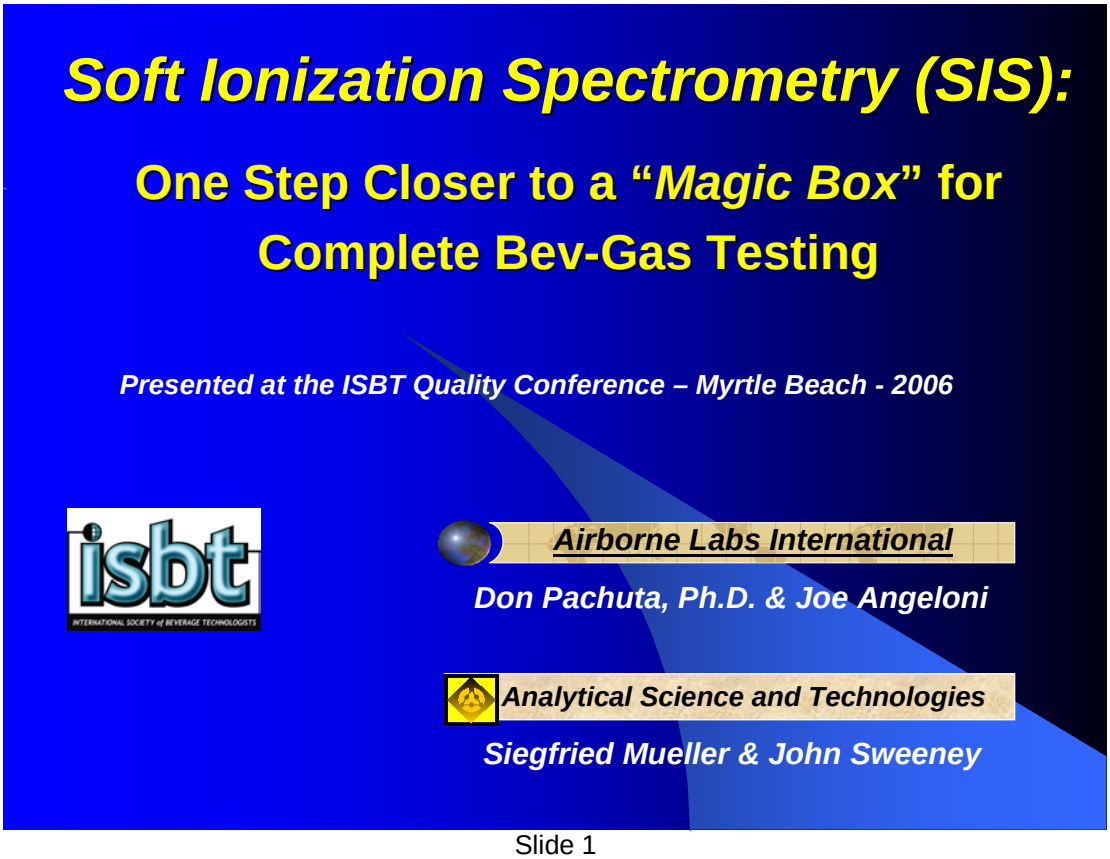


Slide 1

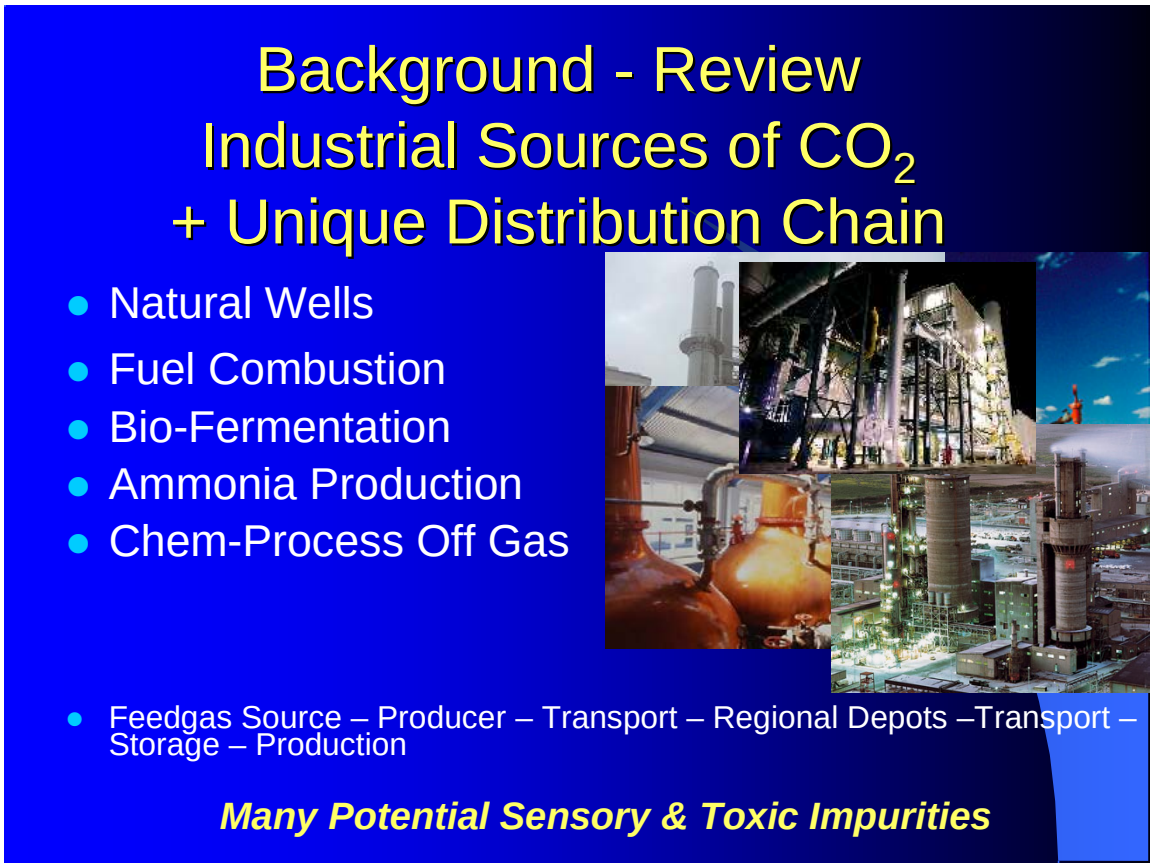
Soft Ionization Spectrometry (SIS): One Step Closer to a "Magic Box" for Complete Bev-Gas Testing

Don Pachuta, Ph.D. & Joe Angeloni, Airborne Labs International Inc.,
Siegfried Mueller & John Sweeney, Analytical Science and Technologies

1. Background Information

Carbon Dioxide (CO_2) quality assurance has recently become an important issue for both large and small international gas suppliers as well as most beverage manufacturers. This interest has been driven in part by a rise in worldwide demand for CO_2, which requires an increased use of non-traditional liquid CO_2 sources and also by efforts to establish harmonized product quality standards. Common CO_2 feedgas sources now include, for example, natural wells, combustion of natural gas, liquid and solid fuels (e.g. coal), bio-fermentation, ammonia production and chemical off-gas streams (see Slide #2). Each feedgas source has a unique set of diverse impurities that must be removed or reduced to acceptable levels. Due to the complex distribution network associated with commercial liquid CO_2, various impurities can also be introduced between the point of CO_2 production and the end-use (e.g. packaged beverage). Many impurities can impart unpleasant sensory and/or potential health-risk properties to a carbonated beverage. To ensure that a proper grade of CO_2 is used for beverage production, demanding "bev-grade" purity guidelines and regulatory standards have been developed by ISBT (ref. "CARBON DIOXIDE Quality Guidelines and Analytical Procedure Bibliography" – 2001) and

other organizations. These purity guidelines (plus manufacturer “add-on” requirements) have been widely adapted by the international beverage community over the past few years.



Slide 2.

2. ISBT CO_2 Bev-Gas Quality Challenges (Slide #3)

A review of ISBT published, extensive CO_2 impurity list and maximum guideline limits is useful for perspective (see Slide# 4). CO_2 transport, storage and handling issues have also been addressed, including sampling procedures, testing frequency and acceptable analytical test methodologies. To meet the challenge of routinely monitoring this *extremely* diverse range of impurities (many of which have low ppb maximum limits), resulting analytical programs and instrumentation have necessarily been quite complex and require multiple analyzer-based systems. The most common systems used-to-date are briefly reviewed (see Slide #5). Due to the stresses of routinely maintaining and operating these multiple analyzer systems, the desire for a less complex “single unit – magic box” analyzer system that meets the requirements of both CO_2 bev-gas producers and consumers has become a common goal for the beverage industry.

ISBT Bev-Gas Quality Challenges

- As beverage production grows, ***continuous*** quality testing becomes v important
- CO_2 Quality Assurance is a complex technical challenge
- All dream of a "Magic Box" that can quickly monitor the ***entire ISBT Impurity List***

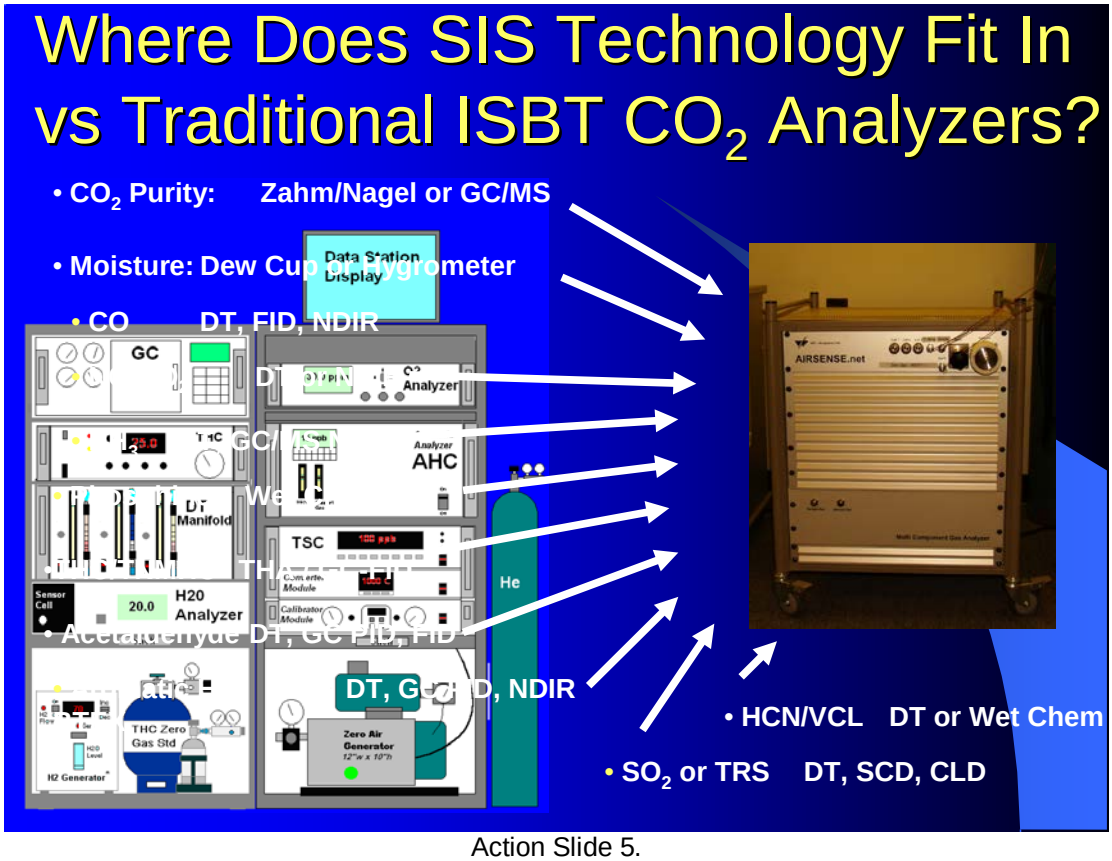
Slide 3.

ISBT CO_2 Purity Guidelines

"The List"

CO_2 Purity	99.9% min	Process
Moisture (H_2O)	20 ppm v/v max	Process
Oxygen (O_2)	30 ppm v/v max	Sensory
Carbon Monoxide (CO)	10 ppm v/v max	Process / Regulatory
Ammonia (NH3)	2.5 ppm v/v max	Process
NO / NO_2	2.5 ppm v/v (ea) max	Regulatory
Non-Volatile Residue (NVR)	10 ppm x/x max	Sensory
Non-Volatile Organic Residue (NVOR)	5 ppm w/w max	Sensory
Phosphine (PH_3)	0.3 ppm v/v max	Regulatory
Total Volatile Hydrocarbons (THC) & TNMHC	50 ppm v/v max – including 20 ppm v/v TNMHC	Sensory
Acetaldehyde (AA)	0.2 ppm v/v max	Sensory
Aromatic Hydrocarbon Content (AHC)	20 ppb v/v max (as benzene)	Regulatory
Total Sulfur Content (TSC*) less SO_2	0.1 ppm v/v max	Sensory
Sulfur Dioxide (SO_2)	1 ppm v/v max	Sensory
Odor of Snow	No foreign odor	Sensory
Appearance in Water	No color or turbidity	Sensory
Odor & Taste in Water	No foreign odor/taste	Sensory
Supplemental HCN / Vinyl Chloride	None detectable–best method	Report

Slide 4.



Action Slide 5.

