

Gas Metrology and Classical Methods

Franklin R. Guenther

NIST

100 Bureau Drive

Gaithersburg MD 20899-8393

NIST Gas NTRM Meeting

☀️ Agenda:

- 🌟 1:30 Welcome
- 🌟 1:45 Summarizing the first 8 years of the gas NTRM Program (Bill Dorko)
- 🌟 2:30 Proposal for new gas NTRM certification program
- 🌟 3:00 Discussion
- 🌟 3:45 ISO TC158 Discussion
- 🌟 4:30 Adjourn

NIST Gas SRM Status

SRM#	Description	!Total
1625	10 cm SO2 Permeation Device	51
1626	5 cm SO2 Permeation Device	22
1658A	1 ppm Methane in Air	51
1659A	10 ppm Methane in Air	20
1660A	4 ppm Methane, 1 ppm Propane in	26
1661A	500 ppm SO2 in N2	20
1662A	1000 ppm SO2 in N2	19
1663A	1500 ppm SO2 in N2	23
1664A	2500 ppm SO2 in N2	25
1665B	3 ppm Propane in Air	22
1666B	10 ppm Propane in Air	2
1667B	50 ppm Propane in Air	4
1668B	100 ppm Propane in Air	4
1669B	500 ppm Propane in Air	1
1671	342 ppm CO2 in Air	21
1672	351 ppm CO2 in Air	21
1674B	7% CO2 in N2	38
1675B	14% CO2 in N2	28
1676A	362 ppm CO2 in Air	0
1677C	10 ppm CO in N2	0
1678C	50 ppm CO in N2	48

1679C	100 ppm CO in N2	13
1680B	500 ppm CO in N2	30
1681B	1000 CO in N2	48
1683B	50 ppm NO in N2	27
1684B	100 ppm NO in N2	55
1685B	250 ppm NO in N2	50
1686B	500 ppm NO in N2	50
1687B	1000 ppm NO in N2	44
1693A	50 ppm SO2 in N2	30
1694A	100 ppm SO2 in N2	59
1696A	3500 ppm SO2 in N2	29
1800	Non-Methane Organic Carbon (NMO)	9
1804A	Ambient Toxic Organics in N2	0
2612A	10 ppm CO in Air	35
2613A	20 ppm CO in Air	32
2614A	45 ppm CO in Air	17
2619A	0.5% CO2 in N2	38
2620A	1.0% CO2 in N2	32
2621A	1.5% CO2 in N2	42
2622A	2.0% CO2 in N2	25
2623A	2.5% CO2 in N2	16
2624A	3.0% CO2 in N2	40

NIST Gas SRM Status

2625A	3.5% CO ₂ in N ₂	40
2626A	4.0% CO ₂ in N ₂	14
2627A	5 ppm NO in N ₂	0
2628A	10 ppm NO in N ₂	0
2629A	20 ppm NO in N ₂	7
2630	1500 ppm NO in N ₂	17
2631A	3000 ppm NO in N ₂	34
2635A	25 ppm CO in N ₂	29
2636A	250 ppm CO in N ₂	17
2637A	2500 ppm CO in N ₂	18
2638A	5000 ppm CO in N ₂	14
2639A	1% CO in N ₂	34
2640A	2% CO in N ₂	40
2641A	4% CO in N ₂	32
2642A	8% CO in N ₂	13
2643A	100 ppm Propane in N ₂	0
2644A	250 ppm Propane in N ₂	0
2645A	500 ppm Propane in N ₂	0
2646A	1000 ppm Propane in N ₂	0
2647A	2500 ppm Propane in N ₂	0
2648A	5000 ppm Propane in N ₂	0
2649A	1% Propane in N ₂	0

