Solutions and Innovations for TOC and VOC Measurements regarding Cleanliness Grades in the High-Tech and Semiconductor Industry

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Clean Event 2023
Veldhoven

Who am I?

- Expert in GC&MS methods
- Visiting professor UGent
- Lean black belt

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Who is Interscience?

- GC&MS instruments
- Sample preparation
- Hands-on training

www.interscience.nl
www.is-x.com

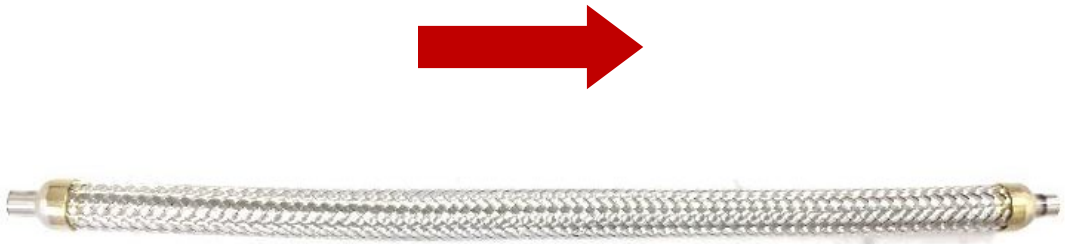
CLEANLINESS TESTING

Assessment of product emission levels

- Routine material emission testing
- Non-routine material emission testing
- Cleanroom air analysis

ROUTINE MATERIAL EMISSION TESTING

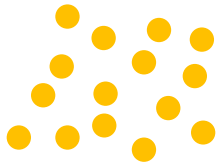
GSA 07 9710

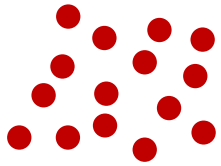


High purity input gas

‘Contaminated’ output gas

ROUTINE MATERIAL EMISSION TESTING





TOC Volatile
TOC_v

TOC Non-Volatile
TOC_{nv}

BP < 150 °C
MW < 120 g/mol

BP > 150 °C
MW > 120 g/mol

ROUTINE MATERIAL EMISSION TESTING

Preferred technique: TD-GC/FID

- Sampling: Microchamber extraction
- Tube: Tenax/graphitized carbon
- Analysis: Thermal desorption
- Quantification: Toluene in nitrogen (3-point calcurve)
- Reportable parameters: TOC_v and TOC_{nv}

ROUTINE MATERIAL EMISSION TESTING

❶ Sampling: microchamber

- Standard method (ISO, ASTM)
- Sample placed in small furnace
- Temperature: 25 – 250°C
- Emissions flushed to tube with nitrogen

