



# Exyte Technology

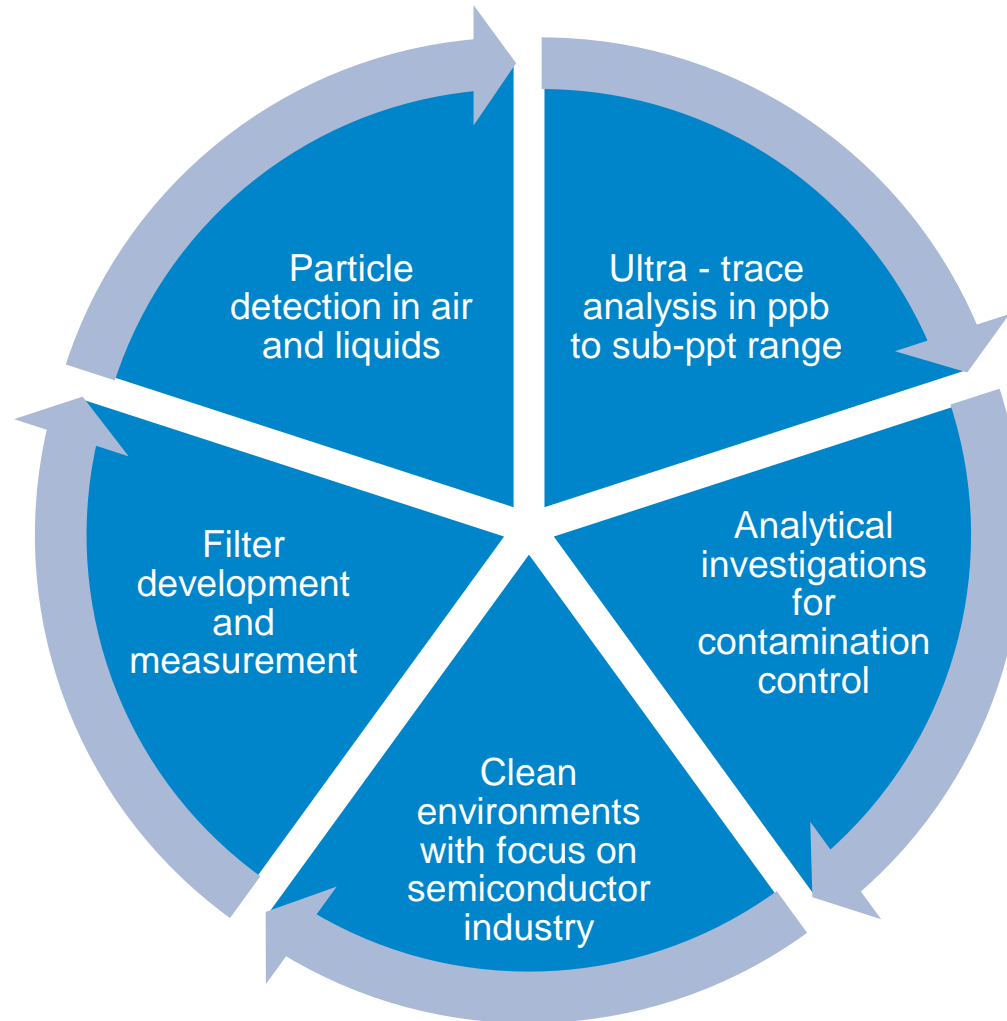
## Controlled Environments

### Trace Analysis

**Exyte Technology GmbH**  
August, 2018

# Our Competences

## Core Competences - Overview



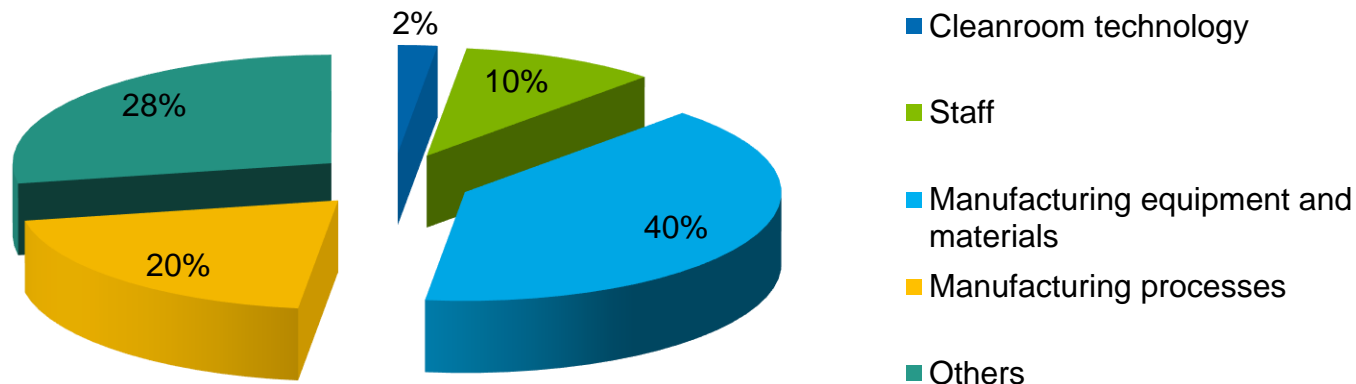
# Measurement and Analysis

## Starting Point

Many industries require clean environmental conditions for optimal product quality, stability of production processes and production yield.

Therefore, cleanroom air, gas supply, materials, components and tools used in minienvironments have to fulfill specified cleanliness levels.

