RoHS analysis with Fischerscope XRAY instruments

for the determination of hazardous substances in electrical and electronic equipment
according to IEC62321 standard

AGENDA

1	Determination of hazardous substances in electrical and electronic equipment according to IEC62321
2	RoHS Analysis
3	Calibration Device RoHS
4	Advantages of Fischer RoHS Setup
5	Question & Answer Session

RoHS Analysis

RoHS = Restriction of the use of hazardous substances in electrical and electronic equipment.

According to Directive 2011/65 EU as amended in 2015, electronic equipment must be tested for the following substances lead, mercury, cadmium, chromium (VI) and certain organic substances containing bromine.

Such hazardous materials can be challenging to manage at the end of the product's life cycle, and because of that the Directive restricts their use at the initial stage, i.e. during product's manufacturing, and helps to keep them out of the waste stream.

The **Fischerscope XRAY RoHS Setup** in conjunction with suitable **Fischerscope XRAY equipment** allows fast and cost-effective screening with classification of the parts to be tested into groups according to the IEC 62321 guidelines:

BL = Samples with contents below the limit values
X = Samples with contents in the limit range
OL = Samples with contents above the limit values

For the samples falling into the groups **BL** or **OL**, a simple analysis can thus be carried out.
Only for samples falling into group **X**, no clear assignment with regard to the limit values, other more time-consuming and costly procedures with higher precision (e.g. ICP-MS, ICP-OES) have to be applied.

RoHS: How to Measure RoHS Samples

Short instruction for the FISCHERSCOPE® on How to Measure RoHS Samples

Switching on the Instrument

1. Plug the FISIM in an USB socket.
2. First switch on the instrument, then the PC and the monitor.
3. If the key switch is present, turn it to the right.
4. To start the WinFTM software, click  .

Positioning Specimen

1. Open the cover of the measurement chamber.
2. Position the specimen into the measurement chamber and focus the video image.
3. Close the cover of the measurement chamber.

Available Measurement Applications

When delivered to the customer, the following measurement applications are available on the instrument:

Nr	Name	Symbol	Application
1	Polymer		All plastics such as PE, PVC, ABS, Polystyrene, Silicone, Resins, etc.
2	Metals		All **uncoated** metal materials such as steel, copper alloys, aluminum alloys and tin compounds.
3	Composites		All samples consisting of layer systems and samples that do not fit into the two other groups.

The table outlines the delivery status of the instrument. You can change the symbols and measurement applications according to your needs.

Starting Measurements

1. To select the correct measurement application, click the appropriate symbol. The measurement application will be loaded and a short test measurement will be started.
   If you need to terminate the running measurement, press the function key **F3**.
   The window *Order No./Operator* appears.
2. If desired, you can enter an order number and the name of the operator in the window *Order No./Operator*.
3. Click **OK**.
   The measurement will be performed.

If the Measurement does not Start

If the measurement is not started and the window *Open* appears instead, enable the communication between instrument and WinFTM

1. In the window *Open* click **Cancel**.
2. In the WinFTM main window select **General** > **Communication ON**.

The Report

When the measurement is finished, the *RoHS Test Report* appears. For each element the report shows whether the RoHS requirements are met.

► To print the report click  .

► To save the report click  .

RoHS Test Report

	Pb ppm	Hg ppm	Cd ppm	Cr ppm	Br ppm
Concentrations	13.37	62.48	N.d.	N.d.	72267
3*σ	10.36	11.78	26.37	23.03	606.0
RoHS Status	BL	BL	BL	BL	X

BL: Below Limit[1]
OL: Over Limit[1]
X: Inconclusive[1] -> further investigations

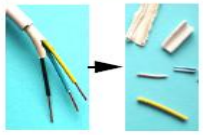
[1]According to IEC 62321

► To close the report click . You can now start a new measurement.

RoHS

NOTES:

- If a FISCHERSCOPE X-RAY XDV-SD(D) is used for the measurement, use a beam trap when making measurements on plastics. If the plastic sample is directly placed on the measuring stage, measurement errors may occur because the stage also generates x-ray fluorescence.
- When measuring on very thin foil material, stack several foils to obtain sufficient information for evaluation.
- When measuring on plastics, all metal parts that are glued or clamped to plastic materials, must be removed to ensure proper measurements.



IEC 62321

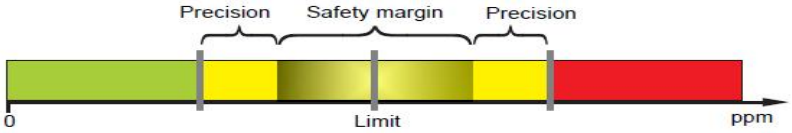
Result information referring to IEC 62321 is presented for each RoHS element, all figures in ppm:

	Polymer	Metals	Composite
Pb	BL ≤ (700-3σ) < X < (1300+3σ) ≤ OL	BL ≤ (700-3σ) < X < (1300+3σ) ≤ OL	BL ≤ (500-3σ) < X < (1500+3σ) ≤ OL
Hg	BL ≤ (700-3σ) < X < (1300+3σ) ≤ OL	BL ≤ (700-3σ) < X < (1300+3σ) ≤ OL	BL ≤ (500-3σ) < X < (1500+3σ) ≤ OL
Cd	BL ≤ (70-3σ) < X < (130+3σ) ≤ OL	BL ≤ (70-3σ) < X < (130+3σ) ≤ OL	LOD[1] < X < (150+3σ) ≤ OL
Cr	BL ≤ (700-3σ) < X	BL ≤ (700-3σ) < X	BL ≤ (500-3σ) < X
Br	BL ≤ (300-3σ) < X		BL ≤ (250-3σ) < X

1. LOD = Limit of detection

Screening procedure according to IEC 62321: The RoHS status of each analysis result is assigned to one of these three classifications.

Surely below the RoHS limit	Safety margin (±30% or ±50% of the RoHS limit) and the instrument precision (3σ)	Surely above the RoHS limit
Below limit (BL)	Inconclusive (X), further investigations necessary	Over limit (OL)



Error Messages

- An error message appears, if the measurement is terminated by the user.
- *Spectrum invalid*: Check, whether the appropriate RoHS symbol has been clicked. View the spectrum of your sample in spectrum mode, other elements may be present. Contact HELMUT FISCHER.
- *No Match*: The instrument was not able to find an application that fits the sample to be measured. Contact HELMUT FISCHER to adjust the existing measuring application or to set up a new measuring application.

HELMUT FISCHER GMBH
INSTITUT FÜR ELEKTRONIK UND MESSTECHNIK

Industriestrasse 21
D-71069 Sindelfingen

www.helmut-fischer.com

On our home page
www.helmut-fischer.com
you will find the addresses of our sole agencies and subsidiary companies around the globe.

RoHS analysis

RoHS: Which elements are measured?

Limit values from Directive 2011/65 EU and substances added in 2015 (RoHS3)

Lead	< 0.1 % (1000 PPM)
Mercury	< 0.1 % (1000 PPM)
Cadmium	< 0.01 % (100 PPM)
Hexavalentes Chrom (CrVI)	< 0.1 % (1000 PPM)
Polybrominated biphenyls (PBB)	< 0.1 % (1000 PPM)
Polybrominated diphenyl ethers (PBDE)	< 0.1 % (1000 PPM)

Substances ingested in 2015, not measurable with XRF:

1. Bis(2-ethylhexyl)phthalat (DEHP), 0,1 % – Used, among other things, as a plasticiser in PVC
2. Benzylbutylphthalat (BBP), 0,1 % – Used, among other things, as a plasticiser in plastics
3. Dibutylphthalat (DBP), 0,1 % – Used, among other things, as a plasticiser in plastics
4. Diisobutylphthalat (DIBP), 0,1 % – Used, among other things, as a plasticiser in plastics

RoHS analysis

RoHS: Which XRAY systems can be used for measurement?

The RoHS setup can be used on Fischerscope XAN 250,XDV-SDD & XDAL 600.
Customized applications are available for Fischerscope XAN 220 and XDV-µ.

XAN 250
Measuring head below, for single samples,
Collimator 1.0mm (standard Settings)

XDV-SDD
Measuring head top, movable table,
single samples and series
Collimator 1.0mm (standard setting)

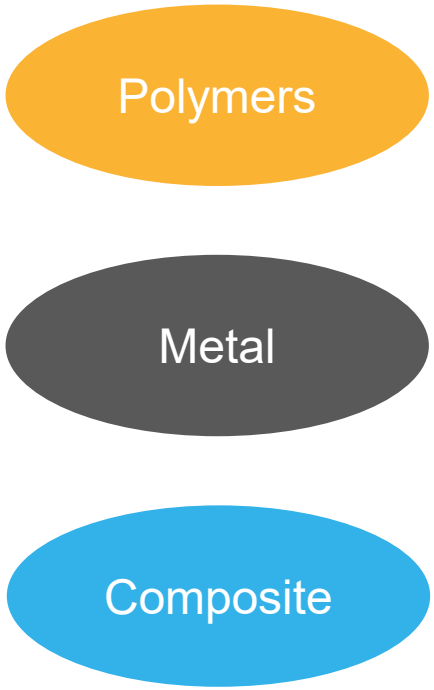
XDAL-600
Measuring head top, Manual operated
scissor table (lab jack)
Collimator 1.0mm (standard setting)

RoHS analysis

Fischerscope XRAY RoHS Setup

Predefined substance classes

The Fischerscope XRAY RoHS setup uses predefined substance classes.



