

**On-site Detection of Siloxanes and other unwanted VOCs
in Landfill/Sewage/Biogas
using
Gas Chromatograph-Ion Mobility Spectrometer (GC-IMS)**

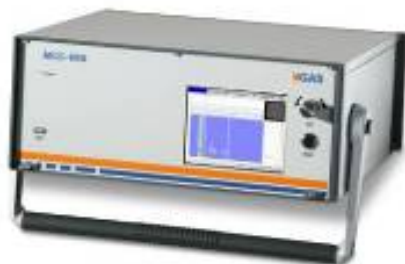
by

G.A.S. Gesellschaft für analytische Sensorsysteme mbH

G.A.S. Instruments with different sampling Techniques



GC-IMS and A-IMS



Measurement of VOCs in gases

Applications

Process Control
Environmental monitoring
Gas quality control

BreathSpec®



Detection of VOCs in exhaled human breath

Applications

Detection of marker Compounds directly in human breath

FlavourSpec®

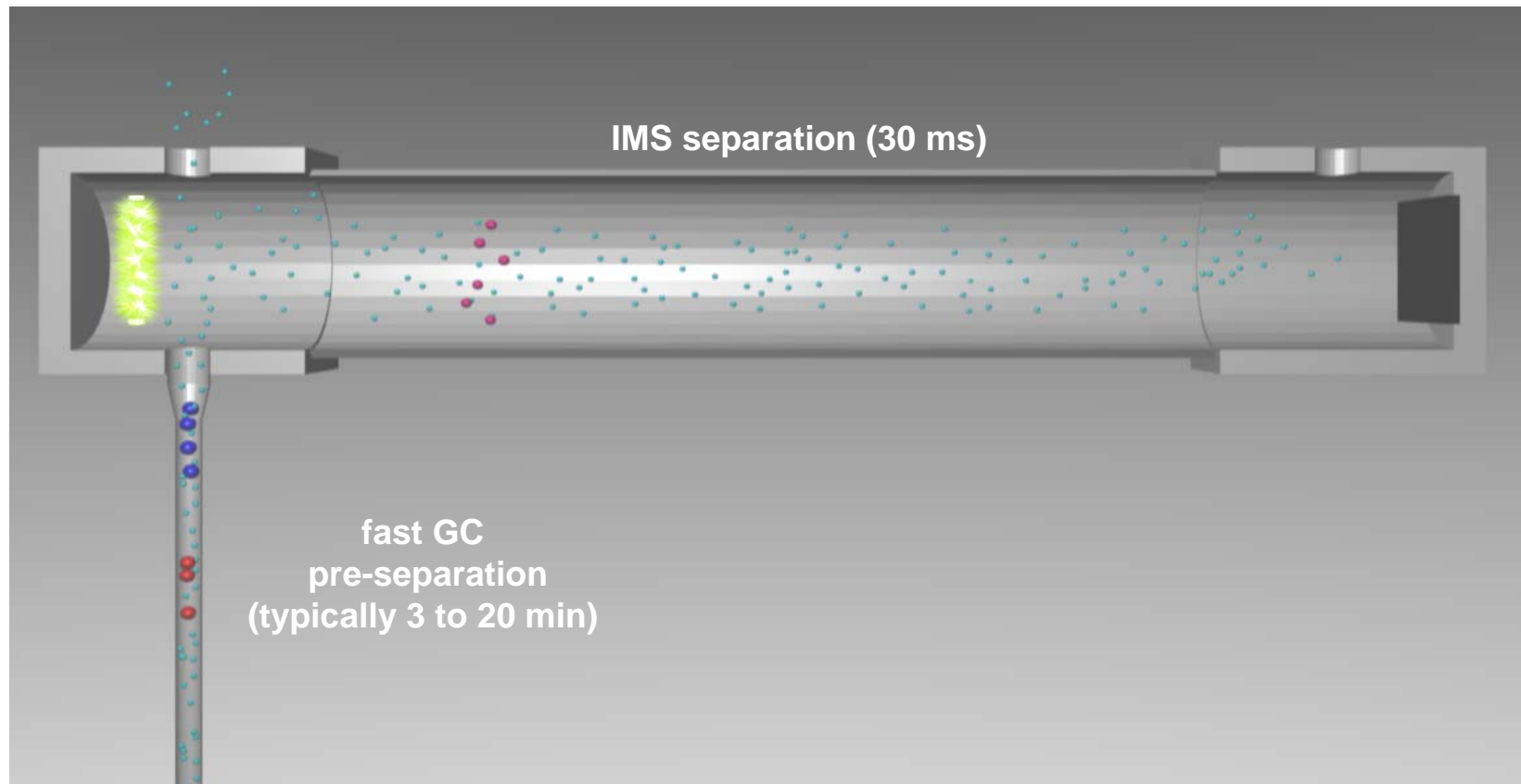


Detection of traces of VOCs in the headspace of solids & liquids in food/beverages