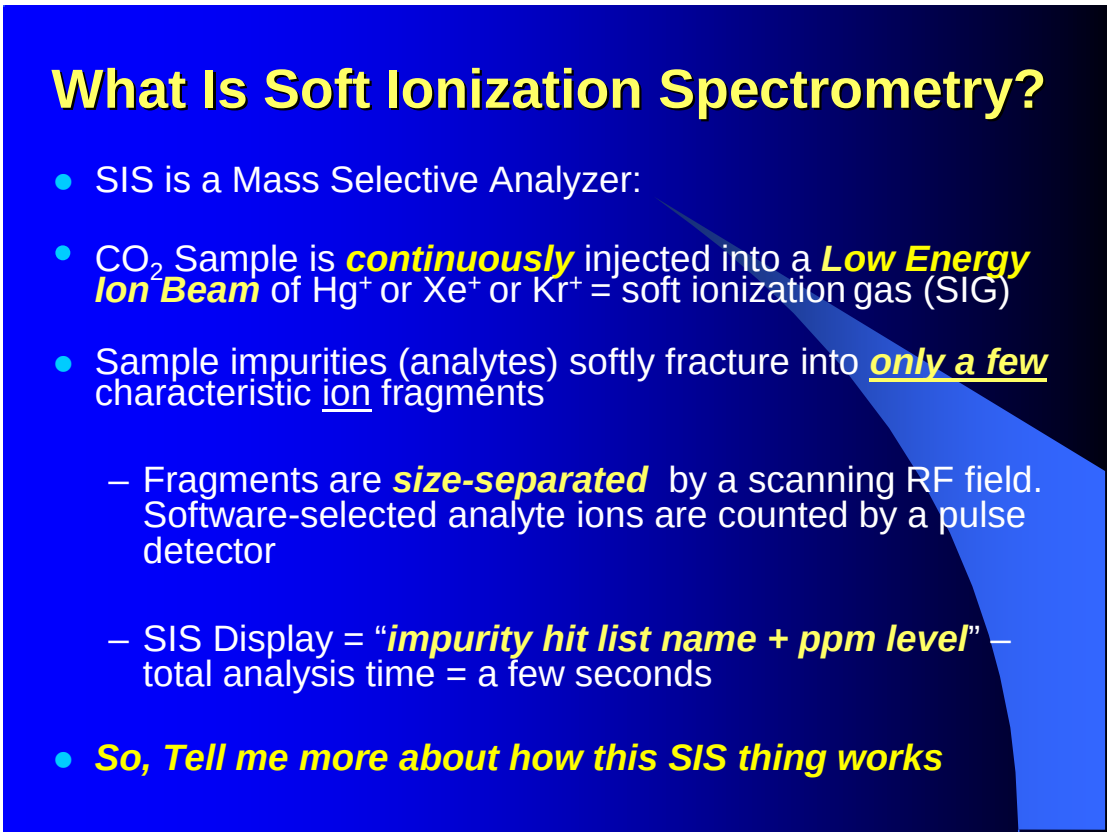
3. <u>Soft Ionization Spectrometry: Technology Description / Overview (Slides 6-11)</u>

Soft Ionization Spectrometry (SIS) is a relatively mature (20+ yr) high-vacuum-based technology that employs a 3-step process of continuous gaseous sample introduction, low energy sample ionization and rapid mass ion separation to identify and quantify a wide molecular mass range of both gaseous organic and inorganic impurities. This technology uniquely employs a focused beam of ionized gas (krypton [Kr], xenon [Xe] or mercury [Hg]) as an energy-transfer medium that "softly" ionizes any sample analytes that it "collides" with. This "soft" ionization method results in a low degree of molecular (parent) ion "fragmentation". As a result, SIS often achieves higher sensitivity and lower detection limits than many other forms of mass spectrometry that employ high-energy heated filaments for <u>direct</u> sample ionization. For continuous measurement of trace CO_2 impurities, this is an important advantage, as "soft-ionization gas" (SIG) excitation does not lead to a significant amount of CO_2 matrix ionization. This key property allows a high percentage of the SIG beam's energy to be available for trace impurity "excitation-ionization" and resulting signal generation. A radio-frequency (rF) scanning ion filter (quadrupole design) is employed to rapidly separate the analyte ions produced, which are sequential scanned onto a pulse-type detector. Due to the high scanning rate at which an rF filter operates, most of the ISBT constituent list can be identified and measured in less than approximately 1 minute. SIS data is typically displayed as a software-selected "hit list" of impurity names and associated ppm or ppb concentration units. No mass spectral interpretation activities or skilled operators are required for performing routine, highly automated SIS operations.

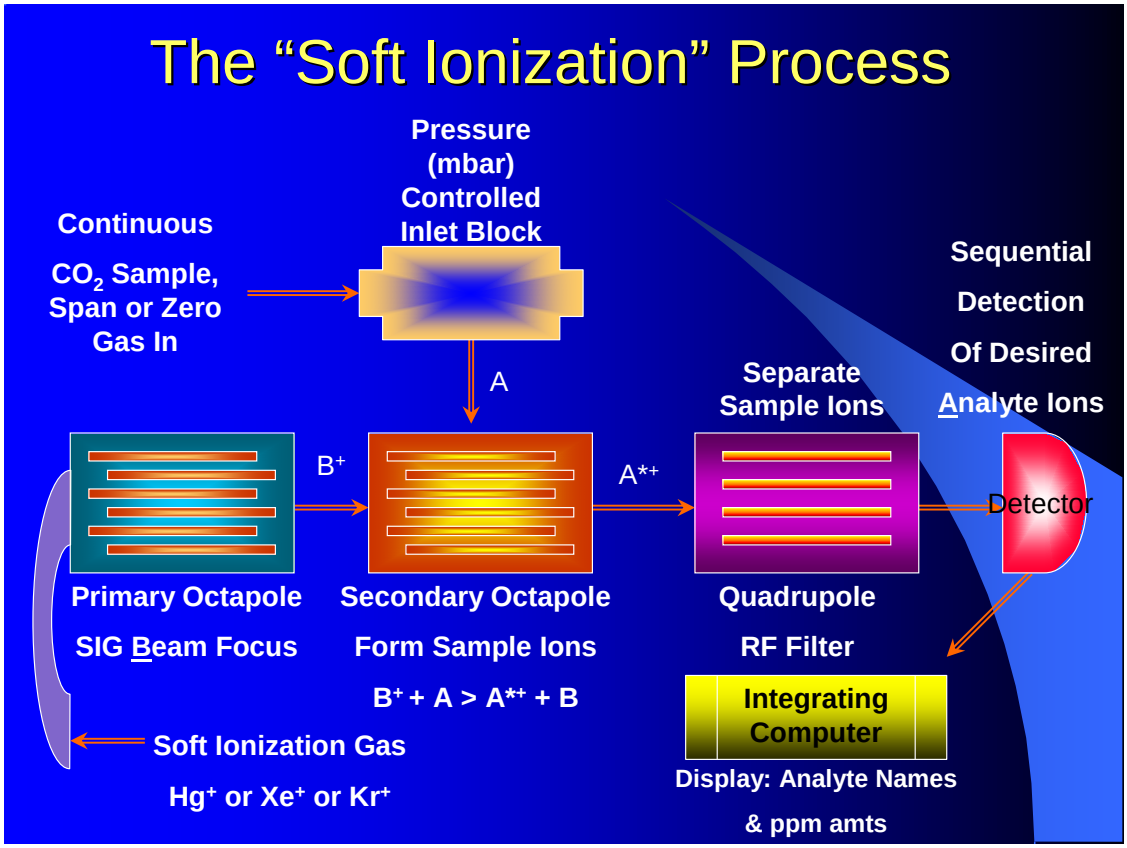
The SIS technology employed in this study was originally developed and octapole-ion-transfer system patented (Austria) by Dr.'s Villinger and Federer during the early 1980's. Because of its rapid analysis capabilities, versatility and low matrix gas interference properties, SIS has been

primarily used in internal combustion engine exhaust studies, air monitoring, as well as in catalyst-high speed kinetics research. In large part, due to the technological challenges that the beverage industry has experienced using traditional analyzer systems to meet well-defined ISBT 2001 CO_2 purity guidelines, SIS technology is being evaluated as a new tool for bev-gas analysis applications. Several SIS systems (35+) have recently been installed at some European CO_2 producer facilities and also at bottling operations performing PET bottle recycling.

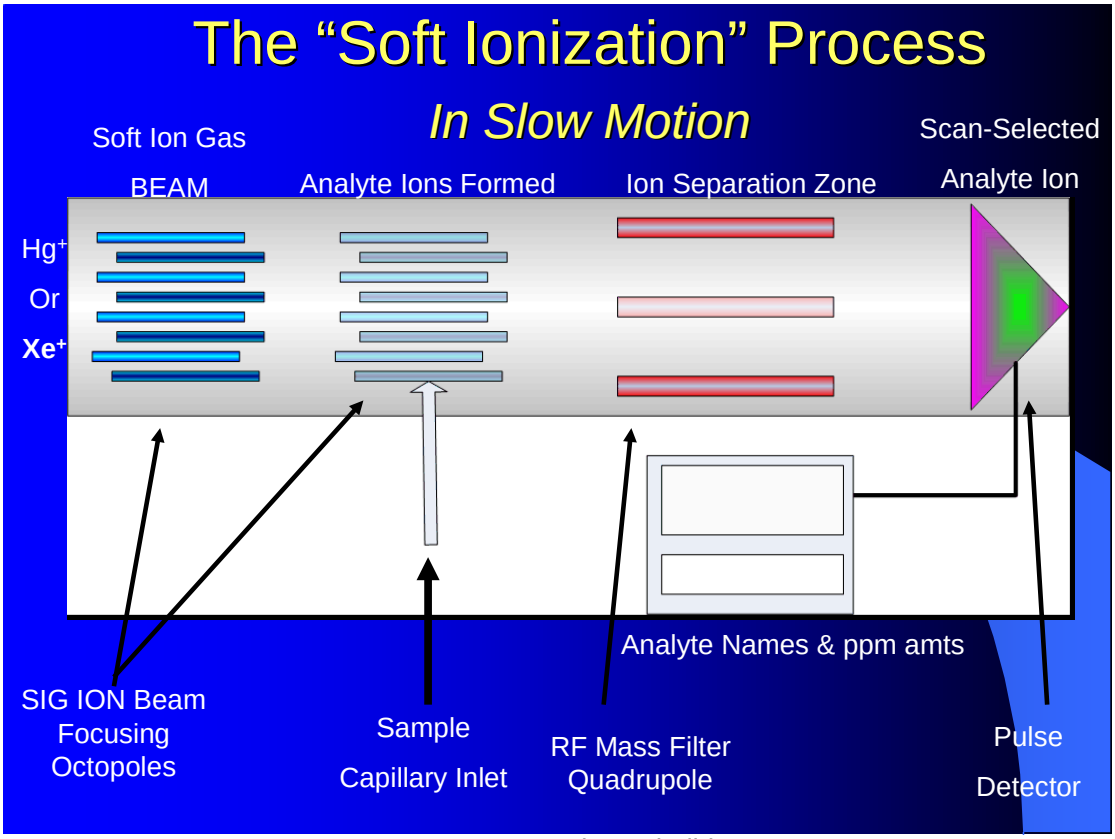
The specific purpose of this scientific assessment study was to determine the potential applicability, benefits and limitations of SIS technology relative to established ISBT CO_2 analytical methodologies. Most of the CO_2 impurities that are normally encountered in practice were studied and the results summarized in this paper.



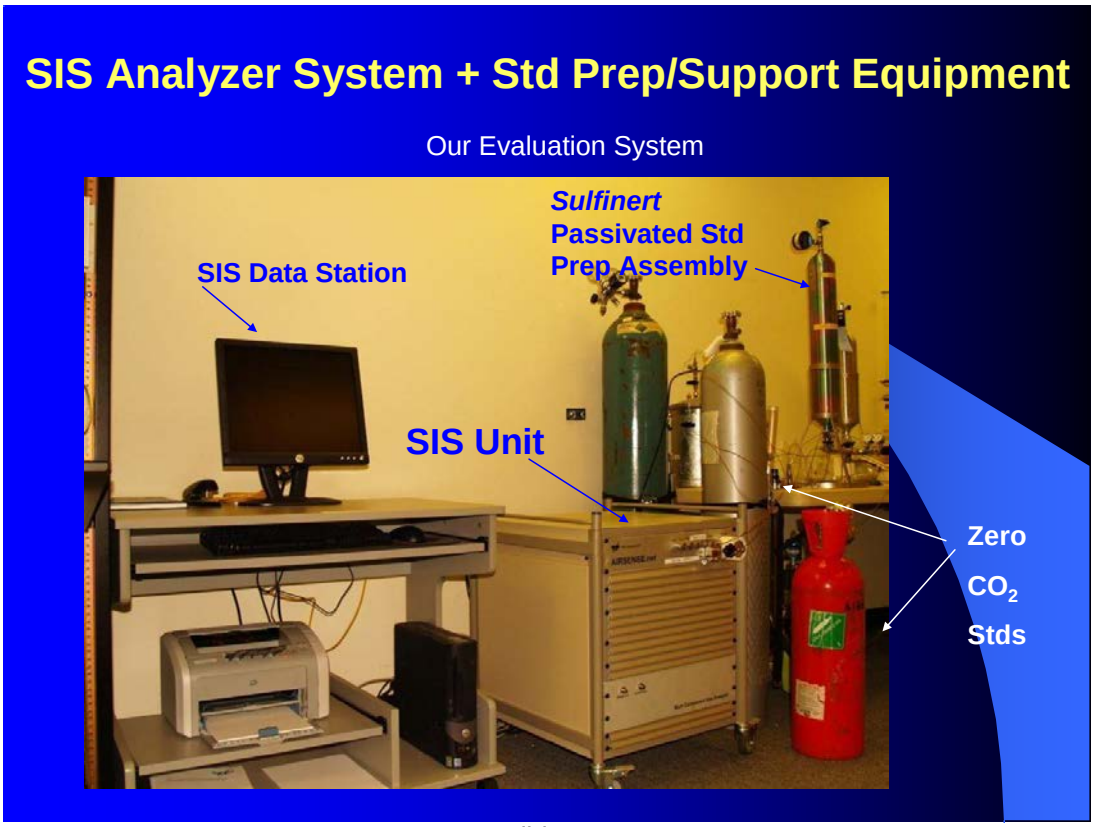
Slide 6.



