



PROPER STANDARD PREPARATION

Don Shelly, LGC Standards
NEMC 2019

LGC Quality | ISO 17034/ISO Guide 34 ISO/IEC 17025 | ISO 9001



I NEED THE STANDARD TODAY!

- “Back in the day” before premade mixes and custom blends, we made our own stock and working standards from “neat” materials
- Neat: the analyte in its purest undiluted form



NECESSARY SKILLS

- How to use a Class A volumetric pipette
- How to use a microliter syringe
- How to use a Class A volumetric flask
- How to use an analytical balance
- Basic algebra



CERTIFIED REFERENCE MATERIAL VS. REFERENCE MATERIAL

CRM (CoA)

- Manufactured by ISO 17034 accredited producer and tested in ISO 17025 accredited lab
- Characteristics determined by metrologically valid procedure for stated properties
- Stated values
- Statement of metrological traceability
- Homogeneity data
- Long term stability results
- **Total uncertainty value**
- Fit for purpose

RM (CoC)

- Product identity
- Purity
- Characterization details
- Fit for purpose

CERTIFIED REFERENCE MATERIAL

CRM (CoA) must follow ISO Guide 31

- 5.2.1 Title of the document
- 5.2.2 Unique identifier of the RM
- 5.2.3 Name of the RM
- 5.2.4 Name and contact details of the RM producer
- 5.2.5 Intended use
- 5.2.6 Minimum sample size
- 5.2.7 Period of validity
- 5.2.8 Commutability
- 5.2.9 Storage information
- 5.2.10 Instructions for handling and use
- 5.2.11 Page number
- 5.2.12 Document version

CERTIFICATE OF ANALYSIS

LGC

DR

EHRENSTORFER™

Certified Reference Material

Product Name

PCB CONGENER MIX, 17 COMPONENTS, 100 µg/mL IN ISOOCTANE

Product Code

DRE-GS09000465IO

Lot Number

371607

Format

Multicomponent Solution

Expiry Date

14 Oct 2021

Storage Temp

≤ -10 °C

REFERENCE MATERIAL CERTIFICATE

ISO 17034

This certificate is designed in accordance with ISO 17034 and ISO Guide 31. This certified reference material (CRM) was designed, produced and verified in accordance with ISO/IEC 17025, ISO 17034 and a registered quality management system ISO 9001.

Compound Name	CERTIFIED Concentration (µg/mL)	Expanded Uncertainty U (µg/mL)	CAS	Lot Number	Combined Purity (%)	Amount (mg)	RT (min)
2,2,5-trichlorobiphenyl (BZ# 18)	100.4	5.2	37680-65-2	515.31P	99	2.50	18.58
2,4,4'-trichlorobiphenyl (BZ# 28)	100.2	5.2	7012-37-5	516.2861P	99	2.50	19.87
2,2,5,5'-tetrachlorobiphenyl (BZ# 52)	99.9	5.2	35693-99-3	518.42P	99.7	2.50	20.67
2,4,4',5-tetrachlorobiphenyl (BZ# 74)	100.4	5.1	32690-93-0	749.5.3.4P	100	2.50	21.88
2,2,3,5,6-pentachlorobiphenyl (BZ# 95)	98.6	5.1	38379-99-6	767.5.6P	99	2.50	22.00
2,2',4,5,5'-pentachlorobiphenyl (BZ# 101)	100	5.1	37680-73-2	520.5.5P	97.3	2.50	22.47
2,2',4,4',5-pentachlorobiphenyl (BZ# 99)	99.12	5.1	38380-01-7	771.257.11P	99.5	2.50	22.56
2,3,3',4',6-pentachlorobiphenyl (BZ# 110)	99	5.1	38380-03-9	779.421.1P	99	2.50	23.14
2,2',3,4',5',6-hexachlorobiphenyl (BZ# 149)	99.79	5.1	38380-04-0	811.421.1P	99	2.50	23.55
2,3',4,4',5-pentachlorobiphenyl (BZ# 118)	99.9	5.1	31508-00-6	539.421.1P	99.5	2.50	23.63
2,2',4,4',5,5'-hexachlorobiphenyl (BZ# 153)	100.1	5.1	35065-27-1	525.5.5.1P	99.9	2.50	24.00
2,3,3',4,4'-pentachlorobiphenyl (BZ# 105)	99.59	5.1	32598-14-4	521.5.9P	99	2.50	24.09
2,2',3,4,4',5'-hexachlorobiphenyl (BZ# 138)	99.7	5.1	35065-28-2	541.5.6P	99.3	2.50	24.47
2,2',3,4',5,5',6-heptachlorobiphenyl (BZ# 187)	101	5.2	52663-68-0	542.4.2.1P	99	2.50	24.71
2,3,3',4,4',5-hexachlorobiphenyl (BZ# 156)	100.6	5.2	38380-08-4	659.421.11P	99	2.50	25.31
2,2',3,4,4',5,5'-heptachlorobiphenyl (BZ# 180)	101.3	5.3	35065-29-3	527.286.1P	98.2	2.50	25.55
2,2',3,3',4,4',5-heptachlorobiphenyl (BZ# 170)	100.1	5.2	35065-30-6	526.4.2.1P	99	2.50	25.99