### Influencing Factors upon the Product Cleanliness

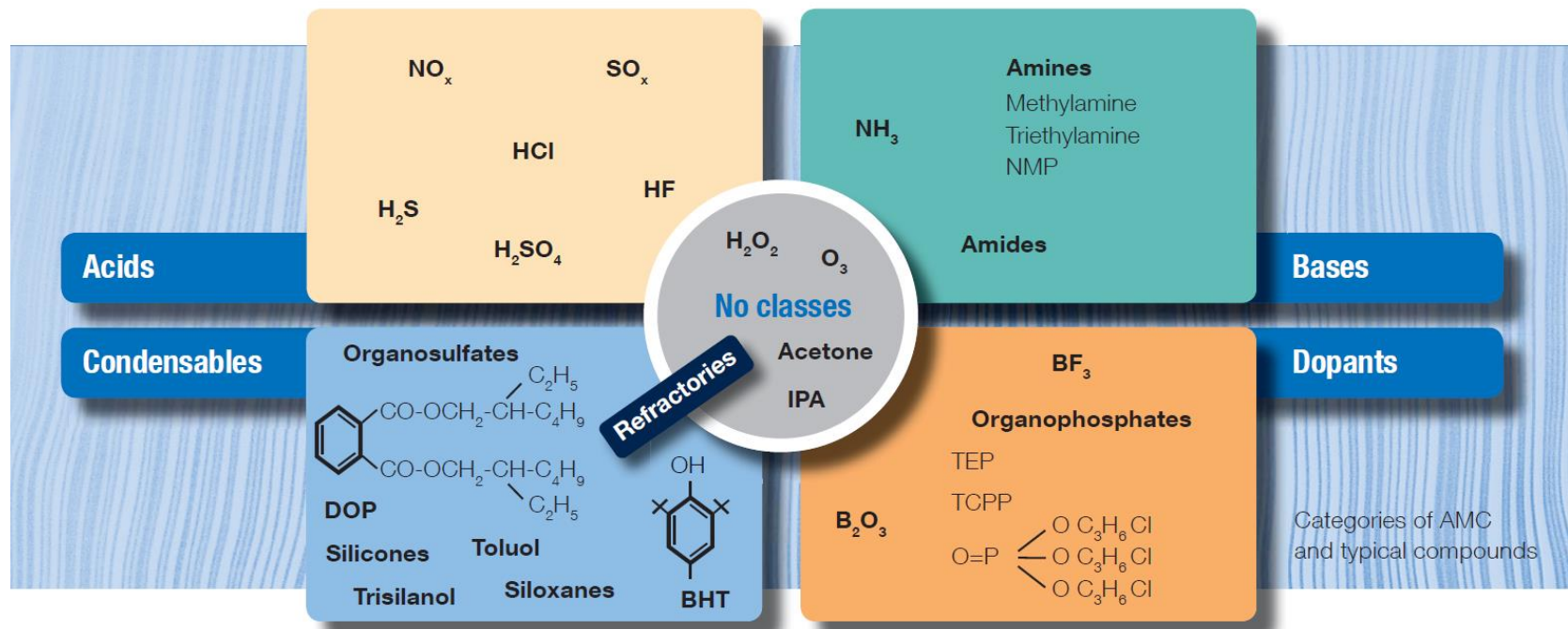


Source: IPA Fraunhofer

# Airborne Molecular Contamination (AMC)

## Definition AMC

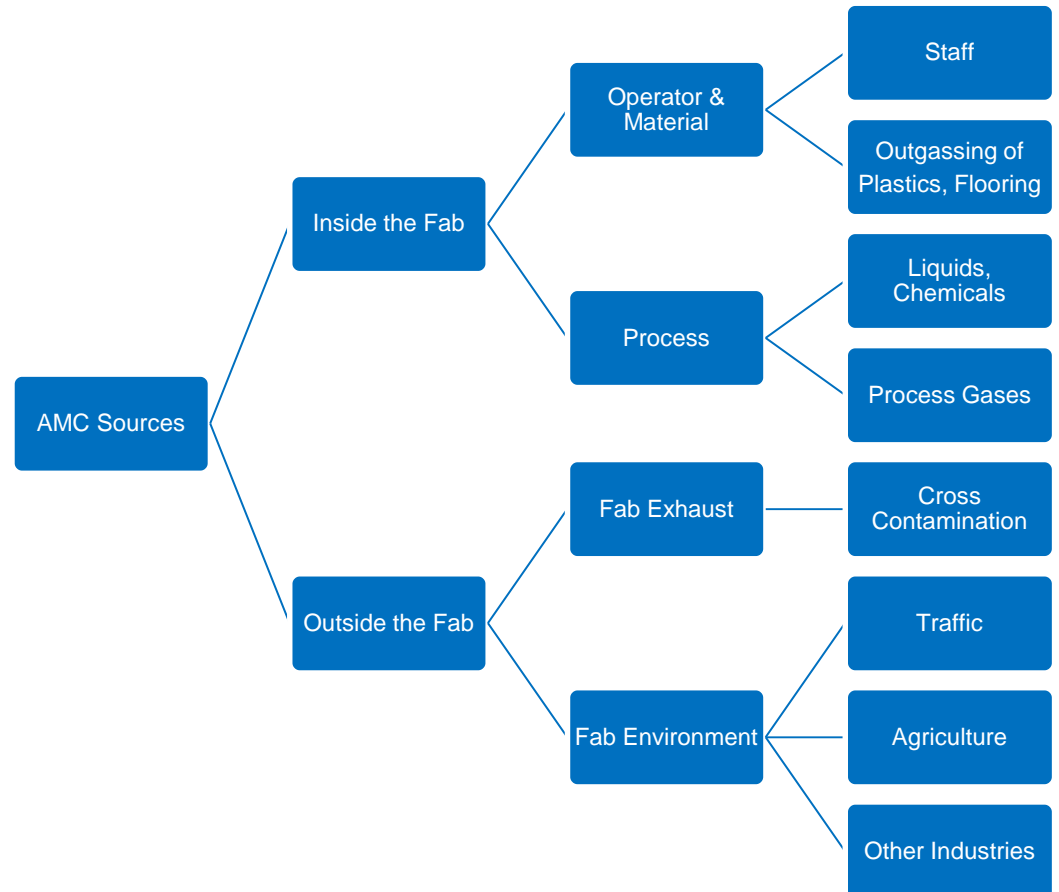
AMC is defined as gas of dust molecules (not particular contamination), present in the atmosphere of a cleanroom or of a controlled environment, which can have a damaging impact of product, process or equipment in cleanroom.  
Definition according to ISO 14644



