial reading for these two cylinders and respective regulators was somewhat lower than in the subsequent experiments. It is believed that the regulators in use were never used or had not been used for several months prior to these nesting studies. The low initial readings could indicate that there is some passivation required for the initial use of the regulators before they maintain long-term stability. Second, it does not appear that taking the first set of results from these experiments and using them for comparative purposes to establish the linearity/stability of an instrument or cylinder is the best practice possible. As indicated by the nesting studies with the Spectra regulator there are three readings very close to one another but very different from the initial experiment. It is more likely that those three readings are more indicative of the cylinder concentration than the initial reading; however this cylinder still fails the 5.0 % difference test. Of practical use, it is also important to note that the two higher flow rates used in this study provided steady flow as observed by a stable indicator on the rotameter while there was significant rotameter instability at a flow of 0.5 L/min. The prescribed flow rate of 1 L/min is optimal as indicated by the results in Table 17. The time to stable concentration reading was not directly affected by either an increase or decrease in flow rate. Whether this is a result of the cylinder itself or the regulator to some extent was not evident from this study. The time to achieve stable output also seems variable to a great extent, especially between the two vendors, and could make operating protocols difficult to follow. One important note, in the case of this study, the gas cylinders were static for at least 4 months after delivery and prior to use. It is unclear at this time what affects this may have had on the results but raises further questions that need to be answered if the use of mercury gas cylinders is to be pursued further.

Proposed Calibrator Stability QC Check Procedure

A simple and rapid quantitative test procedure was developed to detect changes in calibrator output and performance stability over time. This is now provided as a mandatory QC requirement in Section 7.2.2 of the draft EPA 2009 interim traceability protocol for checking a reference generator. This can be performed with a single calibrator unit, and does not require bracketing. It is based on the observation that when a calibrator is new, it provides a highly linear output through the origin. Any changes that occur over time are reflected in changes in the output profile. The procedure was demonstrated with calibrator units that experienced failure during this study. It also can be used for evaluating prior bracketing data. This is especially important if the calibrator being evaluated is a Reference unit. Even if the procedure is not used as an initial check, it can be used in the event that a QC check of a User unit against a Field Reference calibrator indicates a >5 % change in nesting ratio from a prior QC check, to determine which of the calibrators has experienced a change in output.

To perform the procedure, first determine the theoretical ratio between the high concentration and the middle concentration within a span range. For a 10 µg/m^3 span, the theoretical high / medium ratio of 8.1 / 5.7 (µg/m^3) is 1.42. For a high medium concentration ratio of 9.6 / 5.7 (µg/m^3), this theoretical ratio is 1.68.

Table 18. Proposed Diagnostic Ratio Indicator of Calibrator Performance

Calibrator	Date	Experiment	High ug/m^3	High / Medium H / M Ratio	Theoretical / Actual	Percent Difference
Themo 02	8/19/08	118731	8.1	1.43	0.993	-0.7
	1/27/09	118761	9.6	1.6	1.05	5.0
Thermo 03	8/19/08	118731	8.1	1.44	0.986	-1.4
	9/5/08	118744	9.6	1.69	0.994	-0.6
Thermo 03a	12/5/08	118749	9.6	1.74	0.965	-3.4
	12/11/08	118754	9.6	1.81	0.928	-7.2
Thermo 03b	2/17/09	118793	9.6	1.8	0.933	-6.7
Tekran 03	9/3/08	118741	9.6	1.68	1	0.0
	2/13/09	118787	9.6	1.59	1.057	5.7
Tekran 04	12/11/08	118753	9.6	1.66	1.012	1.2
	2/10/09	118781	9.6	1.59	1.057	5.7
	2/17/09	118793	9.6	1.56	1.077	7.7

Theoretical ratio values: 8.10 / 5.70 = 1.42 9.60 / 5.70 = 1.68

To perform the QC check, calibrate the analyzer at the high concentration for the calibrator, then read the concentration value from the analyzer at the high level and then read the analyzer output when the calibrator is set at the medium concentration value without changing the spectrometer calibration. Then divide the theoretical ratio by the actual output ratio. A problem is evident if the concentration output ratio is not within +/- 5 % of the theoretical ratio value. From the data in Table 18, it appears that the cause of failure for Thermo 02, Tekran 03, and Tekran 04 was likely due to the onset of elemental mercury source passivation, since the relative ratios are positive values. Passivation appears to be due to the release of volatiles from the 30-50 feet of PFA tubing between the room in which the compressors were housed and the laboratory. Before any additional nesting experiments are performed, refrigerated air dryers will be instilled in addition to the Tekran air purification manifolds and Thermo carbon cartridges already in place, to provide the highest quality air possible. The problems with the Thermo 03, 03a, and 03b series units appear to be due to some other mechanical difficulty, since the percent differences are negative values.

Power Interruptions and Calibrator Performance

In the early phases of this work, there was some concern about the amount of time that the calibrators and analyzers require to be powered up and under air-flow in order to obtain reliable results. It appears that overnight power to the analyzer and calibrators is necessary. In addition overnight air flow is essential for reliable calibrator operation.

Another issue of concern is the effect of short-term (< 1 hour) power loss on the stability of the calibrators and analyzers. The following discussion describes two instances in which power was lost under different circumstances. The first instance of power loss occurred at a point in which the WRI laboratory building lost power due to a storm for approximately one hour during a 8.1 µg/m^3 sequence of a bracketing experiment between Thermo 03 and Thermo 04 after the systems had appropriate 12-hour minimum initiation time. Upon re-powering the network the instruments behaved as though they had never been initiated properly. In other words, poor stability resulted in making it impossible to zero and span the Reference calibrator (Thermo 03). At that point the prior experiment was halted and the instruments were monitored throughout the day for approximately 3 additional hours. Stability did not return to the system within that time. The second event involved a power outage that was caused by user error in which the power switch on the Thermo 03 Reference calibrator unit was inadvertently turned off during a bracketing experiment when transitioning from a concentration of 8.1 µg/m^3 to 5.7 µg/m^3. The Reference calibrator unit was re-powered up in less than two minutes and the experiment was continued. No drift or non-linear behavior was observed in this case.

We recommend that, in the case of a total power outage that lasts for more than 30 minutes, the instruments be put through normal initiation times/procedures. This includes powering the calibrators and analyzers on under air-flow for at least 12 hours prior to generating data. In the instance of a very short power outage of less than 5 minutes, it is expected that the instruments retain linearity and any experiment underway can be picked up where the outage occurred. For power outages between 5 and 30 minutes, it is recommended that both calibrator and analyzer be put through normal initiation times/procedures until further work can be completed to determine the effects.

<u>Use of User Field Units for CEM Calibration</u>

For certification, a nesting procedure would be conducted in duplicate (3x3x2).

1. To certify a Candidate / User Prime unit from a Vendor Prime Reference unit at a particular span range (i.e. 10 µg/m^3) directly compare the relative outputs of the Candidate calibrator to the Reference calibrator at the three NIST traceable concentration values for that range (High, Medium, and Low, i.e. 9.6, 5.7, and 2.7 µg/m^3). Set both the Candidate and Reference units at the identical set point values and perform the 3x3 nesting study sequence to determine the average relative outputs for the two units at each of the three concentrations. A plot of Candidate output vs. Reference output can be made to evaluate linearity and to archive the line equation for later comparison to see if the relative outputs change significantly in the future.

2. Using the NIST traceable certified values for the Candidate unit, use the average output nested ratios to calculate the actual concentration of the Candidate unit at each of the three certified point values. Calculate a line equation using these three values:

   Actual Candidate Output = Slope*Set point + Intercept.

3. This line equation could then be used to calculate the actual output from the Candidate unit for CEM calibration within the range of set points ranging from -30 % of the Low concentration set point to +30 % of the High concentration set point.

CONCLUSIONS

Experimental procedures for conducting the nesting experiments based on recommendations from the Mercury Standards Working Committee and consistent with results required in the August 2007 EPA Interim Traceability Protocol were optimized. The procedures were subsequently demonstrated with repeated experiments using elemental mercury generators at three concentrations. Results are summarized below.

The use of the previously established standard operating procedures work well for carrying out bracketing comparisons of Reference and Candidate elemental mercury calibrators with only a few updates. The most important of these is that the instruments require a minimum of twelve hours of initiation time with power on and air flow being delivered. The nesting studies indicate differences in relative outputs between Candidate and Reference units. The bulk data also suggest that there is some analyzer drift that occurs but only to a minimal degree. More importantly, the results from the nesting studies demonstrate that the relationship between the Reference and Candidate calibrators remains highly linear between experiments and very little variance is observed when actual responses are plotted against each other.

A new draft QC procedure for verifying proper elemental calibrator performance was developed in this study. The procedure involves output concentration ratios, and it does not require comparison with another calibrator. This procedure is now included as a mandatory QC check in the draft EPA traceability protocol as Section 7.2.2. To successfully operate elemental mercury calibrators over a long period of time, the highest quality air or nitrogen possible must be used to minimize elemental mercury source passivation. Attention to the quality or pretreatment of the PFA tubing might be necessary also.

In order to transfer certification from a Reference to a Candidate, the use of a linear plot of Candidate set point versus the actual Candidate output as determined by bracketing studies was examined. The process involves using the linear relationship of the responses between Candidate and Reference calibrators and their signature, average nesting ratios at each concentration. Plotting the actual Candidate output versus the Candidate set point (calculated using the signature nesting ratios) results in a linear plot that can be used to calculate the actual output of the Candidate calibrator at any given concentration within the NIST certified points used in the nesting procedure. The results presented here show promise for use of this approach as a simple, effective way to transfer certification from a Reference Vendor Prime to a Candidate / User unit.

Results with gas cylinders suggest that cylinder use can be recommended at this time to verify calibrator output. Results were consistent with the previous work by RMB in which they observed that the actual output of the mercury gas cylinders does not always appear to be stable.

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APPENDIX A

Standard Operating Procedure for Thermo Calibrators

Task 2 Standard Operating Procedure
Laboratory Comparison of an Elemental Hg Candidate Calibrator Versus A Reference Calibrator

Thermo Procedure

Western Research Institute   5/23/08 draft

This procedure is based on the 2/21/08 V 3.5 Task 1 field comparison procedure from Richard McRanie and Will Roberson, RMB Consulting

1. Introduction

The purpose of this document is to describe the setup and procedure for a series of tests designed to compare a candidate elemental Hg calibrator with a reference calibrator in a laboratory setting. The purpose of the tests is to evaluate the stability of the conventional "head space" calibrators that are an integral part of every Hg CEMS. The procedure described below is called a 3 X 3 test matrix, which means that three concentrations will be compared three times alternating between the reference calibrator and the candidate calibrator. For some tests, a 3 X 6 matrix will be used. This is essentially the same as the 3 X 3, with three additional comparison steps. A zero reading from the analyzer must be obtained at the beginning and end of each day to confirm that zero drift has not occurred. A Thermo 80i continuous atomic fluorescence Hg analyzer will record the measured concentrations. Concentration ratios between the reference and candidate calibrator at each concentration will be the result. The stability of the ratios over time will be the output of this project.

This procedure is accompanied by a data collection spreadsheet (with instructions) that should be filled out completely and submitted to RMB Consulting & Research, Inc. (RMB) as discussed in the data management section of this procedure. The actual data will be shared with anyone desiring to see it after a brief completeness review. All efforts should be made to provide complete data. Any questions should be addressed to Richard McRanie at RMB.

2. Discussion of Comparison Tests

The calibrator comparison tests will be done using a bracketing procedure employed by the National Institute of Science and Technology (NIST) when traceability is transferred from the NIST Prime calibrator to the Vendor Prime calibrators used by Tekran and Thermo. This procedure is called a bracketing process where the candidate calibrator is bracketed by the Reference Calibrator at each of three Hg concentrations: 2.7, 5.7, and 8.1 $\mu g/m^3$. The bracketing process at each concentration can be represented by the sequence shown in Table 1.

Table 1 – Measurement Sequence, each concentration

Reference Calibrator	Response 1
Candidate Calibrator	Response 2
Reference Calibrator	Response 3
Candidate Calibrator	Response 4
Reference Calibrator	Response 5
Candidate Calibrator	Response 6
Reference Calibrator	Response 7

The concentration of the Candidate Calibrator for each comparison is:

C_{Can1} = (Response 2 / (Avg Response 1,3)) * C_{Ref}
C_{Can2} = (Response 4 / (Avg Response 3,5)) * C_{Ref}
C_{Can3} = (Response 6 / (Avg Response 5,7)) * C_{Ref}

C_{CanI} is the calculated Candidate Calibrator concentration; C_{Ref} is the delivered concentration of the Reference Calibrator

The mean concentration of the candidate calibrator is:

C_{CanM} = Avg (C_{Can1}, C_{Can2}, C_{Can3})

The ratio between the Reference Calibrator and Candidate Calibrator is a useful value when tracking stability over time. This ratio can be calculated as:

R_{Can1} = Response 2 / (Avg Response 1,3)
R_{Can2} = Response 4 / (Avg Response 3,5)
R_{Can3} = Response 6 / (Avg Response 5,7)

The mean ratio is:

R_M = Avg (R_{Cn1}, R_{Can2}, R_{Can3})

This simple bracketing technique is appropriate because the Hg calibrators and analyzers are very linear and the concentrations being compared are very similar in magnitude. The bracketing technique also compensates for drift and other variability in the performance of the calibrators and analyzers.

Note that the comparisons in Table 1 are only for a single concentration and three concentrations will be evaluated during each test. A spreadsheet will be provided to perform all calculations.

3. Written Description of Plumbing Setup

The configuration allows for the alternation of the two calibrators while admitting calibration gas directly to the analyzer. A plumbing diagram for the Thermo arrangement provided by Thermo is contained in the following sections of this procedure. The Reference calibrator will be used for zero gas and this connection will not be disturbed. Both are designed to maintain appropriate flow conditions at the outlet of the calibrator and the inlet of the analyzer so that those conditions do not influence the results obtained. The diagrams must be followed faithfully. All tubing and fittings should be 3/8" PFA unless otherwise noted.

It is critical that the calibrators not be deadheaded which causes excessive backpressure. The atmospheric vents as shown in the respective drawings must be used. In addition, all vents must be routed to a negative pressure vent such as a fume hood so the laboratory interior does not become contaminated with Hg vapor or nitrogen. The calibrators must be in standby condition during any disconnection or connection of fittings.

4. Thermo 81i and 80i Plumbing Diagrams and Setup

The Thermo plumbing diagram for operating the calibrators with ***air*** dilution is shown in Figure 1 at the end of this document. The Thermo plumbing diagram for ***nitrogen*** dilution is shown in Figure 2. Figure 3 is a picture of the nitrogen back pressure regulator that is used to retrofit to all Thermo nitrogen dilution systems. This retrofit is to stabilize the flow through the nitrogen generator and should be left in place at all times. Please follow the instructions on Figure 3 to set the pressure regulator correctly. Note that the Thermo analyzer and calibrator have separate input and output ports for span, probe calibration and zero gas. The diagram was designed to make it clear where all connections should be made for the comparison tests. Mistakes have been made by prior researchers and it was obvious that the plumbing connections had to be perfectly clear. All tubing and fittings shall be 3/8" PFA Teflon. The 3/8" tubing and fittings will minimize backpressure on the calibrator. The 3/8" 3-way valve can be either PFA Teflon or Stainless Steel.

4.1 Place the reference calibrator (81R) on a workbench in reasonable proximity to the candidate / installed calibrator (81I).

4.2 Both calibrators must use the same type of gas: air or nitrogen. If the calibrators use air as the operating medium, plumb cleaned dry zero air to the calibrator port labeled "CDA IN". The same source of air must be used for both calibrators. Note that this air should be connected after full air cleanup. If the calibrators use nitrogen from a nitrogen generator as the operating medium, install the Thermo supplied back pressure regulator assembly (BPR) per Figure 2, and follow the instructions on the figure for the initial setup. If a nitrogen cylinder is used, it is plumbed in the same manner as an air cylinder. The same source of nitrogen must be used for both calibrators.

4.3 Power up the calibrators. (They will power up in Standby Mode)

4.4 Allow the calibrators to operate for at least 12 hours prior to making any comparative measurements.

4.5 Zero and calibrate the 80i Hg analyzer with the Reference calibrator as described below. The Reference calibrator will be used to supply zero gas to the analyzer; this line is connected per Figures 1 and 2.

4.6 Install the 3-way valve manifold and vent tees as shown in Figures 1 and 2 to the IS connections on the reference calibrator, candidate calibrator and analyzer. The vents between the calibrators and the 3-way valve are essential to prevent deadheading the IS output of the calibrators. There should also be a 3/8" vent line tee just adjacent to the analyzer.

4.7 Make sure that all vents are routed properly and ensure that all vents are not kinked or obstructed.

4.8 The 80i Hg analyzer needs to be in Instrument Zero Mode to pull gas through the IZ port, or in Instrument Span mode to pull calibration gas through the IS port. To place either the 80i analyzer or either of the two calibrators into IZ, IS, or standby mode, the Gas Mode Menu works the same way. (Main Menu>Instrument Controls>Gas Mode).

Note: For the sake of this report gas was fed only through the IS port due to a malfunction in the IS inlet of the 80i analyzer. Several steps below weren't necessary because of this. Instead, for steps that involve changing the analyzer from IZ to IS, the tubing was switched from zero gas to cal gas at the prime calibrator.

4.9 Operate the analyzer in Manual Hg(T) mode. (Main Menu>Instrument Controls>Auto/Manual Mode).

4.10 Check all datalogging settings on both the Hg analyzer and the Hg Calibrators. See "Checking Datalogger Settings" Section of this procedure for specific guidance. The logging periods on each component should be set to 1 minute with 60 second averaging. Be sure to synchronize the clocks on the 80i analyzer and the 80i calibrators.

4.11 To change the target concentration on either of the calibrators, go to Main Menu<Instrument Controls<Target Hg Span Conc. Set any of the 6 span values to the three values that will be used for this study (to be determined).

4.12 Once either of the calibrators is in Instrument Span Mode, the target concentration can be changed from the main run screen by pressing either the Up or Down button. The current Span value is displayed on the Time Banner (i.e. Span 1, Span 2....).

4.13 Put all 3 instruments (both calibrators and the analyzer) into Instrument Zero Mode. (Main Menu>Instrument Controls>Gas Mode).

4.14 After the zero reading on the analyzer is stable, adjust the analyzer zero background so that the analyzer reading is zero (Main Menu>Calibration>Hg Bkg).

4.15 After the zero is adjusted put the analyzer in Instrument Span mode (Main Menu>Instrument Controls>Gas Mode).

4.16 Leave the candidate calibrator in Instrument zero mode.

4.17 The Reference Calibrator will be used for the base instrument span calibration. Put the Reference Calibrator in Instrument Span mode and set it to deliver the high-level NIST certified point of the Reference Calibrator for the span to be evaluated (i.e. 8.1 $\mu g/m^3$ for a 10 $\mu g/m^3$ span).

4.18 Position the 3-way valve to direct span gas from the Reference calibrator to the analyzer.

4.19 Allow the calibrator and analyzer to stabilize at the high span value (this may take 10-15 minutes the first time).

4.20 After a stable reading is obtained, adjust the calibration coefficient so that the analyzer reads the exact delivered concentration shown on the Reference Calibrator display (Main Menu>Calibration>Cal Hg Coef).

4.21 Place the analyzer in the Instrument Zero mode and recheck the zero. Readjust if necessary. Once a stable zero response is achieved, allow the analyzer to measure the zero response for a period of at least 5 minutes. Record the start time of the first valid minute of stable zero response in the spreadsheet, and manually note the start time and analyzer readings in a test notebook for the initial zero response.

4.22 Place the analyzer in Instrument Span mode and recheck the span. Readjust if necessary.

Note: It is important that the zero and span be set precisely (within 0.05 µg/m^3) and that adequate time be allowed for the reading to come to a stable value. Longer averaging times will require a longer waiting period. In addition, when the manifold is first connected some time may be needed to condition the lines, valve and fittings.

4.23 After a stable zero and span calibration has been achieved, put all 3 instruments (both calibrators and the analyzer) into Instrument Span Mode.

4.24 Adjust both calibrators to the high-level NIST certified set point of the Reference Calibrator for the span to be evaluated. Both calibrators should be set on the front display to show ***exactly*** the same concentration value.

4.25 Rotate the 3-way valve to direct gas from the Reference Calibrator to the analyzer and allow the reading to stabilize. Record the start time of the first valid minute of stable analyzer response in the spreadsheet, and manually note the start time and analyzer reading in a test notebook for Response 1. The interval prior to the first reading and between readings is 8 minutes, with the final 5 minutes used to calculate a 5-minute average concentration value.

4.26 Rotate the 3-way valve to direct gas from the candidate calibrator to the analyzer and allow the reading to stabilize. Record the start time of the first valid minute of stable analyzer response in the spreadsheet, and manually note the start time and analyzer reading in a test notebook for Response 2.

4.27 Continue this switching between the Reference and candidate calibrators until all 7 responses have been recorded.

Note: It is not expected that the readings on the Reference Calibrator and the Candidate Calibrator will be exactly the same even though the calibrator set point display concentrations are the same. Record the values as indicated by the analyzer. Note that each concentration sequence begins and ends with a reference calibrator response.

4.28 After Response 7 at the high level has been recorded, leave the 3-way valve aligned with the Reference Calibrator, change the display setting on both calibrators to the mid-level setting (5.7 µg/m^3) and allow the calibrator display delivered concentration and analyzer reading to stabilize over an 8-minute interval.

4.29 Record this reading as Response 1 for the midlevel sequence and switch the 3-way valve to direct gas from the candidate calibrator to the analyzer. Allow the analyzer to stabilize and record the reading as Response 2.

4.30 Continue switching between the candidate and Reference Calibrators until 7 responses have been recorded at the midlevel.

4.31 Repeat the sequence at the low level concentration (2.7 μg/m^3).

4.32 After the last response has been completed at the low level, put the analyzer and candidate calibrator into Instrument Zero mode and recheck the zero. Record this final zero reading in the spreadsheet. Do not adjust the zero.

After testing is complete, disconnect the manifold after marking all of the connections and retain the manifold for the next set of tests. It should be plumbed up exactly the same way for each set of tests. Extract the data files from both calibrators and the analyzer as described below. Transmit the data files and spreadsheet file to RMB as discussed in the data management section below. Please check the files for completeness before transmission.

5. Checking Datalogging Configurations

When performing any of the bracketing, nesting or "nose to nose" type Hg calibration gas generator comparisons; one must have access to all the pertinent data generated by the "Reference Calibrator", the "Candidate Calibrator" and the analyzer being used in order to perform the necessary uncertainty and performance calculations. To ensure the data collected are of adequate resolution to properly perform the calculations required by the direct comparison procedures, one must check that the datalogger settings for both the Thermo 80i analyzer and Thermo 81i calibration gas generator(s) are set to record data with a period of 1 minute. Typically the Thermo 80i is set to collect 1 minute data with a 60 second averaging time, but this setting should be confirmed.

However, the default datalogger settings in the Thermo 81i calibrators are set up with a logging period of 60 minutes (i.e. hourly output averages). This default setting is of insufficient resolution to perform the calculations required by the direct comparison procedures. Therefore, during the comparative testing, it is necessary to change the datalogger settings of Thermo's 81i calibration gas generator(s) to record and average the data logged every 1 minute instead of the default setting of every 60 minutes. The bulleted procedures provided below are a guide on how to change the default logger settings and ensure all pertinent measurement data is collected to meet the needs of the direct comparison procedures. If additional assistance and guidance is required, see Chapter 3 of Thermo's 81i Instruction Manual.

The procedures provided in this document were written from the latest firmware updates provided by Thermo Fisher Scientific. For the Model 81i calibrator, this is Version 01.00.16.097 with Firmware 10.19.90. For the Model 80i analyzer, this is Version 01.00.41.253 with Firmware 10.19.90.

<u>Changing Thermo 81i Calibration Gas Generator Data Logging Period</u>
(Note: These same key strokes can also be used to check the datalogger logging period setting of the Thermo 80i).

- Select “Instrument Controls” from the main menu
- Then press the down arrow key until the selection carrot is beside the “Datalogging Settings” option. Then press the “Enter” button.
- Then select “Configure Datalogging”. This menu will display the current logging period settings, memory allocation, and data treatment.
- Select “Logging Period Min” and use the down arrow key to reduce the setting to 1 min, and then press “Enter”.
- Press “Menu Button” to return to “Configure Datalogging” menu. If the “Data Treatment” setting is set to “Avg”, the configuration set-up is complete and the “Run” button can be used to get back to the main screen.
- If “Data Treatment” is not set to “Avg”, scroll down and select “Data Treatment”. Use down arrow to toggle options until “Avg” appears. Then press “Enter”.