ROUTINE MATERIAL EMISSION TESTING

❷ Trapping: adsorbent tube

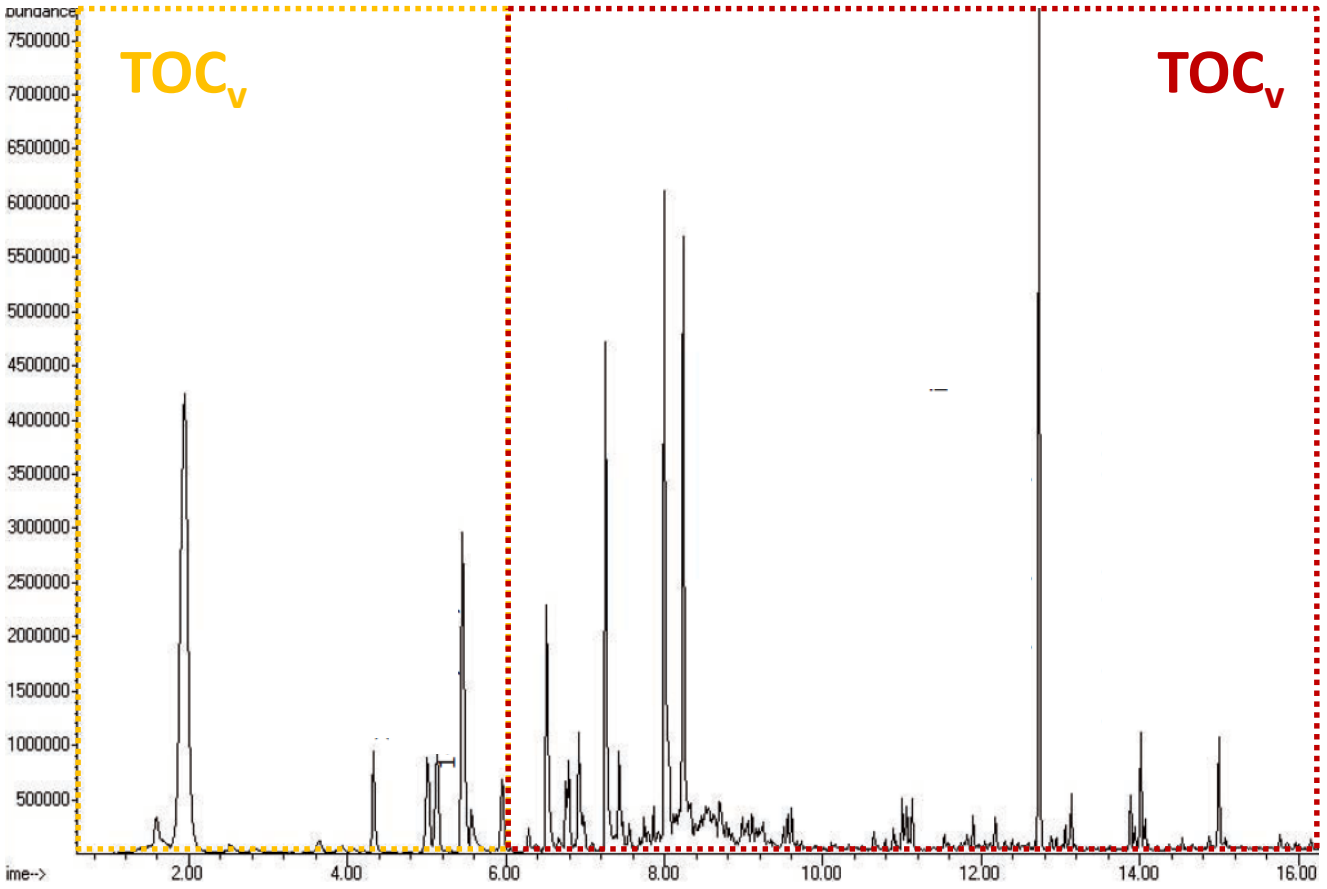
- Tenax/graphitized carbon
  - Tenax = TOC_{nv}
  - Graphitized carbon = TOC_{v}
- RFID tag for traceability

ROUTINE MATERIAL EMISSION TESTING

❸ Analysis: thermal desorption

- Reversed flush desorption, 250°C
- Component refocusing, > 0°C
- Flash injection to GC

ROUTINE MATERIAL EMISSION TESTING



NON-ROUTINE MATERIAL EMISSION TESTING

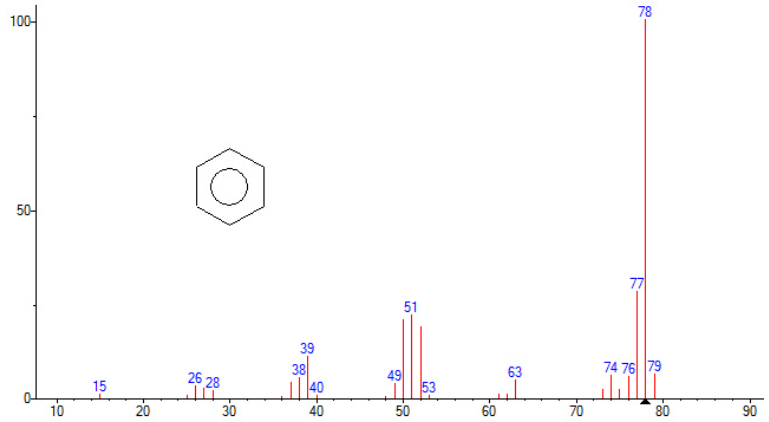
- Identification of unknown components
- Evaluation of new materials
- Troubleshooting QC issues