# Airborne Molecular Contamination (AMC)

## AMC Sources

The reduction of airborne molecular contaminants is a main goal in achieving cleanroom conditions as they can negatively affect manufacturing yields by contaminating wafers, lasers, and other cleanliness sensitive products.



# Our Services

## Portfolio



### Air Quality Monitoring

- AMC Measurement in Air – Qualification of Cleanrooms and Minienvironments
- Material Outgassing and Certification (VOC)
- Quality Control of Gaseous Media
- Laser Certification



### Water Trace Analysis

- Ultrapure Water (UPW) Analysis (Ions, Metals, TOC and Particle)
- Leach Out Tests for Material Testing
- Qualification of UPW Systems and Components (e.g. Tubes, Pipes)



### Filter Technology

- Testing and Development of Filter Materials for Air Filters
- Outgassing Test for AMC- and HEPA/ULPA Filters
- Determination of removal efficiency, filter capacity and remaining life time
- High precision gas dosing with online measurement of gas concentration

# Techniques – TDS-GC/MS

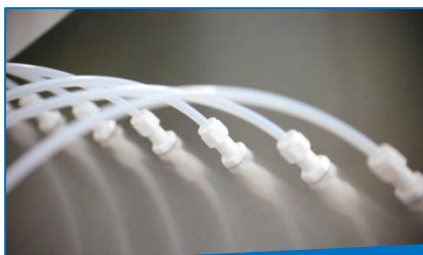
## Trace Analysis



Thermodesorption System



Gas Chromatograph



Sampling Points

**Thermal Desorption Gas Chromatography coupled with Mass Spectrometry (TDS-GC/MS):** Method for identification and quantification of a wide range of volatile and semi-volatile organic compounds in complex chemical environments

### Trace Organic Analysis

- Sub-ppb gas chromatography
- Controlled environment area
  - ISO Class 6.0 for particle contamination

### Equipment

#### 2 Gas Chromatography systems

- Agilent gas chromatograph 7890A and 7890B coupled with mass spectrometer 5975C and 5977A resp.
- Thermodesorption system GERSTEL TDS3
- with Autosampler TDSA2
- Adsorbents: TENAX, CARBO

**Applications:** Evaluation of material and component organic compounds (VOC+SVOC) according to Exyte guidelines and VDI 2083-17 standard; product certification of VOC critical products; air and gas sampling and analysis



# Techniques – TDS-GC/MS

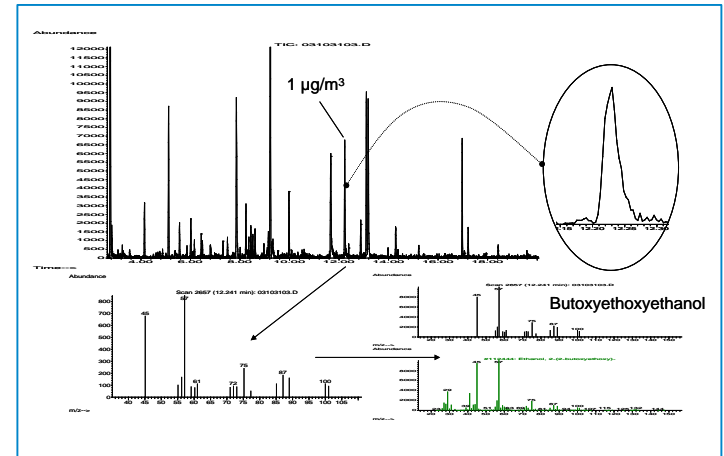
## VOC – Offline Sampling + GC/MS Measurement



