



# **Food and Drug Assurance Laboratories (Pty) Ltd**

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# Discussion points

- Brief overview of chemical residues.
- Why do chemical residue occur in end products?
- Health risks associated with chemical residues
- Risks associated with chemical residues
- Analytical methods
- Maximum residue levels (MRL's)
- Accreditation in the laboratory
- 3 Min video of the inside of the laboratory



# Veterinary Drug Residues in Milk and Milk products

- Trace amounts of pharmaceutical substances that remains after treating dairy animals.
  - Antibiotics
  - Antiparasitic
  - Anticoccidials
  - Non-steroidal anti-inflammatory drugs (NSAIDs)
  - Insecticides
- Environmental chemicals is also of concern
  - Pesticides – from silage and grain products
  - Mycotoxins – especially aflatoxin M1
- Contaminants
  - Melamine – contaminant added to the products
  - PFAS – The forever chemicals



# Health and Operation Risks

- Allergic reactions
- Antibiotic resistance development
- Disruption of gut microbiota
- Toxicity risks from chronic exposure
- Legal and Regulation Issues (locally and internationally)
- Interference with dairy fermentation processes
- Loss of consumer trust



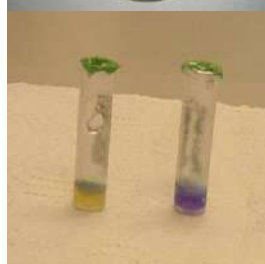
# Sources of residues in milk and milk products

- Improper withdrawal periods after treatment
- Off-label use or overdose
- Poor milking hygiene
- Inadequate record-keeping
- Unregulated feed additives
- Carry over of medicated feed batches to non-medicated feed batches
- Environmental contaminants



# Detection Methods

- Microbial inhibition tests – e.g. Delvotest
- ELISA -enzyme-linked immunosorbent assay – e.g. Neogen, Euro Proxima, Ridascreen
- HPLC - high pressure liquid chromatography with various detectors
  - UV-VIS - non-destructive which **measures the amount of ultraviolet or visible light absorbed** by the sample.
  - FLD - measuring the fluorescence emitted by certain compounds when they are exposed to excitation light (Xenon lamp).
  - MS or MS/MS – (refer to LC-MS/MS) A tandem mass spectrometry is **a two-step technique used to analyse a sample** either by using two or more mass spectrometers





# Screening Tests versus Confirmatory Tests

Aspect	Screening Test	Confirmatory Tests
Purpose	Rapid, initial detection of potential residues	Definitive identification and quantification of specific residues
Sensitivity	Moderate to high	Very high sensitivity and selectivity
Specificity	Lower – may produce false positives	High specificity (low false rate)
Speed	Fast – few minutes	Slower – several hours
Cost	Low cost per sample	High cost – high capital and skilled personnel
Complexity	Simple – often field-usable kits	Complex – requires laboratory infrastructure
Regulatory Acceptance	Routine monitoring not legally binding	Legal confirmation and reporting to authorities



# Maximum residue Limits (MRLs)

## ■ Limits set by:

- Import Country – Every country can set their own MRL limits
- Codex Alimentarius – International Organisation (FAO/WHO)
- SA – Department of Health
- EU – 27 Participating countries in Europe
- US FDA - Food and Drug Administration body of the United State Congress

## ■ Farmers must adhere to withdrawal periods stated on the label of a product or as prescribed by the responsible veterinarian.