Applications

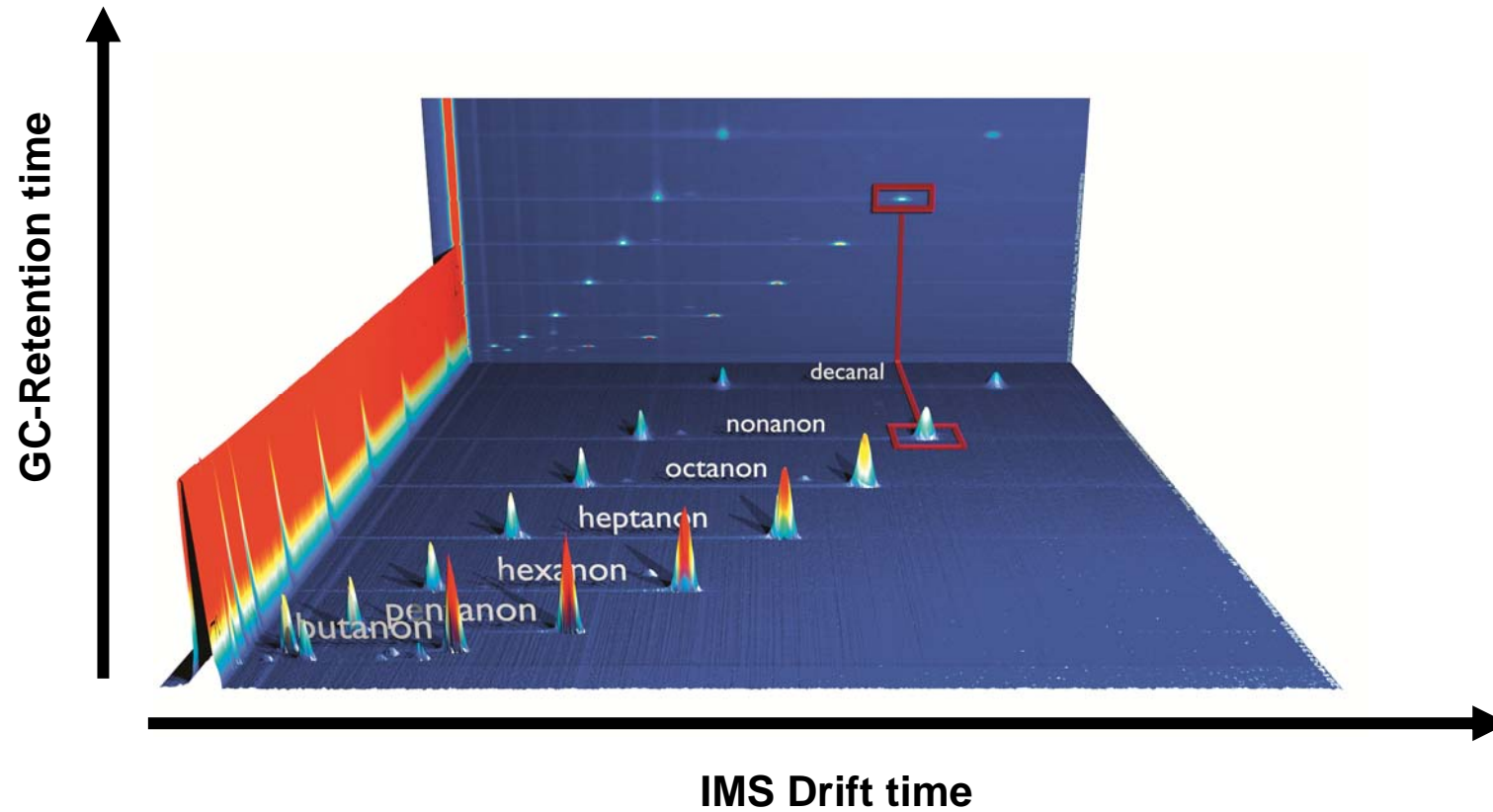
Off-smell monitoring
Raw material and product quality control

GC-IMS Technology: 2-dimensional Separation



Selectivity of GC plus Sensitivity of IMS to achieve excellent analytical Performance

GC-IMS Technology: 2-dimensional Separation



Compound Identification according to NIST-based Library, Validation by test gases

Start System

Measuremen

Local Remote

Setup

Device Di

Plan

Fan regulation

Start 30 °C

Settings

Save

Device overview

Out

Sample Out

Sample Gas

Test Gas

IMS

GC Capillary Column

6-Port Valve + Sample Loop

Drift Gas

N₂

Carrier Gas

EPC1

EPC2

V1

V2

P

T1

T2

T3

Close

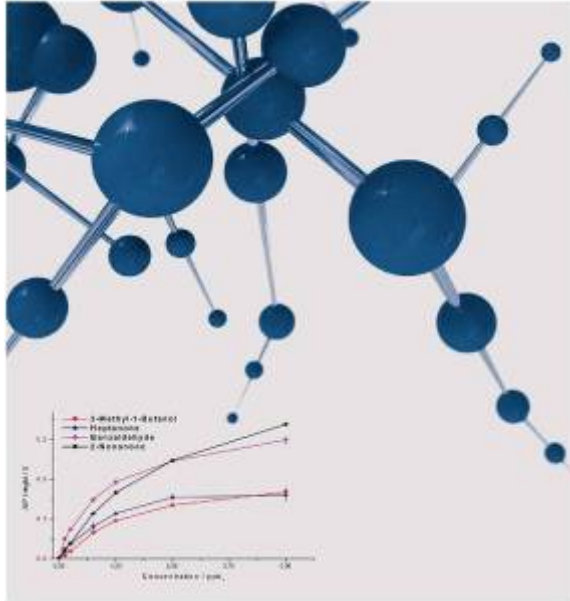
AS
rtmund.de
analytische
me mbH

-IMS-SILOX
F3-00043
2.09
Feb 16 2015
095 / V. 1.20
01.10
2.6.39.4
:7D:2F:AC:A0
168.100.97

smb 192.168.100.97
dhcp

Log 15:07
Feb. 19. 2015

Only power and nitrogen (purge gas) are needed!



IMS is sensitive for a large number of chemical compounds, like:

- ketones
- aldehydes
- alcohols
- ether
- ester
- amines
- aromatic compounds
- organic phosphor compounds
- organic sulfur compounds
- organic nitro compounds
- halogenated compounds
-

➤ **detection limit typically low ppb_v range**

Biogas is produced by the anaerobic digestion or fermentation of biodegradable materials such as biomass, sewage, waste ...

Methan	50 – 60 %
CO ₂	30 – 45 %
N ₂	< 5 %
O ₂	< 2 %
H ₂ S	2 % (200 – 2000 ppm)

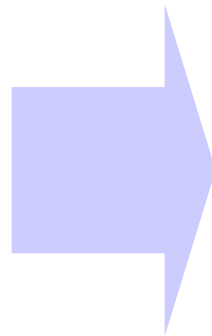
Application: Detection of Siloxanes in Biogas



Different Applications regarding Siloxane Monitoring need to be addressed

Due to the presence of.....