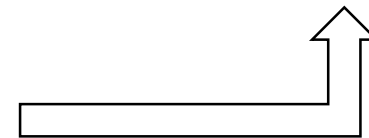
Offline Sampling



TDS Desorption  
GC Separation  
MS Identification



Evaluation

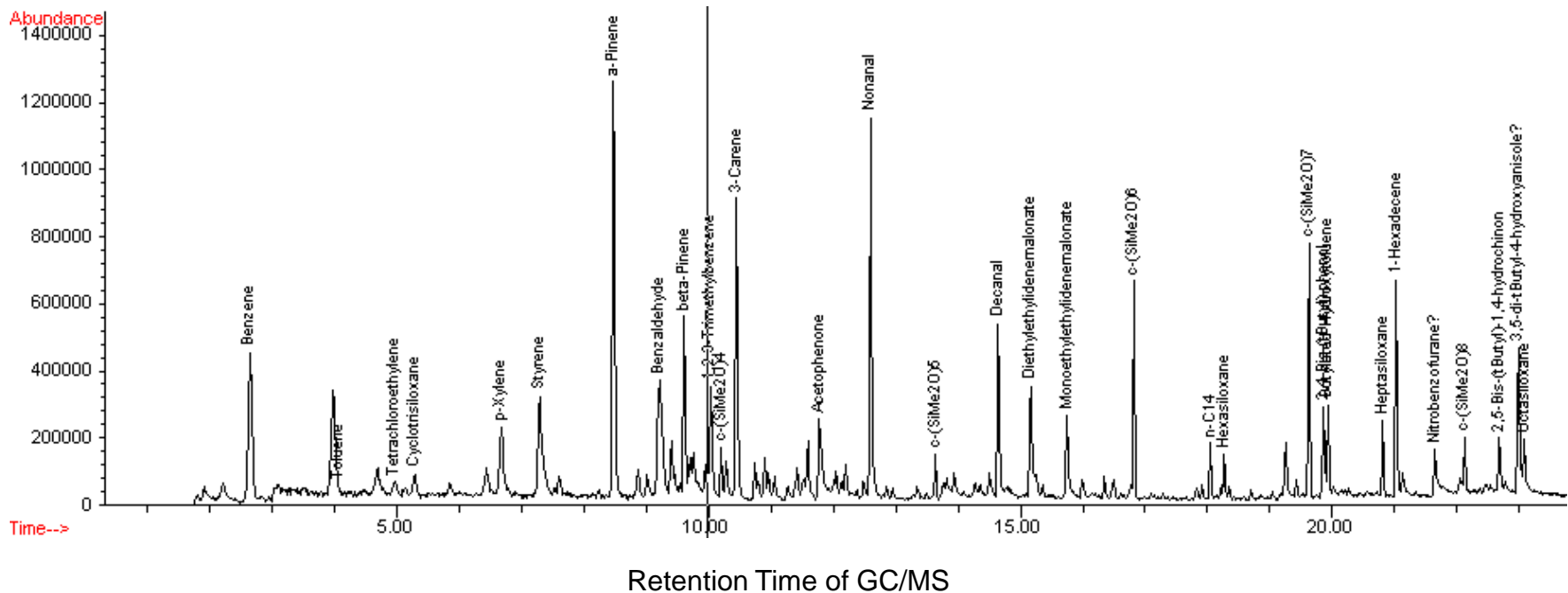




# Qualification and Quality Monitoring

## Production Environments

- Typical GC/MS chromatogram of a cleanroom sample with the identification of every major compound. The gas chromatograph separates the different species while the mass spectrometry allows the unambiguous assignment of a certain molecule.
- The identification of every compound **enables a root course analysis** to locate and eliminate AMC sources.



# Techniques - ICP-MS

## Trace Analysis

### **Inductively Coupled Plasma Mass Spectrometry (ICP-MS):**

Elemental analysis technique for quantitative trace element analysis. Detection of almost all elements of the periodic table.



Autosampler



ICP-MS measurement Device

### **Equipment**

- Perkin Elmer ELAN DRC II

### **Specification**

- Trace elements and dopants: Al, As, B, Ba, Cd, Co, Cr, Cu, Fe, Mn, Ni, Pb, Sn, Sr, Ti, Zn, V (additional elements on request)
- Typical limits of quantitation (LOQs): 1-10 ng/L

### **Application**

- Ultrapure water analysis
- Pre-qualification of ultrahigh purity (UHP) polymer materials (tubes, hoses, membranes etc.) for UPW distribution systems by static or dynamic leach out tests
- Evaluation of water and material testing according to ITRS, SEMI and customer requirements
- Validating of ion exchange resins and filter systems used for UPW systems

## Trace Analysis

**Ion Chromatography (IC):** Method for analyzing anions, cations and other polar substances in aqueous media. A specific instrument design allows the simultaneous analysis of anions and cations from the same sample. Using a specific pre-concentration technique enables ultra-trace analysis down to the lower ng/L-range.

### Trace Ionic Analytical

- ng/L ion chromatography
- UPW facility
- **Controlled environment area**
  - ISO Class 5.0 for particle contamination
  - AMC filtration for Bases, Ammonia level  $<1 \mu\text{g}/\text{m}^3$
  - Controlled airflow at sample handling area



Impinger for Air Sampling



Leach out Test

### Equipment

- **Ion Chromatograph (Dionex ICS 3000)**
  - Cations and Anions systems with conductivity detector
- **Detection Limit**
  - Impinger method for AMC measurement (offline) with LOQ  $< 100 \text{ ng/L}$
  - UPW method (offline) LOQ  $1\text{-}5 \text{ ng/L}$

