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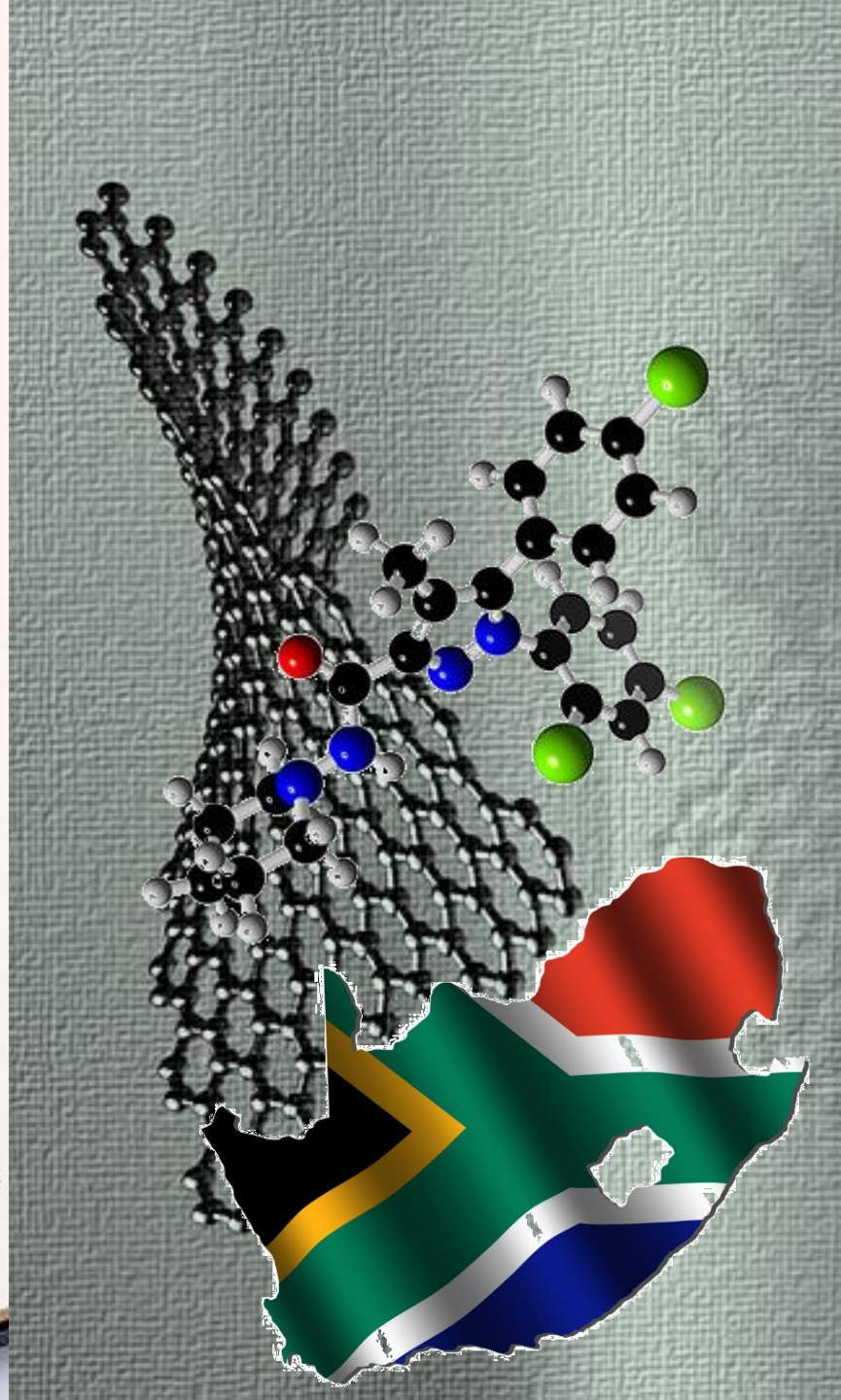
National Metrology Institute of South Africa

South African highlights & expectations

Dr Jayne de Vos

UNEP/ GEF Workshop
Accra, Ghana – 06-08 July 2016



NMISA's mandate

Described broadly, NMISA's functions include:

- Providing a measurement framework that is internationally acceptable;
- Ensuring that South Africa (and the region's) interests are protected in drafting international rules and regulations;
- Assistance with drafting South African National Standards (SANS) and compulsory specifications by guiding attainable measurement limits and ranges;
- Providing accurate and traceable measurements acceptable in a court of law;
- Conducting benchmarking exercises in support of accredited laboratories;
- Coordinating PT comparisons/ inter-laboratory comparisons for industry
- Provide training with regard to measurement science