Slide 7.



Animated Slide 8.



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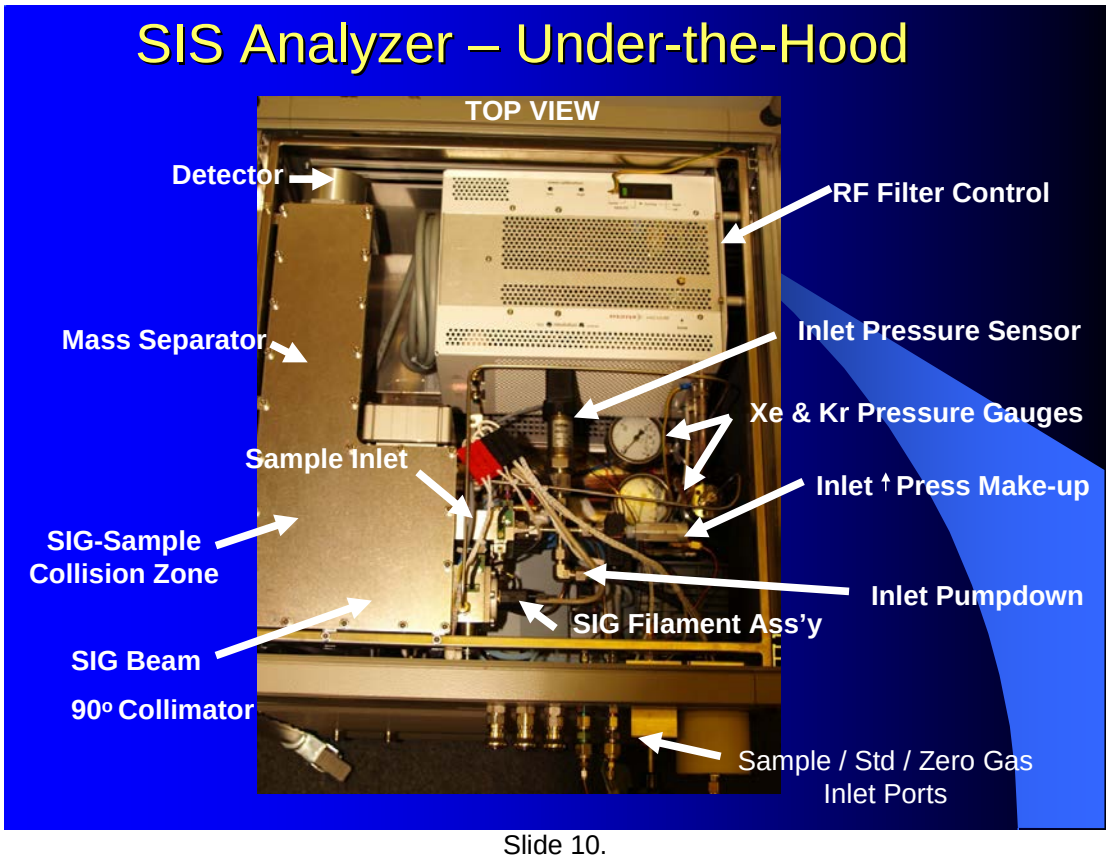
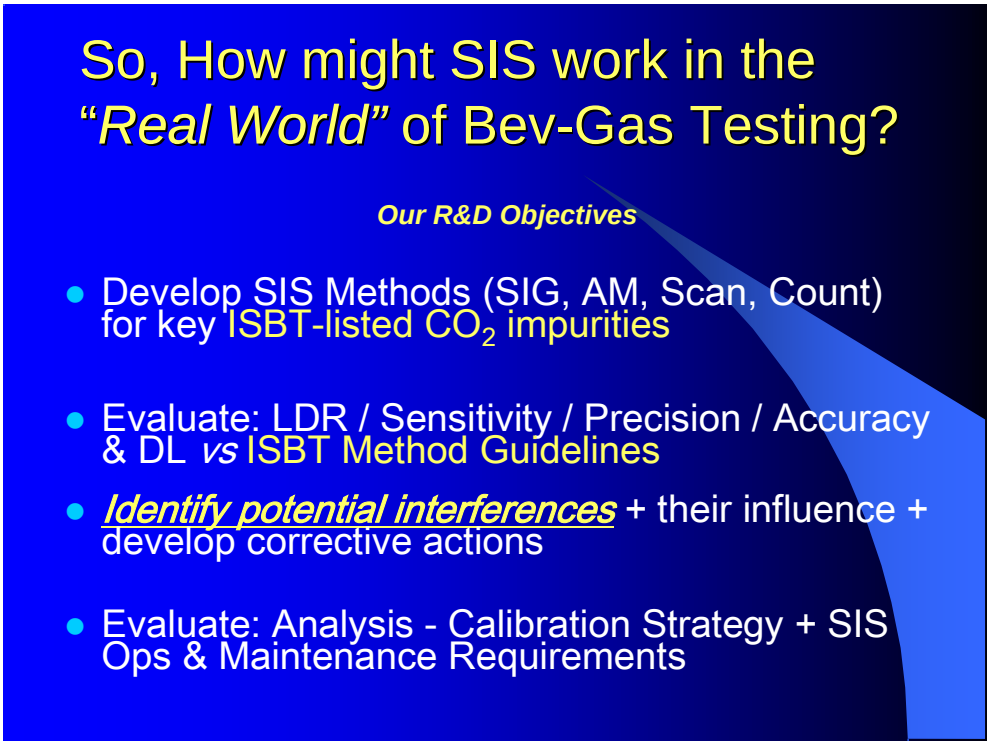


Slide 10.

Slide 11.

4. <u>The SIS Evaluation Study (Slide #12)</u>

Our primary objective was to critically evaluate how SIS technology might perform when complex "real world" conditions of bev-gas impurity challenges are experienced. Identifying potential Interactions between various analytes was a key part of this evaluation. Based upon our international laboratory testing experience, the additional non-ISBT listed analytes reported are common, individual impurities that are known to be present in many of the diverse CO_2 sources employed today.



Slide 12.

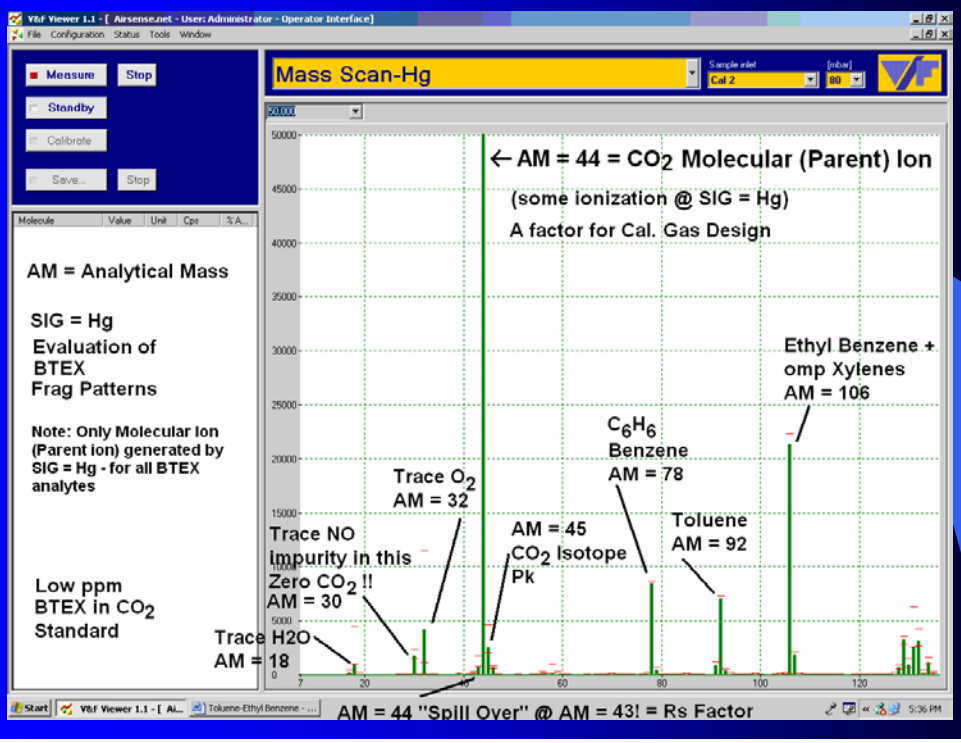
SIS Method Development for CO_2 Samples

As no published or non-proprietary SIS methods were available for measuring trace impurities in multi-sourced, bev-grade CO_2, the initial phase of this study was designed to develop appropriate methods for data evaluation against current ISBT methodology criteria. A commercially available SIS system, passivated standard preparation hardware and certified stock gas standards were used throughout this work. Confirmation of gas standard levels was performed using conventional ISBT methodologies. For each target impurity, method development involved establishing an extensive library of mass spectral data (fragmentation patterns) from approximately mass 10 to 120 amu using Hg^+, Xe^+, Xe^++ Kr^+ and Kr^+ as SIG sources. The energy levels associated with these SIG gases are: $Kr^+ > Xe^+ + Kr^+ > Xe^+ > Hg^+$. Examples of this mass spectral data and some method development challenges associated with analyte ion fragmentation patterns are illustrated in Slides 13 – 14. The conclusion of this method development work was that only Hg^+ and Xe^+ SIG sources are needed to meet current ISBT analytical method guidelines for most target list impurities.

This initial SIS method development phase provided information about the optimal SIG and analytical mass (AM) lines needed for both identification and quantitation of ISBT-listed CO_2 impurities. This 10-120 amu spectral library data also allowed for the evaluation of potential SIS measurement interferences that might be experienced by combinations of common impurities (e.g. presence of acetone on the accurate measurement of acetaldehyde [AA] – see Slide 14).

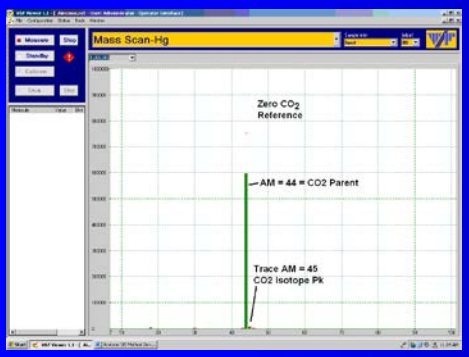
SIG & AM Selection Process

Sometimes its “easy”



Slide 13.

SIG & AM Selection Process – The "Frag Factor"



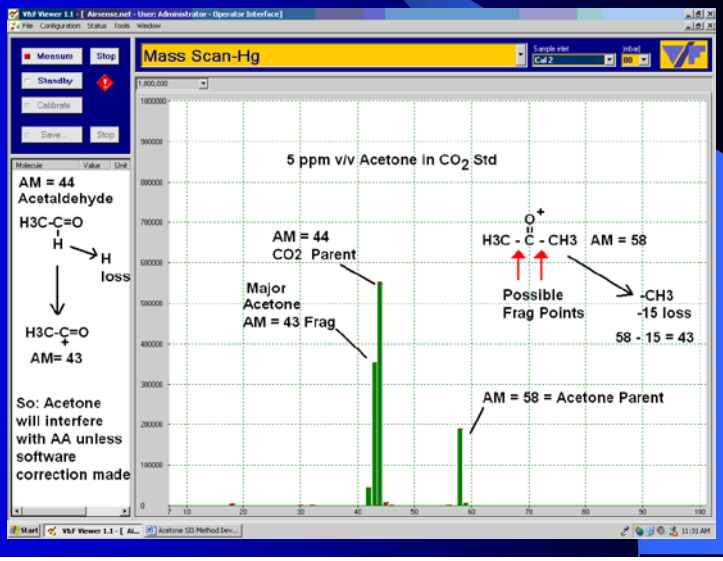
(Sometimes its not-so-easy)

Basic SIG & AM Studies

Needed to ID potential Interferences for design of

Appropriate Software & Calibration Routine

Corrective Measures



Slide 14.

SIS Data Evaluation – Results

Once an optimal SIG and AM was established for each target impurity, a series of target impurity standards was measured to evaluate the linear dynamic range (LDR), precision, accuracy and approximate detection limits (DL) that could be achieved for each individual target analyte. This data is tabulated / summarized and SIS experimental data presented for:

a) **Aromatic Hydrocarbon (AHC)** target analytes in CO_2 (Slides 15 – 17) demonstrate typical data obtained above and below ISBT 20 ppb v/v (as benzene) guideline limits. Comments are provided about any potential or demonstrated interference effects associated with these AHC impurities. No interferences were experienced-to-date.

