



Brussels, 24 July 2024

## Technical clarification on Dechlorane Plus

The Associations above, representing the recycling and waste management industry at European level, would like to share: a procedural comment, experience and data on measuring Dechlorane Plus in polymeric matrixes, and a suggested way forward for the Unintentional Trace Contaminant (UTC) limit for Dechlorane Plus (DP).

### Procedural comment

We commend the European Commission for utilizing the expertise of the European Chemicals Agency to prepare REACH restriction proposals and the subsequent evaluation thereof by the Risk Assessment Committee (RAC) and Socio-Economic Assessment Committee (SEAC) whenever new POP substances are proposed. This process ensures that different policy options are explored and the benefits and drawbacks of for example derogations are quantified holistically. It also gives industry and other stakeholders the opportunity to provide comments and to supply data.

On DP, Plastics Recyclers Europe (PRE), submitted comment #3398 into the REACH Restriction process explaining the recycling technology for WEEE/ELV and that a limit of 0.1% (1,000 ppm) as proposed in the dossier prepared by ECHA would prevent intentional use while not harming recycling<sup>1</sup>. This comment was appreciated and considered to be *very useful for assessing a derogation for the recycling sector* by the SEAC Rapporteur.

We would like to point out that if in this process the limit being considered would have been 1 ppm, the PRE comment would have been substantially different. This would have then been discussed in the RAC/SEAC and the RAC/SEAC opinion would have evaluated the benefit and drawbacks of a lower limit value. In the absence of such a holistic evaluation there are calls from environmental groups and some Member States for a limit that would be as low as is measurable with analytical techniques available today. We call for a more pragmatic approach with limits that are readily measurable and above the level that can be reached in secondary raw materials by recyclers. As such:

***We call upon the European Commission to include in mandates to the ECHA for the preparation of restriction proposals for POP nominees a request to evaluate different options for limit values below 0.1%.***

Obviously, such a measure would help the discussion for future substances and not for DP itself. It should however be noted that in the RAC and SEAC consultation, no calls from Member States nor from environmental NGOs were received for a lower limit value than 0.1%.

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<sup>1</sup> This comment and the response from the RAC/SEAC rapporteurs can be found in the [RCOM document](#) on the [ECHA Website](#).

## Measuring Dechlorane Plus

The measurement of any substance in polymeric material present certain generic difficulties. An explanation of these difficulties can be found in Annex I. There must be an independent, scientific, and verifiable test method available that can be used on a continuous industrial basis – both for recyclers and also authorities challenging evidence.

In the technical clarification on PBDEs provided to the POP Expert Group and published on [CIRCABC](#), it was explained in detail that plastics recyclers must be able to say with confidence that the material that they produce is compliant to UTC limit values. With a limit of 0.1%, this would be feasible since XRF chlorine measurements can be taken with ease and at speed at a high frequency. While chlorine is not the same thing as DP, if the chlorine signal is sufficiently low it is possible to say with confidence that DP cannot be present above the limit of 0.1%.

This method cannot be used anymore with a limit of <0.1%, and periodic and expensive external laboratory testing would have to be performed. We know that there is not a wide availability of such specialist laboratories that are even able to measure DP in polymer matrices.

At the moment, there is no ISO or EN standard specifically for the quantification of DP in polymeric materials, which means that laboratories aiming to implement the measurement of DP have to be creative and either modify existing techniques in-house or develop them themselves and do internal validation work to the extent that they are able. We asked multiple laboratories whether they would be willing to perform a quantification and what their lowest limit of quantification was.

