Mandate

The MiC area is aligning its overarching strategy to establish a comprehensive analytical capability towards a centre of excellence for providing South Africa with traceability through higher order reference measurements.

The Metrology in Chemistry (MiC) area is mandated to provide measurement traceability to industry through reference analysis, the development of reference methods, as well as the preparation and certification of reference materials and dissemination of traceability through the provision of proficiency testing schemes.

Uniquely for the country and Africa!
MiC Main Activities

MiC Traceability

Reference Measurements

Instrument calibration

Ozone meters
Breathalysers

Samples/ PT

Environmental
Food safety
Energy
Automotive
Nanotechnology
Pharmaceutical

Reference Materials

Pure mixtures (calibration)

Elemental solutions
Gas PRMs
EtOH & NaF CRMs

Matrix (bias)

Maize

Pure mixtures (calibration)

Elemental solutions
Gas PRMs
EtOH & NaF CRMs

Maize
Gas Metrology

- GC-FID/ PDHID, FTIR, NDIR, GC-MSD and CRDS
- Preparation of primary gas reference mixtures by gravimetry in N₂ and air matrices
- CO₂; CO; NO; NO₂; SO₂; H₂S; C₃H₈; Stack gas mixtures
- Purity analysis
- Calibration of breathalysers
- Calibration of air pollution analysers
- Certification of gas mixtures
- Uncertainty in Chemical Measurement
Air Pollution projects

**Climate change**
- SA does have a CC response strategy (National Air Quality Act No. 39 of 2004)
- UNFCCC
- 1 April 2010
  - Ambient air monitoring
  - Stack emissions
  - Automotive emissions
- Kyoto protocol (accessed 2002, adopted Dec 1997), 6 GHG (CO₂, CH₄, N₂O, HFC, PFC, SF₆)

**NRL**
- Manage Air Pollution
- SAAQIS (March 2010)
- DEA(T)
- Expand to include
  - dioxins,
  - SPMs,
  - temperature,
  - radio-activity (α, β and γ emissions
  - pesticides & heavy metals in air, etc)
- Host NRL at NMISA

**PRGMs**
- Provide primary reference gas mixtures
- Static gravimetric preparation
- Condensable gas mixtures
- BTEX in N₂
- VOCs
# Primary reference gas mixtures (PRGMs)

<table>
<thead>
<tr>
<th>Description</th>
<th>Concentration</th>
<th>Expanded uncertainty (k=2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon monoxide (CO) in air/nitrogen</td>
<td>1-10 % mol/mol</td>
<td>0,5% relative</td>
</tr>
<tr>
<td>Carbon monoxide (CO) in air/nitrogen</td>
<td>1000 –10 000 μmol/mol</td>
<td>0,5% relative</td>
</tr>
<tr>
<td>Carbon monoxide (CO) in air/nitrogen</td>
<td>100 – 1000 μmol/mol</td>
<td>0,5% relative</td>
</tr>
<tr>
<td>Carbon monoxide (CO) in air/nitrogen</td>
<td>10 –100 μmol/mol</td>
<td>0,5% relative</td>
</tr>
<tr>
<td>Carbon monoxide (CO) in air/nitrogen</td>
<td>1 – 10 μmol/mol</td>
<td>0,5% relative</td>
</tr>
<tr>
<td>Carbon dioxide (CO₂) in air/nitrogen</td>
<td>1-20 % mol/mol</td>
<td>0,5% relative</td>
</tr>
<tr>
<td>Carbon dioxide (CO₂) in air/nitrogen</td>
<td>1000-10 000 μmol/mol</td>
<td>0,5% relative</td>
</tr>
<tr>
<td>Carbon dioxide (CO₂) in air/nitrogen</td>
<td>100 – 1000 μmol/mol</td>
<td>0,5% relative</td>
</tr>
<tr>
<td>Sulphur dioxide (SO₂) in air/nitrogen</td>
<td>1000-10 000 μmol/mol</td>
<td>1% relative</td>
</tr>
<tr>
<td>Sulphur dioxide(SO₂) in air/nitrogen</td>
<td>100 – 1000 μmol/mol</td>
<td>2% relative</td>
</tr>
<tr>
<td>Sulphur dioxide(SO₂) in air/nitrogen</td>
<td>10 – 100 μmol/mol</td>
<td>2% relative</td>
</tr>
<tr>
<td>Nitrogen monoxide (NO) in nitrogen</td>
<td>1000-10 000 μmol/mol</td>
<td>1% relative</td>
</tr>
<tr>
<td>Nitrogen monoxide (NO) in nitrogen</td>
<td>100 – 1000 μmol/mol</td>
<td>2% relative</td>
</tr>
<tr>
<td>Nitrogen monoxide (NO) in nitrogen</td>
<td>10 – 100 μmol/mol</td>
<td>3% relative</td>
</tr>
<tr>
<td>Nitrogen dioxide (NO₂) in air</td>
<td>100 – 1000 μmol/mol</td>
<td>2% relative</td>
</tr>
<tr>
<td>Nitrogen dioxide (NO₂) in air</td>
<td>10 – 100 μmol/mol</td>
<td>3% relative</td>
</tr>
</tbody>
</table>
## Primary reference gas mixtures (PRGMs)