All plastics such as PE, PVC, ABS, polystyrenes, silicones, resins, etc..

All uncoated metal materials, such as steel, copper, tin and aluminium alloys, etc. and others.

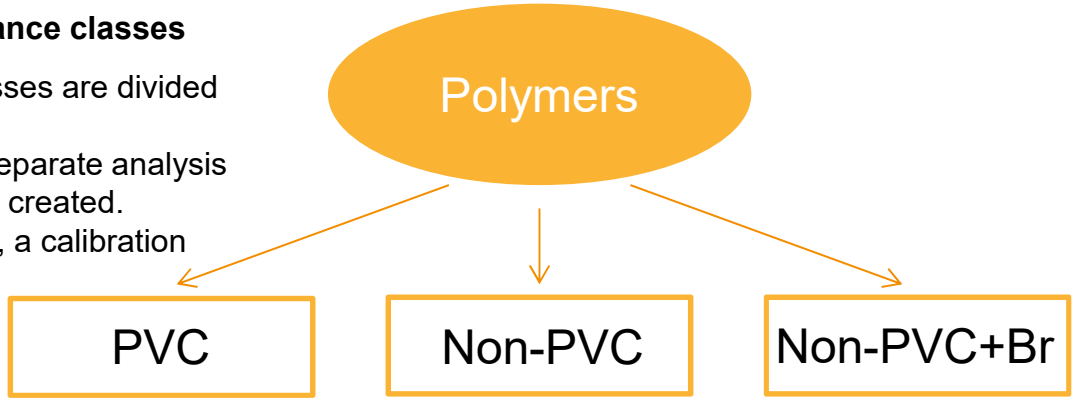
Composite materials, layer systems and samples that cannot be assigned to the polymer and metal groups.

RoHS analysis

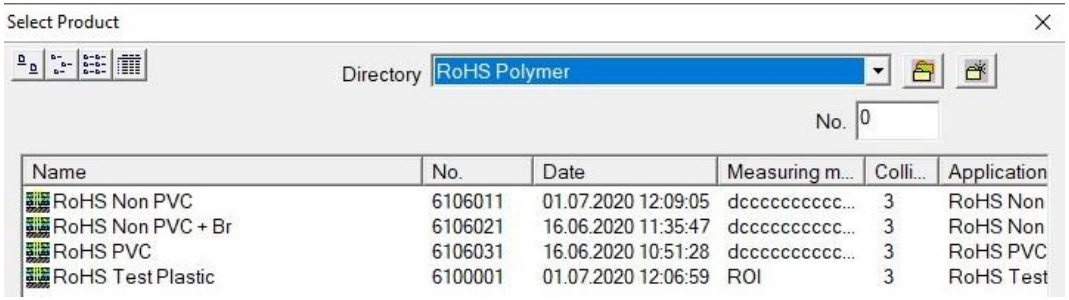
Fischerscope XRAY RoHS Setup

Predefined substance classes

The substance classes are divided into subgroups,
for each of which separate analysis routines have been created.
For each subgroup, a calibration with several several standards is stored.



All plastics, such as PE, PVC, ABS, polystyrenes, silicones, resins, etc.

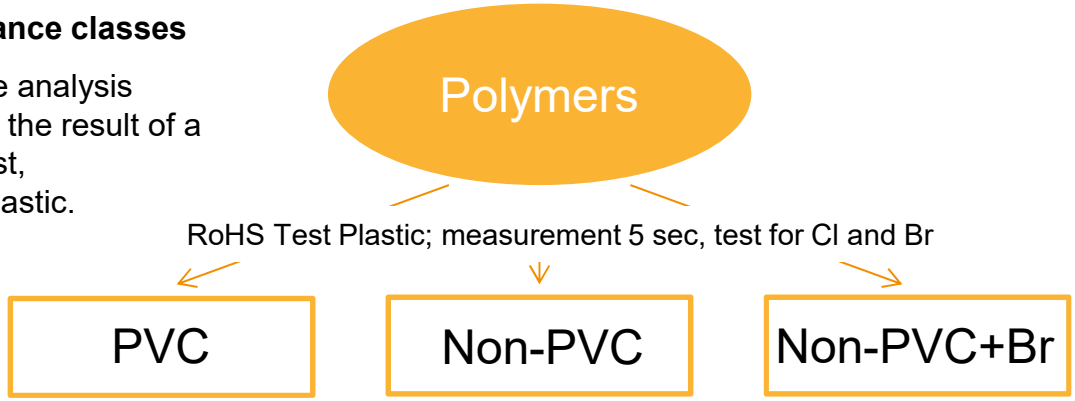


RoHS analysis

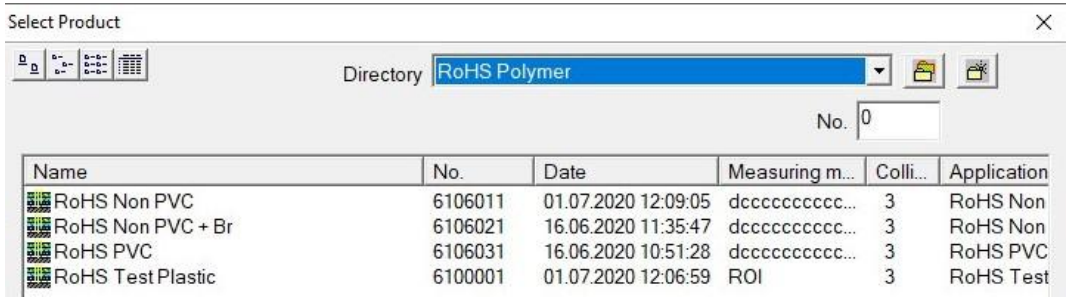
Fischerscope XRAY RoHS Setup

Predefined substance classes

The selection of the analysis routine is based on the result of a substance class test, here: RoHS Test Plastic.



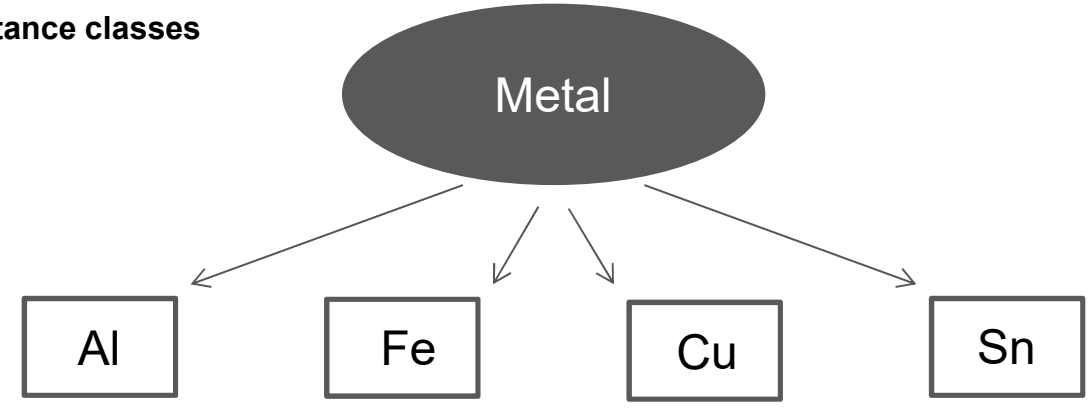
All plastics, such as PE, PVC, ABS, polystyrenes, silicones, resins, etc.



