Pharmbiocon

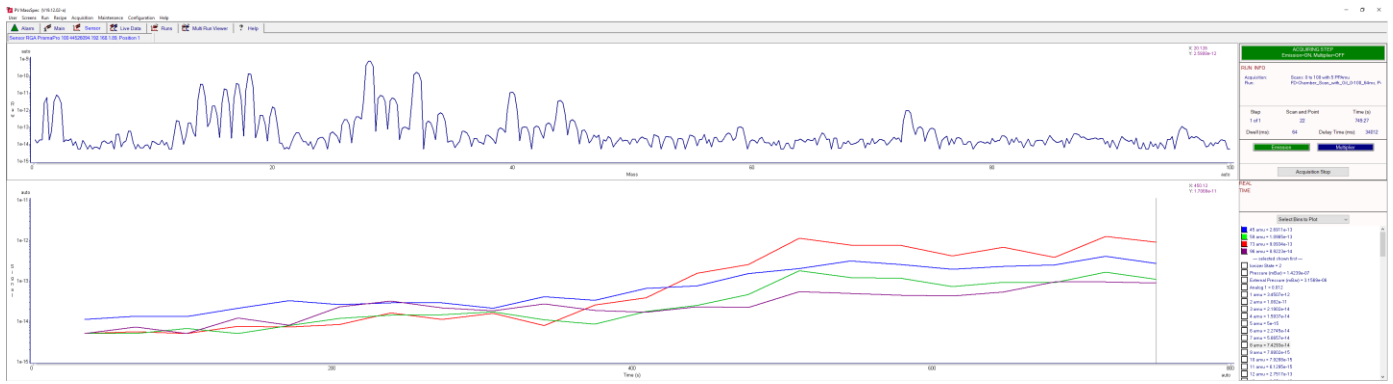
Das Ingenieurbüro im Pharmaumfeld und der Medizintechnikbranche

Basic training mass spectrometer

Content of the training:

- Basics mass spectrometer
- Chemical background
- Interpretation of the measurement result

- Hands-On Part



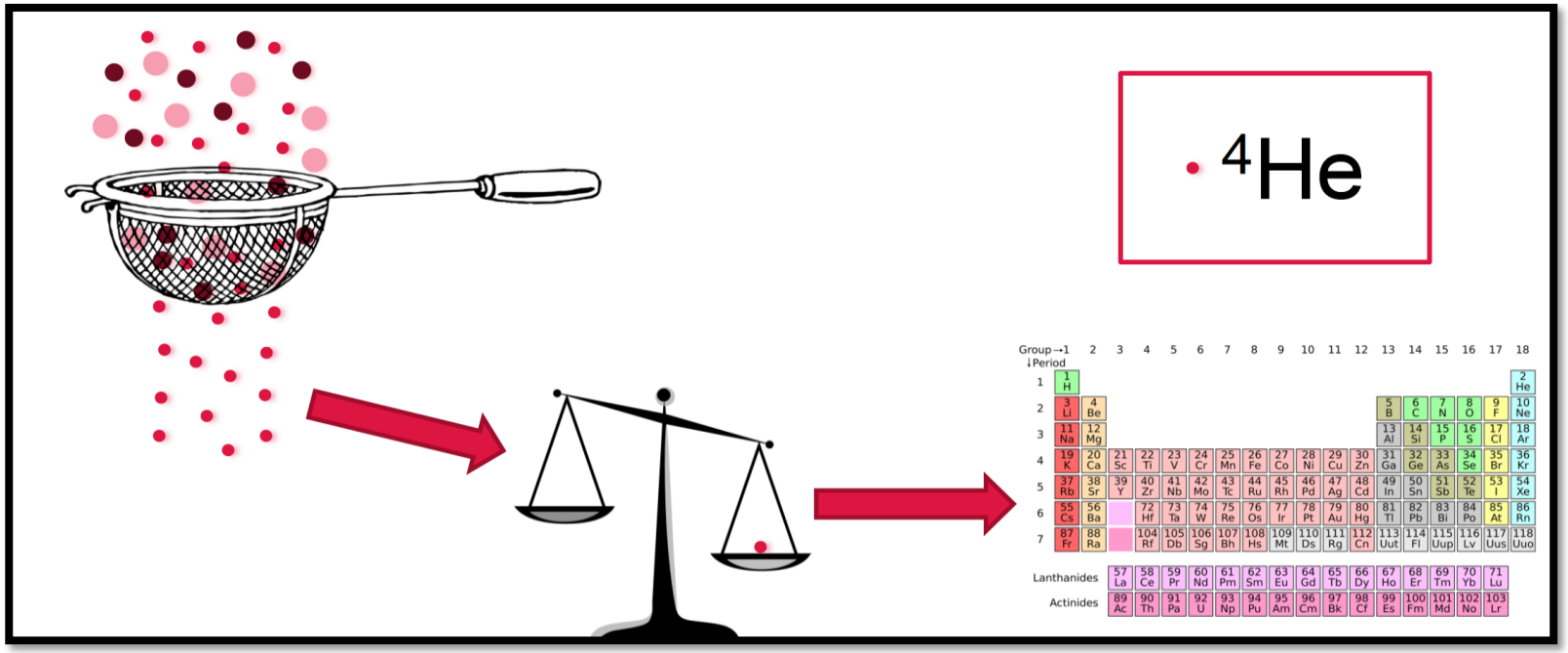
Basic training mass spectrometer

Applications of mass spectrometry:

- For analysis of chemical products in laboratories
- Process analysis in combustion processes, power plant processes, exhaust gas analysis
- Quality control/Vacuumsystems (outgassing, **leak detection**)
- Surface analysis