Washing Agents
Cosmetica
Skin Care Products
Silikone Oils
Waterproofing Materials
Shoe Cream
.....



... induced presence of siloxanes

Tetramethylsilane (TMS)
Trimethylsilanol (MOH)
Hexamethyldisiloxane (L2)
Hexamethylcyclotrisiloxane (D3)
Octamethyltrisiloxane (L3)
Octamethylcyclotetrasiloxane (D4)
Decamethyltetrasiloxane (L4)
Decamethylcyclopentasiloxane (D5)

Generated (typical) Siloxanes

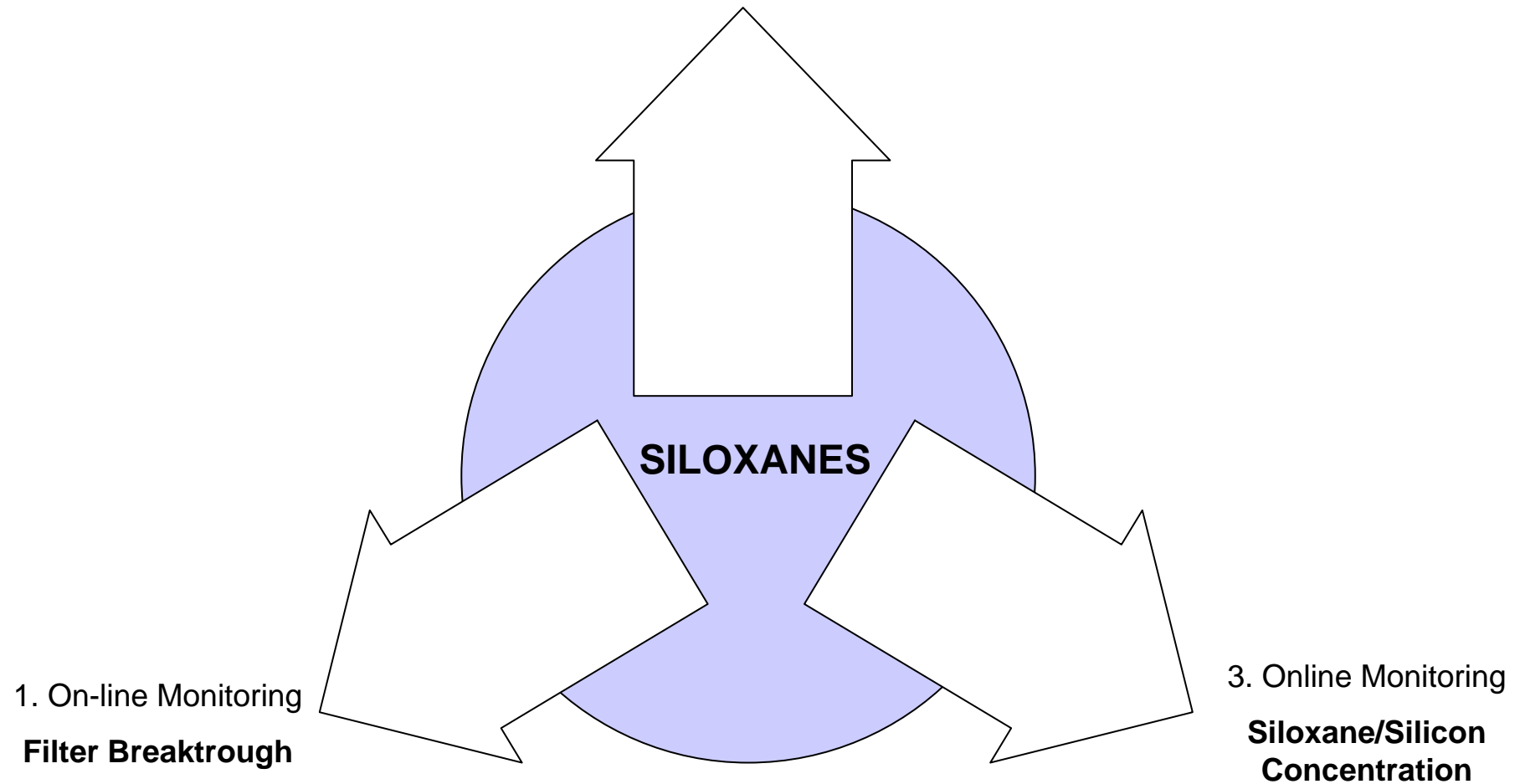
....different parts of the engine can be damaged resulting in:

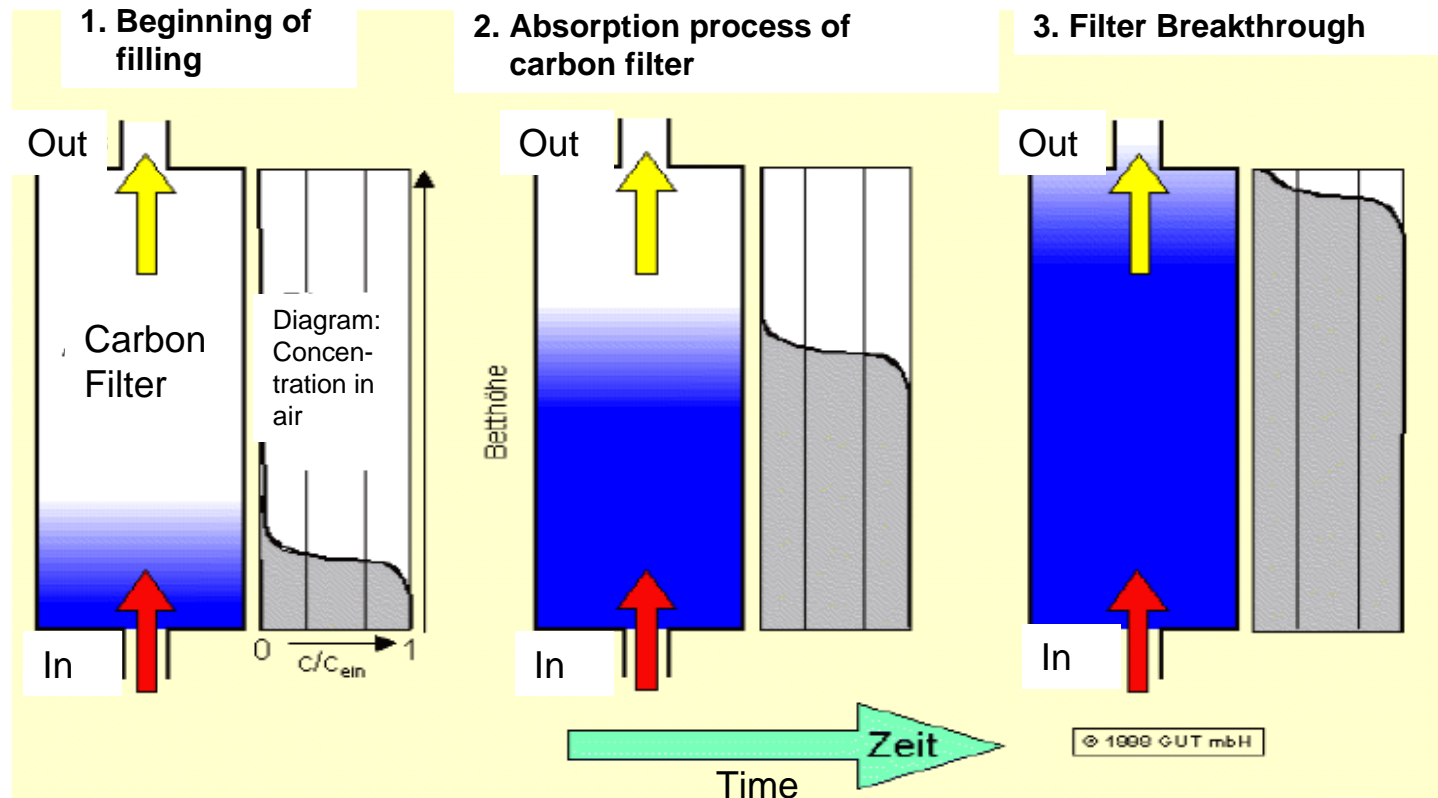
1. Cost intensive break downs of the engine and ...



2. and significant secondary costs: Repair and Down Times!

2. Portable/flexible Instrumentation to test for
Siloxanes/Silicon Concentration





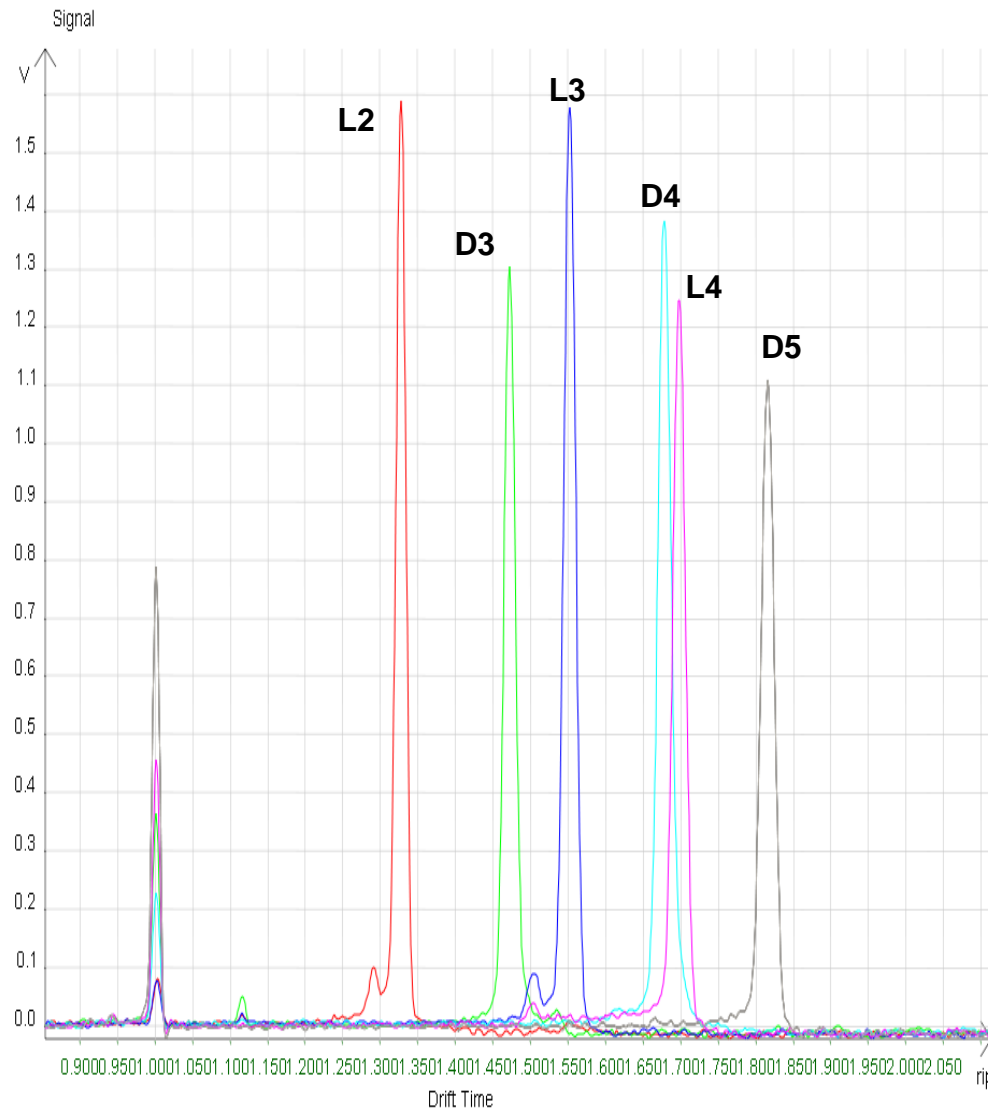
Crucial question for on-site Responsibles: When is the filter saturated?