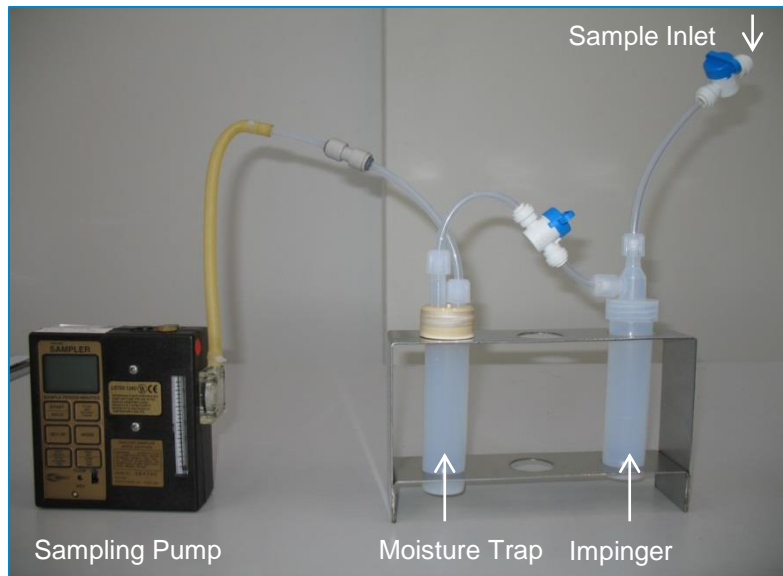
### Following Ions can be determined

- Cations  
( $\text{Li}^+$ ,  $\text{Na}^+$ ,  $\text{NH}_4^+$ ,  $\text{K}^+$ ,  $\text{Mg}^{2+}$ ,  $\text{Ca}^{2+}$ )
- Anions  
( $\text{F}^-$ ,  $\text{Cl}^-$ ,  $\text{NO}_2^-$ ,  $\text{Br}^-$ ,  $\text{NO}_3^-$ ,  $\text{ClO}_3^-$ ,  $\text{SO}_4^{2-}$ ,  $\text{PO}_4^{3-}$ )
- Organic acids (Acetate, Formate, Propionate, Oxalate and Phthalate)

# Techniques - IC

## AMC – Offline Sampling + IC Analysis

Offline Sampling



IC Analysis



# Techniques - TOC

## Trace Analysis

**Total Organic Carbon (TOC):** Continuous monitoring of organics in UPW down to a TOC concentration of 0.1 ppb. Individual samples, e.g. aqueous extracts, can be filled in sample vials and analyzed in a special offline mode.



### Equipment

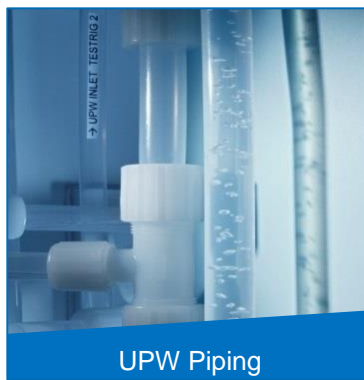
- GE Sievers 500 RLe On-line TOC Analyzer

### Specification

- Measurement range 0.1 - 2,500 ppb

### Application

- UPW analysis
- Qualification of UPW systems
- Qualification of the water quality within the UPW infrastructure
- Pre-qualification of ultrahigh purity (UHP) polymer materials (tubes, hoses, membranes etc.) for UPW distribution systems by static or dynamic leach out tests
- Evaluation of water and material testing according to ITRS, SEMI and customer requirements



# Techniques - Online AMC Measurement

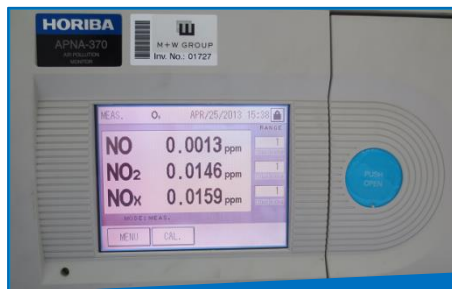
## Online analyzers

Online analyzers are generally used for analyzing gaseous media. They enable fast and continuous analysis with real-time response of airborne molecular contamination and provide results with high accuracy and precision.

Substances of Interest	Analysis Technique	Measuring Range
SO <sub>2</sub> / H <sub>2</sub> S / DMS	UV Fluorescence	0.5 - 10,000 ppb
NO <sub>2</sub> / NO <sub>x</sub>	Chemiluminescence detector	
NH <sub>3</sub>		
Ozone	UV Absorption	
Organic compounds	FID	0 - 10,000 mg C / m <sup>3</sup>

# Techniques - Online AMC Measurement

## Online analyzers



Measurement Device for AMC

### Equipment

- 5 x HORIBA APSA 360
- 3 x HORIBA APNH3 360
- 1 x HORIBA APNA 370
- 1 x HORIBA APOA 370

### Application

Determination of specific AMC compounds (SO<sub>2</sub>/H<sub>2</sub>S/DMS/NH<sub>3</sub>/NO/NO<sub>2</sub>/NO<sub>x</sub>/O<sub>3</sub>) in air and gases

