nmisa
National Metrology Institute of South Africa

Establishing Metrology in Chemistry at the NMISA

- Wynand Louw and Sara Prins -

Your measure of excellence

Your measure of excellence

MiC @ NMISA

- Established since 1996
- Gas metrology
  - Calibration of breath alcohol testers
  - Calibration of Gas Analyzers
  - Preparation of primary gas mixtures
- Inorganic chemistry
  - Certification of reference materials for the mining industry (Au, Pt and other ores)

MiC @ NMISA

- Surface analysis added in 1998 during a re-structuring process at CSIR (the parent company of the then National Metrology Laboratory)
  - Focus on nano-metrology
  - Quantitative XPS and SIMS analysis
  - Dimensional measurements with SEM
  - EDS and OES analysis

MiC @ NMISA

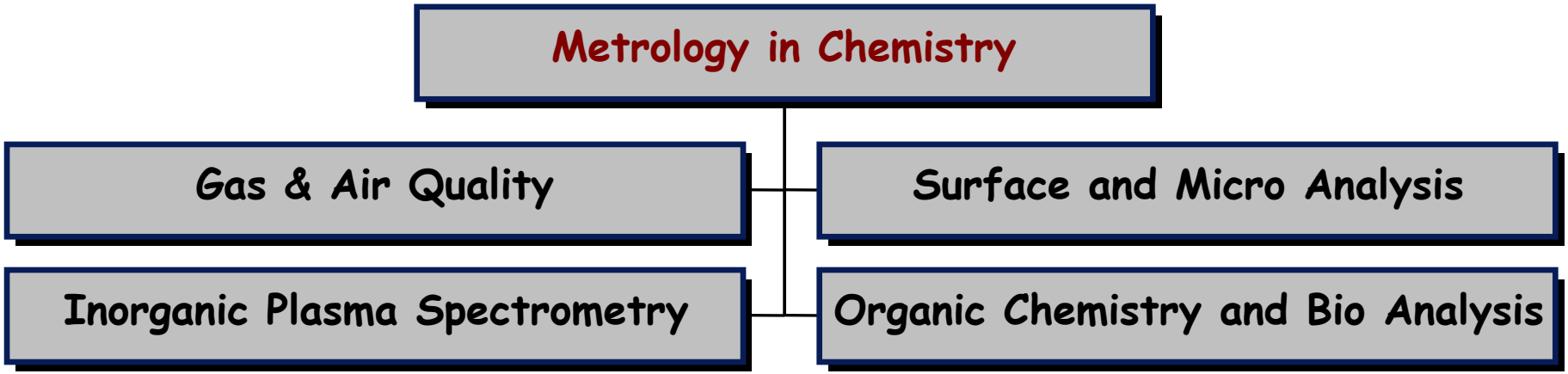
- On request from the Forensic community, started Organic chemistry section in 2002
  - Value assignment of drugs for police forensics
  - Ethanol in aqueous solution CRMs
  - Pesticide reference solution “cocktails”
  - Later, focused on substances of concern in food matrices

MiC @ NMISA

- During 2005, investigated Bio-analysis. The strategy was to sub-contract (and later designate) a laboratory within CSIR to represent SA in bio-analysis
  - At first, good collaboration
  - An expert from CSIR attended BAWG meetings
  - Difference in priority, GMO research vs traceability
  - Finally, the strategy had to be changed and a metrologist was appointed to start BA in NMISA

MiC @ NMISA

- Current situation
- 22 staff, 14 with MSc/PhD
- Operate over 4 technical areas



The MiC Group today

2007/8 International Comparisons

Intercomparisons 07/08		Working Group	Description
1	CCQM-P85	IAWG	Fe, Zn, Cd, Pb, Se and Cr in Bovine liver
2	CCQM-P86	IAWG	Se in pharmaceutical supplement
3	CCQM-K56	IAWG	Ca, Fe, Cu, Zn in whole fat soybean
4	CCQM-K30	IAWG	Pb in wine
5	CCQM-P12.1	IAWG	Cd, Cu and Fe in wine
6	CCQM-52	GAWG	CO_2 in Air
7	CCQM-K51	GAWG	CO in N_2 (PILOT laboratory)
8	BIPM.QM-K1	GAWG	Ambient Ozone
9	Euromet 900	GAWG	CO in N_2
10	CCQM-P20e	OAWG	Theophylline Purity Assessment
11	CCQM-P90	OAWG	Chloramphenicol in milk
12	CCQM P-98	SAWG	Composition of Fe-Ni alloy films
13	CCQM P-95	SAWG	Determination of Light Elements-N doped Carbon layers