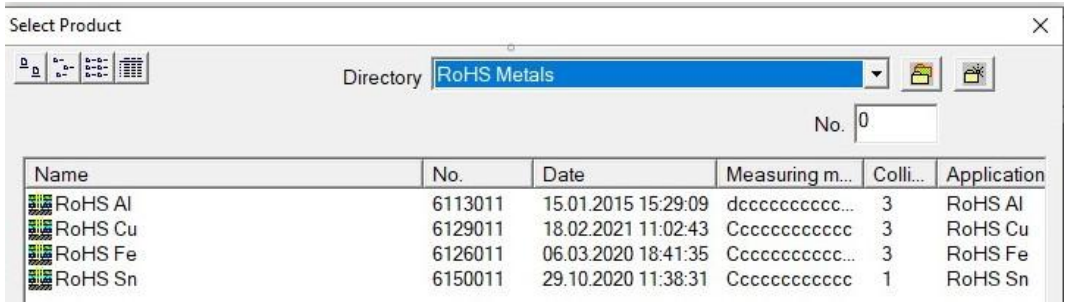
RoHS analysis

Fischerscope XRAY RoHS Setup

Predefined substance classes



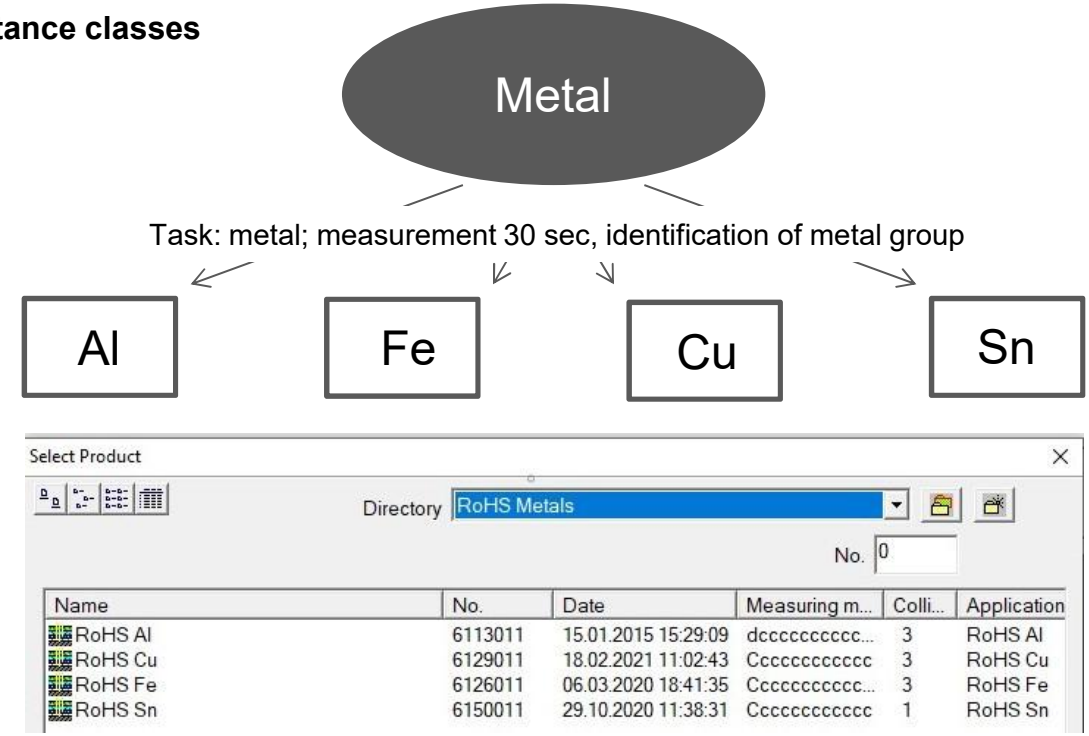
All uncoated metal materials such as steel, copper, tin and aluminium alloys and others.



RoHS analysis

Fischerscope XRAY RoHS Setup

Predefined substance classes



All uncoated metal materials such as steel, copper, tin and aluminium alloys and others.

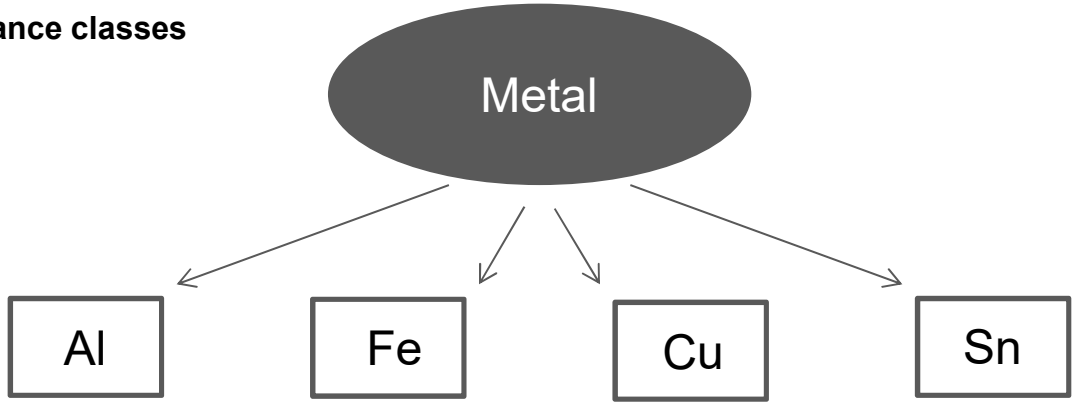
Task: metal
Measurement to identify the metal group, 30sec;
automatic selection and start of the measuring programme
according to metal identification;
Measurement 300 sec

RoHS analysis

Fischerscope XRAY RoHS Setup

Predefined substance classes

For each subgroup a calibration with several standards is stored.



All uncoated metal materials such as steel, copper, tin and aluminium alloys and others.

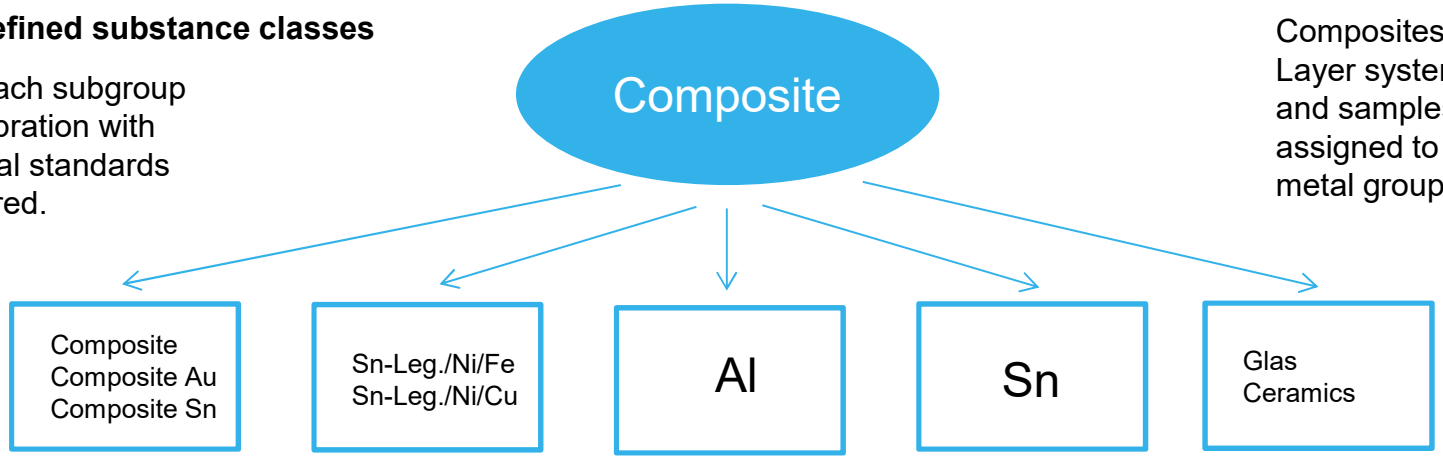
Calibration	2 Al alloys	4 Fe alloys	2 Brass Alloys + 1 alloy with Cr	3 Sn alloys

RoHS analysis

Fischerscope XRAY RoHS Setup

Predefined substance classes

For each subgroup
a calibration with
several standards
is stored.



Composites,
Layer systems
and samples that cannot be
assigned to the polymer or
metal groups.

Calibration

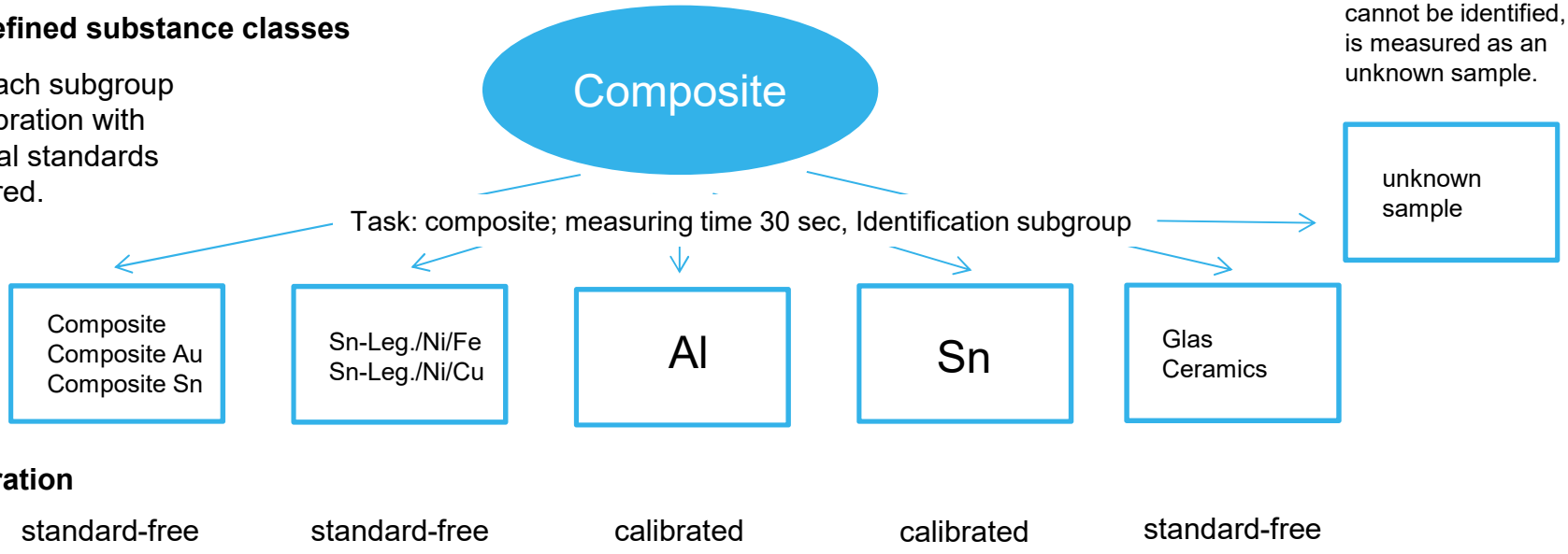
standard-free	standard-free	calibrated	calibrated	standard-free

RoHS analysis

Fischerscope XRAY RoHS Setup

Predefined substance classes

For each subgroup a calibration with several standards is stored.



If the material group cannot be identified, it is measured as an unknown sample.