- Filter material development
- Filter testing

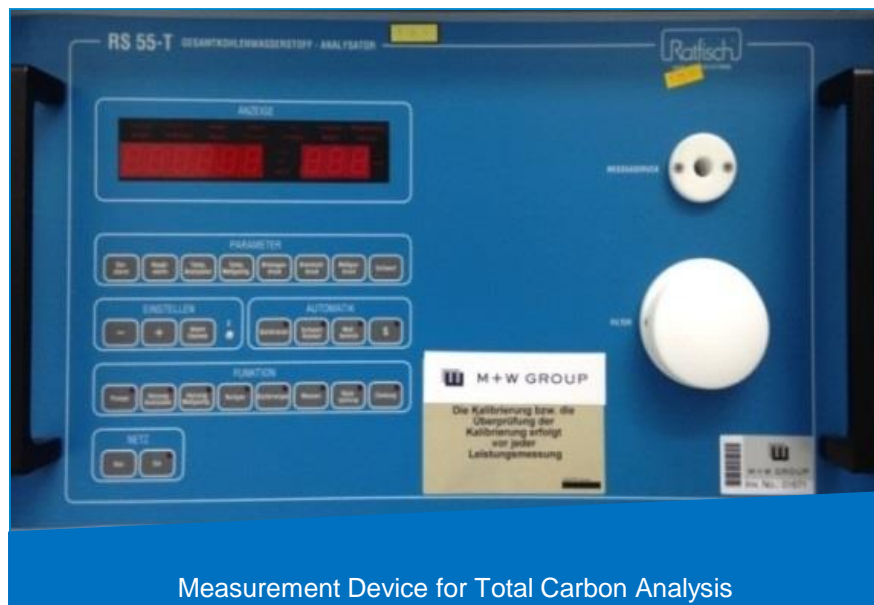
### Specification

Equipment	Type	Range
HORIBA APSA 360	UV Fluorescence detector for SO <sub>2</sub> /H <sub>2</sub> S	0-100 ppb
HORIBA APSA 360	UV Fluorescence detector for SO <sub>2</sub> /H <sub>2</sub> S/DMS	0-10,000 ppb
HORIBA APNH3 360	Chemiluminescence detector for NH <sub>3</sub>	0-10,000 ppb
HORIBA APNA 370	Chemiluminescence detector for NO/NO <sub>2</sub> /NO <sub>x</sub>	0-1,000 ppb
HORIBA APOA 370	UV Absorption for O <sub>3</sub>	0-10,000 ppb



# Techniques - Online AMC Measurement

## Online analyzers



Measurement Device for Total Carbon Analysis

### Equipment

- 3 x Ratfish RS 55-T  
(Flame ionization detector)

### Specification

- Measurement range: 0 - 1,000 mg C/m<sup>3</sup>
- Measurement range: 0 - 10,000 mg C/m<sup>3</sup>

### Application

- Determination of total carbon concentration in air and gases
- Filter material development
- Filter testing

# Techniques - Particle Measurement

## Analysis of Contamination

### Particle Measurement:

Measuring the particle concentration in air and water (particle measurements in air or water can count and size particles)

Analysis	Analysis Medium	Measuring Range
Particles	UPW	$\geq 0.05\mu\text{m}$
Particles	Air	$\geq 0.1\mu\text{m}$

### Equipment

- PMS Lasair II-110 (for measurements in air)
- PMS LS 11 (for measurements in air)
- PMS LS 21 (for measurements in air)

### Specification

- particle size:  $> 0.1 \mu\text{m}$

### Application

- Cleanroom qualification
- Filter testing

### Equipment

- PMS Ultra DI 50  
(for measurements in water)

### Specification

- particle size:  $0.05 - 0.20 \mu\text{m}$

### Application

- Ultrapure water analysis
- Qualification of UPW systems and components

# Ultrapure Water Analysis

## Analysis of Contamination



UPW Distribution System



Piping Line with Valves



Sampling Bottles

### Analysis of contamination in Ultrapure water (UPW)

- Qualification of the water quality within the UPW infrastructure
- Quality monitoring of UPW systems to ensure the performance criteria
- Performance check of systems for media conditioning
- Test of water quality in process tools/ minienvironments for UPW conditioning

### Analysis of UPW

- Analysis parameter: anions and cations, metals und dopants, TOC, bacteria, particle count
- Measurements of particles > 50 nm
- Determination of water resistivity, bacteria and silica loading
- Conformity evaluation according to ITRS and customer specifications

### Equipment

- IC(ppt), ICP-MS (ppt), TOC-Analyzer (ppb), microbiological rapid test

# Ultrapure Water Analysis

## Leaching Test of Polymer Materials

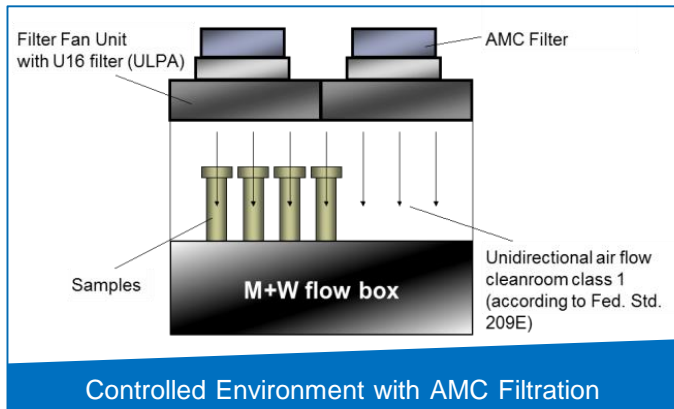


### Performance Evaluation of UHP Polymer Materials for UPW Distribution Systems

- Static leaching tests of UHP polymer materials according to SEMI F40-0699
- Analysis of tubing, piping, valves, fittings, gaskets, membranes
- Pre-cleaning of polymer test samples
- Sample extraction with ultrapure water
- Analysis parameter: anions, cations, metals incl. B, TOC, Particles
- **Equipment:** Ion chromatography (ppt), ICP-MS (ppt), TOC Analyzer (ppb)
- Evaluation according to SEMI specifications F57-0312
- Controlled environment with AMC filtration
- Complete preparation process, flushing, filling and the measuring process, within class ISO 3.0 cleanroom conditions (according to DIN EN ISO 14644, part1)

# Ultrapure Water Analysis

## Leaching Test of Polymer Materials



### Performance validation of UHP polymer materials

- Pre-cleaning of the polymer test sample
- Sample filled with UPW
- After the defined leaching time (7 days at 85 degrees [ $\pm 5$  degrees]) the UPW is analyzed
- Analysis parameter: anions, cations, metals incl. B, TOC, particles
- All measurements are executed and performed in cleanroom
- Installation of AMC filtration systems within the testing area
- Complete preparation process, flushing, filling and the measuring process, within class ISO 5.0 cleanroom conditions (according to DIN EN ISO 14644, part1)