Basics mass spectrometer

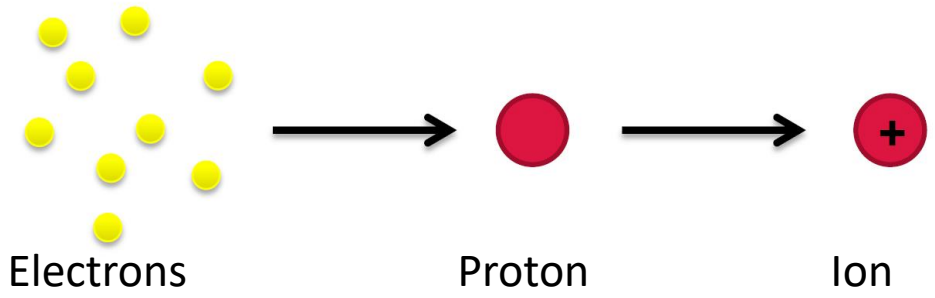
Basic principle of mass spectrometry:

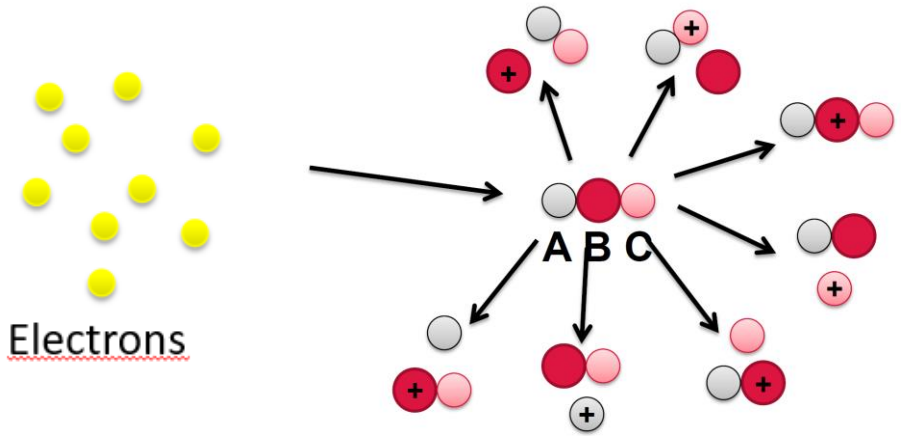


The individual atoms or molecules are separated, the mass/charge ratio is determined and the chemical product is assigned to a specific mass spectrum (fingerprint).

Basics mass spectrometer

Ionization of the gases:

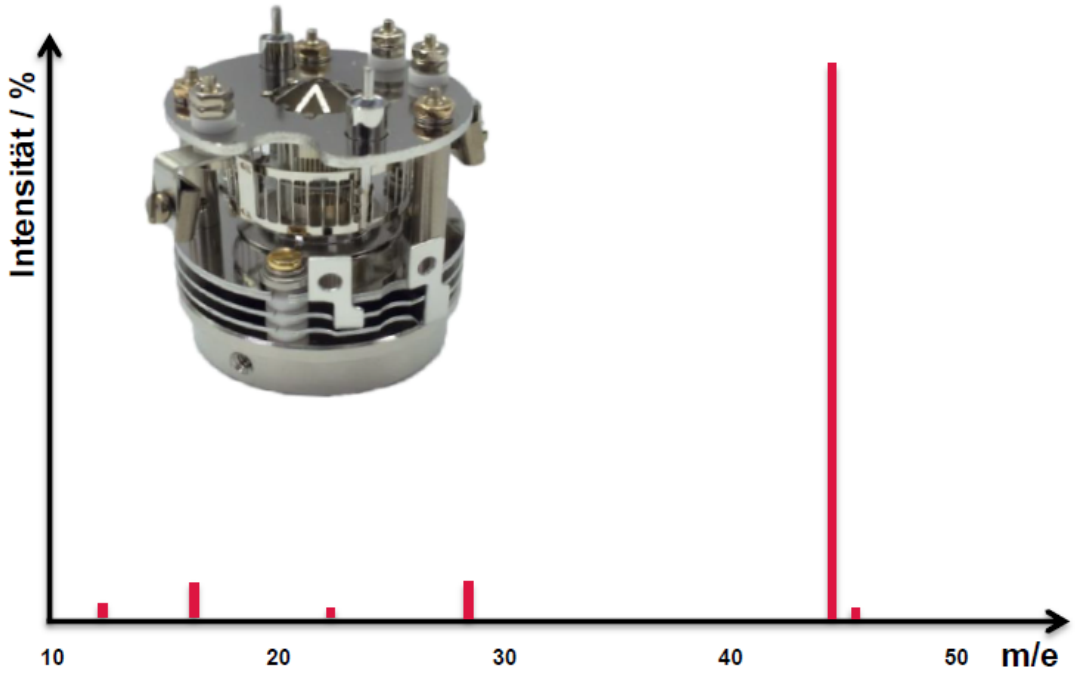




In the ionization process, different fragments are created. Some fragments with high and others with low probability.