Calibration

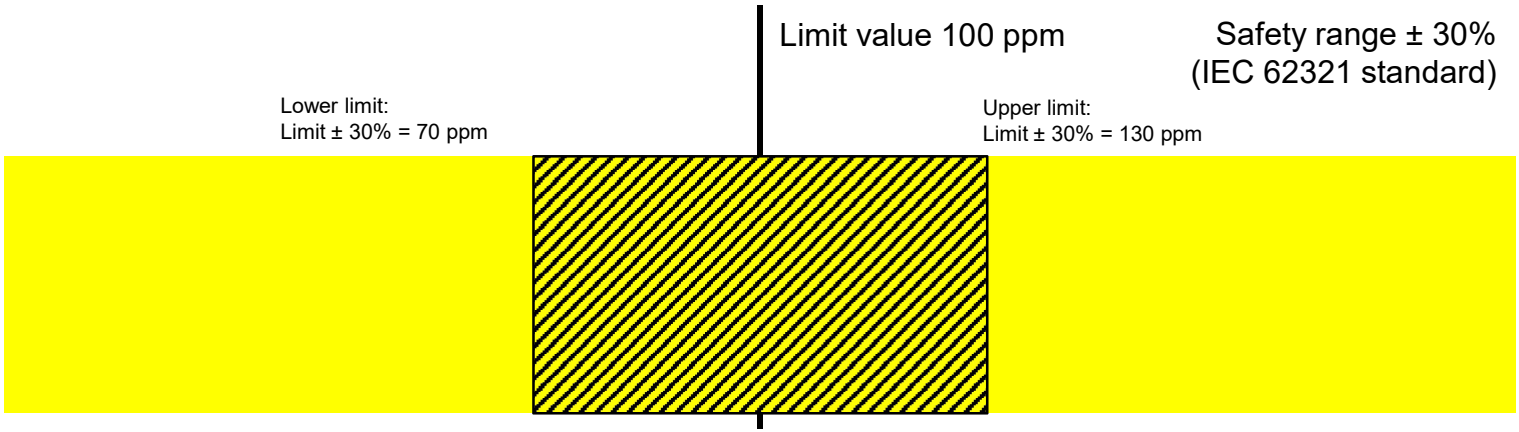
standard-free	standard-free	calibrated	calibrated	standard-free

RoHS analysis

Fischerscope XRAY RoHS Setup - Evaluation

Definition of the limit value, with upper and lower safety range

Example: Cadmium
Limit value 100 ppm



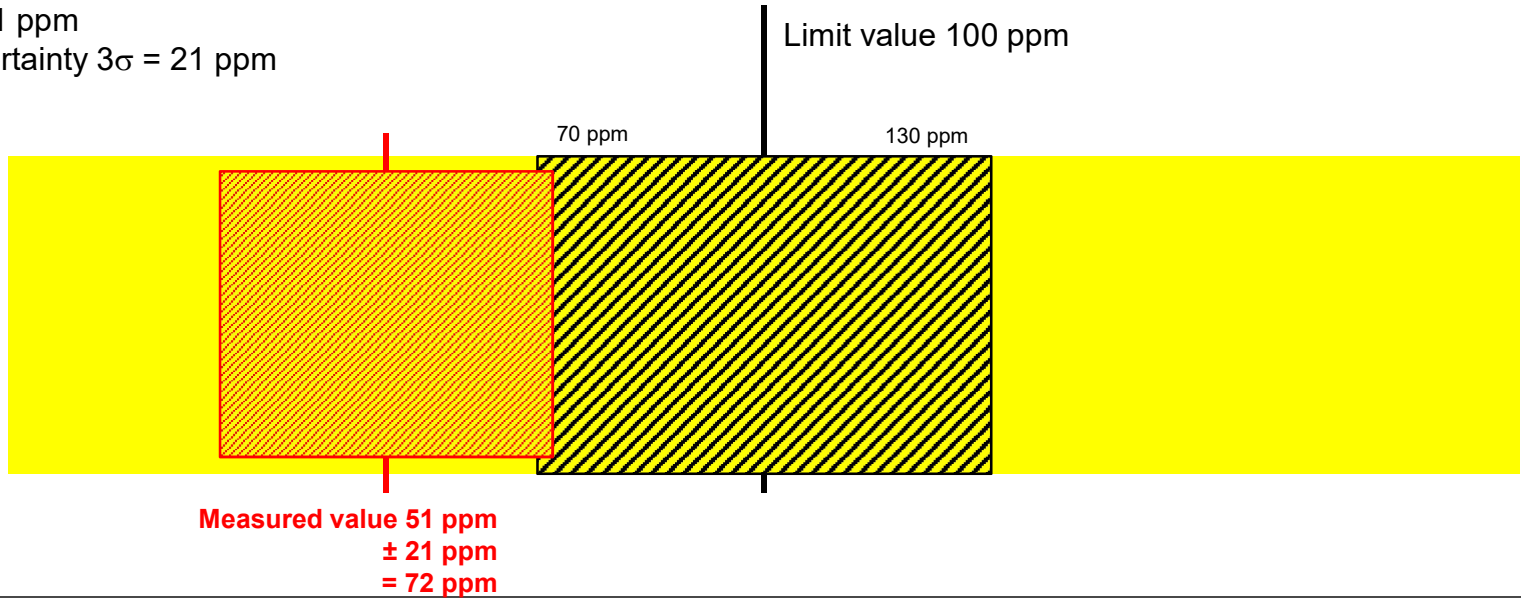
The large safety range is caused by uncertainties in the detection of unknown samples.
Physical effects in the sample matrix (scattering, secondary excitation) can strongly influence the measurement results. These effects are independent of the measurement time and can therefore not be compensated by a longer measurement time.

RoHS analysis

Fischerscope XRAY RoHS Setup - Evaluation

Extension of the safety range by measurement uncertainty

Example: Cadmium
Measuring time: 100 sec
Measured value: 51 ppm
Measurement uncertainty 3σ = 21 ppm



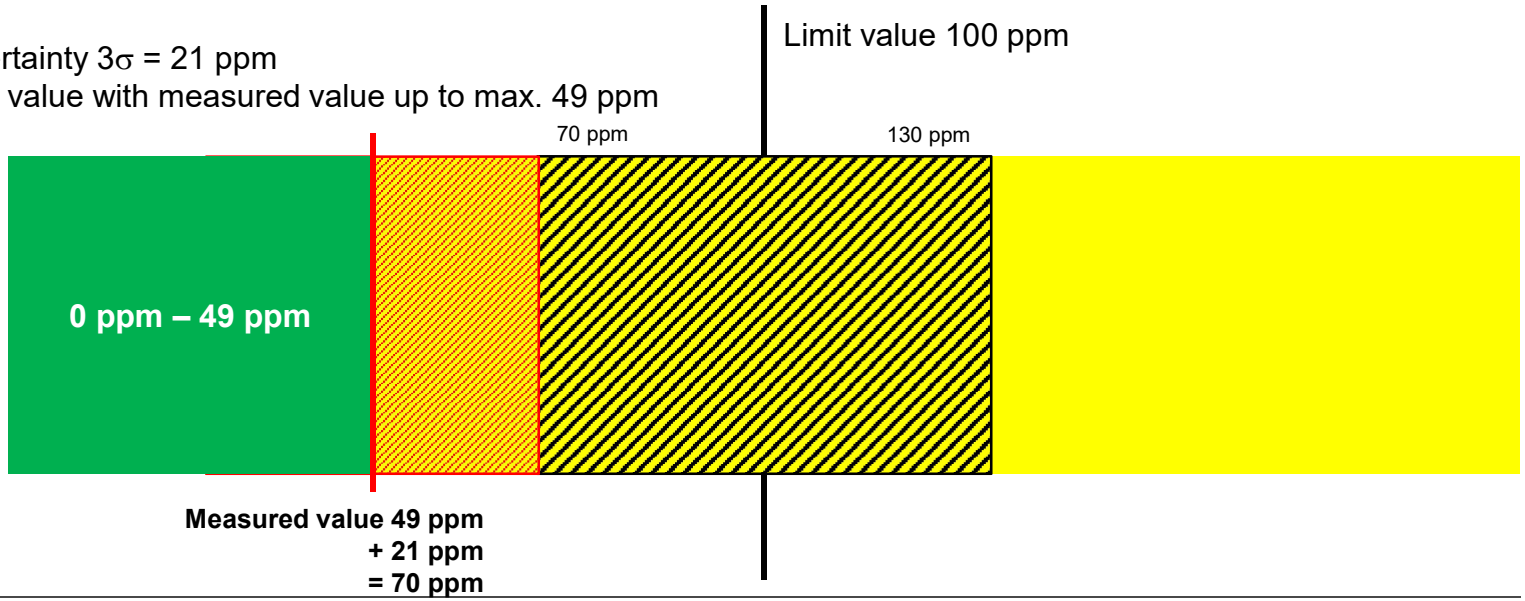
RoHS analysis

Fischerscope XRAY RoHS Setup - Evaluation

Extension of the safety range by measurement uncertainty

Example: Cadmium
Measuring time: 100 sec

Measurement uncertainty 3σ = 21 ppm
Reliably below limit value with measured value up to max. 49 ppm