Selecting Proper Logging Content

Prior to beginning any of the bracketing, nesting or “nose to nose” type Hg calibration gas generator comparisons, the data content being logged by each component should also be checked. There are many different parameters that are of interest during these tests and the bulleted procedure below will ensure that all pertinent parameters are logged. Again, if additional assistance and guidance is required, see Chapter 3 of either Thermo’s 80i or 81i Instruction Manual.

Checking Logging Content Settings for Thermo 81i and Thermo 80i

- From the “Run” screen, press the “Menu” button to get to the main menu
- Then select “Instrument Controls” from the main menu
- Then press the down arrow key until the selection carrot is beside the “Datalogging Settings” option. Then press the “Enter” button.
- Then select “Select Content”

Once the “Select Content” screen is reached, the user is allowed to assign specific parameters to specific data fields. For the Thermo 81i, check that the bulleted items below are selected. Note: the default selections should be pretty close to these settings.

Thermo 81i Data Logger “Selected Content” Settings

Field 1: Hg Concentration	**“CONC”**
Field 2: Hg Span	**“SYSSP”**
Field 3: Hg Flow	**“HGFLO”**
Field 4: Diluent Flow	**“DLFLO”**
Field 5: Cooler Temp	**“CTEMP”**

Field 6: Hg Pressure "PRES"

If any of these settings are different, they should be changed to match this configuration.

These six fields are critically important because future data treatment spreadsheets may be dependent on certain parameters being in specific fields.

For any particular field that is different, scroll down and press "enter" to select that field. "Data in LREC Field X" will then appear. The user will then be able to select what parameter is logged into that particular field. There will be two options to choose from: "Concentrations", and "Other Measurements". The proper data parameter can be selected for Fields 1 and 2 by selecting the "Concentrations" option and the proper data for Fields 3 through 6 can be obtained by selected the "Other Measurements" selection. Once the user has selected either "Concentration" or "Other Measurements", simply scroll down until the desired parameter is highlighted and press "enter". This will place the selected parameter into the selected field. If additional assistance and guidance is required, see Chapter 3 of either Thermo's 80i or 81i Instruction Manual.

If any of parameters required altering, the content must be saved or the changes will be lost. In order to save any changes to the data logger configuration, the user must select the "Commit Content" option from the "DataLogging Setting" menu.

- Main Menu > Instrument Controls> Datalogging Settings >Commit Content

The minimum required logging content during the field candidate for each component is summarized below. (Note: most, if not all of these parameters should be logged by default).

<u>Summary of Data Logger Content</u>

Thermo 81i Hg^0 Calibrator Data Logging Selections Summary

- "Time"
- "Date"
- "Hg Conc" (This is the actual output concentration and appears as "CONC" in data file)
- "Hg Span" (This is the span number and will appear as "SYSSP" in data file)
- "Hg Flow" (Flow rate associated with mercury reservoir will appear as "HGFLO")
- "Dil Flow" (Flow rate associated with the dilution gas will appear as "DLFLO")
- "Cooler Temp" (Cooler temperature and will appear as "CTEMP")

Thermo 80i Analyzer

- "Time"
- "Date"
- "HGT" - Total mercury concentration

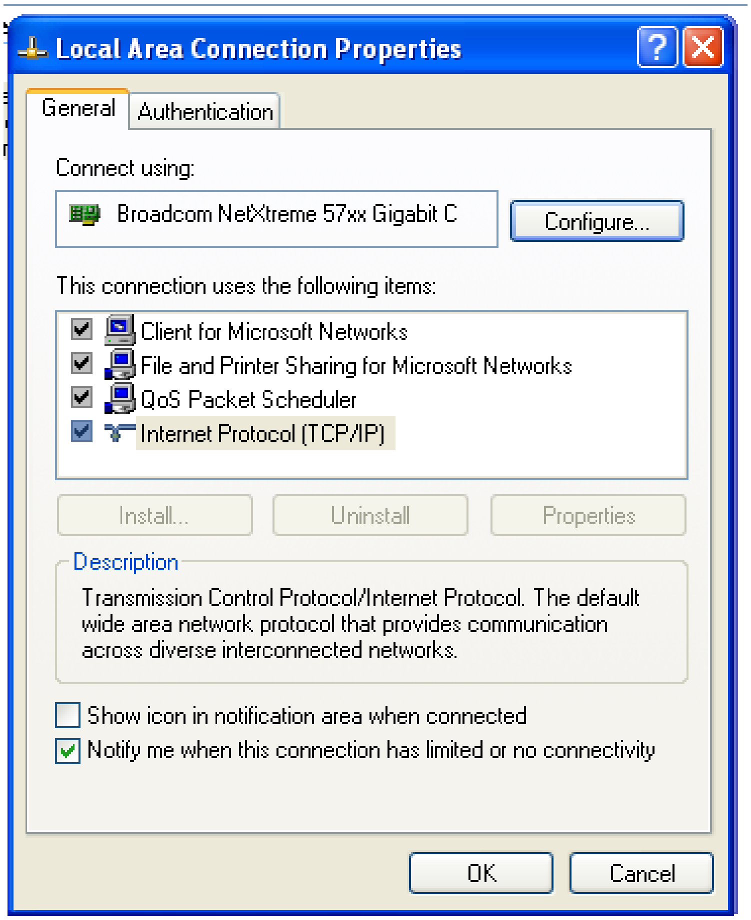
- “RFINT” - Reference intensity
- “SMPLF” - Sample flow rate
- “PMTV” - PMT voltage
- “PRES” - Analyzer chamber pressure

6. Data Extraction

To extract data from either calibrators or the analyzer, iPort software should be used. The iPort software is available at Thermo.com

6.1 Load the iPort software

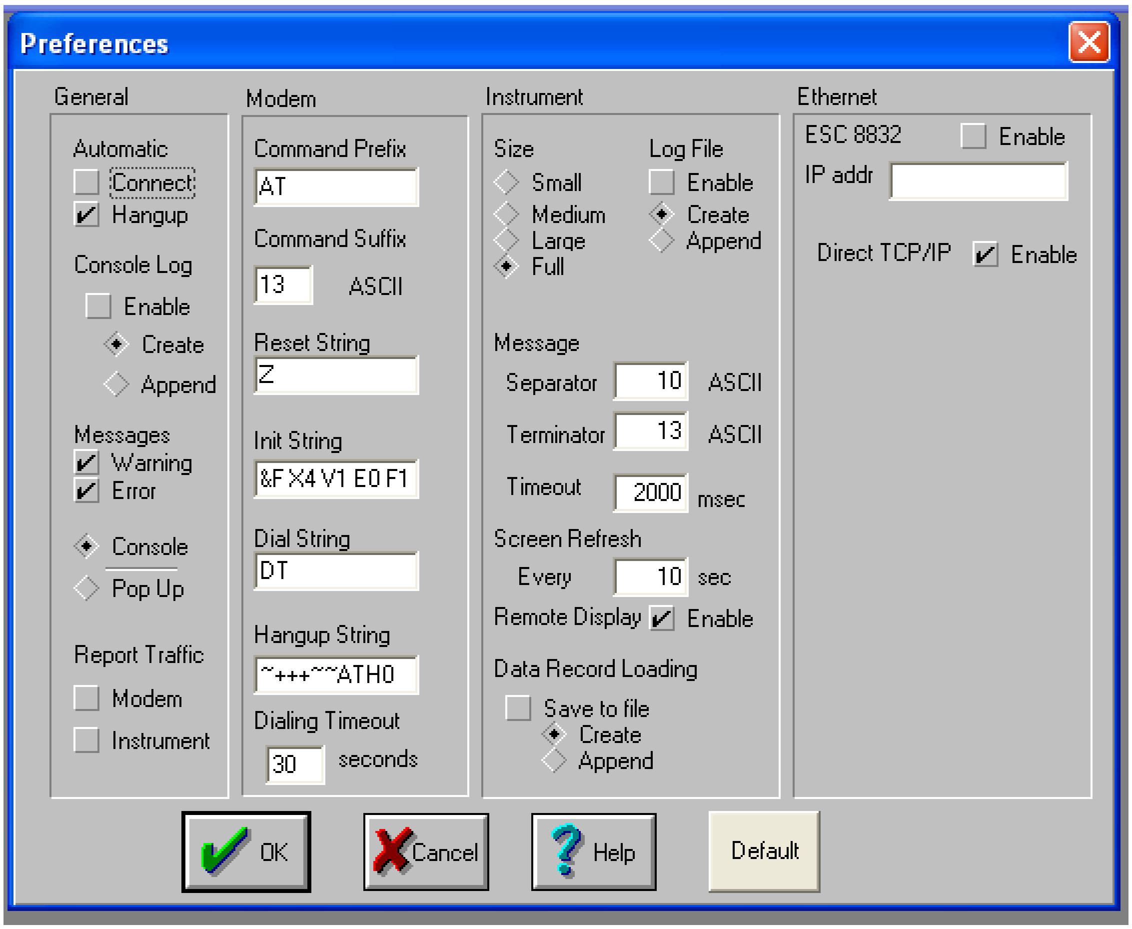
6.2 Configure the laptop or computer to use a local area connection with an appropriate IP, by selecting properties under the TCP/IP item (see image below)(A different IP address may be needed)

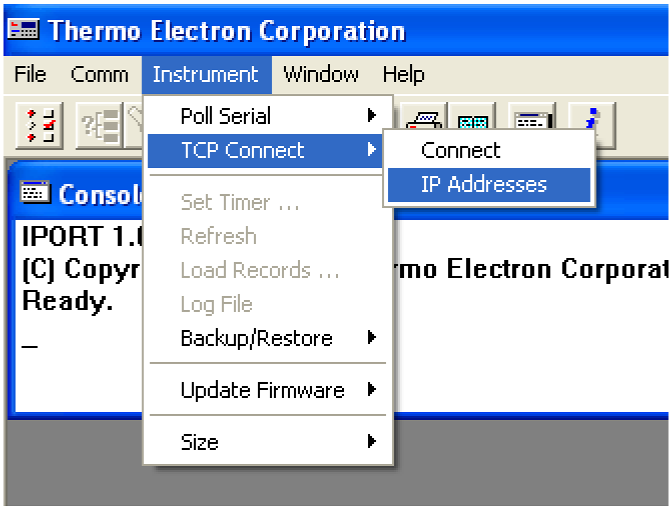


6.3 Connect an Ethernet cable from the laptop to an available port in the hub (located in the instrument rack). A cross-over Ethernet cable can also be connected directly to the back of any instrument.

6.4 Start iPort, select Preferences and check the Direct TCP/IP box, press OK.

6.5 Go to Instrument/TCP Connect/IP Address, double click and enter in the IP address of the desired instrument (see image below). Then select Instrument/TCP Connect/Connect, and the laptop should begin to communicate with the instrument (you should see the instrument front panel displayed on the screen)





6.6 Select Instrument/Load Records.

6.7 Select LREC and enter how many records you wish to download (e.g 1400). Each record is 1 minute worth of data. Check the box to Save File and enter in a file name. Press OK.

6.8 The data will be saved in the iPort directory.

For further information, the iPort manual is available at Thermo.com.

7. Data Management

The completed spreadsheet and extracted data files will be uploaded to RMB's FTP site as soon as possible after the tests are completed. The format for the upload files is date, plant name and unit number followed by ref (reference calibrator file), insta (candidate calibrator file), analy (analyzer file) or ss (spreadsheet file). Using a test performed on February 20, 2008 at Branch 1-2 for example, the reference calibrator file would be named 022008Branch1-2ref.dat and the spreadsheet file would be 022008Branch1-2ss.xls

The site URL is http://www.rmb-consulting.com/mercury.htm. You will note that there are two options – upload data and download data. Unique user names and passwords will be assigned to all participants for upload and download access. Each utility site and unit will be given a confidential code name by RMB prior to the start of the project and this code name will be used to identify all data on the download site. After the data are uploaded, RMB will ensure that all data are present in the proper format. RMB will also change the file names and transfer the files to the download FTP site where all project participants will have access to the data. The objective is to make the data available within 2-3 days after collection.

Figure 1 – Thermo Air Plumbing Diagram

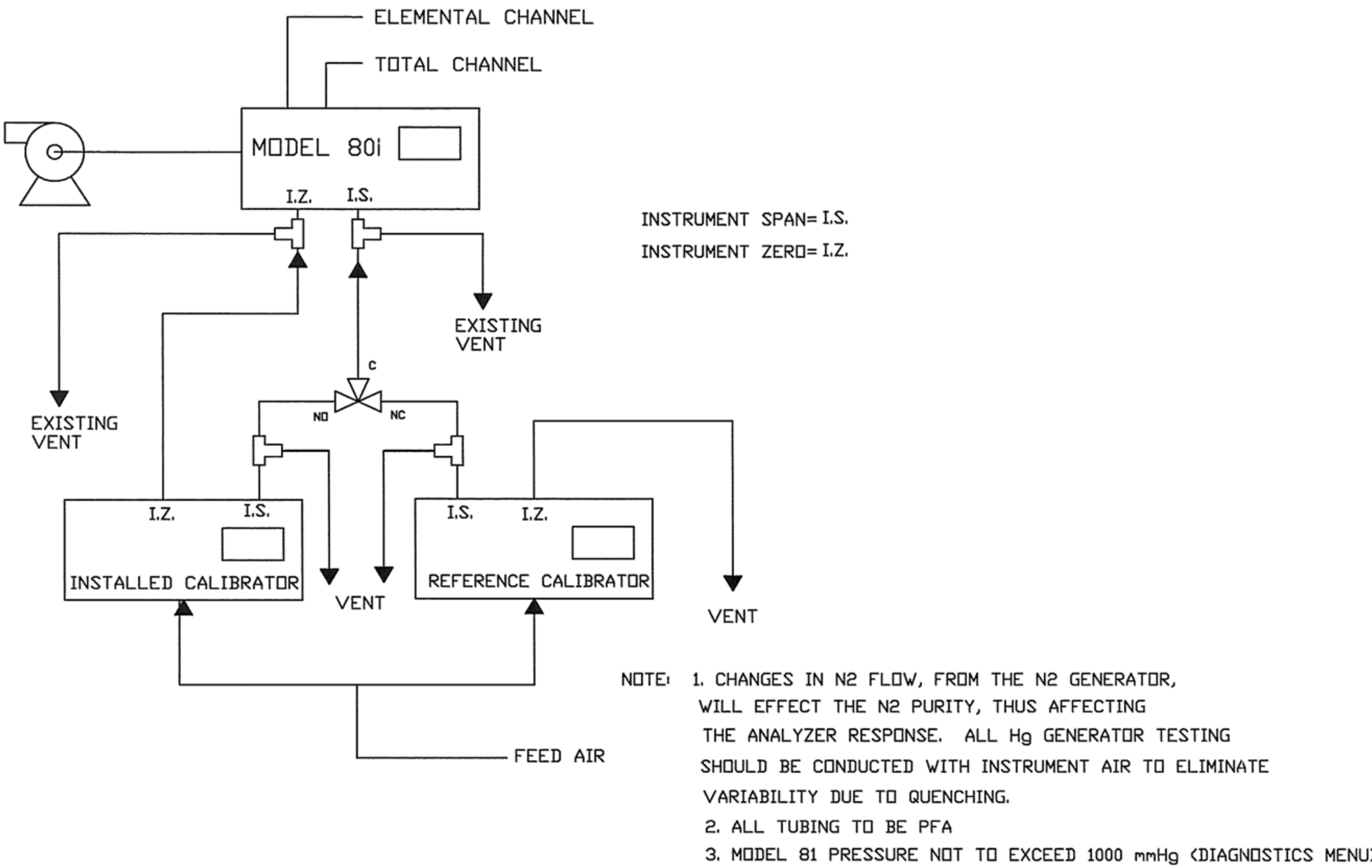


Figure 2 – Thermo Nitrogen Plumbing Diagram

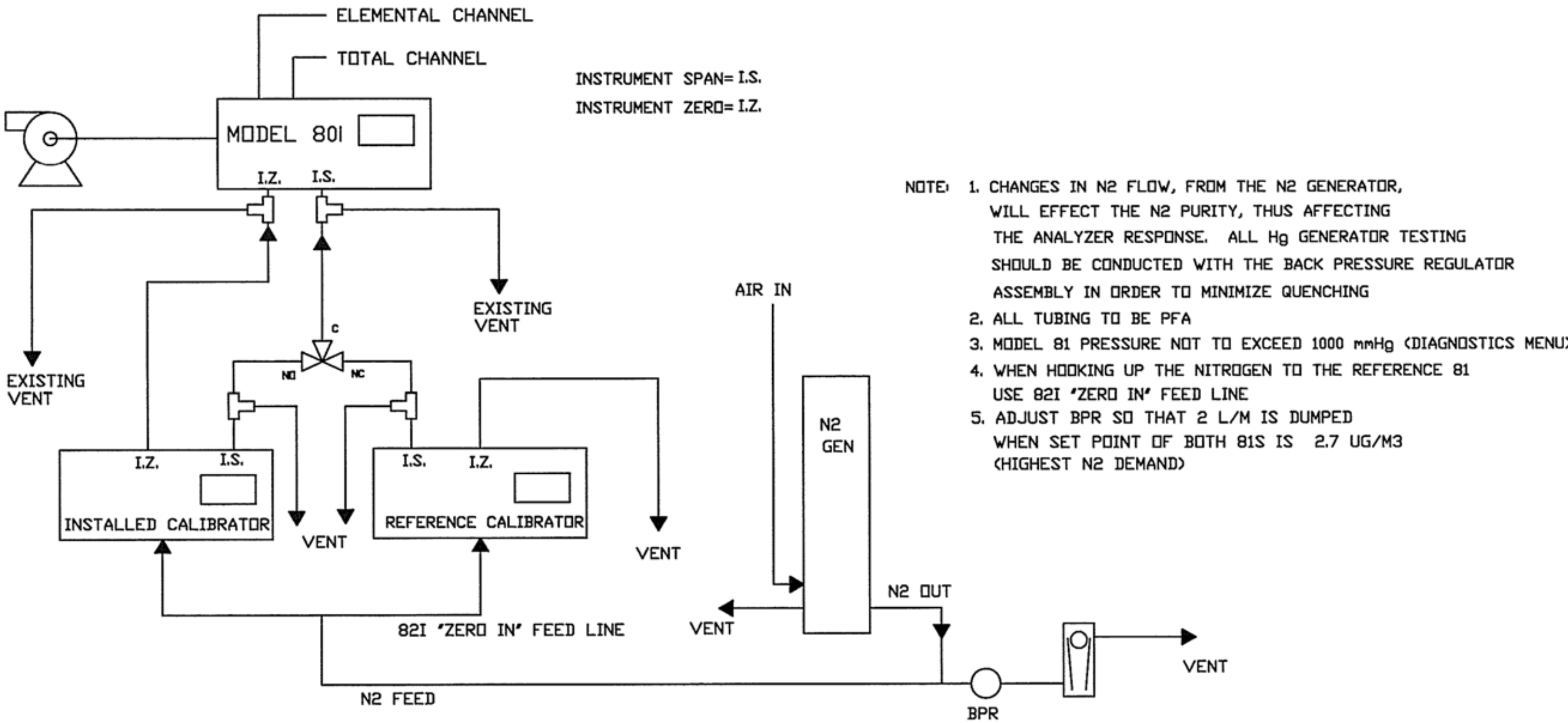
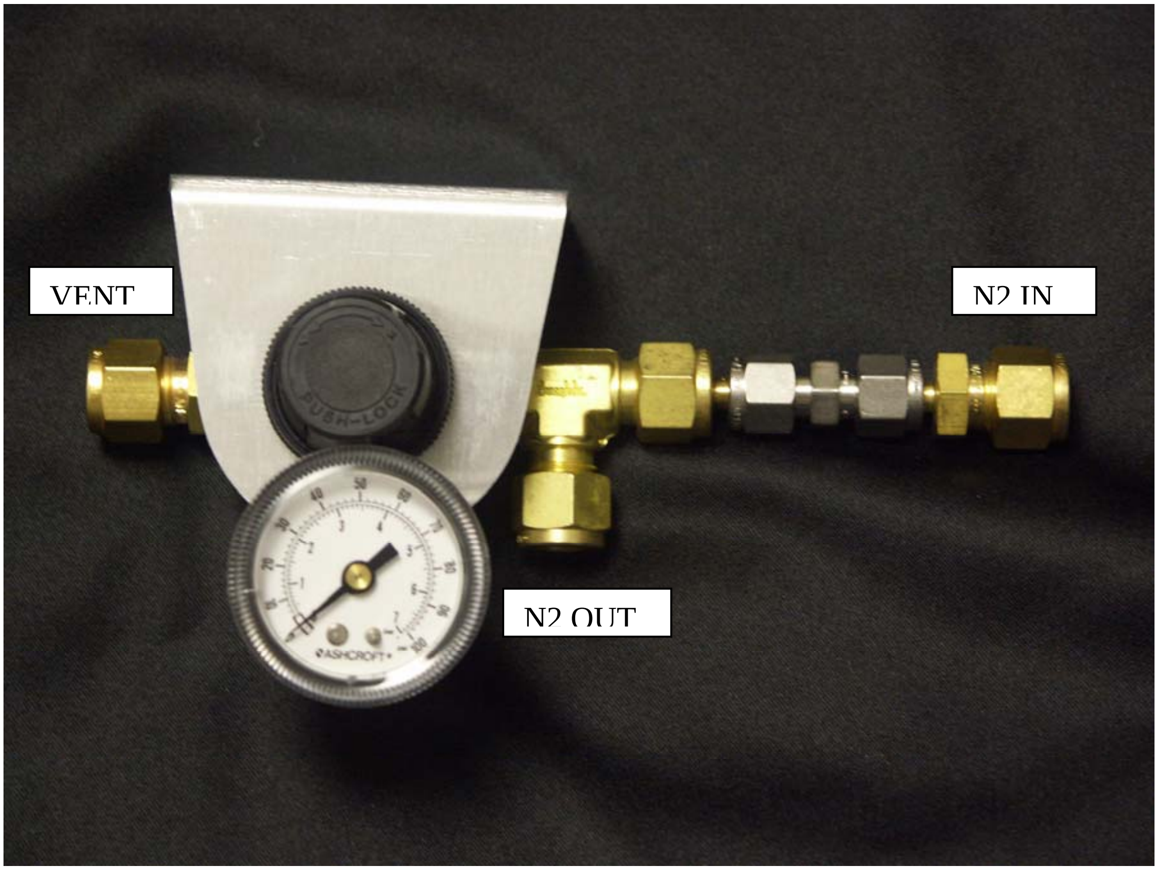


Figure 3 – Nitrogen Backpressure Regulator



APPENDIX B

Standard Operating Procedure for Tekran Calibrators

Task 2 Standard Operating Procedure
Laboratory Comparison of an Elemental Hg Candidate Calibrator Versus A Reference Calibrator

Tekran Procedure

Western Research Institute Version 5/30/08

This procedure is based on the 3/11/08 V 2.0 Task 1 field comparison procedure from Richard McRanie and Will Roberson, RMB Consulting

1. Introduction

The purpose of this document is to describe the setup and procedure for a series of tests designed to compare a candidate elemental Hg calibrator with a reference calibrator in a laboratory setting. The purpose of the tests is to evaluate the stability of the conventional "head space" calibrators that are an integral part of every Hg CEMS. The procedure described below is called a 3 X 3 test matrix, which means that three concentrations will be compared three times alternating between the reference calibrator and the candidate calibrator. A zero reading from the analyzer must be obtained at the beginning and end of each day to confirm that zero drift has not occurred. Concentration ratios between the reference and candidate calibrator at each concentration will be the result. The stability of the ratios over time will be the output of this project.

This procedure is accompanied by a data collection spreadsheet (with instructions) that should be filled out completely and submitted to RMB Consulting & Research, Inc. (RMB) as discussed in the data management section of this procedure. The actual data will be shared with anyone desiring to see it after a brief completeness review. All efforts should be made to provide complete data. Any questions should be addressed to Richard McRanie at RMB.

2. Discussion of Comparison Tests

The calibrator comparison tests will be done using a bracketing procedure employed by the National Institute of Science and Technology (NIST) when traceability is transferred from the NIST Prime calibrator to the Vendor Prime calibrators used by Tekran and Thermo. This procedure is called a bracketing process where the candidate calibrator is bracketed by the Reference Calibrator at each of three Hg concentrations: 2.7, 5.7, and 9.6 µg/m^3. The bracketing process at each concentration can be represented by the sequence shown in Table 1.