SIS - Aromatic Hydrocarbon (AHC) Data

(ISBT CO_2 Limit = 20 ppb v/v as Benzene + report all other target AHC's)

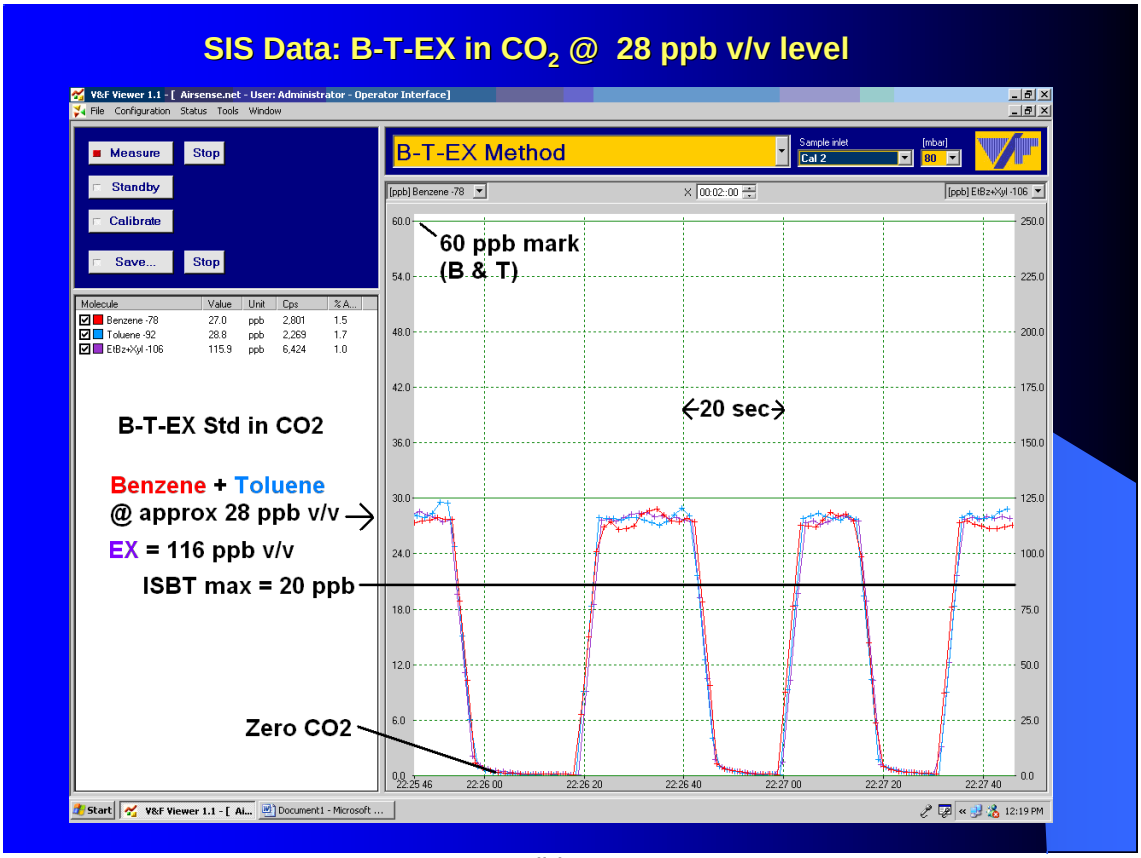
Our Results

AHC	SIG	AM	LDR ppb v/v	DL ppb v/v	Interference (s)
Benzene	Hg	78	LT 0 - 1000+	LT 1	None found-to-date
Toluene	Hg	92	LT 0 - 1000+	LT 1	None found-to-date
Ethyl Benzene + o,m,p Xylenes	Hg	106	1 -1000+	LT 5	None found-to-date Note: EBz + Xylenes measured as Total

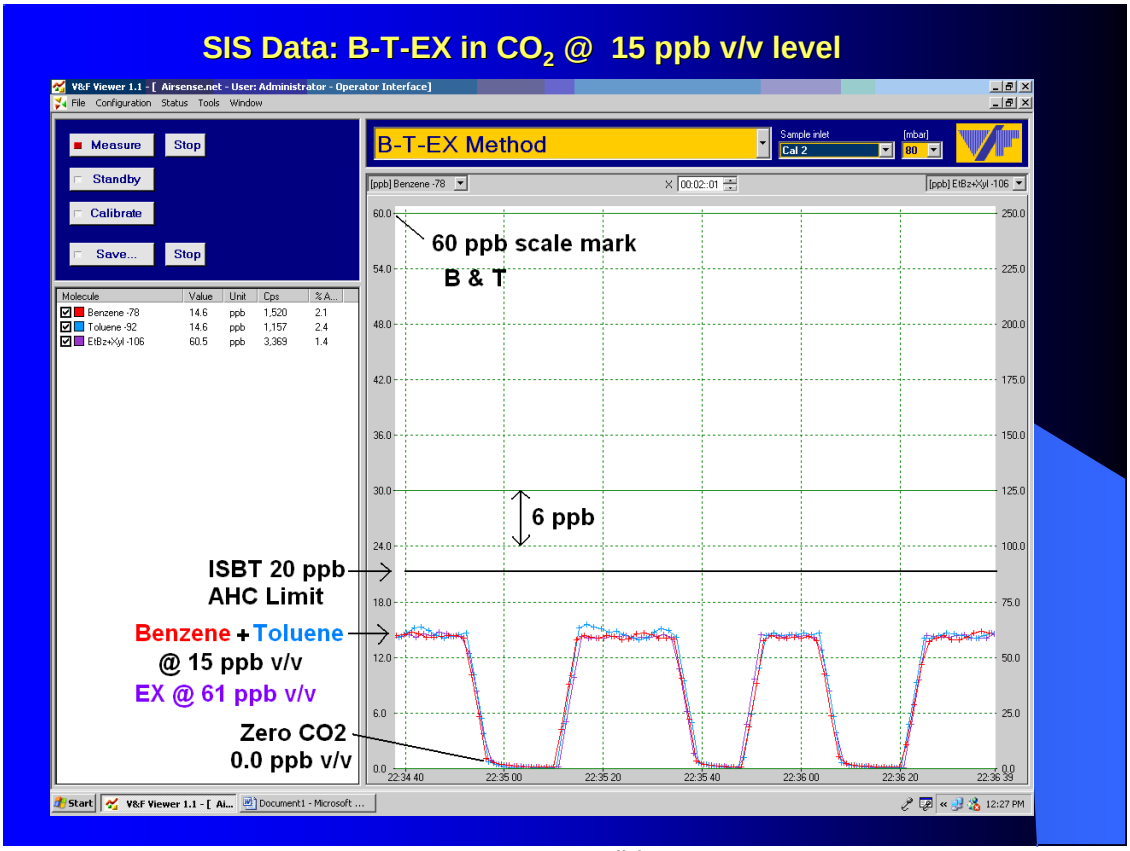
Speciation Information B-T-EX

Use of Benzene Cal Gas for Indirect Cal of TEX works v. well

Slide 15.



Slide 16.



Slide 17.

b) **Volatile Sulfur Compounds (VSC)** target analytes in CO_2 (Slides 18 - 21). Data is shown for the most common VSC impurities both above and below the current ISBT VSC* limit of 0.1 ppm v/v. Detection limit data is also shown which can be compared with other existing VSC analyzers. Note: SO_2 ISBT limits are 1 ppm v/v. Algorithms (as described in ISBT Method 14.0) are needed to convert speciated VSC data into a VSC* value.

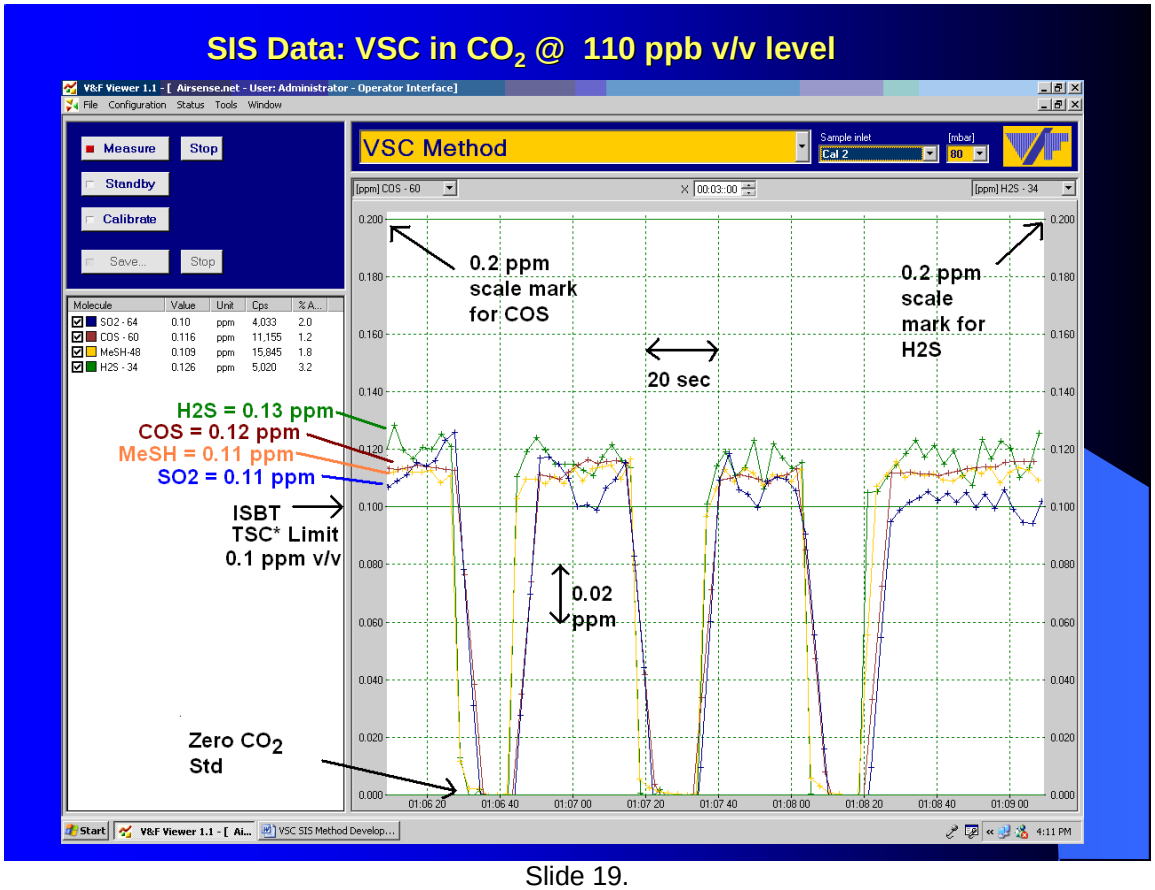
SIS - Volatile Sulfur Compounds (VSC) Data

ISBT Limit = 0.1 ppm v/v as TSC* = sum of VSC target impurities excluding SO_2, SO_2 = 1 ppm v/v max

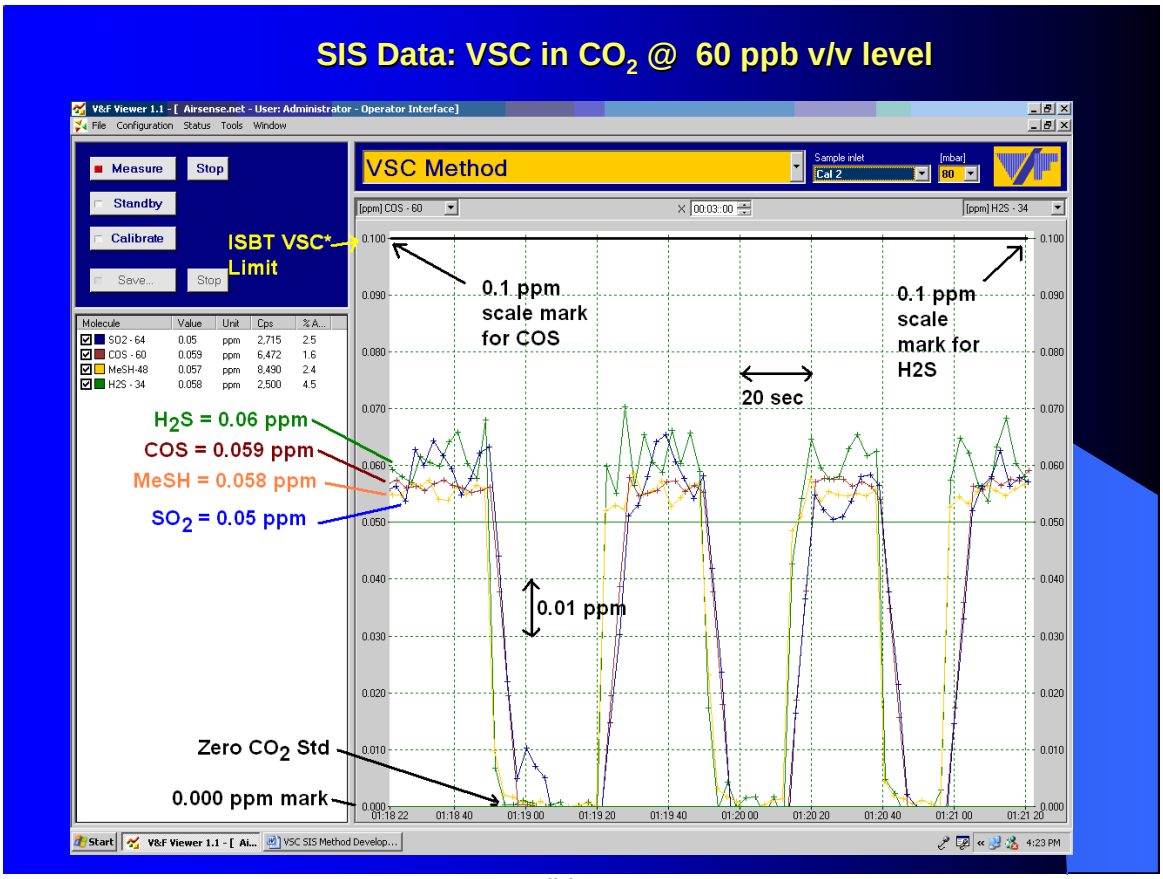
Useful Speciation information achieved

VSC	SIG	AM	LDR ppb v/v	DL ppb v/v	Interference (s)
H_2S	Hg	34	0 – 1000+	LT 5	PH_3 @ AM = 34 (Note: PH_3 = Rare)
COS	Xe	60	0 – 1000+	LT 5	None found-to-date
SO_2	Xe	64	0 – 1000+	5	None found-to-date
MeSH	Hg	48	0 – 1000+	LT 5	None found-to-date
CS_2	Hg	76	0 – 1000+	LT 5	None found-to-date
DMS + EtSH	Hg	62	0 – 1000+	LT 5	Vinyl Chloride (VCL) AM = 62, correct using AM = 64 isotope (Note: VCL = rare) Note: Measures Total DMS + EtSH