Handling within ISO 5.0 Cleanroom Conditions

# Quality Control of Gaseous Media

## Monitoring and Analysis



Gas Mixing System



Gas Distribution System

### Scope of application

- Pure gases are used within many fabrication processes (e.g. semiconductor, LED, flat panel and optical industry)
- Application for purging gases for cleaning and setting up inert atmospheres, dilution and carrier gas, reaction gases and pneumatic drives

### Laboratory services – monitoring and analysis of high purity gases:

- Analysis of clean dry air (CDA), N<sub>2</sub>, He, Ar, CO<sub>2</sub> and others
- Advanced sampling procedure to handle higher pressure and flow
- Sample collection by impinger method for ion chromatography (IC) and ICP-MS analysis
- Sample collection of organic compounds and refractories by sampling with adsorption materials (e.g. Tenax, CarboTrap) and TD-GC/MS analysis



# Filter Technology

## Chemical Filter Testing & Development



Media Test Rig



Specimen



Filter Test

Filter test rigs and air handling units with precise air conditioning for filter tests.

### The filter testing program provides results about:

- Advanced sampling procedure to handle higher pressure and flow
- Determination of removal efficiency
- Filter capacity
- Remaining life time test after a time period of using within its life time cycle
- Filter stability
- Outgassing behavior of filter materials and components

### Parameters

- Standard test condition:
  - temp.  $22\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ ; relative humidity  $45\text{ \%} \pm 5\text{ \%}$
- Dosage of test substances for simulating relevant cleanroom contaminations (e.g.  $\text{SO}_2$ ,  $\text{H}_2\text{S}$ ,  $\text{NH}_3$ ,  $\text{NO}_x$ , Toluene, Ozone,...)



# Filter Technology

## Testing & Development



Media Test Room



Outgassing Test Room

### Online measurement devices and offline sampling

(Impinger method and adsorbent tubes)

#### Online measurement of:

- Gas concentrations ( $\text{NH}_3$ ,  $\text{SO}_2$ ,  $\text{H}_2\text{S}$ ,  $\text{NO}_x$ ,  $\text{O}_3$  and hydrocarbons)
- Temperature, humidity, airflow rate
- Pressure drop

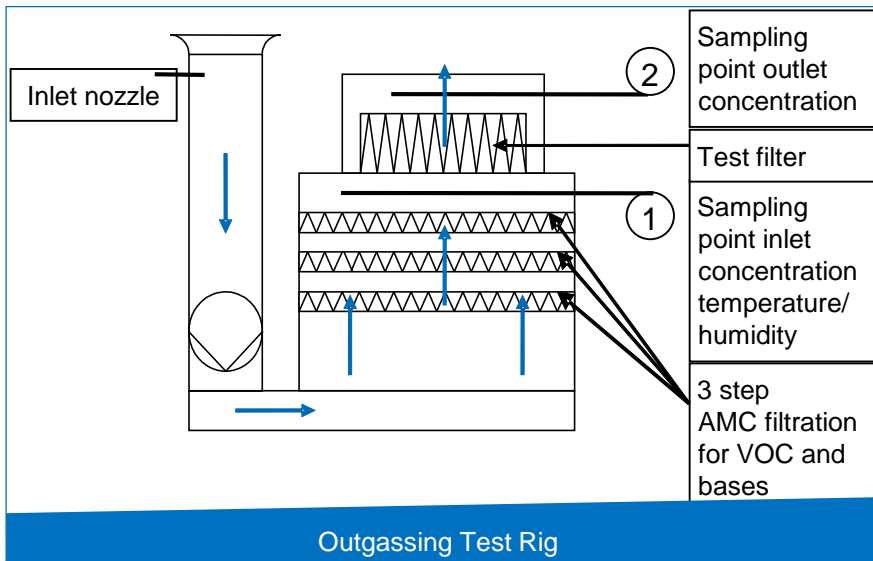
#### High precision gas dosing in ppb and ppm range for:

- $\text{H}_2\text{S}$ , DMS,  $\text{SO}_2$ ,  $\text{NH}_3$ ,  $\text{NO}_x$ , HCl, ozone and others
- Organic compounds like toluene, xylene, HMDSO, PGMEA, IPA and others

#### Testing of several filter materials:

- Modified charcoal
- Ion exchange resins
- Modified inorganic adsorbent materials and others

## Outgassing Test for AMC- and HEPA/ULPA Filters



**Sampling and analysis** of inlet and outlet concentration are performed for anions, cations, VOCs and dopants

**Pre-filtration** necessary to reach very low baseline concentrations:

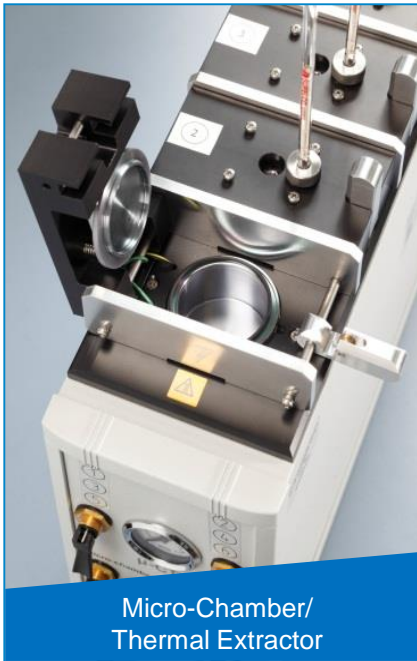
- Anions  $< 0.05 \mu\text{g}/\text{m}^3$
- Cations  $< 0.05 \mu\text{g}/\text{m}^3$
- VOC  $< 1.0 \mu\text{g}/\text{m}^3$
- Boron  $< 0.05 \mu\text{g}/\text{m}^3$