None


The producer certifies that this reference material meets the specification stated in this certificate until the expiry date, provided it is stored unopened at the recommended temperature herein. Product warranties for this reference material are set out in the terms and conditions of purchase.

CERTIFIED BY

HuiChen Stavros, Ph.D.

CERTIFIED ON

10 Apr 2019



RM

Release



Version 1

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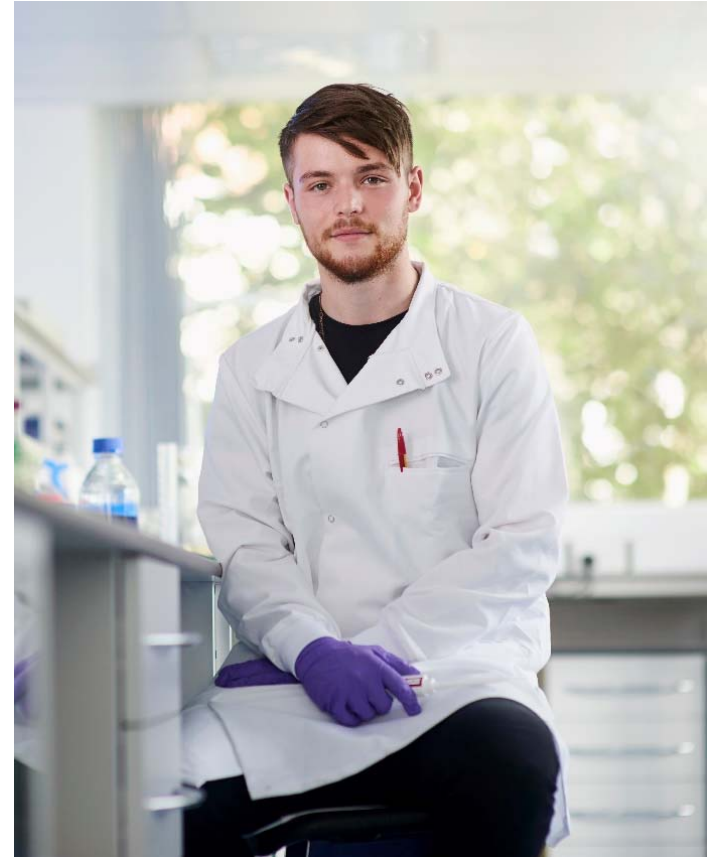
DR EHRENSTORFER™