Source: Act No. 18 of 2006: Measurement Units and Measurement Standards Act, 2006

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NMISA's role

- The primary functions are to:
  - Provide traceability to the international system of units
  - Provide reference measurements
  - Provide calibration services where there is a gap nationally
  - Provide consultation
  - Provide training in the calculation of uncertainty
  - Remain abreast of international developments relating to improved measurement accuracy through dissemination of measurement traceability
- NMISA will establish a capability and then look to partner with industry/ or a laboratory to do the actual measurements; not routine analytical measurements where there is industry capability
- NMISA will also train scientists (industry and research) in the methodology and traceability required to obtain good results
- Serve its stakeholders and industry in support of competitive trade including, but not limited to, the food and feed sectors, as well as supporting environmental and health legislation

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Technical Infrastructure

the dti
Department:
Trade and Industry
REPUBLIC OF SOUTH AFRICA

sanas
South African National Accreditation System

Accreditation:

provides formal recognition to perform calibration and measurement tasks

SABS

Standardisation:

documentary standards for validated methods used in industry

NRCS national regulator for compulsory specifications

Regulation:

administers regulation to ensure compliance and protect human health and safety

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Metrology:

underpins testing and calibration through measurement accuracy and traceability

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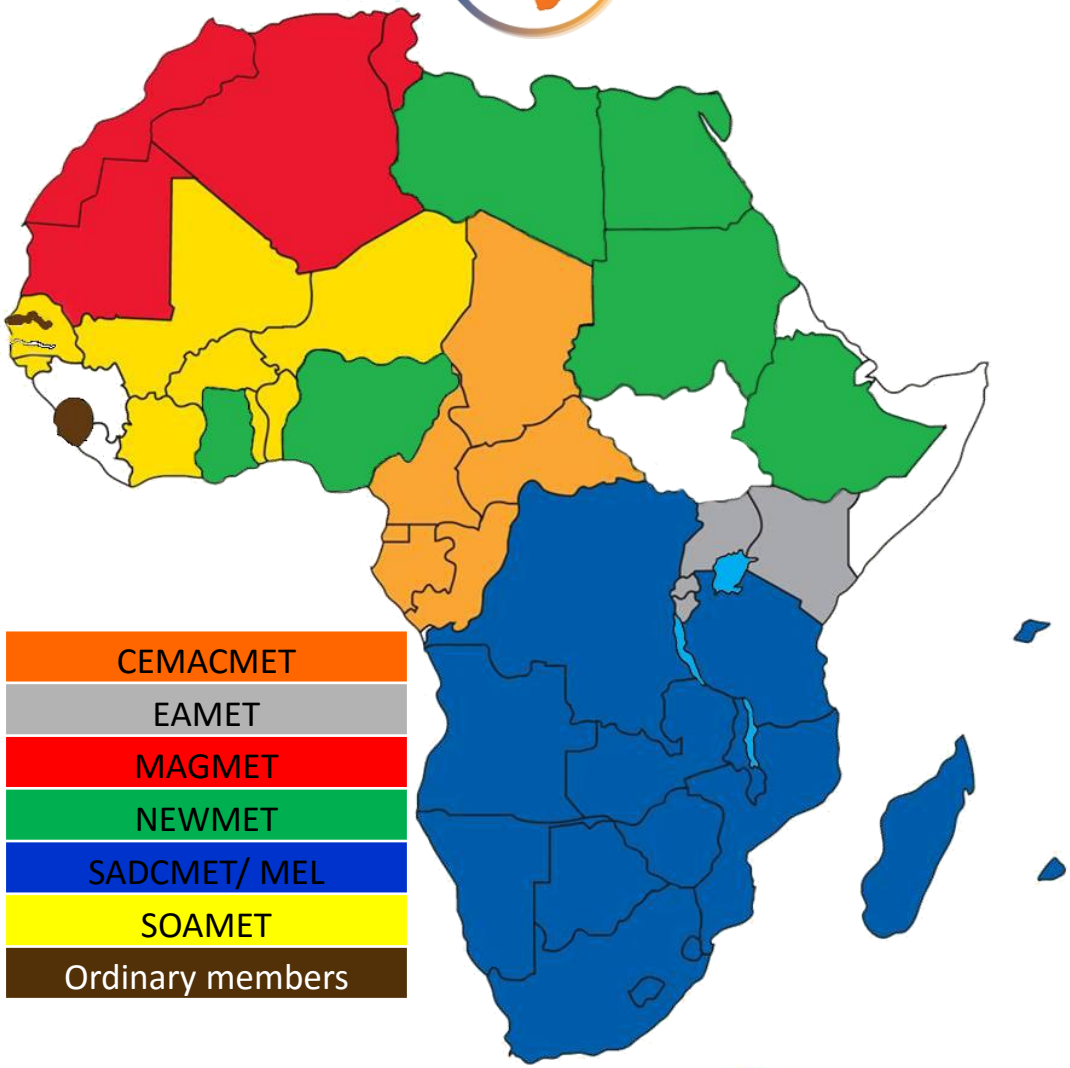
Regional activities

