International Standards

- Identification and Provision of "Higher Order" Certified Reference Materials and Reference Measurement Procedures Required for U.S. Industry Compliance with the EU IVD Directive

- International Benchmarking of NIST Capabilities for Chemical Measurements

- Results of CCQM-K25 Key Comparison for Polychlorinated Biphenyl Congeners in Sediment

- Results from International Key Comparison for Calcium in Serum

- Results from International Comparisons for Selected Components in Natural Gas

- Development and Dissemination of Intrinsic Standards for Chemical Measurements

International Standards

Title: Identification and Provision of "Higher Order" Certified Reference Materials and Reference Measurement Procedures Required for U.S. Industry Compliance with the EU IVD Directive

Authors: W.E. May and V.L. Vilker

Abstract: The recently enacted European Directive 98/79/EC on in vitro diagnostic medical devices (IVD MD) requires, among other things, that "the traceability of values assigned to calibrators and control materials for in vitro diagnostic devices must be assured through available reference measurement procedures and/or reference materials of higher order". At present, neither reference materials nor reference methods are available for more than 300 different chemical or biochemical species that are measured in medical laboratories using IVDs. Reference methods and/or materials exist for only about 30 worldwide

Purpose: Excluding home diagnostics, the overall world-wide invitro diagnostic market is approximately $20 billion. The total IVD market in Europe was about $5.6 billion in 1998 and has shown growth of about 4% per year over the past five years (data from www.edma-ivd.be). Approximately 60% of the IVD products currently on the European market are imported from the US. The U.S. IVD industry (ADVAMED) has asked NIST to work with our counterparts in Europe and the Asia-Pacific to provide the reference materials and methods of "higher order" that are urgently needed to comply with the requirements of the EU IVD Directive. Without timely completion of these standards, the U S. IVD industry's access into the European market will be seriously jeopardized.

Major Accomplishments: To facilitate the identification of "higher order" reference methods and reference materials that are currently available, the Joint Committee on Traceability in Laboratory Medicine (JCTLM) was created at a meeting held at the International Bureau of Weights and Measures (BIPM) in early June 2002. NIST (Willie E. May) is leading the activities of JCTLM Working Group-I, charged with establishing the process for identifying the "higher order" Certified Reference Materials and reference measurement procedures that will be required for IVD industry compliance with the EU IVD Directive.

Three meetings were held this year to review nominations for more than 80 Reference Methods and 435 Reference Materials for Electrolytes, Enzymes, Drugs, and Metabolites and Substrates Coagulation Factors, Non-Peptide Hormones, Nucleic Acids, and Proteins. The higher order standards endorsed by this process will be published in a database maintained by the International Bureau of Weights and Measures (BIPM) and will be publicly available on the BIPM website in February 2004 with links to the NIST CSTL and other relevant websites. See example below for Reference Measurement Procedures and Reference Materials for Cholesterol:

Reference Measurement Procedure					
Procedure Name and/or ID #	Analyte Name	Applicable Matrices	Measurement Principle	Reference Procedure Citation(s) or Document(s)	Reference Procedure Comparability Assessment Studies
NIST definitive method for serum cholesterol	cholesterol	lyophilized, fresh, or frozen serum	ID/GC/MS	Anal Chem 61, 1718-1723 (1989)	CCQM-K6; http://kcdb.bipm.org/appendixB/appbresults/ccqm-k6/ccqm-k6_final_report.pdf; Clin Chem 36, 370-375 (1990)
U. Of Ghent reference method for cholesterol	cholesterol	lyphilized, fresh, or frozen serum	ID/GC/MS	Clin Chem 39,1001-6 (1993) [=part II of Clin Chem 39,993-1000 (1993)]; Eur J Clin Chem Clin Biochem 34, 853-60 (1996); Clin Chem 42, 531-5 (1996)	EUROMET 563
DGKC definitive Method for Serum Cholesterol	cholesterol	lyophilized, fresh, or frozen human serum or plasma	ID/GC/MS	Siekmann et al., Z. anal. Chem. 279, 145-146 (1976)	PTB - National Key Comparison for Accredtation
CDCAbell-Kendall method for cholesterol	cholesterol	lyophillized, fresh or frozen human serum	Spectrophotometry	Cooper, GR, et al, Clin Chem 32: 921-929, 1986	Clin Chem 36, 370-375 (1990)

Reference Materials								
Information about Material				Contact Information	References			Comments
Analyte	Matrix	Material Name and/or ID #	Estimated * Availability (months, as of Jan 2004)	- Producer - Country - Website - Email Address - Phone Number - Fax Number	Commutability Study Information and/or Citations	Other Relevant Publications	Hyperlink to Comparability Assessment Studies	Comments
cholesterol	cholesterol	GBW09203b	60	NRCCRM China Tel: 086-10-64221811 Fax: 086-10-64213149 Email: crmservice@nrccrm.com.cn	Primary calibrator for higher order reference methods			
cholesterol	cholesterol	SRM 911b	21	NIST USA http://ts.nist.gov/ts/htdocs/230/232/232.htm Email:srminfo@nist.gov Tel:(301)975-6776 Fax: (301)948-3730	Primary calibrator for higher order reference methods			
cholesterol	human serum	JCCRM 211		HECTEF Japan http://www.in8.co.jp/hectef/starte.htm Tel: 81-44-813-0055 Fax: 81-44-813-0224			NIST study presented at JCTLM Meeting, June 20, 2003, BIPM, Sevres, France	
cholesterol	human serum (frozen)	SRM 1951b	60	NIST USA http://ts.nist.gov/ts/htdocs/230/232/232.htm Email:srminfo@nist.gov Tel:(301)975-6776 Fax: (301)948-3730	Material prepared following NCCLS Document C37-A "Preparation and Validation of Commutable Frozen Human Serum Pools as Secondary Reference Materials for	Previous lot (1951a) was measured in NIST study presented at JCTLM Meeting, June 20, 2003, BIPM, Sevres, France		
cholesterol	human serum (lyophilized)	SRM 1952a	60	NIST USA http://ts.nist.gov/ts/htdocs/230/232/232.htm Email:srminfo@nist.gov Tel:(301)975-6776 Fax: (301)948-3730		Method used for certification: Anal Chem 61, 1718-1723 (1989)	NIST study presented at JCTLM Meeting, June 20, 2003, BIPM, Sevres, France	
cholesterol	human serum (lyophilized)	SRM 968c	38	NIST USA http://ts.nist.gov/ts/htdocs/230/232/232.htm Email:srminfo@nist.gov Tel:(301)975-6776 Fax: (301)948-3730		Method used for certification: Anal Chem 61, 1718-1723 (1989)	NIST study presented at JCTLM Meeting, June 20, 2003, BIPM, Sevres, France	
cholesterol	human serum (lyophilized)	SRM909b	60	NIST USA http://ts.nist.gov/ts/htdocs/230/232/232.htm Email:srminfo@nist.gov Tel:(301)975-6776 Fax: (301)948-3730		Certification process described: Fresenius' J. Anal. Chem. 361:2 71-80 (1998); Method used for certification: Anal Chem 61, 1718-1723 (1989)	NIST study presented at JCTLM Meeting, June 20, 2003, BIPM, Sevres, France	

Based on this work and input from the medical professional and IVD manufacturing communities, JCTLM WG-I will also establish a list of highest priority needs for new reference procedures/ reference materials. NIST, EU and AP Laboratories have also begun a dialogue regarding sharing the work-load involved in developing and maintaining the more than 100 standards that are currently needed for IVD industry compliance with the EU IVDD. NIST has been asked to provide about 40% of the standards based on our current capabilities and U.S. market share.