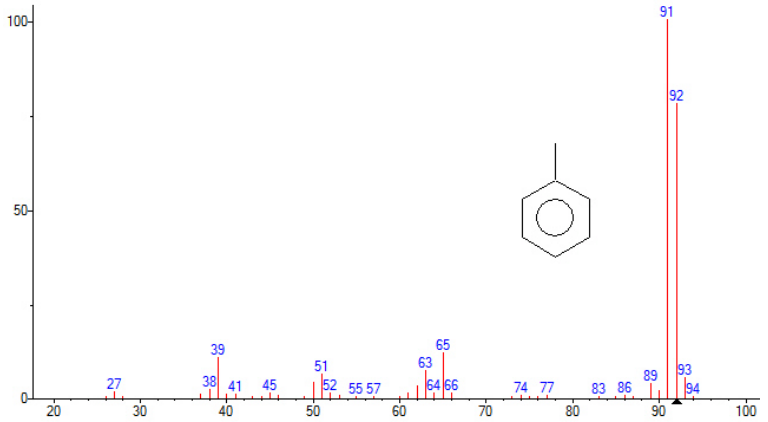
Preferred technique: TD-GC/MS

NON-ROUTINE MATERIAL EMISSION TESTING

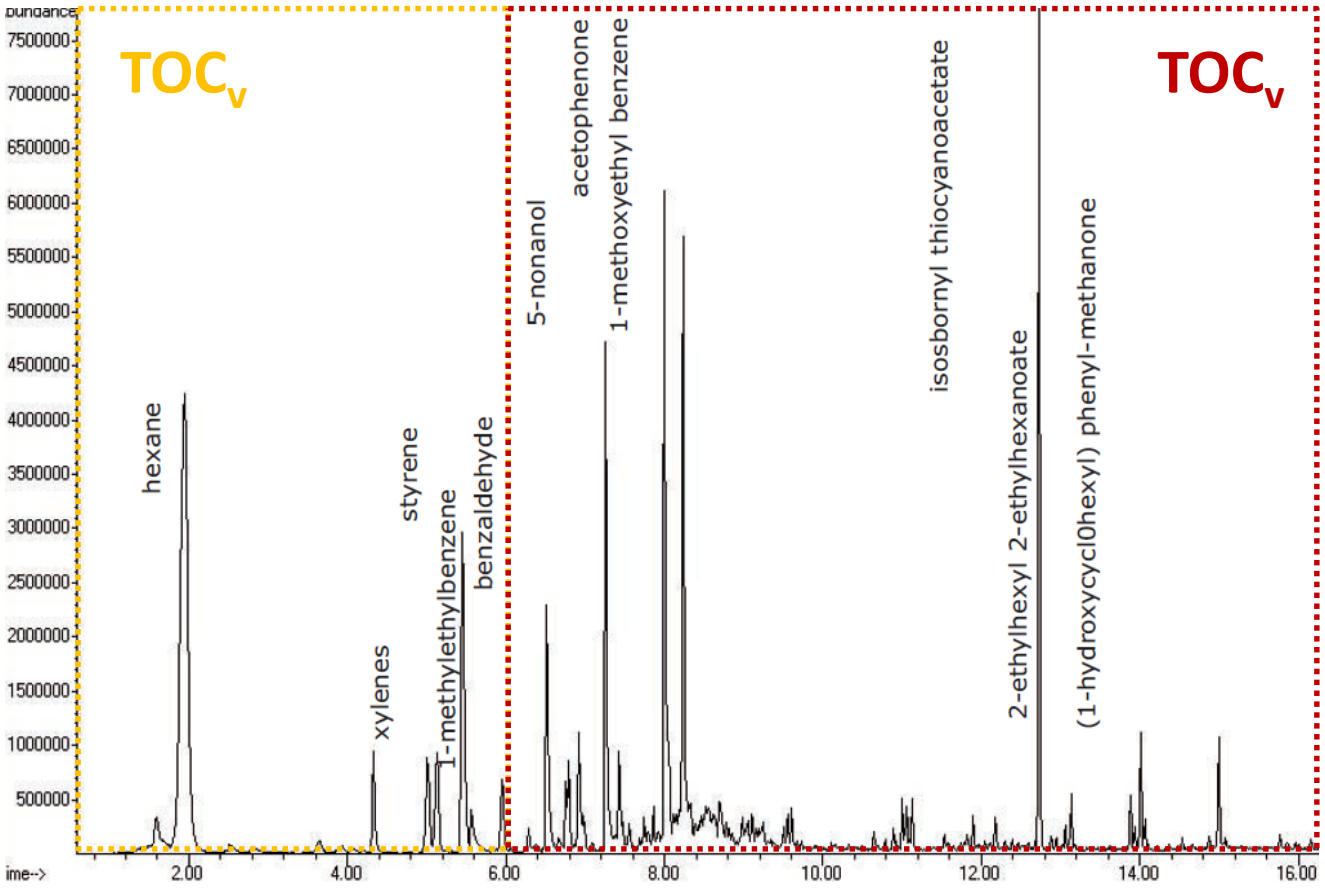
Why GC/MS?