Reference Calibrator	Response 1
Candidate Calibrator	Response 2
Reference Calibrator	Response 3
Candidate Calibrator	Response 4
Reference Calibrator	Response 5
Candidate Calibrator	Response 6
Reference Calibrator	Response 7

Table 1 – Measurement Sequence, each concentration

The concentration of the Candidate Calibrator for each comparison is:

C_{Can1} = (Response 2 / (Avg Response 1,3)) * C_{Ref}
C_{Can2} = (Response 4 / (Avg Response 3,5)) * C_{Ref}
C_{Can3} = (Response 6 / (Avg Response 5,7)) * C_{Ref}

C_{CanI} is the calculated Candidate Calibrator concentration; C_{Ref} is the delivered concentration of the Reference Calibrator

The mean concentration of the candidate calibrator is:

C_{CanM} = Avg (C_{Can1}, C_{Can2}, C_{Can3})

The ratio between the Reference Calibrator and Candidate Calibrator is a useful value when tracking stability over time. This ratio can be calculated as:

R_{Can1} = Response 2 / (Avg Response 1,3)
R_{Can2} = Response 4 / (Avg Response 3,5)
R_{Can3} = Response 6 / (Avg Response 5,7)

The mean ratio is:

R_M = Avg (R_{Can1}, R_{Can2}, R_{Can3})

This simple bracketing technique is appropriate because the Hg calibrators and analyzers are very linear and the concentrations being compared are very similar in magnitude. The bracketing technique also compensates for drift and other variability in the performance of the calibrators and analyzers.

Note that the comparisons in Table 1 are only for a single concentration and three concentrations will be evaluated during each test. A spreadsheet will be provided to perform all calculations.

3. Setup and Operation Instructions for Tekran System

A flow schematic for the Tekran apparatus is shown in Figure 1 on the last page of this document. The schematic was designed to make it clear where all connections should be made for the comparison tests. The diagram shows a CEM controller which will not be present in the laboratory comparison tests. Mistakes have been made by prior researchers due to incorrect plumbing arrangements. It has become clear that the plumbing connections and operation instruction must be made perfectly clear and followed precisely to ensure that the system is being operated as intended by the manufactures. All tubing and fittings shall be 3/8" PFA Teflon except where explicitly stated in the connection procedure below.

It is critical that the calibrators not be deadheaded or have excessive backpressure. The atmospheric vents as shown in the respective drawings must be used. **(Note: ensure all vent caps on the Reference Calibrator have been removed prior to supplying air to calibrator.)** In addition, all manifold vents must be routed outside the laboratory so it does not become contaminated with Hg vapor. The detector/system must be in a standby or "**Idle**" condition during any disconnection or connection of fittings.

Pre-Installation Procedures and Checks

3.1 A kit containing Tee fittings and a toggle valve for the zero air may be used if a switched source of zero air is not conveniently available. The toggle valve, once installed, will allow any mobile calibration system to be reconnected very easily.

3.2 A venting source should be provided and left in place after the test.

3.3 All calibration sequences should be checked to ensure that both calibrators operate at the exact same settings for each concentration and zero concentration point.

4. Standard Calibration Points

The Tekran Model 3310 Reference and Candidate calibrators will normally be operated only at certain predefined standard concentration points. Each CEM span range will have a low, medium and high **L**, **M** and **H** concentration point (Plus a zero "**Z**" concentration point). The calibrator settings and theoretical calculated concentration output for each point of the 10 µg/m^3 span range are shown in Table 2.

Span Range µg/m^3 (20/760)	Point ID	Temp Setting dC	MFC2 Setting slpm	MFC1 Setting sccm	Theoretical Output Conc µg/m^3 (20/760)	EPA Target Conc (20/760)	Percent of Span (EPA)
10.0	P10Z	15.00	20.00	0.00	0.000	0.0	0.0%
	P10L	15.00	20.00	6.73	2.701	2.7	27.0%
	P10M	15.00	20.00	14.21	5.701	5.7	57.0%
	P10H	15.00	20.00	23.69	9.499	9.5	95.0%

Table 2. Tekran 3310 Calibration Settings for 10 µg/m^3 Span

For any certified Reference Calibrator, the actual measured (NIST traceable) output concentration is used in place of the theoretical concentration. These values are shown on a paper certificate provided with each certified analyzer as well as being stored within the calibrator's memory.

The Reference Calibrator will be certified by the manufacturer at the Span value being used at a CEM site. In almost all cases, this will be 10 $\mu g/m^3$. (Note : double check and make sure that both the reference calibrator and candidate calibrator are at the same set points)

5. Apparatus Setup Procedure

The arrangement described below allows both calibrators to be controlled by a notebook PC that is supplied with the calibrators and switching system. The switching valve between the calibrators is automatically controlled by a Model 1110 Control Unit and data from the analyzer is directly captured onto the notebook. See the system flow diagram in Figure 1 at the end of this document for additional plumbing details.

5.1 Connect the 4-port serial to USB converter to an available USB port in the notebook.

5.2 Remove any caps or plugs from the fittings on the rear of the calibrators, especially the vent connection. Failure to perform this step will result in damage to the calibrators.

5.3 Connect a source of Zero Air (> 60 PSI) to the Air Inlet of each calibrator using a ¼" OD section of Teflon line.

5.4 Connect the CAL OUT port of the Reference Calibrator to Port 0 of the Switcher using 3/8" Teflon line.

5.5 Connect the CAL OUT fitting of the Candidate Calibrator to Port 1 of the switcher using a length of 3/8" OD Teflon tubing.

5.6 Connect the 3/8" vent lines to a negative pressure vent or fume hood. Keep these lines as short as practicable and ensure that both lines are of equal length.

5.7 Run a 1/8" Teflon line from the Common port of the switcher to the SAMPLE inlet of the analyzer. Keep this line as short and direct as practical. (<6" if possible.)

5.8 Connect the Switcher unit to the Model 1110 Two Port Sampler Control Unit using the round, two conductor cable, supplied.

5.9 Connect power to the Reference Calibrator, the controller PC, and the switching unit power supply. Allow the calibrator to stabilize for 30 minutes before attempting to use it.

5.10 Double check and make sure all caps have been removed from the calibrator vents before proceeding to step 14.

5.11 Turn on the zero air supply to the Reference Calibrator.

5.12 Connect communication cables between the Controller PC, analyzer, and each calibrator. Port 1 of the Serial Interface is used for the 2537 Analyzer, Port 2 is used for the Candidate Calibrator, and Port 3 is used for the Reference Calibrator.

Model 1110 Two Port Switcher Notes:

The Model 1100 two port switcher should be pre-programmed and will automatically power up in Run mode. Refer to the Model 1110 User Manual for details. For completeness, the Port Switching program parameters are given below.

Init Cycles:	3	
On Cycles:	2	
Off Cycles:	2	
Flag:	1	
Ignore Cal:	0	
HW Pass:	253	(Used only to set keypad debounce parameter)
Debounce:	6	

When the switching valve is Off (Port 0), the CEM (Reference Calibrator) output is sent to the analyzer. When the switching valve is On (Port 1), the Candidate Calibrato*r* is measured. The captured analyzer data is flagged, showing which port was sampled.

6. Procedure for Testing System Connections

6.1 Ensure that all connections have been made and that the 2537 Analyzer and 3310 Calibrators are running properly. The notebook PC should be up and displaying the Windows desktop.

6.2 Ensure that the PC time is the same as the Model 2537 time. (The analyzer will usually remain on standard time year round, while the notebook may be on daylight time.) Set the PC to the analyzer's current time, to within a few seconds. This will ensure that concentration changes occur in sync with analyzer readings.

6.3 Start up the program labeled "**TekCap for Windows**" by double clicking on this icon. This program will capture the raw data from **Port 1** that is generated by the Model 2537 analyzer. The program will begin acquiring analyzer readings automatically. (Note: If the readings do not begin to appear automatically, click the "start" icon in the toolbar menu).

6.4 Start up the program labeled "**Reference Calibrator**" by double clicking on this icon. This program should identify the serial number of the Reference Calibrator attached to **Port 3** of the serial interface.

6.5 Start up the program labeled "**3310 User Calibrator**" by double clicking on this icon. The program should start up and identify the serial number of the Candidate Calibrator plugged into **Port 2** of the interface.

6.6 Ensure that the **Model 1100** valve controller is plugged in and operating. This unit should automatically start running when powered up. (The yellow **RUN** LED on the keypad is lit.) See the **Model 1100 User Manual** for details of the operation of this unit.

Notes:

- You can minimize any of the applications to allow viewing of the other calibrator's status or the latest captured data from the 2537. ***Do not inadvertently exit the applications.***

- If one of the calibrators is an older unit that has not yet been configured for "plug and play" operation, the program will prompt for the serial number of the unit. This entry is required one time only. See **Appendix A** in the User Manual for details.

- Newer versions of the s/w may indicate that certain operations are "**locked**", i.e. available only to service personnel. If a message to this effect appears, click on the lock icon and enter the **user name** and **password** listed on the front of the PC. (This operation is required only once per program provided that you do not exit and restart the application.)

7. Internal 2537 Detector Lamp Calibration

Before beginning the calibrator comparison sequences, Tekran recommends running the internal lamp calibration sequence first (See Internal Detector Calibration Procedure below). Once data capture and control of the calibrators has been established, the Model 2537 should have an internal detector lamp adjustment performed. This takes about 15 minutes and should be performed while the calibrators are stabilizing. The output of this calibration will be recorded onto the raw data file, but is not required (or suitable for) importing for the calibrator intercomparison.

7.1 Ensure that the Model 2537 is running in **Continuous** mode. Press the **<** or **>** arrow keys on the 2537 keypad until the **RUN:CALIB** screen is displayed. See the **Model 2537 User Manual** for details, if required.

7.2 Press **<esc>** immediately followed by **<1>**. The top line of the display should now display **KEYBD** immediately after the screen title.

7.3 The analyzer will complete its existing measurement cycle, and begin to perform a full internal lamp adjustment. This will require about 10 to 15 minutes.

7.4 The analyzer will display a "Calibration Report" to the **TekCap** screen. (See sample data report, next page.) The analyzer will then wait, perform a **Clean** operation and return to continuous monitoring mode. This requires a further 5 minutes.

7.5 Automated or manual calibrator operations can begin as soon as the first "**Cln A**" message appears on the **TekCap** screen. (Note: RMB recommends using the automated procedure detailed in the next section).

8. Calibrator Comparison Test Procedure

Once the lamp detector sequence has been completed, the system is ready to perform the calibrator comparison tests. If using the automatic mode (recommended), set the scheduler for each calibrator to perform the appropriate sequence at the same time as indicated by the Model 2537 "Next Sample". This time will be displayed on Model 2537 immediately following the calibration report which will be generated in the TekCap screen. This report is illustrated below.

```
-
CALIBRATION:  S/N:0257    H/W: 2.21   S/W: 1.11      07-12-23   14:24:58
-
ZERO:  A
Sample  :   150 sec  |  BlArea  :      0
Volume  :  1.20 l    |  BlCorr  :      0/l
Baseline: 0.108 V    |  PkMax   :  .000 V
Bl StDev:   .03 mv   |  PkWid   :    .0 sec
Start   : 07-12-23      14:15:00
-
ZERO:  B
Sample  :   150 sec  |  BlArea  :      0
Volume  :  1.20 l    |  BlCorr  :      0/l
Baseline: 0.108 V    |  PkMax   :  .000 V
Bl StDev:   .04 mv   |  PkWid   :    .0 sec
Start   : 07-12-23      14:17:30
-
SPAN:  A     SOURCE
Sample  :   150 sec  |  Area    :1658143
Volume  :  1.20 l    |  AdjArea :1658143 *
HgAmt   : 2050.4pg   |  RespFctr: 808702
Baseline: 0.108 V    |  PkMax   : 0.664 V
Bl StDev:   .05 mv   |  PkWid   :  16.2 sec
Start   : 07-12-23      14:20:00
-
SPAN:  B     SOURCE
Sample  :   150 sec  |  Area    :1652438
Volume  :  1.20 l    |  AdjArea :1652438 *
HgAmt   : 2050.4pg   |  RespFctr: 805920
Baseline: 0.107 V    |  PkMax   : 0.713 V
Bl StDev:   .05 mv   |  PkWid   :  14.8 sec
Start   : 07-12-23      14:22:30
```

8.1 Automated Mode Settings. *The Scheduler allows an entire calibration sequence to be initiated at some future time automatically and repeated at specified intervals if desired. The "Next Run" time should be set to the "Next Sample" time displayed on the front of Model 2537 which appears immediately following the completion of the Internal Detector Lamp Calibration sequence.*

Note: All timing is done with respect to the PC clock. Ensure that the PC clock is synchronized with the Model 2537 clock.

To set the schedule, use the following procedure:

8.1.1 Click the **Schedule** icon on the left side vertical toolbar.

8.1.2 If the sequence you want to run is already in the list, press **Edit** to bring up the detail screen. (Shown at right, above.) If the sequence you want is not on the list, press **Add** and select it from the list of available sequences to add it to the list.

8.1.3 Edit the **Next Run Date** and **Time** fields to start the calibrator at some convenient time in the future. The time must be far enough in the future to allow both calibrators to be set to start at the same time. Again, RMB recommends setting the start time to the "Next Sample" time shown on the front of the Model 2537 immediately following the completion of an Internal Lamp Calibration sequence.

8.1.4 If the above is not selected, a start time should be chosen so that the Reference calibrator will be sampled first. This can be done by examining the analyzer **Raw Data** capture screen. A pattern of **0,0,1,1,0,0** ... will be visible in the **Flag** field of successive data lines, where **0** indicates that the Reference is being monitored and a **1** indicates that the

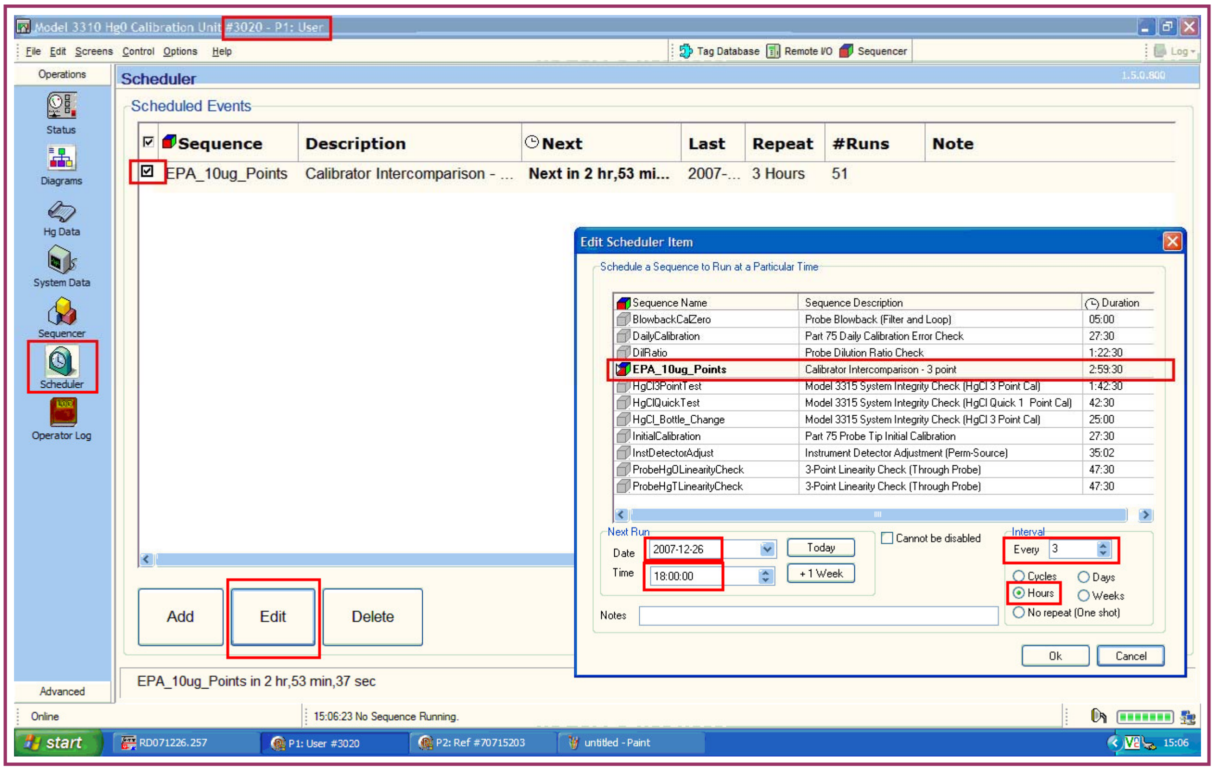
Candidate is being monitored.. Choose a start time 2.5 minutes before the time the next zero flag is scheduled to appear. (Note that the time flagged by the analyzer is the ***end*** time of the data reading's 2.5 minute integration period.)

8.1.5 If desired, select a repeat interval that is just slightly longer than the sequence duration. The repeat period is usually chosen to be the next highest hour. (e.g. if a sequence duration is 2:59:30, choose 3 hours as the repeat interval.

8.1.6 Click **OK** to save the scheduling changes.

8.1.7 Ensure that the **Active** checkbox for this sequence (and ***only*** this sequence) is checked.

8.1.8 Repeat for the other calibrator, ensuring that the schedule settings are identical with the first calibrator.



Notes and Troubleshooting

- In most cases, zero readings will decay quickly over time. Elevated zero readings are caused by residual mercury in the delivery system.
- If both calibrators give extended elevated zero values when compared to the internal (instrument) zero, it is an indication that the calibrators' zero air supply has mercury in it. This would mean changing the zero air canisters in the Model 1304 filter.
- Each output concentration for the Candidate calibrator must be generated and measured at least three times, with the readings bracketed by a Reference calibrator run at the same concentration. The various concentrations are usually produced in the following order. eg: **Z, L, M, H**

8.2 Manual Mode Operation: 3310 Calibrator as a Stand-Alone Unit

This mode of operation allows setting either calibrator to any setting and holding that output concentration indefinitely. Note that following the initial set-up this procedure can be automated for convenience (See next section "Automatic Mode"). This mode is used when using a Thermo analyzer to compare concentration outputs from Tekran and Thermo calibrators, for example. NOTE: NEED A STEP BY STEP PROCEDURE FOR ELECTRONICALLY LOGGING / RECORDING TEKRAN CALIBRATOR SETTINGS ONTO A PC SPREADSHEET WHEN IT IS USED AS A STAND-ALONE UNIT, FOR UPLOADING TO RMB SITE

The display example on the following page displays one of the available calibrator applications.

- The top title bar shows which calibrator the program is controlling.
- The vertical toolbar at left shows the available operations. (In this case, the **Sequencer** is being displayed.)
- The **lock** ion allows unlocking certain specialized program functions, if required.
- The **Sequencer** drop down menu contains the **Execute Step Now** instruction.
- The main screen displays the available sequences on the left side. Expanding a particular sequence and highlighting a step will show the detailed properties for that step on the right.

8.2.1 Click on the Reference Calibrator window and click on Sequencer.

8.2.2 Select the sequence under **EPA-Tests** called: "**EPA_10ug_Points**"

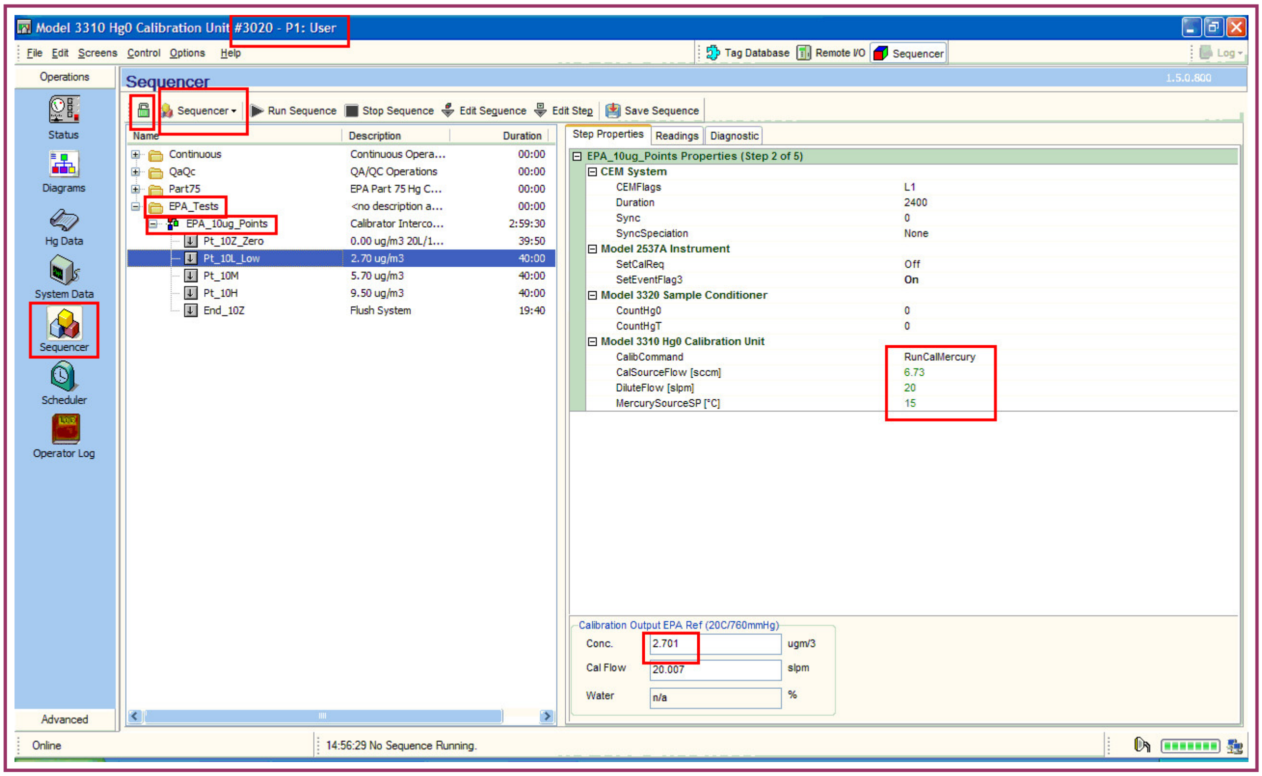
8.2.3 Select the step corresponding to the concentration you want. (eg: **Pt_10L**)

8.2.4 Under the **Sequencer** menu, press **Execute Step Now**. (This will execute and hold this concentration indefinitely.

8.2.5 Record the start time in your log book.

8.2.6 Repeat the above steps for the other calibrator.

8.2.7 When the comparison measurements are completed, click on the "**Default**" step to set both calibrators back into **Idle Mode**.



9. Data Capture and Reduction

Data from the analyzer is captured as an ASCII text file usually named **Tk**yymmdd**.txt**, (where **yymmdd** is the current date) and is stored in the folder named: **c:\TekData**. A shortcut on the screen points directly to this working directory. The first two characters and the three character extension can be varied on a site by site basis to keep the data files distinct. (Often users will use the lower three digits of the 2537 serial number as the extension.)

A sample of raw data captured from the analyzer is shown in the data table below. Note that, for low concentrations, the ng/m^3 values are displayed to three decimal points. At values over 1,000 ng/m^3, the decimals are suppressed. The analyzer displays concentrations in ng/m^3 specified at 0 ºC/760 mm Hg. The analyzer readings have also been calibrated using an internal permeation source, not either of the calibrators.