**DEPARTMENT OF HEALTH  
DEPARTEMENT VAN GESONDHEID**

No. R. 215

10 March 2006

**FOODSTUFFS, COSMETICS AND DISINFECTANTS ACT, 1972 (ACT NO.  
54 OF 1972)**

**REGULATIONS GOVERNING THE MAXIMUM LIMITS FOR VETERINARY MEDICINE  
AND STOCK REMEDY RESIDUES THAT MAY BE PRESENT IN  
FOODSTUFFS**

Sulphonamides., (All substances belonging to the sulphonamide group)	All food producing species.....	Fat, kidney, liver, milk and muscle.....	0.1	The combined total residues of all substance: within the sulphonamide group shall not exceed 0.1
Testosterone.....	Cattle.....	Fat, kidney, liver and muscle.....	Not specified **	
Tetracyclines..... (Group: oxytetracycline, chlortetracycline and tetracycline)	All food producing species.....	Kidney..... Liver..... Muscle.....	1.2*** 0.6*** 0.2***	The combined total residues of all substances within the tetracycline group shall not exceed the limits indicated
	Cattle.....	Milk.....	0.1***	
	Chickens.....	Eggs.....	0.4***	
	Fish * applies only to oxytetracyclin...	Muscle.....	0.1***	



# Multi residue methods versus single methods

- Residue methods:

- **Single method analysis;** One analyte per extraction method.

- Normally very accurate, but very costly if multiple analyte must be included in monitoring plan

- **Multi residue methods**

- More than one analyte sometimes >120 veterinary drugs per method including various groups.
  - More cost effective method, but some analytes might not be recovered during the extraction method



# Monitoring program for milk and milk products

- Risk based system – if it was not used, it cannot be detected! Fewer analytes – lower costs!
- Should include:
  - Antimicrobials – be careful of metabolites
  - Anthelmintics – normally long withdraw periods
  - Non-steroidal anti-inflammatory drugs – low limits applicable
  - Insecticides
  - Environmental contaminants
  - Banned substances
- Milk products
  - Cream, yoghurt, butter - different profile applicable as for milk – more fat soluble compounds is more prevalent



# What does accreditation mean for the client?



*ISO 17025 is the accreditation similar to the ISO 9000 or ISO 18000 system of certification*

- Technical competence is checked and verified
- Confidentiality of results
- Risks management system is in place (2017 version)
- Proficiency testing is done and monitored
- Instrumentation is checked and calibrated
- Records are maintained
- [www.sanas.co.za](http://www.sanas.co.za) – accredited facilities





# Validation of Methods

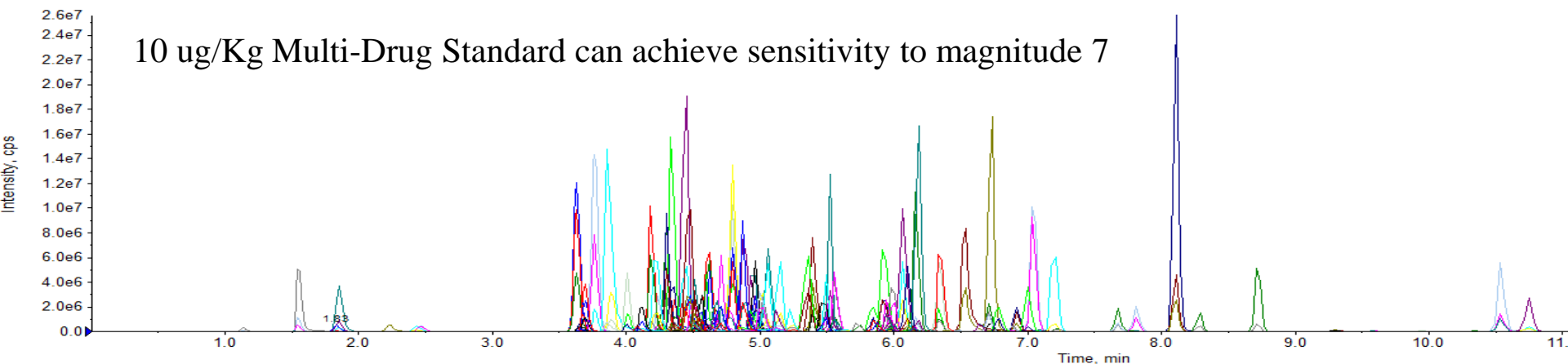
The following technical parameters must be evaluated and included in the validation report

