

# ENVIRONMENTAL TESTING

Organic certified reference materials

Zuzana Antalová  
October 2017



Food &  
Beverage

**MERCK**

# Analytical Testing and Reference Standards ..... go hand in hand



..... for **total confidence** in measurement

Results are only as accurate as the reference!

# Why to buy Standards

## Choosing a supplier for Reference Materials

**NO DOUBTS. NO DELAYS.  
JUST PRECISION!**

Darmstadt, Germany  
Certipur® inorganic and  
elemental CRMs

Laramie, WY, USA  
RT Pharma Secondary  
standards, matrix  
environmental CRMs,  
proficiency testing  
schemes

Round Rock TX, USA  
Cerilliant® clinical,  
toxicology and pharma  
CRMs and RMs

Buchs, Switzerland  
TraceCERT® organic and  
inorganic CRMs, organic  
RMs (pesticides), inorganic  
custom standards

### Trustful partner

- An accredited manufacturer of reference materials that you can **trust to**, we continually **develop new products** and solutions to make your analytical life and research **easier**

### World renowned supply chain

- Global supply, with a majority of standards available to **ship same day**

### Outstanding quality, service

- Supplying **over 22 000** different analytical standards and certified reference materials

## Definition

# What are Reference Materials?

Certified Reference Material	Reference Material
CRM	RM
<ul style="list-style-type: none"><li>• Values(s) characterized by a metrologically valid procedure for the specified property(ies)</li><li>• Certificate provides the<ul style="list-style-type: none"><li>– <b>Property Value</b></li><li>– <b>Associated Uncertainty</b></li><li>– <b>Metrological Traceability</b></li></ul></li><li>• Manufactured by an <b>accredited Manufacturer</b> and value(s) assigned by an accredited laboratory</li></ul>	<ul style="list-style-type: none"><li>• Value is <b>homogenous</b> and <b>stable</b> with regard to the specified property(ies)</li><li>• Fit for its intended use in measurement, calibration of an apparatus or assessment of analytical method</li></ul>

Type of standard

Acronyms

Quality attributes

References

- 1.) ISO Guide 30:2015 Reference materials – Selected terms and definitions
- 2.) ISO Guide 34:2009 General requirements for the competence of reference material producers
- 3.) JCGM 200:2012 International Vocabulary of Metrology

# Certified Reference Materials Production to ISO (International Standardisation Organisation)

## ISO/IEC 17025



Focuses on the  
**MEASUREMENT**

Main objectives of the quality control lab:

- **Characterization of the CRM**
  - Certifying the measurement value and the uncertainty value according to Guide 35
  - Making the reference material traceable to a primary standard

## ISO Guide 34 Changed to ISO17034 from November 2016



Accreditation as a producer of  
**certified reference materials**

- **Production**
  - Raw material selection and purity
  - Production planning and control
- **Characterization** (*everything that is in ISO/IEC 17025*)
  - Measurement methods
  - Uncertainty evaluation
  - Traceability
- **Assessment of homogeneity and stability** (including packaging)

## ISO Guide 31



Defines **content** of the Certificate of Analysis and product labelling

## Certified Reference Materials (CRMs) Merck CRM Manufacture



### Double accreditation as a Reference Material Producer

For CRM producers the combination of ISO/ IEC 17025 *and* ISO Guide 34 is the **highest achievable level of quality and confidence**

We also call it the **"gold standard"** for CRM producers

**All MERCK** standards manufacturing sites are at a minimum double accredited to ISO/IEC 17025 and ISO Guide 34, which is the highest achievable quality level for reference material producers

# ISO Guide 34 & ISO 17025 Accreditation

## Building on a solid foundation

ISO Guide 34: Accreditation as a producer of CRMs

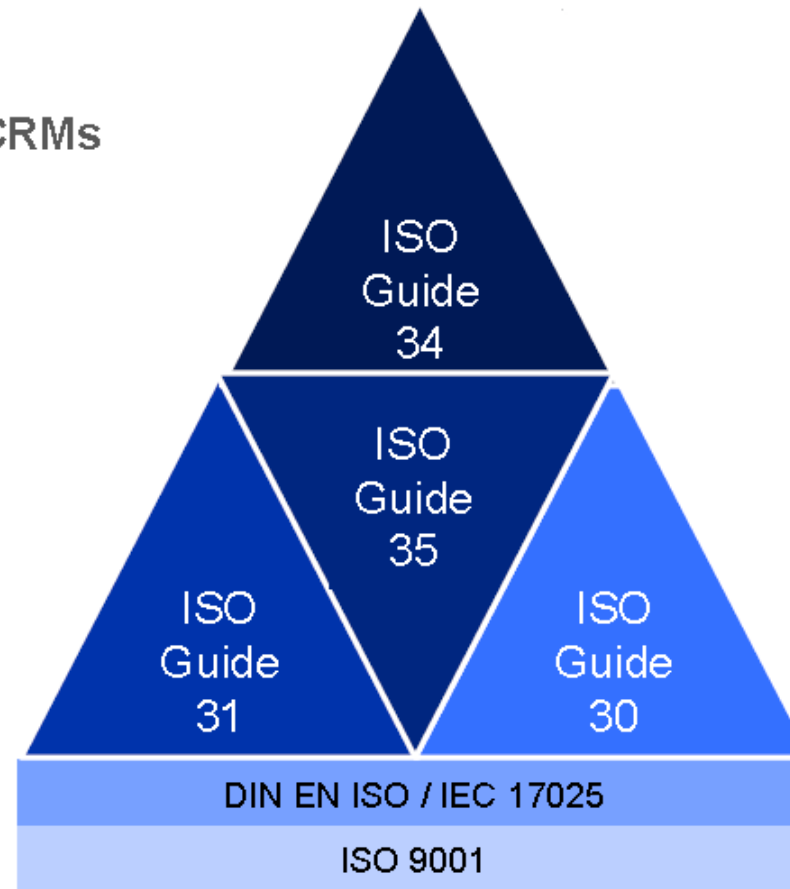
ISO Guide 35: Calculation of the uncertainty

ISO Guide 31: Certificate of Analysis

ISO Guide 30: CRM definition

ISO 17025: Accreditation of the analytical lab

ISO 9001: Documentation



ISO 17025 (General Requirements for the Competence of Testing and Calibration Laboratories) and ISO Guide 34 (General Requirements for the Competence of Reference Material Producers)

# CRM Production

## Merck Manufacturing Sites Having Double Accreditation

**Buchs,  
CH**



Analytical standards and **TraceCERT®** CRMs

**Round  
Rock,  
TX**



**Cerilliant®** clinical, toxicology and pharma standards

**Laramie,  
WY**



Secondary standards, matrix environmental CRMs, proficiency testing schemes

**Bellefonte,  
PA**



Solutions, mixes and customer standards **Supelco®**

**Darmstadt,  
DE**



Merck HQ, Certipure® inorganics



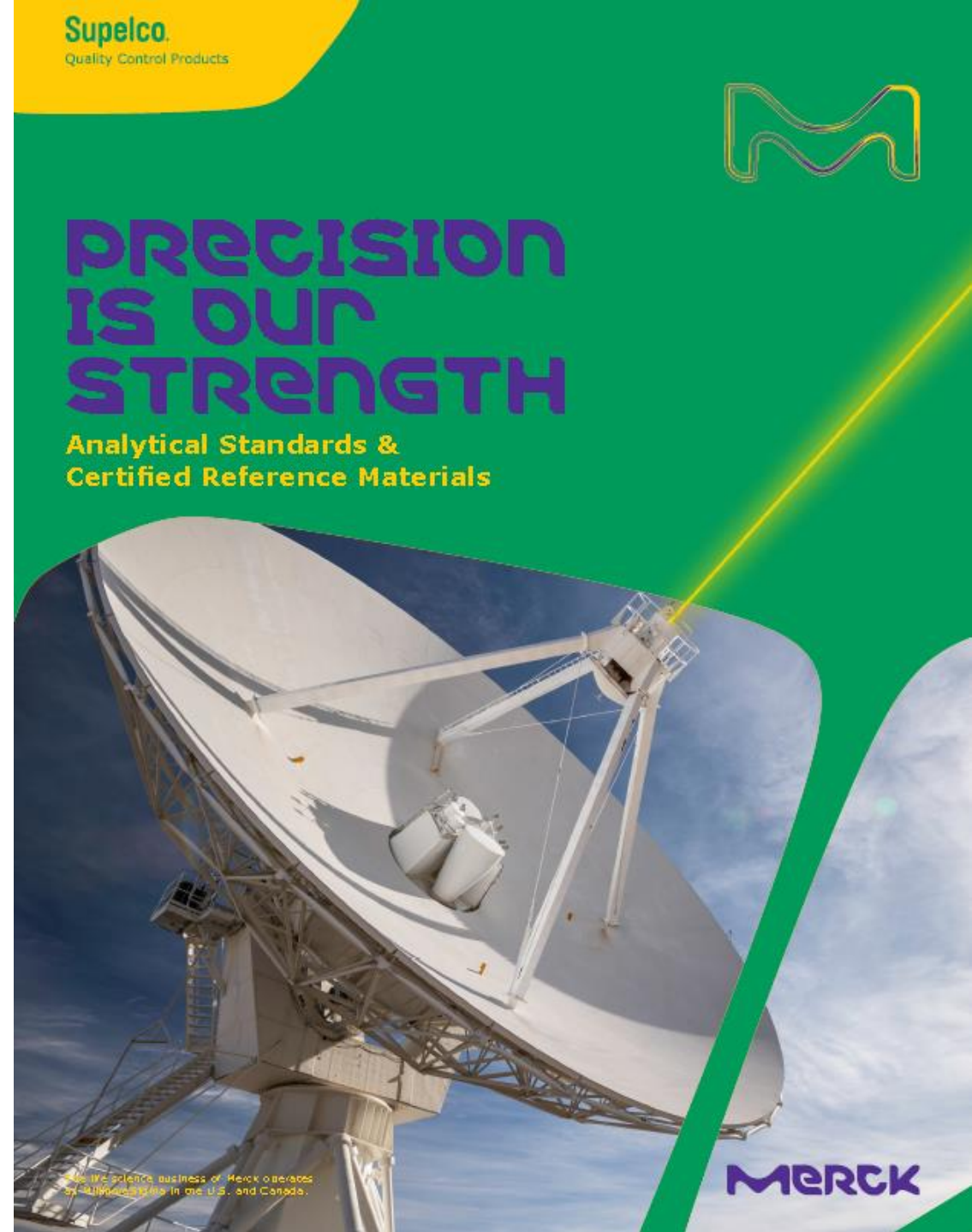
# Why use Certified Reference Materials? Benefits