07-12-22	13:45:00	CONT	B	OK	0	150	1.20	0.108	.043	0.120	29965	32.061	
07-12-22	13:47:30	CONT	A	OK	0	150	1.20	0.108	.036	0.114	17180	18.567	Sampling Ambient Air
07-12-22	13:50:00	CONT	B	OK	1	150	1.20	0.108	.044	0.114	14500	15.514	
07-12-22	13:52:30	CONT	A	OK	1	150	1.20	0.108	.038	0.112	11212	12.110	
07-12-22	13:55:00	CONT	B	OK	0	150	1.20	0.108	.040	0.112	10501	11.243	
07-12-22	13:57:30	CONT	A	OK	0	150	1.20	0.108	.038	0.113	14525	15.688	
07-12-22	14:00:00	CONT	B	OK	1	150	1.20	0.108	.041	0.114	15192	16.255	Transition
07-12-22	14:02:30	CONT	A	OK	1	150	1.20	0.108	.037	0.113	12504	13.514	
07-12-22	14:05:00	CONT	B	OK	0	150	1.20	0.108	.036	0.111	6365	6.810	
07-12-22	14:07:30	CONT	A	OK	0	150	1.20	0.108	.036	0.110	4336	4.683	
07-12-22	14:10:00	CONT	B	OK	1	150	1.20	0.108	.032	0.110	5053	5.410	Sampling Zero Air
07-12-22	14:12:30	CONT	A	OK	1	150	1.20	0.108	.041	0.110	4539	4.902	
07-12-22	14:15:00	CONT	B	OK	0	150	1.20	0.108	.035	0.110	5324	5.697	
07-12-22	14:17:30	CONT	A	OK	0	150	1.20	0.108	.037	0.110	3759	4.062	
07-12-22	14:20:00	CONT	B	OK	1	150	1.20	0.108	.032	0.110	4040	4.323	
07-12-22	14:22:30	CONT	A	NP	1	150	1.20	0.108	.040	.000	0	.000	Transition
07-12-22	14:25:00	CONT	B	OK	0	150	1.20	0.108	.045	0.110	3902	4.178	
07-12-22	14:27:30	CONT	A	OK	0	150	1.20	0.108	.033	0.897	2473358	2672	
07-12-22	14:30:00	CONT	B	OK	1	150	1.20	0.108	.064	1.159	2915525	3119	
07-12-22	14:32:30	CONT	A	OK	1	150	1.20	0.108	.041	1.042	2943388	3181	Low Concentration
07-12-22	14:35:00	CONT	B	OK	0	150	1.20	0.108	.053	1.152	2883596	3085	
07-12-22	14:37:30	CONT	A	OK	0	150	1.20	0.108	.046	1.023	2884190	3115	
07-12-22	14:40:00	CONT	B	OK	1	150	1.20	0.108	.057	1.175	2941741	3150	
07-12-22	14:42:30	CONT	A	OK	1	150	1.20	0.108	.042	1.044	2950358	3187	
07-12-22	14:45:00	CONT	B	OK	0	150	1.20	0.108	.047	1.143	2860143	3060	Transition
07-12-22	14:47:30	CONT	A	OK	0	150	1.20	0.108	.047	1.027	2887009	3120	
07-12-22	14:50:00	CONT	B	OK	1	150	1.20	0.108	.058	1.171	2930951	3136	Medium Concentration
07-12-22	14:52:30	CONT	A	OK	1	150	1.20	0.108	.043	1.926	5778383	6241	
07-12-22	14:55:00	CONT	B	OK	0	150	1.20	0.108	.069	2.250	5973402	6396	
07-12-22	14:57:30	CONT	A	OK	0	150	1.20	0.108	.046	2.033	6124463	6615	
07-12-22	15:00:00	CONT	B	OK	1	150	1.20	0.108	.051	2.294	6078649	6504	
07-12-22	15:02:30	CONT	A	OK	1	150	1.20	0.108	.061	2.040	6141583	6638	Transition
07-12-22	15:05:00	CONT	B	OK	0	150	1.20	0.108	.060	2.291	6077206	6502	
07-12-22	15:07:30	CONT	A	OK	0	150	1.20	0.108	.055	2.026	6098021	6586	
07-12-22	15:10:00	CONT	B	OK	1	150	1.20	0.108	.066	2.294	6083710	6514	
07-12-22	15:12:30	CONT	A	OK	1	150	1.20	0.108	.062	2.031	6123835	6614	
07-12-22	15:15:00	CONT	B	OK	0	150	1.20	0.108	.091	2.279	6152239	6576	High Concentration
07-12-22	15:17:30	CONT	A	OK	0	150	1.20	0.108	.049	3.119	9630386	10408	
07-12-22	15:20:00	CONT	B	OK	1	150	1.20	0.108	0.104	3.671	10045e3	10748	
07-12-22	15:22:30	CONT	A	OK	1	150	1.20	0.108	.068	3.278	10167e3	10981	
07-12-22	15:25:00	CONT	B	OK	0	150	1.20	0.108	.090	3.648	9961378	10666	
07-12-22	15:27:30	CONT	A	OK	0	150	1.20	0.108	.065	3.220	10015e3	10817	Transition
07-12-22	15:30:00	CONT	B	OK	1	150	1.20	0.108	.096	3.688	10084e3	10790	
07-12-22	15:32:30	CONT	A	OK	1	150	1.20	0.108	.077	3.273	10167e3	10981	
07-12-22	15:35:00	CONT	B	OK	0	150	1.20	0.108	0.110	3.616	9897906	10597	Sampling Ambient Air
07-12-22	15:37:30	CONT	A	OK	0	150	1.20	0.108	.057	3.229	10007e3	10808	
07-12-22	15:40:00	CONT	B	OK	1	150	1.20	0.108	.097	3.697	10096e3	10802	
07-12-22	15:42:30	CONT	A	OK	1	150	1.20	0.108	.056	0.431	1016341	1098	
07-12-22	15:45:00	CONT	B	OK	0	150	1.20	0.108	.056	0.120	30134	32.241	
07-12-22	15:47:30	CONT	A	OK	0	150	1.20	0.108	.027	0.114	17322	18.709	Transition
07-12-22	15:50:00	CONT	B	OK	1	150	1.20	0.108	.042	0.114	14278	15.286	
07-12-22	15:52:30	CONT	A	OK	1	150	1.20	0.108	.030	0.112	10627	11.478	
07-12-22	15:55:00	CONT	B	OK	0	150	1.20	0.108	.042	0.112	10204	10.917	
07-12-22	15:57:30	CONT	A	OK	0	150	1.20	0.108	.038	0.114	14776	15.970	
07-12-22	16:00:00	CONT	B	OK	1	150	1.20	0.108	.040	0.114	15622	16.714	
07-12-22	16:02:30	CONT	A	OK	1	150	1.20	0.108	.043	0.113	12984	14.023	
07-12-22	16:05:00	CONT	B	OK	0	150	1.20	0.108	.036	0.111	6297	6.742	
07-12-22	16:07:30	CONT	A	OK	0	150	1.20	0.108	.039	0.110	4357	4.706	
07-12-22	16:10:00	CONT	B	OK	1	150	1.20	0.108	.039	0.110	5513	5.899	
07-12-22	16:12:30	CONT	A	OK	1	150	1.20	0.108	.041	0.110	3590	3.880	
07-12-22	16:15:00	CONT	B	OK	0	150	1.20	0.108	.040	0.110	5065	5.419	
07-12-22	16:17:30	CONT	A	OK	0	150	1.20	0.108	.031	0.110	3296	3.559	

This is the raw data that will be used to perform the ratio and uncertainty calculations. This data file will be imported into a spreadsheet provided by RMB. (See RMB spreadsheet for further details for handling the raw data files and spreadsheet operation) Once the data file has been generated, it may be transferred to another system or a portable drive using the procedure below:

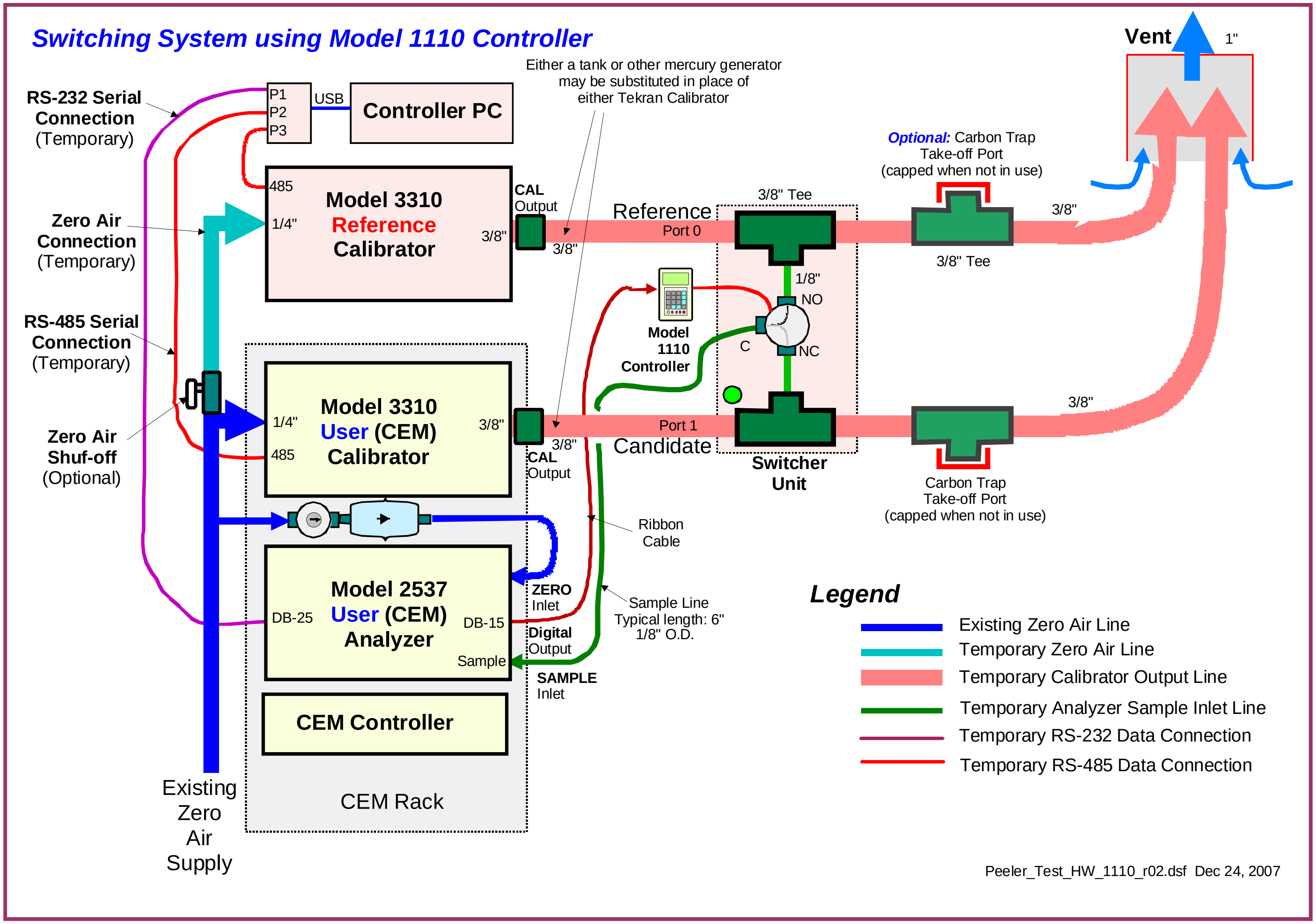
- Mount a USB drive on the PC.
- Click on the **Instrument Data** desktop shortcut. This will display the **c:\TekData** folder.
- Copy the required file onto the USB drive. ***The file copy may be done while the file is still being actively logged to.***

10. Data Management

The completed spreadsheet and extracted data files will be uploaded to RMB's FTP site as soon as possible after the tests are completed. The format for the upload files is date, plant name and unit number followed by ref (reference calibrator file), insta (candidate calibrator file), analy (analyzer file) or ss (spreadsheet file). Using a test performed on February 20, 2008 at Branch 1-2 for example, the reference calibrator file would be named 022008Branch1-2ref.dat and the spreadsheet file would be 022008Branch1-2ss.xls

The site URL is http://www.rmb-consulting.com/mercury.htm. You will note that there are two options – upload data and download data. Unique user names and passwords will be assigned to all participants for upload and download access. Each utility site and unit will be given a confidential code name by RMB prior to the start of the project and this code name will be used to identify all data on the download site. After the data are uploaded, RMB will ensure that all data are present in the proper format. RMB will also change the file names and transfer the files to the download FTP site where all project participants will have access to the data. The objective is to make the data available within 2-3 days after collection.

Figure 1 – Tekran Flow Schematic



APPENDIX C

August - September 2008 Nesting Analyses Summary Data

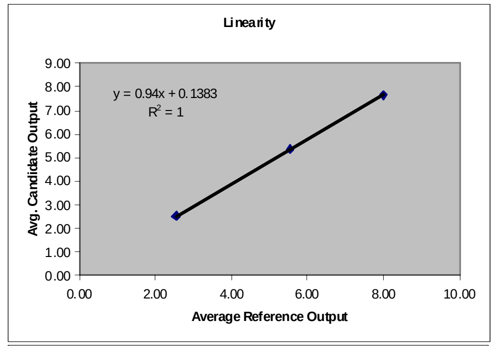
Minimum 12-hour Power and Air Flow

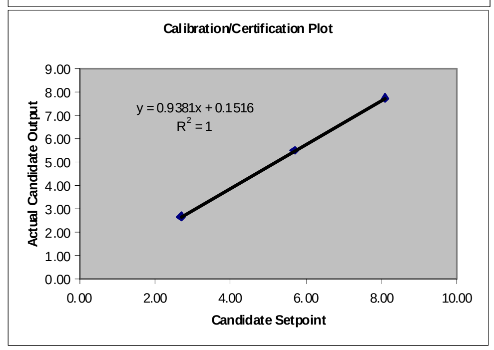
Results of Bracketing Experiments Between Thermo 03 and Thermo 02:

Comparison of WRI Thermo 03cal (REFERENCE) With WRI Thermo 02cal (CANDIDATE)
WRI Test # 118731, 8-19-2008

8.1µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Actual Cand.* Output
Zero Conce	0.01				
Thermo 03	8.02			8.10	7.75
Thermo 02	7.71	0.961			
Thermo 03	8.02				
Thermo 02	7.64	0.954	0.956		
Thermo 03	8.00				
Thermo 02	7.61	0.954			
Thermo 03	7.95				
5.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03	5.57			5.70	5.50
Thermo 02	5.38	0.968			
Thermo 03	5.55				
Thermo 02	5.35	0.964	0.965		
Thermo 03	5.55				
Thermo 02	5.34	0.965			
Thermo 03	5.52				
2.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03	2.57			2.70	2.68
Thermo 02	2.54	0.992			
Thermo 03	2.55				
Thermo 02	2.53	0.992	0.993		
Thermo 03	2.55				
Thermo 02	2.53	0.996			
Thermo 03	2.53				
Zero Conce	-0.05				

Cand. Output = Reference Conc. x Avg. Nested Ratio

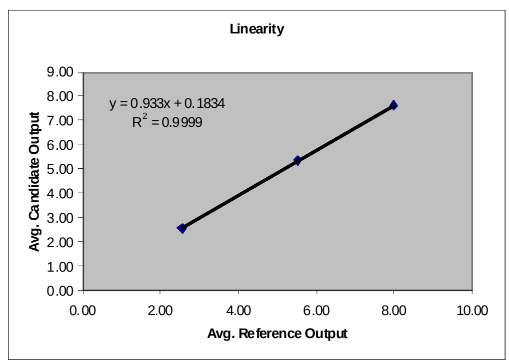


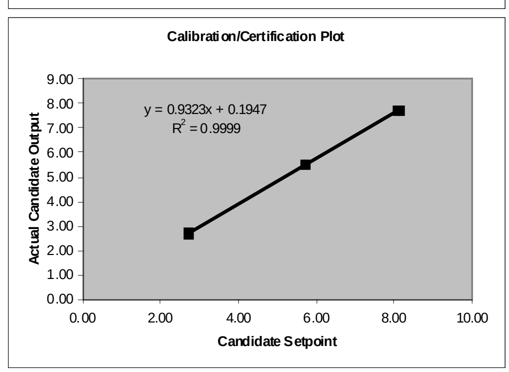


Comparison of WRI Thermo 03cal (REFERENCE) With WRI Thermo 02cal (CANDIDATE)
WRI Test # 118732, 8-19-2008

8.1µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Actual Cand.* Output
Zero Conce	0.02				
Thermo 03	8.04			8.10	7.73
Thermo 02	7.64	0.953			
Thermo 03	7.99				
Thermo 02	7.61	0.954	0.954		
Thermo 03	7.96				
Thermo 02	7.60	0.956			
Thermo 03	7.94				
5.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03	5.55			5.70	5.53
Thermo 02	5.38	0.971			
Thermo 03	5.53				
Thermo 02	5.36	0.970	0.971		
Thermo 03	5.52				
Thermo 02	5.36	0.972			
Thermo 03	5.51				
2.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03	2.56			2.70	2.70
Thermo 02	2.56	1.000			
Thermo 03	2.56				
Thermo 02	2.56	1.000	1.000		
Thermo 03	2.56				
Thermo 02	2.56	1.000			
Thermo 03	2.56				
Zero Conce	-0.01				

Cand. Output = Reference Conc. x Avg. Nested Ratio

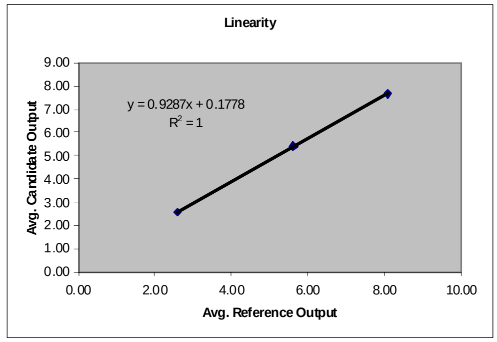


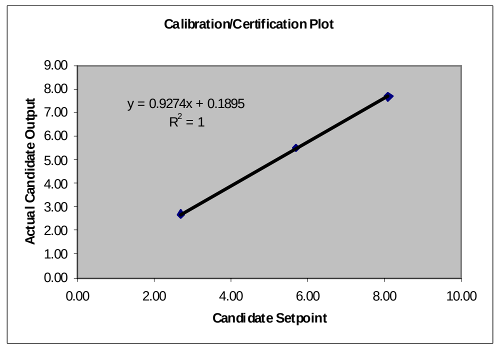


Comparison of WRI Thermo 03cal (REFERENCE) With WRI Thermo 02cal (CANDIDATE)
WRI Test # 118734, 8-21-2008

8.1µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Actual Cand.* Output
Zero Conce	0.03				
Thermo 03	8.10			8.10	7.69
Thermo 02	7.71	0.953			
Thermo 03	8.08				
Thermo 02	7.66	0.949	0.950		
Thermo 03	8.07				
Thermo 02	7.64	0.947			
Thermo 03	8.06				
5.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03	5.64			5.70	5.49
Thermo 02	5.42	0.963			
Thermo 03	5.62				
Thermo 02	5.41	0.963	0.963		
Thermo 03	5.61				
Thermo 02	5.40	0.964			
Thermo 03	5.59				
2.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03	2.62			2.70	2.69
Thermo 02	2.58	0.990			
Thermo 03	2.59				
Thermo 02	2.58	0.996	0.995		
Thermo 03	2.59				
Thermo 02	2.58	0.998			
Thermo 03	2.58				
Zero Conce	-0.01				

:and. Output = Reference Conc. x Avg. Nested Ratio

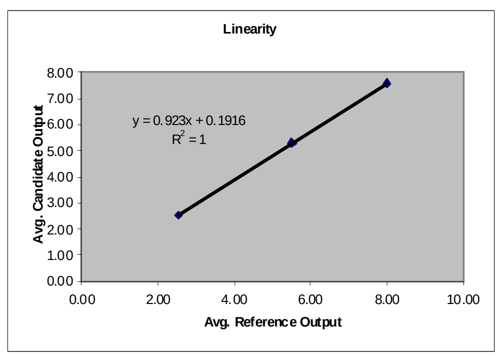


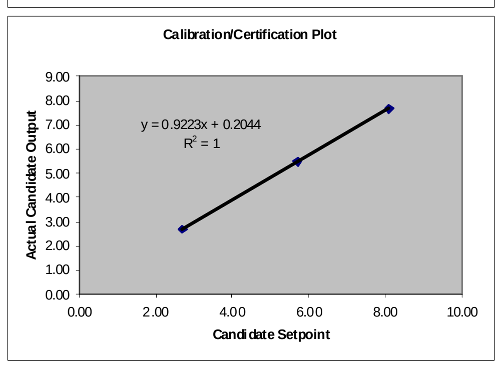


Comparison of WRI Thermo 03cal (REFERENCE) With WRI Thermo 02cal (CANDIDATE)
WRI Test # 118735, 8-21-2008

8.1µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Actual Cand.* Output
Zero Conce	-0.01				
Thermo 03	8.10			8.10	7.67
Thermo 02	7.71	0.957			
Thermo 03	8.02				
Thermo 02	7.52	0.941	0.946		
Thermo 03	7.97				
Thermo 02	7.50	0.942			
Thermo 03	7.95				
5.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03	5.54			5.70	5.48
Thermo 02	5.30	0.959			
Thermo 03	5.51				
Thermo 02	5.29	0.961	0.961		
Thermo 03	5.50				
Thermo 02	5.29	0.963			
Thermo 03	5.49				
2.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03	2.54			2.70	2.69
Thermo 02	2.53	0.996			
Thermo 03	2.54				
Thermo 02	2.53	0.996	0.995		
Thermo 03	2.54				
Thermo 02	2.52	0.994			
Thermo 03	2.53				
Zero Conce	-0.01				

:and. Output = Reference Conc. x Avg. Nested Ratio

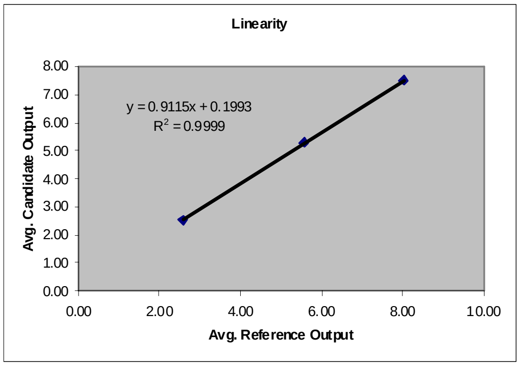


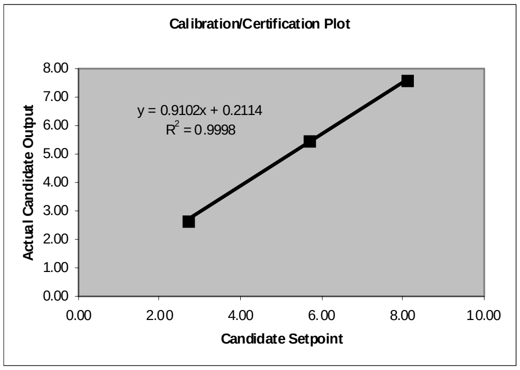


Comparison of WRI Thermo 03cal (REFERENCE) With WRI Thermo 02cal (CANDIDATE)
WRI Test # 118736, 8-22-2008

8.1µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Actual Cand.* Output
Zero Conce	0.03				
Thermo 03	8.07			8.10	7.56
Thermo 02	7.53	0.934			
Thermo 03	8.06				
Thermo 02	7.50	0.933	0.934		
Thermo 03	8.01				
Thermo 02	7.47	0.934			
Thermo 03	7.98				
5.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03	5.57			5.70	5.44
Thermo 02	5.31	0.954			
Thermo 03	5.56				
Thermo 02	5.30	0.953	0.954		
Thermo 03	5.56				
Thermo 02	5.30	0.953			
Thermo 03	5.56				
2.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03	2.59			2.70	2.65
Thermo 02	2.53	0.979			
Thermo 03	2.58				
Thermo 02	2.54	0.984	0.983		
Thermo 03	2.58				
Thermo 02	2.53	0.984			
Thermo 03	2.56				
Zero Conce	-0.01				

Cand. Output = Reference Conc. x Avg. Nested Ratio



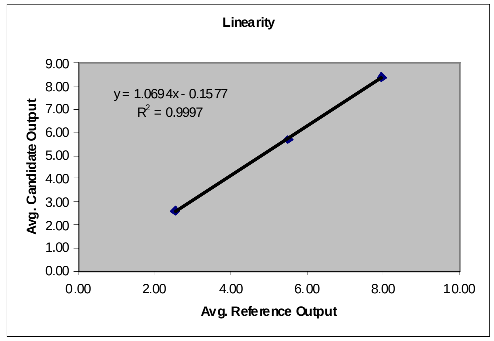


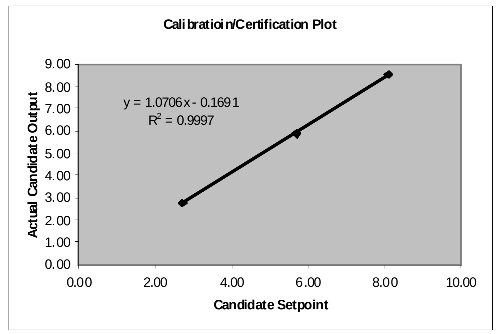
Results of Bracketing Experiments Between Thermo 03 and Thermo 04:

Comparison of WRI Thermo 03cal (REFERENCE) With WRI Thermo 04cal (CANDIDATE)
WRI Test # 118737, 8-26-2008

8.1µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero	0.02				
Thermo 03	8.02			8.10	8.54
Thermo 04	8.43	1.054			
Thermo 03	7.97				
Thermo 04	8.39	1.054	1.054		
Thermo 03	7.95				
Thermo 04	8.36	1.053			
Thermo 03	7.93				
5.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03	5.54			5.70	5.87
Thermo 04	5.71	1.033			
Thermo 03	5.52				
Thermo 04	5.68	1.030	1.031		
Thermo 03	5.51				
Thermo 04	5.66	1.029			
Thermo 03	5.49				
2.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03	2.56			2.70	2.75
Thermo 04	2.59	1.016			
Thermo 03	2.54				
Thermo 04	2.59	1.020	1.018		
Thermo 03	2.54				
Thermo 04	2.58	1.018			
Thermo 03	2.53				
Zero	-0.02				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*

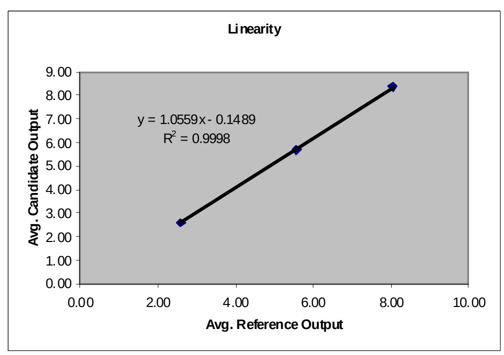


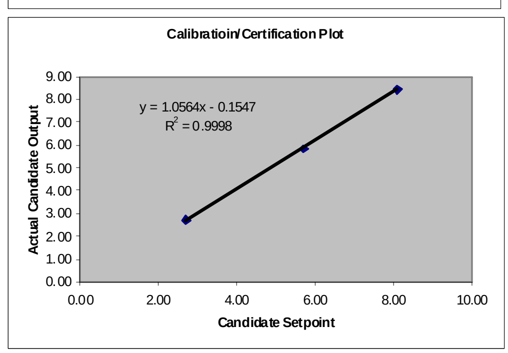


Comparison of WRI Thermo 03cal (REFERENCE) With WRI Thermo 04cal (CANDIDATE)
WRI Test # 118738, 8-26-2008

8.1µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero	0				
Thermo 03	8.05			8.10	8.43
Thermo 04	8.36	1.039			
Thermo 03	8.05				
Thermo 04	8.38	1.042	1.041		
Thermo 03	8.04				
Thermo 04	8.37	1.042			
Thermo 03	8.03				
5.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03	5.59			5.70	5.82
Thermo 04	5.69	1.020			
Thermo 03	5.57				
Thermo 04	5.68	1.020	1.021		
Thermo 03	5.57				
Thermo 04	5.69	1.022			
Thermo 03	5.56				
2.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03	2.59			2.70	2.72
Thermo 04	2.61	1.010			
Thermo 03	2.58				
Thermo 04	2.6	1.008	1.007		
Thermo 03	2.58				
Thermo 04	2.59	1.004			
Thermo 03	2.58				
Zero	-0.01				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*

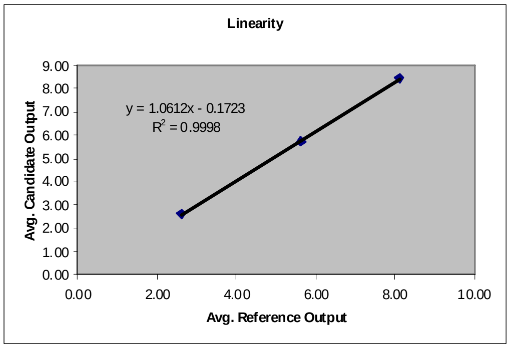


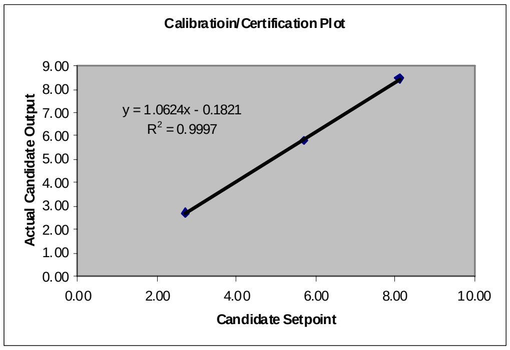


Comparison of WRI Thermo 03cal (REFERENCE) With WRI Thermo 04cal (CANDIDATE)
WRI Test # 118739, 8-27-2008

8.1µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero	0.02				
Thermo 03	8.13			8.10	8.45
Thermo 04	8.47	1.043			
Thermo 03	8.11				
Thermo 04	8.46	1.045	1.044		
Thermo 03	8.08				
Thermo 04	8.42	1.043			
Thermo 03	8.07				
5.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03	5.64			5.70	5.82
Thermo 04	5.77	1.024			
Thermo 03	5.63				
Thermo 04	5.75	1.021	1.021		
Thermo 03	5.63				
Thermo 04	5.72	1.018			
Thermo 03	5.61				
2.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03	2.61			2.70	2.71
Thermo 04	2.62	1.004			
Thermo 03	2.61				
Thermo 04	2.61	1.002	1.004		
Thermo 03	2.6				
Thermo 04	2.61	1.006			
Thermo 03	2.59				
Zero	0.02				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*



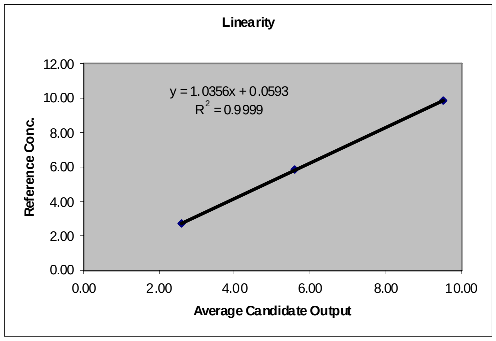


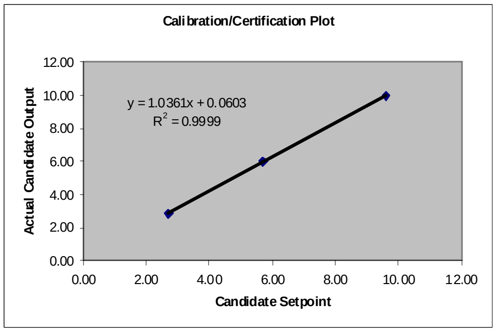
Results of Bracketing Experiments Between Thermo 03 and Tekran 03:

Comparison of WRI Thermo 03cal (REFERENCE) With WRI Tekran 03cal (CANDIDATE)
WRI Test # 118741, 9-03-2008

9.6 µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero Conce	0.01				
Thermo 03	9.58			9.60	9.99
Tekran 03	9.92	1.039			
Thermo 03	9.51				
Tekran 03	9.91	1.043	1.041		
Thermo 03	9.50				
Tekran 03	9.88	1.041			
Thermo 03	9.49				
5.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03	5.60			5.70	6.00
Tekran 03	5.88	1.051			
Thermo 03	5.59				
Tekran 03	5.87	1.052	1.053		
Thermo 03	5.57				
Tekran 03	5.88	1.055			
Thermo 03	5.58				
2.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03	2.61			2.70	2.84
Tekran 03	2.73	1.048			
Thermo 03	2.60				
Tekran 03	2.73	1.052	1.051		
Thermo 03	2.59				
Tekran 03	2.73	1.054			
Thermo 03	2.59				
Zero Conce	-0.01				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*

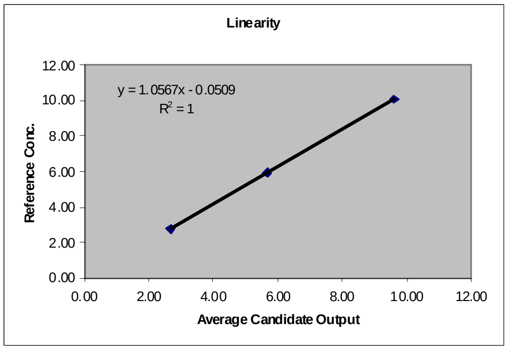


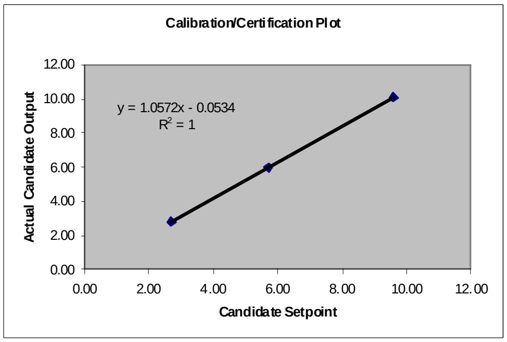


Comparison of WRI Thermo 03cal (REFERENCE) With WRI Tekran 03cal (CANDIDATE)
WRI Test # 118742, 9-04-2008

9.6 µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero Conce	0.01				
Thermo 03	9.62			9.60	10.09
Tekran 03	10.11	1.052			
Thermo 03	9.60				
Tekran 03	10.10	1.052	1.052		
Thermo 03	9.60				
Tekran 03	10.09	1.050			
Thermo 03	9.61				
5.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03	5.65			5.70	5.98
Tekran 03	5.94	1.050			
Thermo 03	5.66				
Tekran 03	5.93	1.048	1.048		
Thermo 03	5.66				
Tekran 03	5.93	1.047			
Thermo 03	5.67				
2.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03	2.67			2.70	2.80
Tekran 03	2.77	1.039			
Thermo 03	2.66				
Tekran 03	2.76	1.036	1.037		
Thermo 03	2.67				
Tekran 03	2.76	1.036			
Thermo 03	2.66				
Zero Conce	0.02				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*

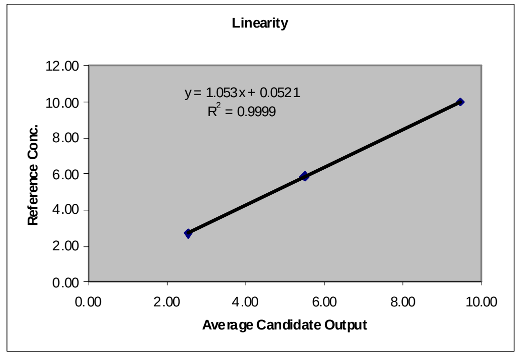


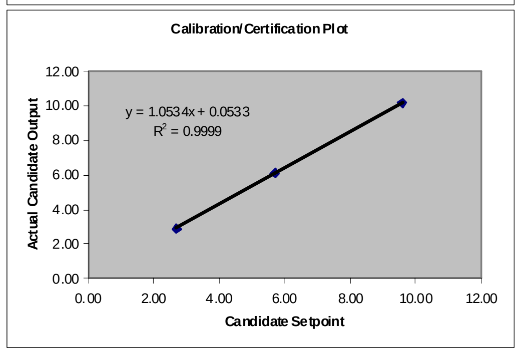


Comparison of WRI Thermo 03cal (REFERENCE) With WRI Tekran 03cal (CANDIDATE)
WRI Test # 118743, 9-05-2008

9.6 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**	**Reference Conc.**	**Act. Cand.* Output**
Zero Conce	0.01				
Thermo 03	9.55			9.60	10.15
Tekran 03	10.02	1.053			
Thermo 03	9.49				
Tekran 03	10.01	1.058	1.058		
Thermo 03	9.44				
Tekran 03	10.02	1.063			
Thermo 03	9.42				
5.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03	5.52			5.70	6.09
Tekran 03	5.87	1.064			
Thermo 03	5.51				
Tekran 03	5.88	1.069	1.068		
Thermo 03	5.49				
Tekran 03	5.88	1.071			
Thermo 03	5.49				
2.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03	2.55			2.70	2.88
Tekran 03	2.72	1.067			
Thermo 03	2.55				
Tekran 03	2.72	1.067	1.067		
Thermo 03	2.55				
Tekran 03	2.72	1.067			
Thermo 03	2.55				
Zero Conce	0.00				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*

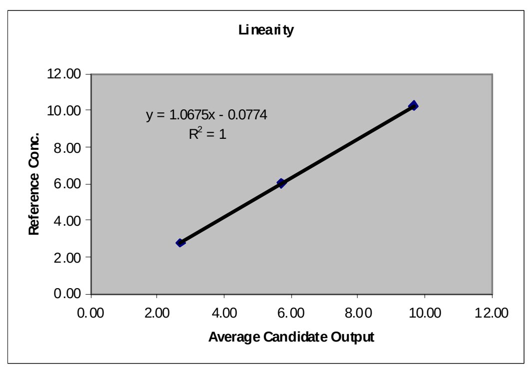


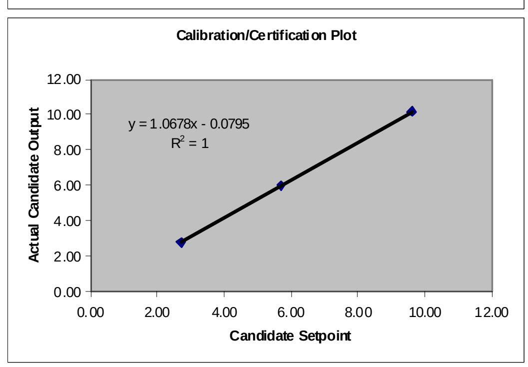


Comparison of WRI Thermo 03cal (REFERENCE) With WRI Tekran 03cal (CANDIDATE)
WRI Test # 118744, 9-05-2008

9.6 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**	**Reference Conc.**	**Act. Cand.* Output**
Zero Conce	0.01				
Thermo 03	9.65			9.60	10.17
Tekran 03	10.30	1.067			
Thermo 03	9.65				
Tekran 03	10.21	1.057	1.059		
Thermo 03	9.67				
Tekran 03	10.19	1.053			
Thermo 03	9.68				
5.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03	5.71			5.70	6.02
Tekran 03	6.03	1.055			
Thermo 03	5.72				
Tekran 03	6.05	1.058	1.055		
Thermo 03	5.72				
Tekran 03	6.02	1.053			
Thermo 03	5.71				
2.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03	2.68			2.70	2.80
Tekran 03	2.78	1.037			
Thermo 03	2.68				
Tekran 03	2.78	1.035	1.037		
Thermo 03	2.69				
Tekran 03	2.79	1.037			
Thermo 03	2.69				
Zero Conce	0.01				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*





APPENDIX D

December 2008 Nesting Analyses Summary Data

Results of Bracketing Experiments Between Thermo 03a and Thermo 02

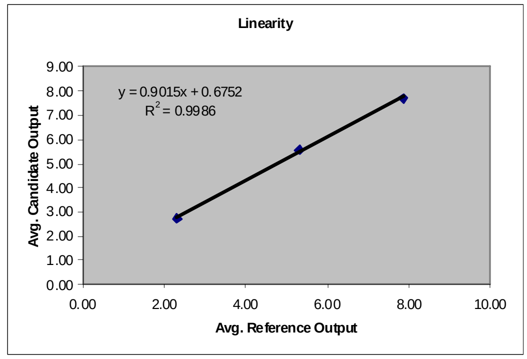
Comparison of WRI Thermo 03a cal (REFERENCE) With WRI Thermo 02 cal (CANDIDATE)
WRI Test # 118751, 12-11-2008

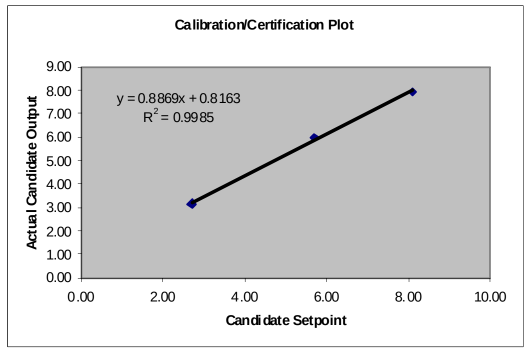
8.1µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero	-0.03				
Thermo 03a	7.93			8.10	7.94
Thermo 02	7.73	0.978			
Thermo 03a	7.88				
Thermo 02	7.70	0.980	0.980		
Thermo 03a	7.84				
Thermo 02	7.70	0.983			
Thermo 03a	7.82				

5.7µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio		
Thermo 03a	5.34			5.70	5.98
Thermo 02	5.58	1.046			
Thermo 03a	5.33				
Thermo 02	5.58	1.050	1.049		
Thermo 03a	5.30				
Thermo 02	5.58	1.051			
Thermo 03a	5.32				

2.7µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio		
Thermo 03a	2.34			2.70	3.16
Thermo 02	2.73	1.172			
Thermo 03a	2.32				
Thermo 02	2.72	1.172	1.172		
Thermo 03a	2.32				
Thermo 02	2.71	1.171			
Thermo 03a	2.31				
Zero	-0.05				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*





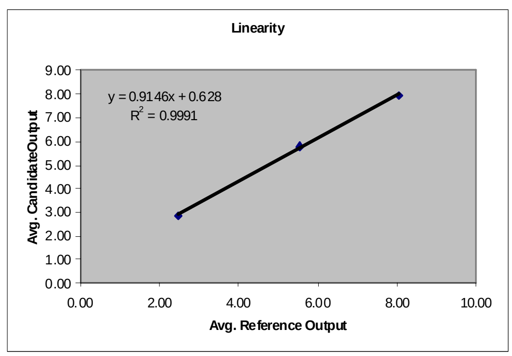
Comparison of WRI Thermo 03a cal (REFERENCE) With WRI Thermo 02 cal (CANDIDATE)
WRI Test # 118752, 12-11-2008

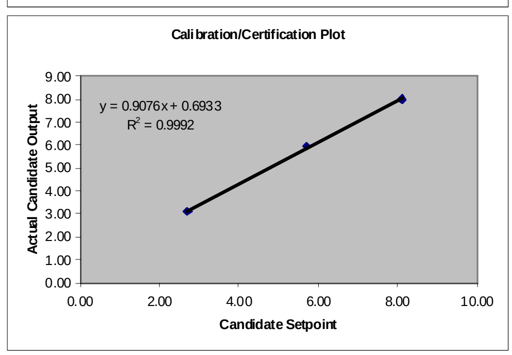
8.1µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero	-0.05				
Thermo 03a	8.11			8.10	8.00
Thermo 02	7.96	0.986			
Thermo 03a	8.04				
Thermo 02	7.95	0.991	0.988		
Thermo 03a	8.00				
Thermo 02	7.92	0.986			
Thermo 03a	8.06				

5.7µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio		
Thermo 03a	5.51			5.70	5.95
Thermo 02	5.78	1.045			
Thermo 03a	5.55				
Thermo 02	5.79	1.041	1.043		
Thermo 03a	5.57				
Thermo 02	5.80	1.043			
Thermo 03a	5.55				

2.7µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio		
Thermo 03a	2.48			2.70	3.11
Thermo 02	2.86	1.153			
Thermo 03a	2.48				
Thermo 02	2.85	1.149	1.151		
Thermo 03a	2.48				
Thermo 02	2.85	1.152			
Thermo 03a	2.47				
Zero	-0.01				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*



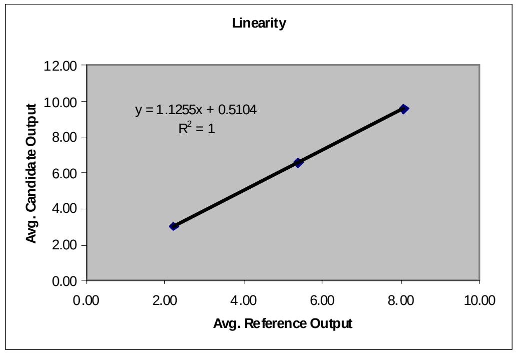


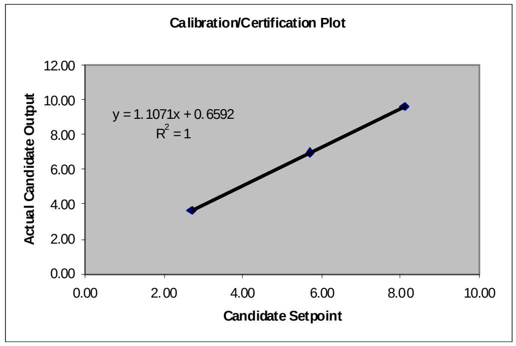
Results of Bracketing Experiments Between Thermo 03a and Thermo 04a:

Comparison of WRI Thermo 03a cal (REFERENCE) With WRI Thermo 04a cal (CANDIDATE)
WRI Test # 118755, 12-12-2008

8.1µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero	0.01				
Thermo 03a	8.15			8.10	9.63
Thermo 04a	9.61	1.186			
Thermo 03a	8.06				
Thermo 04a	9.60	1.191	1.189		
Thermo 03a	8.06				
Thermo 04a	9.59	1.191			
Thermo 03a	8.05				
5.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03a	5.40			5.70	6.96
Thermo 04a	6.59	1.220			
Thermo 03a	5.40				
Thermo 04a	6.60	1.222	1.221		
Thermo 03a	5.40				
Thermo 04a	6.58	1.221			
Thermo 03a	5.38				
2.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03a	2.23			2.70	3.65
Thermo 04a	3.02	1.354			
Thermo 03a	2.23				
Thermo 04a	3.02	1.354	1.353		
Thermo 03a	2.23				
Thermo 04a	3.01	1.350			
Thermo 03a	2.23				
Zero	0.04				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*

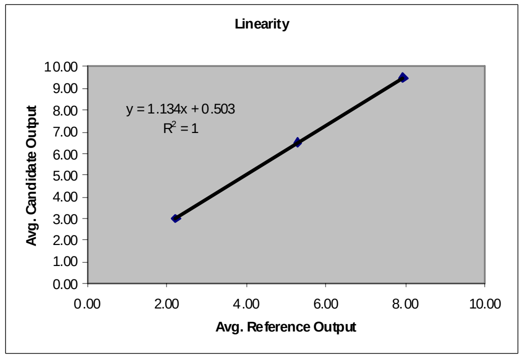


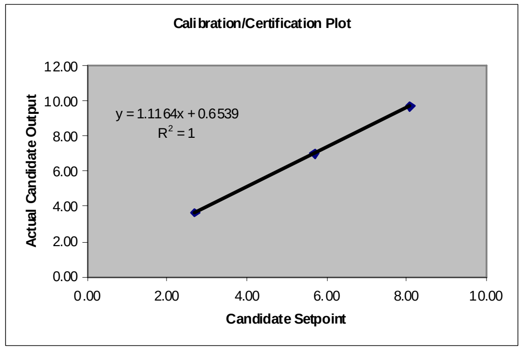


Comparison of WRI Thermo 03a cal (REFERENCE) With WRI Thermo 04a cal (CANDIDATE)
WRI Test # 118757, 12-16-2008

8.1µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero	-0.03				
Thermo 03a	7.98			8.10	9.69
Thermo 04a	9.50	1.193			
Thermo 03a	7.95				
Thermo 04a	9.50	1.198	1.197		
Thermo 03a	7.91				
Thermo 04a	9.49	1.200			
Thermo 03a	7.91				
5.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03a	5.31			5.70	7.02
Thermo 04a	6.53	1.233			
Thermo 03a	5.28				
Thermo 04a	6.51	1.231	1.232		
Thermo 03a	5.30				
Thermo 04a	6.51	1.232			
Thermo 03a	5.27				
2.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03a	2.23			2.70	3.67
Thermo 04a	3.02	1.357			
Thermo 03a	2.22				
Thermo 04a	3.01	1.356	1.358		
Thermo 03a	2.22				
Thermo 04a	3.02	1.360			
Thermo 03a	2.22				
Zero	-0.04				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*



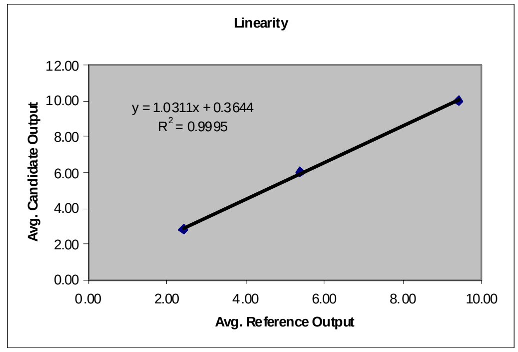


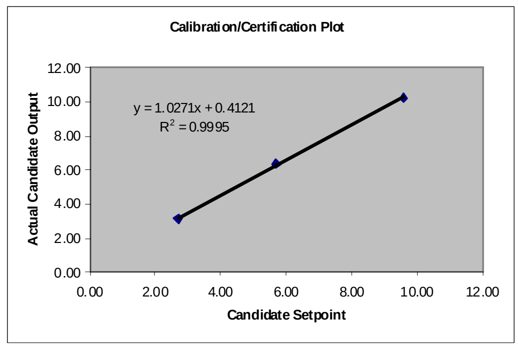
Results of Bracketing Experiments Between Thermo 03a and Tekran 03:

Comparison of WRI Thermo 03a cal (REFERENCE) With WRI Tekran 03 cal (CANDIDATE)
WRI Test # 118749, 12-05-2008

9.6µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero	0.00				
Thermo 03a	9.45			9.60	10.23
Tekran 03	10.03	1.064			
Thermo 03a	9.41				
Tekran 03	10.02	1.065	1.066		
Thermo 03a	9.40				
Tekran 03	10.03	1.069			
Thermo 03a	9.37				
5.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03a	5.41			5.70	6.36
Tekran 03	6.01	1.112			
Thermo 03a	5.40				
Tekran 03	6.02	1.116	1.115		
Thermo 03a	5.39				
Tekran 03	6.02	1.118			
Thermo 03a	5.38				
2.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03a	2.42			2.70	3.13
Tekran 03	2.81	1.161			
Thermo 03a	2.42				
Tekran 03	2.80	1.159	1.161		
Thermo 03a	2.41				
Tekran 03	2.80	1.162			
Thermo 03a	2.41				
Zero	-0.04				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*

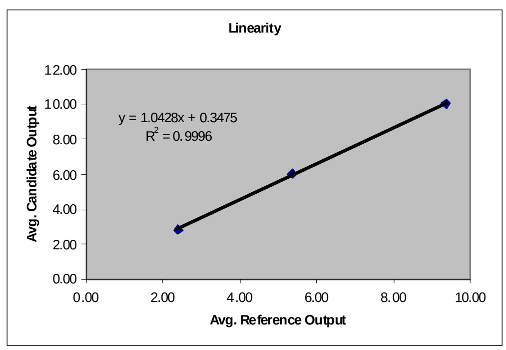


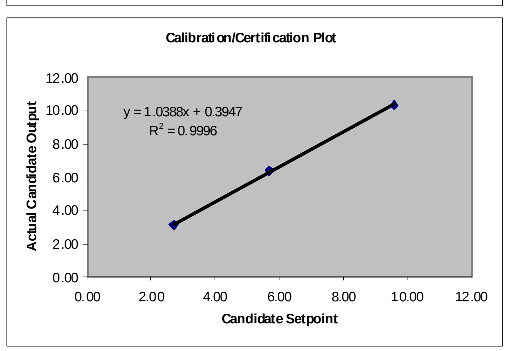


Comparison of WRI Thermo 03a cal (REFERENCE) With WRI Tekran 03 cal (CANDIDATE)
WRI Test # 118750, 12-05-2008

9.6µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero	-0.04				
Thermo 03a	9.39			9.60	10.33
Tekran 03	10.07	1.074			
Thermo 03a	9.37				
Tekran 03	10.07	1.075	1.076		
Thermo 03a	9.36				
Tekran 03	10.09	1.080			
Thermo 03a	9.33				
5.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03a	5.38			5.70	6.40
Tekran 03	6.02	1.121			
Thermo 03a	5.36				
Tekran 03	6.03	1.125	1.123		
Thermo 03a	5.36				
Tekran 03	6.02	1.122			
Thermo 03a	5.37				
2.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03a	2.40			2.70	3.15
Tekran 03	2.81	1.168			
Thermo 03a	2.41				
Tekran 03	2.81	1.166	1.168		
Thermo 03a	2.41				
Tekran 03	2.81	1.168			
Thermo 03a	2.40				
Zero	-0.04				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*



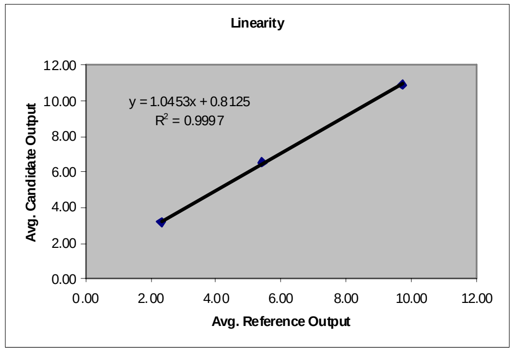


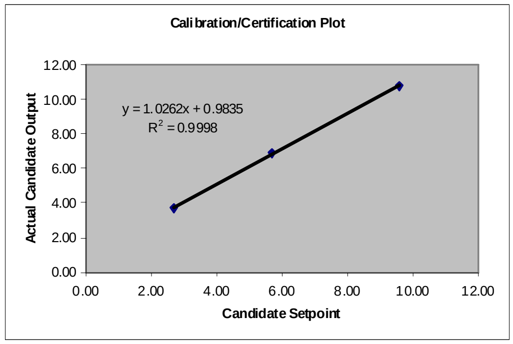
Results of Bracketing Experiments Between Thermo 03a and Tekran 04:

Comparison of WRI Thermo 03a cal (REFERENCE) With WRI Tekran 04 cal (CANDIDATE)
WRI Test # 118753, 12-11-2008

9.6µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero	0.00				
Thermo 03a	9.74			9.60	10.81
Tekran 04	10.96	1.127			
Thermo 03a	9.71				
Tekran 04	10.94	1.128	1.126		
Thermo 03a	9.69				
Tekran 04	10.87	1.122			
Thermo 03a	9.68				
5.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03a	5.44			5.70	6.90
Tekran 04	6.55	1.205			
Thermo 03a	5.43				
Tekran 04	6.57	1.211	1.210		
Thermo 03a	5.42				
Tekran 04	6.57	1.213			
Thermo 03a	5.41				
2.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03a	2.33			2.70	3.72
Tekran 04	3.17	1.363			
Thermo 03a	2.32				
Tekran 04	3.19	1.381	1.377		
Thermo 03a	2.30				
Tekran 04	3.19	1.387			
Thermo 03a	2.30				
Zero	-0.02				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*

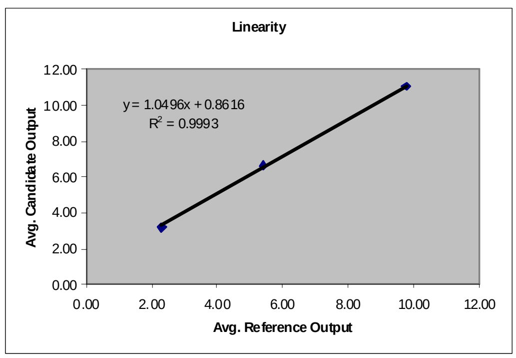


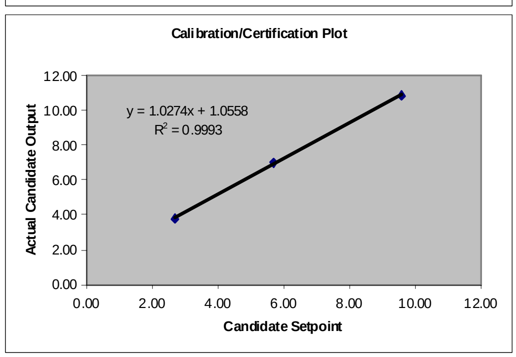


Comparison of WRI Thermo 03a cal (REFERENCE) With WRI Tekran 04 cal (CANDIDATE)
WRI Test # 118754, 12-11-2008

9.6 µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero	-0.02				
Thermo 03a	9.77			9.60	10.87
Tekran 04	11.06	1.132			
Thermo 03a	9.77				
Tekran 04	11.08	1.134	1.133		
Thermo 03a	9.77				
Tekran 04	11.04	1.132			
Thermo 03a	9.74				
5.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03a	5.4			5.70	7.02
Tekran 04	6.64	1.230			
Thermo 03a	5.4				
Tekran 04	6.64	1.232	1.231		
Thermo 03a	5.38				
Tekran 04	6.64	1.232			
Thermo 03a	5.4				
2.7µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Thermo 03a	2.29			2.70	3.77
Tekran 04	3.2	1.400			
Thermo 03a	2.28				
Tekran 04	3.2	1.397	1.396		
Thermo 03a	2.3				
Tekran 04	3.2	1.391			
Thermo 03a	2.3				
Zero	-0.03				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*





APPENDIX E

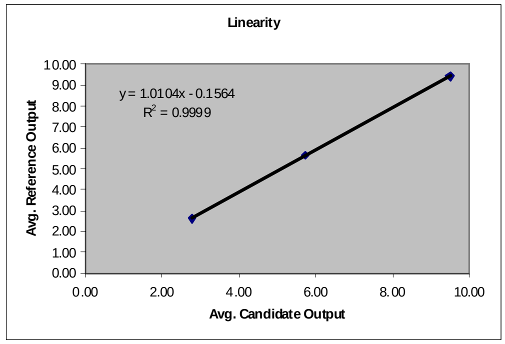
January 2009 Nesting Analyses Summary Data

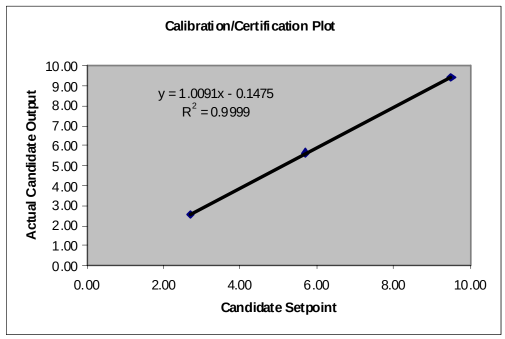
Results of Bracketing Experiments Between Tekran04 and Tekran 03:

Comparison of WRI Tekran 04cal (REFERENCE) With WRI Tekran 03cal (CANDIDATE)
WRI Test # 118759, 01/23/2009

9.6 µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero					
Tekran 04	9.462			9.50	9.426
Tekran 03	9.408	0.991			
Tekran 04	9.530				
Tekran 03	9.466	0.995	0.992		
Tekran 04	9.501				
Tekran 03	9.426	0.991			
Tekran 04	9.522				
5.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	5.730			5.70	5.634
Tekran 03	5.659	0.988			
Tekran 04	5.726				
Tekran 03	5.662	0.990	0.988		
Tekran 04	5.710				
Tekran 03	5.650	0.987			
Tekran 04	5.738				
2.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	2.784			2.70	2.561
Tekran 03	2.623	0.942			
Tekran 04	2.782				
Tekran 03	2.643	0.951	0.948		
Tekran 04	2.774				
Tekran 03	2.638	0.951			
Tekran 04	2.772				
Zero	-0.020				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*

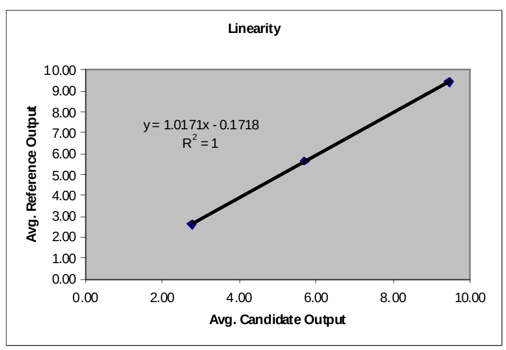


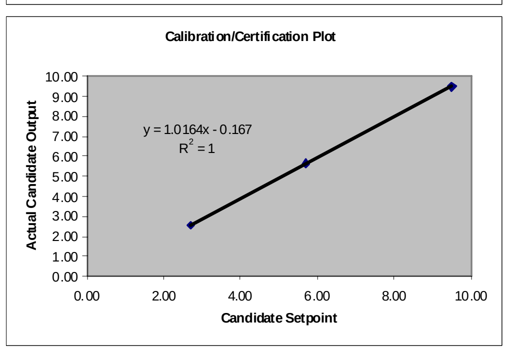


Comparison of WRI Tekran 04cal (REFERENCE) With WRI Tekran 03cal (CANDIDATE)
WRI Test # 118760, 01/23/2009

9.6 µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero					
Tekran 04	9.451			9.50	9.478
Tekran 03	9.423	0.997			
Tekran 04	9.454				
Tekran 03	9.431	0.997	0.998		
Tekran 04	9.466				
Tekran 03	9.456	0.999			
Tekran 04	9.458				
5.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	5.693			5.70	5.649
Tekran 03	5.651	0.991			
Tekran 04	5.708				
Tekran 03	5.659	0.991	0.991		
Tekran 04	5.711				
Tekran 03	5.660	0.991			
Tekran 04	5.712				
2.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	2.763			2.70	2.564
Tekran 03	2.627	0.950			
Tekran 04	2.770				
Tekran 03	2.636	0.951	0.950		
Tekran 04	2.776				
Tekran 03	2.631	0.949			
Tekran 04	2.770				
Zero	0.001				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*



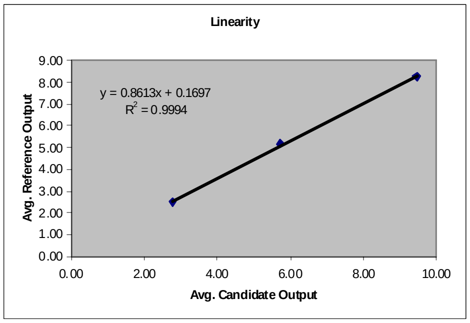


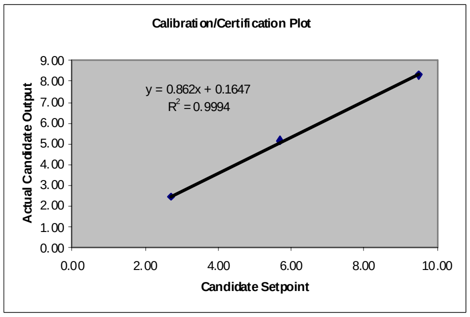
Results of Bracketing Experiments Between Tekran04 and Thermo02:

Comparison of WRI Tekran 04cal (REFERENCE) With WRI Thermo02cal (CANDIDATE)
WRI Test # 118761, 01/27/2009

9.6 µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero					
Tekran 04	9.449			9.50	8.319
Thermo 02	8.357	0.885			
Tekran 04	9.440				
Thermo 02	8.282	0.876	0.876		
Tekran 04	9.467				
Thermo 02	8.203	0.866			
Tekran 04	9.477				
5.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	5.723			5.70	5.159
Thermo 02	5.176	0.904			
Tekran 04	5.727				
Thermo 02	5.189	0.906	0.905		
Tekran 04	5.728				
Thermo 02	5.188	0.905			
Tekran 04	5.736				
2.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	2.781			2.70	2.447
Thermo 02	2.521	0.907			
Tekran 04	2.777				
Thermo 02	2.516	0.907	0.906		
Tekran 04	2.772				
Thermo 02	2.511	0.905			
Tekran 04	2.775				
Zero	-0.051				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*

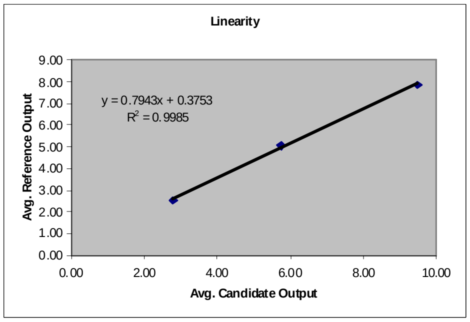


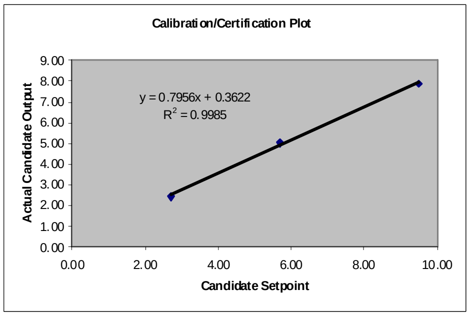


Comparison of WRI Tekran 04cal (REFERENCE) With WRI Thermo02cal (CANDIDATE)
WRI Test # 118762, 01/27/2009

9.6 µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero					
Tekran 04	9.469			9.50	7.867
Thermo 02	7.886	0.831			
Tekran 04	9.506				
Thermo 02	7.868	0.828	0.828		
Tekran 04	9.493				
Thermo 02	7.838	0.825			
Tekran 04	9.513				
5.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	5.744			5.70	5.019
Thermo 02	5.077	0.884			
Tekran 04	5.737				
Thermo 02	5.061	0.880	0.881		
Tekran 04	5.761				
Thermo 02	5.048	0.877			
Tekran 04	5.752				
2.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	2.800			2.70	2.442
Thermo 02	2.517	0.899			
Tekran 04	2.799				
Thermo 02	2.540	0.909	0.905		
Tekran 04	2.788				
Thermo 02	2.524	0.905			
Tekran 04	2.790				
Zero	0.003				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*



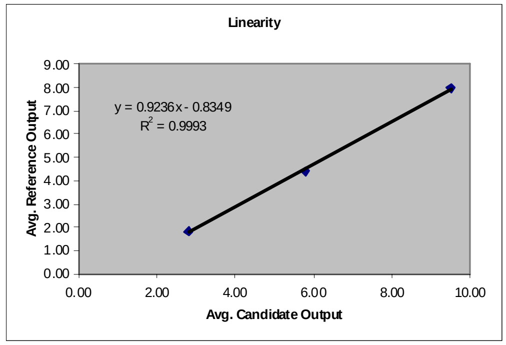


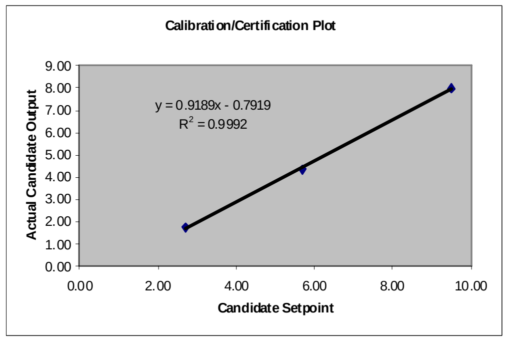
Results of Bracketing Experiments Between Tekran04 and Thermo03a:

Comparison of WRI Tekran 04cal (REFERENCE) With WRI Thermo03acal (CANDIDATE)
WRI Test # 118765, 01/29/2009

9.6 µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero					
Tekran 04	9.500			9.50	7.982
Thermo 03a	7.994	0.841			
Tekran 04	9.513				
Thermo 03a	8.039	0.844	0.840		
Tekran 04	9.527				
Thermo 03a	7.953	0.835			
Tekran 04	9.514				
5.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	5.781			5.70	4.346
Thermo 03a	4.408	0.762			
Tekran 04	5.788				
Thermo 03a	4.414	0.762	0.762		
Tekran 04	5.792				
Thermo 03a	4.417	0.763			
Tekran 04	5.788				
2.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	2.810			2.70	1.745
Thermo 03a	1.823	0.648			
Tekran 04	2.815				
Thermo 03a	1.816	0.645	0.646		
Tekran 04	2.812				
Thermo 03a	1.818	0.645			
Tekran 04	2.822				
Zero	-0.009				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*

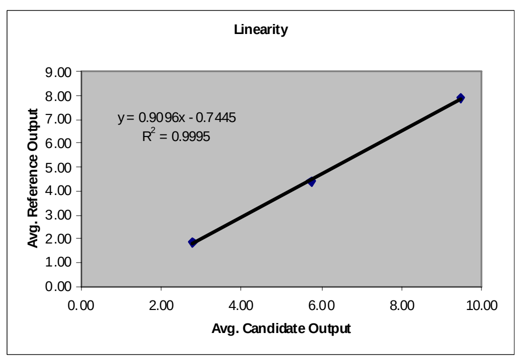


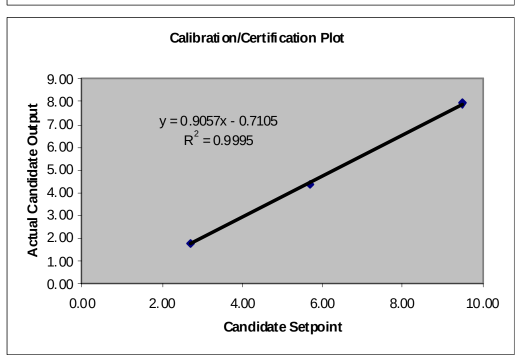


Comparison of WRI Tekran 04cal (REFERENCE) With WRI Thermo03cal (CANDIDATE)
WRI Test # 118766, 01/29/2009

9.6 µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero					
Tekran 04	9.483			9.50	7.928
Thermo 03a	7.912	0.834			
Tekran 04	9.493				
Thermo 03a	7.900	0.834	0.835		
Tekran 04	9.463				
Thermo 03a	7.919	0.836			
Tekran 04	9.476				
5.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	5.751			5.70	4.373
Thermo 03a	4.415	0.767			
Tekran 04	5.761				
Thermo 03a	4.416	0.768	0.767		
Tekran 04	5.745				
Thermo 03a	4.410	0.767			
Tekran 04	5.756				
2.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	2.799			2.70	1.779
Thermo 03a	1.841	0.658			
Tekran 04	2.797				
Thermo 03a	1.842	0.658	0.659		
Tekran 04	2.800				
Thermo 03a	1.851	0.660			
Tekran 04	2.808				
Zero	-0.004				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*



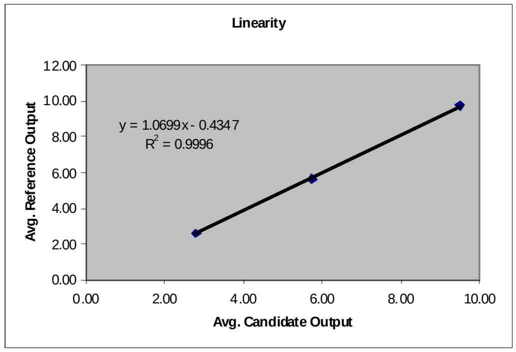


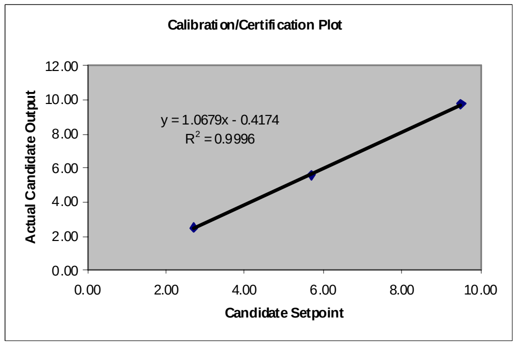
Results of Bracketing Experiments Between Tekran04 and Thermo04a

Comparison of WRI Tekran 04cal (REFERENCE) With WRI Thermo04acal (CANDIDATE)
WRI Test # 118763, 01/28/2009

9.6 µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero					
Tekran 04	9.477			9.50	9.764
Thermo 04a	9.784	1.031			
Tekran 04	9.497				
Thermo 04a	9.756	1.027	1.028		
Tekran 04	9.502				
Thermo 04a	9.739	1.025			
Tekran 04	9.502				
5.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	5.748			5.70	5.587
Thermo 04a	5.647	0.981			
Tekran 04	5.760				
Thermo 04a	5.639	0.980	0.980		
Tekran 04	5.752				
Thermo 04a	5.633	0.979			
Tekran 04	5.753				
2.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	2.785			2.70	2.512
Thermo 04a	2.601	0.932			
Tekran 04	2.795				
Thermo 04a	2.601	0.931	0.930		
Tekran 04	2.793				
Thermo 04a	2.594	0.928			
Tekran 04	2.798				
Zero	-0.035				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*

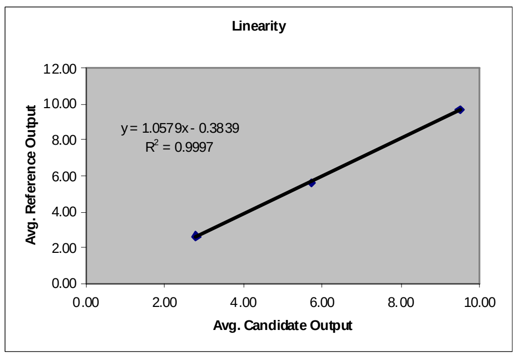


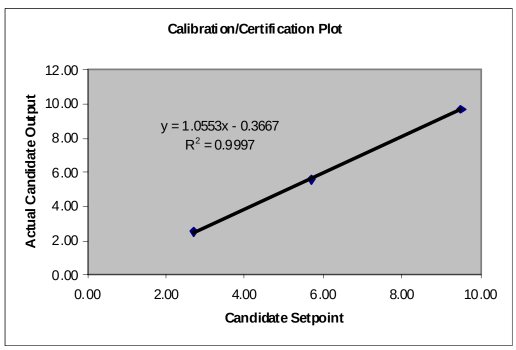


Comparison of WRI Tekran 04cal (REFERENCE) With WRI Thermo04acal (CANDIDATE)
WRI Test # 118764, 01/28/2009