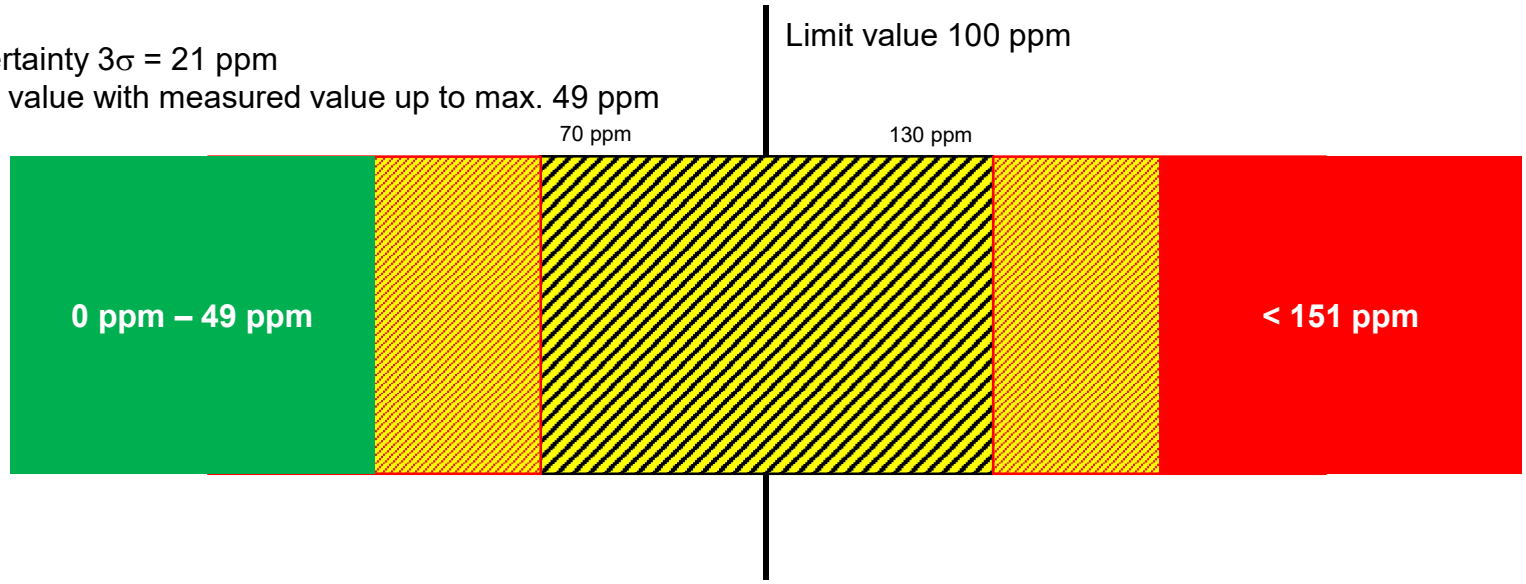
RoHS analysis

Fischerscope XRAY RoHS Setup - Evaluation

Extension of the safety range by measurement uncertainty

Example: Cadmium
Measuring time: 100 sec

Measurement uncertainty 3σ = 21 ppm
Reliably below limit value with measured value up to max. 49 ppm



RoHS analysis

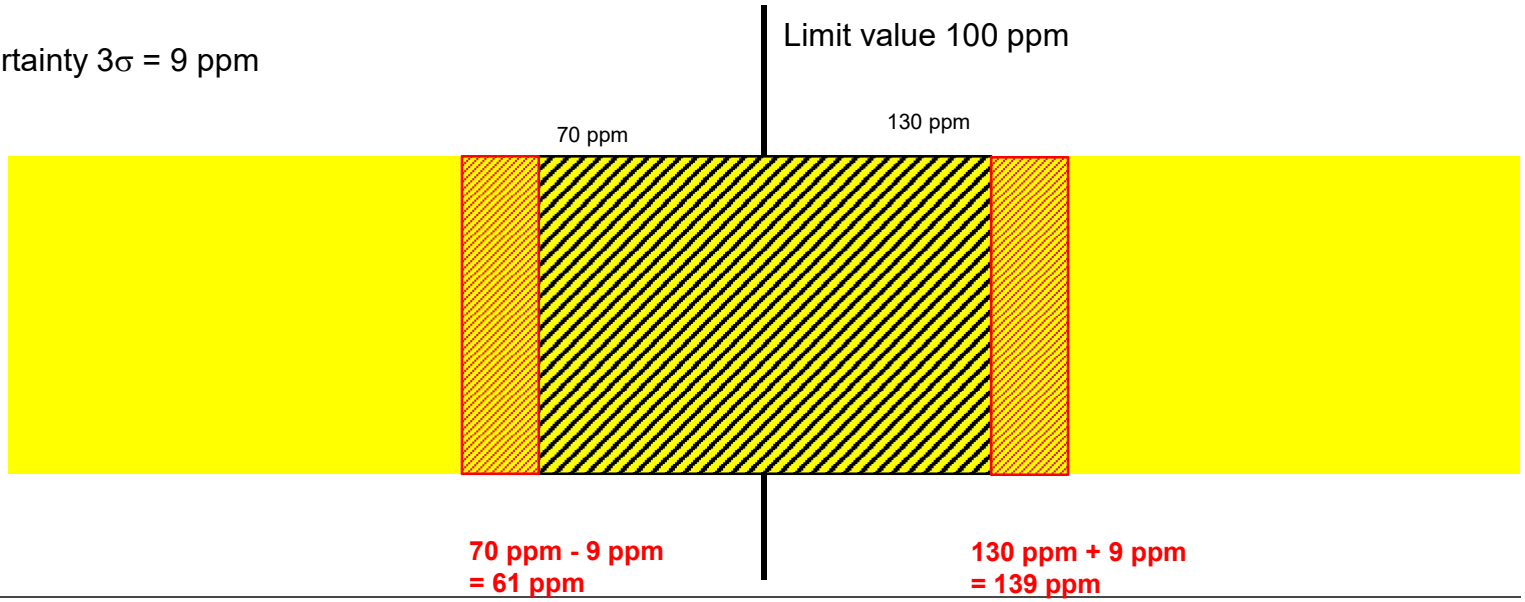
Fischerscope XRAY RoHS Setup - Evaluation

Extension of the safety range by measurement uncertainty - Influence of the measurement time

Example: Cadmium
Measuring time: 500 sec

Measurement uncertainty 3σ = 9 ppm

The lower measurement uncertainty results from the dependence of σ on 1/root(measuring time



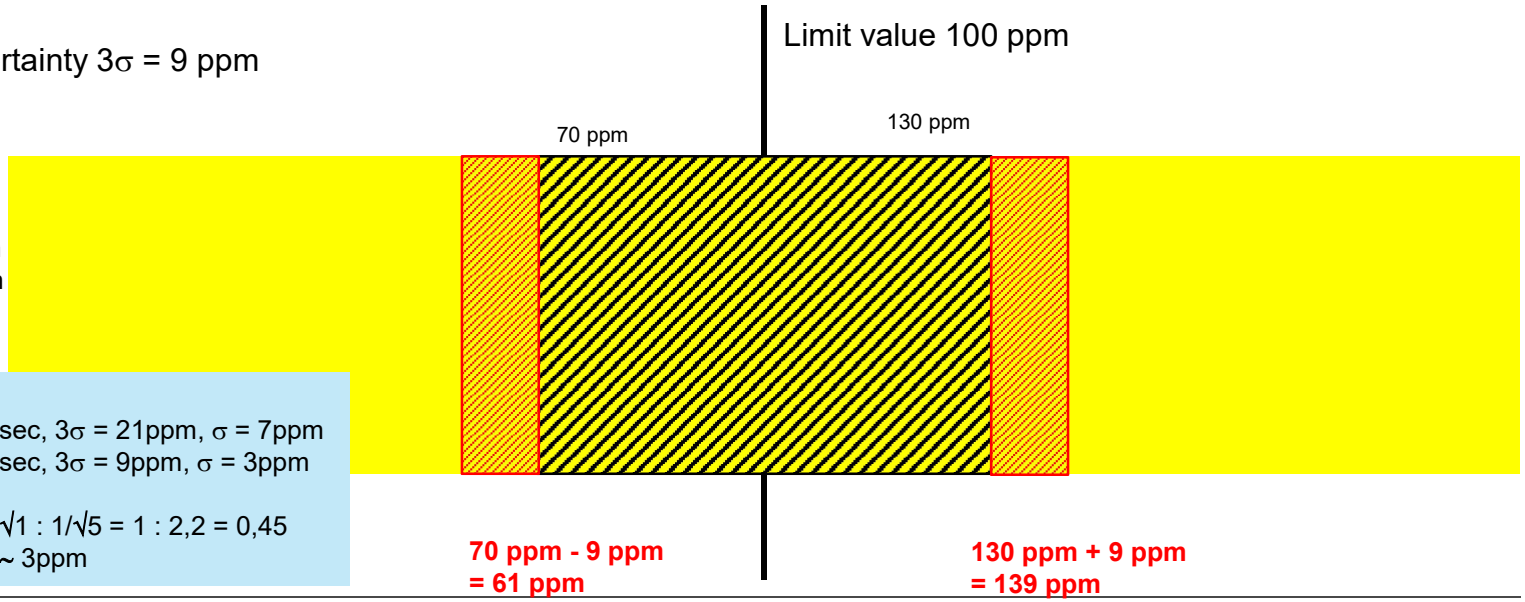
RoHS analysis

Fischerscope XRAY RoHS Setup - Evaluation

Extension of the safety range by measurement uncertainty - Influence of the measurement time