- **High Reliability**
- **Accurate Quantitative Values**
- **Comparable Results**

## ISO/IEC 17025

General requirements for the competence of  
**testing and calibration laboratories**

- **Usage of CRMs is mandatory**



Supelco.  
Quality Control Products

PRECISION  
IS OUR  
STRENGTH

Analytical Standards &  
Certified Reference Materials

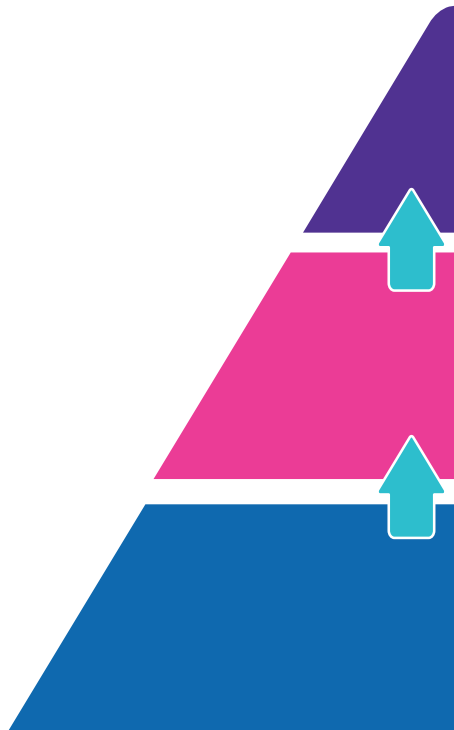
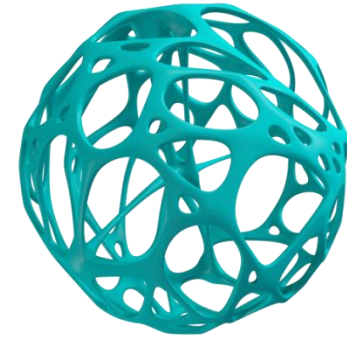
MERCK

© 2014 The science business of Merck operates  
as MilliporeSigma in the U.S. and Canada.

The advertisement features a large satellite dish pointing towards a bright yellow laser beam against a blue sky with clouds. The background is a vibrant green with a yellow and purple wavy logo in the top right corner.

What are the different types of standards in our portfolio?

## The Hierarchy of Standards



**Metrology Reference Standard (NMI), and Pharmacopeial (Primary) Reference Standard**

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**Certified Reference Material (CRM), ISO**

A CRM is considered to provide the highest level of accuracy and traceability for a measurement outside of a National Metrology Institute (NMI) material

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**Analytical Standard**

these are provided with a Certificate of Analysis, with high purity, this does not make them the same as a CRM, the certificate can be issued by anyone, no rules about who is authorized to issue certificates and who is not

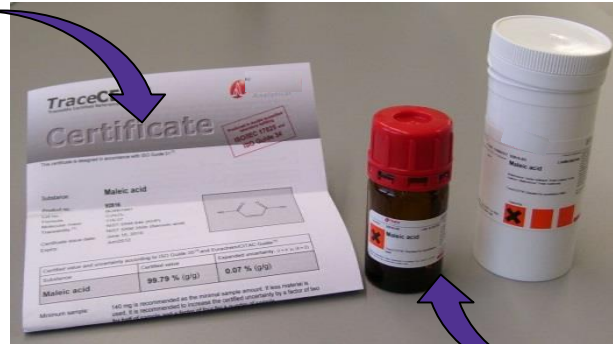
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# Certified Reference Material (CRM) and Analytical Standard

## What is the difference?

**ISO/IEC 17025**  
(Guide for testing laboratories)

**ISO Guide 31**  
(Guide for documentation, certificates)



**ISO Guide 34**  
(Guide for the CRM Producer)

Parameters	Analytical Standard	CRM
Stability	✓	✓
Homogeneity		✓
Uncertainty		✓
Traceability		✓

# Which type to choose from our portfolio?

## Pure Chemical, Standard or CRM?

### Research Grade Chemical

Used: *in a variety of applications, synthesis*

Is provided with a CoA

Purity varies

Is not suitable to be used as a standard

### Analytical Standard

Used: *Where a standard is needed for calibration*

Qualitative identification (such as impurities) or quantitative

*Method development*

Performance controls

Impurity identification

Verification and checks of analytical systems

Also known as a:

- Standard, internal standard, calibrator, control

### Certified Reference Material (CRM)

Used: *Where confidence in the measurement is critical*

As a calibration standard in 17025 regulated labs

*Method validation*

Where uncertainty of measurement is needed

Where traceability is needed (e.g. final product testing)

Instrument qualification (IQ/OQ)

Proficiency testing

# Analytical Standards and Certified Reference Materials Formats available



## Solid and liquid ('neats')

- To create your own calibration concentrations

## Solutions

- Ready to use, for spiking biological samples or for use as calibrators

## Mixes

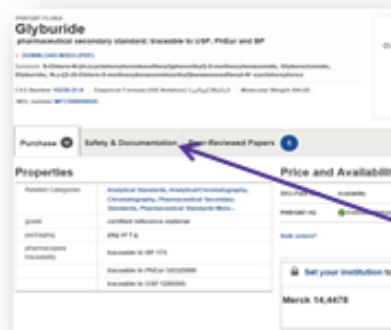
- Convenient combinations of commonly screened compounds, such as multicomponent ICP CRMs

# Analytical Standards and CRMs

## Did you receive your Certificate of Analysis?

### RM delivered with their CoAs:

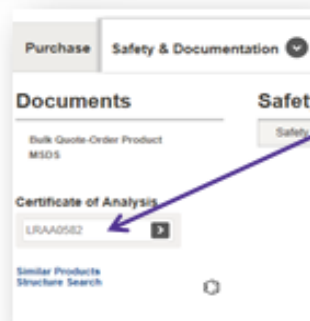
- TraceCERT® (organic)
- Pestanal®



### RMs not delivered with CoA:

## Cerilliant® Certified Reference Materials

- Electronic Certificate – product web page or CoA search tool



# CoA

the real **value** behind

# Analytical Standard Certificate of analysis

**SIGMA-ALDRICH®**

3050 Spruce Street, Saint Louis, MO 63103 USA  
Email USA: techserv@sial.com Outside USA: eurtechserv@sial.com

## Certificate of Analysis

**Product Name:** GLYPHOSATE  
PESTANAL™, analytical standard

**Product Number:** 45521

**Batch Number:** BCBS2439V

**Brand:** Sigma-Aldrich

**CAS Number:** 1071-83-6

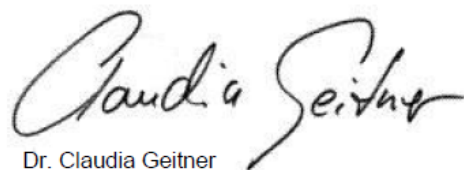
**Formula:** (HO)<sub>2</sub>P(O)CH<sub>2</sub>NHCH<sub>2</sub>CO<sub>2</sub>H

**Formula Weight:** 169.07

**Expiration Date:** AUG 2021

**Quality Release Date:** 08 SEP 2016

TEST	SPECIFICATION	RESULT
PURITY (HPLC AREA %)	≥ 98.0 %	98.7%
WATER	≤ 1.0 %	0.48%
PROTON NMR SPECTRUM	CONFORMS TO STRUCTURE	CONFORMS