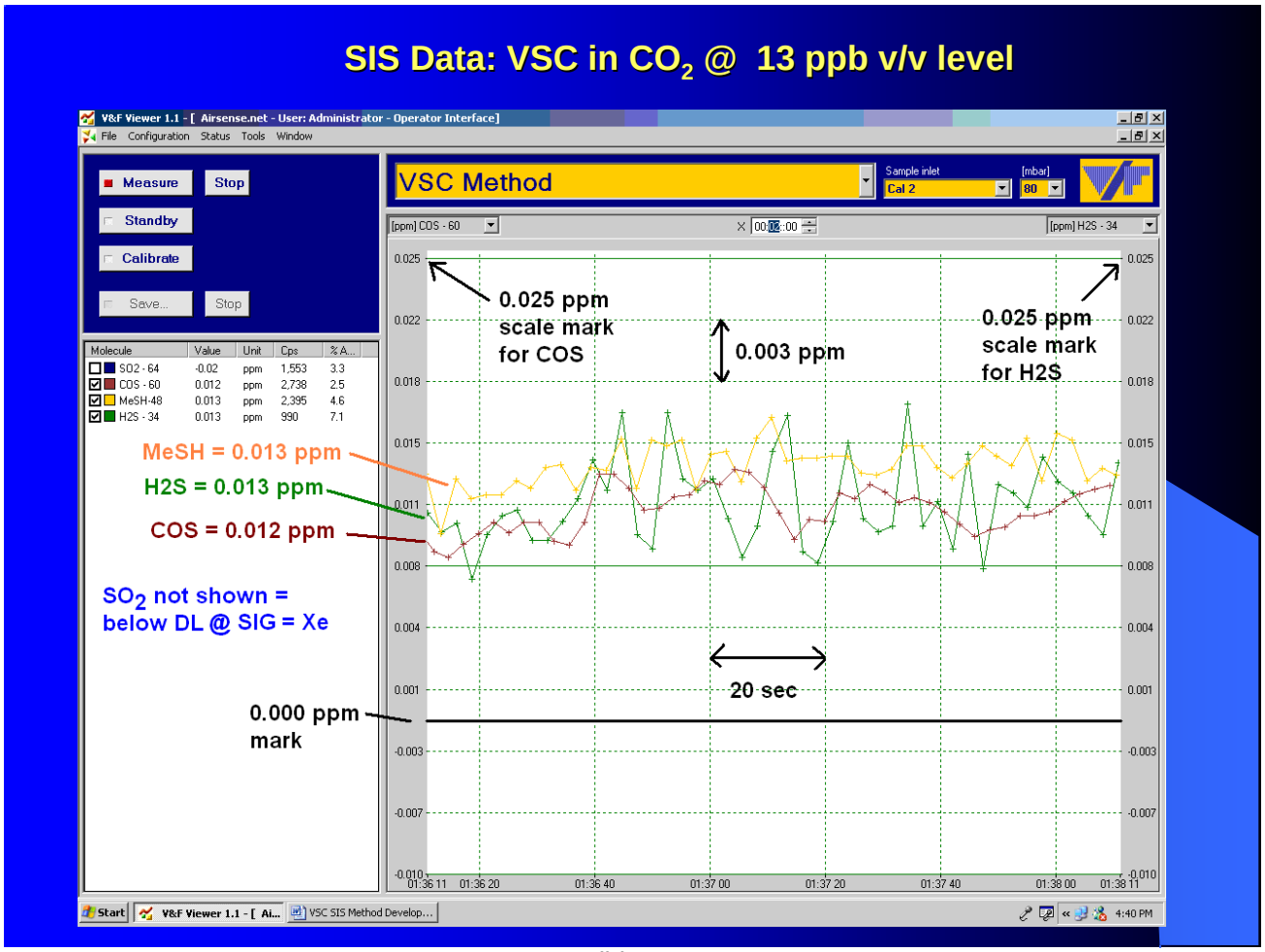
Slide 18.



Slide 19.



Slide 20.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Calibrate
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Stop
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80
[ppm] COS - 60
[ppm] H2S - 34
Molecule Value Unit Cps %A...
SO2 - 64 -0.02 ppm 1,553 3.3
COS - 60 0.012 ppm 2,738 2.5
MeSH-48 0.013 ppm 2,395 4.6
H2S - 34 0.013 ppm 990 7.1
MeSH = 0.013 ppm
H2S = 0.013 ppm
COS = 0.012 ppm
SO_2 not shown = below DL @ SIG = Xe
0.000 ppm mark
0.025 ppm scale mark for COS
0.003 ppm
0.025 ppm scale mark for H2S
20 sec
0.025
0.022
0.018
0.015
0.011
0.008
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VSC SIS Method Develop...
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Slide 21.

c) **<u>Volatile Oxygenated Compounds (VOX)</u>** target analytes in CO_2 (Slides 22-28)

In this VOX series, a specific example of a "worst-case" interference effect is demonstrated along with typical precision, repeatability and sensitivity that was achieved above and below the ISBT 0.2 ppm v/v AA limit. This example specifically shows the extreme positive interference effect that acetone potentially has on obtaining an accurate AA measurement. SIS Software-correction, however, was found to be capable of adequately eliminating this high level of spectral interference. For THC + TNMHC parameters, algorithms are required to convert speciated VOX data into equivalent THA-FID data as ppm v/v Methane response.

SIS - Volatile Oxygenated Compounds (VOX) Data

ISBT Limit = 0.2 ppm v/v as Acetaldehyde (AA) + Speciation information needed for THC/TNMHC calcs etc.

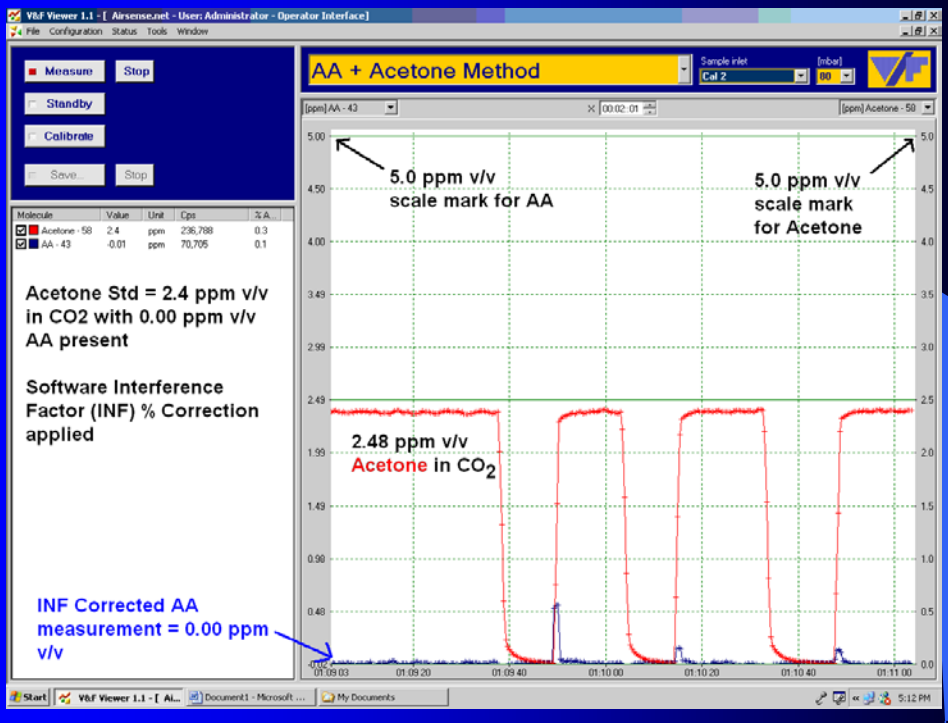
VOX	SIG	AM FR = Frag	LDR ppm v/v	DL ppm v/v	Interference (s)
AA	Hg	43^{fr}	0 - 5+	LT 0.1	Acetone = Major INT, i-PA = INT, i-Butanes = INT, n-Butane= minor INT
DME	Hg	46	0 - 5+	LT 0.1	No effect on AA, NO_2 = INT
MeOH	Xe	31^{fr}	0 - 5+	LT 0.1	No effect on AA, EtOH / nPA = INT
EtOH	Hg	45^{fr}	0 – 5+	LT 0.1	No effect on AA, DME = minor INT
N-Butanol	Hg	56^{fr}	0 - 5+	LT 0.1	No effect on AA
Acetone	Hg	58	0 - 5+	LT 0.1	Major AA effect, Butanes = INT
EtOAc	Hg	70^{fr}	0 - 5+	LT 0.1	No effect on AA

Other VOX impurities studied to date: n / i -Propanols, i -Butanols, Ethylene Oxide

Slide 22.

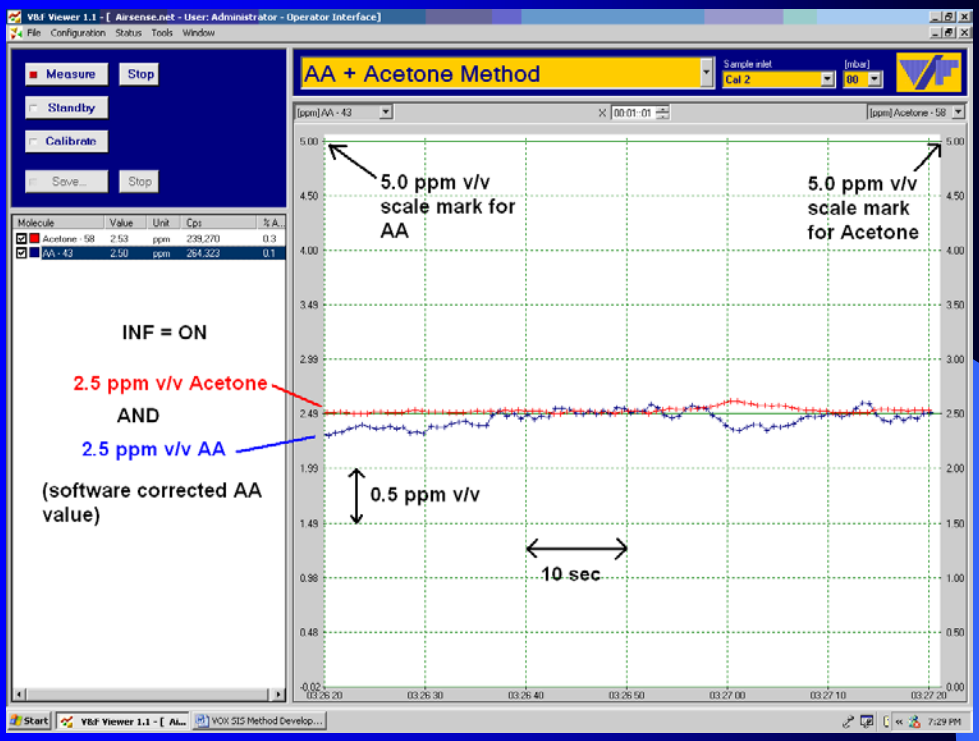
SIS Data: VOX in CO_2 @ 2.5 ppm v/v Acetone / 0.00 AA – INF Corrected

Key to Success is having accurate Interference Correction Data

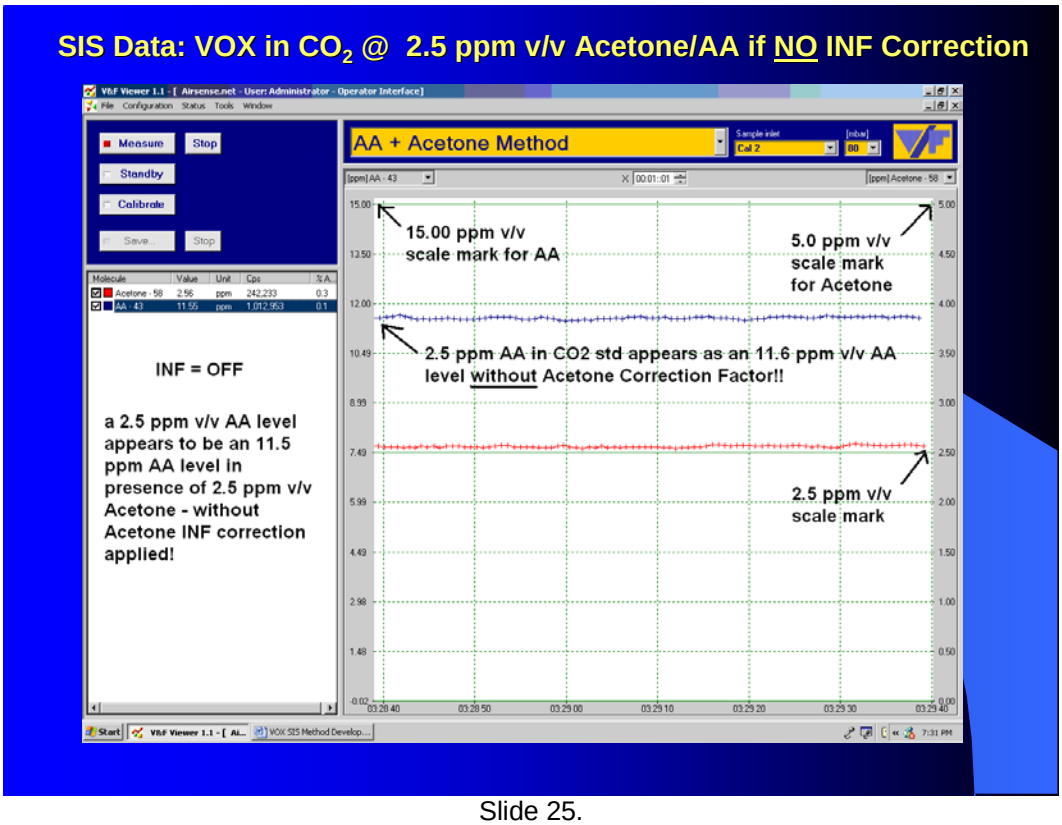


Slide 23.