- 42 principal members (end 2015)
- In support of the CFTA and accredited testing facilities;
  - Produce calibration solutions
  - Introduce a PT scheme category within the comparison framework
  - Services database for metrology institutes (peer reviewed acceptance of capabilities for intra-Africa trade)
- With AFRAC, facilitate technique accreditation for NMIs in Africa

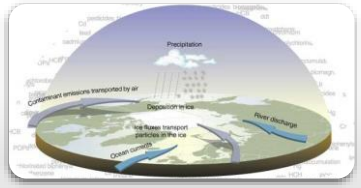


Why do we have to measure POPs?

Countries and trade regions **impose regulations** and directives to trade goods, **protect** the health of their people and the environment

Stricter **legislation** and the initiation of environmental programs are being applied globally to **limit these emissions**

The Stockholm Convention (SC) is a global **treaty** to protect human health and the environment from persistent organic pollutants (POPs)

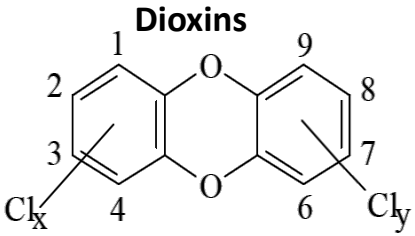


In implementing the SC, Governments must **take measures to eliminate or reduce** the release of POPs into the environment, making the analysis of environmental pollutants a **legal obligation**

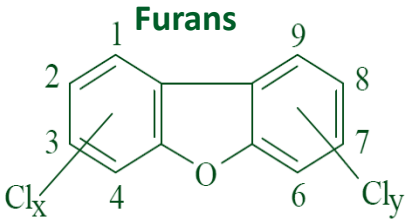
South Africa has **signed** this Convention and therefore has an **obligation** to undertake appropriate research, monitoring, and co-operation pertaining to POPs, and more particularly, PCDD/Fs

Why do we need this in SA?

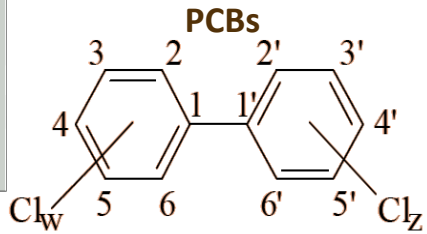
The list of potentially hazardous chemicals is ever increasing; the lack of analytical services to monitor these toxins; it's a compliance issue



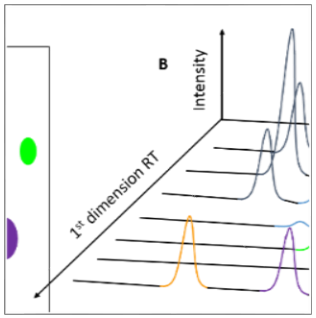
South Africa, as with most developing countries, has no established GC-HRMS facility for dioxin analysis



Necessitates the development of an alternative solution to measure PCDD/Fs at ultra-trace levels amidst challenging high levels of contaminants



Investigate selectivity, understand LOD/ LOQ to reliably quantify PCDD/Fs in the presence of interferences and assess the accuracy of the GC×GC-TOFMS method



Research outcomes & collaboration

University of Pretoria

NORTH-WEST UNIVERSITY

Environmental Monitoring

- Metrologically traceable reference measurement for the accurate quantification of dioxins and brominated flame retardants in the South African environment
- Developed methods for the extraction, analysis and quantification of PFCs in various environmental matrices
- Pesticide analysis in environmental matrices, as well as human biomonitoring for new generation pesticides and their metabolites
- Analysis of PAHs in abiotic and biotic matrices
- Close collaboration with UP, NWU and ULg
- IVM, MOE, Restek, LECO Africa and EPA Taiwan

Environment Canada

IVM Institute for Environmental Studies

Ontario

Research highlights[2]

National Research Foundation

South African WATER RESEARCH COMMISSION
Supporting sustainable development through research funding, knowledge creation and dissemination

ORASECOM
One River, Four Nations

SANCOR

- **Data generation** of persistent organic pollutants (POPs) and toxicants in abiotic and biotic matrices
  - Perfluorinated compounds (PFCs)
  - Polychlorinated biphenyls (PCBs)
  - Organochlorine pesticides (OCPs)
  - Polycyclic aromatic hydrocarbons
- **Training Programmes**
  - Introduction to the analysis of POPs
  - Use of the QuEChERS extraction technique for the analysis of PAHs
  - Analytical Proficiency
  - Analytical quality assessment and quality control