Dr. Claudia Geitner  
Manager Quality Control  
Buchs, Switzerland

- High chromatographic purity
- Identity Confirmed (NMR)
- (sometimes) Tested for Impurities (Water, Solvents, inorganics)

45521 Glyphosate

Lot Number: BCBS2439V	Sample Name: T38562_001_LC
Dionex Ultimate 3000	
<b>Pump :</b> LPG-3400A	<b>Injection Time:</b> 07.09.16 19:43
<b>Autosampler:</b> WPS-3000	<b>Processed By:</b> Mikael Berthet
<b>Detector:</b> PDA-3000	<b>Vial Number:</b> BE4
<b>Column:</b> Supelco Ascentis Express C18, 2.7 um	<b>Column S/N:</b> -
<b>Column Dim.:</b> 100 x 4.6 mm	<b>Sample Type:</b> unknown
<b>Mobile Phase:</b>	<b>Injection Volume:</b> 2.0 µl
%A : Acetonitrile	<b>Flow:</b> 1.50 ml/min
%B : H2O	<b>Column Temp. (°C):</b> 25.0
%C : TB pH 2.4	<b>Run Time:</b> 15.00 min
%D : HPS pH 2.4	
<b>Gradient :</b> see Figure 1	
<b>Sample Prep.:</b> 400 ul sample solution (1 mg/ml sample in Buffer pH 9) and 600 ul (5 mg/ml DNBC in acetonitrile)	

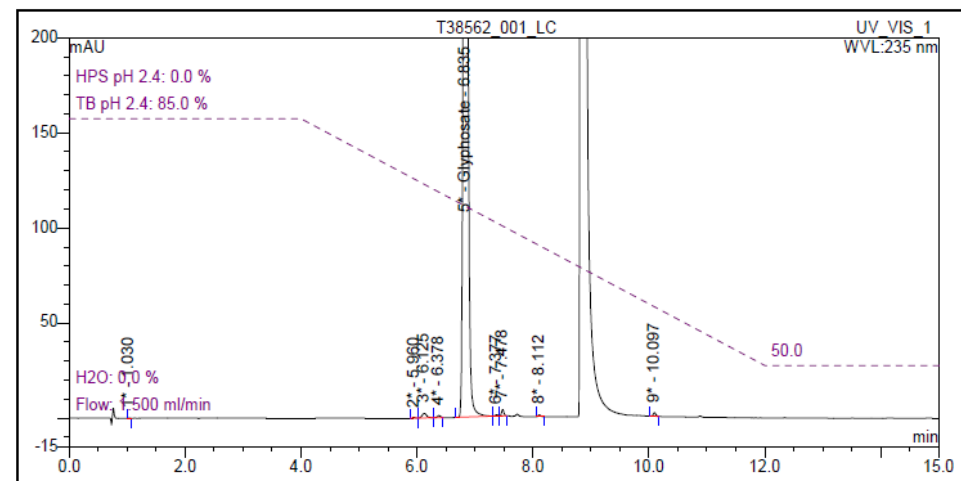


Figure 1: Zoomed Chromatogram

No.	Ret.Time min	Peak Name	Area mAU*min	Height mAU	Amount	Rel.Area %
1	1.030	n.a.	0.00695	0.21985	n.a.	0.01
2	5.960	n.a.	0.01555	0.23788	n.a.	0.03
3	6.125	n.a.	0.21857	2.38727	n.a.	0.43
4	6.378	n.a.	0.06830	1.05527	n.a.	0.14
5	6.835	Glyphosate	49.73302	638.84435	n.a.	98.73
6	7.377	n.a.	0.03779	0.67942	n.a.	0.08
7	7.478	n.a.	0.15080	3.30383	n.a.	0.30
8	8.112	n.a.	0.04084	0.92191	n.a.	0.08
9	10.097	n.a.	0.09856	1.87811	n.a.	0.20
Total:			50.37038	649.52789		100.00

Table 1: Integration

**MERCK**



# Certified Reference Materials Certificate of Analysis

- Values **traceable** (NIST, SI)
- Proper calculated **uncertainty**
- **Homogeneity**
- **Stability** tested

All guaranteed if manufactured under **ISO 17025** and **ISO Guide 34**



**TraceCERT®** **SIGMA-ALDRICH®**  
Traceable Certified Reference Materials

## Certificate

This certificate is designed in accordance with ISO Guide 31<sup>[1]</sup>.

Product name: **Pyrocatechol**

Product no.: **50550**

Lot no.: **BCBM6265V**

Formula: **C<sub>6</sub>H<sub>6</sub>O<sub>2</sub>**

Molecular mass: **110.11 g/mol**

Traceability<sup>[2]</sup>: **NIST SRM 350b (Benzole assay)**

Certificate issue date: **February 29, 2016**

Expiry: **FEB 2018**

Certified value and uncertainty according to ISO Guide 35 <sup>[3]</sup> and Eurachem/CITAC Guide <sup>[4]</sup>		
Substance	Certified value as mass fraction (g/g)	Expanded uncertainty, $U = k \cdot u$ ( $k = 2$ ) as mass fraction (g/g)
<b>Pyrocatechol</b>	<b>99.8 %</b>	<b>0.2 %</b>

Minimum sample: The sample is solid at room-temperature. 10 mg is recommended as the minimal sample amount. If less material is used, it is recommended to increase the certified uncertainty by a factor of two for half of sample and a factor of four for a quarter of sample.

Drying instruction: This material does not require drying before use.

Intended use: Use this certified reference material (CRM) as calibrant for chromatography or any other analytical technique.

Storage and handling: The CRM should be stored in the original bottle at room-temperature (20-25°C). After use the bottle should be tightly closed and protected from excessive moisture and light. Store under Argon.

CRM operations: *A. Rück*  
Dr. A. Rück

Certification body: *Mano Bachmann*  
Dr. K.D. Bachmann

Certificate page 1 of 4      Sigma-Aldrich Produkte GmbH, Industriestraße 25, 9471 Bucha/Schweizland, Tel. +41-81-756-2611, Fax +41-81-756-5856      **SIGMA-ALDRICH®**

ISO Guide 31

Traceability statement

Expiry date

Content

Expansion factor

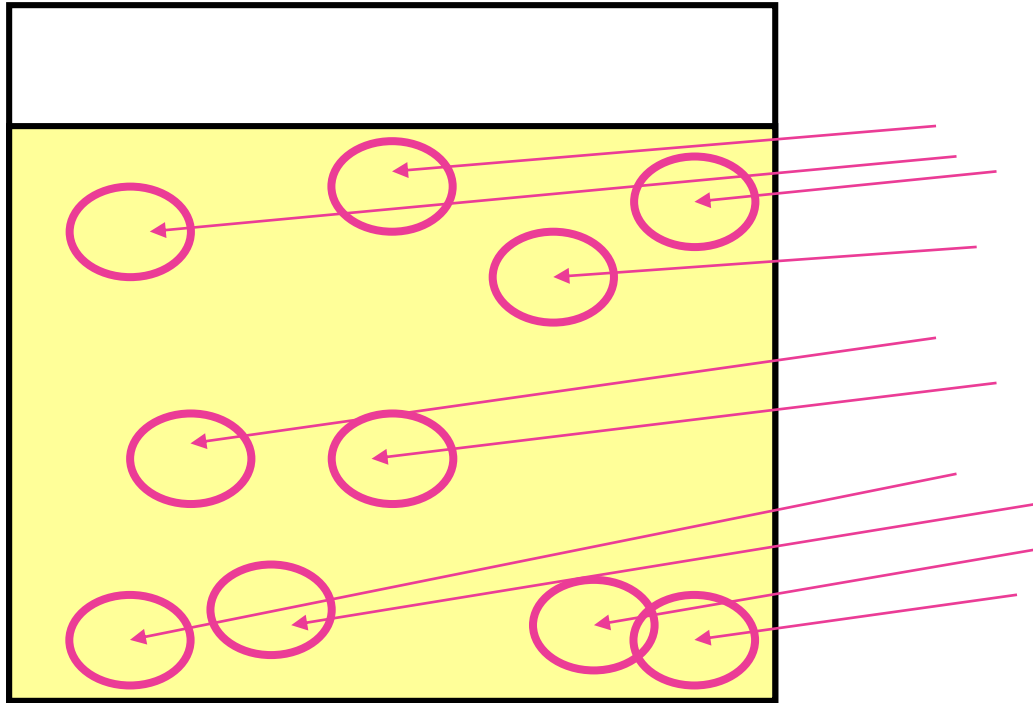
Expanded measurement uncertainty

Homogeneity

Stability

ISO/IEC 17025 + ISO Guide 34 accreditation

Certified Reference Materials  
**ISO Guide 34 Requirement: Homogeneity**



- 10 samples measured
- Samples taken from randomly chosen spots  
(top – middle – bottom)
- Contribute to overall uncertainty

## Certified Reference Materials ISO Guide 34 Requirement: : Stability Tests

- Long term stability tests at RT
- Stress tests: 3 months at 45°C



# Certified Reference Materials

## Standard and Expanded uncertainty

Certified value: is determined during batch release, stated in the respective CoA

Measurement uncertainty: during measurements, different influences need to be taken into account, which affect the measurement results and are represented as the "**Measurement uncertainty**"

The combined **standard uncertainty uc** is obtained from the standard uncertainties of the characterization(measurement), the homogeneity and the stability.

$$u_{CRM} = \sqrt{u^2_{\text{Characterisation}} + u^2_{\text{Homogeneity}} + u^2_{\text{Stability}}}$$

**Ucharacterization** is the uncertainty in accordance to DIN EN ISO/IEC 17025 which includes the contributions of the primary reference material and the measuring system (measurement uncertainty)

**Uhomogeneity** is the between-bottle variation in accordance to ISO Guide 34. The assessment of homogeneity is performed by analysis of a representative number of systematically chosen sample units,

**Ustability** is the uncertainty obtained from short-term and long-term stability in accordance to ISO Guide 34. The stability studies are the basis for the quantification of the minimum shelf life of this reference material for the unopened bottle.