Staff in the Analytical Chemistry and Biotechnology Divisions are developing “higher order” reference materials and methods urgently needed for US IVD industry compliance with the requirements of the EU IVD Directive including:

Health Stautus Marker	Disease State
• Cardiac Troponin-I	heart attack occurrence and damage
• Cadmium and Mercury	toxic metal poisoning
• Folates	neural tube defects
• HER-2 Neu	breast cancer
• Homocysteine	heart disease risk
• Glycated Hemoglobin	diabetes status
• Prostate-Specific Antigen	prostate cancer
• Triiodothyronine and Thyroid Stimulating Hormone	thyroid function
• Trinucleotide Repeat	mental retardation
• Speciated Iron	hemochromatosis, anemia

In 2003, work will be completed for standards for Toxic Metals in Urine, Cardiac Troponin-I and Electrolytes in Human Serum.

Impact: Clinical measurement results that are reliable and comparable over both space and time are essential for optimal patient care, most efficient use of available healthcare funds, and full utilization of the potential of new information technology tools. Incorrect interpretation of measurement results by the physician can lead to incorrect diagnosis and treatment, additional unnecessary tests and medical procedures, and increased pain and suffering for the patient. Measurements are responsible for 10%-15% of the $1.5 T annual costs of healthcare in the United States. A significant portion (25% - 30%) of health-related measurements is performed for non-diagnostic reasons (re-tests, error prevention and detection). The "German Health Report of 1998" states explicitly that "the costs of repeat measurements amount to 1.5 billion US $ per year in Germany." If normalized to the U.S. GDP for that year, these costs would be $7.4 B. Even modest improvements in measurement accuracy and quality assurance will result in multi-billion dollar savings in health-care costs. In addition to measurements reliability and related cost issues, timely completion of these standards will also assure U S. IVD industry's continued access to the European market.

Future Plans: Work will continue on the high priority list presented above. SRMs will be completed for Homocystein, Folates, Thyroid Markers in 2004. Work will begin on standards for gene expression including RNA standards and a fluorescence standard for microarray scanning devices. JCTLM Review Teams for Blood gases, Blood Groupings, Microbial Serology, Non-Electrolyte metals and Vitamins will be established for providing lists of "higher order" methods and materials by December 2004.

International Standards

Title: International Benchmarking of NIST Capabilities for Chemical Measurements

Authors: W.E. May, G.C. Turk, S.A. Wise, F.R. Guenther, G.W. Kramer, R.R. Greenberg, and R.M. Parris

Abstract: Traceability to stated references and global confidence in this realization are the basis for mutual recognition and confidence in data used to facilitate and underpin international trade and decisions regarding health, safety, commerce, and scientific studies In October 1999, the Directors of National Metrology Institutes (NMIs) for the thirty-eight member states of the Meter Convention, and representatives of two international organizations signed a Mutual Recognition Arrangement on national measurement standards and calibration and measurement certificates issued by national metrology institutes (MRA). The NIST Analytical Chemistry Division has ongoing major activities to meet the MRA requirements for NMIs:

1. Declaring and documenting calibration and measurement capabilities
2. Evidence of successful participation in formal, relevant international comparisons
3. Demonstration of system for assuring quality of each NMI's measurement services

Purpose: Traceability to stated references and global confidence in this realization are the basis for mutual recognition and confidence in data used to facilitate and underpin international trade and decisions regarding health, safety, commerce, and scientific studies In October 1999, the Directors of National Metrology Institutes for the thirty-eight member states of the Meter Convention, and representatives of two international organizations signed a Mutual Recognition Arrangement on national measurement standards and calibration and measurement certificates issued by national metrology institutes (MRA). This MRA provides an open, transparent, and comprehensive framework for obtaining reliable quantitative information on the comparability of metrological services needed for mutual recognition of national measurement standards and measurement certificates issued by national metrology institutes around the World. This Arrangement also provides governments and other parties with a secure technical foundation for wider agreements related to international trade, commerce, and regulatory affairs.

Major Accomplishments: For implementation of this MRA, the signatory NMIs agreed to:

1. declare and document their calibration and measurement capabilities (CMCs)[Appendix C]
    - CSTL's Analytical Chemistry Division has over 1100 CMCs for Chemical Measurements included in ~3000 CMCs for Chemical Measurements published in the CIPM MRA Appendix C (http://kcdb.bipm.org/AppendixC/default.asp)

2. participate in relevant international comparisons to support their CMCs [Appendix B] (http://kcdb.bipm.fr/BIPM/KCDB/, http://icdb.nist.gov)
    - CSTL's Analytical Chemistry Division has participated in >75 international comparisons to meet requirements of the CIPM MRA. In FY03, the comparisons in which ACD participated were for the determination of a variety of organic and

inorganic components in matrices such as gaseous mixtures, sediments, food, water, fuel, and serum. Results of selected recent comparisons are shown below.

3. implement and document the existence of a system for assuring the quality of the measurement services provided.
   - CSTL's Analytical Chemistry Division's Quality Manual that summarizes and formalizes policies and approaches for addressing quality-related issues concerning the services that it provides is being updated to assure appropriate compliance with ISO/EC 17025 and ISO Guide 34.

The implementation of the CIPM MRA is carried out by a Joint Committee of Regional Metrology Organizations (RMOs) and the BIPM (JCRB). The JCRB is made up of representatives from each RMO and the BIPM, and provides oversight for results included in the Key Comparisons Database (Appendix B of the MRA), as well as the determination of the degree(s) of equivalence of results from individual NMIs. RMOs have the responsibility for carrying out supplementary comparisons and other actions within their regions to support mutual confidence in the validity of calibration and measurement certificates through the Joint Committee of the RMOs and the BIPM (JCRB). They are also responsible for approval of calibration and measurement capabilities (CMCs) of their member NMIs.

Systema Interamericano de Metrologia (SIM) is the metrological regional organization (RMO) for the Americas. CSTL provides the Chair for the Chemical Metrology Working Group and SIM representative to the JCRB. In order to assure the effective, fair and metrologically sound implementation of the MRA. CSTL staff have led the critical review of both SIM and international chemistry CMC data for Appendix C of the BIPM.

CSTL staff also play a leadership role the International Committee of Weights and Measures-Consultative Committee on the Quantity of Material (CCQM). The CCQM has formed seven working groups: (1) Gas Analysis, (2) Organic Analysis, (3) Inorganic Analysis, (4) Electrochemistry (5) Biometrology, (6) Surface Analysis and (7) Key Comparisons and CMC Quality. These working groups are responsible for selecting and overseeing the operation of key comparisons that address chemical measurement-related issues important for international trade, environmental, health, and safety-related decision making. CSTL staff are active in all seven and has provided formal leadership for the Organic Analysis, Biometrology and Key Comparisons Working Groups.

Impact: NIST and other National Metrology Institutes around the world have the responsibility for establishing, maintaining, and disseminating the highest level of metrological references for a given country or economy. The calibration and measurement services that these NMIs provide must be of high quality and delivered to our customers in a consistent and transparent manner.