CERTIFICATE OF ANALYSIS

	<div>REFERENCE MATERIAL CERTIFICATE</div>	<div>ISO 17034</div>
<div>Intended Use</div> <p>This CRM is intended for use in a laboratory as a calibration and quality control standard or in method development for analytical techniques.</p>	<div>Traceability</div> <p>The balances used for gravimetric measurements are calibrated with weights traceable to the national standards (NIST). The calibration of the balances is verified daily internally and annually by an external accredited calibration service. Only Class A glassware is used for volumetric measurements.</p> <div>Homogeneity</div> <p>Random replicate samples of the final packaged CRM have been analysed to prove homogeneity consistent with ISO 17034.</p> <div>Storage</div> <p>The CRM should be stored in the original sealed bottle at the indicated temperature.</p>	<div>Method of Preparation</div> <p>The certified value is based on gravimetric and volumetric preparation of this CRM. This CRM has been confirmed by the appropriate analytical techniques.</p> <div>Batch Information</div> <p>Solvent: Isooctane, Lot no. 17040656, 25 mL</p> <p>None</p>
<div>Uncertainty</div> <p>The certified value(s) and uncertainty(ies) are determined in accordance with ISO 17034 with an 95% confidence level ($k=2$). Uncertainty is based on the Total Combined Uncertainty, including uncertainties of preparation, purity of neat materials, homogeneity, long-term stability testing, and transportation stability.</p> <div>LGC Group</div> <p>7290-B Investment Drive North Charleston, SC 29418 United States T +1 843 763 4884 F +1 866 509 5146 E dr.ehrenstorfer@lgcgroup.com</p>	<div>The producer of this reference material is registered to ISO 9001:2015 under IZ391-IS4 by NSF-ISR and accredited to ISO 17025:2005 and ISO 17034:2016 by A2LA with the accreditation numbers 3031.01 and 3031.02.</div> <div>  </div> <div> ISO 17034 Accredited Reference Material Producer Cert. No. 3031.02 </div>	<div>Instructions for Use</div> <p>The CRM should be used shortly after opening to avoid concentration changes due to evaporation. It is recommended to use 1 μL as the minimum sample size. If storage after opening is necessary, it should be transferred to an amber vial with minimum head space and a Teflon lined silicon septum. If handled as recommended, use period after opening is a maximum of 8442 days for an estimated 5% drift in concentration as a result of analyte and/or solvent transpiration. Visit the support section of our website lgcstandards.com for a series of Dr. Ehrenstorfer Tech Tip videos and frequently asked questions.</p>
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CRM: TOTAL COMBINED UNCERTAINTY

- Characterization:
Manufacturing Process
- Homogeneity:
Manufacturing Process
- Transportation:
Shipping, impacts shelf life
- Long term stability:
Chemistry; impacts shelf life and is the major contributor to uncertainty



SOLVENT SELECTION

- Solvent miscible/soluble with analytes
- Intermediate solvent
- Solvent analyte interaction
- Homogeneity
- Precipitation of analyte
- Preservatives
- Fit for purpose (Electron Capture Detectors)



SOLVENT MISCIBILITY/SOLUBILITY

- **Miscibility:** will a solvent form a solution with another solvent
- Methanol and hexane are not miscible
- **Solubility:** will the solvent dissolve the analyte
- Polar analytes in polar solvents and non-polar analytes in non-polar solvents
- Fit for purpose (Electron Capture Detectors)



STABILITY

- Organic mixtures are sealed in ampules so the primary variables we measure are temperature and composition.
- Composition changes primarily through chemical reactions
 - Analyte \rightarrow Analyte
 - Analyte \rightarrow Solvent
- Chemical reaction rates change with temperature
 - Can use Arrhenius Equation



SHELF LIFE DETERMINATION

- Classical Method: real time storage data
 - Test retains at intervals
- Historical Data/Product Knowledge
- Accelerated Method: conservative estimate of shelf life
 - Stress product with appropriate environmental variables to accelerate product failure (95% recovery or worse)
 - Used when creating custom products



ACCELERATED METHODS

- Assumes that product failure and the rate of degradation increases if environmental conditions are made more harsh than the stated storage conditions
- It is critical that the certified values for the analytes are within the stated uncertainties for the specified shelf life



ACCELERATED STRESS EXPERIMENT

- One sample retain is placed at storage temperature
- Additional retains are heat stressed at 3 or more temperatures between the storage temperature and 100o C for a given time
- All retained samples are analyzed sequentially
- Results entered into Arrhenius equation and plotted