Publications & posters

Trends in Analytical Chemistry, Vol. 46, 2013 Trends

Experience in South Africa of combining bioanalysis and instrumental analysis of PCDD/Fs

Jayne de Vos, Laura Quinn, Claudine Roos, Rialet Pieters, Peter Gorst-Allman, Egmont Rohwer, John P. Giesy, Henk Bouwman

Contents lists available at ScienceDirect

Journal of Chromatography A

journal homepage: www.elsevier.com/locate/chroma

Establishing an alternative method for the quantitative analysis of polychlorinated dibenzo-*p*-dioxins and polychlorinated dibenzofurans by comprehensive two dimensional gas chromatography–time-of-flight mass spectrometry for developing countries

Jayne de Vos[a,*], Peter Gorst-Allman[b], Egmont Rohwer[c]

Contents lists available at SciVerse ScienceDirect

Chemosphere

journal homepage: www.elsevier.com/locate/chemosphere

Levels of PCBs in wild bird eggs: Considering toxicity through enzyme induction potential and molecular structure

L.P. Quinn[a,b,*], C. Roos[a], R. Pieters[a], K. Løken[c], A. Polder[c], J.U. Skaare[c], H. Bouwman[a]

Accred Qual Assur (2015) 20:273–280
DOI 10.1007/s00769-015-1122-7

PRACTITIONER'S REPORT

Coordination of organochlorine pesticides in water proficiency testing schemes in South Africa

Désirée Prevoo-Franzsen[1] · Nontete Nhlapo[1] · Laura Quinn[1] · Maria Fernandes-Whaley[1]

PERFLUORINATED COMPOUNDS IN WATER, SEDIMENT AND WILD BIRD EGGS FROM THE ORANGE-SENQU RIVER, SOUTH AFRICA

Swiegelaar CR[1,2], Quinn LP[1,2], Prevoo-Franzsen D[1], Bouwman H[2]

METHOD VALIDATION FOR THE QUANTIFICATION OF PFOS AND PFOA IN FISH

Swiegelaar CR[1,2*], Prevoo-Franzsen D[1], Bouwman H[2], Quinn LP[1,2]

ANALYTICAL ADVANTAGES OF SELECTIVE PRESSURISED LIQUID EXTRACTION COMBINED WITH TIME OF FLIGHT MASS SPECTROMETRY FOR THE QUANTIFICATION OF POLYBROMINATED DIPHENYL ETHERS IN SEDIMENT

Brits M[1,2,3*], Quinn LP[1,4], De Vos J[1], Weiss, JM[2], Rohwer E[3], De Boer J[2].

[1]NMISA, Organic Analysis Section, Meiring Naudé Road, Brummeria, Pretoria, South Africa, [2]VU University, Institute for Environmental Studies (IVM), De Boelelaan 1087, 1081 HV Amsterdam, The Netherlands, [3]University of Pretoria, Department of Chemistry, Pretoria, South Africa, [4]North-West University, Unit for Environmental Sciences and Management, Potchefstroom, South Africa

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Research projects

Green economies

- Primary reference gas mixtures (PRGMs)
- Traceability to GAW Cape Point
- Particle size for suspended particulate matter
- Silicon nano-wires and organic solar cells
- Surface analysis (elemental and chemical composition)
- Environmental monitoring (PAHs, PFCs, PCBs, Dioxins, BFRs, PFOS and pesticides)

African Food and Feed Certified Reference Materials program

- Certification of toxic/ nutritional elements in wheat flour
- Multi-mycotoxins in maize RMs, CRMs and reference measurements
- Amino acid content in infant formula (nutritional labelling)
- Phthalates in PVC food packaging
- Hazardous organic chemicals in South-African fish
- Dioxins and dioxin-like PCBs in food and feed, etc

Integrated Human Capital Development Program

- Improve staff qualifications
- Appoint qualified and experienced research staff
- Interns and Bursars
- Contract: retired, experienced individuals to partner with students and assist with training
- External training provided by NMISA