Two contacted laboratories indicated that they have already performed DP analysis in polymeric matrix by modifying an existing technique in-house. They adapted the procedures described in ISO 22818<sup>2</sup>, a standard originally developed for the identification and quantification of short- and medium-chain chlorinated paraffins in textile products, or in IEC 62321-6<sup>3</sup>, a standard developed for the determination of polybrominated biphenyls and polybrominated diphenyl ethers in polymers in electrotechnical products. In both methods, extraction is done in toluene, followed by separation of extract components by gas chromatography, and detection with mass spectroscopy. The claimed determination limit was ranging between 1 mg/kg (1 ppm) for a targeted analysis to 50 mg/kg (50 ppm) for a screening analysis<sup>4</sup>, which is more commonly used for lab detection of contaminants in WEEE. The cost per sample was quoted between 150 for screening to 233 – 250 for targeted analysis EUR.

One laboratory mentioned that in some samples analysed in the past for private companies, if any DP was found, it was usually in cable sheeting, a stream normally associated with demolition waste rather than WEEE/ELV, at concentrations varying from 0.1% to 0.2% (i.e. 1000 ppm to 2000 ppm).

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<sup>2</sup> Full title: *Textiles — Determination of short-chain chlorinated paraffins (SCCP) and middle-chain chlorinated paraffins (MCCP) in textile products out of different matrices by use of gas chromatography negative ion chemical ionization mass spectrometry (GC-NCI-MS)*

<sup>3</sup> Full title: *Determination of certain substances in electrotechnical products – Part 6: Polybrominated biphenyls and polybrominated diphenyl ethers in polymers by gas chromatography–mass spectrometry (GC-MS)*

<sup>4</sup> Other laboratories using other methods may come to much higher determination limits.

PRE submitted 18 samples for testing with a focus on WEEE/ELV derived recyclate since it is in these sectors that one would expect the use of flame retardants. However, materials from the PVC and HDPE recycling sectors were also included because they are used in sectors with very long service life (construction and demolition for PVC, wheelie bins/container park plastics for HDPE) or with quite high recycling rates (Drums/IBCs for HDPE). Packaging derived plastics recyclate was excluded since it has a very short service life, and it makes little sense to use a very expensive flame retardant in packaging as there is normally no fire safety standard demanding this. The results of the analysis by the external laboratory can be found in Table 1.

Table 1 Results of the PRE Measurement Campaign. \* There are considerable uncertainties in the reported values including the limit of quantification (see text below).

SampleID	Recycling Sector	Source Material	Polymer	Dechlorane Plus* (mg/kg)
24V0001	WEEE/ELV	WEEE - SDA	PS/ABS/PP	<1.0
24V0014		ELV - Bumpers	PP	<1.0
24V0009		ELV - ASR	PE/PP	<1.0
24V0015		WEEE/ELV - Mixed SLF/ASR	PE/PP	<1.0
24V0025		WEEE - LDA	PP	<1.0
24V0030		WEEE - TEE	PS	<1.0
24V0038		WEEE - CRT/FPD/Small IT	PS	<1.0
24V0043		WEEE/ELV- Mixed SLF/ASR and Bumpers	PP	<1.0
24V0049		WEEE - SLF	ABS	<1.0
24V0057		WEEE - SLF	PS	3.1
24V0058		General Household	PE/PP	<1.0
24_C0001		PVC	Window Profiles	PVC
24_C0005	PVC Film		PVC	<1.0
24_C0011	Construction Profiles		PVC	<1.0
24_C0008	Window Profiles		PVC	<1.0
24_E0005	HDPE	Drums/IBCs	HDPE	<1.0
24_E0015		Bulky Plastics from Container Park	HDPE	<1.0
24_E0016		Wheelie bins	HDPE	<1.0

The results show that DP was detected in recyclate derived from mixed WEEE shredder light fraction PS recyclate at 3.1 ppm. However, it should be noted that the standard used (ISO 22818) was originally designed for SCCPs and MCCPs in textiles, which brings substantial uncertainty and concerns for the recycling industry.

