

# ANSWERING THE URGENT CALL FOR CHLORINATED PARAFFIN STANDARDS



## MIXTURES

ISO 17034  
ISO/IEC 17025  
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### What are chlorinated paraffins?

Chlorinated paraffins (CP) also known as polychlorinated n-alkanes (PCA), are produced as complex mixtures of thousands of isomers of different carbon chain length and chlorination degree.

### CPs are subdivided according to their carbon chain length:

Very short chain CPs (vSCCPs, C6–9)

Short chain CPs (SCCPs, C10–13)

Medium chain CPs (MCCPs, C14–17)

Long chain CPs (LCCPs, C>17)

Very long chain CPs (vLCCPs, >C21)

The degree of CP chlorination can vary between 30 and 70 wt%

### Where are they used?

CPs are used as high-temperature lubricants in metal-working machinery and as flame retardant plasticizers in vinyl plastics. Less common applications include the use as flame retardants in textiles, rubber, paints, adhesives and as sealants.



## What are the concerns?

The total global production remains largely unknown, but is believed to exceed at least two million metric tonnes per year. CPs show resistance to degradation, and some show bioaccumulation and toxic potential. They are suspected to be carcinogenic to humans according to the International Agency for Research on Cancer (IARC).

Short-chain CPs have been prohibited by the Stockholm Convention on Persistent Organic Pollutants (POPs) in the EU since 2017 (Regulation (EC)850/2004) and placed on several monitoring lists such as the EU Water Framework Directive. However, due to their persistence and long-range transport, CPs will be in the environment for decades.

## Analytical challenges

One of the (many) challenges researchers face when determining CPs is the lack of suitable and generally accepted reference materials (RM). Current commercially available individual congener standards (native and labelled) have a chlorine pattern that is different than those found in industrial mixtures and the environment. CP mixture standards are not well-characterised nor purity assessed. The available labelled congener standards aimed for use as internal standard do not ionize on most commonly used detection methods (i.e. ESI and APCI). For longer chained CPs (C>17) standards in general are scarce. This all results in semi-quantitative analysis.

## The CHLOFFIN project

In October 2019 the Eurostars CHLOFFIN project was launched to address the lack of suitable standards for CPs. The three year collaboration between Chiron, Vrije Universiteit and European Commission, Joint Research Centre aimed to deliver:

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40 Native individual congener standards of CPs

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8 <sup>13</sup>C labelled individual congener standards of CPs

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10 Single chain mixtures

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1 Matrix Certified Reference Material (CRM)

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The lack of suitable standards for Chlorinated Paraffins has presented significant challenges for their analysis and regulation.”



## Synthesis & Certification

Single chain CP mixtures have been made by chlorination catalysed by UV light. NMR techniques have been used to determine the chlorine content of these single chain mixtures and technical mixtures. The results have been compared with an accredited titration method and by elemental analysis and were found to be consistent. Furthermore, the single chain mixtures were analysed by GC-MS and GC-FID, high resolution MS and by GC-GC. Water, solvent, and ash content were determined by Thermogravimetric analysis (TGA). CRMs were produced by a combination of purity determination by GC-FID, identity by NMR and excess water, solvent and ash by TGA in addition to stability and homogeneity assessment.

Single congener mixtures have been prepared for quantification together with defined mixtures and single chain mixtures.

**CLF-5248**, a synthetic mixture of well-defined, single SCCP Cl6 congeners, (common calibrant, CC), and **CLF-5371**, a complex mixture of SCCP single chain mixtures (Calibration QC, CQC) were applied in the certification of the first ever matrix RM for the mass fraction of SCCPs. The SCCPs certification was performed on ERM®-CE100, an already commercially available fish tissue CRM. The certification programme was coordinated by the European Commission Joint Research Centre

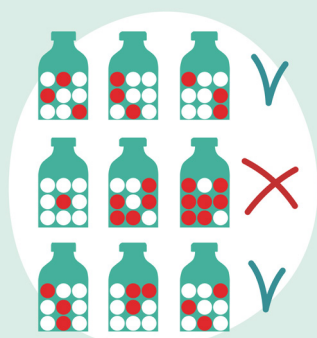


### ERM®-CE100

(JRC) and was performed in accordance with ISO 17034:2016 and ISO Guide 35:2017.