On-site Solution: GC-IMS-SILOX



Sensitive, rugged and easy to use on-site and on-line tool !

Chromatogram of L2-L4, D3-D5



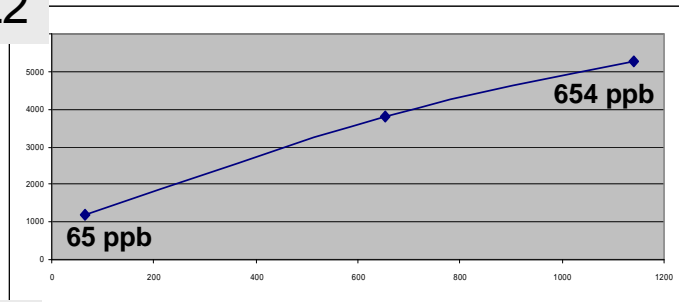
Reliable detection of individual Siloxanes!

GC-IMS-SILOX: Calibration using Permeation Tubes

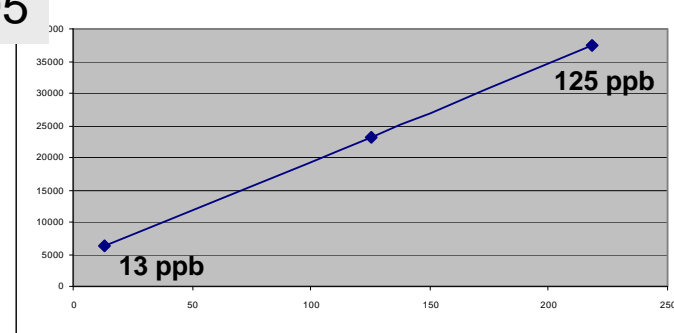


Calibration with test gases of L2, D4 and D5.

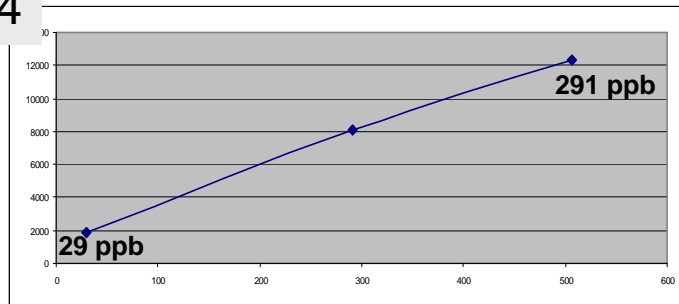
L2



D5



D4



Detection limit requirements in NL for siloxanes are:

L2: 31 ppb, D4: 16 ppb, D5: 13 ppb

Total silicon: 0.8 mg/m³

Customized 'Siloxanes Solution': Set-up on-line Monitoring



Start
System
Info

Last Quantification Results

09.01.2017 / 09:46:38

TOTAL Si	1.3 mg/m³
TOTAL SiO2	2.7 mg/m³
TOTAL SILOXANES	3.8 mg/m³
L2	0.1 mg/m ³
L3	1.8 mg/m ³
L4	0.2 mg/m ³
L5	n.d.
D3	0.7 mg/m ³
D4	0.5 mg/m ³
D5	0.5 mg/m ³
D6	n.d.

Clear
Report...
◀ History

MEA

←

Start measurement manually

CAL

←

Start calibration manually

INTERVAL

←

Start automatic measurement cycle

SCOPE

RESULTS

1F3-00065

READY
SILOX1

SMB
192.168.100.59
DHCP
AUTO

10:13
Sep. 16.
2016

Log

100% remote and continuous Operation using 'INTERVAL' Mode!

Interval Mode

Measurement - Start Time: Fri, 12. Dec 2014 11:00

Measurement - Every: 1 hours

Calibration - Start Time: Fri, 12. Dec 2014 13:00

- Every: 1 days

Substance Calibration Definitions [Edit...](#)

Next Measurement: Fri, 12. Dec 2014, 11:00
Next Calibration: ---

[Start](#) [Cancel](#)

Example: Automatic Measurement once every hour!

Start System Info

Last Quantification Results

09.01.2017 / 09:46:38

TOTAL Si	1.3 mg/m ³
TOTAL SiO ₂	2.7 mg/m ³
TOTAL SILOXANES	3.8 mg/m ³
L2	0.1 mg/m ³
L3	1.8 mg/m ³
L4	0.2 mg/m ³
L5	n.d.
D3	0.7 mg/m ³
D4	0.5 mg/m ³
D5	0.5 mg/m ³
D6	n.d.