Basics mass spectrometer

CO2 fragmentation

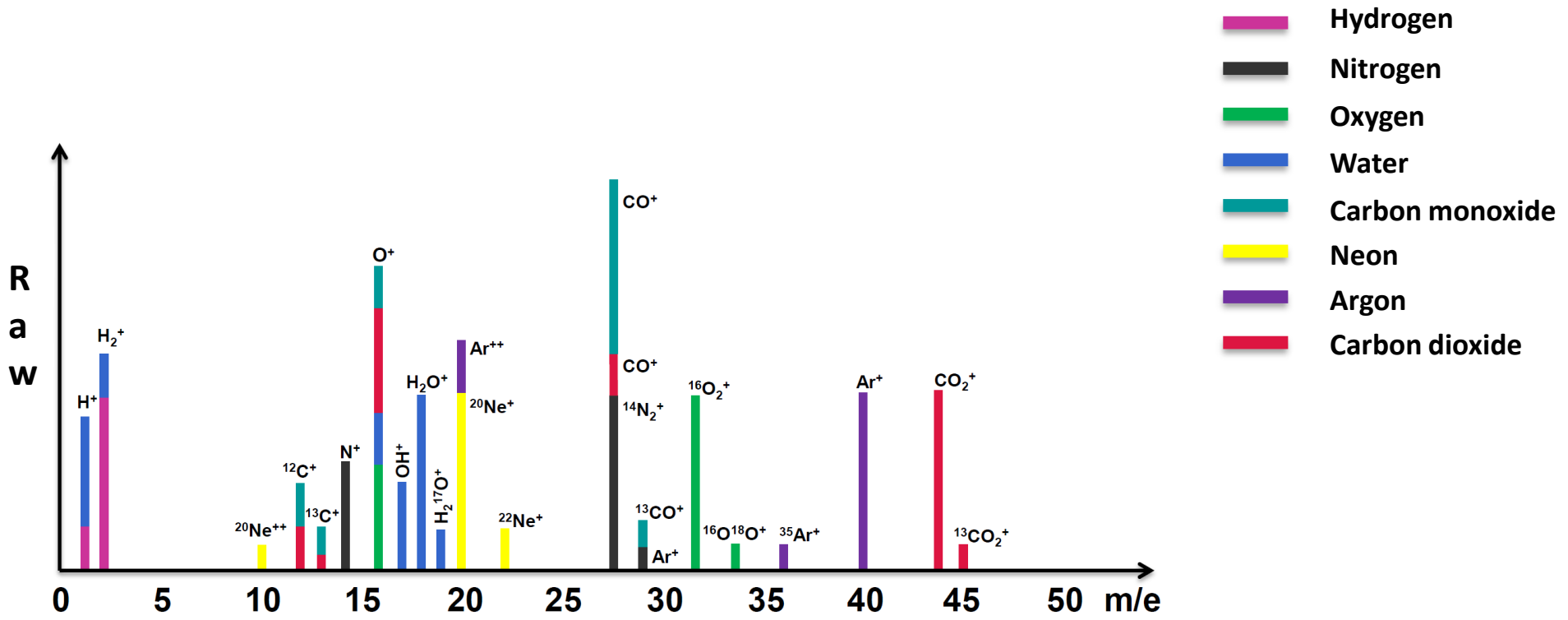


Fragments of CO2

m/e	Intensität	Ion
12	2,46	$^{12}C^{+}$
16	6,24	$^{16}O^{+}$
22	1,78	$^{12}C^{16}O_2^{++}$
28	6,55	$^{12}C^{16}O^{+}$
29	0,06	$^{13}C^{16}O^{+}$
44	**100,00**	$\mathbf{^{12}C^{16}O_2^{+}}$
45	1,16	$^{13}C^{16}O_2^{+}$
46	0,41	$^{12}C^{16}O^{18}O^{+}$

Basics mass spectrometer

Applications of mass spectrometry:

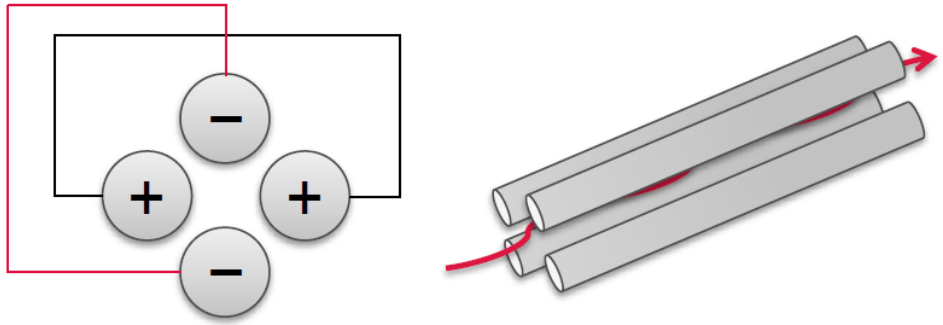


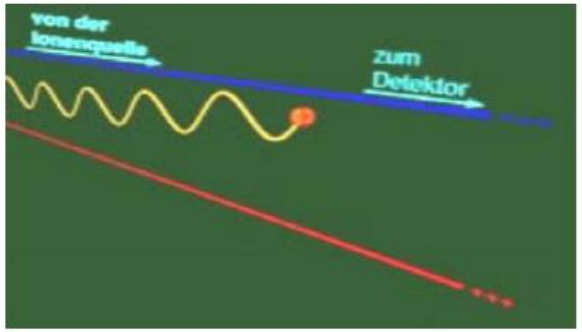
Due to the Gaussian normal distribution, a unique mass spectrum is created for each molecule

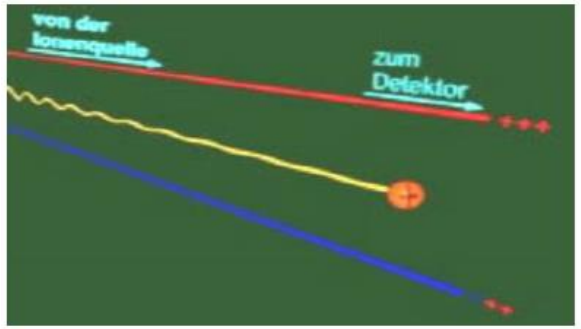
Basics mass spectrometer

Separation of masses:

- 4 cylindrical metal bars
- opposite bars are electrically connected
- an AC voltage field is applied to a DC voltage field
- stable flight paths for certain ions
- with a suitable m/e- relation, the ion reaches the detector
- if the relation does not fit, the ion is deflected before it hits the detector

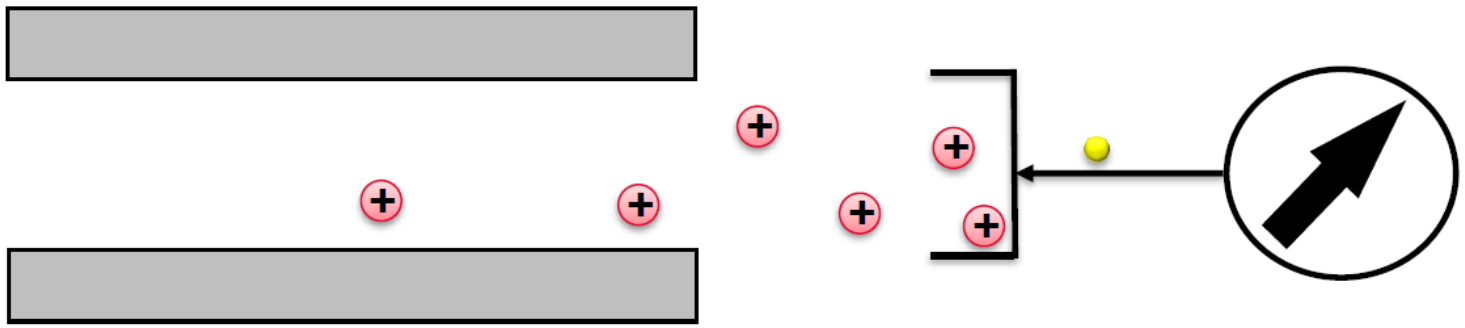






Basics mass spectrometer

Detector:

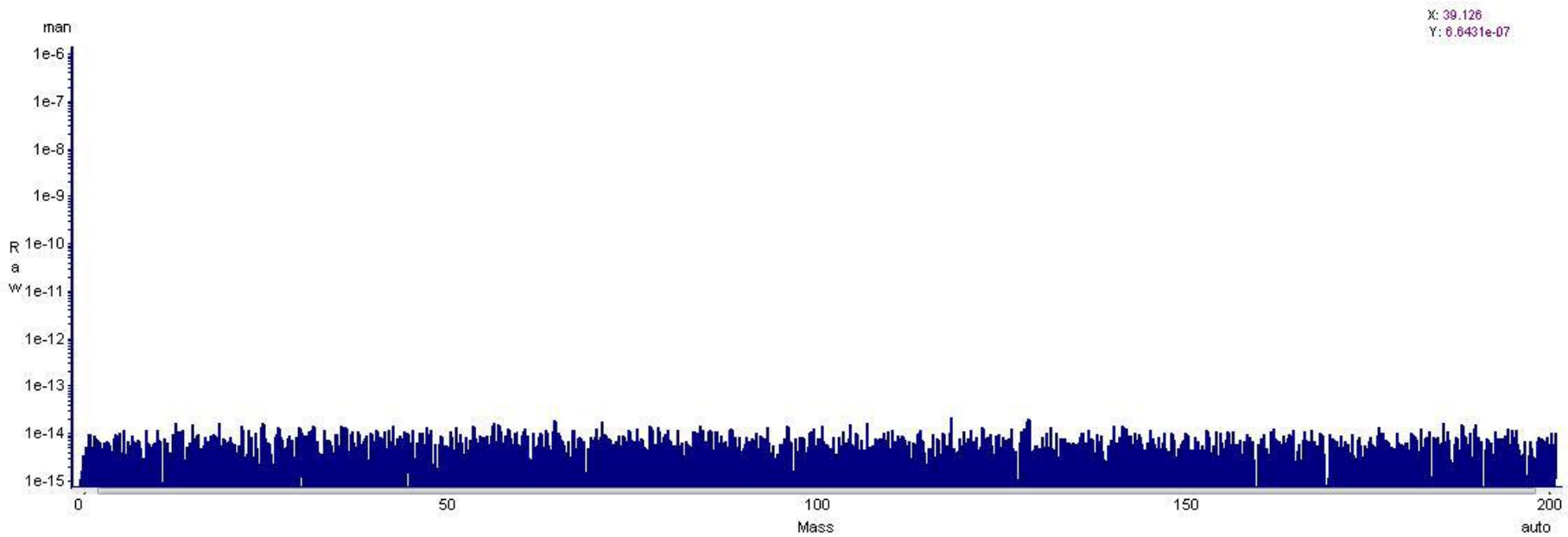


- If the m/e- relation is correct, the corresponding ion flies through the magnetic field and hits the detector.
- The relation m/e- is adjusted for each mass, so that only the corresponding mass crosses the path.