9.6 µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero					
Tekran 04	9.507			9.50	9.691
Thermo 04a	9.691	1.018			
Tekran 04	9.525				
Thermo 04a	9.693	1.019	1.020		
Tekran 04	9.505				
Thermo 04a	9.697	1.023			
Tekran 04	9.450				
5.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	5.716			5.70	5.576
Thermo 04a	5.609	0.981			
Tekran 04	5.724				
Thermo 04a	5.609	0.977	0.978		
Tekran 04	5.752				
Thermo 04a	5.611	0.977			
Tekran 04	5.738				
2.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	2.794			2.70	2.523
Thermo 04a	2.592	0.926			
Tekran 04	2.804				
Thermo 04a	2.657	0.946	0.935		
Tekran 04	2.811				
Thermo 04a	2.612	0.931			
Tekran 04	2.800				
Zero	-0.025				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*





APPENDIX F

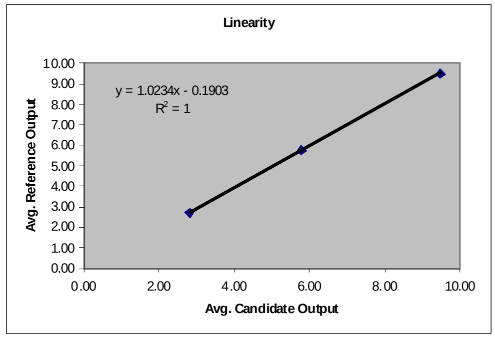
February 2009 Nesting Analyses Summary Data

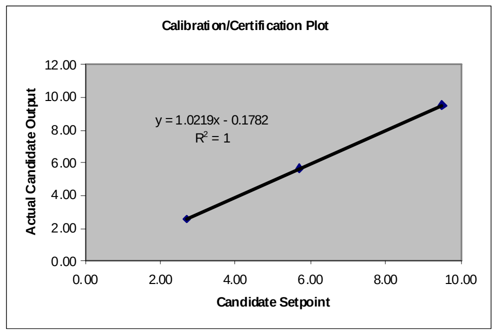
Results of Bracketing Experiments Between Tekran04 and Tekran 03:

Comparison of WRI Tekran 04cal (REFERENCE) With WRI Tekran03cal (CANDIDATE)
WRI Test # 118771, 02/02/2009

9.6 µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero					
Tekran 04	9.475			9.50	9.523
Tekran 03	9.490	1.001			
Tekran 04	9.486				
Tekran 03	9.511	1.003	1.002		
Tekran 04	9.485				
Tekran 03	9.516	1.004			
Tekran 04	9.481				
5.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	5.799			5.70	5.662
Tekran 03	5.749	0.992			
Tekran 04	5.793				
Tekran 03	5.752	0.993	0.993		
Tekran 04	5.791				
Tekran 03	5.764	0.995			
Tekran 04	5.791				
2.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	2.826			2.70	2.572
Tekran 03	2.697	0.954			
Tekran 04	2.828				
Tekran 03	2.684	0.952	0.953		
Tekran 04	2.815				
Tekran 03	2.680	0.952			
Tekran 04	2.814				
Zero	-0.026				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*

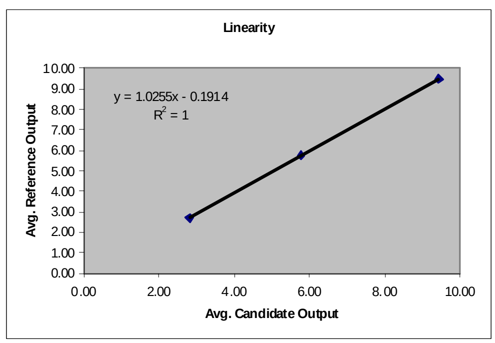


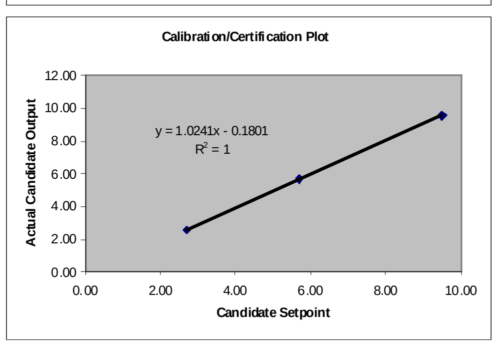


Comparison of WRI Tekran 04cal (REFERENCE) With WRI Tekran03cal (CANDIDATE)
WRI Test # 118772, 02/02/2009

9.6 µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero					
Tekran 04	9.427			9.50	9.540
Tekran 03	9.467	1.003			
Tekran 04	9.444				
Tekran 03	9.483	1.004	1.004		
Tekran 04	9.438				
Tekran 03	9.495	1.005			
Tekran 04	9.455				
5.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	5.767			5.70	5.678
Tekran 03	5.743	0.995			
Tekran 04	5.773				
Tekran 03	5.749	0.996	0.996		
Tekran 04	5.770				
Tekran 03	5.752	0.997			
Tekran 04	5.769				
2.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	2.816			2.70	2.574
Tekran 03	2.686	0.954			
Tekran 04	2.814				
Tekran 03	2.689	0.954	0.953		
Tekran 04	2.823				
Tekran 03	2.684	0.952			
Tekran 04	2.818				
Zero	-0.002				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*

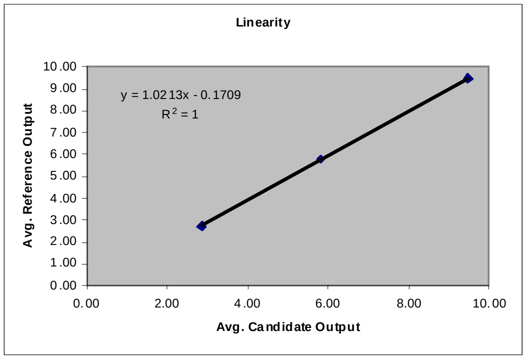


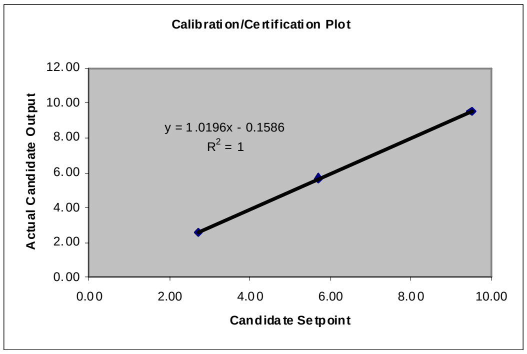


Comparison of WRI Tekran 04cal (REFERENCE) With WRI Tekran03cal (CANDIDATE)
WRI Test # 118776, 02/05/2009

9.6 µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero					
Tekran 04	9.486			9.50	9.517
Tekran 03	9.483	1.000			
Tekran 04	9.478				
Tekran 03	9.499	1.003	1.002		
Tekran 04	9.465				
Tekran 03	9.461	1.002			
Tekran 04	9.415				
5.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	5.806			5.70	5.678
Tekran 03	5.783	0.996			
Tekran 04	5.801				
Tekran 03	5.781	0.996	0.996		
Tekran 04	5.811				
Tekran 03	5.786	0.996			
Tekran 04	5.803				
2.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	2.835			2.70	2.580
Tekran 03	2.728	0.959			
Tekran 04	2.856				
Tekran 03	2.728	0.954	0.956		
Tekran 04	2.866				
Tekran 03	2.731	0.955			
Tekran 04	2.854				
Zero	0.010				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*

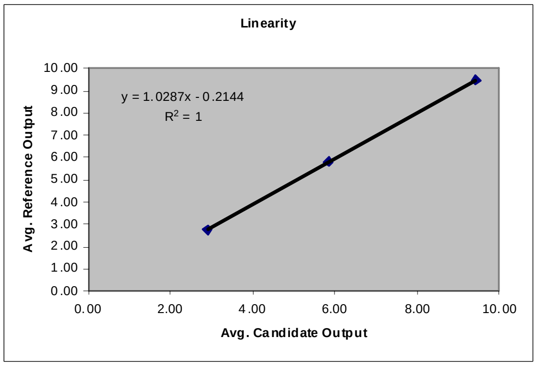


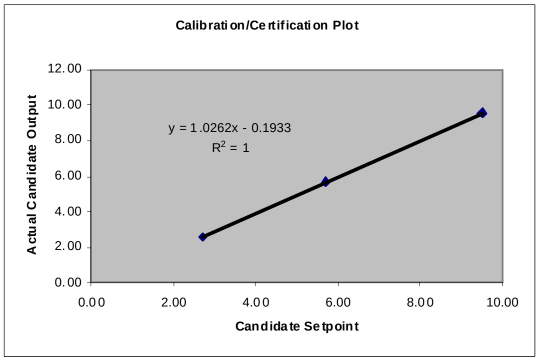


Comparison of WRI Tekran 04cal (REFERENCE) With WRI Tekran03cal (CANDIDATE)
WRI Test # 118778, 02/06/2009

9.6 µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero					
Tekran 04	9.438			9.50	9.548
Tekran 03	9.492	1.006			
Tekran 04	9.434				
Tekran 03	9.466	1.004	1.005		
Tekran 04	9.415				
Tekran 03	9.460	1.005			
Tekran 04	9.417				
5.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	5.844			5.70	5.673
Tekran 03	5.817	0.995			
Tekran 04	5.853				
Tekran 03	5.821	0.995	0.995		
Tekran 04	5.845				
Tekran 03	5.821	0.996			
Tekran 04	5.844				
2.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	2.893			2.70	2.568
Tekran 03	2.749	0.950			
Tekran 04	2.892				
Tekran 03	2.751	0.951	0.951		
Tekran 04	2.892				
Tekran 03	2.755	0.951			
Tekran 04	2.899				
Zero	0.014				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*

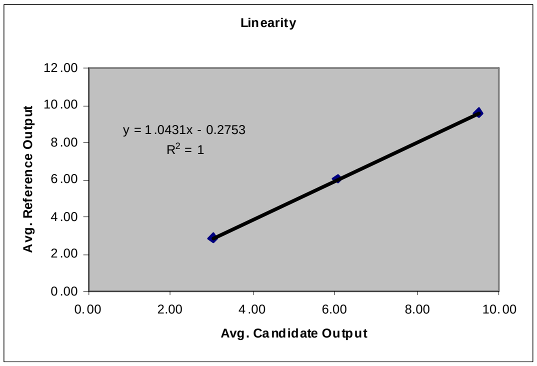


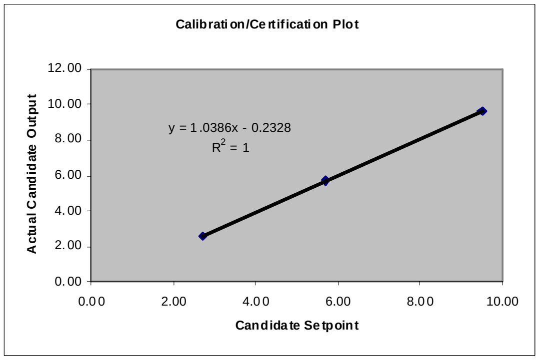


Comparison of WRI Tekran 04cal (REFERENCE) With WRI Tekran03cal (CANDIDATE)
WRI Test # 118787, 02/13/2009

9.6 µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero					
Tekran 04	9.477			9.50	9.631
Tekran 03	9.592	1.012			
Tekran 04	9.476				
Tekran 03	9.615	1.014	1.014		
Tekran 04	9.483				
Tekran 03	9.625	1.015			
Tekran 04	9.487				
5.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	6.070			5.70	5.695
Tekran 03	6.045	0.996			
Tekran 04	6.071				
Tekran 03	6.064	1.001	0.999		
Tekran 04	6.047				
Tekran 03	6.054	1.001			
Tekran 04	6.054				
2.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	3.027			2.70	2.567
Tekran 03	2.868	0.947			
Tekran 04	3.028				
Tekran 03	2.883	0.952	0.951		
Tekran 04	3.029				
Tekran 03	2.887	0.953			
Tekran 04	3.029				
Zero	-0.022				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*





Results of Bracketing Experiments Between Tekran04 and EPA Tekran 05:

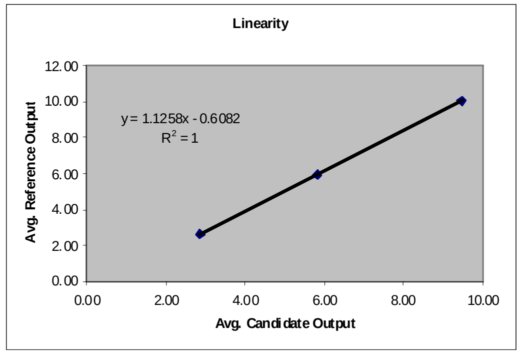
Comparison of WRI Tekran 04cal (REFERENCE) With EPA Tekran 05cal (CANDIDATE)
WRI Test # 118773, 02/03/2009

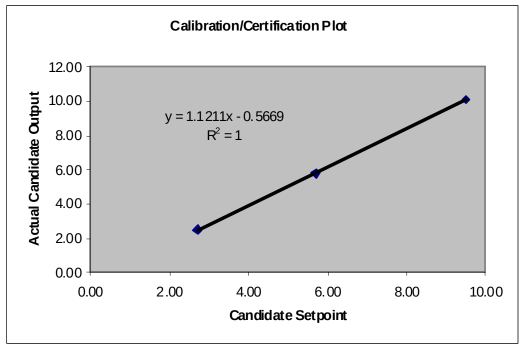
9.6 µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero					
Tekran 04	9.456			9.50	10.096
EPATekran 05	10.061	1.063			
Tekran 04	9.473				
EPATekran 05	10.078	1.064	1.063		
Tekran 04	9.464				
EPATekran 05	10.050	1.061			
Tekran 04	9.480				

5.7 µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio		
Tekran 04	5.822			5.70	5.795
EPATekran 05	5.905	1.014			
Tekran 04	5.829				
EPATekran 05	5.929	1.018	1.017		
Tekran 04	5.818				
EPATekran 05	5.927	1.018			
Tekran 04	5.821				

2.7 µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio		
Tekran 04	2.851			2.70	2.476
EPATekran 05	2.610	0.916			
Tekran 04	2.850				
EPATekran 05	2.614	0.916	0.917		
Tekran 04	2.859				
EPATekran 05	2.621	0.920			
Tekran 04	2.840				
Zero	-0.006				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*





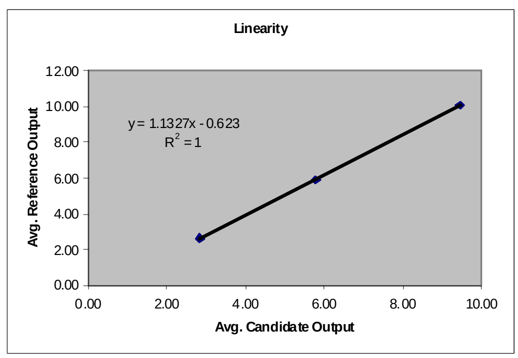
Comparison of WRI Tekran 04cal (REFERENCE) With EPA Tekran 05cal (CANDIDATE)
WRI Test # 118774, 02/03/2009

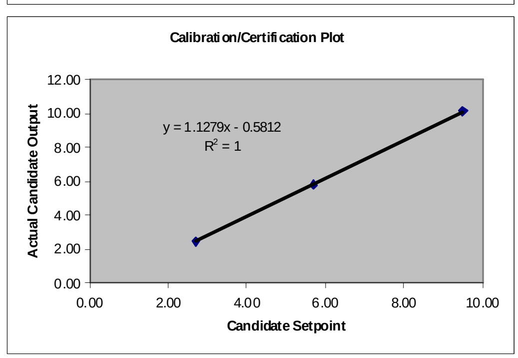
9.6 µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero					
Tekran 04	9.451			9.50	10.147
EPATekran 05	10.115	1.070			
Tekran 04	9.449				
EPATekran 05	10.084	1.067	1.068		
Tekran 04	9.457				
EPATekran 05	10.093	1.067			
Tekran 04	9.457				

5.7 µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio		
Tekran 04	5.797			5.70	5.818
EPATekran 05	5.910	1.020			
Tekran 04	5.790				
EPATekran 05	5.917	1.021	1.021		
Tekran 04	5.802				
EPATekran 05	5.922	1.021			
Tekran 04	5.800				

2.7 µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio		
Tekran 04	2.841			2.70	2.481
EPATekran 05	2.607	0.917			
Tekran 04	2.843				
EPATekran 05	2.617	0.920	0.919		
Tekran 04	2.845				
EPATekran 05	2.615	0.919			
Tekran 04	2.845				
Zero	-0.012				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*

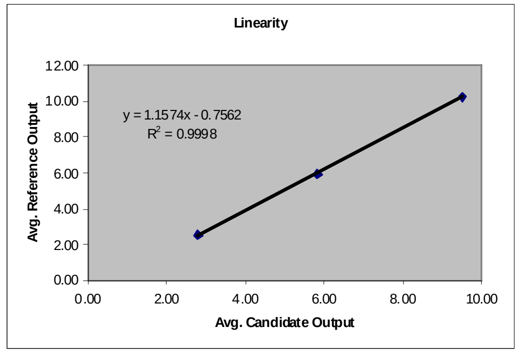


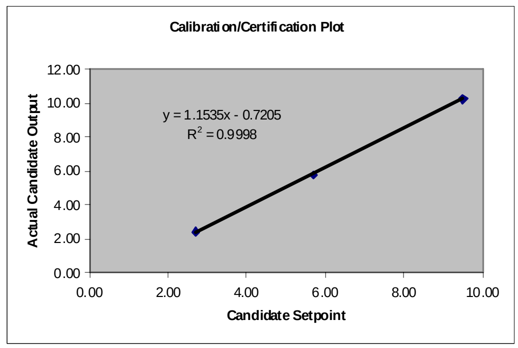


Comparison of WRI Tekran 04cal (REFERENCE) With EPA Tekran 05cal (CANDIDATE)
WRI Test # 118779, 02/09/2009

9.6 µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero					
Tekran 04	9.497			9.50	10.264
Tekran 05	10.331	1.086			
Tekran 04	9.536				
Tekran 05	10.267	1.079	1.080		
Tekran 04	9.500				
Tekran 05	10.227	1.077			
Tekran 04	9.491				
5.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	5.834			5.70	5.797
Tekran 05	5.932	1.017			
Tekran 04	5.830				
Tekran 05	5.919	1.016	1.017		
Tekran 04	5.822				
Tekran 05	5.927	1.018			
Tekran 04	5.823				
2.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	2.790			2.70	2.427
Tekran 05	2.507	0.898			
Tekran 04	2.793				
Tekran 05	2.514	0.898	0.899		
Tekran 04	2.806				
Tekran 05	2.519	0.900			
Tekran 04	2.792				
Zero	-0.154				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*

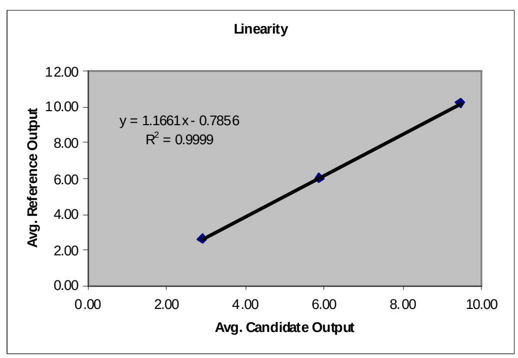


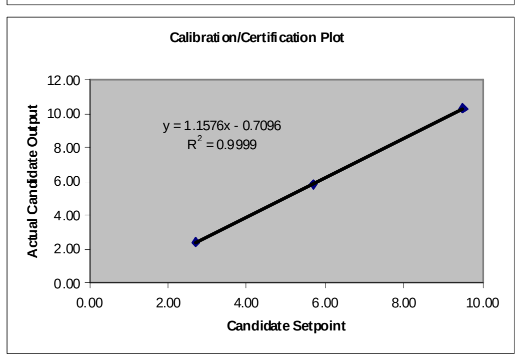


Comparison of WRI Tekran 04cal (REFERENCE) With EPA Tekran 05cal (CANDIDATE)
WRI Test # 118780, 02/09/2009

9.6 µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero					
Tekran 04	9.460			9.50	10.310
Tekran 05	10.242	1.083			
Tekran 04	9.455				
Tekran 05	10.262	1.086	1.085		
Tekran 04	9.447				
Tekran 05	10.260	1.087			
Tekran 04	9.431				
5.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	5.883			5.70	5.838
Tekran 05	6.033	1.025			
Tekran 04	5.886				
Tekran 05	6.008	1.022	1.024		
Tekran 04	5.870				
Tekran 05	6.024	1.025			
Tekran 04	5.881				
2.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	2.907			2.70	2.444
Tekran 05	2.635	0.907			
Tekran 04	2.903				
Tekran 05	2.635	0.906	0.905		
Tekran 04	2.916				
Tekran 05	2.631	0.903			
Tekran 04	2.910				
Zero	0.005				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*

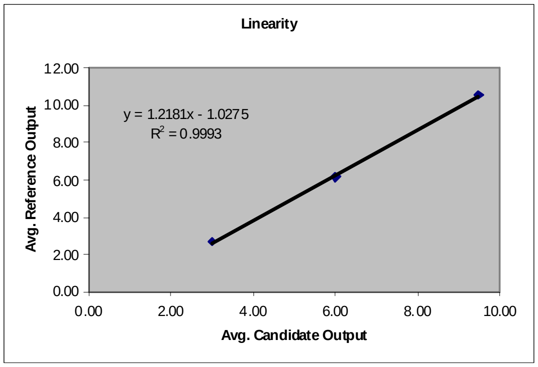


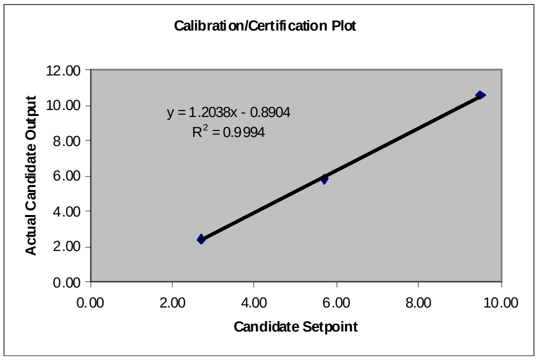


Comparison of WRI Tekran 04cal (REFERENCE) With EPA Tekran 05cal (CANDIDATE)
WRI Test # 118786, 02/12/2009

9.6 µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero					
Tekran 04	9.487			9.50	10.596
Tekran 05	10.584	1.114			
Tekran 04	9.514				
Tekran 05	10.586	1.115	1.115		
Tekran 04	9.471				
Tekran 05	10.583	1.117			
Tekran 04	9.476				
5.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	6.014			5.70	5.856
Tekran 05	6.168	1.026			
Tekran 04	6.009				
Tekran 05	6.188	1.030	1.027		
Tekran 04	6.010				
Tekran 05	6.176	1.027			
Tekran 04	6.021				
2.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	3.008			2.70	2.424
Tekran 05	2.695	0.897			
Tekran 04	2.998				
Tekran 05	2.683	0.895	0.898		
Tekran 04	2.997				
Tekran 05	2.704	0.901			
Tekran 04	3.008				
Zero	-0.003				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*



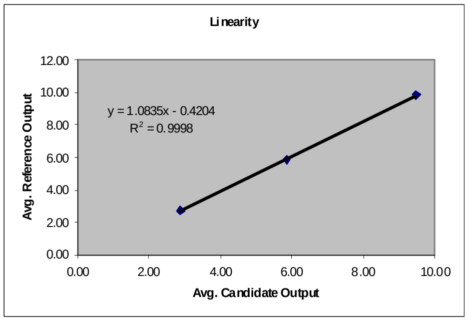


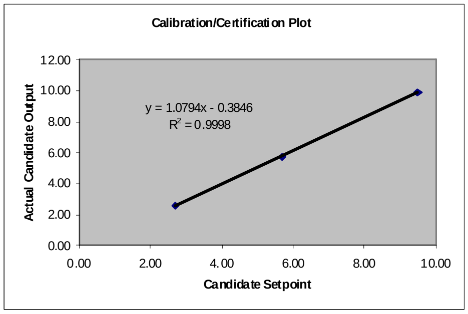
Results of Bracketing Experiments Between Tekran04 and EPA Thermo 02