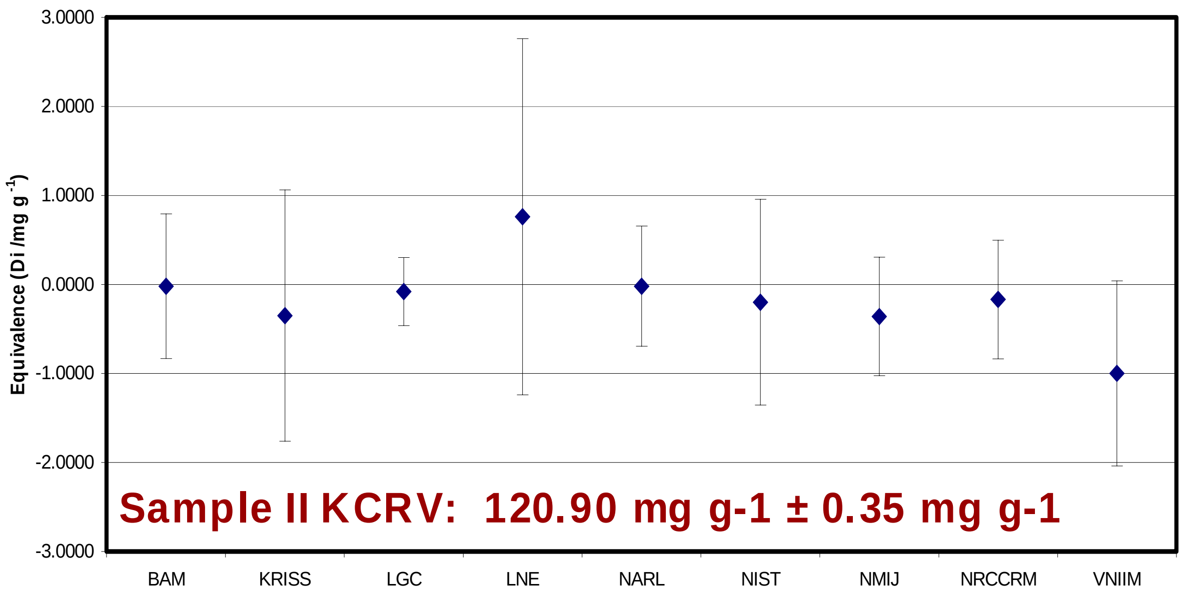
Future Plans:
Over 25 additional CCQM studies are planned to be conducted over the next two years and the Analytical Chemistry Division has already committed to coordinate at least 7 of these. In addition, the Division's Quality System for its measurement services will be presented to and assessed by the SIM Quality System Task Force. The results of this review will be reported to

the CIPM JCRB the complete the requirements for maintaining our CMCs in the CIPM Database.

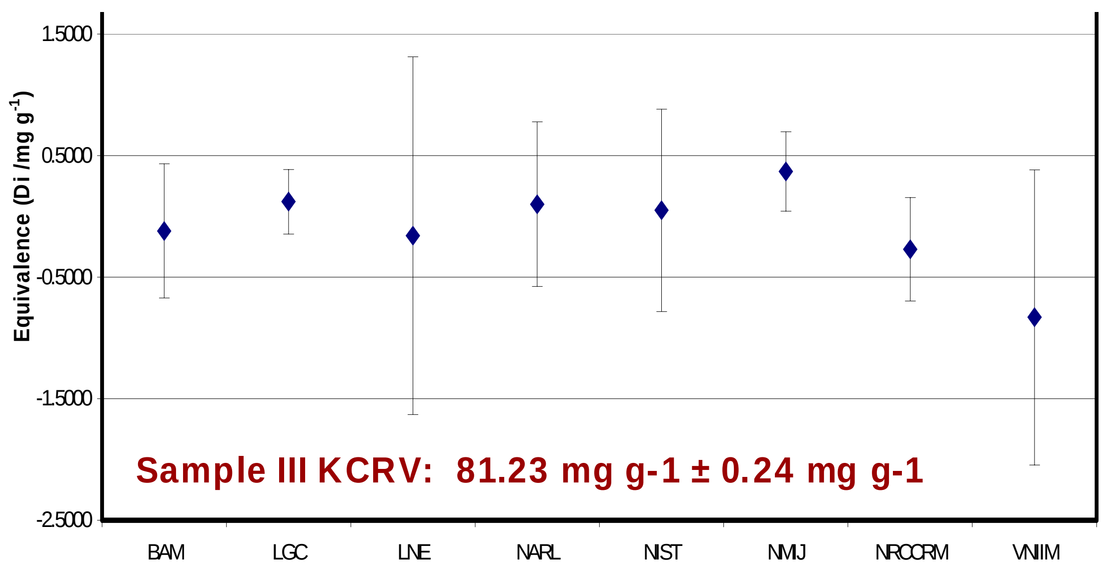
Examples of NIST participation in recently completed international comparisons

CCQM-K27: Determination of Ethanol in Aqueous Matrices
Study Period: 2002

CCQM-K27a Forensic level
Samples II (aqueous solution spiked gravimetrically with ethanol)



CCQM-K27b Commodity level
Sample III (commercial red wine stabilized by irradiation)