Firstly, the way of applying chlorinated paraffins to textile is very different than the use of DP in thermoplastics. Chlorinated paraffins are applied to the textile material by impregnation resulting in the majority of the chlorinated paraffins to be present at the surface of the textile fibres and only to a substantially lesser degree inside of the polymeric matrix. When DP is used in thermoplastics material it is incorporated into the solid polymer matrix in the melt phase and thus uniformly distributed throughout the solid material. Furthermore, the surface to volume ratio of textiles is huge compared to solid thermoplastics materials such as the plaques that were supplied to the laboratory. Since the ISO 22818 standard's extraction procedure is designed to extract chlorinated paraffins present at the surface of a textile material with a huge surface to volume ratio it is extremely likely that the additive DP that is uniformly distributed- and encapsulated- in a solid polymer matrix will be less than complete. This would result in severe underreporting of the actual concentration of DP.

Furthermore, it is uncertain if the clean-up procedure described in ISO 22818 does not also remove DP to some extent, which again would result in underreporting of the actual concentration.

The way to address and quantify these uncertainties would be to perform method validation work. In a first step a laboratory would purchase "Certified Reference Material" (CRM), several different plastics materials with known concentrations of Dechlorane Plus and perform the method as written down for chlorinated paraffins and note the "recovery", i.e. the detected concentration divided over the actual concentration in the material. If this recovery is inconsistent across the different reference materials or simply rather low the methods would need to be further adapted (e.g. by selecting more harsh extraction conditions or increasing the duration) and repeat the work. It normally is a long and laborious process.

We have contacted the laboratory to inquire how much validation work was done to determine the reliability of the limit of quantification and measured DP. They indicated that they simply presumed that the procedure of ISO 22818 would work perfectly with complete extraction, no destruction of the compound of interest and so on and so forth. We inquired if they would be willing to perform the required validation work to determine the recovery. They would be interested to do so, however, they indicated that no such CRM is available to them at present, and it is doubtful whether it exists at all anywhere in the world. They can produce such CRMs themselves and subsequently donate this to CRM libraries, however they would need to somehow get a hold of sufficient DP to manufacture the CRM themselves<sup>5</sup>. Since CRMs for DP in solid polymer matrixes are not available, it is likely that not a single accredited laboratory exists that has been able to perform measurements with known recovery rates.

All this means that should a full method development and validation study be done for DP in a variety of solid polymeric materials, the actual power to detect DP could increase substantially. For example, if the recovery of DP with the followed method would be 10, 1, or 0.1% then the measured concentration would be 30.7, 307, or 3070 ppm and the true limit of detection of the current method would be 10, 100, or 1000 ppm. As such we advocate that:

***There must be an independent, scientific, and verifiable test method available that can be used on a continuous industrial basis – both for recyclers and also authorities challenging evidence.***

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<sup>5</sup> This is not an easy thing to do since, while analytical standards of pure DP, these cost tens to hundreds of EUR for milligrams of material. To run a laboratory extruder to create CRM, several hundreds of grams to a few kilograms would be needed. To obtain such volumes of a POP substance is highly complex given the POP status of the substance.

## Proposed way forward

While the REACH Restriction development did not assess lower limits, it did assess what would happen if a limit of 0.1% were set and what would happen if various derogations would be given or not (see Figure 1). In their assessment, the only thing that could result in higher levels of emissions to the environment would be the granting of time limited derogations. When providing no derogation or when derogations expire the emissions decrease to 0 tons per year. Implying at least that a limit of 0.1% should result in cessation of all emissions of DP.