Comparison of WRI Tekran 04cal (REFERENCE) With EPA Thermo02cal (CANDIDATE)
WRI Test # 118775, 02/04/2009

9.6 µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero					
Tekran 04	9.467			9.50	9.894
EPA Thermo 02	9.870	1.043			
Tekran 04	9.467				
EPA Thermo 02	9.863	1.041	1.041		
Tekran 04	9.491				
EPA Thermo 02	9.864	1.041			
Tekran 04	9.456				
5.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	5.856			5.70	5.714
EPA Thermo 02	5.872	1.003			
Tekran 04	5.851				
EPA Thermo 02	5.858	1.001	1.002		
Tekran 04	5.852				
EPA Thermo 02	5.866	1.003			
Tekran 04	5.846				
2.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	2.885			2.70	2.560
EPA Thermo 02	2.734	0.948			
Tekran 04	2.883				
EPA Thermo 02	2.734	0.949	0.948		
Tekran 04	2.882				
EPA Thermo 02	2.733	0.948			
Tekran 04	2.884				
Zero	-0.015				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*

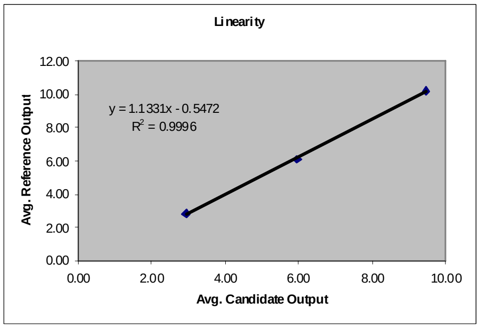


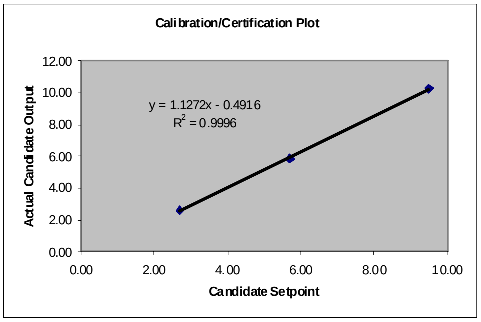


Comparison of WRI Tekran 04cal (REFERENCE) With EPA Thermo02cal (CANDIDATE)
WRI Test # 118781, 02/10/2009

9.6 µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero					
Tekran 04	9.456			9.50	10.254
EPA Thermo 02	10.179	1.076			
Tekran 04	9.459				
EPA Thermo 02	10.201	1.079	1.079		
Tekran 04	9.448				
EPA Thermo 02	10.228	1.083			
Tekran 04	9.444				
5.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	5.924			5.70	5.850
EPA Thermo 02	6.107	1.029			
Tekran 04	5.942				
EPA Thermo 02	6.099	1.025	1.026		
Tekran 04	5.956				
EPA Thermo 02	6.108	1.024			
Tekran 04	5.969				
2.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	2.932			2.70	2.599
EPA Thermo 02	2.830	0.964			
Tekran 04	2.939				
EPA Thermo 02	2.828	0.962	0.963		
Tekran 04	2.938				
EPA Thermo 02	2.823	0.961			
Tekran 04	2.936				
Zero	-0.056				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*

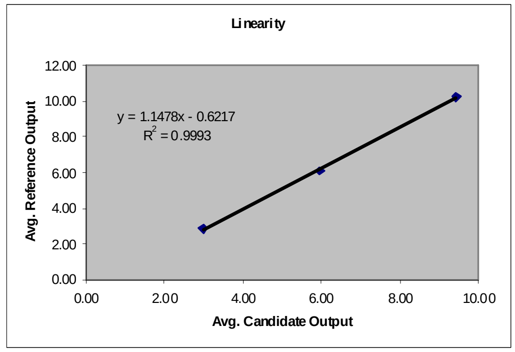


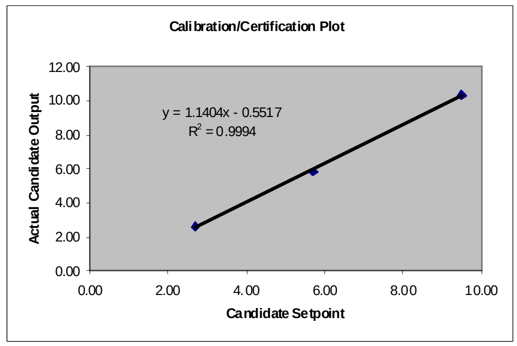


Comparison of WRI Tekran 04cal (REFERENCE) With EPA Thermo02cal (CANDIDATE)
WRI Test # 118782, 02/10/2009

9.6 µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero					
Tekran 04	9.444			9.50	10.332
EPA Thermo 02	10.256	1.087			
Tekran 04	9.426				
EPA Thermo 02	10.270	1.089	1.088		
Tekran 04	9.429				
EPA Thermo 02	10.236	1.086			
Tekran 04	9.416				
5.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	5.950			5.70	5.836
EPA Thermo 02	6.106	1.025			
Tekran 04	5.964				
EPA Thermo 02	6.095	1.023	1.024		
Tekran 04	5.954				
EPA Thermo 02	6.104	1.024			
Tekran 04	5.970				
2.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	2.970			2.70	2.590
EPA Thermo 02	2.852	0.959			
Tekran 04	2.976				
EPA Thermo 02	2.847	0.957	0.959		
Tekran 04	2.973				
EPA Thermo 02	2.857	0.962			
Tekran 04	2.969				
Zero	0.008				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*

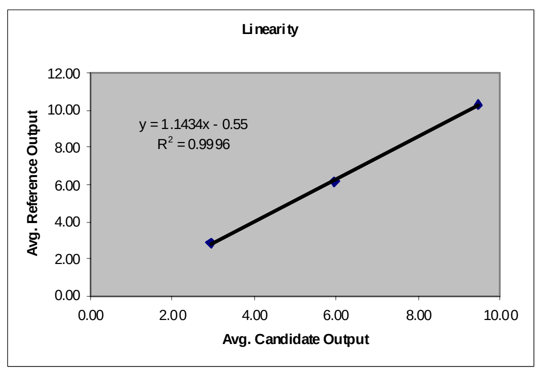


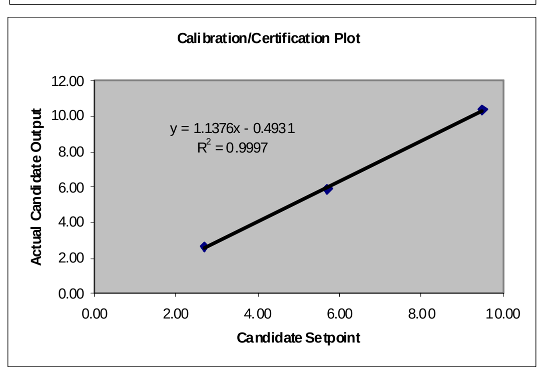


Comparison of WRI Tekran 04cal (REFERENCE) With EPA Thermo02cal (CANDIDATE)
WRI Test # 118785, 02/12/2009

9.6 µg/m3 Nominal	5-min avg µg/m3	Nested Ratio	Avg Nested Ratio	Reference Conc.	Act. Cand.* Output
Zero					
Tekran 04	9.469			9.50	10.351
EPA Thermo 02	10.279	1.086			
Tekran 04	9.457				
EPA Thermo 02	10.302	1.089	1.090		
Tekran 04	9.455				
EPA Thermo 02	10.325	1.093			
Tekran 04	9.440				
5.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	5.951			5.70	5.908
EPA Thermo 02	6.155	1.035			
Tekran 04	5.944				
EPA Thermo 02	6.164	1.037	1.036		
Tekran 04	5.945				
EPA Thermo 02	6.173	1.038			
Tekran 04	5.955				
2.7 µg/m3 Nominal	**5-min avg µg/m3**	**Nested Ratio**	**Avg Nested Ratio**		
Tekran 04	2.940			2.70	2.625
EPA Thermo 02	2.854	0.972			
Tekran 04	2.934				
EPA Thermo 02	2.848	0.971	0.972		
Tekran 04	2.930				
EPA Thermo 02	2.853	0.974			
Tekran 04	2.930				
Zero	-0.083				

** Act. Cand. Output = Reference Conc. x Avg. Nested Ratio*

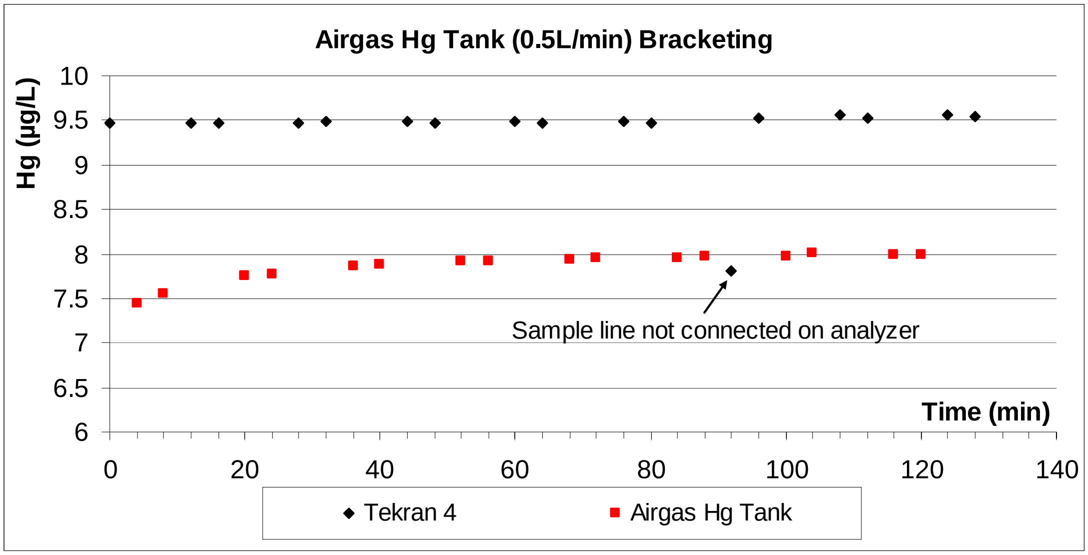




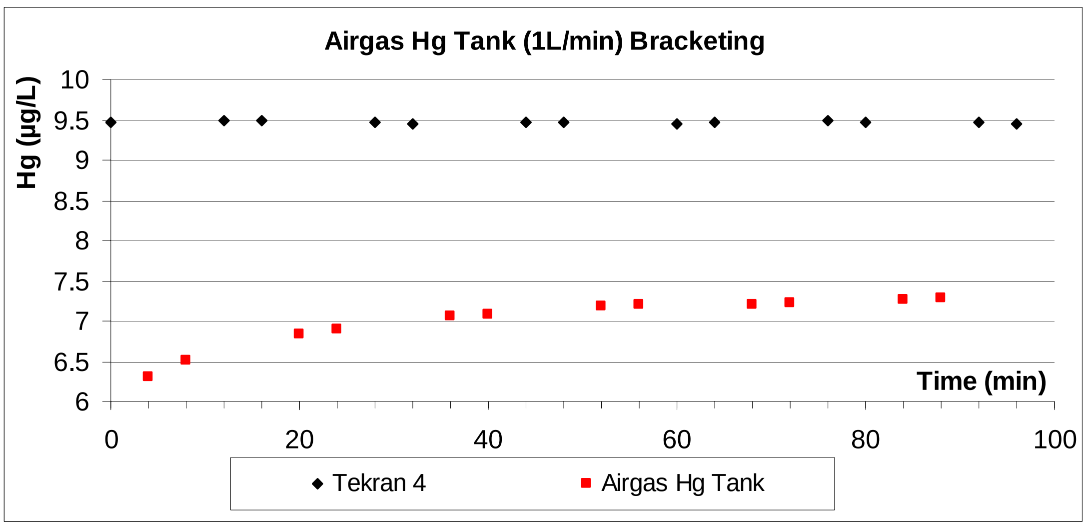
APPENDIX G

Gas Cylinder Nesting Results

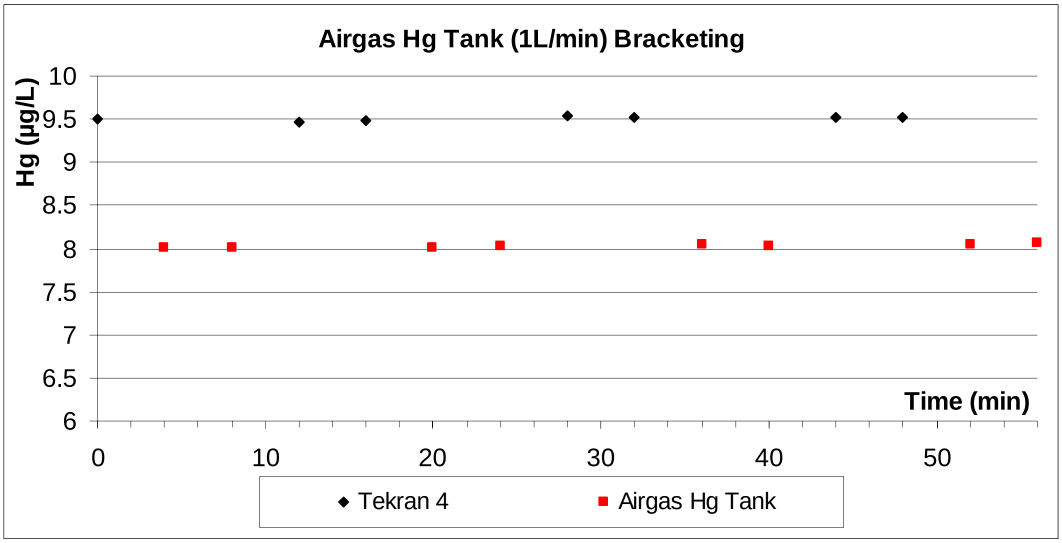
Airgas Cylinder Nesting Results



2/11/09

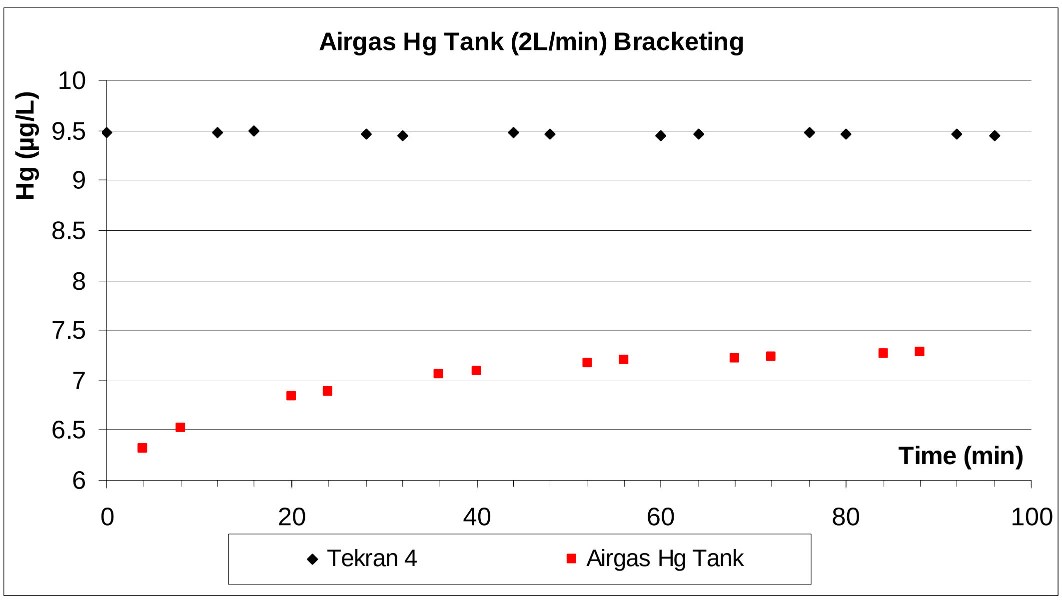


1/30/09



Airgas Hg Tank (1L/min) Bracketing
Hg (µg/L)
10
9.5
9
8.5
8
7.5
7
6.5
6
Time (min)
0
10
20
30
40
50
Tekran 4
Airgas Hg Tank

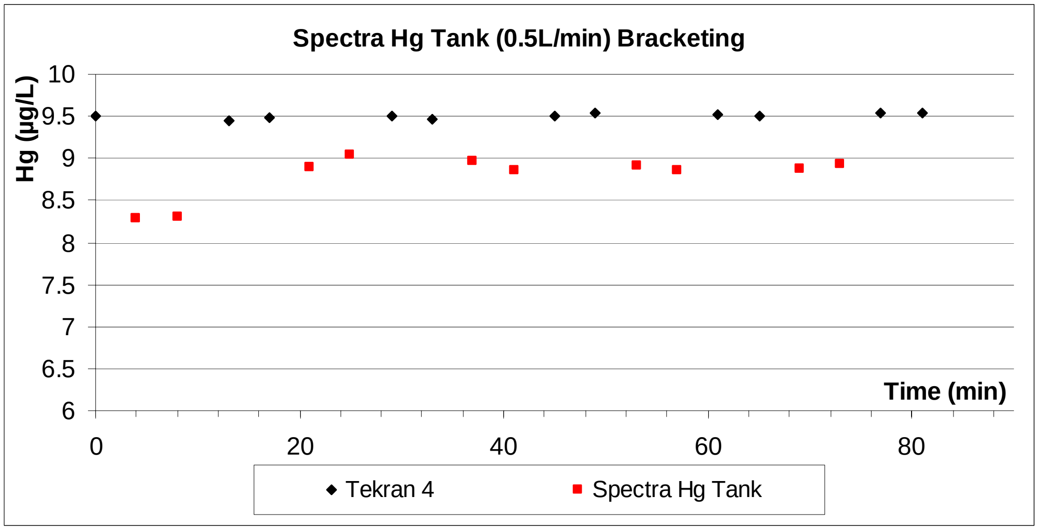
2/16/09



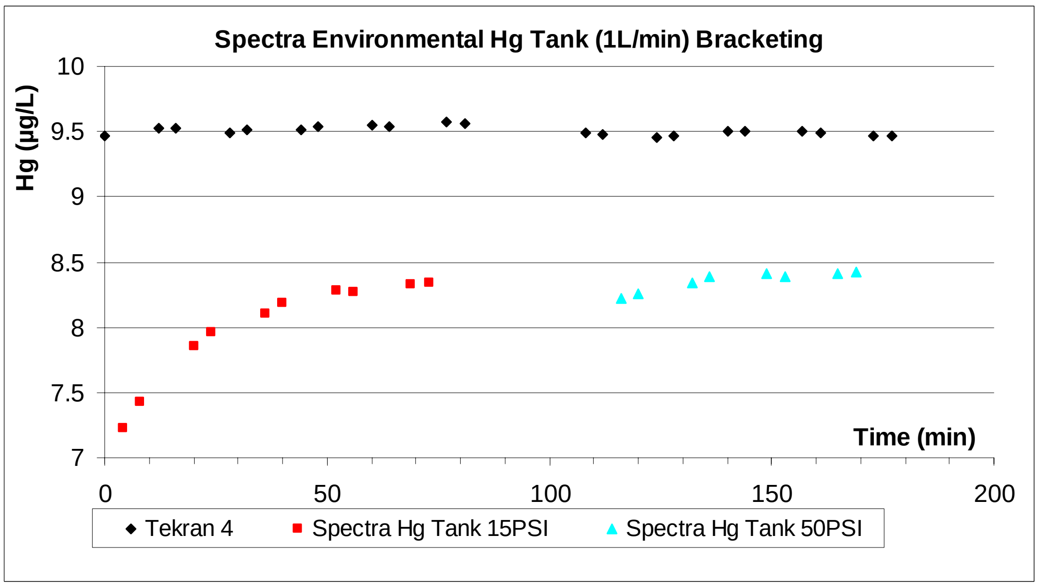
Airgas Hg Tank (2L/min) Bracketing
Hg (µg/L)
10
9.5
9
8.5
8
7.5
7
6.5
6
Time (min)
0
20
40
60
80
100
Tekran 4
Airgas Hg Tank

2/11/09

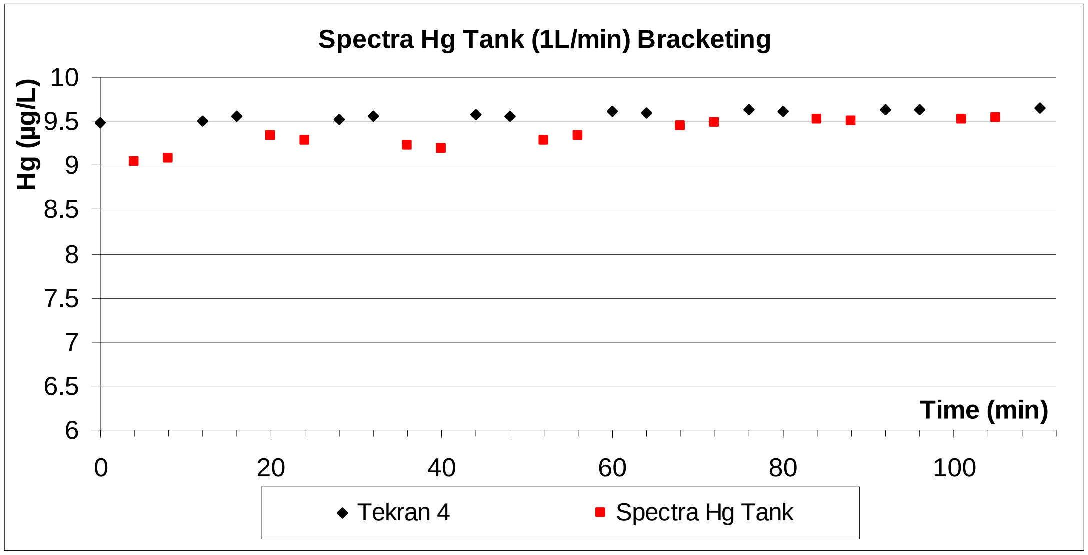
Spectra Gases Cylinder Nesting Results



2/13/09

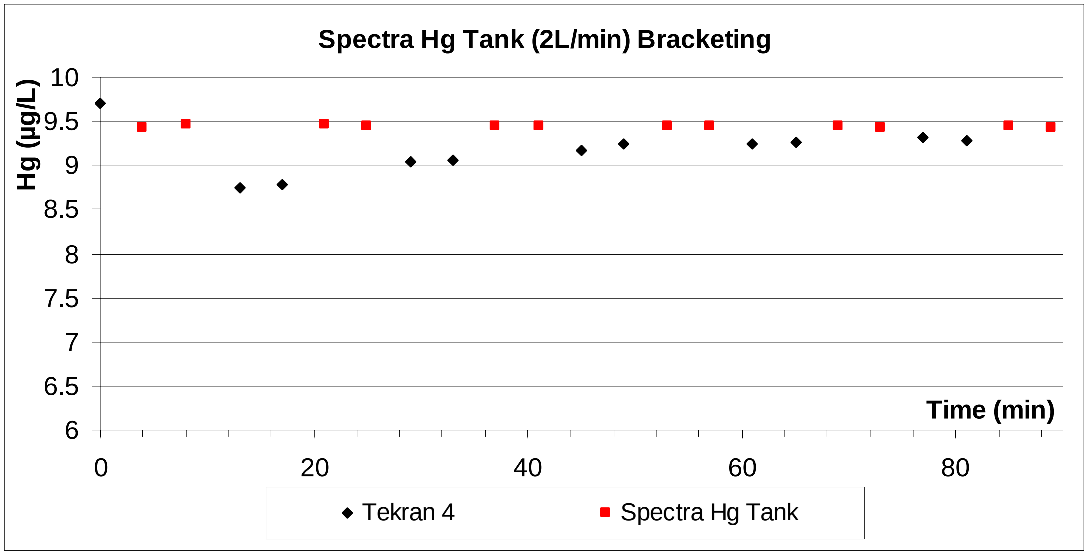


1/30/09



Spectra Hg Tank (1L/min) Bracketing
Hg (µg/L)
10
9.5
9
8.5
8
7.5
7
6.5
6
Time (min)
0
20
40
60
80
100
Tekran 4
Spectra Hg Tank

2/16/09



Spectra Hg Tank (2L/min) Bracketing
Hg (µg/L)
10
9.5
9
8.5
8
7.5
7
6.5
6
Time (min)
0
20
40
60
80
Tekran 4
Spectra Hg Tank

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