## Certified Reference Materials **Organics**

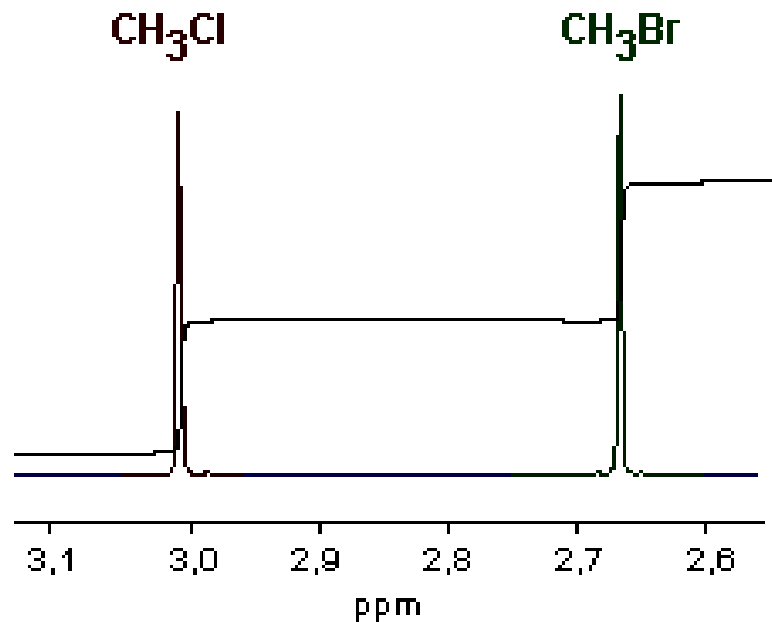
Traceability is a Challenge: ca. 50 Million Organic Compounds

-> Not always a Standard with known content is available

## Quantitative $^1\text{H-NMR}$ (qNMR)

signal intensity is:

- proportional to the No. of protons
- independent of chemical structure
- no need of chemically identical reference material





## **Sigma-Aldrich Buchs:**

ISO 17025 and ISO Guide 34  
Double Accreditation  
for CRM production using qNMR

600MHz Bruker NMR

$^1\text{H}$ ,  $^{31}\text{P}$  and  $^{19}\text{F}$  qNMR

## Certified Reference Materials Prerequisites for qNMR

- Solubility
- No reaction between analyte and IS
- No Overlap for at least one signal
- No interfering impurities

## Advantages of qNMR

- Traceability
- Low uncertainties for the measurement
- Non-Destructive
- Structure verification
- Information about impurities





# Organic *TraceCERT*: CRMs for Chromatography

## Product Groups

Amino Acids

Polyaromatic Hydrocarbons (PAHs)

Pesticides

Antibiotics

Phthalates

REACH SVHC



**More than 200 products available so far  
-> Continuously growing  
neats -> solutions**

**[www.sigma-aldrich.com/organiccrm](http://www.sigma-aldrich.com/organiccrm)**

## Reference Materials

... an overview of the  
complete range for  
chromatography

# Food and Beverage Testing Portfolio

**Food Microbiological Control**



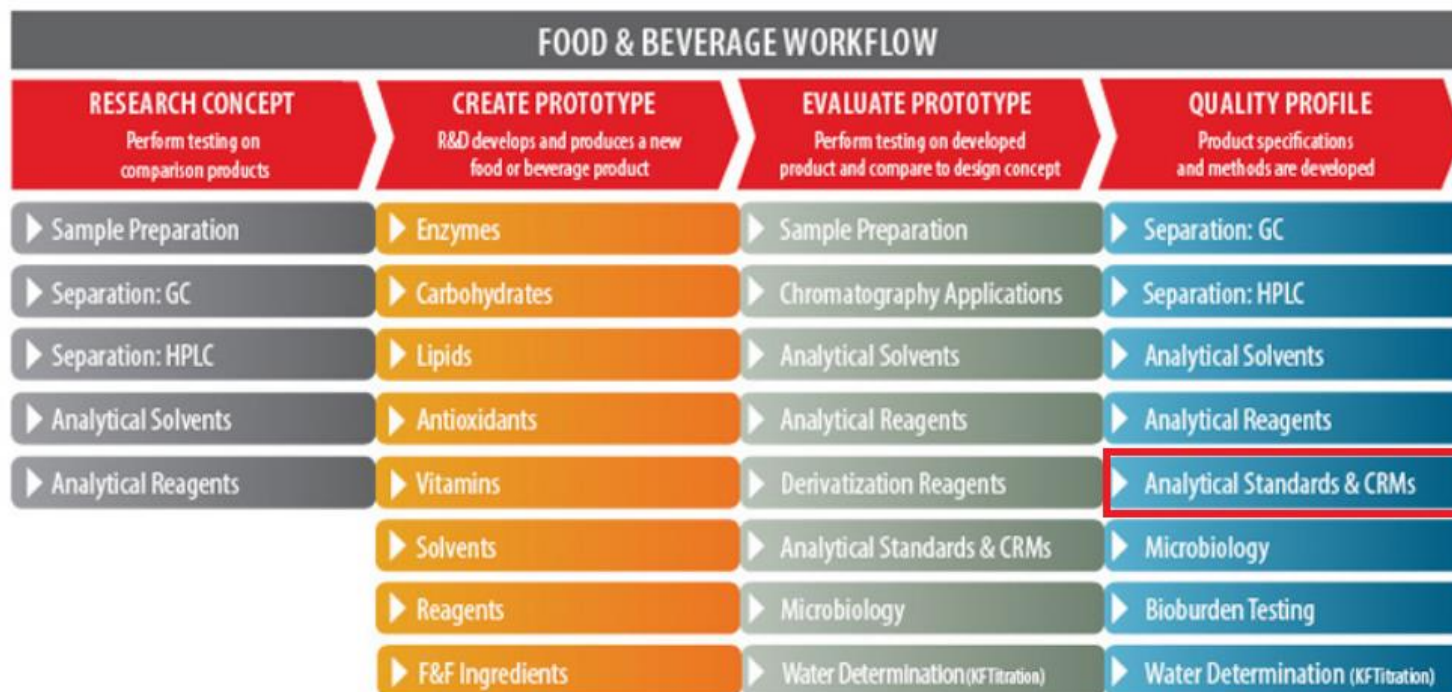
[LEARN MORE](#)

**Food and Beverage Chemical Analysis**



[LEARN MORE](#)

## Food and Beverage



- Neats and Solutions - (1415)
- Matrix CRMs (IRMM) - (104)
- Biological Reference Materials (Microorganisms) - (83)
- GMO Reference Materials, Qualitative - (109)
- Physical Properties - (7)

Key areas in ....

