

## **COLLECTIVE EXPERT APPRAISAL: SUMMARY AND CONCLUSIONS**

**regarding the expert appraisal for recommending occupational exposure limits  
for chemical agents  
concerning the assessment of measurement methods for nine substances listed  
in the appendix of the European Directive (EU) 2019/1831**

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**This document summarises the work of the Working Group on Metrology.**

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### **Presentation of the issue**

Prior to the transposition of European occupational exposure limits (OEL) into French law, ANSES is mandated by the Ministry of Labour to conduct an assessment of the measurement methods available for the substances listed in the European Directives.

### **Scientific and legal background**

European objectives, intended to protect workers from risks associated with exposure to chemical agents, are set via European directives, in particular in the form of occupational exposure limits (OELs).

Since the European Commission relies on recommendations issued by European scientific expert committees (SCOEL<sup>1</sup> or RAC<sup>2</sup>) for the establishment of European OELs, ANSES does not reassess the health effects of the substances in question when European directives establishing OELs are published.

However, given that neither SCOEL nor RAC undertakes in-depth assessments of the available measurement methods with regard to the European OELs, ANSES is asked to undertake these assessments so that the French Ministry of Labour can have all of the information necessary to establish the binding or indicative nature of the limit values in national law.

Directive (EU) 2019/1831 of the Commission of 24 October 2019 establishes a fifth list of indicative occupational exposure limit values for ten chemical agents pursuant to Council Directive 98/24/EC and amending Commission Directive 2000/39/EC.

Of these 10 chemical agents, trimethylamine was covered by an expert appraisal conducted by ANSES in 2015; the corresponding Opinion, common to 10 other substances, was published in January 2019 (ANSES, 2019). Therefore, trimethylamine was not included in this expert appraisal.

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<sup>1</sup> SCOEL: Scientific Committee on Occupational Exposure Limits

<sup>2</sup> RAC: Committee for Risk Assessment

As part of the memorandum of understanding on occupational exposure limits and biological limit values (OELs and BLVs) established between the Ministry of Labour and ANSES, the Directorate General for Labour (DGT) mandated ANSES to undertake the metrological expert appraisal only for the substances mentioned in the following table, with regard to the OELs established in Directive (EU) 2019/1831.

In light of the question asked, the relevance of the values laid down by European Directive (EU) 2019/1831 has not been examined.

**Table 1: List of substances assessed in this expert appraisal**

Substance	CAS number	OELs laid down by the directive (UE) 2019/1831 (mg.m <sup>-3</sup> )	
		8h-OEL	15min-STEEL <sup>(1)</sup>
Aniline	62-53-3	7.74	19.35
Chloromethane	74-87-3	42	-
2-Phenylpropane (cumene)	98-82-8	50	250
n-Butyl acetate	123-86-4	241	723
Isobutyl acetate	110-19-0		
sec-Butyl acetate	105-46-4		
4-Aminotoluene	106-49-0	4.46	8.92
Isoamyl alcohol	123-51-3	18	37
Phosphoryl trichloride	10025-87-3	0.064	0.13

<sup>(1)</sup> STEEL : Short term exposure limit

## Organisation of the expert appraisal

The expert appraisal on the assessment of measurement methods with regard to OELs established under European Directives falls within the sphere of competence of the Working Group on Metrology (Metrology WG).

This report has been prepared from metrology reports developed individually for each substance according to the methodology of the Metrology WG validated by the Expert Committees "Health reference values" and "Assessment of the risks related to air environments" (Anses, 2020a).

This expert appraisal was therefore conducted by a group of experts with complementary skills. It was carried out in accordance with the French Standard NF X 50-110 "Quality in Expertise Activities".

## Description of the method

Each assessment report, individually prepared by the Metrology WG for each substance, presents the various protocols for measuring the respective substance in workplace atmospheres grouped together based on the methods they use

These methods were then assessed and classified based on the performance requirements set out particularly in the French Standard NF EN 482: "Workplace atmospheres - General requirements for the performance of procedures for the measurement of chemical agents" and

the decision-making criteria listed in the methodology report (Anses, 2020a). The list of the main sources consulted is detailed in the methodology report (Anses, 2020a).