Basics mass spectrometer

Detector:

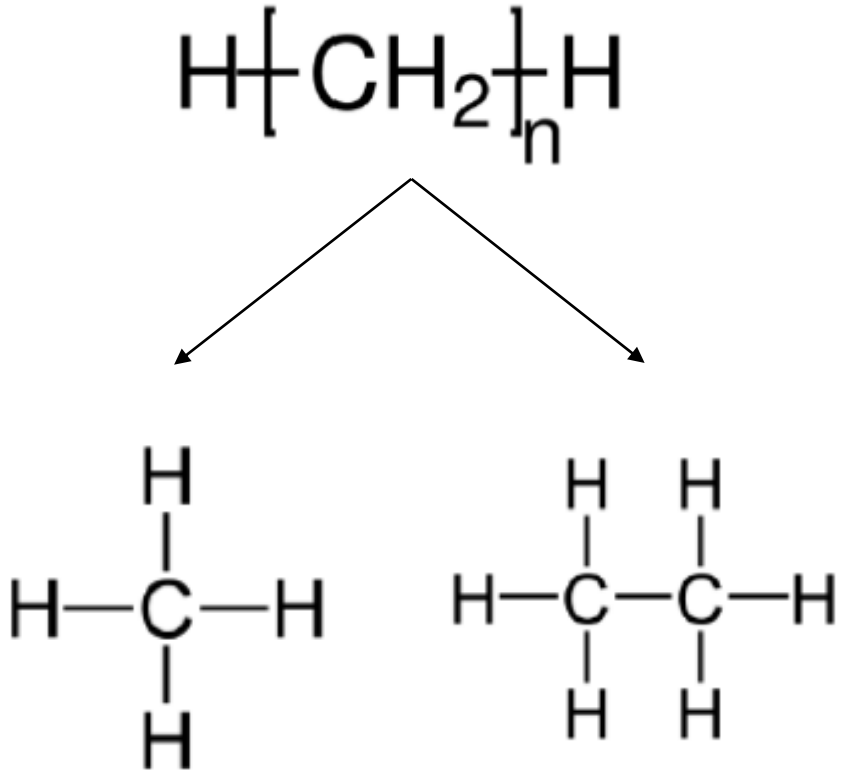
Background signal of the detector



- The detector outputs a minimum intensity independent of the gas analysis
- This signal is called “ground” and results from minimal voltage changes

Chemical background

Alkanes (hydrocarbons)

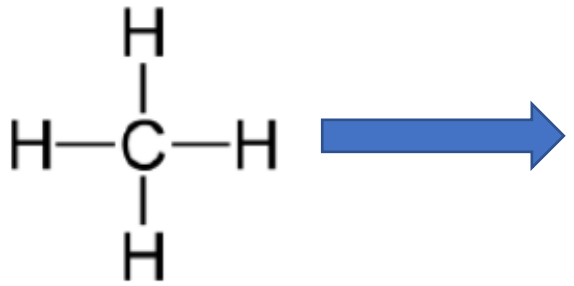


In the following we will have a look at which fragments are produced during ionization and which mass spectrum results from it

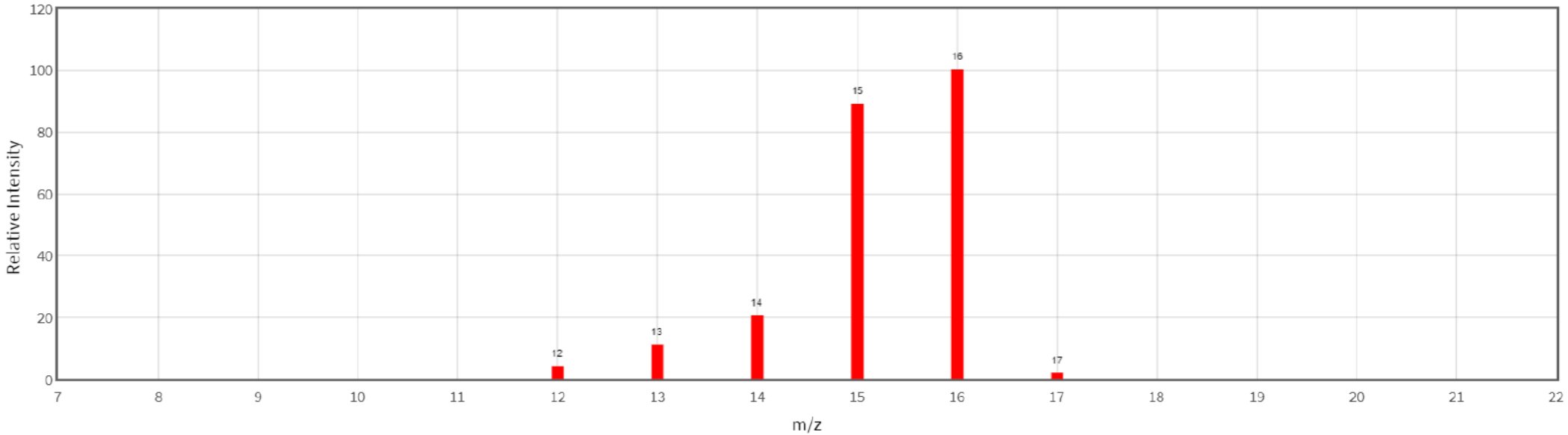
The fragments occur with different probabilities, the most frequently occurring fragment is set to intensity 100% and the others are set in relation to it

Chemical background

Methan



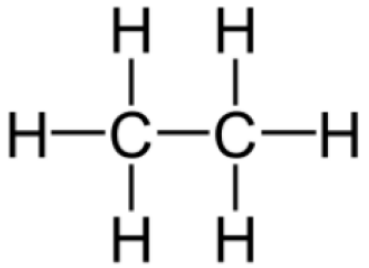
CH_4^+ → 17 amu
CH_4 → 16 amu
CH_3 → 15 amu
CH_2 → 14 amu
CH → 13 amu
C → 12 amu
H_2 → 2 amu
H → 1 amu