SOLVENT → ANALYTE INTERACTION

- Analyte-solvent interaction can impact solution pH and redox potential over time
- Acetonitrile can break down into Acetamide over time and become basic
 - This will degrade urea-based pesticides
- Chlorine free radicals in methylene chloride can be protonated by methanol and form HCl
 - Acid sensitive analytes can decompose or partition from solution



STABILITY

- Benzaldehyde: Benzidine reacts with HCl to form new derivatives
- Acetonitrile turns basic as it ages causing urea-based pesticides to degrade
- We use preservatives to maintain the correct pH
- 0.1% formic acid in acetonitrile
- Ammonium hydroxide in methanol



STABILITY

- Test your standard regularly to determine its shelf life
- Store at the temperature that is appropriate for the standard (usually $< -10^{\circ}\text{C}$)
Hint: see the storage temperature on CoA
- Standards using water: store in the refrigerator
- EPA Method 1664 standards: store at room temperature
- Avoid light: primarily for benzidine, PAHs, organophosphates, etc.

MORE ABOUT STABILITY

- After the product is opened, it is the lab's responsibility to determine its shelf life
- You must store the product at the temperature that is written on the CoA
- Avoid light for photosensitive analytes such as benzidine, PAHs, organophosphates, etc.



AS PREVIOUSLY MENTIONED...

- Always keep in mind that chemicals interact
- Analyte → solvent interactions can impact pH and redox potential over time
- Chlorinated hydrocarbons in methanol: acids are formed over time
- Ketones degrade very quickly under these conditions
- PAHs will degrade quickly in poor quality ethyl acetate

IN AND OUT OF THE FREEZER / REFRIGERATOR

- Most standards need to be brought to room temperature (around 20° C) before diluting or using
- You may need to shake or sonicate the standard before using
- Humidity is not your friend
 - When you open your flask/vial/bottle you are exposing the air in the container to humidity
 - This humidity will condense into water and dilute your standard when it's refrigerated again
 - Eventually, you will see lower recoveries

POSSIBLE SOLUTIONS TO HUMIDITY

- Avoid humidity
- Add a blanket of nitrogen to the sample container before refrigerating again
- Sub-vial your standard so that you are not exposing all of your standard to humidity
 - Many small containers is always better than one big one





THE CERTIFICATE OF ANALYSIS AND PURITY

 **EHRENSTORFER™**

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THE MATH

Calculate the mass of neat material needed

Adjust for Purity (98%)

$$0.1021 \text{ g.} \times 0.98 = 0.1000$$

Stock

$$\frac{0.10 \text{ g}}{10 \text{ mL}} = \frac{100 \text{ mg}}{10 \text{ mL}} = \frac{10 \text{ mg}}{\text{mL}} = 1\% \text{ solution}$$

THE MATH

Calculate volume of stock needed to make a Working

Working

$$\frac{10 \text{ mg}}{\text{mL}} \times (x) \text{ mL}$$

$$= 10 \mu\text{g/mL} = 10 \text{ ppm}$$

100 mL

THE MATH

Make all units the same

micrograms to milligrams

$$\frac{10 \text{ mg}}{\text{mL}} \times (\text{x}) \text{ mL}$$

$$100 \text{ mL} = 0.010 \text{ mg/mL}$$

THE MATH

Algebra

Multiply (mass needed) times the (final volume)

$$\frac{0.010 \text{ mg}}{\cancel{\text{mL}}} \times 100 \cancel{\text{ mL}} = 1 \text{ mg}$$

Divide product by the stock concentration

$$\frac{1 \cancel{\text{ mg}}}{10 \cancel{\text{ mg}}/\text{mL}} = 0.10 \text{ mL}$$

THE MATH

Check our math

$$\frac{\frac{10 \text{ mg}}{\cancel{\text{mL}}} \times 0.100 \cancel{\text{ mL}}}{100 \text{ mL}} = \frac{1 \text{ mg}}{100 \text{ mL}}$$
$$= 0.010 \text{ mg/mL} = 10 \text{ } \mu\text{g/mL}$$



OKAY, LET'S
WEIGH IT OUT!