NMISA Chemistry CMCs

- Submitted CMCs:
  - Gas
  - Inorganic
  - Organic
  - Surface Analysis
- CMC review
  - Review group established in AFRIMETS
  - Will still receive assistance from APMP
- CCQM-KCWG participation
  - Angelique Botha (GAWG)
  - Mare Linsky (IAWG)
  - Wynand Louw (SAWG)

Gas Metrology

- Initially, the facility was established for gravimetric preparation of gaseous reference materials and calibration of breath alcohol analyzers
- First national standard was for evidential breath analysis; certification of secondary reference gas mixtures from ethanol in nitrogen
- Accredited to ISO 17025 for gravimetric preparation of gas mixtures
- Accredited to ISO 34 for CRM production

Gas PRMs

Available

- carbon dioxide (CO_2) in nitrogen (N_2) and air
- carbon monoxide (CO) in nitrogen
- sulphur dioxide (SO_2) in nitrogen and air
- nitrogen monoxide (NO) in nitrogen
- nitrogen dioxide (NO_2) in air

Coming soon

- Stack gas PRMs (CO_2, CO, NO, SO_2 and propane)
- Ethanol in nitrogen condensed gas

Other Services

- Calibration of gas analysers (for ambient air quality monitoring, occupational health and safety applications and emissions monitoring. Breathalysers are also calibrated)
- Purity analysis of client's gas mixtures
- Gravimetric preparation of special mixtures on request
- Intercomparisons and proficiency testing (national audit sample for South African National Accreditation System -SANAS)

Gas Planned Expansions

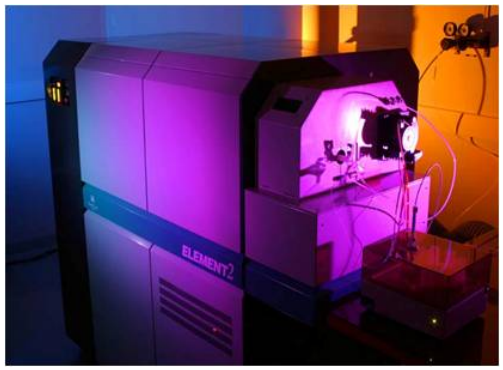
- Corrosive gas analysis and PRMs
- CRDS for trace impurities
- Condensed gas mixtures
- Air flow meter calibrations

Intercomparison History

Intercomparisons		Working Group	Description
1	CCQM-P73		30 to 70 µmol/mol CO in nitrogen
2	CCQM-52	GAWG	CO_2 in Air
3	CCQM-K51	GAWG	CO in N_2 (PILOT laboratory)
4	BIPM.QM-K1	GAWG	Ambient Ozone
5	Euromet 900	GAWG	CO in N_2
6	CCQM-K66	GAWG	Purity of methane (CH_4)
7	CCQM-XX	GAWG	Stack gas emissions – this intercomparison has not yet been assigned a number

Inorganic

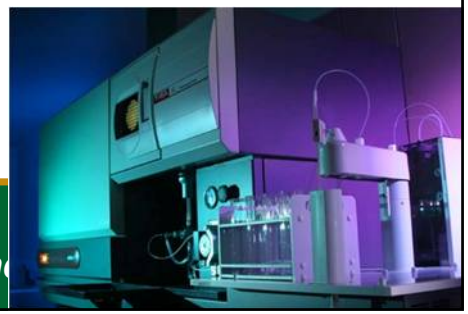
- Initially established for trace and ultra-trace reference analysis of Inorganic elements in ore samples
- Invested in state-of-the-art equipment:
  - -ICP-MS with isotope dilution
  - -Advanced sample preparation to support
  - -Investment in reference materials