Analytical capability

Chemical class	Matrix
Pesticide and pesticide metabolite residues	
Pyrethroid, triazines, carbamates , organophosphates, organophosphates and aniline pesticides	Plant material
Pyrethroid , organophosphates, carbamates and associated metabolites	Urine and breast milk
Organochlorine and organophosphates pesticides	Water
Organochlorine pesticides	Soil, sediment and mussel
Priority pollutants	
Polycyclic aromatic hydrocarbons	Soil, sediment, mussel, fish and bird eggs
Phthalates	PVC
Persistent organic pollutants	
Polychlorinated dibenzo-*p*-dioxins	Soil and sediment
Polychlorinated dibenzofurans	Soil and sediment
Polybrominated diphenyl ethers	Soil, sediment
Polychlorinated biphenyls	Soil, sediment and mussel
Perfluorinated compounds	Soil, sediment, water, bird eggs and fish
Food contaminants	
Melamine	Infant formula
Mycotoxins	Maize and wine
Pharmaceutical residue	
Benzodiazepine	Wine
Antibiotic residue	Milk and pork meat

Chemical class	Matrix
Gas Analysis	
Volatile Organic compounds	Nitrogen
Green-house gasses	Nitrogen
Stack emission gases (CO_x, SO_x, NO_x)	Nitrogen/Air
Indoor and outdoor Air Quality Standards (medical gases)	Nitrogen/Air
Air Pollution gases	Nitrogen/Air
Automotive exhaust gas mixtures for emissions	Nitrogen
Ethanol (breath alcohol)	Nitrogen/ Air
BTEX	Nitrogen
Metal analysis in food matrices	
Al, As, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Zn	Water
Essential (Fe & Zn) and Toxic Elements (Cd, Pb)	Bovine liver
Trace metals (Ni, Pt, Cd, Cr, Hg, Pb)	Algae
Essential element (Se)	Wheat flour
Toxic elements (Sn, Pb and Cd)	Tomato paste
Trace elements (Zn)	Soybean
Trace elements (Pd, Cd, Ca and Fe)	Wine
Trace elements (Cd)	Rice
Essential (Ba, Ca, Cu, Fe, Sr, and Zn) and toxic elements (Cd, Ni and Pb)	Tobacco
Essential elements (K, Cu)	Infant formula

Inter-laboratory benchmarking

NMI comparisons (CCQM)

Coordinating laboratory	Description	Status
2011 - 2012		
GLHK/ NIM China	CCQM P136 - Endosulfan II and endosulfan sulfate in green tea	Completed
2012 - 2013		
BIPM	Purity assessment L-valine	Completed
NIM China	CCQM K103 Melamine in milk powder	Completed
2014 - 2015		
BIPM/NIM	CCQM K115 - Peptide purity	Completed
GLHK	CCQM K126 - Carbamazepine in Surface Water	Completed
IRMM	CCQM K102 - PBDEs in sediment	Completed
2015 - 2016		
BIPM	CCQM.K55d - Purity assignment Folic acid (mass balance)	Completed
BIPM	CCQM.P117d - Purity assignment Folic acid (Q-NMR)	Completed
2016 - 2017		
UME	CCQM K138 - Aflatoxin in figs	In progress
NIST	CCQM.K95.1 - PAHs in yerba mate tea	In progress
NIST	CCQM.K131 - PAHs in acetonitrile calibration solution	In progress
UME	CCQM K138 - Aflatoxin in figs	Registered

International comparisons

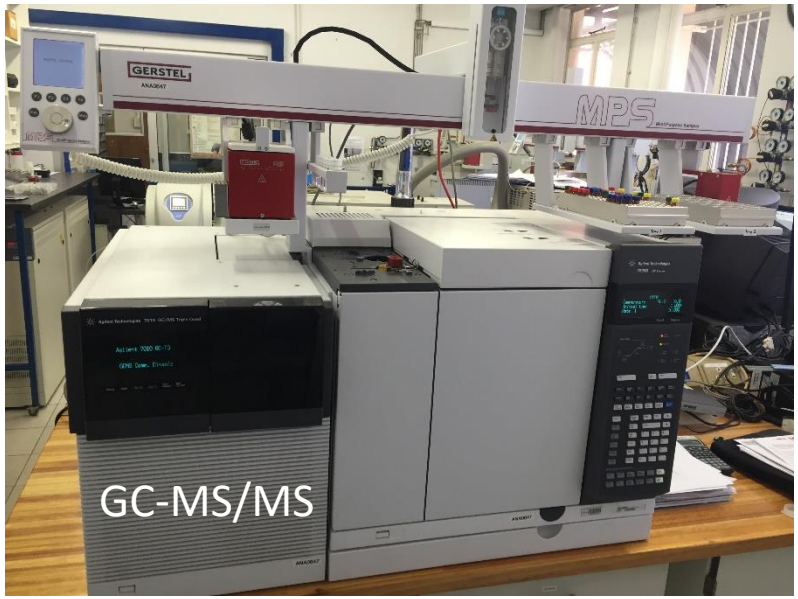
Coordinating laboratory	Description	Status
2012 - 2013		
ERA USA	ERA CW 8 - OCPs in drinking water	Completed
2013 - 2014		
FERA, UK	FAPAS 2998 – Multimycotoxins in maize	Completed
FERA, UK	FAPAS 04223 – Mycotoxins in maize	Completed
2014 - 2015		
ERA USA	ERA EF 13 - OPPs in effluent	Completed
2015 - 2016		
FERA, UK	FAPAS 04280 - Aflatoxins in fig slurry	Completed
FERA, UK	FAPAS 04276 - Mycotoxins in maize	Completed
FERA, UK	FAPAS 17156 - Ochratoxin-A in wine	Completed
FERA, UK	FAPAS 3060 - Melamine in milk	Completed
2016 - 2017		
Quasimeme	PAH and PFCs	Registered
UNEP	OCPs, PCBs, PCDD/Fs, PBDEs and PFCs	Registered