## Food & Beverage Reference Materials

### Food Composition/Nutrition Labeling

#### Flavors

Focus on EU positive list of authorized flavoring substances (EFSA 10/2012; EC1223/2009)

- flavor enhancers
- sweeteners

#### Food components

- Carbohydrates
- Vitamines
- Aminoacids
- Lipids & Fatty acids / FAME
- Proteins
- Food additives
- Preservatives



### Food Safety

#### Safety

- Pesticides and pesticide metabolites
- Toxins (mycotoxins)
- Veterinary drug residues
- Processing/Packaging Contaminants

#### Physical Properties

- pH
- Conductivity

# Complete Range for Chromatography



- Alcohol
- Pharmaceuticals & Illicit Drugs
- Steroids/Hormones
- Thyroid & Other Biomarkers
- Vitamins & Phytochemicals
- Internal Standards
- Environmental
- Explosives & Chemical Warfare
- View All

**TraceCERT®**

- **Organic CRMs TraceCERT**
  - CRMs for Chromatography (neats)
  - CRMs for Quantitative NMR (neats)
  - Supelco CRMs for Chromatography (solutions)

## Certified Standards for Chromatography

The organic TraceCERT products for chromatographic methods comprise many products for environmental, **food and beverage** as well as for clinical testing

IRMM (Institute for Reference Materials and Measurements):  
Merck is authorized distributor of their standards  
Certified Reference Materials (BCR, ERM, IRMM)

**Vetranal®** analytical standards



- Amino Acids, Peptides & Proteins - (84)
- Carbohydrate Standards - (87)
- Carotenoids - (26)
- DNA Standards for Food Authenticity Testing - (10)
- Flavors and Fragrance Standards - (681)
- Food & Agriculture CRMs - (104)
- Food Color Additives - (62)
- Food Residuals - (117)
- Lipids - FA/FAME/Oils/Sterols Standards - (161)
- E numbers (Food Additives) - (150)
- Organic Acid Standards - (39)
- Phytopharma - (881)
- Preservative & Antioxidants - (25)
- Sweeteners - (34)
- Trigger for Food Allergies & Intolerances - (110)
- Vitamins - (64)

# Environmental Testing

## Pesticides

- more than 1700 high purity pesticide and pesticide metabolite standards and CRMs for environmental analysis under the PESTANAL® and TraceCERT® brands

### Products:

- Pesticides
- CRM Matrix Standards and Proficiency Testing (PT)
- Pesticide Metabolite Standards
- Isotope labeled internal standards
- Multi-component solutions

[www.sigmaaldrich.com/pesticides](http://www.sigmaaldrich.com/pesticides)

### pesticides TRACECERT®

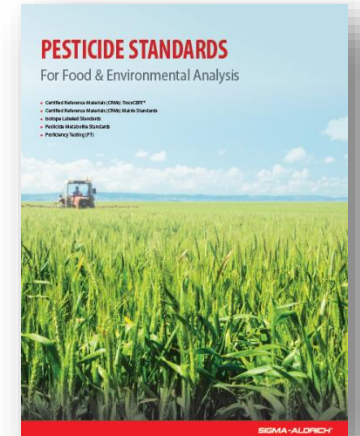
- the neat CRMs are certified by quantitative NMR (qNMR) and traceable to NIST SRM, 50 and 100 mg
- the CRM solutions are produced gravimetrically - Mass Balance Method (100% - impurities) using NIST traceable weights for balance calibration (various concentrations, solvents), 1 ml

### pesticides Matrix CRMS sigma-Aldrich RTC

- “Real world” soil or sediment natural matrices or as natural matrices, in which selected analytes have been fortified to give analytical profiles that meet the needs of analysts
- CRMs for Solids, Non-Potable and **Drinking Water** ([www.sigmaaldrich.com/ecrm](http://www.sigmaaldrich.com/ecrm))

### pesticide Metabolite standards and isotope labeled standards pestanal®

- possible conversion of active compounds of some pesticides by the target insects or plants or degradation in the environment to their metabolites
- neat standards and solutions in various solvents, all are analytical standards – no CRMs



# Pesticides Products for determining Fipronil and its metabolites

Description	Product
Fipronil certified reference material, TraceCERT®	16785
Fipronil, Pestanal, analytical standard	46451
Fipronil-(pyrazole-13C3, cyano-13C), analytical standard	79157
Fipronil sulfone, Pestanal, analytical standard	32333
Fipronil carboxamide, Pestanal, analytical standard	34519
Fipronil sulfide, Pestanal, analytical standard	34520
Fipronil-desulfinyl, Pestanal, analytical standard	41865

**TraceCERT®**  
Traceable Certified Reference Materials

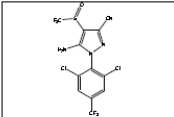
**SIGMA-ALDRICH**

## Certificate

This certificate is designed in accordance with ISO Guide 31<sup>19</sup>.

**Product name:** Fipronil

**Product no.:** 16785  
**Lot no.:** BCB58032V  
**Formula:** C<sub>12</sub>H<sub>9</sub>Cl<sub>2</sub>F<sub>6</sub>N<sub>2</sub>O<sub>2</sub>S  
**Molecular mass:** 437.16 g/mol  
**Traceability<sup>19</sup>:** NIST SRM 390b (Benzoic acid)  
**Certificate issue date:** November 14, 2016  
**Expiry:** OCT 2018



Substance	Certified value as mass fraction (g/g)	Expanded uncertainty, (u = k · u, k = 2) as mass fraction (g/g)
Fipronil	96.7 %	0.6 %

**Minimum sample:** The sample is solid at room-temperature. 10 mg is recommended as the minimal sample amount. If less material is used, it is recommended to increase the certified uncertainty by a factor of two for half of sample and a factor of four for a quarter of sample.

**Drying instruction:** This material does not require drying before use.

**Intended use:** Use this certified reference material (CRM) as calibrant for chromatography or any other analytical technique.

**Storage and handling:** The CRM should be stored in the original bottle at 2-8°C. Warm to room temperature before opening. After use the bottle should be tightly closed and protected from excessive moisture and light. Store under Argon.

**CRM operators:** A. Rück, Dr. A. Rück

**Certification body:** Dr. K.C. Schmidt

ISO Guide 34, ISO/IEC 17025, ISO 9001

Sigma-Aldrich Product Certificate, Issued in accordance with ISO 9001:2015, ISO 17025:2017 and ISO 9001:2015

**SIGMA-ALDRICH** is now **Merck**

PRODUCTS SERVICES INDUSTRIES

My Sign in ACCOUNT SUPPORT ORDER

## Testing Food for Fipronil Contamination

**Background**

In early June, eggs contaminated with the pesticide Fipronil were discovered in Belgium and the Netherlands. A treatment against ticks containing fipronil had been incorrectly used to treat poultry and, if consumed, fipronil can be harmful to humans.

The EU's executive alarm system, the Rapid Alert System for Food and Feeds (RASFF), alerted several European countries that eggs contaminated with Fipronil may have crossed their borders. Both eggs and poultry meat meant for human consumption have been affected by this scandal.

**What is fipronil?**

Fipronil is an insecticide used against pests including ants, bees, lice, ticks, cockroaches and also red mites which are commonly found in poultry. It has been shown to be acutely toxic in animal experiments when ingested orally, absorbed through the skin, or when inhaled, and should therefore not be used on food-producing animals.

The maximum allowed fipronil residue levels (the sum of both fipronil and its sulfone metabolite) for chicken eggs or chicken meat is 0.005 mg/kg of body weight.

**How to accurately test for fipronil and fipronil metabolites.**

Samples are best prepared using the QuEChERS method followed by testing using GC/MS, GC/MS/MS or LC/MS/MS techniques. We offer a complete range of analytical standards, certified reference materials, solvents, and columns for analyses of your food samples. Additionally we also develop application methods for determining fipronil and its sulfone metabolite in raw eggs, chicken meat and mayonnaise.

**Reference Materials and Standards**

Our high quality Pestanal® analytical standards and TraceCERT® certified reference materials can help ensure that measurements lower than the maximum allowed residue level can be detected, and our double accredited manufacturing process to ISO/IEC 17025 and ISO 17034 gives you the assurance of accuracy.

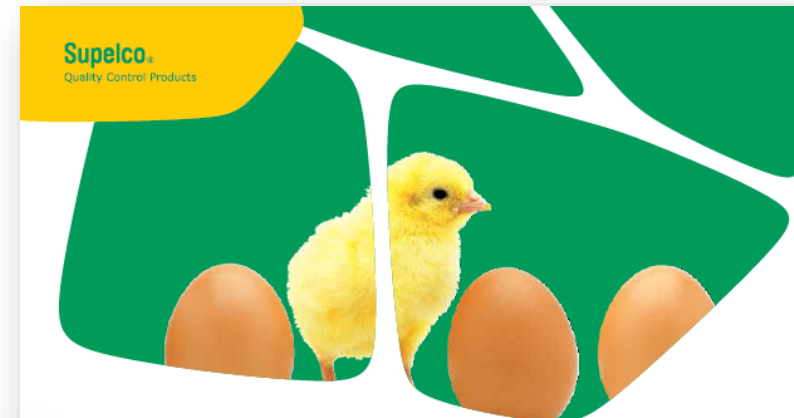
**Materials**

**Related Products**

- Analytical Standards
- Certified Reference Materials
- Environmental Pesticide Standards
- QuEChERS

**Additional information**

- Application, Analysis of Fipronil in Eggs
- Fipronil Flyer



## Food Alert — Fipronil in eggs and egg containing products

In early June, eggs tainted with the pesticide Fipronil were discovered in Belgium and The Netherlands. The EU's executive alarm system, the Rapid Alert System for Food and Feeds (RASFF), alerted France, Sweden, Britain, and Switzerland that eggs contaminated with Fipronil may have crossed their borders. On the basis of this notice, the German Federal Institute for Risk Assessment (BfR) has prepared a risk assessment of individual measurements of fipronil levels detected in eggs and poultry meat.