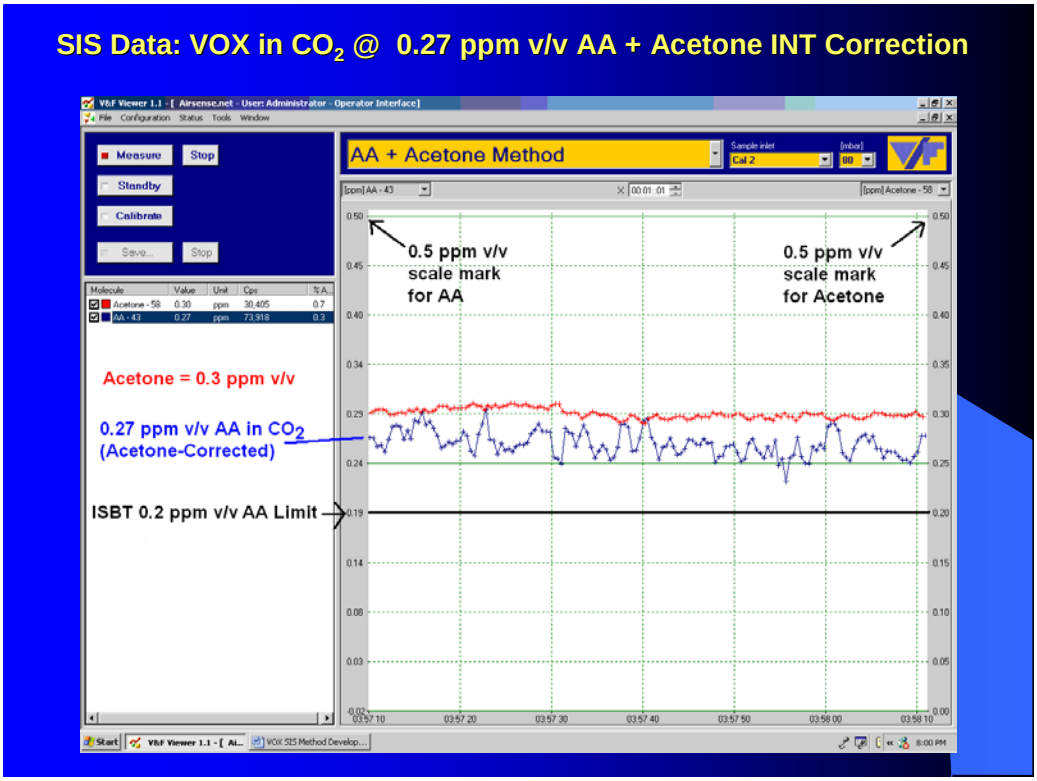
SIS Data: VOX in CO_2 @ 2.5 ppm v/v Acetone + AA – INF Corrected



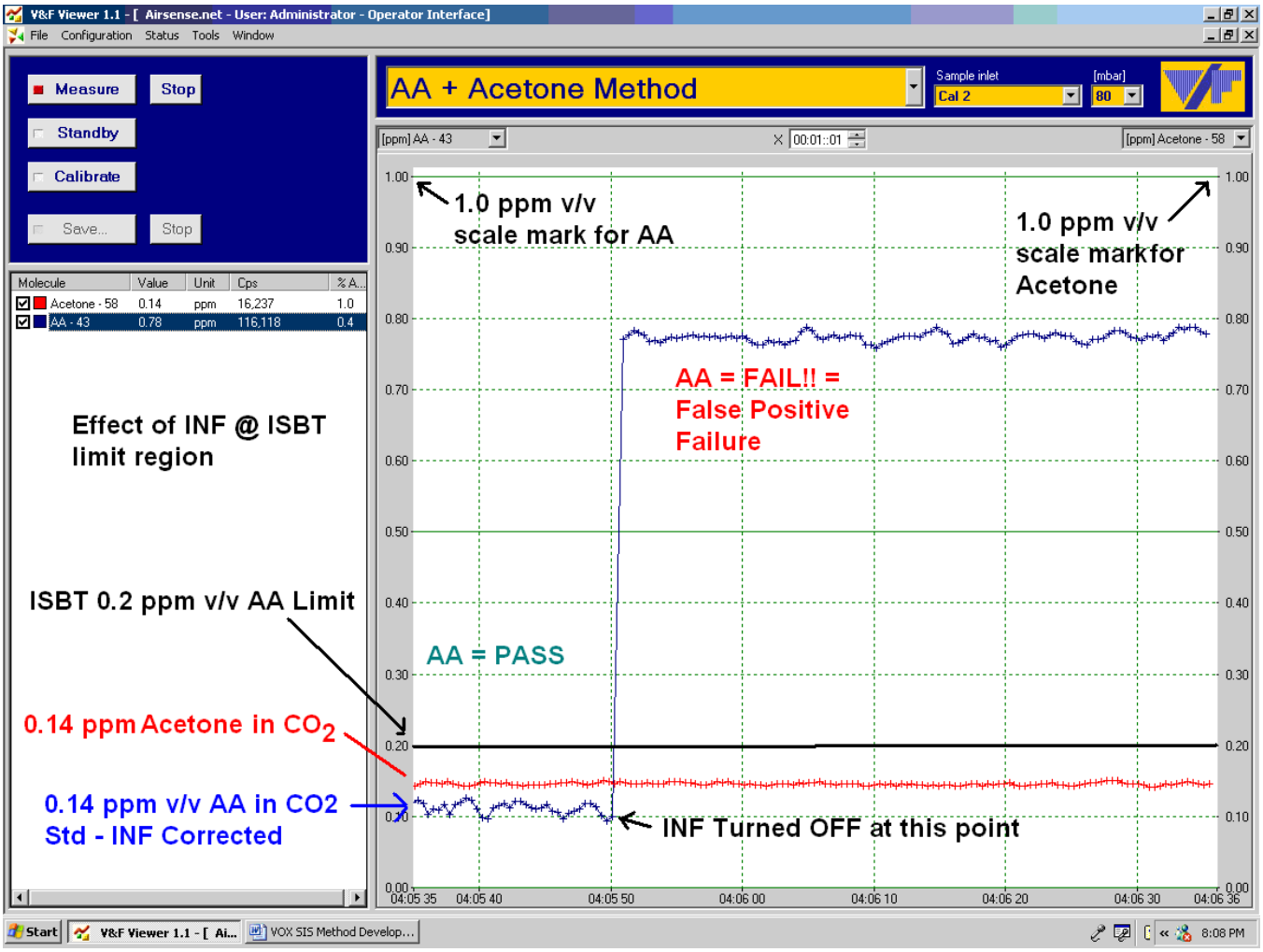
Slide 24.



Slide 25.



Slide 26.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80
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00:01::01
[ppm] Acetone - 58
Molecule Value Unit Cps %A...
Acetone - 58 0.14 ppm 16,237 1.0
AA - 43 0.78 ppm 116,118 0.4
1.0 ppm v/v scale mark for AA
1.0 ppm v/v scale mark for Acetone
AA = FAIL!! = False Positive Failure
Effect of INF @ ISBT limit region
ISBT 0.2 ppm v/v AA Limit
AA = PASS
0.14 ppm Acetone in CO2
0.14 ppm v/v AA in CO2 Std - INF Corrected
INF Turned OFF at this point
1.00
0.90
0.80
0.70
0.60
0.50
0.40
0.30
0.20
0.10
0.00
04:05 35
04:05 40
04:05 50
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04:06 10
04:06 20
04:06 30
04:06 36
Start
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VOX SIS Method Develop...
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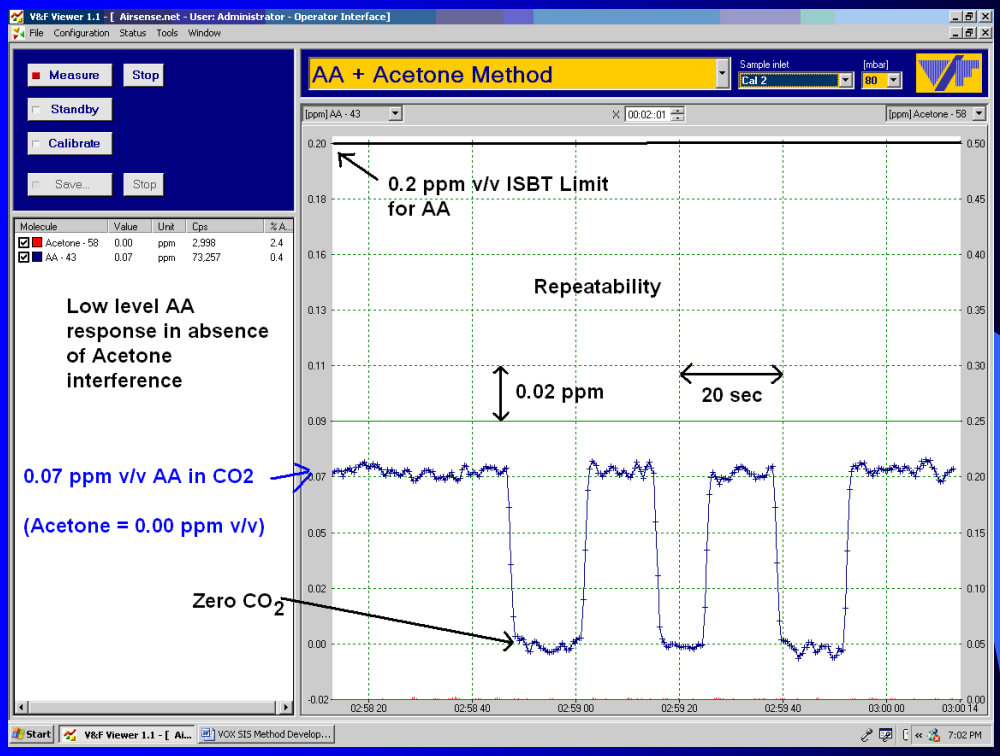
Slide 27.

SIS Data: VOX in CO_2 @ 0.07 ppm v/v AA – if No Acetone present



Slide 28.

d) **Volatile Hydrocarbons (VHC)** target analytes in CO_2 (Slides 29 – 31)

In this VHC series, specific examples of dual, more common, less extreme forms of spectral interferences (from ethane and n-butane), software-correction performance as well as expected precision and repeatability data are demonstrated for the measurement of propane (@AM=29). SIS performance for methane to n-butane target analytes at levels just above the 20 ppm v/v (as methane) TNMHC limits are demonstrated. For calculating TNMHC data from primarily VHC + VOX data, an algorithm must be employed using speciated VHC + VOX data. Methane is easily speciated from non-methane organic impurities.