Bureau International des Poids et Mesures

NMISA coordinated proficiency testing schemes

- Aqueous ethanol proficiency testing scheme, 3 rounds, 3 levels each
- Aqueous sodium fluoride proficiency testing scheme, 1 round, 2 levels
- Organochlorine pesticides in water proficiency testing scheme, 1 round
- Organophosphorus pesticides in water proficiency testing scheme, 1 round

Strengthening analytical infrastructure[1]

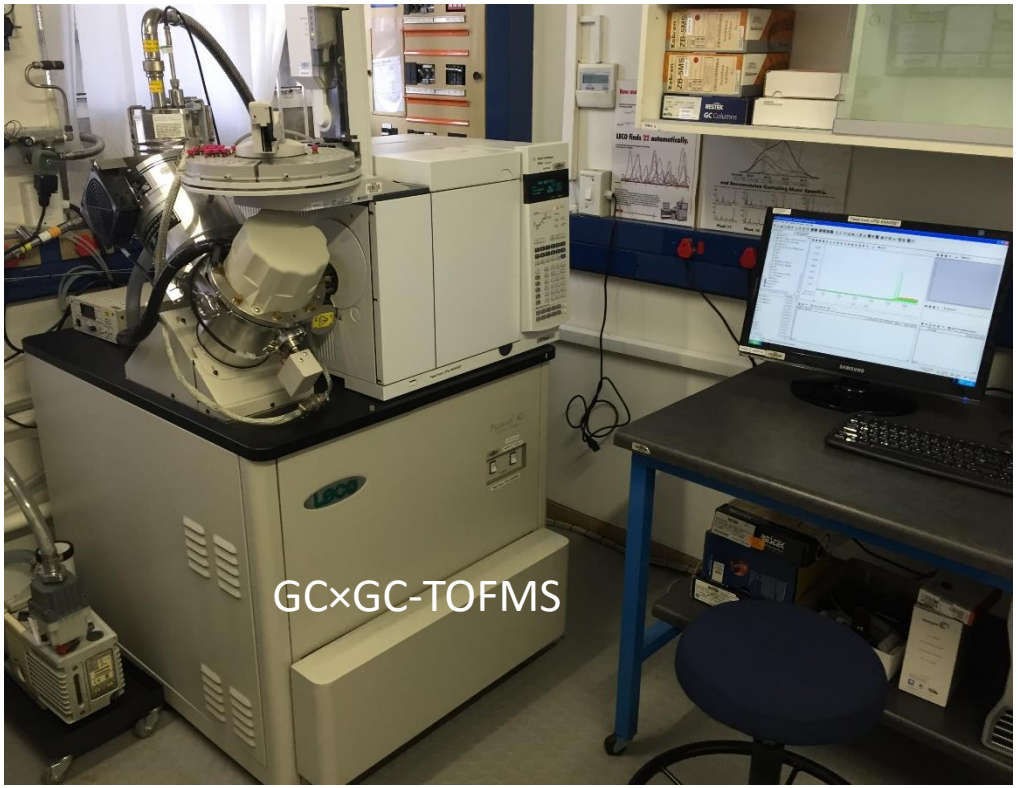


Inlet system:

- Gas chromatography electron impact ionization (GC-EI)
- Gas chromatography electron capture negative ionization (GC-ECNI)

Detector:

- Triple quadrupole mass spectrometer (MS/MS)