SCOPE

RESULTS

MEASUREMENT MODE: MEA

FUNCTION: CAL

INTERVAL

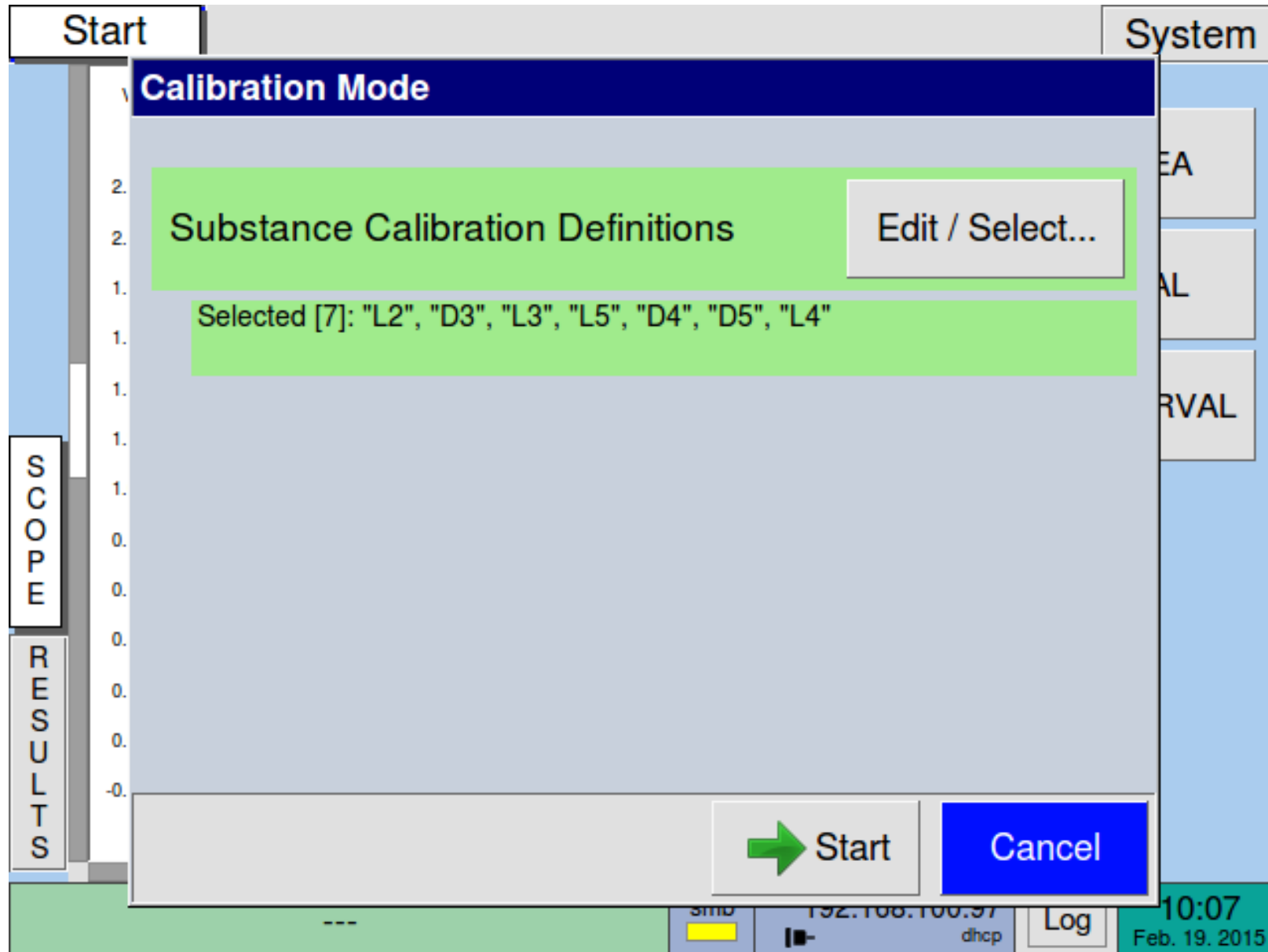
Clear Report... History

1F3-00065 READY SILOX1 SMB 192.168.100.59 10:13
AUTO DHCP Sep. 16. 2016 Log

Display of last result of individual siloxanes, 'Total Siloxanes' and 'Total Silicon' (Si).

Start		System	Info
SCOPE	Last Quantification Results		MEA
			CAL ← Calibration Button
			INTERVAL
RESULTS	09.01.2017 / 09:46:38		
	TOTAL Si	1.3 mg/m ³	
	TOTAL SiO ₂	2.7 mg/m ³	
	TOTAL SILOXANES	3.8 mg/m ³	
	L2	0.1 mg/m ³	
	L3	1.8 mg/m ³	
	L4	0.2 mg/m ³	
	L5	n.d.	
	D3	0.7 mg/m ³	
	D4	0.5 mg/m ³	
D5	0.5 mg/m ³		
D6	n.d.		
Clear		Report...	← History
1F3-00065	READY SILOX1	SMB 192.168.100.59 DHCP AUTO	10:13 Sep. 16. 2016 Log

Easy one-point Calibration for on-site Operation available*!
** Availability of certified test gas still key problem.*



The screenshot shows a software interface with a 'Calibration Mode' dialog box. The dialog has a dark blue header with the text 'Calibration Mode'. Below the header, there is a green box containing the text 'Substance Calibration Definitions' and an 'Edit / Select...' button. Underneath this, another green box displays 'Selected [7]: "L2", "D3", "L3", "L5", "D4", "D5", "L4"'. At the bottom of the dialog, there are two buttons: a green arrow button labeled 'Start' and a blue button labeled 'Cancel'. The background interface includes a 'Start' button at the top left, a 'System' label at the top right, a vertical 'SCOPE' label on the left, and a 'RESULTS' label below it. On the right side, there are several buttons labeled 'EA', 'AL', and 'RVAL'. The bottom status bar shows 'SMB', '192.168.100.97', 'dhcp', 'Log', and the date/time '10:07 Feb. 19. 2015'.