The material was characterised by an intercomparison of 9 laboratories of demonstrated competence, adhering to ISO/IEC 17025:2017, and applying different analytical procedures. The assigned values include results obtained by GC and LC-based analytical methodologies coupled with different MS detectors. The certified value and uncertainty for SCCPs are traceable to the International System of Units (SI). This first matrix RM for these analytically challenging pollutants bring the comparability of SCCPs analysis a step forward, securing better accuracy and traceability of measurement results.

### RM Certification Steps:



homogeneity



storage stability



transport stability



characterisation

### Reference:

Nuclear magnetic resonance as a tool to determine chlorine percentage of chlorinated paraffin mixtures.

Valderhaug, S., Liu, H., Gorovoy, A., Johansen, J.E., van Mourik, L., de Boer, J., Gautuna, O.R. – Chemosphere Volume 308, Part 3, December 2022, 136312.





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## Single Chain Mixtures

Chiron No.	Name	Mol. Formula	CAS
<b>CLF14574.9</b>	Chloroparaffin single chain mixture C9 Cl4-Cl7; 48.5% Cl (NMR)	C9ClxH20-x (x=4 to 7)	N/A
<b>CLF14575.10</b>	Chloroparaffin single chain mixture C10 Cl2-Cl6; 52.5% Cl (NMR)	C10ClxH22-x (x=2 to 6)	N/A
<b>CLF14803.10</b>	Chloroparaffin single chain mixture C10 Cl4-Cl8; 58.4% Cl (NMR)	C10ClxH22-x (x=4 to 8)	N/A
<b>CLF14576.11</b>	Chloroparaffin single chain mixture C11 Cl4-Cl7 52.3% Cl (NMR)	C11ClxH24-x (x=4 to 7)	N/A
<b>CLF14808.11</b>	Chloroparaffin single chain mixture C11 Cl4-Cl8 57.6% Cl (NMR)	C11ClxH24-x (x=4 to 8)	N/A
<b>CLF15318.12</b>	Chloroparaffin single chain mixture C12 Cl2-Cl7; 53.8% Cl (NMR)	C12ClxH26-x (x=2 to 7)	N/A
<b>CLF14809.12</b>	Chloroparaffin single chain mixture C12 Cl4-Cl8; 57.3% Cl (NMR)	C12ClxH26-x (x=4 to 8)	N/A
<b>CLF14577.13</b>	Chloroparaffin single chain mixture C13 Cl2-Cl7; 45.9% Cl (NMR)	C13ClxH28-x (x=2 to 7)	N/A
<b>CLF14687.13</b>	Chloroparaffin single chain mixture C13 Cl5-Cl8(Cl9); 60.0% Cl (NMR)	C13ClxH28-x (x=5 to 8)	N/A
<b>CLF14870.14</b>	Chloroparaffin single chain mixture C14 Cl2-Cl6, 49.2% Cl (NMR)	C14ClxH30-x (x=2-6)	N/A
<b>CLF14811.14</b>	Chloroparaffin single chain mixture C14 Cl4-Cl9; 58.7% Cl (NMR)	C14ClxH30-x (x=4-9)	N/A
<b>CLF14998.15</b>	Chloroparaffin single chain mixture C15 Clx-ClY; 47.7% Cl (NMR)	C15ClxH32-x	N/A
<b>CLF14999.15</b>	Chloroparaffin single chain mixture C15 Clx-ClY; 59.3% Cl (NMR)	C15ClxH32-x	N/A
<b>CLF15000.16</b>	Chloroparaffin single chain mixture C16 Clx-ClY; 51.5% Cl (NMR)	C16ClxH32-x	N/A
<b>CLF15001.16</b>	Chloroparaffin single chain mixture C16 Clx-ClY; 58.4% Cl (NMR)	C16ClxH32-x	N/A
<b>CLF15002.17</b>	Chloroparaffin single chain mixture C17 Clx-ClY; 56.3% Cl (NMR)	C17ClxH34-x	N/A
<b>CLF15003.17</b>	Chloroparaffin single chain mixture C17 Clx-ClY; 60.3% Cl (NMR)	C17ClxH34-x	N/A
<b>CLF15004.18</b>	Chloroparaffin single chain mixture C18 Clx-ClY; 56.9% Cl (NMR)	C18ClxH36-x	N/A
<b>CLF15005.18</b>	Chloroparaffin single chain mixture C18 Clx-ClY; 58.4% Cl (NMR)	C18ClxH36-x	N/A
<b>CLF15133.19</b>	Chloroparaffin single chain mixture C19 Clx-ClY; 45.0% Cl (NMR)	C19ClxH38-x	N/A
<b>CLF15134.19</b>	Chloroparaffin single chain mixture C19 high chlorine	C19ClxH38-x	N/A
<b>CLF15135.20</b>	Chloroparaffin single chain mixture C20 Clx-ClY; 38.0% Cl (NMR)	C20ClxH40-x	N/A
<b>CLF15136.20</b>	Chloroparaffin single chain mixture C20 Clx-ClY; 59.0% Cl (NMR)	C20ClxH40-x	N/A
<b>CLF9826.X</b>	Chlorinated paraffins (Cl: 38 % from NMR), technical mix	CP mix C12; C14; C16 rich	61788-76-9