- Establish reputation by participation in numerous CCQM and APMP international (key/pilot) comparisons
- Can now focus on the dissemination to industry

Inorganic – Specific Projects

- IAEA - Proficiency test study for analytical laboratories of the Nuclear Reactors Research Centres in Africa
- Value-assigned Pb content in wheat samples for the SA Grain Laboratory
- Trace measurements for nanotechnology projects
- PGM analysis consultation to the automotive industry

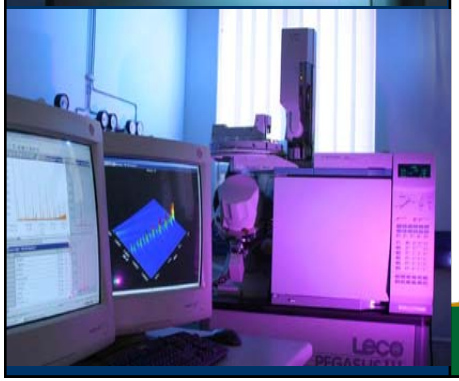


Current Inorganic Focus

- Trace and ultra-trace element analysis of food, environmental and inorganic materials
- Metal speciation analysis
- Certification of reference materials through international collaboration and comparison studies
- Assisting industry with reference analysis and characterisation of reference materials
- Consulting on laboratory methods, procedures, method validation and promoting concepts of traceability and measurement uncertainty
- Statistical analysis and data processing of analytical results for laboratory comparisons and proficiency testing studies

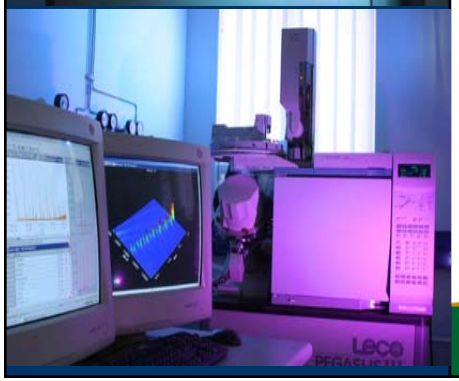
New Horizons

- Metal Speciation Analysis:
  - Amount of metal
  - Chemical form of the metal is important too!!
    - Bio-availability
    - Toxicity
    - Therapeutic effects

Organic

- Established during 2001/2002
- Focus was on forensic and pharmaceutical applications
  - Significant capital investment for basic equipment
  - Human capital development by extended visits to established NMIs
- Established facility for production of CRMs on request of Department of Health



Current Projects

- POPs (Chlorinated pesticides, PCBs, dioxins, furans and brominated flame retardants)
- Mycotoxins
- Biofuels (biodiesel)
  - Methods to determine the FAMEs (fatty acid methyl esters)
- Wine adulteration
  - Methoxypyrazines in Sauvignon Blanc

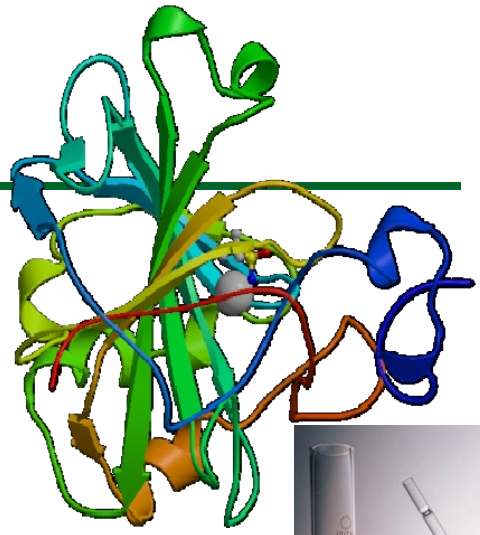
Biofuels in South Africa

- Government Strategy Document in 2007
- Tax incentives proposed
- Standards development for supply chain management in progress to protect all stakeholders
- Very stringent specification standards for biofuels (based on EU standards)
- Analysis anticipated to be CRM sensitive (based on the stockfeed used)