<table>
<thead>
<tr>
<th>Description</th>
<th>Concentration</th>
<th>Expanded uncertainty (k=2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen sulphide (H$_2$S) in nitrogen</td>
<td>1000-10 000 µmol/mol</td>
<td>4% relative</td>
</tr>
<tr>
<td>Hydrogen sulphide (H$_2$S) in nitrogen</td>
<td>100 – 1000 µmol/mol</td>
<td>4% relative</td>
</tr>
<tr>
<td>Hydrogen sulphide (H$_2$S) in nitrogen</td>
<td>10 – 100 µmol/mol</td>
<td>4% relative</td>
</tr>
<tr>
<td>Methane (CH$_4$) in nitrogen/helium</td>
<td>1 – 10 % mol/mol</td>
<td>1% relative</td>
</tr>
<tr>
<td>Methane (CH$_4$) in nitrogen/helium</td>
<td>1000 – 10000 µmol/mol</td>
<td>1% relative</td>
</tr>
<tr>
<td>Methane (CH$_4$) in nitrogen/helium</td>
<td>100 – 1000 µmol/mol</td>
<td>1% relative</td>
</tr>
<tr>
<td>Methane (CH$_4$) in nitrogen/helium</td>
<td>10 – 100 µmol/mol</td>
<td>1% relative</td>
</tr>
<tr>
<td>Methane (CH$_4$) in nitrogen/helium</td>
<td>1 – 10 µmol/mol</td>
<td>1 to 2 % relative</td>
</tr>
<tr>
<td>Propane (C$_3$H$_8$) in nitrogen/helium</td>
<td>1 – 10 % mol/mol</td>
<td>1% relative</td>
</tr>
<tr>
<td>Propane (C$_3$H$_8$) in nitrogen/helium</td>
<td>1000 – 10000 µmol/mol</td>
<td>1% relative</td>
</tr>
<tr>
<td>Propane (C$_3$H$_8$) in nitrogen/helium</td>
<td>100 – 1000 µmol/mol</td>
<td>1% relative</td>
</tr>
<tr>
<td>Propane (C$_3$H$_8$) in nitrogen/helium</td>
<td>10 – 100 µmol/mol</td>
<td>1% relative</td>
</tr>
<tr>
<td>Propane (C$_3$H$_8$) in nitrogen/helium</td>
<td>1 – 10 µmol/mol</td>
<td>1 to 2 % relative</td>
</tr>
<tr>
<td>Automotive emission gases</td>
<td>Multi-component mixtures</td>
<td>1 to 2 % relative</td>
</tr>
<tr>
<td>Stack gas emission gases</td>
<td>Multi-component mixtures</td>
<td>1 to 2 % relative</td>
</tr>
</tbody>
</table>
## 2009 to 2011 Inter-comparisons

