

Quantitative Gas Analysis

Determination of the concentration of gas species as a part of the hole gas mixture.

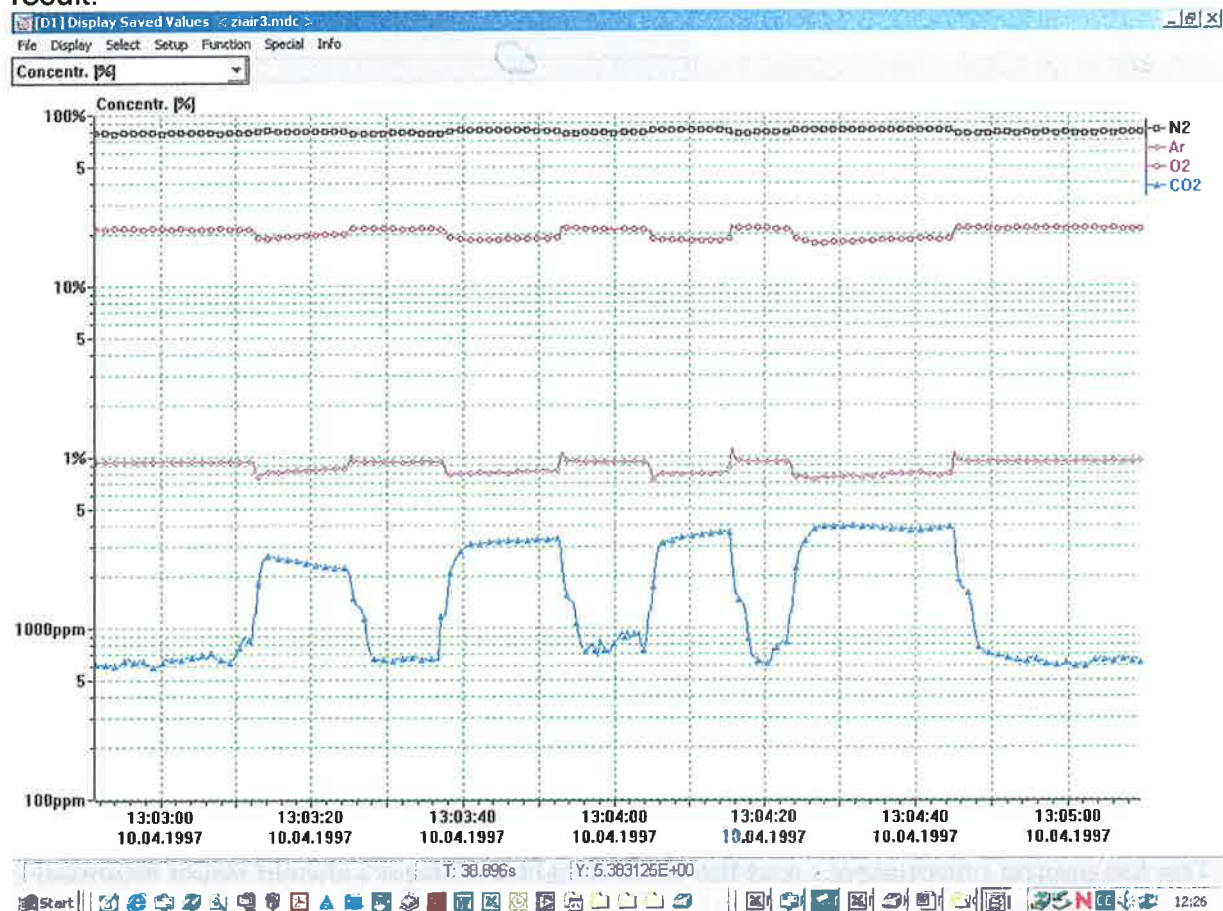
predictions:

- QMS with QuadStar software (QAM-module)
- calibration gases

procedure with the QuadStar software:

- tuning of the ion source parameters (" tune up ")
- offset calibration
- mass scale adjust
- zero gas measurement (background determination)
- gas specific calibration with calibration gases with known concentration
- measurement of the unknown gas mixture

result:



Tuning of the ion source parameter

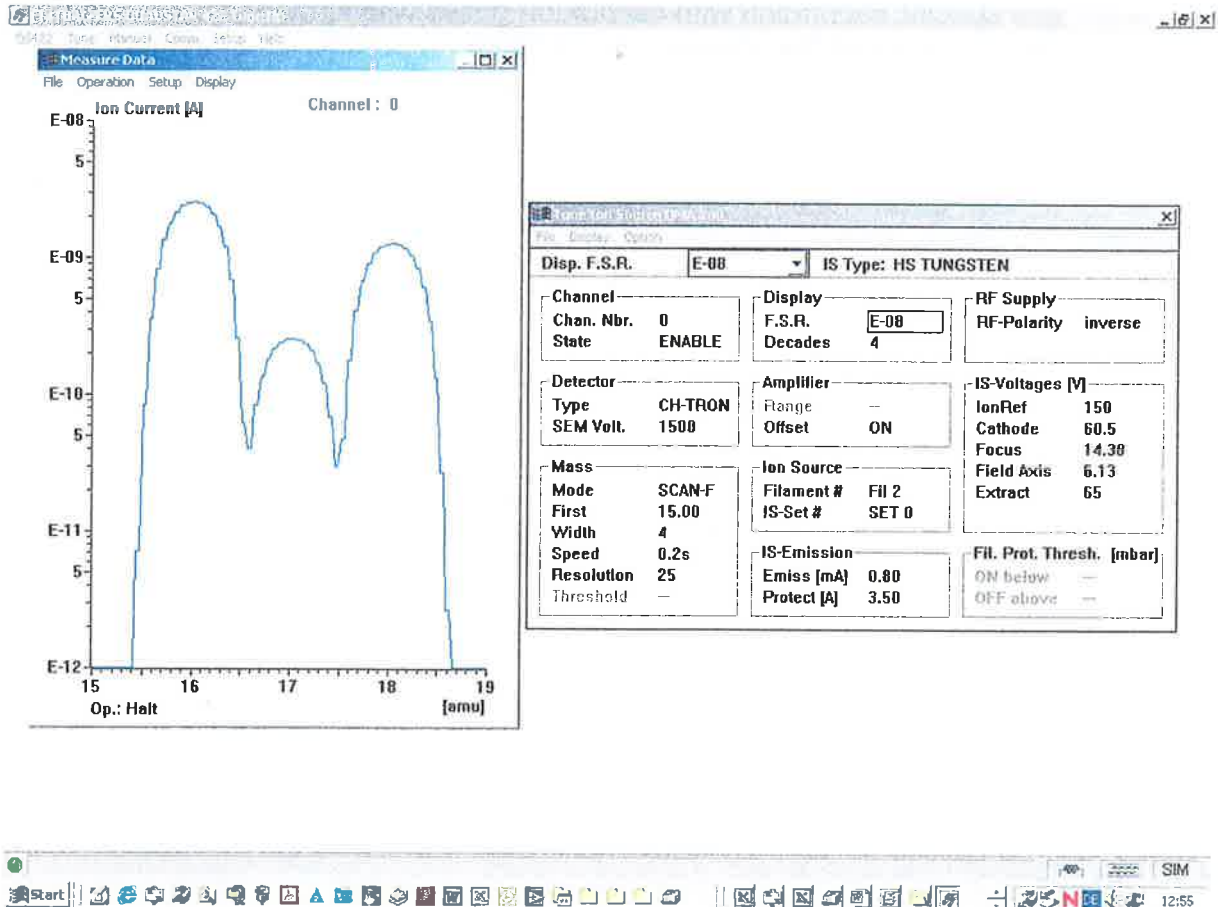
prediction: sample gas inlet

“Tuneup” menu, → “Ion source”

optimization of the ion source parameters regarding of:

- maximum of intensity
- best resolution for the interesting masses
- peak shape
- stability

result:



Remark:

The ion source parameters (and the SEM detector voltage) should never changed during the following measurements !!!

Offset calibration

Determination of the offset of the electrometer preamplifier in each range.
(without gas inlet or with emission off)

Measure

"Tuneup" menu, → "offset", → "parameters"



- select mass 5,5 amu for QMA 400/430/410 and 50,5 amu for PRISMA
- Set detector type to Faraday and resolution to 1 (Prisma)

result:

operation → remeasure



Range	Previous Offset Values			New Offset Values		
	Fast	Normal	Slow	Fast	Normal	Slow
1E-05	0.0000E-05	0.0000E-05	0.0000E-05	0.0000E-05	0.0000E-05	0.0000E-05
1E-06	0.0000E-06	0.0000E-06	0.0000E-06	0.0000E-06	0.0000E-06	0.0000E-06
1E-07	0.0053E-07	0.0053E-07	0.0060E-07	0.0054E-07	0.0054E-07	0.0060E-07
1E-08	0.0000E-08	0.0000E-08	0.0000E-08	0.0000E-08	0.0000E-08	0.0000E-08
1E-09	0.0000E-09	0.0000E-09	0.0000E-09	0.0000E-09	0.0000E-09	0.0000E-09
1E-10	0.0054E-10	0.0054E-10	0.0060E-10	0.0055E-10	0.0055E-10	0.0060E-10
1E-11	0.0668E-11	0.0000E-11	0.0000E-11	0.0680E-11	0.0000E-11	0.0000E-11
1E-12	****	0.0054E-12	0.0060E-12	****	0.0054E-12	0.0060E-12