## Mixtures of single chain SCCPs mixtures

Generation	Chiron No.	Name	Mix No.	Composition [CAS]	Concentration/Wt. %	Concentration / Solvent
Single chain	CLF-5246-100-AN	Common Calibrant SCCP mix of single chain mixes, C10-C13	CLF Mix 1	CLF14575.10 Chloroparaffin single chain mixture C10 C12-C16; 52.5% Cl (NMR) [N/A] 7 µg/mL  CLF14576.11 Chloroparaffin single chain mixture C11 C14-C17 52.3% Cl (NMR) [N/A] 16 µg/mL  CLF15318.12 Chloroparaffin single chain mixture C12 C12-C17; 53.8% Cl (NMR) [N/A] 12 µg/mL  CLF14577.13 Chloroparaffin single chain mixture C13 C12-C17; 45.9% Cl (NMR) [N/A] 65 µg/mL	100 µg/mL (total) in Acetonitrile	
Single chain	CLF-5252-100-AN	Common Calibrant SCCP mix of single chain mixes, C10-C13	CLF Mix 4	CLF14575.10 Chloroparaffin single chain mixture C10 C12-C16; 52.5% Cl (NMR) [N/A] 7 µg/mL  CLF14576.11 Chloroparaffin single chain mixture C11 C14-C17 52.3% Cl (NMR) [N/A] 16 µg/mL  CLF15318.12 Chloroparaffin single chain mixture C12 C12-C17; 53.8% Cl (NMR) [N/A] 12 µg/mL  CLF14687.13 Chloroparaffin single chain mixture C13 C15-C18(C19); 60.0% Cl (NMR) 65 µg/mL  CAS for mixture: [85535-84-8]	100 µg/mL (total) in Acetonitrile	
Single chain	CLF-5371-10-AN 	Calibration QC of SCCP single chain mixtures	CLF Mix 5	CLF14575.10 Chloroparaffin single chain mixture C10 C12-C16; 52.5% Cl (NMR) [N/A] 0.7 µg/mL  CLF14576.11 Chloroparaffin single chain mixture C11 C14-C17 52.3% Cl (NMR) [N/A] 1.6 µg/mL  CLF15318.12 Chloroparaffin single chain mixture C12 C12-C17; 53.8% Cl (NMR) [N/A] 1.2 µg/mL  CLF14577.13 Chloroparaffin single chain mixture C13 C12-C17; 45.9% Cl (NMR) [N/A] 2.0 µg/mL  CLF14687.13 Chloroparaffin single chain mixture C13 C15-C18(C19); 60.0% Cl (NMR) [N/A] 4.5 µg/mL  CAS for mixture: [85535-84-8]	10 µg/mL (total) in Acetonitrile	
Single chain	CLF-5371-10-IO 	Calibration QC of SCCP single chain mixtures	CLF Mix 5	CLF14575.10 Chloroparaffin single chain mixture C10 C12-C16; 52.5% Cl (NMR) [N/A] 0.7 µg/mL  CLF14576.11 Chloroparaffin single chain mixture C11 C14-C17 52.3% Cl (NMR) [N/A] 1.6 µg/mL  CLF15318.12 Chloroparaffin single chain mixture C12 C12-C17; 53.8% Cl (NMR) [N/A] 1.2 µg/mL  CLF14577.13 Chloroparaffin single chain mixture C13 C12-C17; 45.9% Cl (NMR) [N/A] 2.0 µg/mL  CLF14687.13 Chloroparaffin single chain mixture C13 C15-C18(C19); 60.0% Cl (NMR) [N/A] 4.5 µg/mL  CAS for mixture: [85535-84-8]	10 µg/mL (total) in Isooctane	