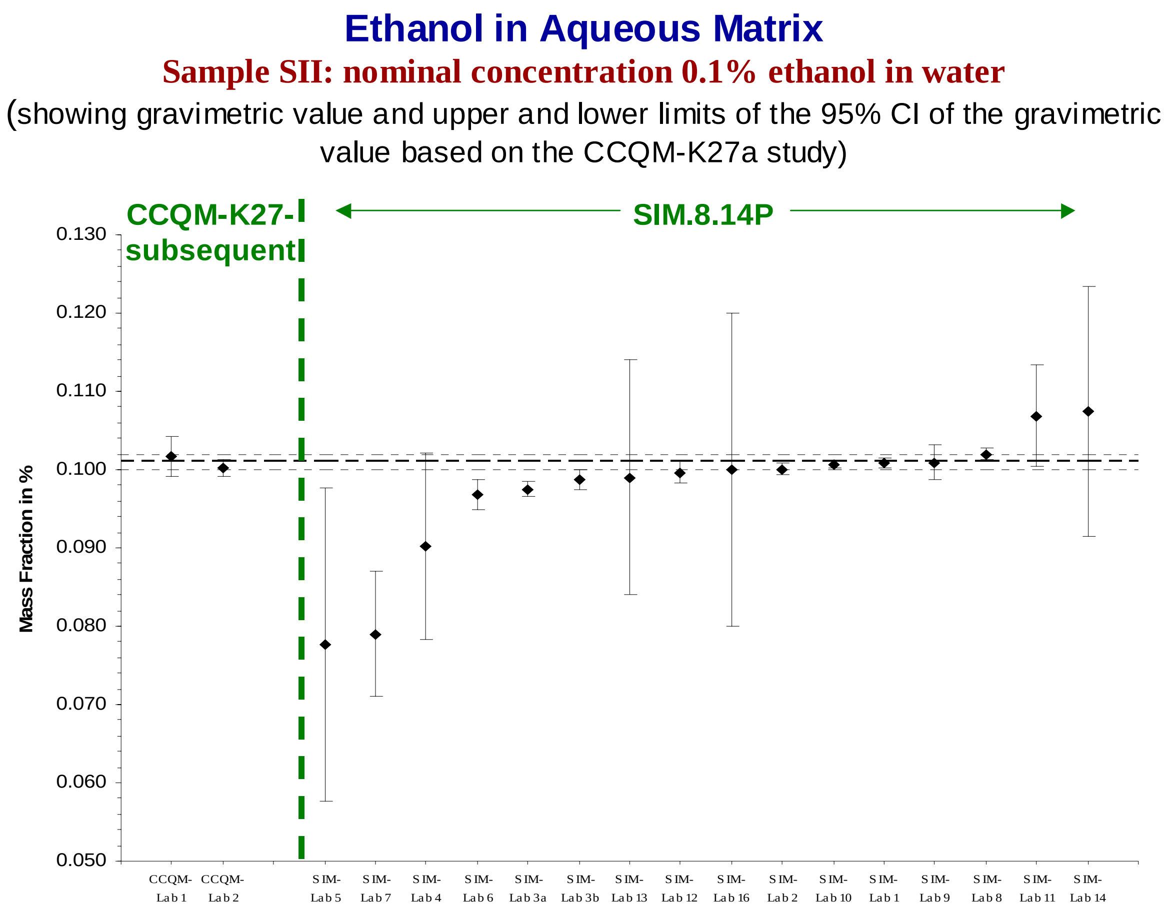


Determination of Ethanol in Aqueous Matrix

Study Period: 2003

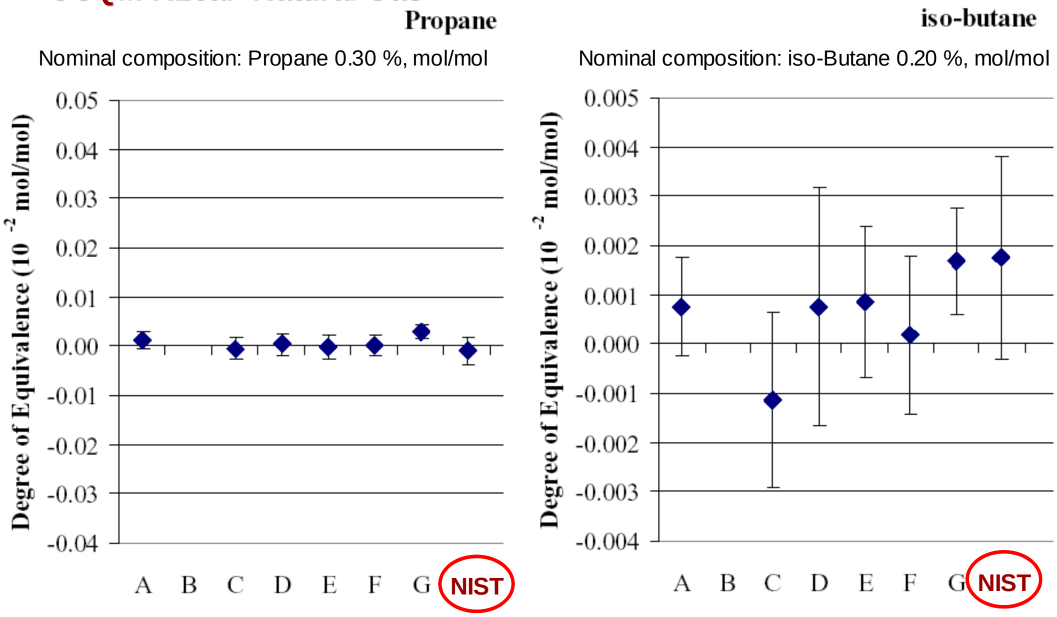
As SIM Pilot Comparison: SIM.8.14P (16 participants)

As Key Comparison: CCQM-K27-subsequent (4 participants)

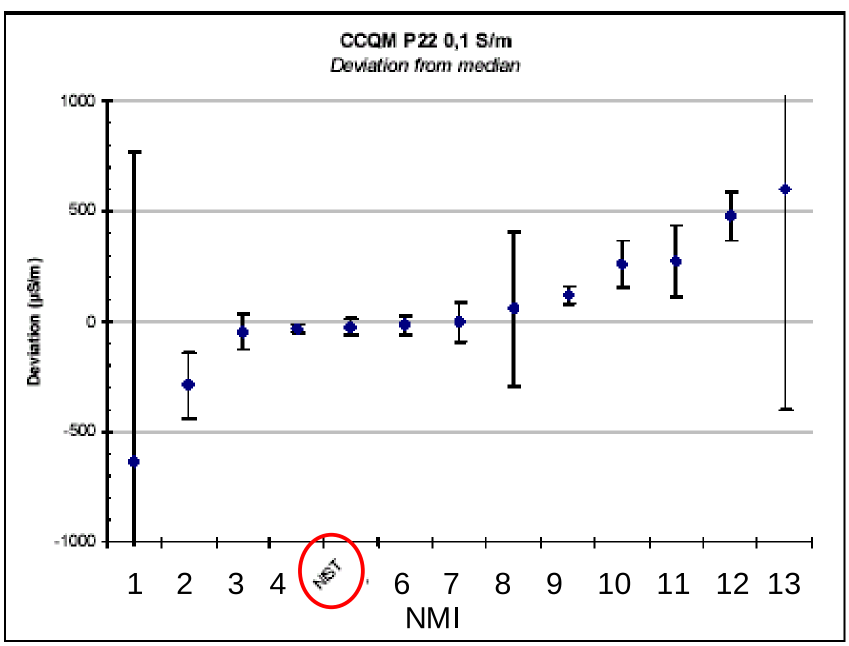


Determination of Components of Natural Gas

CCQM-K16a: Natural Gas



CCQM-P22:
Electrolytic Conductivity
(primary and secondary measurements)