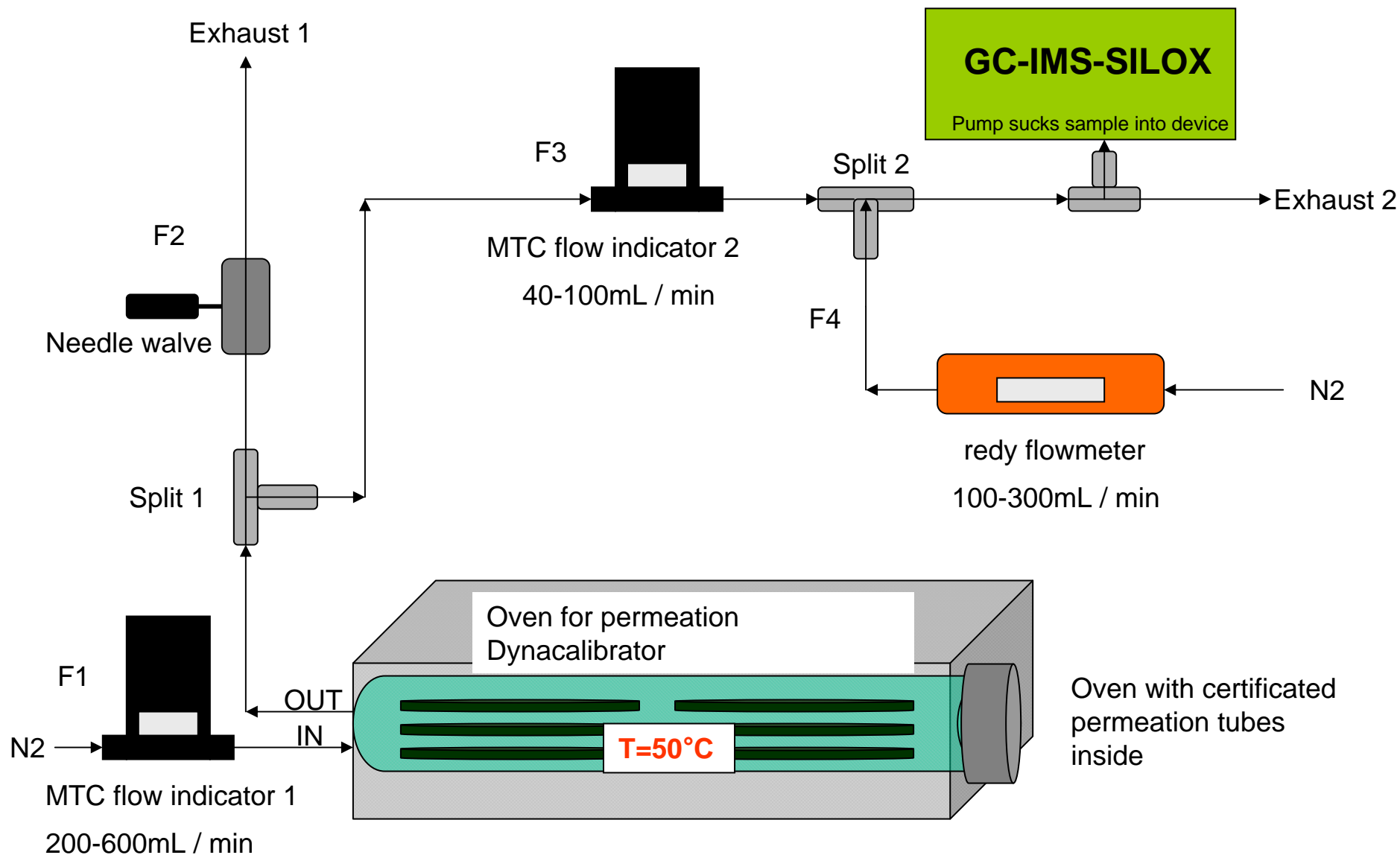
GC-IMS-SILOX: Set Calibration Gas Values



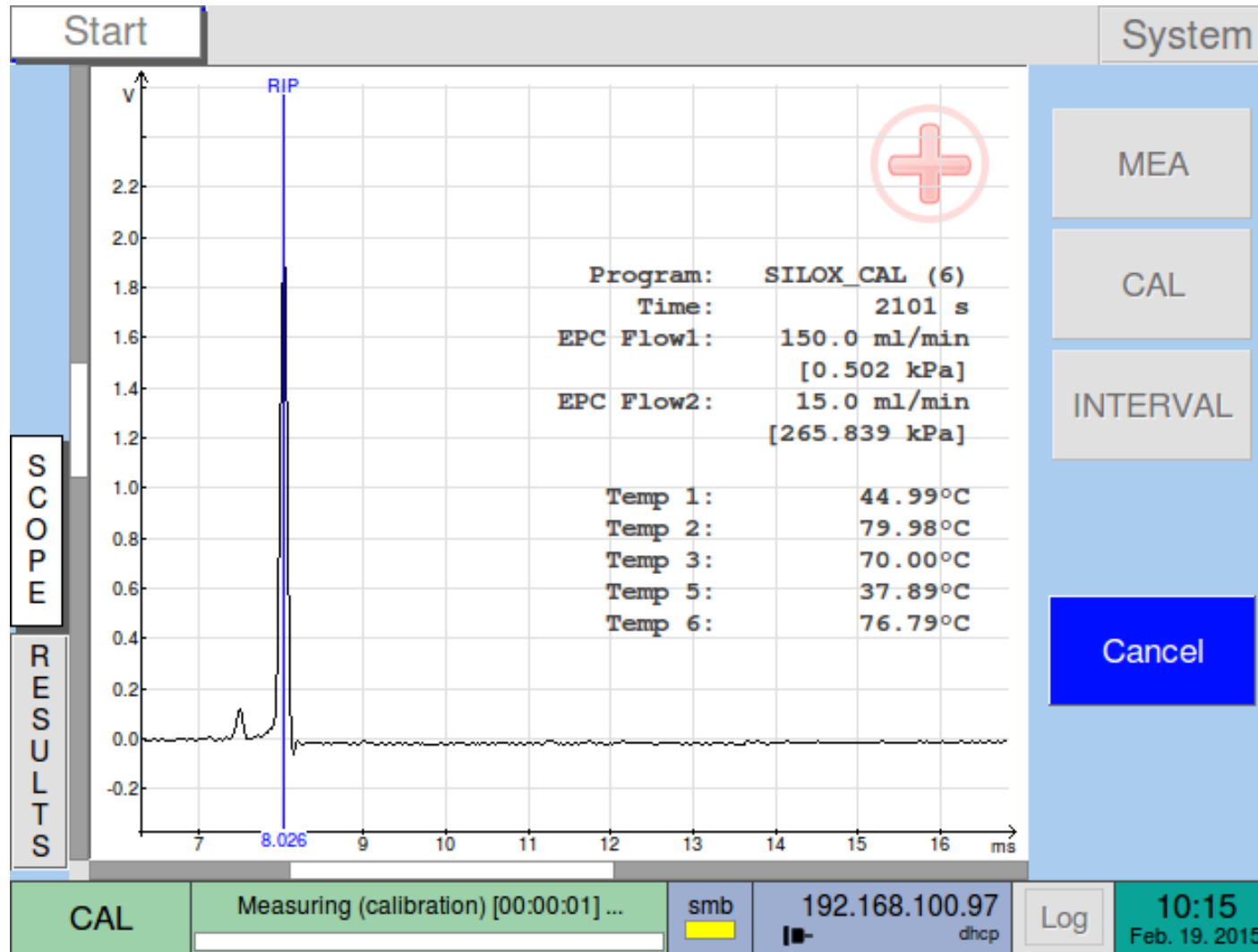
Concentration in Test Gas				
Min:	0.000	2.020	mg/m3	
Max:	5.700			
7	8	9	Clear	+
4	5	6	←	+
1	2	3		-
0	.			-
			OK	Cancel