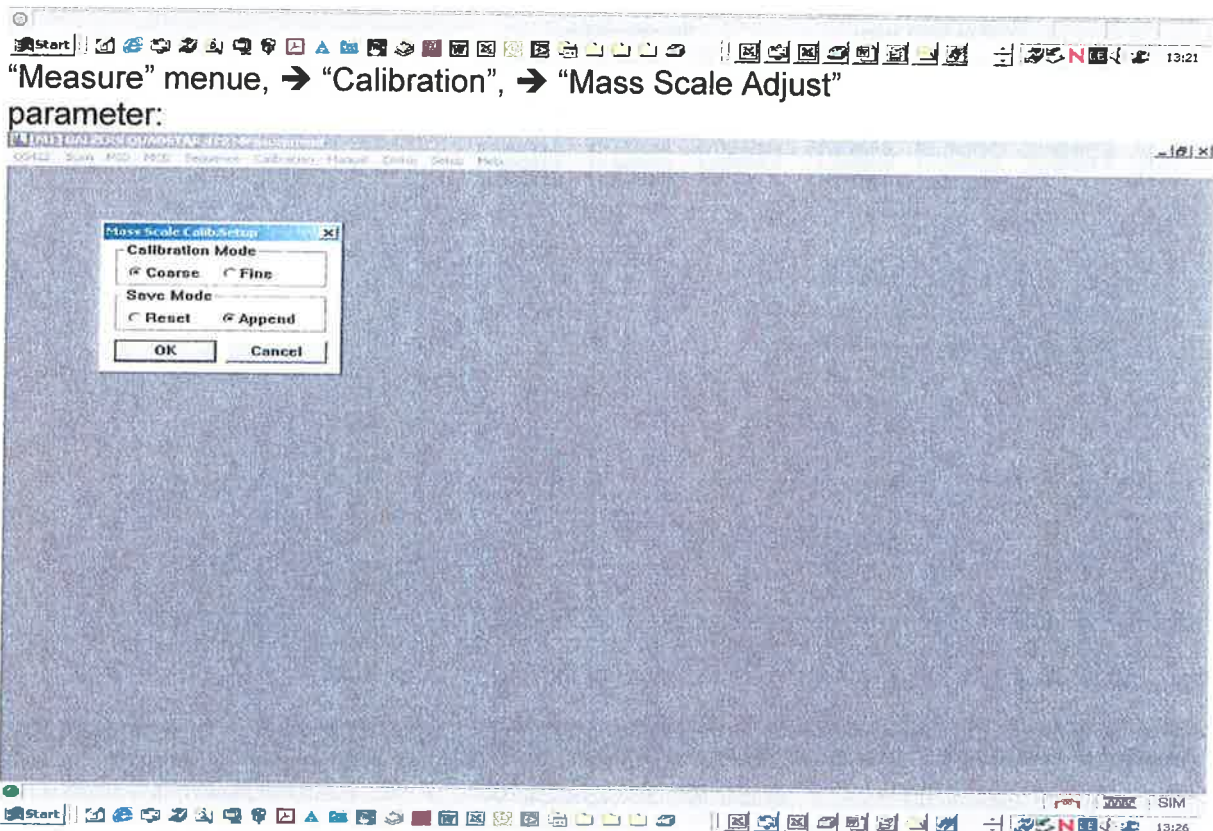
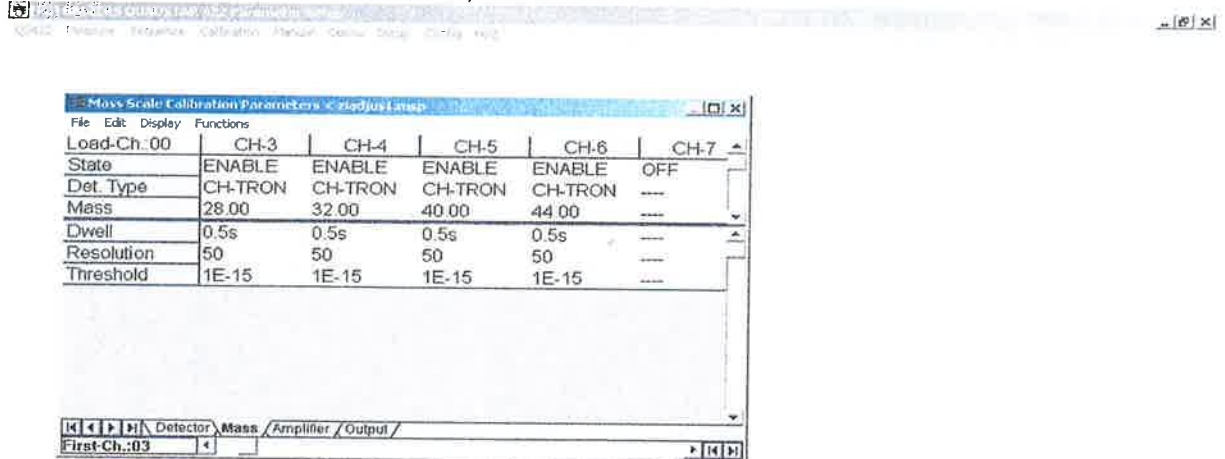
Mass scale adjust

Adjust of the peak maximum of each mass number of interest.

predictions: - gas inlet of the sample gas (or calibration gases)

parameter:

“Parset” menu, → “Calibration”, → “Mass Scale...”



Remarks:

- "coarse" → "fine"
- in the "append" mode, new mass numbers will be append, existing mass numbers will be overwritten

result:



Nbr	Old Mass	New Mass	Intensity	State
0	28.00	28.00	9.990E-09A	ok
1	32.00	32.00	8.308E-09A	ok
2	40.00	39.97	1.662E-09A	ok
3	44.00	44.00	6.286E-10A	ok



Remark:

- all existing parameter files (MID and MCD) will be corrected with the actual mass scale correction

Zero Gas Measurement

Determination of the intensity at the interesting mass numbers caused by the "background" of the analyze chamber or by a "zero gas" inlet.