Example: Cadmium
Measuring time: 500 sec

Measurement uncertainty 3σ = 9 ppm



The lower measurement uncertainty results from the dependence of σ on 1/root (measuring time)

Ex:
Measurement time 100sec, 3σ = 21ppm, σ = 7ppm
Measurement time 500sec, 3σ = 9ppm, σ = 3ppm

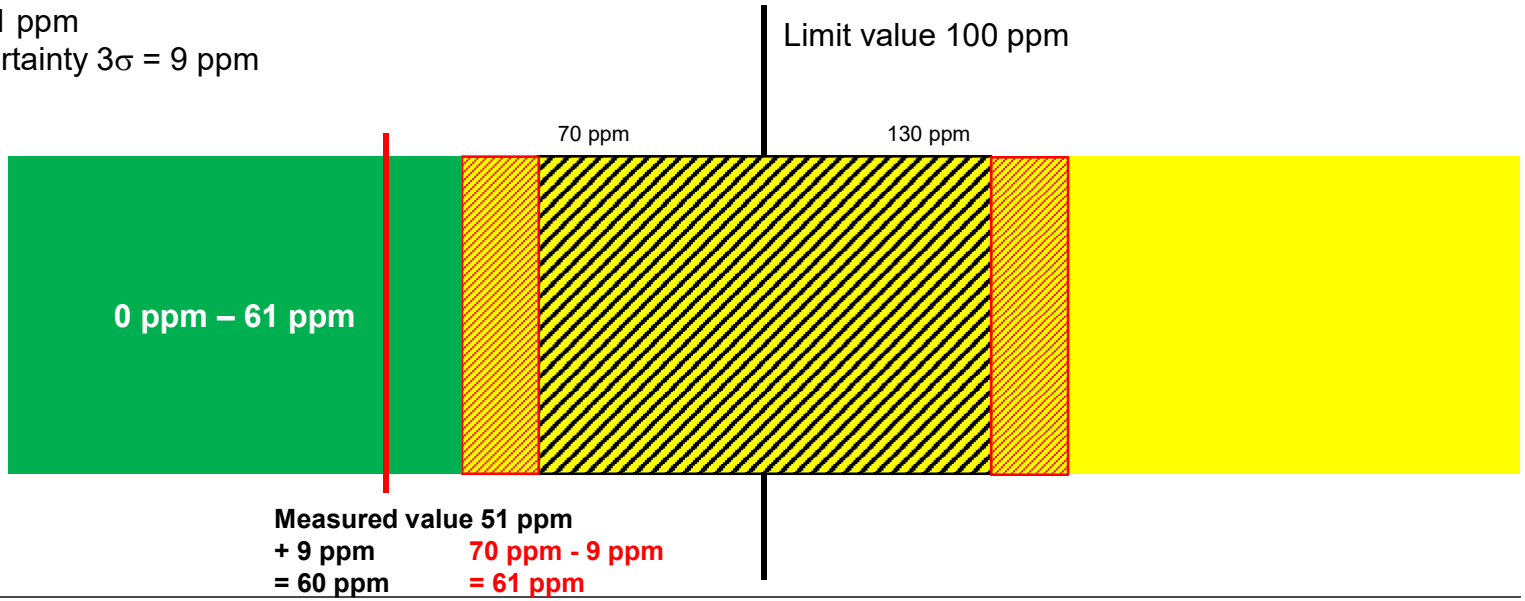
1/√(100) : 1/√(500) = 1/√1 : 1/√5 = 1 : 2,2 = 0,45
7ppm x 0,45 = 3,2ppm ~ 3ppm

RoHS analysis

Fischerscope XRAY RoHS Setup - Evaluation

Extension of the safety range by measurement uncertainty - Influence of the measurement time

Example: Cadmium
Measuring time: 500 sec
Measured value: 51 ppm
Measurement uncertainty 3σ = 9 ppm



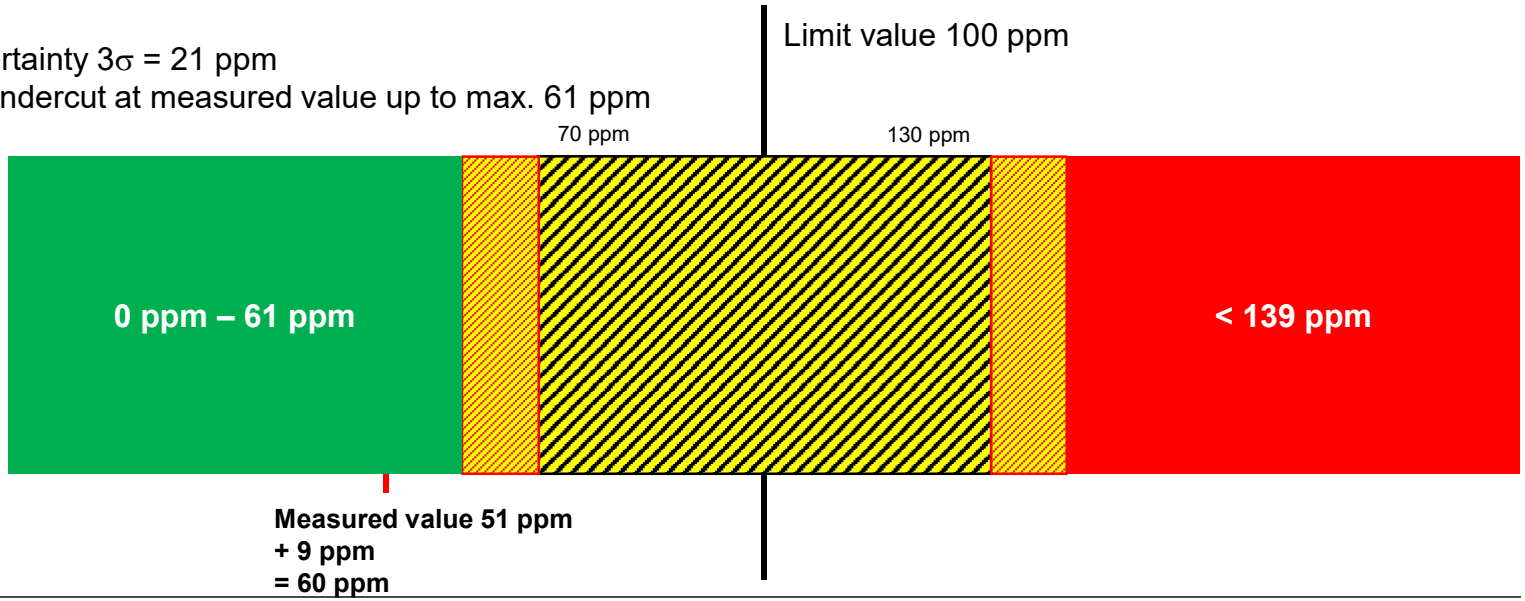
RoHS analysis

Fischerscope XRAY RoHS Setup - Evaluation

Extension of the safety range by measurement uncertainty - Influence of the measurement time

Example: Cadmium
Measuring time: 500 sec

Measurement uncertainty 3σ = 21 ppm
Limit value safely undercut at measured value up to max. 61 ppm

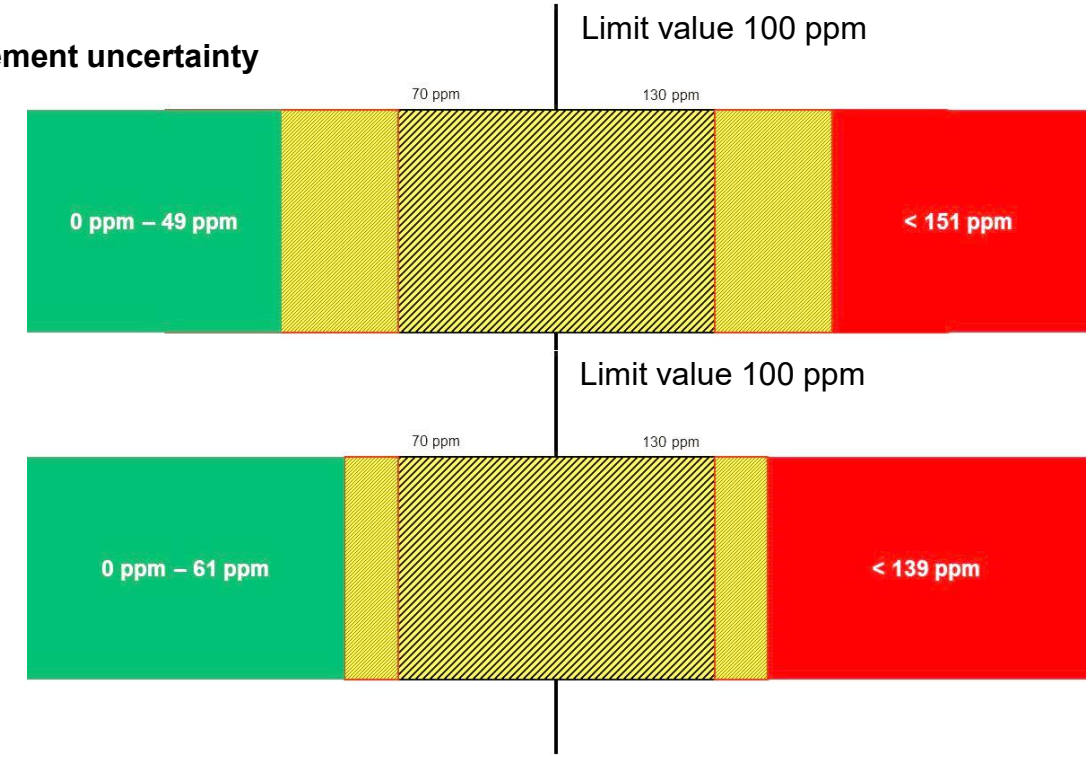


RoHS analysis

Fischerscope XRAY RoHS Setup - Evaluation

Extension of the safety range by measurement uncertainty
Influence of measurement time