These methods were classified as follows:

- category 1A: validated methods (all of the performance criteria are met);
- category 1B: partially validated methods (the essential performance criteria are met);
- category 2: indicative methods (essential criteria for validation are not clear enough or else the method requires adjustments that need to be validated);
- category 3 : methods not recommended because they are unsuitable (essential validation criteria are not fulfilled)
- category 3\*: methods not recommended because they cannot be evaluated (essential validation criteria are not documented).

NB : For the measurement of aerosols and substances in mixed phases, an initial classification is established with regard to the performance criteria for sampling methods. A second classification is then established with regard to the performance criteria for analytical methods. The final classification of the method corresponds to the least favourable of these two classifications.

A detailed comparative study of the methods in categories 1A, 1B and 2 was conducted with respect to their various validation data and technical feasibility, in order to recommend the most suitable method(s) for measuring concentrations for comparison with OELs.

The expert appraisal was carried out by the Metrology WG between 29 October 2020 and 9 February 2021.

The details concerning the adoption of each measurement method assessment report are given in the following table.

**Table 2 : Adoption dates of the individual reports by the Metrology Working Group**

Substance		Adoption date by the WG
Name	CAS number	
Aniline	62-53-3	09/02/2021
Chloromethane	74-87-3	25/01/2021
2-Phenylpropane (cumene)	98-82-8	25/01/2021
n-Butyl acetate	123-86-4	09/02/2021
Isobutyl acetate	110-19-0	
sec-Butyl acetate	105-46-4	
4-Aminotoluene	106-49-0	08/12/2020
Isoamyl alcohol	123-51-3	05/01/2021
Phosphoryl trichloride	10025-87-3	08/12/2020

The overall report, as well as the summary and conclusions of the collective expert appraisal, were adopted by the Metrology WG for public consultation on 09/02/2021.

This collective expert appraisal work and the summary report were submitted to public consultation from 02/03/2021 to 02/04/2021. The people or organizations that contributed to the

public consultation are listed in appendix 8 of the report (only available in French). The comments received were reviewed by the Working Group on Metrology who finally adopted this version on 16/04/2021.

## Results of the collective expert appraisal

The results of the assessment of the measurement methods are summarised below for each substance. References for all the protocols and the headings of the methods identified and evaluated for each substance are listed in Table 3. In this table, the protocols with the most complete validation data are highlighted in bold, and the measurement methods recommended at the end are noted in blue.

### Assessment of the measurement methods for aniline

Eight methods for the measurement of aniline in workplace air were identified and assessed:

- Method 1: Active sampling on filter impregnated with sulphuric acid using a GSP1 device, acetonitrile/ammonia elution, analysis by gas chromatography - detection by mass spectrometry (GC/MS)
- Method 2: Active sampling on 2 filters impregnated with sulphuric acid using a closed face cassette (CFC) or an IOM device, methanol/water+sodium hydroxide elution, analysis by high-performance liquid chromatography - ultra-violet detection (HPLC/UV)
- Method 3: Active sampling on filter impregnated with sulphuric acid using a CFC, acetonitrile/water + dansyl chloride elution, analysis by ultra performance liquid chromatography - detection by tandem mass spectrometry (UPLC-MS/MS)
- Method 4: Active sampling on filter impregnated with sulphuric acid using a CFC, ethanol elution, analysis by GC/MS
- Method 5: Active sampling on silica gel tube, ethanol/water elution, analysis by gas chromatography – flame ionisation detection (GC/FID)
- Method 6: Active sampling on XAD-7 tube impregnated with phosphoric acid, methanol/water + ammonium hydroxide elution, analysis by GC/FID
- Method 7: Active sampling on Tenax tube, thermal desorption, analysis by GC/FID
- Method 8: Active sampling on silica gel tube, methanol + potassium hydroxide elution, analysis by gas chromatography – nitrogen phosphorus detection (GC/NPD)

Due to its vapour pressure, aniline is found in workplace air in vapour and particulate forms. That is why methods 5, 6, 7 & 8, enabling only the gas phase to be sampled, have been classified in Category 3 for regulatory technical control of the 8h-OEL and the 15min-STEEL, as well as for short-term exposure monitoring.

Method 1 uses a GSP1 sampling device. Although this device was adapted from the GSP3.5, considered as indicative of the inhalable fraction (ANSES, 2020b), no validation data are available for the GSP1 with regard to the inhalable fraction, whether or not this sampler is used with an impregnated filter. This sampling device has therefore been classified in Category 3\* in terms of its compliance with regard to the inhalable fraction.