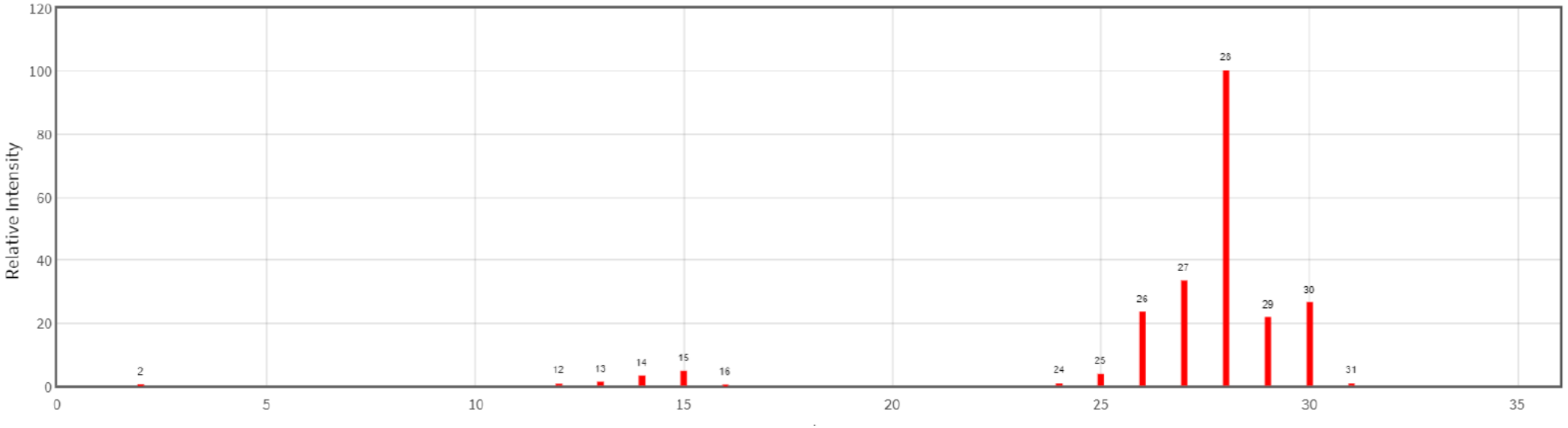


Due to the different probability of the resulting fragments, a unique mass spectrum results like a fingerprint