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TECHNICAL TIPS: INORGANICS

- Ba and S are not compatible in the same mix
- P and Zr are not compatible in the same mix
- Zr and As not compatible over 100 ppm
- Pb needs to be pre-complexed in straight HCL before added to HCl mix
- TL not stable in HCl
- Sb requires high HCl % to remain stable
- Ba sensitive to high levels of HNO₃
- Follow your lab's SOP
- Read and follow the method
- Always dilute into the same matrix
- Make fresh working standards daily
- Don't put unused standards back into the bottle (contamination)
- Don't reuse old bottles
- Determine your shelf life

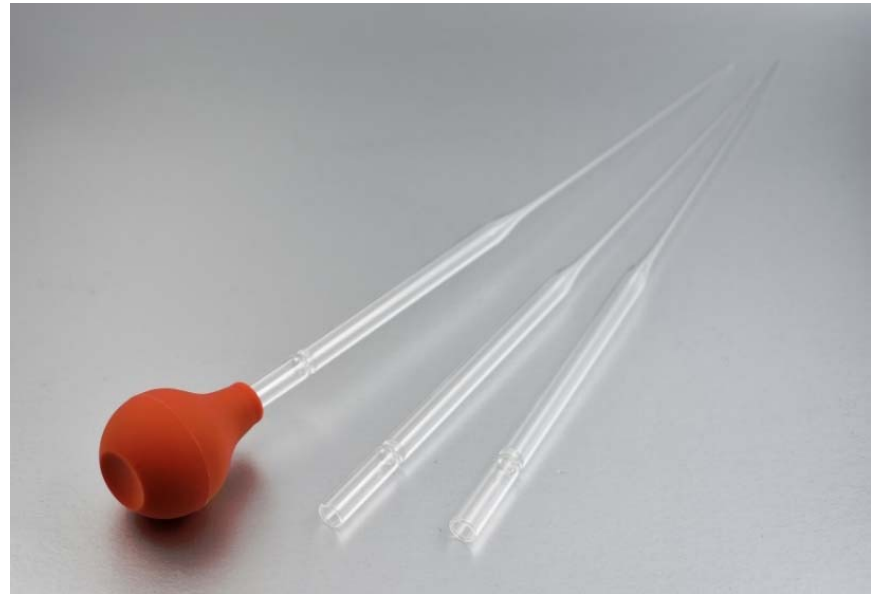
TECHNICAL TIPS: ORGANICS

- Choose a work space that will minimize chances of contamination
- Have everything you will need ready
- Ensure your analytical balance is calibrated and stable
- Only use clean glassware
- Use only Class A glassware
- Allow reference material containers containing sticky substances to sit upright at least 24 hours before use when possible
- Let reference materials reach room temperature before use
- Do not use tools that show excessive wear
- Only use high purity solvents without preservatives when possible
- Record everything
- Follow your SOPs
- Determine product shelf life
- Do not use materials that will contaminate your standard