2650	2% Propane in N ₂	0
2657A	2% Oxygen in N ₂	42
2658A	10% Oxygen in N ₂	39
2659A	21% Oxygen in N ₂	36
2660	Oxides of Nitrogen in Air	43
2727	1.6% CO, 600 ppm Propane and 11	0
2728	8% CO, 3000 ppm Propane and 14	0
2730	5 ppm H ₂ S in N ₂	84
2731	20 ppm H ₂ S in N ₂	54
2735	800 ppm NO in N ₂	31
2736	2000 ppm NO in N ₂	0
2740	10% CO in N ₂	22
2741	13% CO in N ₂	1
2745	16% CO ₂ in N ₂	29
2750	50 ppm CH ₄ in Air	18
2751	100 ppm CH ₄ in Air	11
2764	0.25 ppm Propane in air	29

Gas Standards Survey

Priority List

Low Concentration NO (0.1 - 1 ppm)

Hydrogen Chloride (0.5 - 500 ppm)

Ammonia (1-200 ppm)

Low Concentration SO₂ (0.5 to 1 ppm)

Chlorine (1 - 1000 ppm)

Global Warming gases

Mercury

CCQM Intercomparisons

- ✦ BTX – ongoing
- ✦ Ethanol
- ✦ CO, CO₂, Propane
- ✦ Future
 - ✦ Global Warming
 - ✦ Low Conc NO, SO₂
 - ✦ Ozone
 - ✦ Others?

Group Structure

☀ Classical Methods

- ☀ 4 Scientists
- ☀ STRS Support – 30%
- ☀ SRM support – 60 %

☀ Gas Metrology

- ☀ 7 Scientists
- ☀ 4 Technicians
- ☀ STRS Support – 27 %
- ☀ OA Support – 6 %
- ☀ Standards Activities
 - ☀ SRM – 20 %
 - ☀ Customer – 36 %
- ☀ Ozone Program
Added in 1999

International Activities

☀ Classical Methods

✳ CCQM

- pH, Conductivity, Purity assessment

✳ SIM

- pH Intercomparison
- Course

✳ Bilateral

- Conductivity – DFM, OMH

☀ Gas Metrology

✳ CCQM

- BTX Intercomparison

✳ SIM

- Auto Exhaust Intercomparison
- Course

✳ Bilateral

- NMI – Inorganic Gas
- NPL – Organic, IR DB

Traceability

☀ Industry Requirements

- ☀ To a National Standard
- ☀ Global Economy requires global traceability
 - ☀ European Directive for health monitors – just the beginning
 - ☀ Specialty Gas Industry – 3 to 4 global companies

Gas NTRM Program

- ☀ SGC Produces NTRM
- ☀ SGC Analyzes Batch
- ☀ NIST Reviews data, selects cylinders
- ☀ NIST analyzes selected cylinders
- ☀ NIST certifies batch using NIST/SGC data
- ☀ SGC retains NTRM batch for use/sale

Program Results

- ☀ 8 years experience with program
 - ★ Steady growth to \$500K annually
 - ☀ Approx 80 batches
 - ☀ 2500 - 3000 cylinders
 - ☀ 125000 – 150000 traceable standards
 - EPA Protocol Gas Standards
- ☀ Accepted by EPA
- ☀ Global Community
 - ★ Acceptance, copied

NTRM - Future

- ☀ Effectively replace most gas SRMs
 - ☀ Promote NTRM sale to end users
- ☀ Remove individual batch certification by NIST

New Gas NTRM Program

- ☀ SGC is certified to produce gas NTRMs
 - ✳ Must have successful history of NTRM production
 - ✳ Accredited to ISO 17025
 - ✳ Analytical systems are certified for NTRM certification
 - ✳ Using p -NTRM certified at NIST

New Gas NTRM Program

- ☀ SGC sends candidate *p*-NTRM to NIST
 - ✳ Concentration suite to cover range
- ☀ NIST certifies *p*-NTRM against PSMs
 - ✳ Best uncertainty possible
 - ✳ Better than available SRMs
 - ✳ Certified for 1 year only