Organic CRMs

- Aqueous ethanol solutions
  - 4% for the 10 mg / 100 g solution
  - 2% for the 20 mg / 100 g solution
  - 1.3% for the 50 mg / 100g solution
  - 1% for conc between 55 and 500 mg / 100 g
  - 1.2% for conc between 1 g / 100 g and 20 g / 100 g
- NaF solutions
  - 0.3 to 3 g / 100 ml

Bio-Analysis



- 1st official bio-appointment in Feb 2008
- Business plan: ‘Market’ needs assessment, national bio-facility overview, BAWG direction
- Initial focus will be measurements in serum
  - Testosterone in human urine

Organic Comparisons

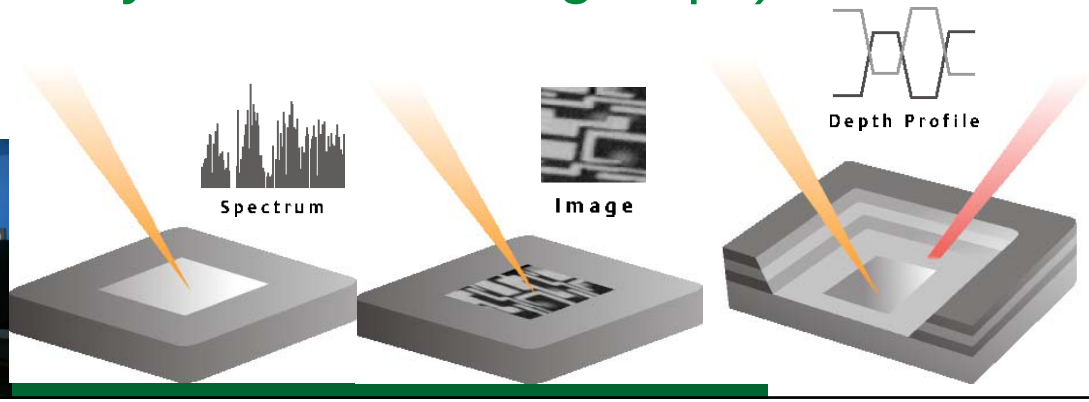
Intercomparisons 07/08		Working Group	Description
1	CCQM-P20e	OAWG	Theophylline Purity Assessment
2	CCQM-P90	OAWG	Chloramphenicol in milk
3	CCQM-K27	OAWG	Ethanol in aqueous matrix
4	CCQM-P20a-d	OAWG	Purity assessment of Atrazine and Chlorpyrifos
5	CCQM-P31a	OAWG	PAHs in Organic Solution
6	CCQM-P31b	OAWG	PCBs in Organic Solution

Surface Analysis

- ‘Inherited’ in 1998 a well-established highly skilled group of surface analysts
- Immediately became involved in SAWG’s first international pilot comparison (thickness of ultra-thin layers (2-18 nm) of SiO_2 on Si)
- Proficiency testing schemes:
  - electron microscopy magnification calibration
  - elemental analysis by EDS
- Very active in SAWG, VAMAS, ISO 201/202/229

Surface Analysis

- XPS / SEM-EDS-EBSD / TOF-SIMS / XRD / GD-OES
- Study materials by looking at the interaction of ‘radiation’ (x-rays, electrons, ions) and matter (metals, polymers, ceramics, bio-matter):
  - Imaging (nano-scale and elemental mapping)
  - Elemental composition and binding energies
  - Crystal structure
  - Surface layers and coatings (thickness and composition)
  - Surface chemistry (catalysis, functional groups)
- Polymer analysis