<table>
<thead>
<tr>
<th>International Number</th>
<th>Laboratory</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 CCQM-P73</td>
<td>Gas</td>
<td>Gravimetry: 30 to 70 ppm NO in nitrogen</td>
</tr>
<tr>
<td>2 CCQM-K52</td>
<td>Gas</td>
<td>360 ppm CO₂ in air</td>
</tr>
<tr>
<td>3 CCQM-K53</td>
<td>Gas</td>
<td>Gravimetry: 100 ppm O₂ in N₂</td>
</tr>
<tr>
<td>4 CCQM-K51 and Euramet 900a</td>
<td>Gas</td>
<td>5 ppm CO in nitrogen</td>
</tr>
<tr>
<td>5 BIPM.QM-K1</td>
<td>Gas</td>
<td>Ambient level ozone</td>
</tr>
<tr>
<td>6 CCQM-K66</td>
<td>Gas</td>
<td>Methane gas purity</td>
</tr>
<tr>
<td>7 CCQM-K74 and CCQM-P110</td>
<td>Gas</td>
<td>NO 10ppm by FTIR and other</td>
</tr>
<tr>
<td>8 EURO.QM-K4.2</td>
<td>Gas</td>
<td>Ethanol-in-nitrogen</td>
</tr>
<tr>
<td>9 EURAMET 1113</td>
<td>Gas</td>
<td>Preparative study for automotive emission gases</td>
</tr>
<tr>
<td>10 CCQM-K51</td>
<td>Gas</td>
<td>5 µmol/mol CO in N₂ (Pilot Laboratory)</td>
</tr>
<tr>
<td>11 APMP.QM-K41</td>
<td>Gas</td>
<td>10 µmol/mol H₂S in nitrogen</td>
</tr>
<tr>
<td>12 CCQM-K74</td>
<td>Gas</td>
<td>10 µmol/mol NO₂ in nitrogen</td>
</tr>
</tbody>
</table>
International key comparison

CCQM-K51 Carbon monoxide in nitrogen

Degrees of equivalence (%) vs Laboratory
Organic (Bio)

- GC-FID/ GC-MS/ GCXGC-TOFMS/ HPLC/ LC-MS\(^n\)/ HPLC-DAD, Fluorescence/ DSC/ TGA/ KF
- Gravimetry, titrimetry, IDMS
- Purity assignment
- Reference materials (forensic and wine industry)
  - Aqueous Ethanol solutions (Concentration range: 0.01 mg/100 g – 20 g/100 g)
  - Sodium Fluoride (0.3 g/ 100ml to 3 g/ 100 ml)
- Proficiency testing schemes
  - Provincial: DOH Chemistry laboratories
  - National: Afrimets EtOH PT scheme (AFRIMETS.QM-K27)
- Food safety
- POPs
Inorganic Chemistry

• **What’s in it?**
  - ICP-MS, LA-ICP-MS, ICP-OES
  - Trace and ultra-trace element analysis in food and environmental samples
    - Se in wheat
    - Sn in tomato paste
    - Cd in rice
    - Pd, Cd, Ca, Fe in wine
    - Ca, Fe, Cu, Zn in fat soybean
    - Cd, Fe, Pb and Zn in bovine liver
    - Cd, Cr, Ni, Hg, Pb and Pt in algae
  • Metals in manufacturing materials
    - Cr, Mn, Mo and Ni in low alloy steel
    - RoHS (Cd, Cr, Hg and Pb) in polypropylene
    - Pb in lead-free solder

• **Application:** Reference analysis and certification of materials/foods
Environmental projects (POPs)

**CROC**
- Consortium for the Rehabilitation of the Oliphant River Catchment
- SANParks
- Crocodile deaths
- Ecotoxicology of the Sabie River
- Biodiversity modelling (Italians)