Substance Calibration	
<input type="button" value="Toggle"/>	<input type="button" value="Unselect All"/>
<input checked="" type="checkbox"/> L2	Concentration in Test Gas 2.020 mg/m3
<input type="checkbox"/> D4	Attributes
<input type="checkbox"/> D3	Last Auto-Calibration ---
<input type="checkbox"/> L5	Correction Factor 1.000
<input type="checkbox"/> D5	Type Standard
<input type="checkbox"/> L3	Concentration Range 0.000 mg/m3 - 5.700 mg/m3
<input type="checkbox"/> L4	Import Timestamp 2015-10-22 09:54:54
	Fingerprint f11eba4372ccb7b9 cae26c4ff0d35fcc
	Averaging
<input type="button" value="Import Calibration"/>	<input type="button" value="Close"/>

Setup for Calibration of the Siloxanes



GC-IMS-SILOX: Calibration Running



Contamination: GC-IMS-SILOX Cleaning Mode



Start System

Measurements Information

Cleaning in progress

EPC1 $\frac{ml}{min}$	EPC2 $\frac{ml}{min}$	T1 $^{\circ}C$	T2 $^{\circ}C$	T3 $^{\circ}C$	T4 $^{\circ}C$	T5 $^{\circ}C$	T6 $^{\circ}C$
150.0	15.0	46.4	>80	71.0	---	38.1	76.8

V

ms

EPC Flow1: 150.0 ml/min
[0.499 kPa]

EPC Flow2: 15.0 ml/min
[266.154 kPa]

Temp 1: 46.35 $^{\circ}C$

Temp 2: 80.47 $^{\circ}C$

Temp 3: 71.03 $^{\circ}C$

Temp 5: 38.12 $^{\circ}C$

Temp 6: 76.84 $^{\circ}C$

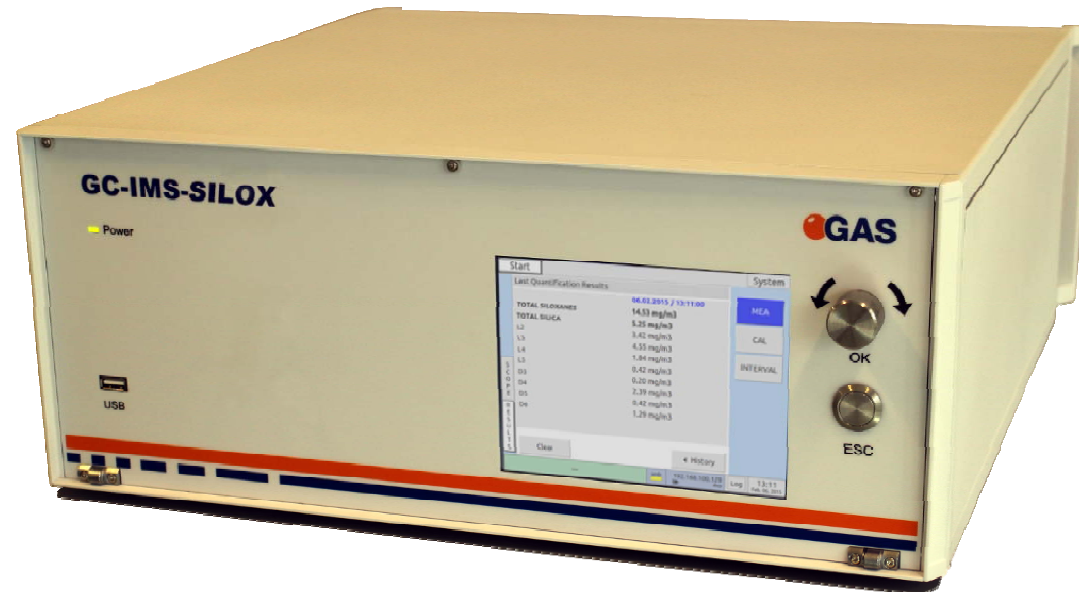
Time remaining: 8:59:43 Skip

smb

192.168.100.97
dhcp

Log

15:28
Feb. 19, 2015



- Easy to operate – no analytical specialist needed
 - Precise (<10% at span)
 - Fast: Typically <15 minutes run times
 - Online and/or portable on-site Operation
- Fully automatic measurement at user defined intervals
 - Digital Documentation

- 1. Most relevant siloxanes in biogas L2-L5, D3-D5 (D6 and TMSOL optional) can be measured and quantified on-site and on-line.**
- 2. Individual siloxanes can be measured starting from 0.03 mg/m³ while the determination of the 'total silicon' goes up to ~3mg/m³. Other calibration ranges on request.**
- 3. Filter breakthrough can be detected and full filter capacity can be used.**

Thank you for your attention!

For further questions

please contact

Thomas Wortelmann

G.A.S. mbH Gesellschaft für analytische Sensorsysteme mbH

Otto-Hahn Str. 15

44227 Dortmund, Germany

+49 231 97426550

www.gas-dortmund.de

A member of