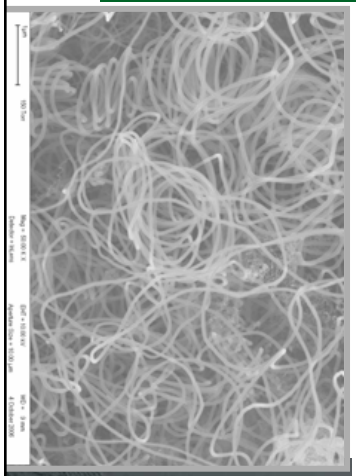
Intercomparisons

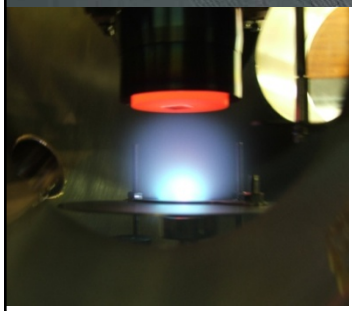
Intercomparisons		Working Group	Description
1	CCQM-K32	SAWG	SiO2 on Si
2	CCQM P80/81	SAWG	C and N for TiC and VN coatings on steel
3	VAMAS TWA2	VAMAS	Static TOF-SIMS Inter-laboratory study
4	CCQM P-98	SAWG	Composition of Fe-Ni alloy films
5	CCQM P-95	SAWG	Determination of Light Elements-N doped Carbon layers
6	CCQM K-67	SAWG	Composition of Fe-Ni alloy films (by XPS)
7	CCQM P-108	SAWG	Composition of Fe-Ni alloy films (by SEM/EDS)
8	VAMAS - TWA33#1	VAMAS	Determination of the shape, size and size distribution of nano-filler particles

‘New’ Metrology

- Nanometrology
- Materials Metrology

Nanometrology





- In MiC
  - Thin films – thickness and composition
  - Particle size analysis of nano-particles
- In Dimensional Metrology
  - AFM nano-scale measurements
  - Round robin with 9 participating labs in SA
  - X-Y measurements only at moment
  - Traceability to national length standard (laser) through diffraction grating