**NIP & POPs**
- Stockholm Convention
- NIP
- Dioxins/ furans
- PCBs
- PAHs
- Pesticides
- BFRs
- Speciation
- Heavy metals
- Establishing a Centre of Excellence for Toxic Substances (Dioxins)
- Water quality & the right to clean air

**Collaborative projects**
- SAPS FSL
- AFMA
- CSIR
- UP
- NWU
- University Liége
- Norwegian collaboration
- DEA (NRL)
- DWA RQS
- LNE
- METAS?
Meeting SA and International Food Safety Regulations

- **Nutritional content**
  - Protein content: amino acid

- **Mycotoxins**
  - Multiple toxins in maize & wine

- **Pesticide residues & Toxic elements**
  - Fruits & vegetables

- **Veterinary drug residues**
  - Chloramphenicol in pork and milk

- **Processing contaminants**
  - Melamine in infant formula & milk, methoxy-pyrazines in wine

- **Industrial & environmental chemicals**
  - POPs

- **SAGL PT scheme**
  - Nutritive elements

- **Wine industry**
  - PT schemes/reference measurement ISO 17025 labs

- **Veterinary Institutes & Meat industry**
  - Dairy & wine industry

- **Heavy metals & Toxic elements**
Projects in bio-analysis

- Traceable protein measurements to support the diagnostics and pharmaceutical sectors

- Microbiology in Africa
  - investigate traceability
  - Counting the bacteria based on DNA content
  - Imaging techniques for cell counting
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<thead>
<tr>
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<tbody>
<tr>
<td>1 CCQM-K55.a</td>
<td>Org</td>
<td>Characterization of organic substances for chemical purity: b-Estradiol</td>
</tr>
<tr>
<td>2 CCQM-P90</td>
<td>Org</td>
<td>CAP in Milk</td>
</tr>
<tr>
<td>3 CCQM-P86.1</td>
<td>Org &amp; IPS</td>
<td>Total Se and Se-speciation analysis of Se-rich wheat flour</td>
</tr>
<tr>
<td>4 CCQM-K55.b</td>
<td>Org</td>
<td>Characterization of organic substances for chemical purity: Aldrin</td>
</tr>
<tr>
<td>5 CCQM-P122</td>
<td>Org</td>
<td>Chloramphenicol in pig muscle</td>
</tr>
<tr>
<td>6 CCQM-KC-79</td>
<td>Org</td>
<td>Comparison of Value-Assigned CRMs and Proficiency Testing Materials for Ethanol in Aqueous Matrix, 0.1 mg/g to 500 mg/g</td>
</tr>
<tr>
<td>7 FAPAS #1795</td>
<td>Org</td>
<td>Ochratoxin-A in wine</td>
</tr>
<tr>
<td>8 FAPAS #3027</td>
<td>Org</td>
<td>Melamine in milk powder</td>
</tr>
<tr>
<td>9 CCQM-P55.1</td>
<td>Bio</td>
<td>Mole concentration of peptides in solution</td>
</tr>
<tr>
<td>10 PEAAL-Comparison no. 002/2010</td>
<td>Org</td>
<td>Interlaboratory comparison in anhydrous bioethanol</td>
</tr>
<tr>
<td>11 APMP_QM_P19</td>
<td>Org</td>
<td>Melamine in milk powder</td>
</tr>
<tr>
<td>12 CCQM-P129</td>
<td>Org</td>
<td>Determination of ethanol and water in bioethanol fuel</td>
</tr>
</tbody>
</table>
Sample 1 Peptide

[Graph showing peptide concentrations]
International melamine comparison
## 2009 to 2010 Inter-comparisons