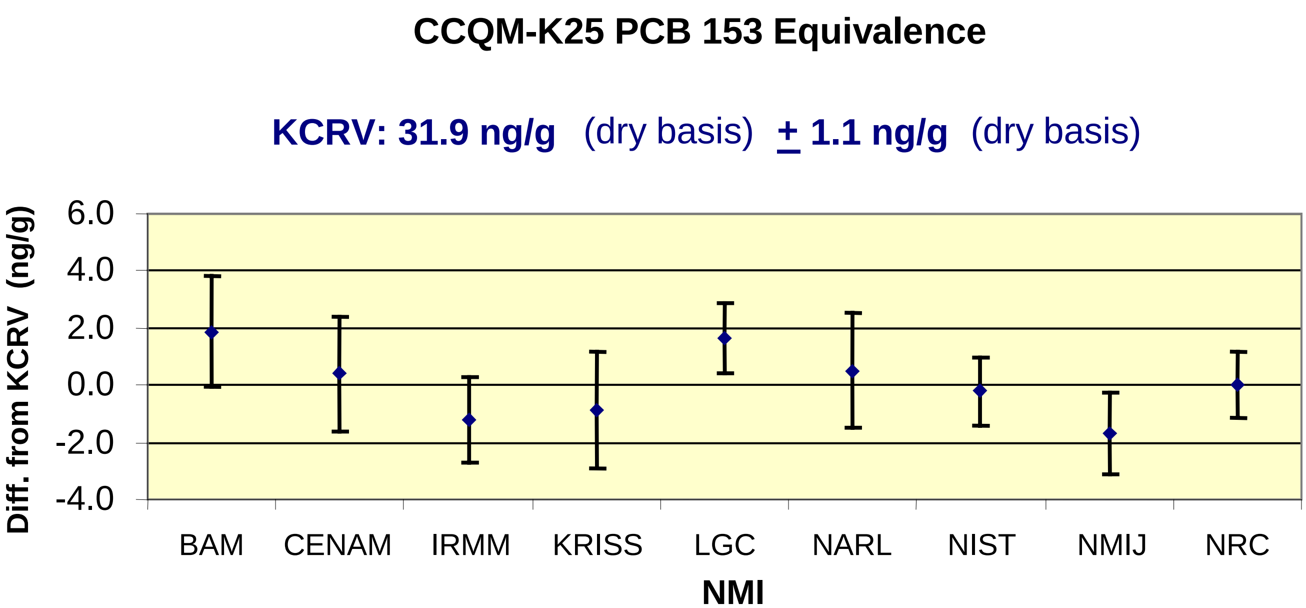


Nominal 0.1 S/m (1000 µS/cm)

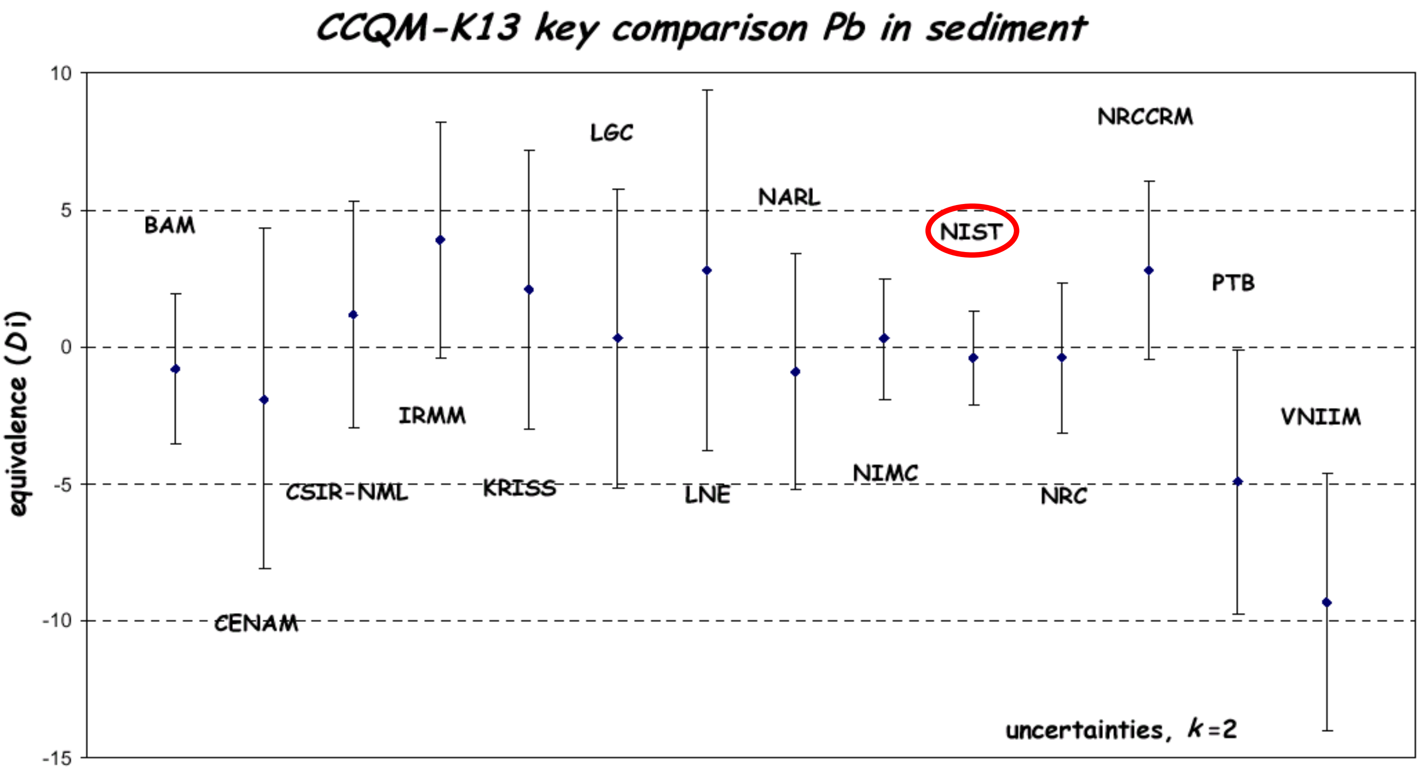
CCQM-K25: PCB Congeners in Sediment

The five PCB congeners measured in CCQM-K25 were selected to be representative of the approximately 150 congeners found in environmental samples. These five congeners also provided the typical analytical measurement challenges encountered including problematic GC separations and a wide volatility range and concentration range for the individual congeners. The results for PCT 153 are shown below.

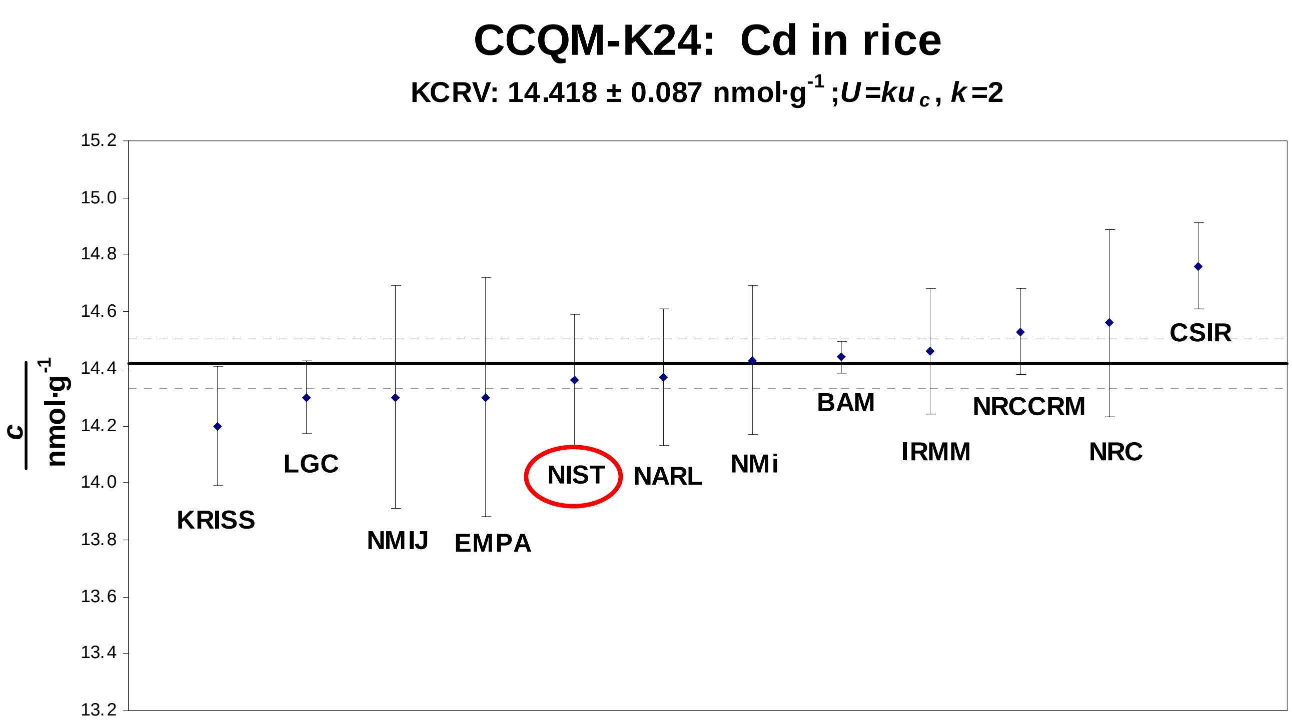
- PCB 28 - volatile and potential coelution with PCB 31
- PCB 101 - potential coelution with minor congener, PCB 90
- PCB 105* - lower concentration and potential elution order changes with PCB 153 and/or PCB 132
- PCB 153 - potential coelution with PCB 132
- PCB 170 - lower concentration and potential coelution with PCB 190