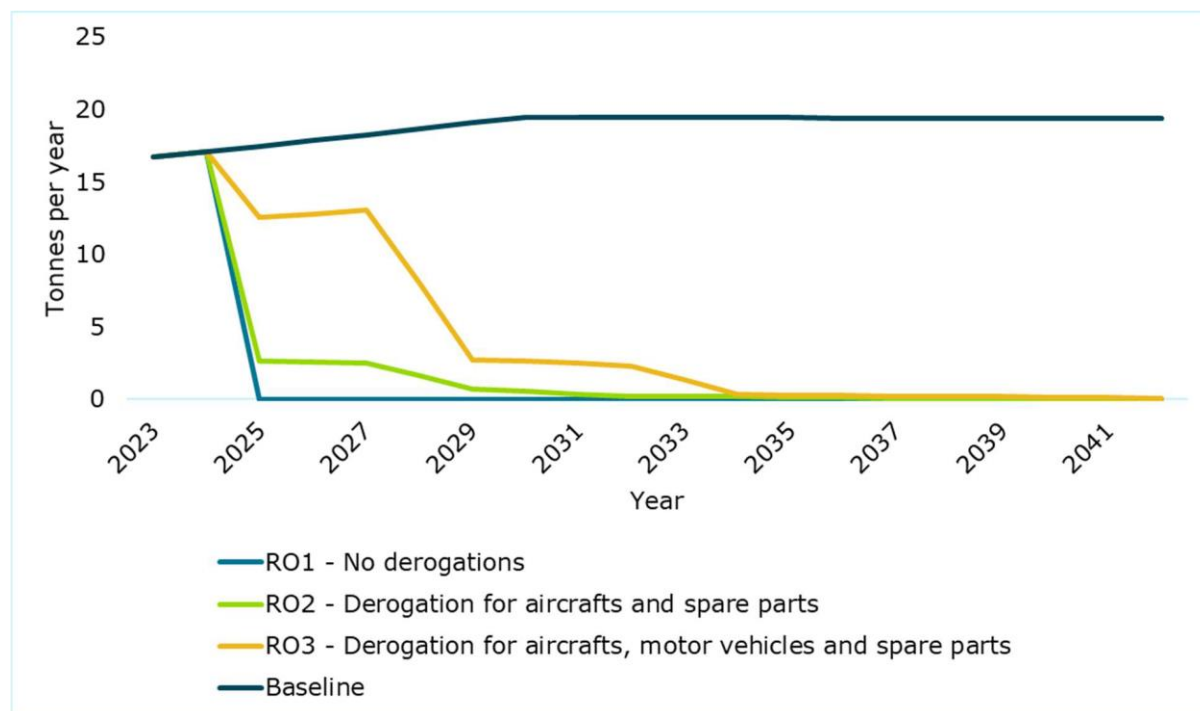


Figure 1 Continued emissions of DP under each restriction scenario and the baseline. Source: [ECHA RAC/SEAC Annex to the Background Document](#) (page 247)

This would be entirely logical since a limit of 0.1% would prevent all intentional use of DP. About two orders of magnitude more would be needed to functionally make plastics material flame retardant with DP. At such levels, the density sorting process utilised at WEEE/ELV plastics recyclers will cause accumulation of the DP in the heavy fraction of the recyclers, which is directed towards incineration.

Furthermore, it would allow recyclers to utilise XRF technology to say with a strong degree of confidence whether the batch of material supplied to the market complies with the POP Regulation limit.

As such:

***We strongly advocate that the UTC for Dechlorane Plus should be set at 0.1%.***

**For and on behalf of the following industry Associations:**



*FEAD is the European Waste Management Association, representing the private waste and resource management industry across Europe, including 19 national waste management federations and 3,000 waste management companies. Private waste management companies operate in 60% of municipal waste markets in Europe and in 75% of industrial and commercial waste. This means more than 320,000 local jobs, fuelling €5 billion of investments into the economy every year.*



EERA is the voice of WEEE recycling in Europe being the professional association for the e-waste recycling and reprocessing industry. Our mission is to achieve a level playing field for fair competition in the WEEE value chain, harmonisation of regulations, effective and efficient recycling and reprocessing with prevention of pollution, minimization of emissions and a high quality of secondary raw materials and components.