<table>
<thead>
<tr>
<th>International Number</th>
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</tr>
</thead>
<tbody>
<tr>
<td>1 CCQM-K67</td>
<td>SA</td>
<td>Composition of a Fe-Ni binary alloy thin film</td>
</tr>
<tr>
<td>2 VAMAS TWA33#1</td>
<td>SA</td>
<td>Determination of the shape, size and size distribution of nano-filler particles</td>
</tr>
<tr>
<td>3 VAMAS TWA2#</td>
<td>SA</td>
<td>VAMAS 2009 ORGANIC DEPTH PROFILING INTERLABORATORY STUDY</td>
</tr>
<tr>
<td>4 CCQM K67/P108</td>
<td>SA</td>
<td>Ni/Fe in thin film alloys</td>
</tr>
<tr>
<td>5 VAMAS TWA37</td>
<td>SA (Nano)</td>
<td>Determination of reproducibility and repeatability of grain size measurement by Electron Back Scattered Diffraction (EBSD)</td>
</tr>
<tr>
<td>1 CCQM-P119</td>
<td>Inorganic</td>
<td>Pb in Pb-free solder</td>
</tr>
<tr>
<td>2 CCQM-K60</td>
<td>Inorganic</td>
<td>Analysis of total Se in wheat flour</td>
</tr>
<tr>
<td>3 CCQM-K56</td>
<td>Inorganic</td>
<td>Ca, Fe, Zn and Cu in Whole Fat Soybean Powder</td>
</tr>
<tr>
<td>4 AFRA IV-12</td>
<td>Inorganic</td>
<td>Research Reactor Programme for Socio-economic Development, Trace elements in nutrition additives, medicinal plants and food materials</td>
</tr>
<tr>
<td>5 INCT-OSTL-5 INCT-PVTL-6</td>
<td>Inorganic</td>
<td>Collaboration study on the certification of new tobacco leaves candidate reference materials</td>
</tr>
<tr>
<td>6 CCQM-K57/P118</td>
<td>Inorganic</td>
<td>Toxic Metals (Pt, Ni, Cr, Cd, Hg &amp; Pb) in Algae</td>
</tr>
<tr>
<td>7 CCQM-P106</td>
<td>Inorganic</td>
<td>Cd, Cr, Hg and Pb in Polypropylene</td>
</tr>
<tr>
<td>8 CCQM-K87</td>
<td>Inorganic</td>
<td>Chromium, cobalt and lead (pure elemental solution)</td>
</tr>
<tr>
<td>9 SADC Water PT No 7</td>
<td>Inorganic</td>
<td>Al, As, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Zn in water</td>
</tr>
</tbody>
</table>
Pb in solder
Toxic metals in Algae

Pt in P118 Algae (k=2)

Concentration (mg/kg)
Surface Analysis

Techniques

Applications

- Imaging of surface topography and chemical species up to nano scale
- Elemental and chemical composition of the surface
- Crystal structure
- Sputter depth profiles on thin films and coatings from a few nm to microns (thickness and composition)
- Samples include: catalysts, carbon nanotubes, metal alloys, glass, automotive coatings etc…
- Polymer analysis: biopolymers and their nanocomposites, polyolefins, etc.
- Corrosion metrology: Vapour Phase Corrosion Inhibitor testing for the metal packaging industry
Projects supporting industry

- Improve measurement accuracy
  - XPS SiO$_2$ layers
  - XPS glass standards

- Provide accurate measurements to industry
  - Nano-crystalline Si
  - Imaging, bioanalysis
  - Pharmaceuticals

- Catalysts
  - Failure
  - Corrosion

- Metals beneficiation
  - Toxic elements (plastics)
  - Mining (speciation)

- Polymers & plastics
  - Bio & oxo-degradable plastics
  - Food
  - Product development
Projects supporting dissemination

- **PT schemes**
  - DoH Chemical laboratories
  - AFRIMETS
  - Pesticides in water

- **Water PT**
  - SADC: heavy metals in water

- **Gas PSMs**
  - PSGMs & PRGMs

- **Org CRM s**
  - EtOH and EtOH in glucose
  - NaF

- **IPS CRM s**
  - Elemental calibration solutions