New Gas NTRM Program

- ☀ SGC uses *p*-NTRM to certify NTRM
 - ✳ Within range of suite, using approved analytical system
 - ✳ Certified value and uncertainty generated by NIST software
 - ✳ Certificate generated by software
 - ✳ Data sent to NIST quarterly

New Gas NTRM Program

☀ NIST Quality Assurance

- ☀ Through *p*-NTRM and Software
- ☀ May call for NTRM periodically
- ☀ Will send challenge cylinder to SGC
 - ☀ SGC certifies and send certificate to NIST
 - ☀ Certification must agree with NIST analysis
 - Within stated uncertainty

New Gas NTRM Program

- ☀ Elimination of most gas SRMs
 - ☀ Some always necessary
- ☀ NTRM Production not limited
- ☀ p -NTRM available to SIM countries
- ☀ International Acceptance
- ☀ NIST able to turn to new gas standards

ISO TC 158: Analysis of Gases

- ☀ No active US Participation
- ☀ Participating Members (Voting):

Belgium	Portugal
Czech Republic	Russian Federation
Germany	Spain
Hungary	Sweden
Italy	Ukraine
Netherlands (S)	United Kingdom
Poland	

Working Groups

1. Terminology
2. Quality assurance of gas analysis
3. Gravimetric methods
4. Comparison methods and certificates
5. Static and dynamic volumetric methods
6. Quantities of composition

Standards

- FDIS 6141: Requirements on certificates for gases and gas mixtures
- DIS 6142: Preparation of calibration gas mixtures – Gravimetric method
- DIS 6143.2: Determination of the composition of calibration gas mixtures – comparison methods
- DIS 6144.2: Preparation of calibration gas mixtures – static volumetric methods
- CD 6145-1: Preparation of calibration gas mixtures – Dynamic volumetric methods – methods of calibration

Standards

- CD 6145-2: Preparation of calibration gas mixtures – Dynamic volumetric methods – Volumetric pumps
- 6145-3: Preparation of calibration gas mixtures – Dynamic volumetric methods – periodic injections into a flowing gas stream
- 6145-4: Preparation of calibration gas mixtures – Dynamic volumetric methods – continuous injection method
- 6145-5.2: Preparation of calibration gas mixtures – Dynamic volumetric methods – Capillary calibration devices
- 6145-6: Preparation of calibration gas mixtures – Dynamic volumetric methods – Critical orifices

Standards

- ☀ 6145-7: Preparation of calibration gas mixtures – Dynamic volumetric methods – Thermal mass flow controllers
- ☀ 6145-8: Preparation of calibration gas mixtures – Dynamic volumetric methods – Diffusion
- ☀ 6145-9: Preparation of calibration gas mixtures – Dynamic volumetric methods – Saturation methods
- ☀ 6145-10: Preparation of calibration gas mixtures – Dynamic volumetric methods – Permeation methods
- ☀ 7504: Vocabulary

Standards

- ☀ 14166: Pure gases – Requirements for presentation of the results of gas purity analysis
- ☀ 14167: General quality assurance aspects in the use and preparation of reference gas mixtures – guidelines
- ☀ 14912: Quantities of composition
- ☀ 15796: Detection and correction of analytical bias
- ☀ 16664: Handling of calibration gases and gas mixtures - guidelines

U.S. Participation

☀ ANSI

- ☀ Requires formation of Technical Activity Group (TAG)
 - ☀ Freely open to all who wish to participate
 - ☀ Reports to ANSI
 - ☀ Requires yearly financial support to ANSI
 - \$3155 for TAG
 - \$490 per Working Group
 - ☀ Must be accredited by ANSI

Possible TAG Secretariat

- ☀️ ASTM through D22

- ☀️ Will they take on another ISO TG?

- ☀️ NIST

- ☀️ Is this a NIST Role??

- ☀️ Requires support from?

- ☀️ Others?