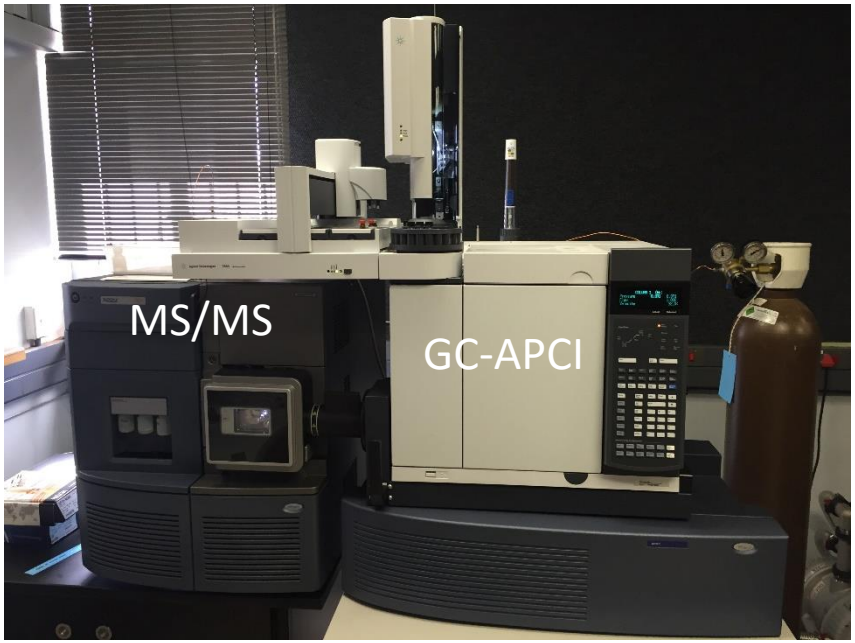
Inlet system:

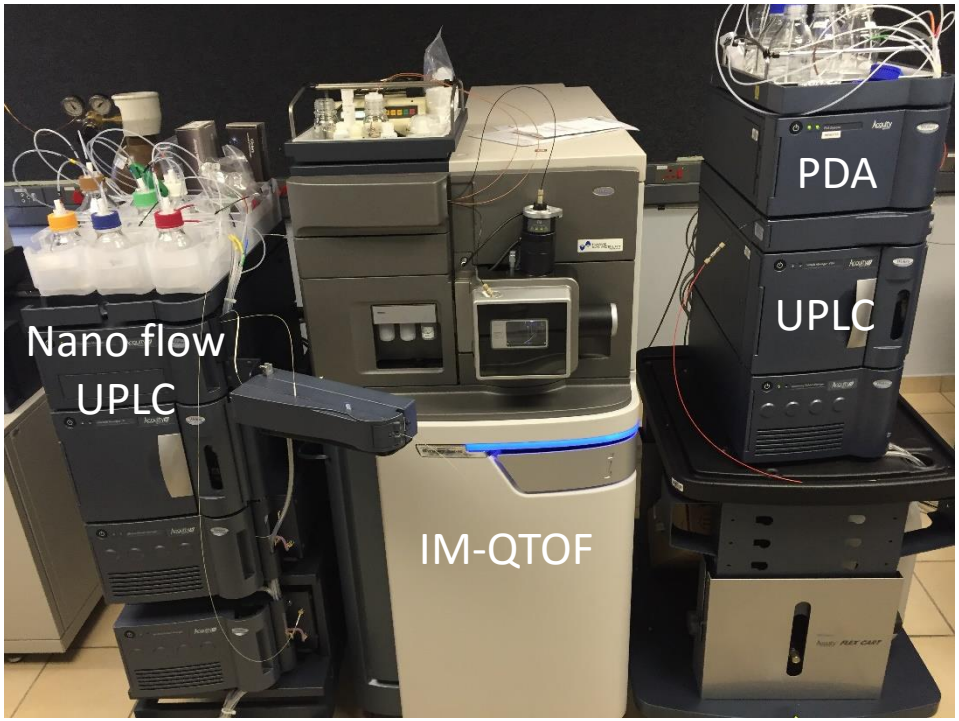
- Comprehensive two-dimensional gas chromatography - electron impact ionization (GC×GC-EI)

Detector:

- Time-of-flight mass spectrometer (TOFMS)

Strengthening the analytical infrastructure[2]