→ $I_{res.}$ ($m/e = ?$)

predictions:

- inlet of the zero gas (very pure gas)
- or closed gas inlet, determination of the residual gas components

parameter:

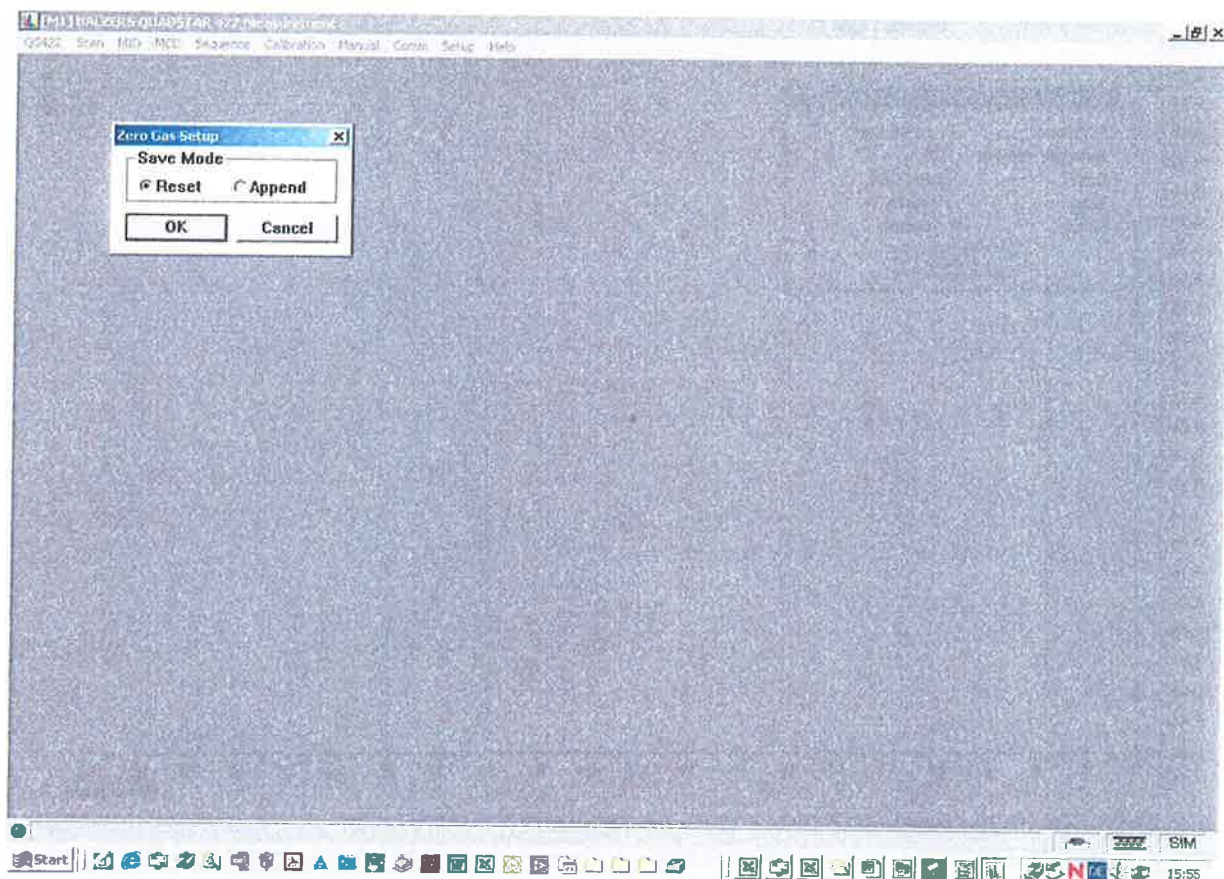
"Parset" menue, → "MID",



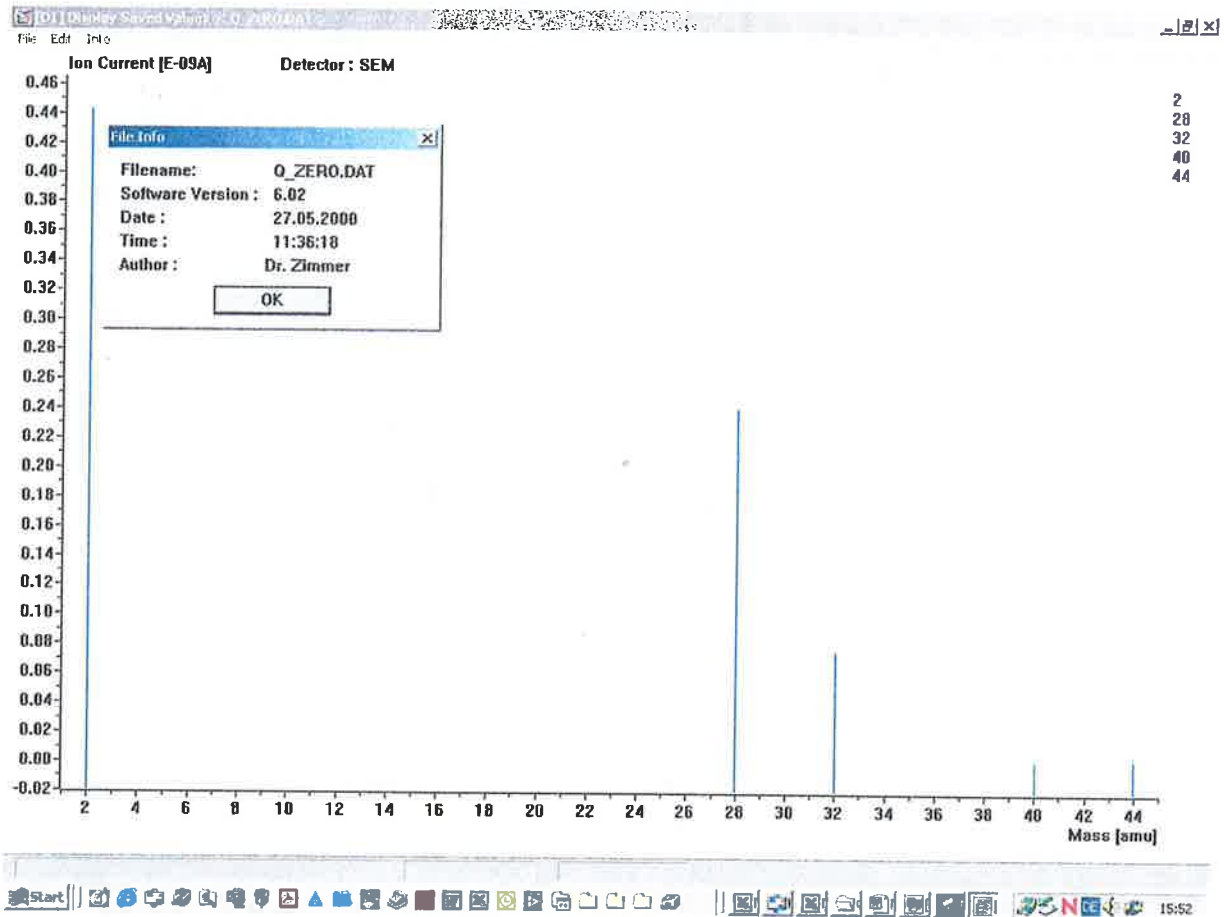
Load-Ch.:00	CH-5	CH-6	CH-7	CH-8	CH-9
State	ENABLE	SKIP	ENABLE	ENABLE	ENABLE
Det. Type	CH-TRON	CH-TRON	CH-TRON	CH-TRON	CH-TRON
Mass	28.00	29.00	32.00	40.00	44.00
Dwell	0.5s	0.5s	0.5s	0.5s	0.5s
Resolution	50	50	50	50	50