TECHNICAL TIPS: PIPETTORs



TECHNICAL TIPS: SOURCES OF CONTAMINATION



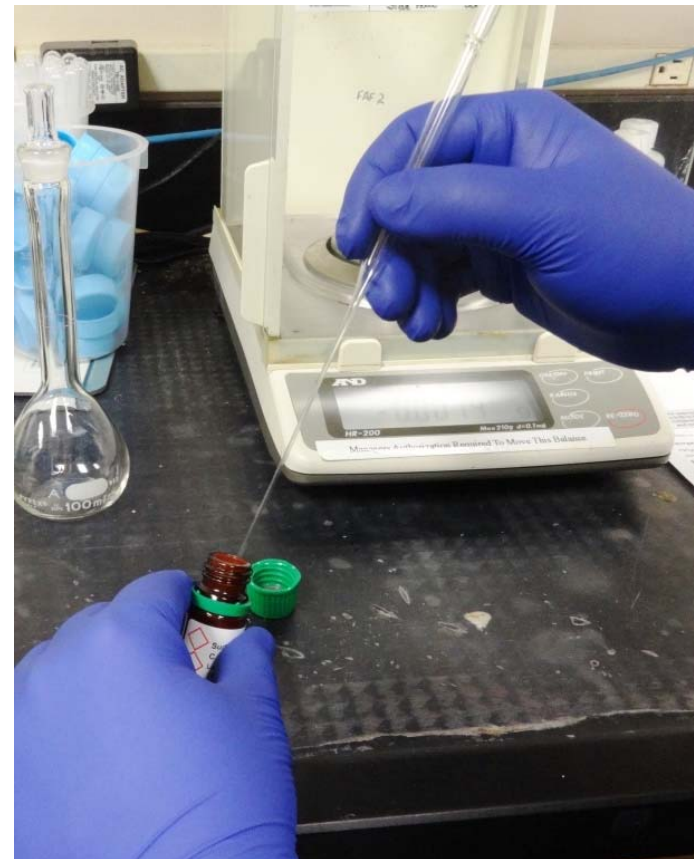
ANALYTICAL BALANCES: ISSUES

- Electrostatic charge
- Not on a stable surface
- Vibrations
- Leaning on the bench
- Wind
- Escaping volatiles
- Hygroscopic materials



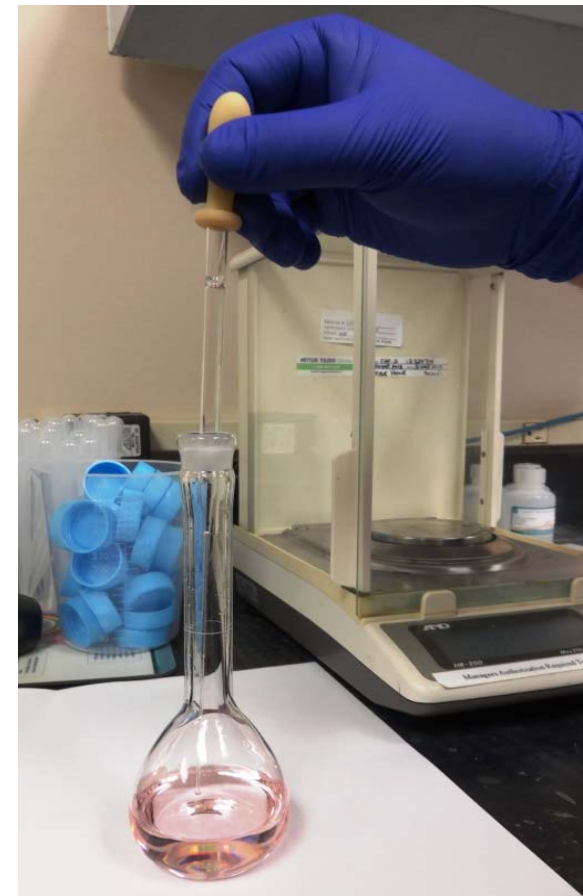
WEIGHING OUT STICKY ORGANICS (LIKE PCBS)

- Write down the tare weight of a glass rod or Pasteur pipette
- Gently rub the pipette along the inner walls of the reference material vial
- Take the mass of the pipette and write it down



WEIGHING OUT STICKY ORGANICS (LIKE PCBS)

- Place the end of the pipette into a Class A volumetric flask containing the proper solvent and rinse the material off the pipette and into the solvent
- Allow the solvent to evaporate from the pipette and then take its mass; write it down
 - It should be close to your tare weight
- Determine the difference between the pipette's mass with material and the mass after drying the pipette
 - This difference is the mass added to your flask of solvent



WEIGHING OUT STICKY ORGANICS (LIKE PCBS)

- Add your solvent to the Class A volumetric flask until the meniscus sits on the line
- Stopper and invert 3 or 4 times to form a homogeneous solution
- Store per your SOP



POWDERS ARE MUCH EASIER

- Just weigh out what you need
- Write it down
- Completely transfer contents to your flask
- Add the proper solvent
- Invert 3 or 4 times



An aerial photograph of a vast lavender field, showing rows of dark purple plants. A white road with power lines runs diagonally across the field. The text "THE VIAL IS EMPTY!" is overlaid on the left side of the image.

**THE VIAL IS
EMPTY!**

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SOUND LIKE A HASSLE?

When in doubt, purchase your
standards from an ISO 17034
accredited professional





CONTACT INFORMATION

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