EuRIC is an umbrella organisation for European Recycling Industries. Through its Members, EuRIC represents companies involved in the collection, processing, recycling, transport and trade of a variety of recyclables (metals, paper, plastics, glass and beyond) across Europe. By servicing its Members, EuRIC contributes to promote recycling, which is first and foremost a business activity driven by an ecosystem of thousands of Small and Medium-size Enterprises (SMEs) and fewer but equally important larger companies. All of them are local and global actors



Plastics Recyclers Europe (PRE) is an organization representing the voice of the European plastics recyclers who reprocess plastic waste into high quality material destined for production of new articles. Recyclers are important facilitators of the circularity of plastics and the transition towards the circular economy. Plastics recycling in Europe is a rapidly growing sector representing over €8.7 billion in turnover, 11.3 million tonnes of installed recycling capacity, more than 730 recycling facilities, and over 30.000 employees.

## Annex I: Detailed Explanation of Analytical Difficulties

In a first step, the substance needs to be extracted from the polymer matrix. Since the polymer matrix severely limits the release of substances the extraction method used tends to need to be a very harsh one, typically a combination of a strong solvent, higher temperatures, and other means of adding energy to the system (e.g. ultrasonic agitation). In validation studies, one would normally perform additional analysis to determine if the extraction is exhaustive (i.e. did all of the substance of interest come out of the sample?) and whether or not the harsh extraction conditions did not destroy (a fraction of) the substance of interest. Both effects could result in underreporting of the actual concentration of the substance of interest.

Sometimes, such an extract is then injected directly into a chromatographic column in which the different substances in the extract are separated before being directed to a detector (typically mass spectroscopy). However, when performing such harsh extractions not just the substance of interest is extracted. Oligomers of the polymer, other additives, substances that the polymer absorbed during its service life, and other things are extracted as well. In the best (very unlikely) case, the chromatographic column is able to separate the substance of interest from all other extract components and a low limit of detection is possible. In an intermediate case (more likely), there is overlap between other extract components and the substance of interest which can be overcome to an extent with modern MS techniques. However, a higher limit of detection is normally the result. In a worst-case scenario (which is more likely than one would like), the complex extract which contains also higher molecular weight substances clogs the chromatographic column making the measurement unreliable **and** requiring cleaning of the chromatographic column (which is something laboratories try to avoid).

One way to deal with this issue of overlapping peaks and/or clogging columns is to try a less harsh extraction technique, however this then can have drawbacks in terms of exhaustiveness of the extraction. Another is to dilute the extract, which results in a higher limit of detection. Finally, there is also the option to perform "sample cleanup".

Sample cleanup is a procedure whereby the sample is treated in such a way to remove substances that are not the substance of interest. A classical example of this is two phase extraction. In two phase extraction two solvents that do not dissolve with one another are added to an extract and well mixed by shaking. Afterwards the system is allowed to settle and two layers form in the beaker (this looks similar to what would happen if one would add water and olive oil to a glass). If the substance of interest has a property (e.g. hydrophilicity) that makes it move to one of the layers/phases (e.g. water rather than oil) then this layer can be used for further analysis. All raw extract components that have affinity of the other layer will thus be removed. Of course, two phase extraction has been complemented by many other techniques, but one thing that they all share is that they are laborious processes. Typically adding 100 – 200 EUR/sample to the cost at commercial laboratories and resulting in a longer time until the results are received.

Furthermore, also these kinds of steps tend to require validation studies. To return to the two-phase extraction, normally the separation is not absolute in the sense that every single molecule of the substance of interest moves to one of the phases. Normally, there is a partition of the substance of interest predominantly to one of the phases, but some is lost to the other phase. This needs quantification to determine the recovery percentage required to back calculate to the concentration of the substance in the original sample.

The above are just some of the practical issues in measuring substances in polymeric matrixes. There are quite a few more. Just to name one: not all polymer matrixes are equal. Polyolefins tend to release their substance of interest (and other matrix components) more readily than styrenics polymer for example. An example of a good validation study can be found in the Joint Research Centre's work on [Polycyclic Aromatic Hydrocarbons](#), which shows that proper validation work for analytical standard method for specific substances present at very low levels can take years to develop.