- Identification of unknown components based on unique MS spectra
- Quantification of known & unknown components based on peak integration
- Full scan/SIM mode for trace and ultratrace analysis in a single run





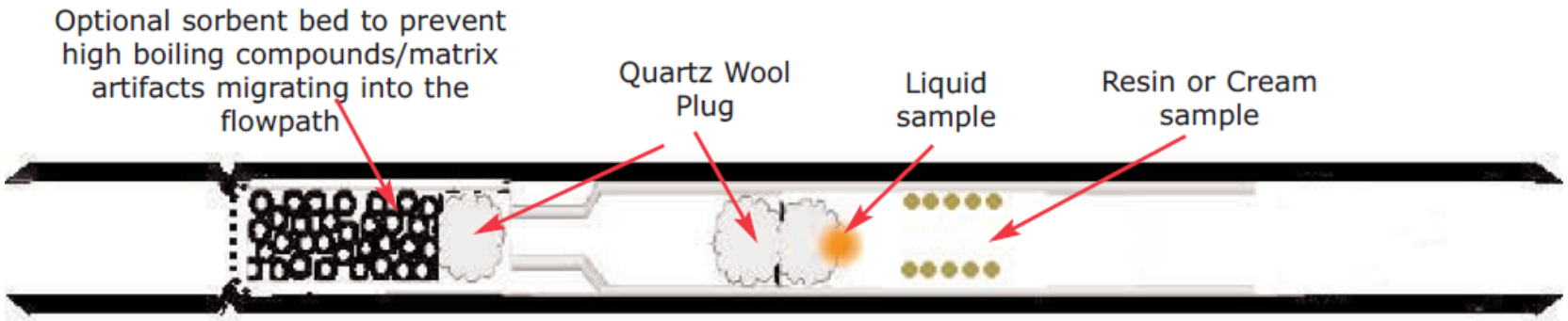
NON-ROUTINE MATERIAL EMISSION TESTING



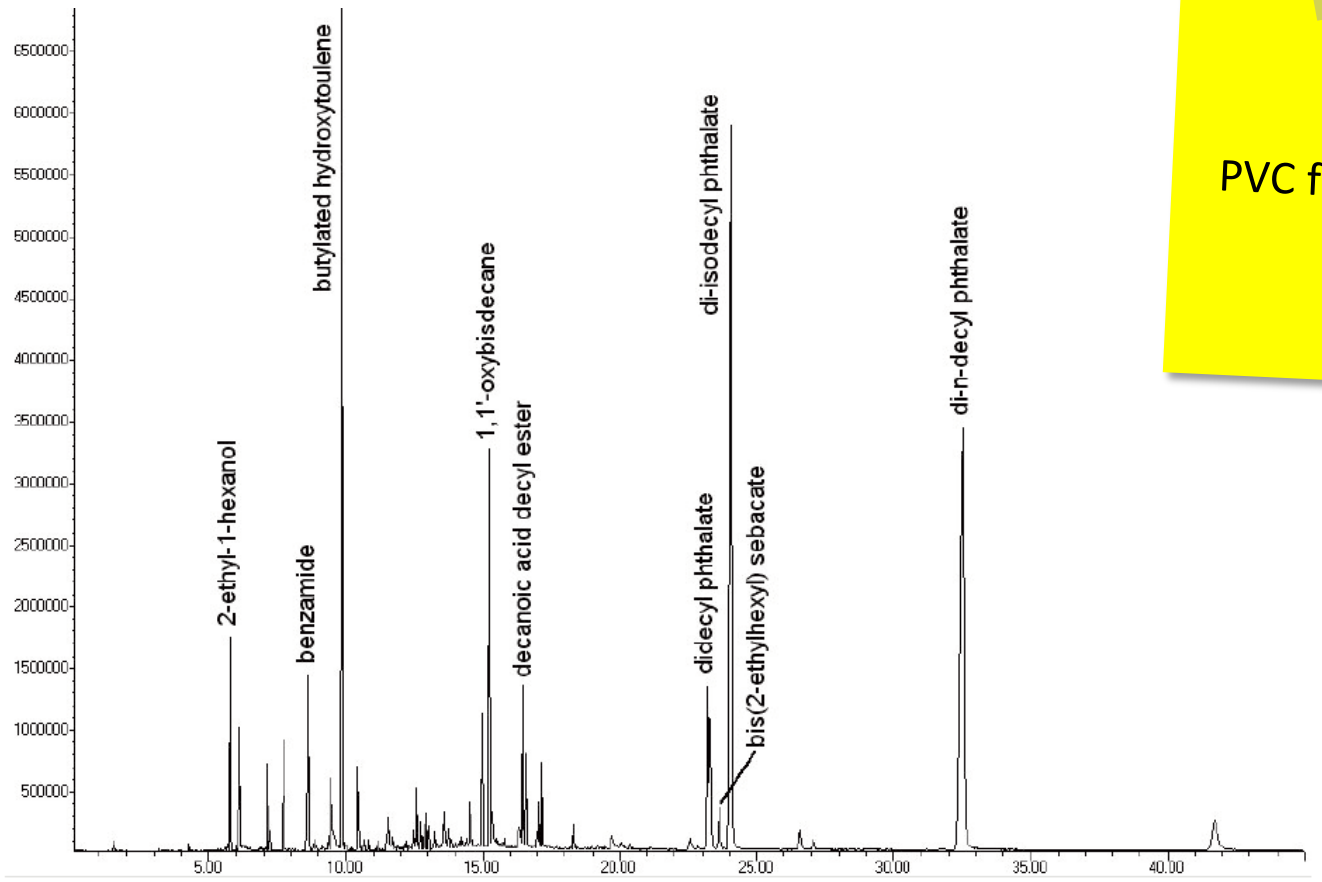
NON-ROUTINE MATERIAL EMISSION TESTING

Direct thermal desorption of materials

- Insert small piece of material in empty tube
- Grind in case of bulk solids
- Use PTFE liner in case of resins/adhesives