Chemical background

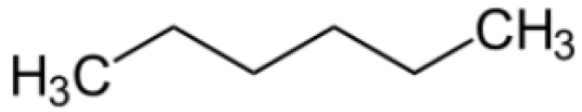
Ethan

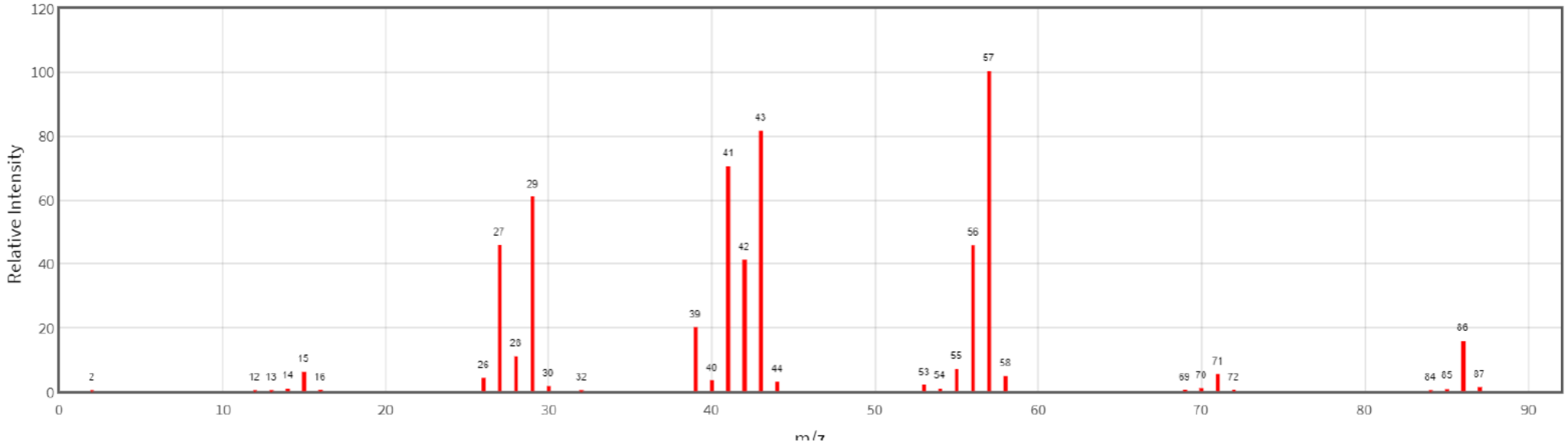




Chemical background

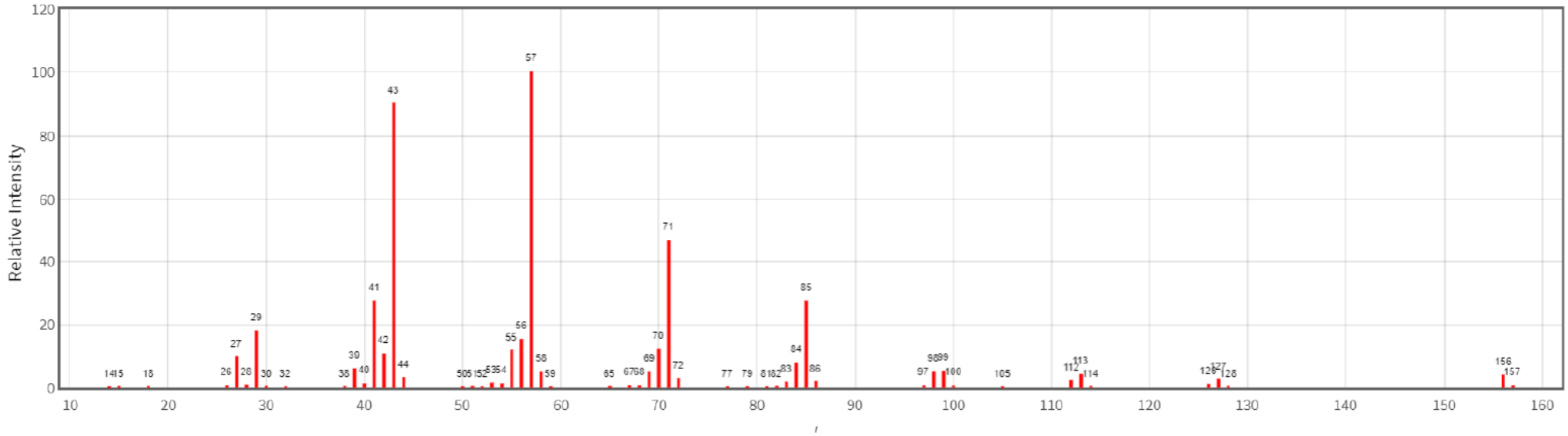
n-Hexan





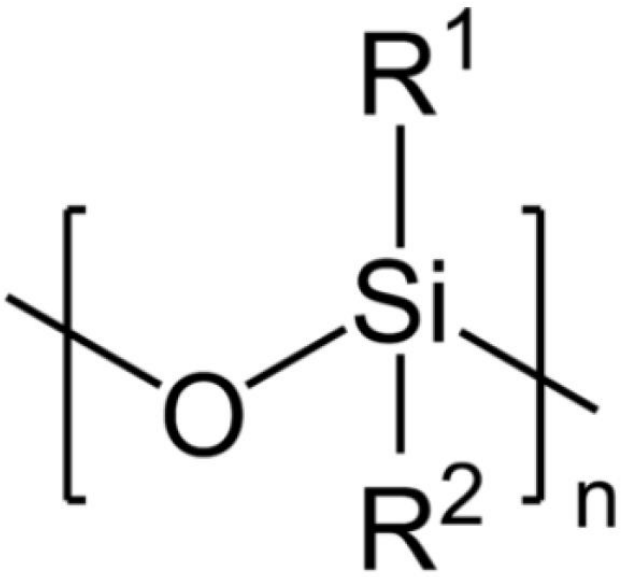
Chemical background

n-Hexan



Chemical background

Silicone oils

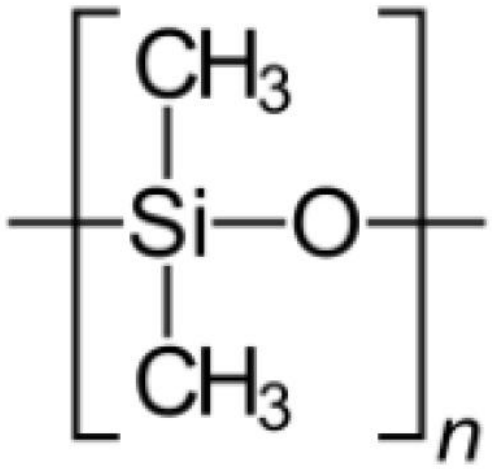


The repeating unit of the siloxane polymer

- Siloxane Polymer
- Silicone oils are clear, colorless, nontoxic, neutral, odorless, tasteless, chemically inert, temperature-stable over a wide range, hydrophobic liquids with a molecular mass of 162 to 150,000 g/mol

Chemical background

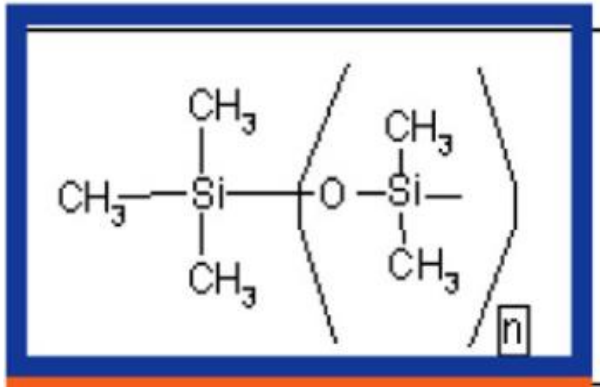
Polydimethylsiloxane (C_2H_6OSi)