### Are you testing for Fipronil?

Maximum residue levels of 0.005 mg/kg for the total of Fipronil and its sulfone metabolite are allowed for food consumption. Testing using GC/MS, GC/MS/MS or LC/MS/MS techniques with high quality Pestanal Reference Material and TraceCERT® Certified Reference Materials ensure that levels lower than the maximum residue level in chicken eggs and chicken meat are achievable. Double accredited manufacturing to ISO/IEC 17025 and ISO 17034 gives you the assurance of accuracy and of correct decision making.

### What is Fipronil?

Fipronil is a broad-spectrum insecticide used to combat insects such as fleas, lice, ticks, cockroaches and mites. The use of this insecticide on food-producing animals (livestock) is not permitted.

The life science business of Merck operates as MilliporeSigma in the U.S. and Canada.

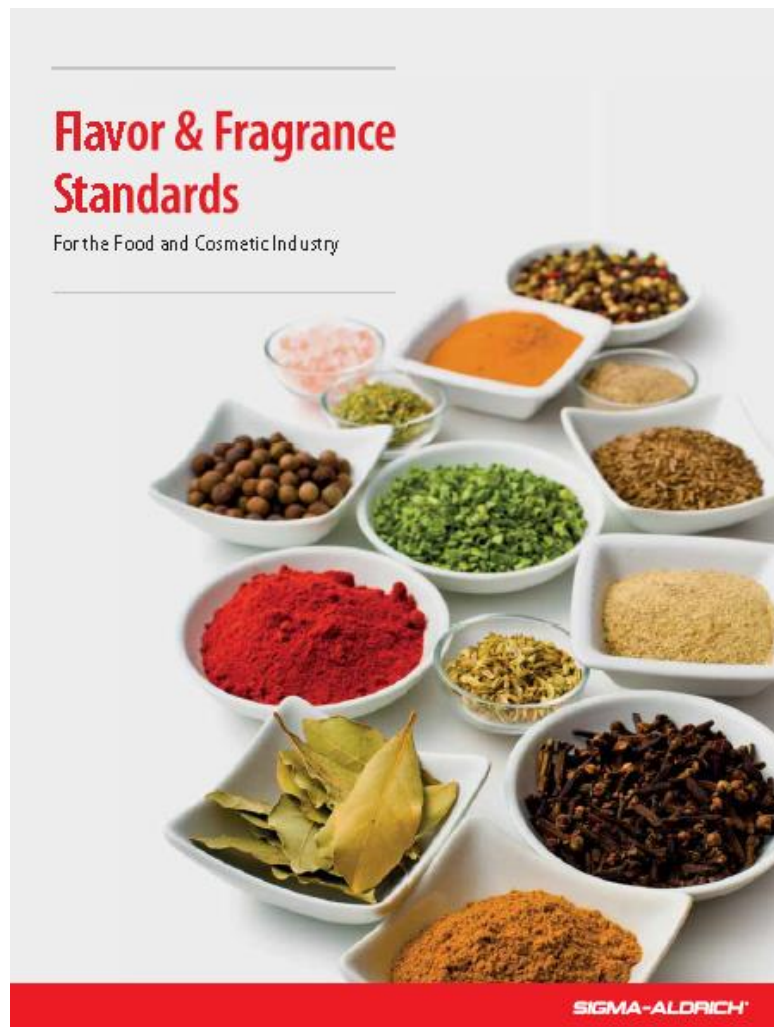


**MERCK**

**MERCK**

# Food & Beverage Testing

## Flavors & Fragrances



Certified reference materials (CRMs)  
*TraceCERT®*

- Allergenic compounds
- Sweeteners
- Flavor enhancers
- Essential oils

CRMs fulfilling both ISO 17025 and ISO Guide 34  
the neat CRMs are certified by quantitative NMR (qNMR) and traceable to NIST standards

[Learn more](#)



# Flavors & Fragrances Analysis Workflow

## Sample Preparation

Automated Solutions  
for Sample Preparation

Analytical Reagents

Derivatization Reagents

Solid Phase Microextraction (SPME)

Solid Phase Extraction (SPE)

Filtration Technologies & Millex®

## Separation

Columns with Superior Resolution  
for F&F Compounds

### GC

Ionic Liquid GC Columns – Unique Selectivity  
for essential oil separations

SLBms GC-MS Columns – Durable and  
sensitive columns e.g. for allergen analysis

Equity-1 GC Columns – Ideal where a non-  
polar column is needed

SUPELCOWAX® 10 GC Columns – Polar  
column e.g. for aromatics and F&F  
compounds

Watercol™ GC Columns – Measurement of  
aqueous samples and/or Water

### HPLC

Chiral GC Columns – Widest selectivity  
range for enantiomer separations

Ascentis® Express U/HPLC Columns –  
Fused-Core® Technology for highest  
efficiency

Chromolith® HPLC Columns – Lowest back  
pressure & highest robustness against dirty  
samples

Purospher® STAR UHPLC Columns –  
Capacity, Inertness & highest reproducibility

## Detection & Analysis

Reliable Products  
for Accurate Results

### Standards

Flavor & Fragrance Standards

Antioxidant & Preservative Standards

Phytochemical Standards

Color Standards

Stable Isotopes

Microbiology Certified Reference Materials

### Reagents

Analytical Reagents

Separation – GC

Separation – HPLC & UHPLC

Spectroscopy

Inorganic Trace Analysis

Karl Fischer with Aquastar®

Microbial Testing Solutions

Solvents

Labware

# Flavors & Fragrances

## Analytical workflow

### Properties

Related Categories: Analytical/Chromatography

volatiles in coffee

volatiles in coffee in Analytical/Chromatography

3 Product Results | Match Criteria: Analytical/Chromatography

APPLICATION ▲

**GC Analysis of Volatiles in Green Arabica Coffee Beans using 50/30 µm DVB/Carboxen/PDMS Fiber for SPME,**

**GC Analysis of Volatiles in Roasted Arabica Coffee Beans using 50/30 µm DVB/Carboxen/PDMS Fiber for SPME,**

**GC Analysis of Volatiles in Roasted Coffee Beans using a 50/30 µm DVB/Carboxen/PDMS Fiber for SPME,**

sample:	Roasted Coffee Beans
liner:	0.75 mm I.D.
applicability:	application for GC
applicability:	application for SPME
Featured Industry:	Food and Beverage

Show Fewer Properties ▲

### GC Analysis of Volatiles in Roasted Coffee Beans on Omegawax™ after SPME using a 50/30 µm DVB/Carboxen/PDMS Fiber

application for SPME, application for GC

Overview Safety & Documentation Protocols & Articles Related Products

#### Properties

Related Categories	Analytical/Chromatography, Analysis (Coffee), Capillary Columns/Retention Gas Industry / application, More...
test parameters	sample/matrix: 2 g of coffee beans SPME fiber: 50/30 µm polydimethylsiloxane (PDMS) extraction: headspace period of 10 min, at 80 °C exposure of 40 min, alternate clockwise and counter-clockwise at 600 rpm description process: 2 µl of coffee beans column: Omegawax 250 µm (24138) oven: 40 °C (6 min), 4 to 230 °C (2 min) inj. temp.: 280 °C

#### Description

**Analysis Note**  
Headspace-SPME followed by GC analysis was used to analyze volatile compounds in roasted coffee. SPME is ideal for analyzing the headspace of solid samples like coffee beans. The Carboxen/DVB/PDMS fiber is the fiber of choice for analyzing the wide range of analytes commonly found in roasted coffee beans. The relatively polar Omegawax 250 GC column provided high resolution.

**Legal Information**  
Omegawax is a registered trademark of Sigma-Aldrich.

**Other Notes**  
Complementary products: Birtines, View, Gas Purification and Gas Management, Inlet Liners, Glass Wool, and Specialized Hand Tools, Septa and Specialized Hand Tools, Column Ferrules, Nuts, and Specialized Hand Tools

#### Analyte

Description	Methyl acetate
	2-Methylfuran
	Ethyl acetate
	2-Butanone
	2,3-Dihydro-5-methylfuran
	Ethyl alcohol
	2,5-Dimethylfuran
	2,3-Pentanedione

### Description

**Analysis Note**  
Headspace-SPME followed by GC analysis was used in this application to analyze volatile compounds in roasted coffee. SPME is ideal for analyzing the headspace of solid samples like coffee beans. The Carboxen/DVB/PDMS fiber is the fiber of choice for analyzing the wide range of analytes commonly found in roasted coffee beans. The relatively polar Omegawax 250 GC column provided high resolution.