SIS - Volatile Hydrocarbons (VHC) Data

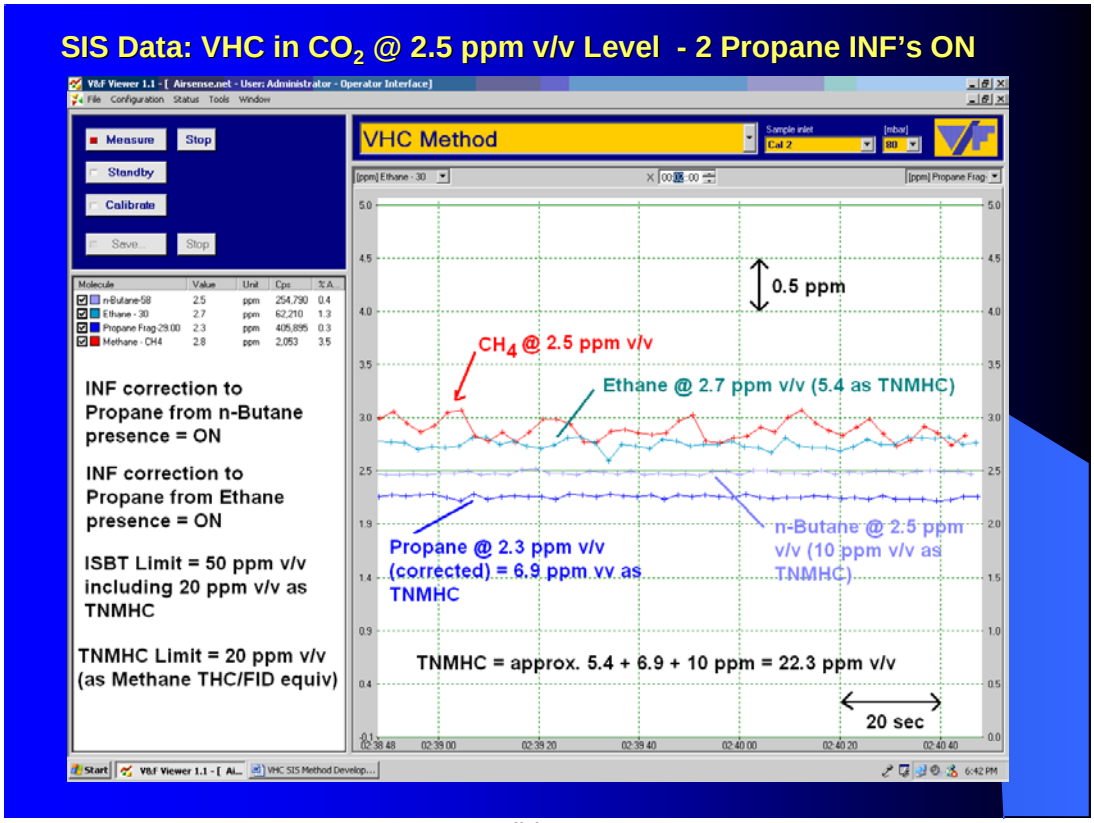
ISBT Limit = 50 ppm v/v THC as Methane including 20 ppm v/v max as Total Non-Methane HC's

VHC	SIG	AM fr= Frag	LDR ppm v/v	DL ppm v/v	Interference (s)
CH_4	Xe	16	0 – 100+	LT 0.5	None found-to-date
Ethane	Xe	30	0 – 20+	LT 0.1	NO = INT (AM = 30)
Propane	Xe	29^{fr}	0 – 10+	LT 0.1	Ethane = Minor INT, ETO = INT @ Frag = 29, n-Butane = Minor INT
N-Butane	Hg	58	0 - 5+	LT 0.1	Minor INT for AA,
N-Pentane[1]	Hg	42^{fr}	0 - 5+	LT 0.1	ID @ AM = 72 Parent

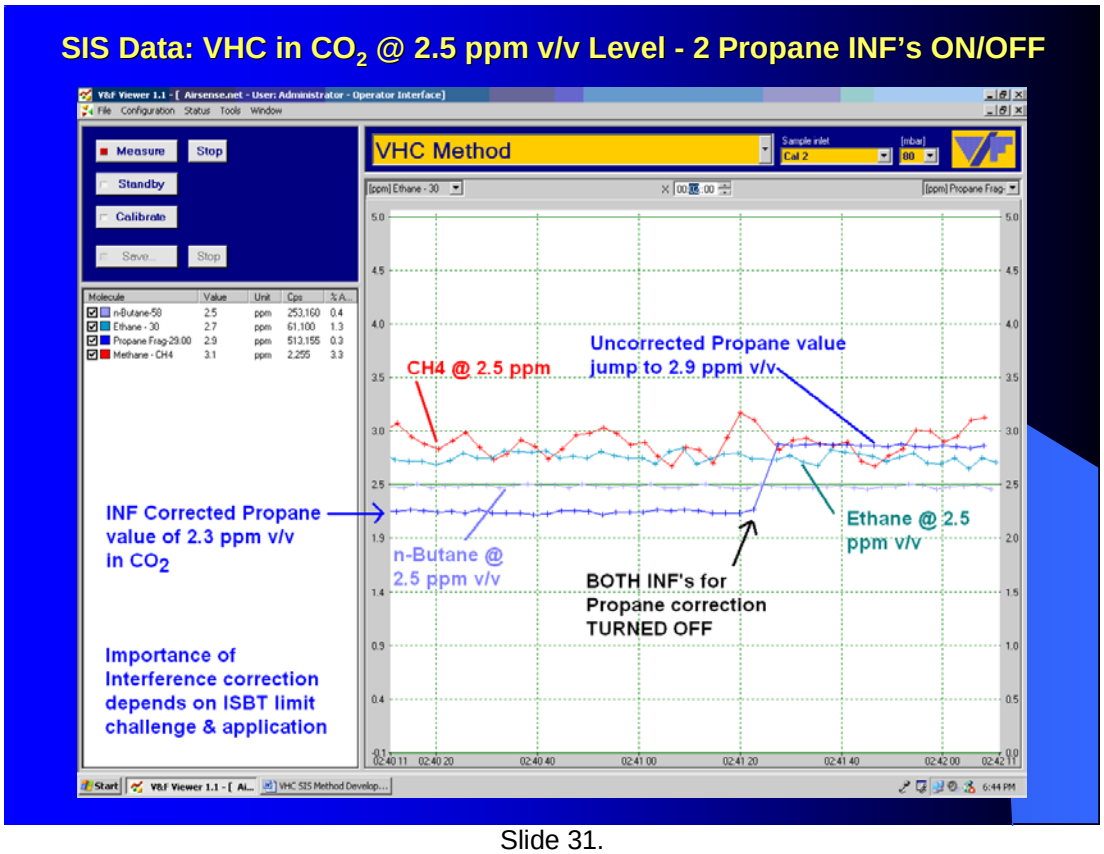
Note 1: n-Pentane C-C-C-C-C $\longrightarrow$ $C\text{-}C\text{-}C^{+}$ (AM=42)

Other VHC's not studied-to-date

Slide 29.



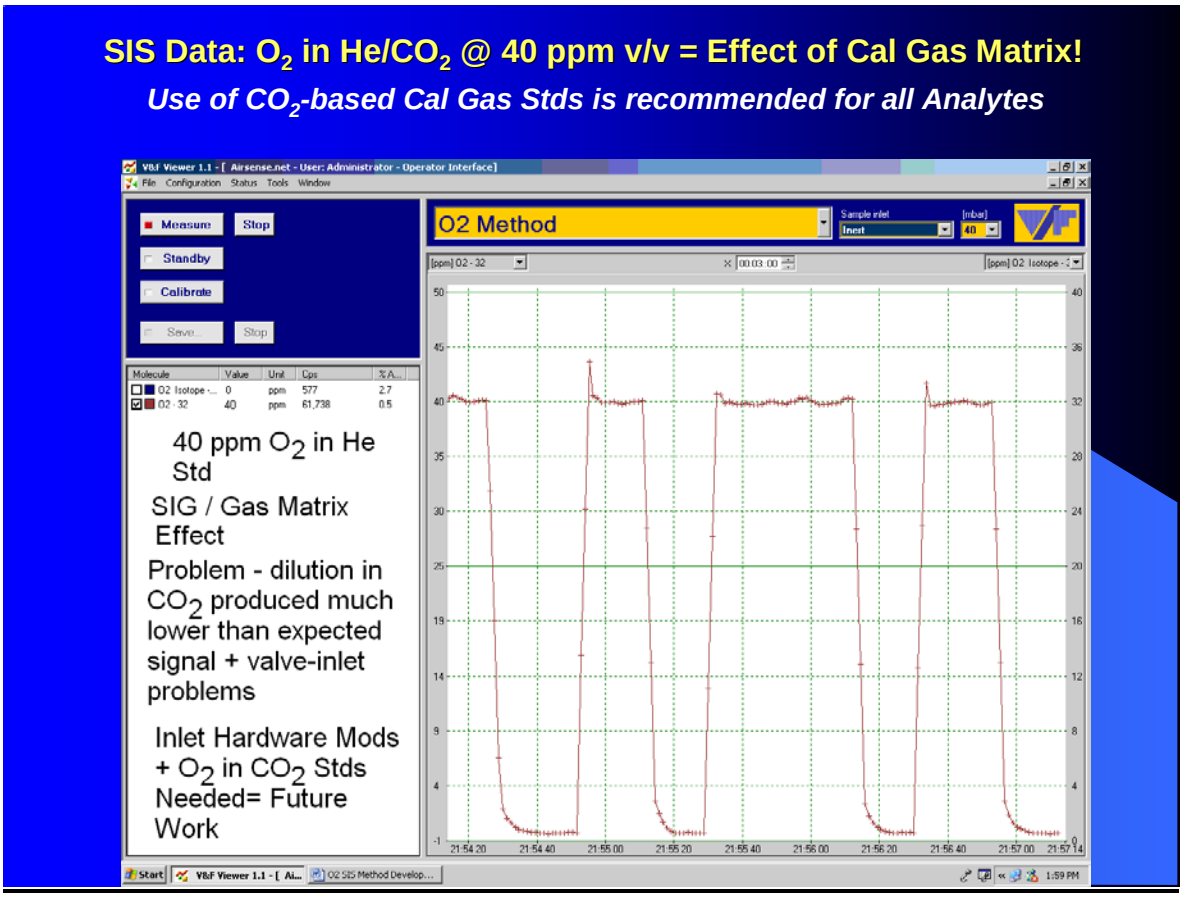
Slide 30.



Slide 31.

e) **Oxygen (O_2) in CO_2** (Slide 32)

This method development series indicated the importance of using calibration gas standards that are comprised of the same matrix gas as the sample (CO_2), especially when higher energy SIG sources such as Xe^+ are employed. An initial 40 ppm v/v O_2 gas standard in helium (He) produced an excellent SIS signal as shown. However, when this 40 ppm v/v O_2 in He standard was diluted 1:1 in zero-grade + O_2 scrubbed CO_2, the net signal counts dropped dramatically and the expected 20 ppm v/v O_2 result was not achieved. Instead a value below 5 ppm (relative to the He std) was observed. This result emphasized the point that CO_2, while only ionized with a relatively low efficiency in the SIG beam, is not an "inert" bystander in the process and that the SIG beam intensity felt by an analyte in CO_2 will be less than that in a totally SIG non-ionizable gas such as helium. In addition, the inlet system of the SIS model employed was not equipped with the make-up hardware modification needed to prevent transient, trace O_2 incursion during inlet switching. Because of this factor, this aspect of our O_2 method study was suspended, with plans to resume this work at a later date.

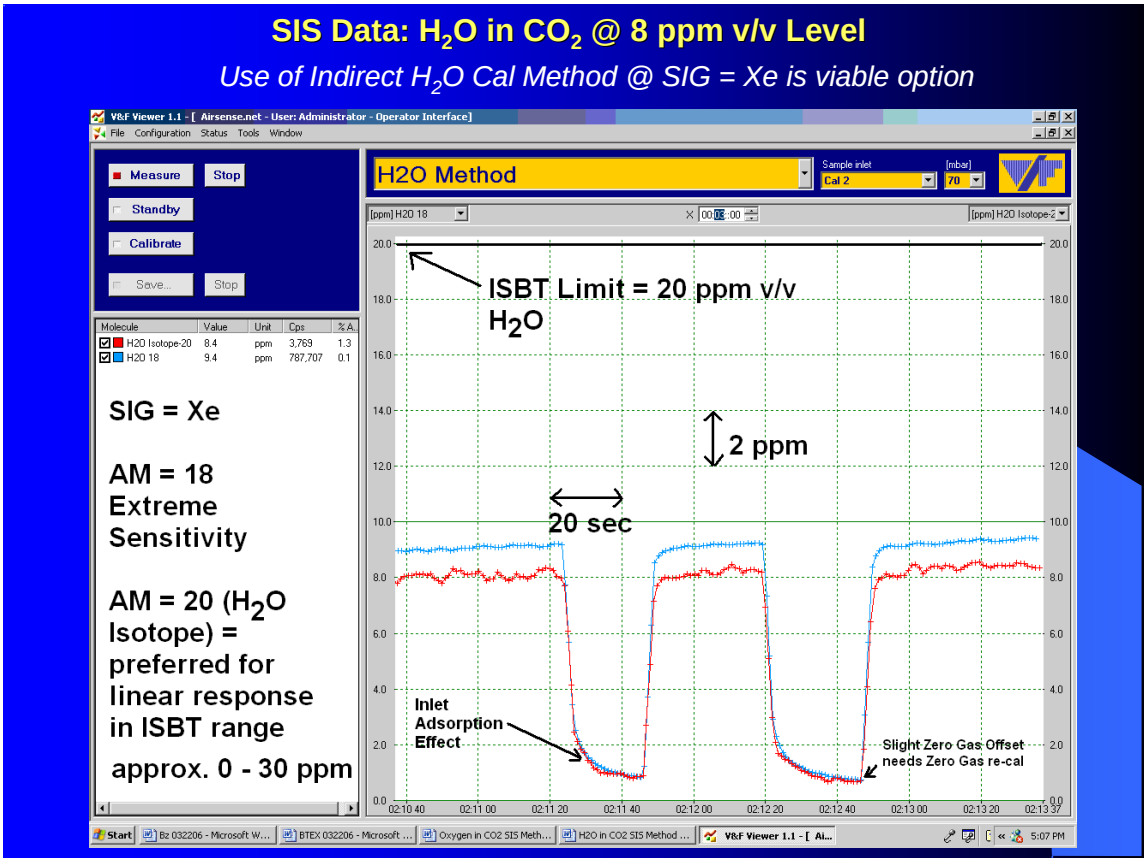


Slide 32

f) **Water Vapor (H_2O) in CO_2** (Slide 33)

In this H_2O series, two AM lines (AM = 18 + AM = 20 (an isotope line) were used for evaluation. This dual AM evaluation was performed because of the extremely high sensitivity that H_2O exhibits with a SIG = Xe^+. Use of lower energy Hg+ as a SIG does not produce a significant signal with H_2O in CO_2. For the typical range of H_2O measurements needed for bev-gas applications (e.g. 20 ppm v/v ISBT limit), the less sensitive isotope AM=20 line is recommended. Good linearity, precision equivalent to ISBT Method 3.0 and a low ppm DL were obtained with AM = 20. No interferences were encountered. Use of a passivated SIS inlet system is also suggested from the SIS signal response curve data for H_2O.