Determination of Cadmium and Lead in Sediment



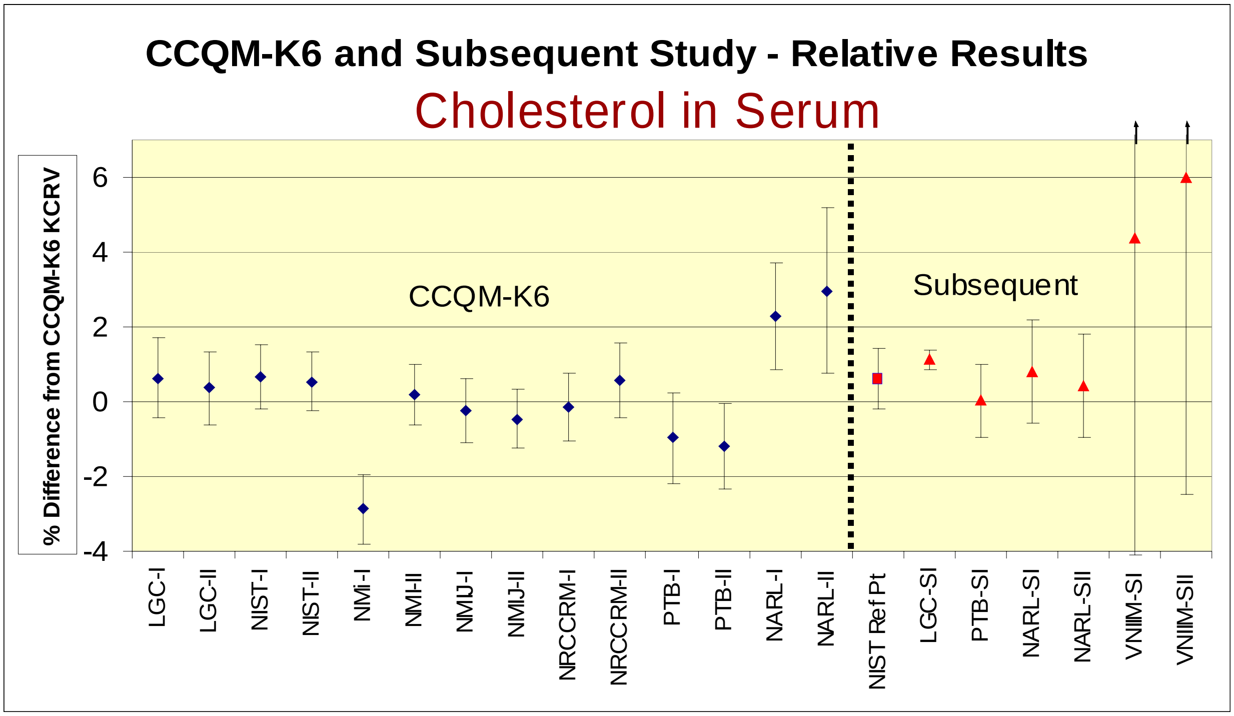
Determination of Cadmium in Rice



Expanded uncertainties with coverage factor of k=2

Uncertainty of prescribed drying protocol was major component of NIST expanded uncertainty.

Determination of Cholesterol in Serum



K6 results are plotted as % differences from KCRVs

Subsequent results are plotted relative to NIST results in K6S and are offset by average (NIST-KCRV) result from K6 (NIST Ref Pt)

International Standards

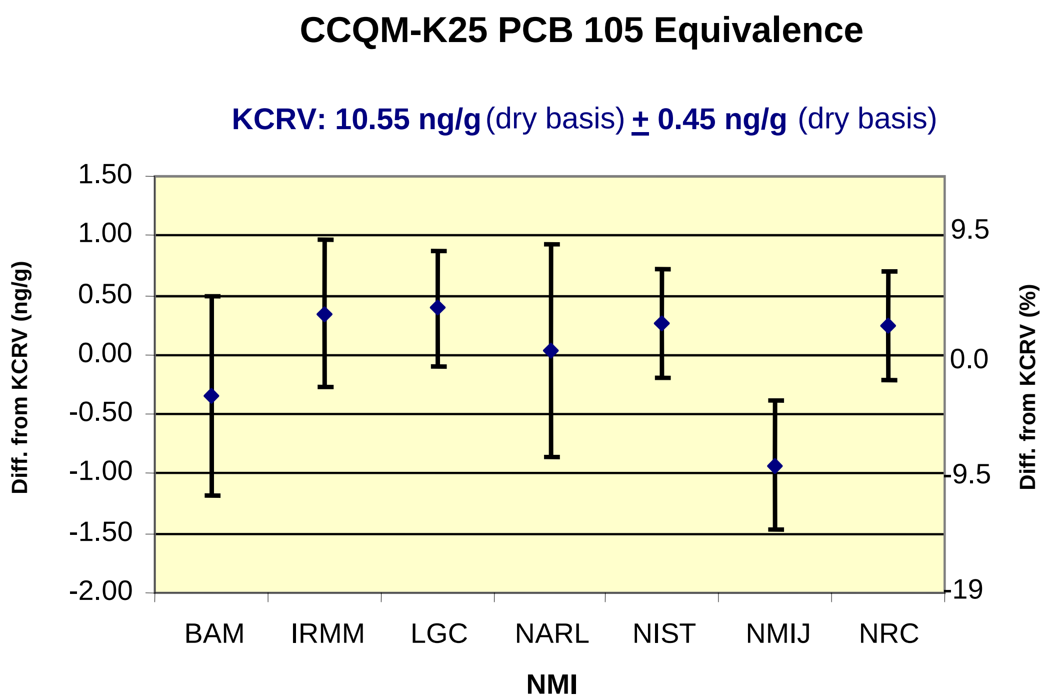
Title: Results of CCQM-K25 Key Comparison for Polychlorinated Biphenyl Congeners in Sediment

Authors: M.M. Schantz and S.A. Wise

Abstract: Polychlorinated biphenyls (PCBs) consist of 209 possible congeners depending on the substitution of the chlorine atoms around the biphenyl molecule. PCBs have been widely used as industrial fluids, flame retardants, diluents, hydraulic fluids, and dielectric fluids for capacitors and transformers. As a class of compounds, they are environmentally stable and tend to bioaccumulate. Of the 209 possible congeners, approximately 150 congeners have been reported in the environment. A CCQM key comparison for PCB congeners in sediment was conducted with NIST serving as the coordinating laboratory. For the key comparison, five target PCB congeners were selected as representative of the measurement of individual congeners in environmental samples. The target congeners included some potential problematic gas chromatography (GC) separations, and they spanned the volatility range and the typical concentration range for the 150 congeners found in environmental samples. Nine laboratories participated in the key comparison. All but one of the participants used gas chromatography/mass spectrometry (GC/MS) with carbon-13 labeled PCB congeners as internal standards/surrogates (ID-GC/MS).