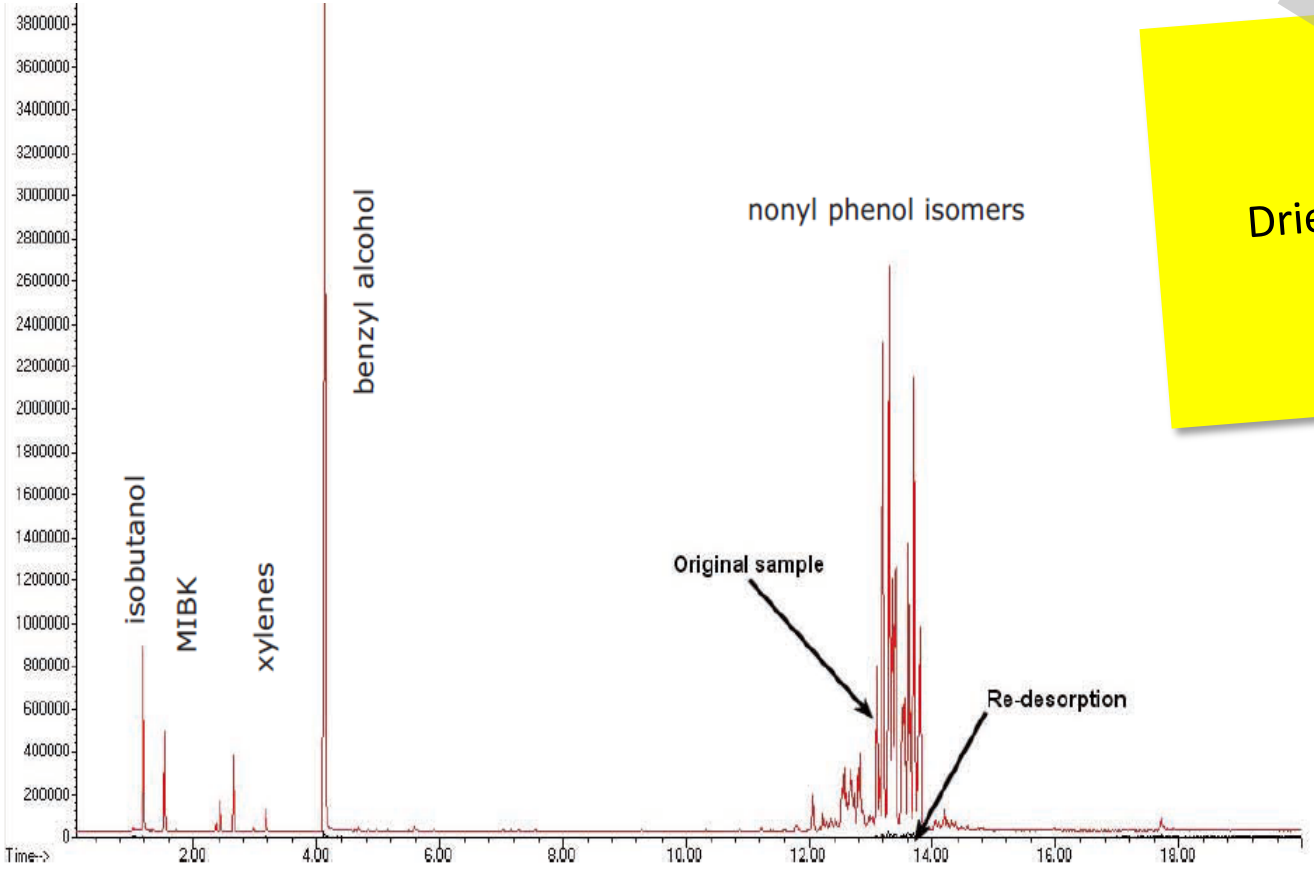


NON-ROUTINE MATERIAL EMISSION TESTING



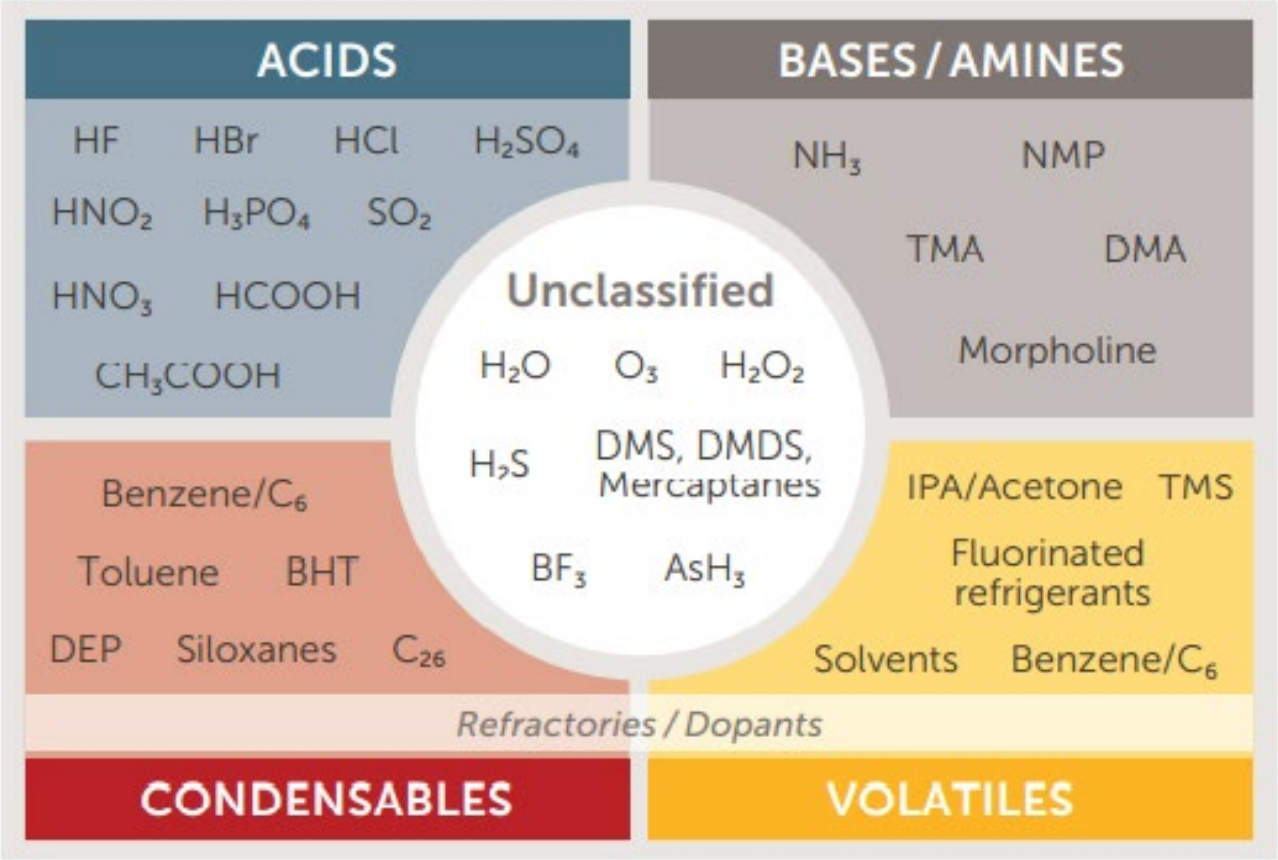
PVC foam sheet

NON-ROUTINE MATERIAL EMISSION TESTING



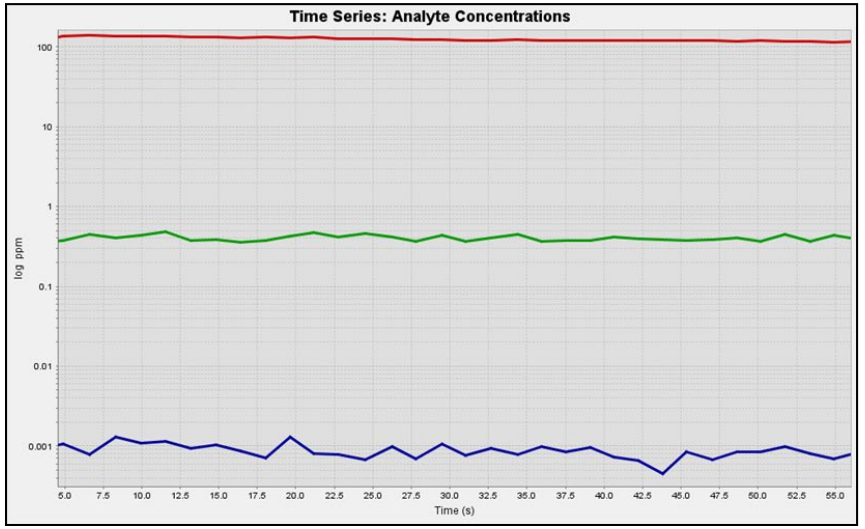
Dried paint

INNOVATION: SIFT-MS



INNOVATION: SIFT-MS

- Selected ion flow tube MS
- Direct analysis in real-time of:
  - volatile organic components
  - volatile inorganic components



HCl [125 ppm]

Benzene [40 ppb]

Dimethyl sulfide [870 ppt]

TARGET COMPONENTS

Compound Class	Compound Class
Alkanes	Nitrogen heterocycles
Alkenes and dienes	Nitrated organics
Alkynes and diynes	Oximes
Aromatic hydrocarbons	Nitriles
Terpines	Halogenated alkanes and alkenes
Alcohols and diols	Halogenated aromatics
Aldehydes and dialdehydes	Miscellaneous halogenated organics
Ketones	Thiols
Carboxylic acids	Thioalcohols
Esters	Thioacids
Ethers	Thioethers
Cellosolves	Sulfur heterocycles
Oxygen heterocycles	Sulfoxides
Epoxides	Organophosphorus compounds
Amines	**Inorganics**
Amides	

Inorganics
NH3, PH3, AsH3, GeH3
B2H6,
SiH4, siloxanes,
H2S,
HF, HCl, HBr,
Cl2, Br2,
SO2, CO2, N2O

APPLICATIONS

- TOC analysis
- Material emissions
- Online monitoring

imec
SAMSUNG

Material emissions

- Similar strategy as TD
- Immediate response
- Ideal for fast troubleshooting
- Includes the emission of inorganics!

On-line monitoring

- Multipoint sampling
- High temporal resolution
- Organics + inorganics!

CONCLUDING REMARKS

More info?

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