### Sampling conditions

- Max. sample size: 1,200 mm × 1,200 mm
- Air flow rate: 1,000 – 2,500 Nm<sup>3</sup>/h (according to specification)
- Temperature: 22°C
- Humidity: 45 %

# Outgassing of Materials

## Standards and Procedures



### Outgassing test of cleanroom materials

#### Exyte standard procedure

- VOC emissions from materials
- Certification of cleanroom materials (vendor qualification)
- Testing of technical components used in minienvironments (e.g. sealing materials, adhesives)
- Sampling with test setup according to Exyte procedure (90 °C, 3h)
- TD-GC/MS

### Outgassing tests according to CSM standards

#### Method according to VDI 2083 part 17

- Industry association managed by Fraunhofer IPA
- Sampling with a micro test chamber
- Testing procedure at room temperature (22 °C)
- Evaluation of TVOC ( $\geq C_6 \dots \leq C_{16}$ )
- Material-specific ISO class ISO-AMC<sub>m</sub> for VOC (=  $\lg[\text{TVOC}_{\text{norm}} \text{ g/m}^3]$ , based on  $V=1 \text{ m}^3$ ,  $A=1 \text{ m}^2$ ,  $n=1/\text{s}$ )

# Outgassing of Materials

## Evaluation

### Outgassing Test of Cleanroom Materials

**Evaluation** of the results according to Exyte acceptance criteria for cleanrooms

- **Evaluation parameters**
  - (1) Total Organics ( $\Sigma$ TVOC + SVOC)
  - (2) SVOC: high boilers >C16, like siloxanes and phthalates
  - (3) Amines
  - (4) Organophosphorous compounds
- Mainly relevant for Exyte applications
- Exyte data base of tested cleanroom suitable materials
- According to this test, Exyte guarantees the AMC class of its cleanrooms

Exyte specification	Air contacted area covered by the tested material (in relation to net cleanroom area)		
	> 50%	5 to 50 %	< 5%
$\Sigma$ (TVOCs + SVOCs) ( $\mu\text{g/g}$ )	1000	2000	10000
SVOCs ( $\mu\text{g/g}$ )	250	500	2500
Amines ( $\mu\text{g/g}$ )	150	300	1500
Organophosphates ( $\mu\text{g/g}$ )	10	10	10

# Outgassing of Materials

## Micro-Emission Chamber



Micro-Chamber/  
Thermal Extractor

### Micro-emission chamber

- Versatile and compact device especially designed for outgassing tests for construction materials (e.g. molding materials, resins, seals, coatings, paints)
- Fast emissions screening as part of routine industrial quality control complying with international standard methods (e.g. ISO 12219-3)
- Procedure: Material samples are heated at a certain temperature and the released chemicals (VOCs) are collected on thermo-desorption tubes and analyzed by TD-GC/MS. This enables the prequalification of materials for optical and laser devices.

# Contamination by Outgassing within Cleanrooms



## Product Control for Optical and Laser Devices

### **Optoelectronic products (LEDs) and laser systems**

- Contamination of optics due to outgassing of organic compounds, especially condensable compounds
- Sources of organic outgassing: seals, circuit boards, cable insulation, cleaning agents, coatings, resins, plasticizers, and others
- Routine product control

### **Screening analysis of organic compounds by TD-GC/MS**

- Analysis of total VOC
- Evaluation of low-, medium- and high-boiling organic compounds
- Separate determination of amines and siloxanes
- Identifying by MS library search and quantifying of the 20 most abundant peaks
- Issuance of product certificate

# Contamination by Outgassing within Cleanrooms

## Product and Component Emission Tests

### Certification of VOC critical products and components

- VOC emission test (similar to outgassing test)
- Especially laser certification for several external customers
- Component tests for product development (vendor qualification)
- Method according to customer specifications
- Sample taking by customers using special test chambers
- Tests performed at room temperature
- CDA is used as flushing gas

exyte

# Material Outgassing Certificate

Hereby we confirm that the

Coating material

Manufactured by

John Doe Ltd.  
123 Anywhere Street  
Anytown, State 12345

was tested according to M+W Products standard outgassing procedure and fulfills the M+W Products specifications for semiconductor cleanrooms for above 50% coverage of the net cleanroom area.  
This certificate is valid for a period of three years from date of issue.

Outgassing Results

Σ (VOCs+SVOCs)	[µg/g]	9.4
TVOCs	[µg/g]	6.1
SVOCs	[µg/g]	3.3
Amines	[µg/g]	< 0.1
Organophosphates	[µg/g]	< 0.1

Detailed information can be obtained from M+W Products test report number 131154aa.  
November 29, 2013

Executed by

Approved by

Rubén López-Trigo

Dr. Michael Franzke

 CERT  
M+W Products GmbH · Lotzbergstraße 30 · 70499 Stuttgart · Germany · [www.mwgroup.net](http://www.mwgroup.net)





Thank you for your attention!

**Exyte Technology GmbH**  
Analysis Services  
Rosine-Starz-Straße 2-4  
71272 Renningen  
Germany

Telefon: +49 711 8804 8000  
[info@exyte-technology.net](mailto:info@exyte-technology.net)