“Measure” menu, → “Calibration”, → “Zero Gas....”



result: in file "Q_zero.dat"



Remarks:

- you can determine the background at more mass numbers than you need (in the example $m/e = 2$)
- in the "append" mode, new mass numbers will be appended, existing mass numbers will be overwritten

Gas Specific Calibration

Determination of the calibration factors (matrix) for each mass number and gas species.

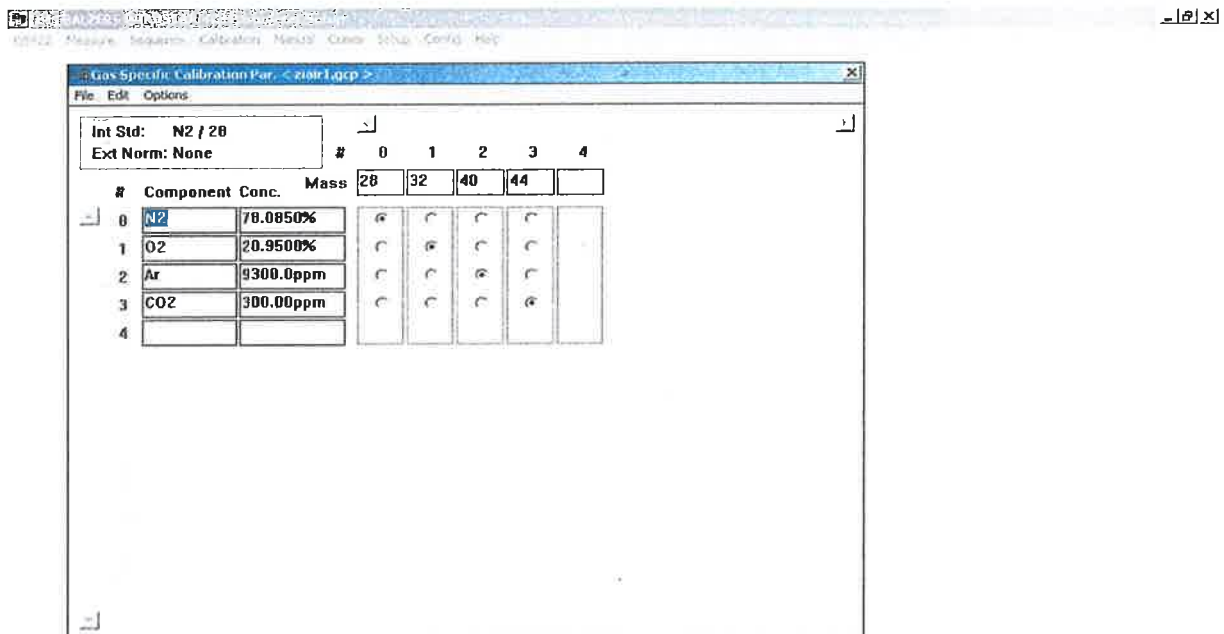
$$\rightarrow K(C_x) = I_{cal}(x) / C_x, \text{ with } I_{cal}(x) = I_{tot}(x) - I_{res}(x)$$

predictions:

- calibration gas mixtures with non overlapped fractal pattern
- calibration gas inlet open

parameter:

"Parset" menu, \rightarrow "Calibration", \rightarrow "Gas Specific Sensity"



Remarks:

"Internal Standard" means not only, that the calibration factor is equal "1", it means also that the component which is defined as "internal standard" change not the (former determind) calibration factor.