- Estimation of the uncertainty of the results
- Detection limit/quantification limit
- Selectivity of the method
- Linearity of the range for quantitative results
- Repeatability/reproducibility
- Robustness of the method





# Validated Multi Drug Method



## ■ Method Capabilities

- Detection of >120 analytes/drugs
- 12 antibiotic classes (*Eg*: Coccidiostats, Tetracyclines, Macrolides, 3 groups of anthelmintics plus metabolites, *etc.*)
- Various biological species
- Validated against international requirements (EU and Codex Alimentarius)

<b>ANTIBIOTICS</b> (Concentration in µg/kg)	<b>Aim 96</b> <b>(FARM</b> <b>TEST)</b>	<b>BRT</b> <b>“BLUE</b> <b>STAR”</b>	<b>DELVO</b> <b>TEST SP</b>	<b>CHARM</b> <b>II</b>	<b>HPLC</b> <b>UV/FLD</b>	<b>EU</b> <b>MRL</b>	<b>U.S.</b> <b>MRL</b>	<b>SA</b> <b>MRL</b>
<b>BETA LACTAMS</b>								
Amoxicillin	4	-	3	10	10	4	10	4
<b>Ampicillin</b>	<b>4</b>	<b>2</b>	<b>4</b>	<b>9</b>	<b>10</b>	<b>4</b>	<b>10</b>	<b>4</b>
Cephapirin	10	-	8	4.5	-	-	20	50
Penicillin G	3	.003	3	4.8	10	4	5	4
<b>SULFA DRUGS</b>								
Sulfamethoxine	10	-	100	4	30	100	10	100
<b>Sulfamethazine</b>	<b>15</b>	<b>60</b>	<b>500</b>	<b>10</b>	<b>30</b>	<b>100</b>	<b>10</b>	<b>100</b>
<b>TETRACYCLINES</b>								
<b>Chlortetracycline</b>	<b>150</b>	<b>600</b>	<b>500</b>	<b>25</b>	<b>30</b>	<b>100</b>	<b>30</b>	<b>100</b>
Doxycycline	50	-	-	100	30	100	-	100
Oxytetracycline	100	500	200	30	30	100	30	100
Tetracycline	100	250	200	10	30	100	30	100
<b>MACROLIDES</b>								
Erythromycin	100	75	400	25	-	-	50	50
Tylosin	20	20	100	50	50	50	50	100
<b>AMINOGLYCOSIDES</b>								
Gentamycin	30	200	250	24	-	200	30	50
Streptomycin	500	400	4000	12.5	50	200	125	50
<b>AMPHENICOL</b>								
<b>Chloramphenicol</b>	<b>&gt;1000</b>	<b>4000</b>	<b>5000</b>	<b>0.5</b>	<b>10</b>	<b>0</b>	<b>0</b>	<b>10</b>



# Scope of the laboratory

- Identification and Quantification of small organic molecules in both food and feed
- Laboratory make use of chromatography as the separation mode – ability to discriminate between two different compounds
- Specialises in Confirmatory Techniques





# Overview of the services of the Laboratory

## *Residues of Veterinary drugs*

- For Registration of drugs
- Monitoring of residues
- Therapeutic levels

## *Medicated Feed*

Antimicrobials, Growth stimulants,  
Coccidiostats, Multi antimicrobial  
and Ionophores

## *Chromatography Confirmatory Tech.*

GC-MS/MS  
HPLC-UV/FLD  
LC-MS/MS (x4)  
LC-TOF

*Environmental Analyses  
Pesticides/Insecticides  
Dedicated Research projects*

*Mycotoxin Analyses in  
Feed and Biological  
Matrices (for milk M1)*

ISO 17025 Accredited Laboratory



## We analyse accurately!

*We deliver our analytical services to all parts of the globe. Whether you are a first time consumer, have worked with us before but have a new sample or test, or simply have a question, we look forward to hear from you.*