Purpose: The five PCB congeners chosen were PCB 28, PCB 101, PCB 105, PCB 153, and PCB 170. PCB 28 (2,4,4'-trichlorobiphenyl) is volatile and under certain conditions coelutes with PCB 31 (2,4",5-Trichlorobiphenyl). PCB 101 (2,2',4,5,5'-pentachlorobiphenyl) has the potential coelution with a minor congener, PCB 90 (2,2',3,4',5-pentachlorobiphenyl). PCB 153 (2,2',4,4',5,5'-hexachlorobiphenyl) is typically one of the most abundant congeners and potentially coelutes with PCB 132 (2,2',3,3',4,6'-hexachlorobiphenyl). PCB 105 (2,3,3',4,4'-pentachlorobiphenyl) is a congener with a lower concentration than the other congeners selected and which may change elution order with PCB 132 depending on the analytical conditions. Finally, PCB 170 (2,2',3,3',4,4',5-heptachlorobiphenyl) is one of the less volatile congeners, is typically found at lower concentrations, and can potentially coelute with PCB 190 (2,3,3',4,4',5,6-heptachlorobiphenyl). The ability of a laboratory to measure these five congeners should indicate their ability to measure the suite of 150 PCB congeners found in sediments.

Major Accomplishments: This Key Comparison study demonstrated a high level of equivalence in capabilities of the participating NMIs to successfully measure five PCB congeners (congener numbers 28, 101, 105, 153, and 170) in a moderately contaminated sediment using ID-GC/MS-based methods. The uncertainties of the key comparion reference values (KCRVs) ranged from 2% to 5% indicating excellent agreement among the participants. The following figure of equivalence for PCB 105 shows the excellent agreement among the laboratories and the comparison to the KCRV. Similar degrees of equivalence were demonstrated for the other four congeners.



Impact: The five PCB congeners measured in CCQM-K25 were selected to be representative of the approximately 150 congeners found in environmental samples. These five congeners also provided the typical analytical measurement challenges encountered in the measurement of individual congeners, including problematic GC separations and a wide volatility range and concentration range for the individual congeners. The ability of the laboratories to measure these five congeners should translate to the ability to measure the typical suite of PCB congeners found in sediments at concentrations encountered in moderately contaminated sediments (> 5 ng/g dry basis).

Future Plans: To help understand differences among the laboratories in calculating uncertainties, a pilot study will be conducted using a calibration solution, an extract, a spiked extract, and a tissue sample with the same five PCB congeners as the targets. The primary focus of this study will be to identify sources of uncertainties for each step of the analytical method.

International Standards

Title: Results from International Key Comparison for Calcium in Serum

Authors: S.E. Long and K.E. Murphy

Abstract: The Analytical Chemistry Division successfully participated in a key comparison of calcium in blood serum (CCQM-K14), which was conducted by the Inorganic Analysis Working Group of the CCQM. The calcium was determined using isotope dilution inductively coupled plasma – mass spectrometry. The result was in excellent agreement with the other participating laboratories and almost identical to the key comparison reference value chosen to represent the amount content of calcium in the serum material.

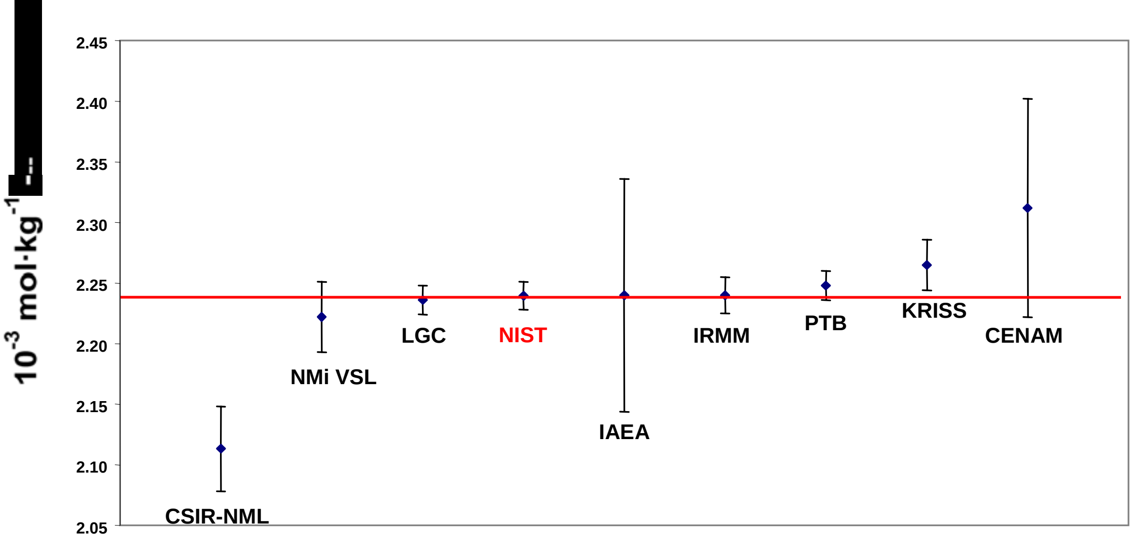
Purpose: Successful participation in CCQM key comparison studies is an important requisite for the demonstration of analytical measurement competence by national metrology institutes (NMIs). Key comparisons are an important component of the CIPM Mutual Recognition Arrangement and establish a quantitative measure of the degree of equivalence of national measurement standards of signatory NMIs.

Major Accomplishments: Under the auspices of the CCQM Inorganic Analytical Working Group (IAWG), the key comparison, CCQM-K14, was aimed at addressing the need for better measurement methodology for routine clinical measurements of total calcium in blood serum. It is evident that the current measurement system is not satisfactory from a clinical diagnostic standpoint, and as a result, the impact on health-care costs may be considerable. Following successful participation by ACD in the pilot study, which was conducted during FY 01, calcium was determined in an un-modified frozen human serum, which was distributed by European Commission's Institute for Reference Materials and Measurements (IRMM) as the pilot institute. Eight other national metrology institutes also participated. Calcium was determined using an isotope dilution "cold-plasma" inductively coupled plasma – mass spectrometry (ICP-MS) measurement. The result (2.2395 ± 0.0115 $mmol.kg^{-1}$) was in excellent agreement with the other laboratories and also the proposed value for the key comparison reference value, which was 2.240 ± 0.008 $mmol.kg^{-1}$. The analytical data from the key comparison are shown in Figure 1. It is interesting to note the shift from using thermal ionization mass spectrometry (TIMS) to ICP-MS for making such measurements, which has been made possible by the advent of systems for reducing or eliminating spectral interferences. Of the nine participants in the study, only two used TIMS. The remainder used a combination of high-resolution sector field, collision cell and cold-plasma ICP-MS, while one laboratory used ICP optical emission spectrometry.