Example:

Now we have for the same measurement task 3 calibration gas mixtures.

- a) Ar / CO₂
- b) N₂ / O₂
- c) Ar / O₂

possible calibration procedure:

1. Ar / O₂ , internal standard : Ar
2. N₂ / O₂ , internal standard : O₂
3. Ar / CO₂ , internal standard : Ar

“Measure” menu, → “Calibration”, → “Gas Specific Sensity”

result:



Nbr	Mass	Component	Concentration	Intensity	Calib.Factor
0	28.00	N2	78.0850%	9.985E-09A	* 1.000E+00
1	32.00	O2	20.9500%	8.306E-09A	3.101E+00
2	39.97	Ar	9300.0ppm	1.857E-09A	1.394E+01
3	44.00	CO2	300.00ppm	6.209E-10A	1.639E+02



Remarks:

- “*” shows the internal Standard (in the example N₂ at 28)

MCD measurement

Determination of the concentration of gas species in a unknown gas mixture.

$$\rightarrow C(x) = I(x) / K(x) \quad \text{with } I(x) = I_{\text{total}}(x) - I_{\text{res}}(x)$$

predictions:

- all calibration procedures done
- no change in the ion source parameters, same detector, same SEM voltage (use the feature "common voltage"), same resolution parameter
- sample gas inlet open

parameter:

"Parset" menu, \rightarrow "Measure", \rightarrow "MCD..."

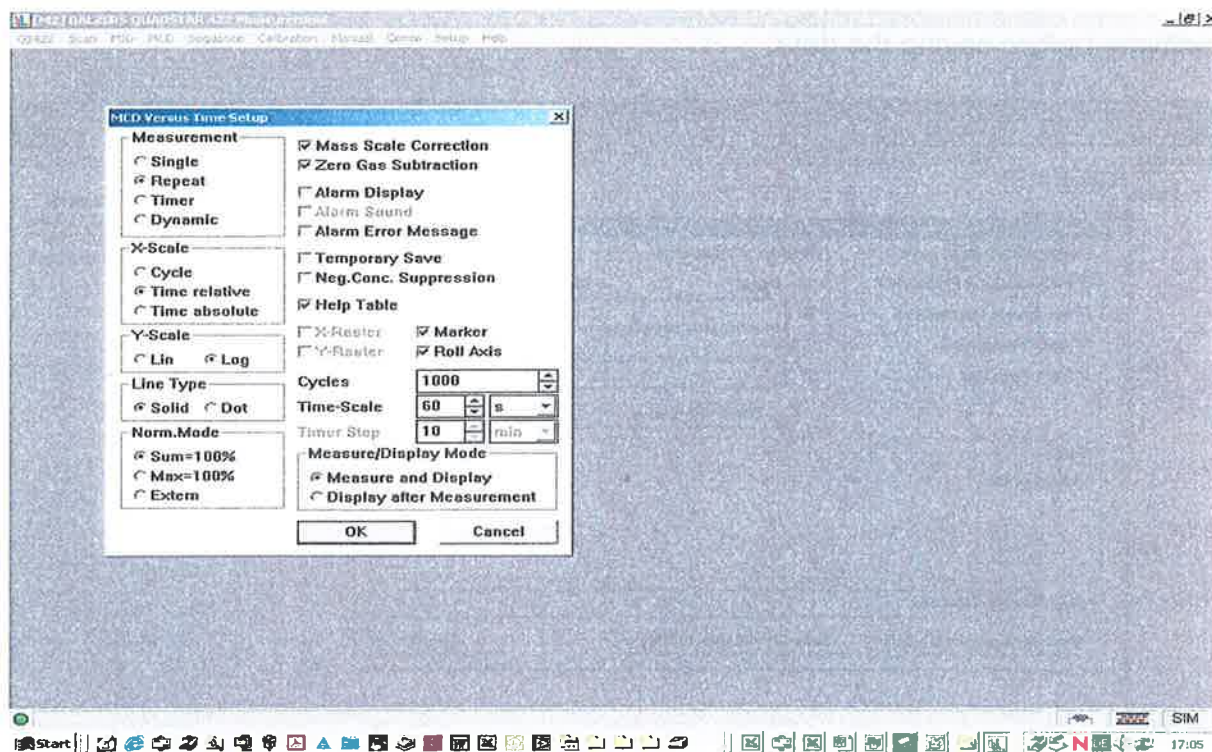
The screenshot shows the SIMION software interface. The main window displays a table of mass spectrometry data. Below it, the 'MCD Channel Parameters' dialog box is open, showing settings for four channels (CH-0 to CH-4).

#	Comp.	Mass	28	32	40	44	
0	N2		1	0	0	0	
1	Ar		0	0	13.94	0	
2	O2		0	3.101	0	0	
3	CO2		0	0	0	163.9	
4							

Load-Ch.:00	CH-0	CH-1	CH-2	CH-3	CH-4
State	MATRIX	MATRIX	MATRIX	MATRIX	OFF
Det. Type	CH-TRON	CH-TRON	CH-TRON	CH-TRON	---
Mass	28	32	40	44	---
SEM Voltage	<< 1500 >>	<< 1500 >>	<< 1500 >>	<< 1500 >>	---
Dwell	0.1s	0.1s	0.1s	0.2s	---
Resolution	50	50	50	50	---
Threshold	---	---	---	---	---