## Single congener mixtures

Generation	Chiron No.	Name	Mix No.	Composition [CAS]	Concentration/Wt. %	Concentration / Solvent
2 <sup>nd</sup>	CLF-5247-100-AN	Common Calibrant SCCP mix of single congeners, C10-C13	CLF Mix 2	CLF12284.10 1,2,5,6,9,10-Hexachlorodecane [189350-94-5] 4 µg/mL		100 µg/mL (total) in Acetonitrile
				CLF14069.11 1,2,4,5,8,9-Hexachloroundecane [N/A] 13 µg/mL		
				CLF14072.12 1,2,5,6,9,10-Hexachlorododecane [N/A] 13 µg/mL		
				CLF14131.13 1,2,6,7,10,11-Hexachlorotridecane [N/A] 70 µg/mL		
2 <sup>nd</sup> & 3 <sup>rd</sup>	CLF-5248-100-AN 	Common Calibrant mix of SCCP single congeners	CLF Mix 3	CLF12284.10 1,2,5,6,9,10-Hexachlorodecane [189350-94-5] 4 µg/mL		100 µg/mL (total) in Acetonitrile
				CLF14069.11 1,2,4,5,8,9-Hexachloroundecane [N/A] 13 µg/mL		
				CLF14072.12 1,2,5,6,9,10-Hexachlorododecane [N/A] 13 µg/mL		
				CLF14131.13 1,2,6,7,10,11-Hexachlorotridecane [N/A] 35 µg/mL		
2 <sup>nd</sup> & 3 <sup>rd</sup>	CLF-5248-100-IO 	Common Calibrant mix of SCCP single congeners	CLF Mix 3	CLF12284.10 1,2,5,6,9,10-Hexachlorodecane [189350-94-5] 4 µg/mL		100 µg/mL (total) in Isooctane
				CLF14069.11 1,2,4,5,8,9-Hexachloroundecane [N/A] 13 µg/mL		
				CLF14072.12 1,2,5,6,9,10-Hexachlorododecane [N/A] 13 µg/mL		
				CLF14131.13 1,2,6,7,10,11-Hexachlorotridecane [N/A] 35 µg/mL		
1 <sup>st</sup> & 2 <sup>nd</sup>	CLF-4784-100-IO	C10 SCCP Mixture	CLF Mix 6	CLF1666.10 1,2-Dichlorodecane [34619-32-4] 33.58 %Cl		100 µg/mL in Isooctane
				CLF1662.10 1,1,3-Tetrachlorodecane [51755-60-3] 50.64 %Cl		
				CLF1671.10 1,2,9,10-Tetrachlorodecane [205646-11-39] 50.64 %Cl		
				CLF1659.10 1,1,3,9,10-Hexachlorodecane [601523-26-69] 61.97 %Cl		
				CLF1622.10 1,1,1,3,8,10,10,10-Octachloroundecane [601523-23-3] 67.88 %Cl		

# Single congener mixtures

Generation	Chiron No.	Name	Mix No.	Composition [CAS]	Concentration/Wt. %	Concentration / Solvent
1 <sup>st</sup> & 2 <sup>nd</sup>	CLF-4785-100-IO	C11 SCCP Mixture	CLF Mix 7	CLF1667.11 1,2-Dichloroundecane [81246-86-8] 31.49 %Cl		100 µg/mL in Isooctane
				CLF1649.11 1,1,3-Tetrachloroundecane [56686-55-6] 48.22 %Cl		
				CLF1674.11 1,2,10,11-Tetrachloroundecane [210049-49-3] 48.22 %Cl		
1 <sup>st</sup> & 2 <sup>nd</sup>	CLF-4786-100-IO	C12 SCCP Mixture	CLF Mix 8	CLF1650.11 1,1,3,10,11-Hexachloroundecane [601523-28-8] 58.60 %Cl		100 µg/mL in Isooctane
				CLF1623.11 1,1,3,9,11,11-Octachloroundecane [601523-25-5] 65.67 %Cl		
1 <sup>st</sup> & 2 <sup>nd</sup>	CLF-4786-100-IO	C12 SCCP Mixture	CLF Mix 8	CLF1668.12 1,2-Dichlorododecane [75121-23-2] 29.64 %Cl		100 µg/mL in Isooctane
				CLF1663.12 1,2-Dichlorododecane [3922-28-9] 29.64 %Cl		
				CLF1651.12 1,1,3-Tetrachlorododecane [14983-60-9] 46.03 %Cl		
1 <sup>st</sup> & 2 <sup>nd</sup>	CLF-4787-100-IO	C13 SCCP Mixture	CLF Mix 9	CLF1675.12 1,2,11,12-Tetrachlorododecane [210115-98-3] 46.03 %Cl		100 µg/mL in Isooctane
				CLF1652.12 1,1,3,11,12-Hexachlorododecane [865306-22-5] 56.42 %Cl		
				CLF1624.12 1,1,3,10,12,12-Octachlorododecane [601523-21-1] 63.60 %Cl		
1 <sup>st</sup> & 2 <sup>nd</sup>	CLF-4787-100-IO	C13 SCCP Mixture	CLF Mix 9	CLF1669.13 1,2-Dichlorotridecane [701920-72-1] 28.00 %Cl		100 µg/mL in Isooctane
				CLF1653.13 1,1,3-Tetrachlorotridecane [67095-50-5] 44.02 %Cl		
				CLF1654.13 1,1,3,12,13-Hexachlorotridecane [865306-23-6] 54.40 %Cl		
1 <sup>st</sup> & 2 <sup>nd</sup>	CLF-4787-100-IO	C13 SCCP Mixture	CLF Mix 9	CLF1625.13 1,1,3,11,13,13-Octachlorotridecane [865306-24-7] 61.67 %Cl		100 µg/mL in Isooctane