Impact: The veracity of the analytical measurement system for calcium in blood serum has been demonstrated. Such measurements provide documented performance for the NIST quality system which is an integral component of its measurement services. The final report for CCQM K-14 concludes that the analytical procedures developed during the study are also relevant to other analytes in serum, so the comparison study has extended benefits. The material will be used in the future for external quality assurance programs.

Future Plans: Participation in appropriate CCQM / IAWG comparison exercises will continue to demonstrate the quality of the analytical measurement capabilities of NIST which underpin measurement services for its customer base.

Figure 1: CCQM-K14 Calcium in Serum



International Standards

Title: Results from International Comparisons for Selected Components in Natural Gas

Authors: G.C. Rhoderick and F.R. Guenther

Abstract: NIST participated in the CCQM-K16 Natural Gas Key Comparison in which the final report was released in October of 2002. The study evaluated the degree of equivalence for the given measurand in natural gas for National Metrology Institutes (NMI). Unlike past comparisons, this natural gas included heavier hydrocarbons and was designed to emulate real natural gas at both high and low calorific value. Due to shipping problems, NIST was able to analyze only the cylinder containing the low calorific natural gas mixture. NIST's results show excellent agreement with the gravimetric key comparison reference values. A similar Pilot Study was organized by NIST for the SIM countries. Results of the SIM 8-P8 pilot study on natural gas reveal much larger deviations than those of the CCQM key comparison.

Purpose: These comparisons were designed to evaluate the capabilities of National Metrology Institutes to certify natural gas standards, and are intended to provide evidence for calibration and measurement capabilities listed by each NMI in the BIPM MRA Database.

Major Accomplishments: NIST completed the analysis of the low calorific value natural gas standard in FY03 and released the report to the pilot laboratory. The high calorific valued natural gas standard was not analyzed by NIST, as the cylinder was lost in shipment and there was not enough time to replace it before the Key Comparison ended. Table 1 lists the NIST results of the comparison.

Table 1. Agreement between NIST value and Preparation Value (given as % mol/mol).

Compound	Reference Value	Reference Value	NIST Value Uncertainty	NIST Value Uncertainty	Absolute Difference Δ_x	Relative Difference $\Delta_{x/x}$
Methane	81.6826	0.0027	81.77	0.46	0.087	0.11 %
Nitrogen	12.1205	0.0007	12.11	0.16	-0.010	-0.09 %
CO_2	4.0446	0.0005	4.055	0.026	0.010	0.26 %
Helium	0.50813	0.00016	0.5158	0.0026	0.0077	1.51 %
Ethane	0.7489	0.0002	0.7493	0.0046	0.0004	0.05 %
Propane	0.29144	0.00010	0.2905	0.0026	-0.0009	-0.32 %
iso-Butane	0.20165	0.00019	0.2034	0.0019	0.0018	0.87 %
n-Butane	0.20260	0.00007	0.2024	0.0018	-0.0002	-0.10 %
iso-Pentane	0.04861	0.00011	0.0483	0.0006	-0.00031	-0.64 %
n-Pentane	0.04982	0.00011	0.0500	0.0007	0.00018	0.36 %
neo-Pentane	0.04972	0.00006	0.0495	0.0007	-0.0002	-0.44 %
n-Hexane	0.05061	0.00011	0.0502	0.0008	-0.0004	-0.81 %

A similar Pilot Study was organized by NIST for the SIM countries. Results of the SIM 8-P8 pilot study on natural gas reveal much larger deviations than those of the CCQM key comparison. As this was a pilot study, the results are not to be made publicly available. All the participating labs submitted methane values that were within ≤ 1.0 % of the certified concentration. However, the agreement among labs for all of the remaining compounds was poor. One lab was consistently within ± 1.5 % of the certified values with the exception of carbon dioxide (4.6 %) and n-butane (1.7 %). Therefore much work remains to be done in SIM to bring the comparability among SIM NMIs in line with the CCQM.

Future Plans: There will be another Natural Gas CCAM Key Comparison in FY2004. However since NIST has participated in the CCQM-K16 natural gas comparison with good results, NIST has decided to forgo this comparison. A SIM pilot study on automobile emission gases, piloted by NIST, is ongoing in FY2004, and will be completed by March 2004. A new SIM pilot study on industrial gas emission (sulfur dioxide) will be piloted by NIST, and initiated in late FY2004.

International Standards

Title: Development and Dissemination of Intrinsic Standards for Chemical Measurements

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Abstract: Intrinsic standards "based on well characterized laws of physics, fundamental constants of nature, or invariant properties of materials" (ANSI/NCSL Z540) have many uses in physical and chemical metrology. Such standards can reduce the need for (1) labor-intensive artifact standard production by National Metrology Institutes (NMIs) and (2) repeated costly laboratory measurement comparisons associated with mutual recognition agreements.

Purpose: Intrinsic standards enable NMIs to concentrate efforts on metrology rather than artifact production or calibration, and offer end-users of commercial standards ready access to traceability through data. Economy of scale encourages users to purchase the physical realization of an intrinsic standard (an artifact transfer standard) rather than constructing it themselves. Thus, the production of artifact standards devolves from the NMI to the commercial sector, which is better suited to production.

Major Accomplishments: Three separate efforts within the Analytical Chemistry Division report significant progress. (1) In collaboration with the NIST Physics Laboratory (NIST/PL), we have prepared the first of three manuscripts reporting improved spectral line positions for mercury and argon atomic lines in low-pressure discharges. These positions serve as secondary length standards and support higher accuracy in the determination of atomic line positions in such analytical plasmas as the Inductively Coupled Plasma (ICP). (2) SRD 79 Quantitative Infrared Database is an on-going project providing quality-assured quantitative infrared absorption reference data to support open-path Fourier transform infrared (FT-IR) measurements, such as those described in EPA Method TO-16. New spectra based on gravimetrically prepared samples are added to the database as they are acquired. Comparisons with Pacific Northwest National Laboratories continue to play a key role in validating the data. (3) Samples of SRM 2034 (dilute holmium oxide solution sealed into fused silica cuvettes) were distributed to fifteen NMIs. Results from these laboratories, NIST/PL, and our own reference instruments will form the basis for an internationally supported intrinsic wavelength standard for UV/visible chemical spectrophotometry.

Impact: Intrinsic standards provide a passive means of support for our customers, requiring attention only at wide intervals to improve uncertainties or respond to emerging needs. End users or standards producers may construct artifacts deriving from these standards – such as atomic pen lamps, bottled gas standards, and sealed solution wavelength standards – with traceability provided by data. This provides economic benefits and improved responsiveness to NIST and other NMIs, commercial standards producers, and end users alike.

Future Plans: The atomic wavelength project is essentially complete, awaiting only the publication of the three manuscripts. The database for molecular gas cross sections is a continuing project, with improvements planned including Internet distribution. Long-term plans for the database include investigating calibration transfer issues and international comparisons of the data. The intrinsic wavelength standard should be completed in FY04, with the possible extension to simultaneous coverage of wavelength and absorbance in the future.