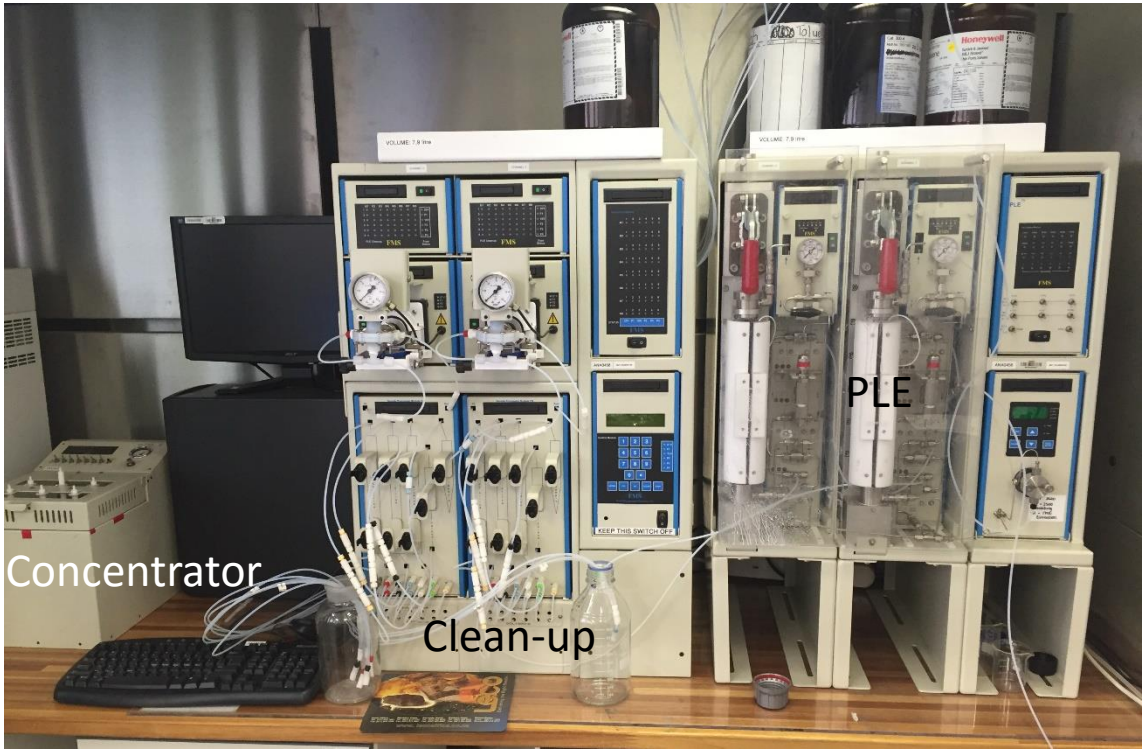
Interchangeable inlet systems:

- High performance liquid chromatography
- Ultra high performance liquid chromatography
- Nano flow ultra-high performance liquid chromatography
- Gas chromatography atmospheric pressure chemical ionization
- Solid sample introduction probe

Detectors:

- Ion mobility accurate-mass quadrupole time-of-flight mass spectrometer (IM-QTOF)
- Triple quadrupole mass spectrometer (MS/MS)
- Photodiode Array (PDA) Detector

Strengthening analytical infrastructure[3]



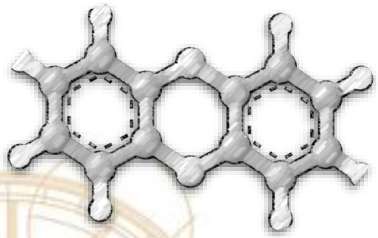
Accelerated Solvent Extraction (ASE)

TRP Total-Rapid-Prep system performs the pressurised liquid extraction (PLE) , multi-column clean-up and concentration

Broad vision

Trace Laboratory

PCDDs
PCDFs
PBDEs
PCBs
PFCs
PAHs
Pesticides
New generation POPs



Toxicological Testing

Reporter gene bio-assay
Acute Toxicity assays
Mutagenicity assays
Broad spectrum screening

CRM Production

Combined Trace and Toxicological Environmental CRM
Sediment
Soil
Fish
Mussel
NONE EXIST

Additional considerations

- The instruments are used for other projects within the section and as routine analysis becomes the primary objective, other activities may be delayed
- Limited/ no research; focus will be routine analysis and product development
- Will need budget to employ more qualified analysts
- Cost benefit to industry – time benefit
- Shift in current operations
- Large capital investment in laboratory space and infrastructure
- Initial investment with little to no return
- Improved collaboration with other institutes involved in monitoring POPs (AISA, DEA, etc)

Expectations

- Support from the expert laboratories in initial setup to establish a routine testing facility for PCDD/Fs
- Further collaboration in terms of the emerging POPs
  - Mr Martin Brits (DST/NRF scholarship under the VU Amsterdam-NRF Desmond Tutu Doctoral Programme and UP)
  - Ms Caitlin Swiegelaar (NWU/ NRF – Orasecom)
- NMISA focus will be geared more toward training and QA/ QC
- NMISA has planned an MSc bursar for 2017/18 (POP extraction techniques)
- NMISA has planned funding for a senior scientist for 2017/18 and 2018/19

QUESTIONS?

Dr Jayne de Vos (bjdevos@nmisa.org)