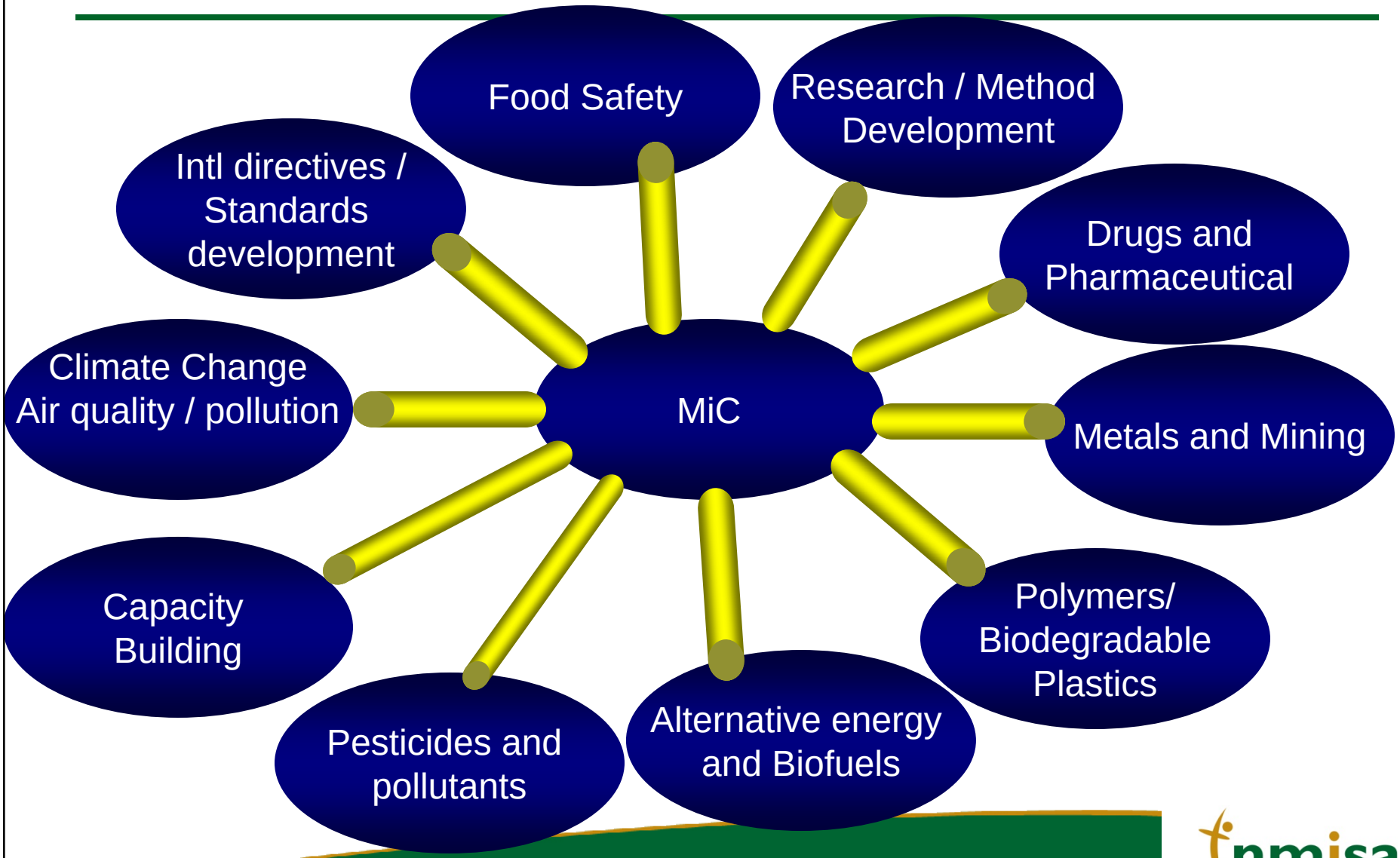
Materials Metrology

- "*the metrology of material property measurements*"
- Need for international comparability on materials properties
- Usually these have derived / non-standard units
  - Tensile strength, toughness, thermal expansion, thermal conductivity
- Environmental information of materials

The Way Forward…

- Gathering our information
  - National directives and government focus areas
  - International metrology trends (CCQM, WHO, JCTLM, WMO, IAEA, IFCC, IUPAC, ISO, REMCO, ILAC and CITAC)
  - TBTc, international directives for trade/compliance
  - Regional interaction
  - Technical Advisory Forums
  - Conferences, workshops
  - Stakeholder interaction (e.g. Food Safety Initiative)

Strategizing…



Our successes

- We started small and build skill and capacity in specific focus areas
- Dedicated, well trained personnel
- Clear deliverables
- Support (institute, government)
- Internal collaboration
- Continuous learning
- Communication with industry

Our failures

- A needs assessment for MiC in SA was performed by Physical Metrologists
- Started activities based on “physical” perspective of metrology, thus Gas
- Inorganic and Organic started at the request of the mining industry and forensics. Too industry focused and most of the needs could more easily have been addressed through the importation of CRMs
- Not enough energy was spent on the integration of MiC into the institute, “cultural” differences between physicists / engineers and chemists

Hindsight...

- Our advice for starting MiC would be to ensure a proper needs assessment, focusing on national priorities and issues that can not be addressed by “importing” traceability
- Then design laboratories and appoint a mix of experienced and young analytical staff
- Send experienced staff to other NMIs for exposure, periods of 2-12 weeks
- Spend the first two years on getting staff proficient in their methods and refining activities. Interact with other analytical laboratories at national level.

Hindsight...

- Start to participate in regional and international pilot studies
- Design a schedule for participation in regional and international key comparisons with the assistance of the regional MiC chair and the CC WG chair
- Your laboratories will now be at a level to successfully join the international MiC world
- Most importantly, don’t try to do everything at once. It takes time and a lot of money, always anticipate 30-50% more than budgeted originally!

Focus, Focus, Focus!

Contacts at NMISA

Who	email	Expertise
Dr Wynand Louw	wlouw@nmisa.org	AFRIMETS, general NMISA
Jayne de Vos	BJdeVos@nmisa.org	Metrology in Chemistry
Daphney Shikwambani	dshikwambani@nmisa.org	Metrology in Chemistry General contacts
Donald Masuku	dmasuku@nmisa.org	SADCMET