1. Dimethylformaldehyde	15. β-pinene	27. γ-Terpinene	39. 2-Ethyl-6-methylpyrazine
2. Methyl acetate	16. Sabinene	28. 2-Methylpyrazine	40. 2-Ethyl-5-methylpyrazine
3. 2-Methylfuran	17. 3-Pentene-2-one	29. p-Cymene	41. 2,3,5-Trimethylpyrazine
4. Ethyl acetate	18. 2,3-Hexanedione	30. 2,5-Dimethylpyrrole	42. Furfural
5. 2-Butanone	19. Methyl-1H-pyrrole	31. Acetoin	43. Acetol acetate
6. 2,3-Dihydro-5-methylfuran	20. 3,4-Hexanedione	32. 2,5-Dimethylpyrazine	44. 2-Furfuryl-5-methylsulfide
7. 2-Methyl butanal	21. 2-Vinyl-5-methylfuran	33. 2,6-Dimethylpyrazine	45. Furfuryl formate
8. 3-Methyl butanal	22. β-Myrcene	34. 2-Ethylpyrazine	46. 2-Acetyl furan
9. Ethyl alcohol	23. α-Terpinene	35. 2,3-Dimethylpyrazine	47. Pyrrole
10. 2,5-Dimethylfuran	24. Pyridine	36. 4-Heptanone	48. Furfuryl acetate
11. 2,3-Butanedione	25. Limonene	37. 2-Methyl-2-cyclopentenone	49. Linalool
12. 3-Hexanone	26. Pyrazine	38. 3-Ethylpyridine	50. Linalyl acetate
13. 2,3-Pentanedione			51. 5-Methylfurfural
14. Hexanal			52. 2-Furfuryl furan
			53. N-Methyl-2-formylpyrrole
			54. γ-Butyrolactone
			55. 1-(2-Furyl)-3-butanone
			56. 2-Acetyl-1-methylpyrrole
			57. Furfuryl alcohol
			58. N-Acetyl-4(5H)pyridine
			59. 1-(5-Methyl-2-furyl)-2-propanone
			60. Furfuryl pyrrole
			61. 2-Methoxyphenol
			62. 3-Butenone
			63. Phenylethyl alcohol
			64. 4-Pyran-4-one
			65. 2-Acetylpyrrole
			66. Furfuryl ether
			67. Pyrrole-2-carboxaldehyde

# Food & Beverage Testing

## Food additives (E numbers)

- E100–E199 (color additives) - (29)
  - E200–E299 (preservatives) - (30)
  - E300–E399 (antioxidants, acidity regulators) - (23)
  - E400–E499 (thickeners, stabilizers, emulsifiers) - (8)
  - E500–E599 (acidity regulators, anti-caking agents) - (3)
  - E600–E699 (flavor enhancers) - (7)
  - E700–E799 (antibiotics) - (10)
  - E900–E999 (miscellaneous) - (17)
  - E1000–E1599 (additional chemicals) - (17)
- some E number food dyes are not allowed in the EU and US, some are still used **illicitly** in food products
  - a broad range of neat standards – analytical standards and CRMs

**E numbers** are codes for **food additives** that have been assessed for use within the European Union (the "E" prefix stands for "Europe"). They are commonly found on food labels throughout the European Union. Safety assessment and approval are the responsibility of the European Food Safety Authority (EFSA).

e.g., E numbers are used as codes for **food color additives**

E numbers 100-199 include not only artificial food colors derived from petroleum, but also natural additives originating from sources such as vegetables, insects, or mixtures made from natural foodstuffs

## Food Testing Phytochemical Standards

Natural product **CRMS, primary and secondary standards** for the testing of plant, herb, or dietary supplements for identity, strength, or purity. Also designed for nutrition research and metabolomics.

Range includes alcohols and phenols, aldehydes and ketones, alkaloids, flavonoids, glucosinolates, and isoprenoids to organic acids and esters, phenylpropanes, quinones, and tannins.

Suitable for **LC OR LC-MS/MS** and other analytical techniques.

Manufactured by **HWI Analytik** and exclusive to Merck  
[sigma-aldrich.com/medicinalplants](https://sigma-aldrich.com/medicinalplants)



# Food Testing Mycotoxins



[www.sigmaaldrich.com/mycotoxins](http://www.sigmaaldrich.com/mycotoxins)

Because of the regulations limiting levels in foodstuffs for at least 100 countries, we provide a wide range of mycotoxin standards that includes:

- **Neats**
- Mycotoxin **Single-Component Standard Solutions** – various concentrations and solvents
- Mycotoxin **Mixture Standard Solutions** For Multi-Analyte Detection
- **13C Isotopically Labeled Internal Standards Solutions** for LC-MS
- **Dried Down** Mycotoxin Standards
- Certified Matrix Reference Materials (CRMs) **IRMM®**, produced with raw materials to more accurately resemble actual samples in their natural state, compound feed, maize, wheat and peanut butter reference materials for an accurate determination of detection limits as well as validation of methods

# Mycotoxin Reference Materials (Neats)

## Mycotoxin Single-Component Standard Solutions

Cat. No.	Product Description	Pkg (mg)
32927	3-Acetyldeoxynivalenol	5
32928	15-Acetyldeoxynivalenol	5
32754	Aflatoxin B <sub>1</sub>	5
32755	Aflatoxin B <sub>2</sub>	5
32756	Aflatoxin G <sub>1</sub>	5
32757	Aflatoxin G <sub>2</sub>	5
32943	Deoxynivalenol	5
32936	Fumonisin B <sub>1</sub>	5

Cat. No.	Product Description	Pkg (mg)
33438	Fusarenon-X	5
32932	Neosolaniol	5
32929	Nivalenol Hydrate	5
32937	Ochratoxin A	5
32759	Patulin	5
32609	Sterigmatocystin	5
33947	T-2 Toxin	5
32939	Zearalenone	5



Cat. No.	Product Description	Concn (µg/g)	Solvent	Pkg (mg)
34132	3-Acetyldeoxynivalenol	100	Acetonitrile	2
34133	15-Acetyldeoxynivalenol	100	Acetonitrile	2
34129	Acetyl-Deoxynivalenol-D <sub>3</sub>	100	Acetonitrile	2
34029	Aflatoxin B <sub>1</sub>	2	Acetonitrile	2
34034	Aflatoxin B <sub>2</sub>	0.5	Acetonitrile	2
34032	Aflatoxin G <sub>1</sub>	2	Acetonitrile	2
34033	Aflatoxin G <sub>2</sub>	0.5	Acetonitrile	2
34031	Aflatoxin M <sub>1</sub>	0.5	Acetonitrile	2

## Mycotoxins

### **<sup>13</sup>C Isotopically Labeled Internal Standards Solutions for LC-MS**

Cat. No.	Product Description	Concn (µg/g)	Solvent	Pkg (mg)
32962	3-Acetyldeoxynivalenol- <sup>13</sup> C <sub>17</sub>	25	Acetonitrile	1
32764	Aflatoxin B <sub>1</sub> - <sup>13</sup> C <sub>17</sub>	0.5	Acetonitrile	1
32771	Aflatoxin B <sub>2</sub> - <sup>13</sup> C <sub>17</sub>	0.5	Acetonitrile	1
32772	Aflatoxin G <sub>1</sub> - <sup>13</sup> C <sub>17</sub>	0.5	Acetonitrile	1
32777	Aflatoxin G <sub>2</sub> - <sup>13</sup> C <sub>17</sub>	0.5	Acetonitrile	1

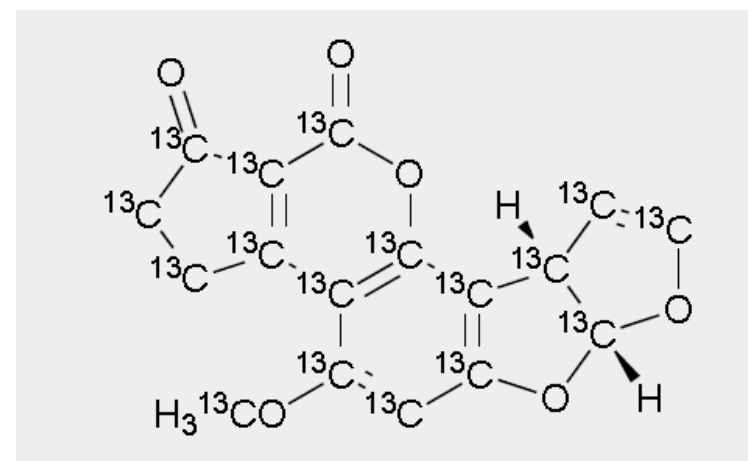


Figure 1. Molecular structure of a fully <sup>13</sup>C isotope-labeled Aflatoxin B<sub>1</sub>-<sup>13</sup>C<sub>17</sub>

# Dried Down Mycotoxin Standards – analytical standards

## Mycotoxin Mixture Standard Solutions For Multi-Analyte Detection

Cat. No.	Product Description	Concn (µg/g)	Pkg (mg)
35758	Alternariol	100	0.1
35762	Alternariol-9-methyl ether	100	0.1
37012	Beauvericin (BEA)	100	0.1
35878	Citreoviridin A	100	0.1
35970	Meleagrín	100	0.1
37025	Retrorsín	50	0.05
35976	Stachybotrylactam	100	0.1
35977	Tentoxin	100	0.1