Note: the SIS detector tends to exhibit some non-linear behavior (due to the high count-rate signals) experienced when AM = 18 is employed and if moisture is present around the ISBT limit. This data clearly indicates that SIS can be used to detect ppb v/v levels of H_2O if ever needed (using the AM=18 line).



Slide 33.

g) <u>Other ISBT-Listed Impurities in CO_2</u> (Slide 34)

A data table is presented that summarizes the SIS evaluation for measurement of:

Nitric Oxide (NO), Nitrogen Dioxide (NO_2), Ammonia (NH_3), Hydrogen Cyanide (HCN) and Vinyl Chloride (VCl) in CO_2.

For each of these analytes, SIS exhibited an LDR, precision and detection limits that were equivalent or superior to the relevant ISBT Analytical Methods, as described in the 2001 guideline. Minimal interferences were found, and in most cases, interferences were correctable by proper selection of SIG, AM, program measurement sequencing or software-correction. For HCN, even lower detection limits were achievable with a SIG = Xe^+ + Kr^+.

Our results for CO indicated that SIS does not have sufficient sensitivity for measuring CO down to or below the 10 ppm v/v ISBT limit in a CO_2 matrix.

SIS – Data for Other ISBT Listed CO_2 Impurities

Impurity	SIG	AM FR = Frag IS = Isotope	LDR ppm v/v	DL ppm v/v	Interference (s)
NO	Hg	30	0 - 5+	LT 0.05	None found-to-date
NO_2	Hg	46	0 - 5+	LT 0.05	DME = INT AM = 46
NH_3	Hg	17	0 - 5+	LT 0.05	None found-to-date
HCN	Xe	27	0 - 5+	LT 0.1	VCl = INT HCN Lower DL @ SIG = Xe + Kr
VCl	Hg	64^{is}	0 - 5+	LT 0.05	Use VCl Isotope AM = 64

Note 1: PH_3 study not completed SIG = Hg, AM = 34

Note 2: CO work was not successful for this ISBT application

Slide 34.

5. Summary: SIS applicability to ISBT CO_2 List (Slide 35)

This table is a "Yes/No + Comment" type summary of our experimental evaluation study. In most cases, SIS technology was found to produce data that met or exceeded the measurement criteria described in the 2001-published ISBT Analytical Methods Manual. From this standpoint, for the analytes and conditions described, this technology is a viable option for bev-gas quality assurance applications.

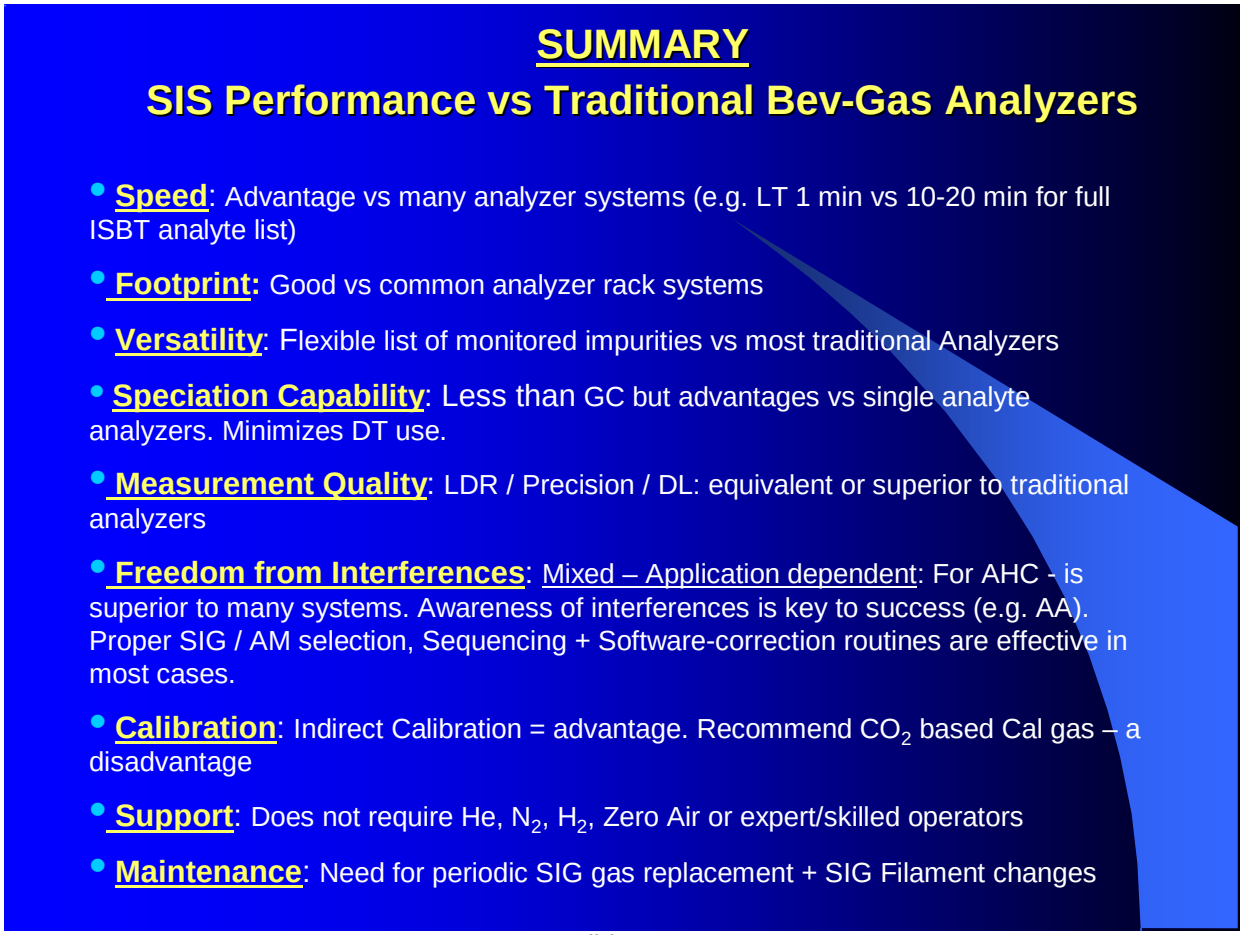
SIS Applicability to ISBT CO_2 List

ISBT LISTED IMPURITY	SIS Measurable?	Comments
CO_2 Purity	NO (but positive CO_2 ID)	Can't do N_2, H_2, Ar
Moisture (H_2O)	**YES**	Can go sub-ppm range
Oxygen (O_2)	**Maybe** (hardware mods)	Needs O_2 in CO_2 Stds
Carbon Monoxide (CO)	NO	Poor Sensitivity issues
Ammonia (NH_3)	**YES**	Needs passivated inlet
NO / NO_2	**YES** (+ speciation)	Some minor INF's (not common)
Non-Volatile Residue (NVR)	NO	Can't do particulates / non volatiles
Non-Volatile Organic Residue (NVOR)	NO (but C6+ = Yes)	Can't do V. Hi MW oils/grease
Phosphine (PH_3)	**YES** (+ H_2S) PH_3= RARE	Can't distinguish from H_2S
Total Volatile Hydrocarbons (THC) & TNMHC	THC = **YES** TNMHC = **YES**	Get CH_4 + need Algorithm Sum of all TNMHC's vs THA-FID signal
Acetaldehyde (AA)	**YES** (+ VOX speciation)	Needs corrections for INF's
Aromatic Hydrocarbons (AHC)	**YES** (B-T-EX)	V good precision / low DL's no known INF's
Total Sulfur Content (TSC*) less SO_2	**YES** (+ speciation)	Need Algorithm Sum of VSC's
Sulfur Dioxide (SO_2)	**YES**	Easily Speciated from COS
HCN	**YES**	V. Low DL no common INF
Vinyl Chloride	**YES**	V Low DL

Slide 35.

6) **Summary SIS Performance vs Traditional Bev-Gas Analyzers (Slide 36)**

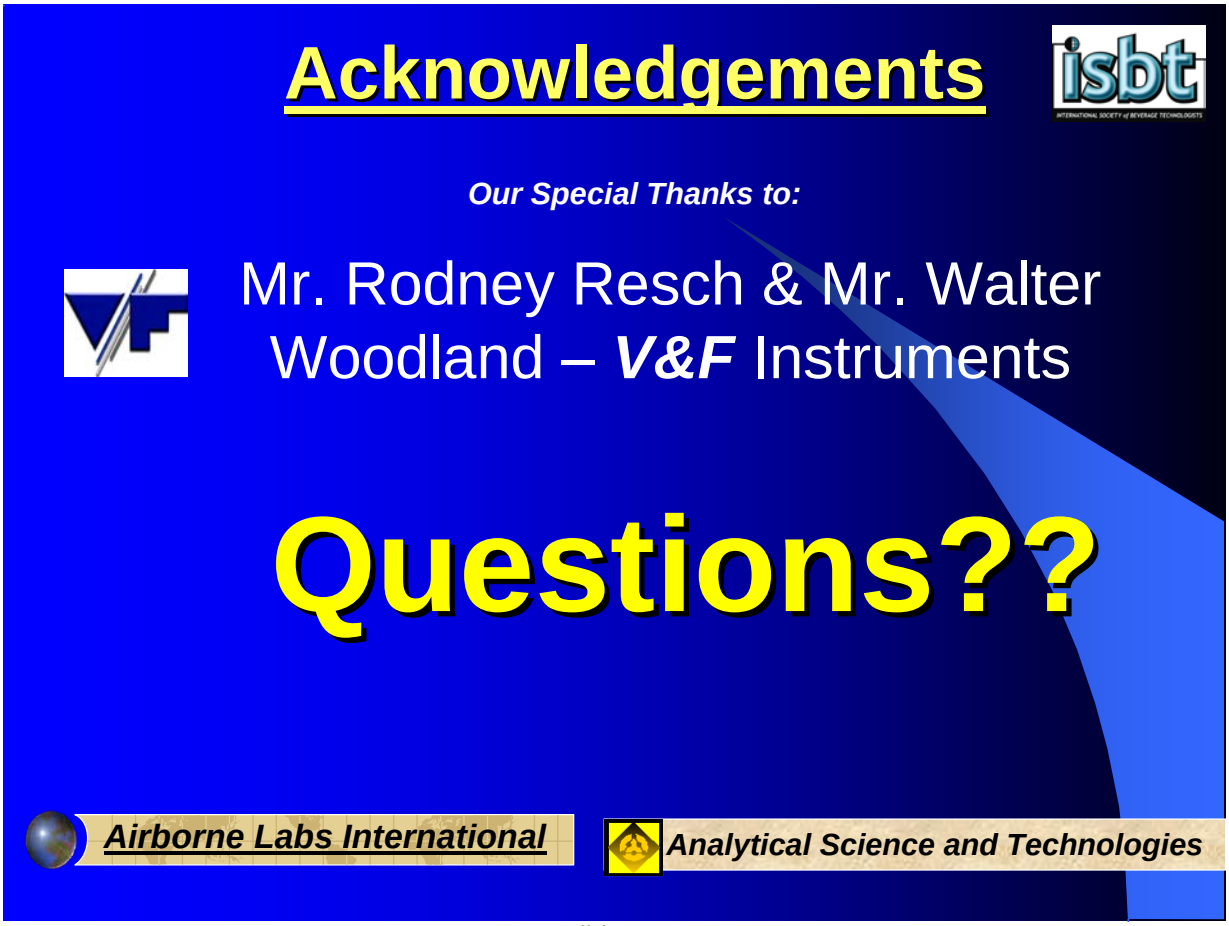
Both the advantages and perceived limitations associated with SIS technology, as experienced in this work are summarized relative to analyzers that are currently common and familiar to the beverage industry. In many cases, these results indicate potential operational advantages can be obtained with SIS for many CO_2 quality control applications. The key to successful application of SIS (as demonstrated by acetone / AA data and VHC data) relates to having a thorough appreciation for the potential interferences that may be encountered in a specific application. In most cases (e.g. a CO_2 producer plant with a well-defined feedgas stream) this interference assessment will be relatively straightforward. For bottling plants that may receive CO_2 from a variety of supplier sources, this assessment will be more complex. Based on the interference work performed to date, it is expected however, that available strategies (e.g. involving measurement sequencing, proper use of software-correction routines and optimal calibration gas standards) can adequately minimize or eliminate many potential interference issues.



Slide 36.

7) **Acknowledgements** (Slide 37)

Thanks and appreciation is given to the manufacturers of the SIS equipment that was on-loan for this evaluation.



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