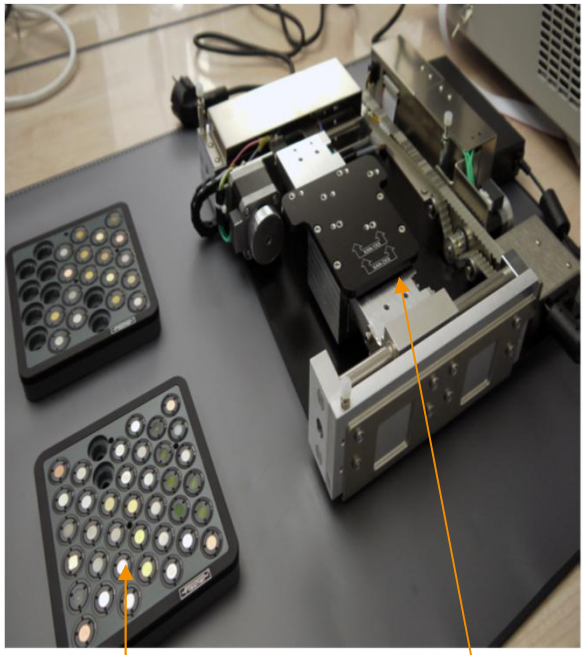
Measuring time: 100 sec
Reliably below limit value
with measured value up to max. 49 ppm



Measuring time: 500 sec
Reliably below limit value
with measured value up to max. 61 ppm

$\sigma \sim 1/\sqrt{}$ (Measurement time)

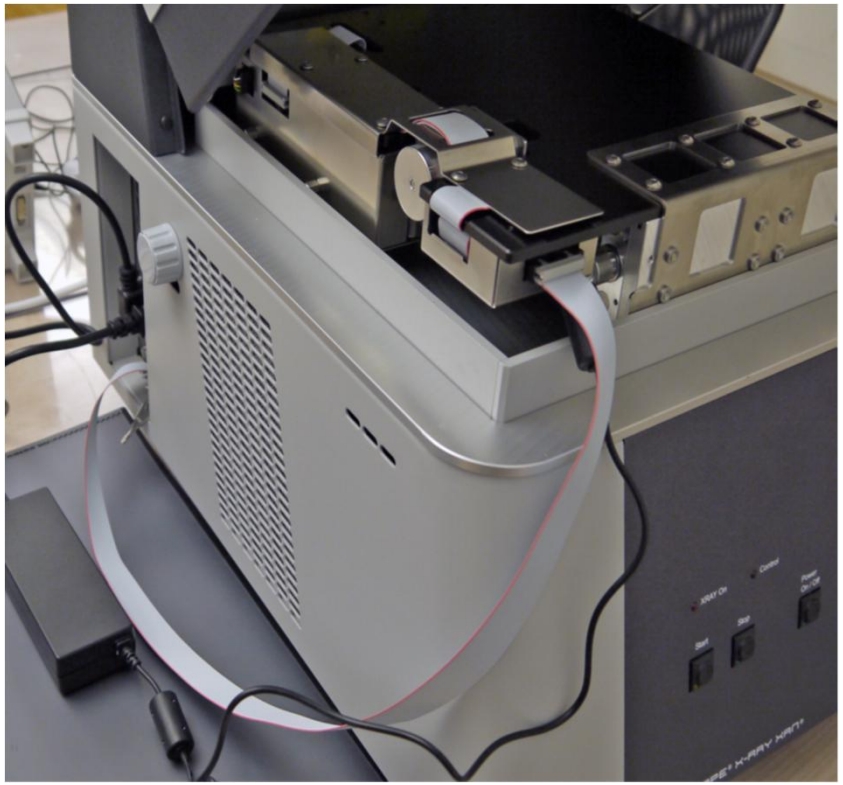
Calibration Device RoHS Setup

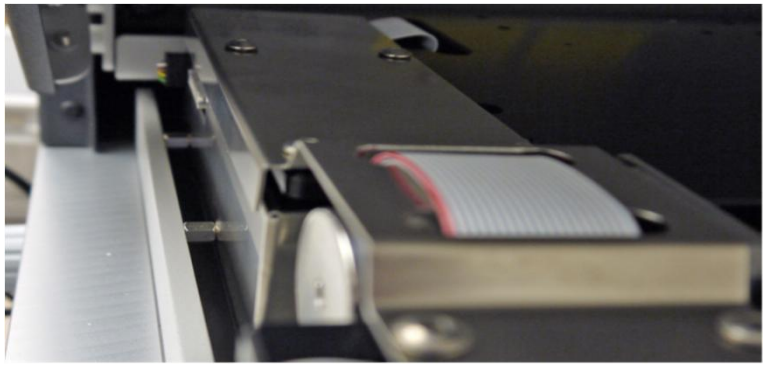


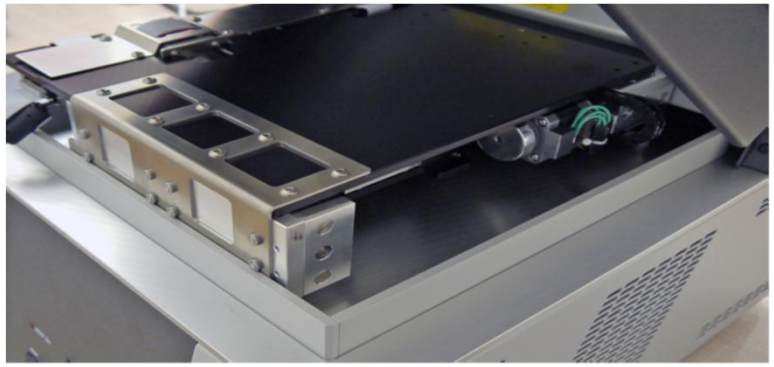
Calibration standards holder

Calibration device

Calibration Device RoHS Setup

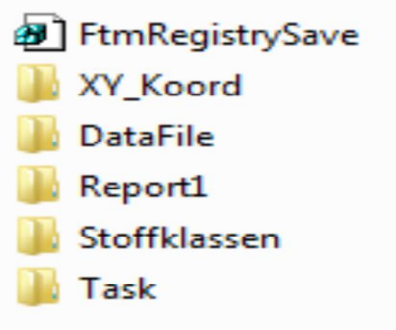






RoHS: Calibration

New instruments (at Fischer Sindelfingen):
Files to be copied into WinFTM:



- Icons + colors of tolerance limits
- Coordinates for the calibration task
- Products + Applications
- Reports (for the different Products)
- Class of materials for Metals und Composite
- Tasks (calibration, automated selection of products)

- start calibration task
- start refreshing of the classes of materials
- check calibration (testsamples + N6 Block)
- create certificate

RoHS: calibration

2.) instruments after repair (Service)

Main Problem: originally installed setup might be changed !!!!

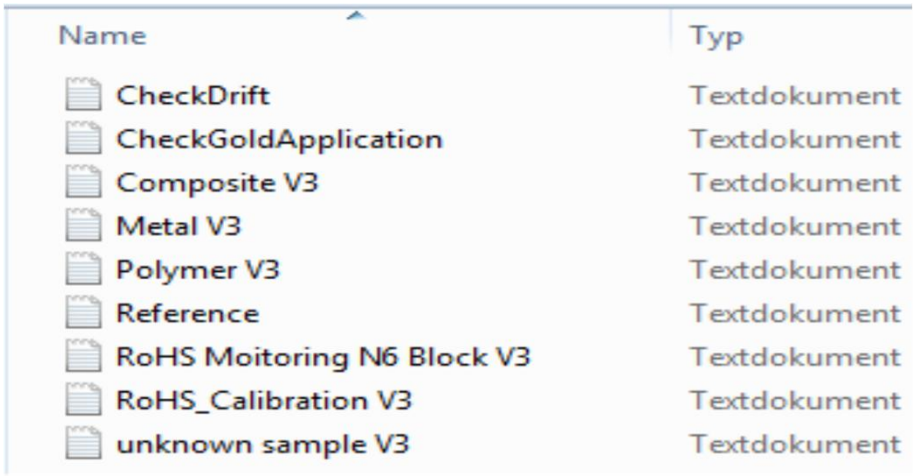
→ calibration task fails or measures wrong standards
→ RoHS applications have to be checked and maybe changed to the originally ones before starting the calibration task
be carefull: some products use the same application (Composite)
→ V3 calibration task: needs WinFTM 6.33. or higher (Recommended 6.49 for DPP+)

RoHS: Calibration - blocks needed for RoHS calibration:

RoHS V2 (calibration block)

RoHS V3 (calibration block 605-204)