At the bottom of the dialog box, there is a control bar with the following text: $\left[\leftarrow \right] \left[\rightarrow \right] \backslash$ Detector / Mass / Amplifier / Output / Trip / Display /

“Measure” menue, → “MCD”, → “Setup”, → “Versius Time ”

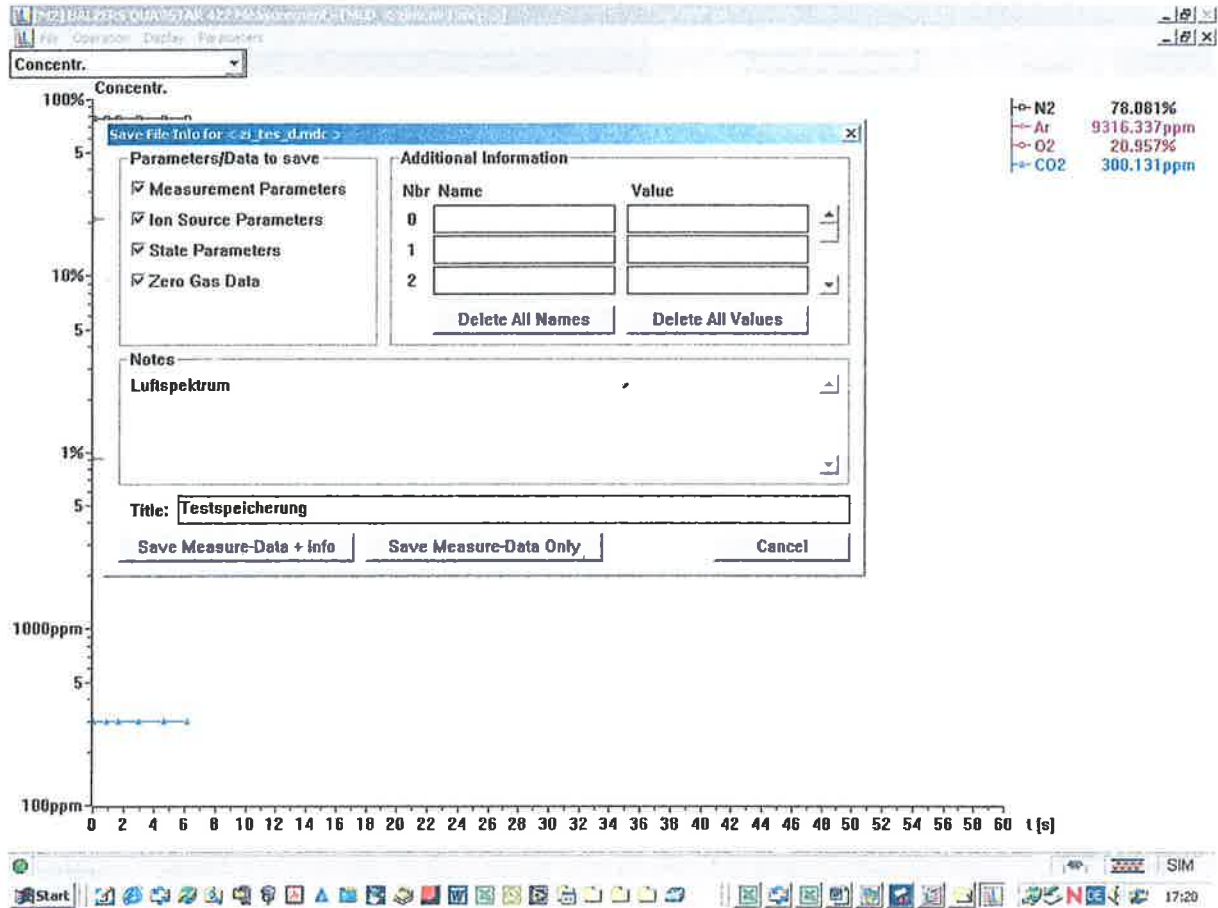


“Measure” menue, → “MCD”, → “Versius Time”
result:



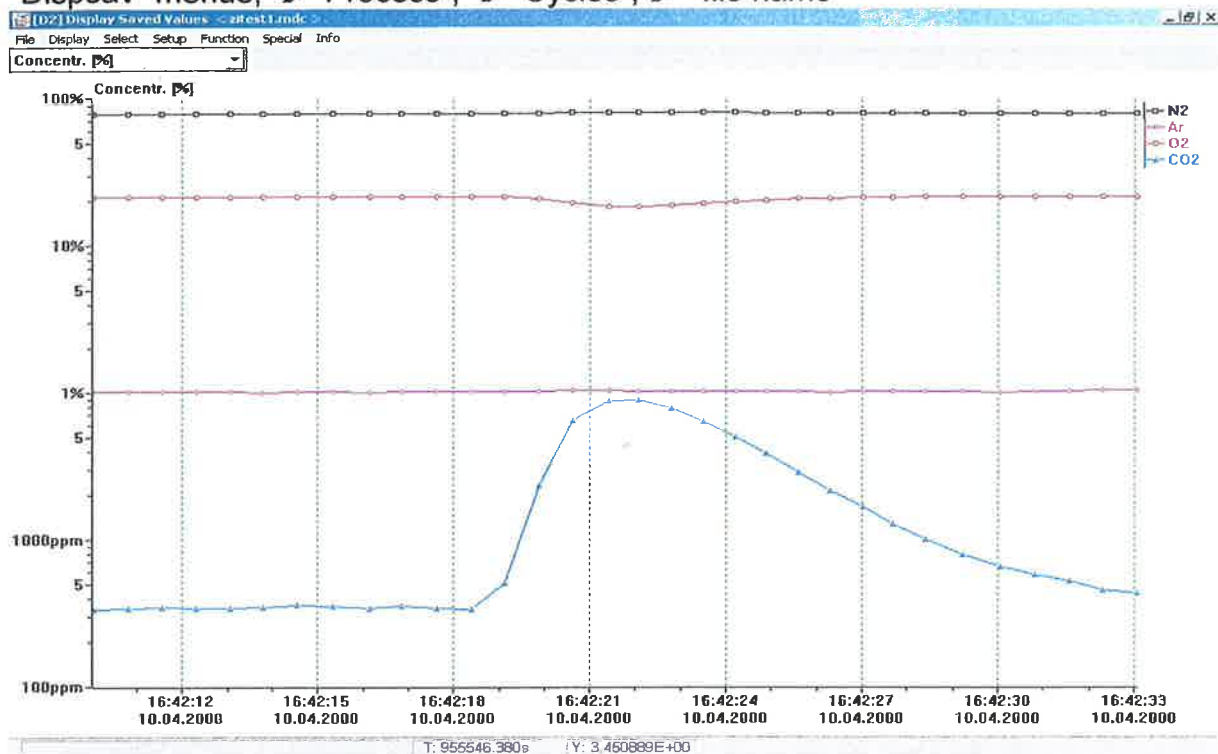
Display Saved Values

activate before saving the data:

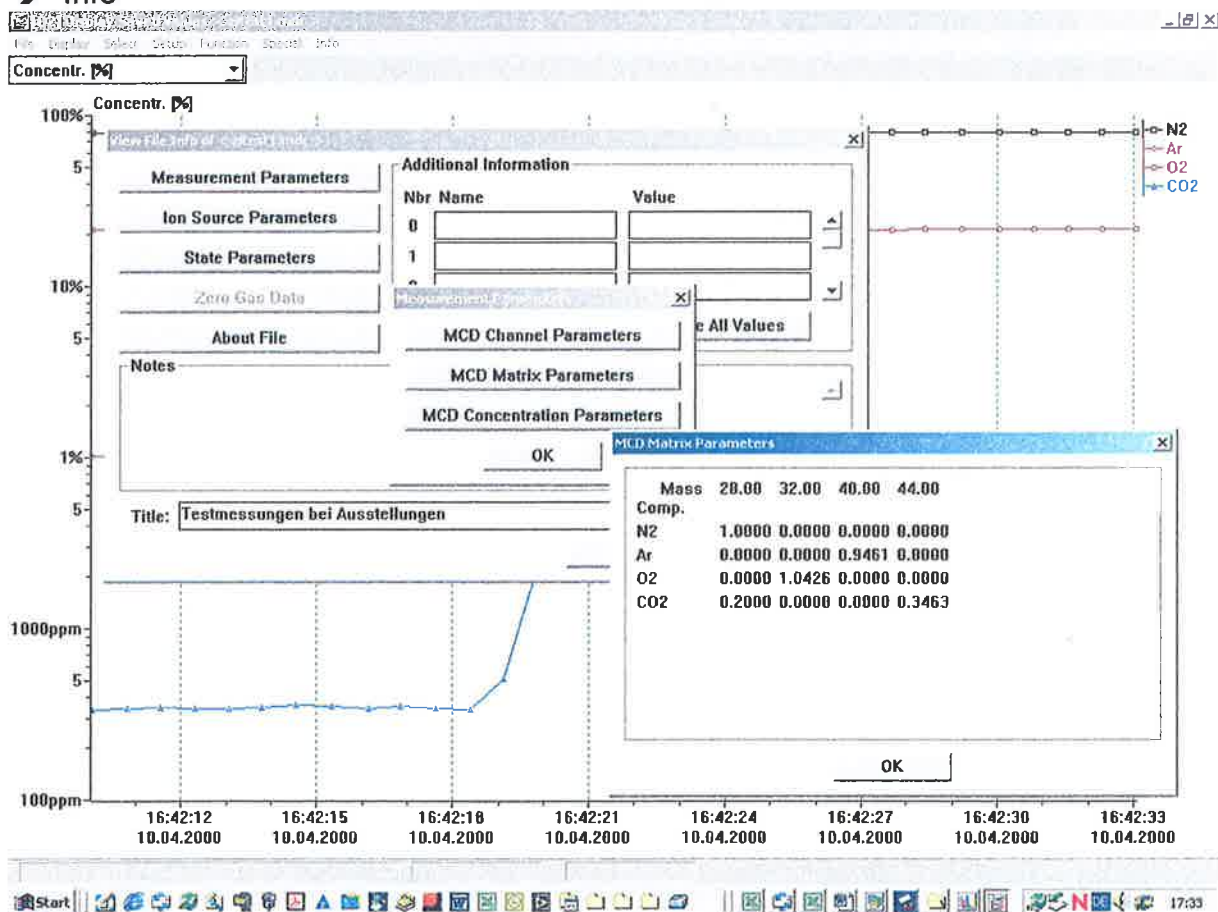


Display Saved Values

“Dispsav” menue, → “Process”, → “Cycles”, → “file name”



→ “Info”



Work with the Process Control Module (Sequencer)