# Single congener mixtures

Generation	Chiron No.	Name	Mix No.	Composition [CAS]	Concentration/Wt. %	Concentration / Solvent
1 <sup>st</sup> , 2 <sup>nd</sup> & 3 <sup>rd</sup>	CLF-5133-ASS(50)-10	C10-13 SCCPs Mix, 56.0 % CI ("Hordalub 80")	CLF Mix 16	CLF1666.10 1,2-Dichlorodecane [34619-32-4] 33.58 %CI 0.44 Wt. %		
				CLF13255.10 1,2,4,5-Tetrachlorodecane, isomermix [N/A] 50.64 %CI 1.80 Wt. %		
				CLF12590.10 2,3,4,5-Tetrachlorodecane, isomermix [N/A] 50.64 %CI 1.8 Wt. %		
				CLF12284.10 1,2,5,6,9,10-Hexachlorodecane [189350-94-5] 61.97 %CI 5.96 Wt. %		
				CLF1674.11 1,2,10,11-Tetrachloroundecane [210049-49-3] 48.22 %CI 4.125 Wt. %		
				CLF12728.11 4,5,7,8-Tetrachloroundecane [N/A] 48.22 %CI 4.125 Wt. %		
				CLF12285.11 1,2,3,4,5,6-Hexachloroundecane, isomermix [N/A] 58.60 %CI 24.75 Wt. %		50 µg/mL (total) in Isooctane
				CLF13398.12 1,2,4,5-Tetrachlorododecane [N/A] 46.03 %CI 2.40 Wt. %		
				CLF12425.12 2,3,4,5-Tetrachlorododecane [N/A] 46.03 %CI 2.40 Wt. %		
				CLF1652.12 1,1,1,3,11,12-Hexachlorododecane [865306-22-5] 56.42 %CI 30.80 Wt. %		
				CLF1624.12 1,1,1,3,10,12,12,12-Octachlorododecane [601523-21-1] 63.61 %CI 4.40 Wt. %		
				CLF1654.13 1,1,1,3,12,13-Hexachlorotridecane [865306-23-6] 28.00 %CI 13.09 Wt. %		
				CLF1625.13 1,1,1,3,11,13,13,13-Octachlorotridecane [865306-24-7] 44.02 %CI 3.91 Wt. %		
1 <sup>st</sup> & 2 <sup>nd</sup>	CLF-5138-ASS(50)-10	C14-C17 MCCP Mix, 52 % CI	CLF Mix 21	CLF1677.14 1,2,13,14-Tetrachlorotetradecane [221155-23-3] 42.18 %CI 28.35 Wt. %		
				CLF1678.14 1,1,1,3,12,14,14,14-Octachlorotetradecane [865306-26-9] 59.84 %CI 22.25 Wt. %		
				CLF8506.15 1,1,1,3,14,15-Hexachloropentadecane [N/A] 50.76 %CI 16.50 Wt. %		50 µg/mL (total) in Isooctane
				CLF8507.16 1,1,1,3,14,16,16,16-Octachlorohexadecane [N/A] 56.50 %CI 16.50 Wt. %		
				CLF8508.17 1,1,1,3,15,17,17,17-Octachloroheptadecane [N/A] 54.96 %CI 16.50 Wt. %		



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