RoHS V3: Tasks

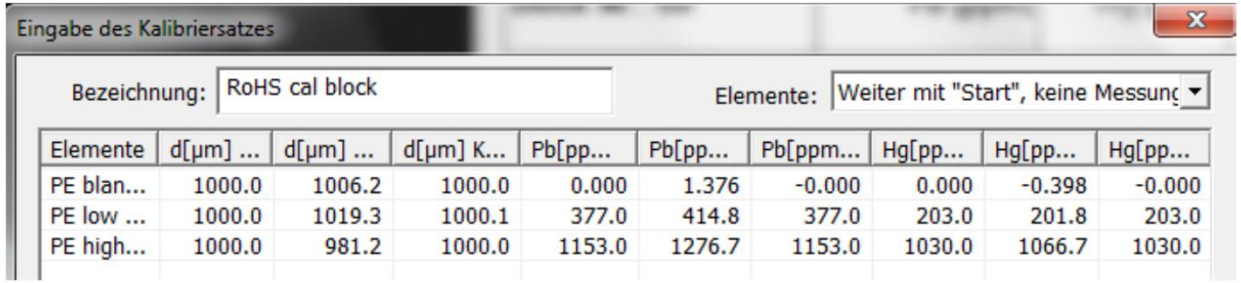


→ selection of product
→ selection of product
→ selection of product

→ measurement of N6 block
→ runs calibration
→ measurement of unknown samples

RoHS: check RoHS Setup

1.) check calibration set: max 20% or 100ppm difference



Elemente	d[µm] ...	d[µm] ...	d[µm] K...	Pb[pp...	Pb[pp...	Pb[ppm...	Hg[pp...	Hg[pp...	Hg[pp...
PE blan...	1000.0	1006.2	1000.0	0.000	1.376	-0.000	0.000	-0.398	-0.000
PE low ...	1000.0	1019.3	1000.1	377.0	414.8	377.0	203.0	201.8	203.0
PE high...	1000.0	981.2	1000.0	1153.0	1276.7	1153.0	1030.0	1066.7	1030.0

2.) measure testsamples (refreh class of materials first, if spectra library has changed or recalculated)
3.) check N6-Block + create certificate (only for RoHS Setup V3) (N6-Block needs recertification after1-2 years)

RoHS analysis

Fischerscope XRAY RoHS Setup - Evaluation

Classification of measurement results
according to IEC 62321 (Determination of components in electrotechnical products)

Below limit (BL)	« Inconclusive » (X) , further investigations are neccessary	Over limit (OL)
Measured value safely below the RoHS limit value	Measured value within the unsafe range, Measurement with more precise methods necessary	Measured value safely above the RoHS limit value

RoHS analysis

Fischerscope XRAY RoHS Setup - Evaluation

Classification of the measurement results

	Polymer	Metals	Composite
Pb	BL≤(700-3σ) < X < (1300+3σ) ≤ OL	BL≤(700-3σ) < X < (1300+3σ) ≤ OL	BL≤(500-3σ) < X < (1500+3σ) ≤ OL
Hg	BL ≤(700-3σ) < X < (1300+3σ) ≤ OL	BL ≤(700-3σ) < X < (1300+3σ) ≤ OL	BL ≤(500-3σ) < X < (1500+3σ) ≤ OL
Cd	BL ≤(70-3σ) < X < (130+3σ) ≤ OL	BL ≤(70-3σ) < X < (130+3σ) ≤ OL	LOD < X < (150+3σ) ≤ OL
Cr	BL ≤(700-3σ) < X	BL ≤(700-3σ) < X	BL ≤(500-3σ) < X
Br	BL ≤(300-3σ) < X		BL ≤(250-3σ) < X

LOD = Limit of Detection

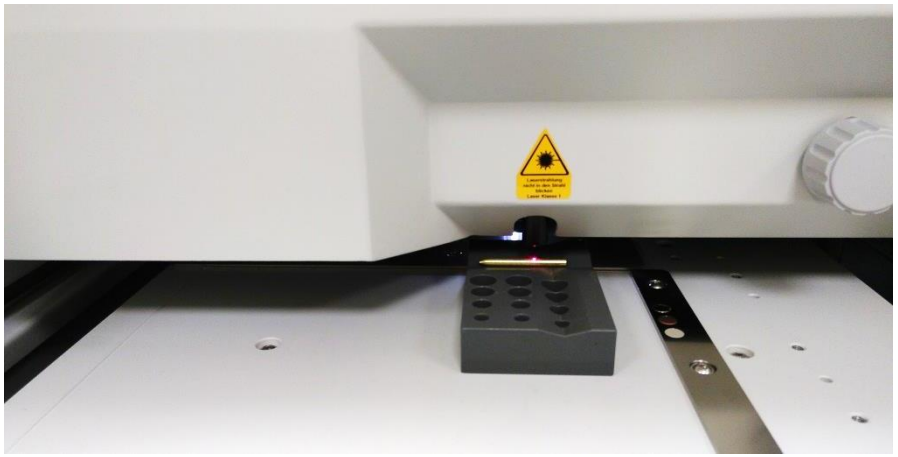
RoHS analysis

Fischerscope XRAY RoHS

Analysis example

Samples: Brass tubes CuZn37, CuZn38Pb2

Brass tubes on sample rest for round samples (Sample support: Helmut Fischer GmbH)



Positioning on the measuring table of a Fischerscope XDV-SDD

RoHS analysis

Fischerscope XRAY RoHS

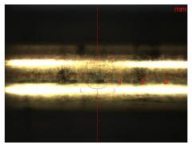
Automated protocol generation after analysis

Samples: Brass tubes CuZn37, CuZn38Pb2

CuZn37

RoHS Test Report

Sample: RoHS **Material: RoHS Metals**



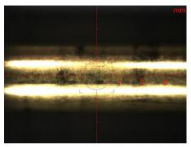
	Test Result				
	Pb ppm	Hg ppm	Cd ppm	Cr ppm	Br ppm
Concentrations	179.4	N.d.	N.d.	N.d.	N.d.
3*σ	29.65	29.95	35.36	28.64	15.07
RoHS Status	BL	BL	BL	BL	BL

BL: Below Limit¹
OL: Over Limit¹

CuZn38Pb2

RoHS Test Report

Sample: RoHS **Material: RoHS Metals**



	Test Result				
	Pb ppm	Hg ppm	Cd ppm	Cr ppm	Br ppm
Concentrations	16821	39.09	46.19	N.d.	25.11
3*σ	137.5	32.29	40.70	31.62	16.89
RoHS Status	OL	BL	X	BL	BL

BL: Below Limit¹
OL: Over Limit¹

RoHS analysis

Fischerscope XRAY RoHS

Automated protocol generation after analysis

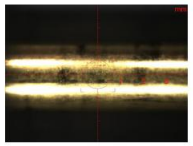
Samples: Brass tubes

	Polymer	Metals
Pb	BL≤(700-3σ) < X < (1300+3σ) ≤ OL	BL≤(700-3σ) < X < (1300+3σ) ≤ OL
Hg	BL ≤(700-3σ) < X < (1300+3σ) ≤ OL	BL ≤(700-3σ) < X < (1300+3σ) ≤ OL
Cd	BL ≤(70-3σ) < X < (130+3σ) ≤ OL	BL ≤(70-3σ) < X < (130+3σ) ≤ OL
Cr	BL ≤(700-3σ) < X	BL ≤(700-3σ) < X
Br	BL ≤(300-3σ) < X	

CuZn38Pb2

RoHS Test Report

Sample: RoHS **Material: RoHS Metals**



Test Result

	Pb ppm	Hg ppm	Cd ppm	Cr ppm	Br ppm
Concentrations	16821	39.09	46.19	N.d.	25.11
3*σ	137.5	32.29	40.70	31.62	16.89
RoHS Status	OL	BL	X	BL	BL

BL: Below Limit¹
OL: Over Limit¹

RoHS analysis

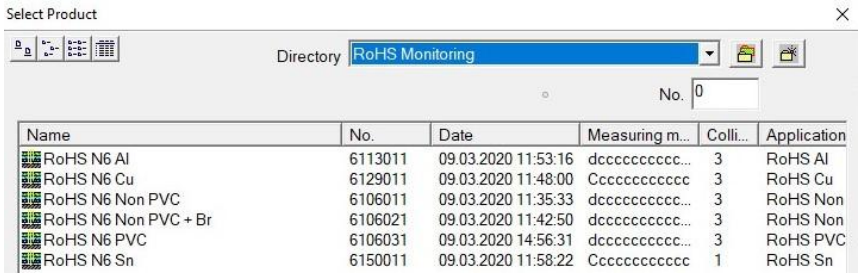
Fischerscope XRAY RoHS

Test set: RoHS N6 block

The test set contains samples for the different substance classes.

Substance class identification and correctness of the determination of the RoHS elements can be checked with this.

The standards can also be used for high-precision monitoring of measuring equipment. Preconfigured measurement programmes are available for this purpose.



RoHS analysis

Fischerscope XRAY systems and RoHS setup

XDAL 600

XDV-SDD

XAN 250

Advantages for the user

- Coordinated with the IEC 62321 guideline
- simple operation of the Fischerscope
- no time-consuming sample preparation
- fast analyses (compared to physico-chemical methods, typ. measuring times of 5 min. or less)
- measurement with small measuring points possible for fine structures
- immediate and easy interpretation of results
- automated report generation
- no additional costs, saving of other time- and cost-intensive methods
- simple and fast monitoring of measuring equipment to ensure correct results
- accessories (sample holder, radiation trap) available for easy and safe measurement

In addition, many years of experience and competent application support.