Cat. No.	Product Description	Components	Concn (µg/g)	Solvent	Pkg (mg)
34036	Aflatoxin Mix 4	B <sub>1</sub> , G <sub>1</sub> B <sub>2</sub> , G <sub>2</sub>	2 0.5	Acetonitrile	1, 2
33415	Aflatoxin Mix 4	B <sub>1</sub> , G <sub>1</sub> , B <sub>2</sub> , G <sub>2</sub>	20 (each)	Acetonitrile	2
34143	Fumonisin Mix	FB <sub>1</sub> , FB <sub>2</sub>	50 (each)	Acetonitrile/Water	2
32926	Trichothecene Mix	3-AcDON, DON, NIV, FusX, HAT-2, T-2, DAS, ZON	10 (each)	Acetonitrile	1
34134	B-Trichothecen Mix	DON, NIV, 3 – AcDON, 15 – AcDON	100	Acetonitrile	2





## Matrix Mycotoxin CRMs

# Certified Matrix Reference Materials (CRMs) For Mycotoxin Analysis

we offer compound feed, maize, wheat and peanut butter reference materials from IRRM

Cat. No.	Product Description	Component	Concn (µg/g)	Pkg (mg)
BCR375	Compound Feed (Aflatoxin blank)	Aflatoxin B <sub>1</sub>	<1	50
ERMBE375	Compound Feedingstuff (Aflatoxins, very low level)	Aflatoxin B <sub>1</sub>	2.6	2×75
		Aflatoxin B <sub>2</sub>	0.20	—
		Aflatoxin G <sub>1</sub>	0.4	—
		Aflatoxin G <sub>2</sub>	<0.2	—
ERMBE376	Compound Feedingstuff (Aflatoxins, high level)	Aflatoxin B <sub>1</sub>	12.9	2×75
		Aflatoxin B <sub>2</sub>	0.68	—
		Aflatoxin G <sub>1</sub>	5.2	—
BCR262R	Defatted Peanut Meal (Aatoxin B1, blank)	Aflatoxin B <sub>1</sub>	<3.0	100
ERMBC716	Maize (Zearalenone, very low level)	Zearalenone	<5	60
ERMBC717	Maize (Zearalenone, low level)	Zearalenone	83	60
BCR377	Maize Flour (Deoxynivalenol, blank)	Deoxynivalenol	<50	150
BCR401R	Peanut Butter (Aflatoxin, low level)	Aflatoxin B <sub>1</sub>	<0.2	100
		Aflatoxin B <sub>2</sub>	(each)	—
		Aflatoxin G <sub>1</sub>	—	—
		Aflatoxin G <sub>2</sub>	—	—
BCR471	Wheat (Ochratoxin A, blank)	Ochratoxin A	<0.6	55

# Mycotoxin Solutions CRMs from IRMM®

## Certified Reference Material (CRM) Solutions – TraceCERT®



Cat. No.	Component	Concn (µg/g)	Solvent	Pkg (mg)
ERMAC057	Aflatoxin B <sub>1</sub>	3.79	Acetonitrile	4
ERMAC058	Aflatoxin B <sub>2</sub>	3.80	Acetonitrile	4
ERMAC059	Aflatoxin G <sub>1</sub>	3.78	Acetonitrile	4
ERMAC060	Aflatoxin G <sub>2</sub>	3.80	Acetonitrile	4
IRMM315	4-Deoxynivalenol	25.1	Acetonitrile	4
IRMM316	Nivalenol	24.0	Acetonitrile	4
BCR423RM	Aflatoxin M <sub>1</sub>	9.93	Chloroform	2.5
ERMAC699	Zearalenone	9.95	Acetonitrile	4

Cat. No.	Component	Concn (µg/g)	Solvent	Pkg (mg)
CRM46323	Aflatoxin B <sub>1</sub>	3	Benzene:Acetonitrile (98:2)	1
CRM44647	Aflatoxin B <sub>1</sub>	20	Methanol	1
CRM46324	Aflatoxin B <sub>2</sub>	3	Benzene:Acetonitrile (98:2)	1
CRM46325	Aflatoxin G <sub>1</sub>	3	Benzene:Acetonitrile (98:2)	1
CRM46326	Aflatoxin G <sub>2</sub>	3	Benzene:Acetonitrile (98:2)	1
CRM46319	Aflatoxin M <sub>1</sub>	10	Acetonitrile	1

# Food & Beverage Standards: Components

## Lipids & Fatty Acids

A diverse range of reference materials suitable for nutritional studies and food labelling, including -

› Cyclopropane Fatty Acid Derivates

› Fatty Acid Methyl Esters (FAMES)

› Glycerides & Glycerols

› Edible Oils

› Fatty Acid Tryptamides

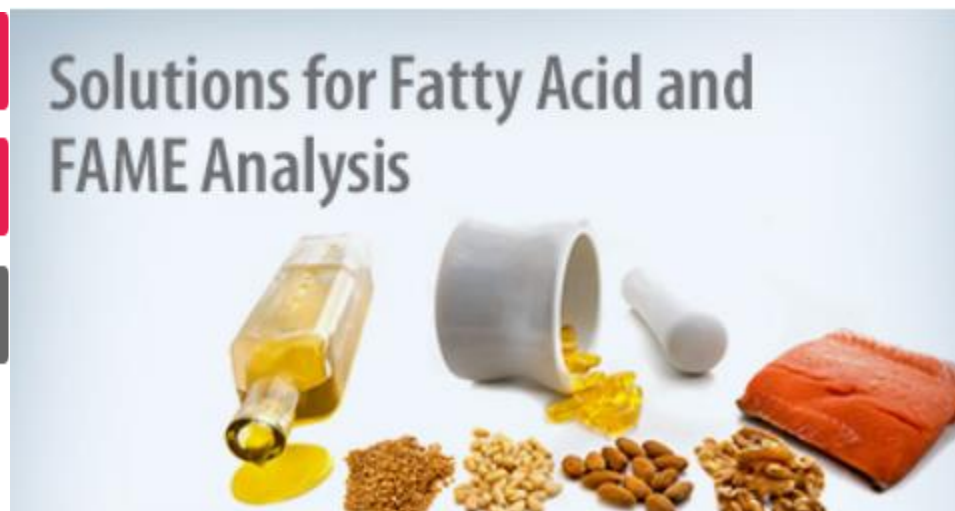
› Sterols

› Fatty Acid Amides

› Free Fatty Acids

› View All

› Fatty Acid Degradation Products



Neats and solutions  
Analytical standards, CRMs

# Analytical Standards and Certified Reference Material

## Our Custom Capabilities

### Organic Custom Standards

We can prepare chemical standards for you:

#### 1. environmental

2. food & beverage
3. pesticide & metabolite
4. petroleum
5. chemical
6. Pharmaceutical

- Includes Safety Data Sheet (SDS)
- Certificate of Composition

### Custom Services from Cerilliant®

- Custom Reference Standards
- Custom Organic Synthesis
- Analytical Services
- Custom Packaging
- Inventory Management and Distribution of Client Reference Materials

Specifications

**Mixture Description (for label) :**

**Solvent(s) :**

**Volume or Weight / ampul:**

1 mL  100 mg

5 mL  500 mg

10 mL  Other (please specify)

**Number of units :**  
(typically minimum order quantity is 4)

**Concentration Units :**

ng/mL  vol / vol %

µg/mL  wt. / wt. %

mg/mL  wt. / vol.

Other (please specify)

Components	CAS No. (Optional)	Concentration

<http://www.sigmaaldrich.com/analytical-chromatography/analytical-standards/standards-quote.html>

# Analytical Standards and Reference Materials

## Further information sources

Further  
information

### Analytix

Journal for new topics in Standards and reagents

- Published 5 times per year.
- Subscribe at [sigma-aldrich.com/analytix](https://sigma-aldrich.com/analytix)

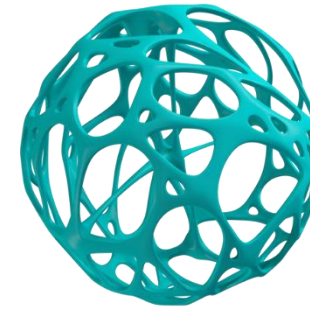
### Standards Explorer

- Quickly find the standard you are looking for
- Various search parameters, including CAS #, agency method

### Reporter

- Quarterly issued, analytical applications
- Subscribe at [sigmaaldrich.com/reporter](https://sigmaaldrich.com/reporter)

# Our Advantage



## Supply Chain

World Wide distribution  
Compliance Expertise  
Fast delivery (high stock levels)

## Customer & Technical Service

Highly educated personnel  
Presence in most countries / languages  
eCommerce

## Bundling Possibilities convenient & cost saving

One of the largest portfolio  
Analytical reagents  
Consumables, Sample Preparation,  
Lab Water, Labware

## Technical expertise

Five double accredited sites (ISO  
17025 and ISO Guide 34)  
High percentage of in-house  
production



Let us know what else you need to better support your work

**Thank you !**