Masses

- H – 1
- C – 12
- O – 16
- Si – 28

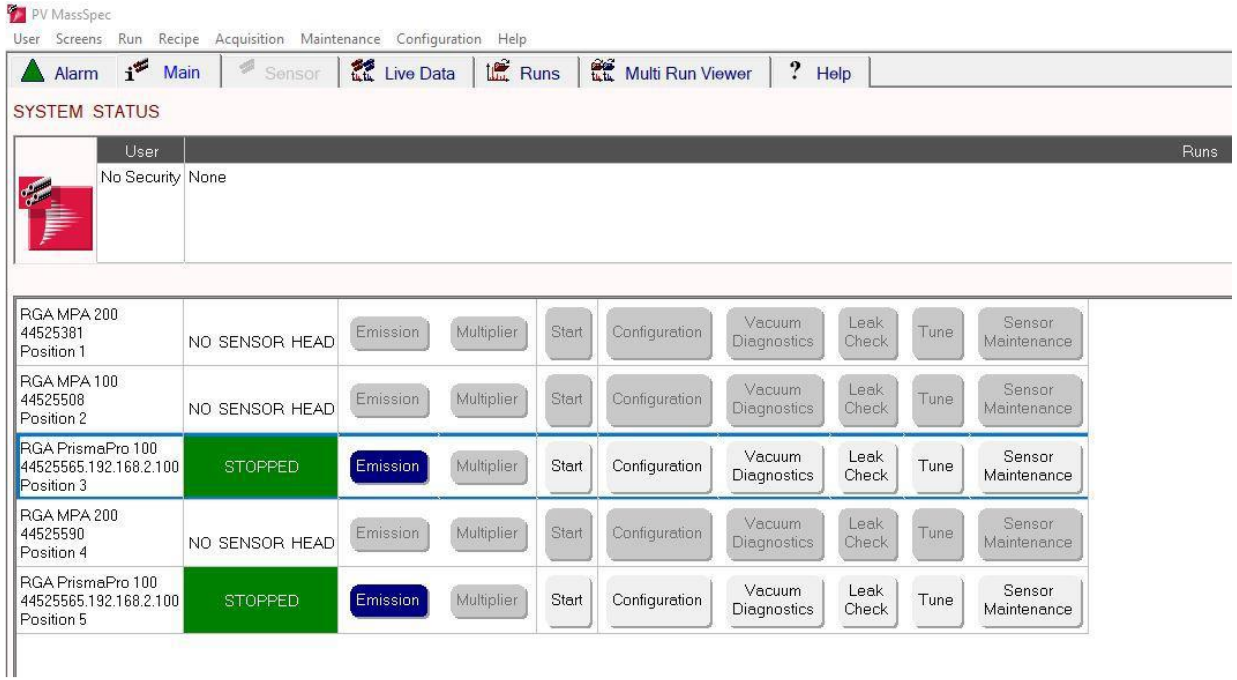
Sum formula: $SiOC_2H_6$



n=0	n=1	n=2
73amu	147amu	221amu
SiC_3H_9	$Si_2OC_5H_{15}$	$Si_3O_2C_7H_{21}$

Software

Pv MassSpec

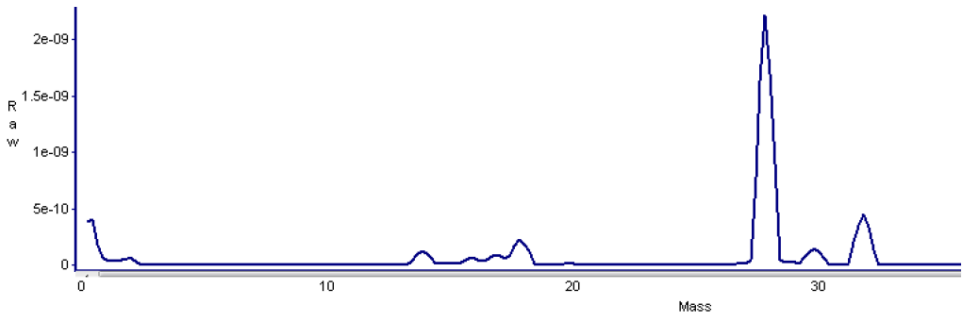


- Recording and evaluation of measurements with one software
- compatible with Windows 10
- manage multiple devices simultaneously
- compatible with PrismaPro (and PrismaPlus

PV MassSpec Measuring Modes

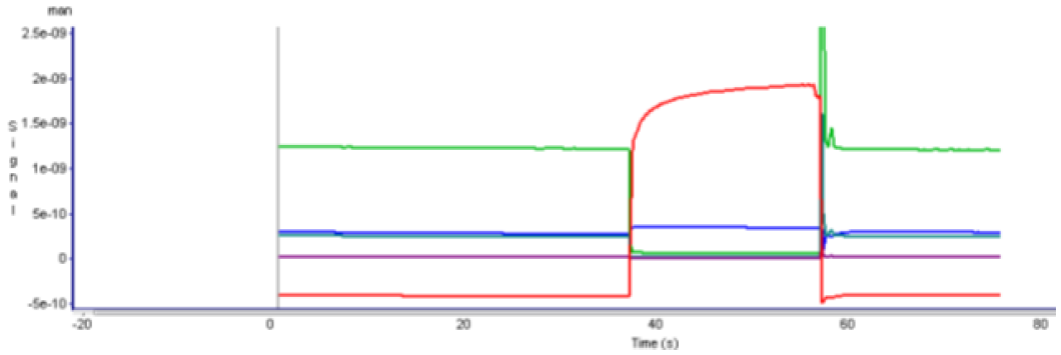
Analog Scan:

- Total mass spectrum
- Mass range selectable



MID Mode:

- Selected masses
- Development over time



Interpretation of the measurement result

Scan of the chamber with silicone oil

- During the scan over all masses, all fragments are detected which are created during the ionization of the air molecules
- If there are traces of evaporated silicone oil in the atmosphere, these fragments are also detected
- The indicators for silicone oil are mainly 45 amu, 59 amu and 73,74,75 amu
- During the scan over all masses, each mass is sampled several times, resulting in parabolic peaks
- The intensity is always relative to the main peak N_2 Intesity = 10
- If the limited sample of silicone oil evaporates, the intensity decreases over time until finally all the oil has evaporated
- In the case of a leak, no decrease in intensity will be seen, as it is not a limited quantity here
- When the chamber is clean, only the fragments of air can be seen