This method covers the range of 0.1 to 2 times the 8h-OEL with two 4h samples, but without additional data on the influence of high humidity on the capacity of the sulphuric acid-impregnated filter, the analytical method has been classified in Category 2 for regulatory technical control of the 8h-OEL.

For a 15min sampling, the method has been validated over a range of concentrations that covers 0.64 to 2 times the 15min-STEEL. However, the quantification limit of the method is below 0.1 times the 15min-STEEL and covers the lower part of the targeted range of concentrations. The protocol does not provide any information regarding the influence of potential interfering compounds or environmental conditions, including humidity; however, over a period of 15

minutes, these influences are considered limited. The analytical method has therefore been classified in Category 1B for regulatory technical control of the 15min-STEL and short-term exposure monitoring.

Therefore, measurement method 1 has been classified in Category 3\*, corresponding to the overall classification (sampling + analysis), for regulatory technical control of the 8h-OEL and the 15min-STEL, as well as for short-term exposure monitoring.

For an 8h or 15min sampling, methods 2, 3 & 4 do not cover the targeted concentration ranges. Moreover, the CFC recommended in these methods is not compliant with regard to the inhalable fraction (ANSES, 2020b), and neither is the IOM device recommended by method 2, since deposition on the walls is not taken into account. Therefore, methods 2, 3 & 4 have been classified in Category 3 for regulatory technical control of the 8h-OEL and the 15min-STEL, as well as for short-term exposure monitoring.

#### Assessment of the measurement methods for chloromethane

Three methods for the measurement of chloromethane in workplace air were identified and assessed:

- Method 1: Active sampling on an adsorbent tube, solvent desorption, analysis by GC/FID
- Method 2: Passive sampling, thermal desorption and analysis by GC/FID or MS or an other selective detector
- Method 3: Passive sampling, solvent desorption, analysis by GC/FID or MS or an other selective detector

Method 1 uses two activated charcoal tubes mounted in series. This method is not able to reach 0.1 times the 8h-OEL. Data on the breakthrough volume do not allow the sampling conditions to be adjusted to reach this level. Therefore, this measurement method has been classified in Category 3 for regulatory technical control of the 8h-OEL.

Methods 2 and 3, described by generic standards and protocols for measuring volatile organic compounds (VOCs), have been classified in Category 3\* for technical control of the 8h-OEL. Only the diffusive uptake rate is specified for a given sampling medium for each of these methods (Spherocarb for method 2 and activated charcoal (ORSA-5) for method 3) in the data specific to chloromethane.

A literature search did not find any additional validation data.

#### Assessment of the measurement methods for 2-phenylpropane (cumene)

Four methods for the measurement of 2-phenylpropane in workplace air were identified and assessed:

- Method 1: Active sampling on an adsorbent tube, solvent desorption, analysis by GC/FID or GC/MS
- Method 2: Active sampling on an adsorbent tube, thermal desorption, analysis by GC/FID or GC/MS
- Method 3: Passive sampling on an adsorbent tube, thermal desorption, analysis by GC/FID
- Method 4: Passive sampling on an adsorbent medium, solvent desorption, analysis by GC/FID

Method 1, described by 11 protocols, has complete validation data meeting the requirements of the NF EN 482 standard. It covers the range from 0.1 to 2\*8h-OEL with an 8h sampling and the range from 0.1 to 2\*15min-STEL with a 15min sampling, using a low- or high-capacity activated

charcoal tube. The influence of interfering compounds and environmental conditions is mentioned but not studied.

Method 1 has therefore been classified in Category 1B for regulatory technical control of the 8h-OEL and the 15min-STEEL, as well as for short-term exposure monitoring.

Methods 2, 3 & 4 have been classified in Category 3\* for regulatory technical control of the 8h-OEL and the 15min-STEEL, as well as for short-term exposure monitoring, due to the absence of validation data available in the protocols describing these methods.

☐ Assessment of the measurement methods for n-butyl acetate, isobutyl acetate and sec-butyl acetate

Five methods for the measurement of n-butyl acetate, isobutyl acetate and sec-butyl acetate in workplace air were identified and assessed:

- Method 1: Active sampling on an adsorbent tube, solvent desorption, analysis by GC/FID
- Method 2: Active sampling on an adsorbent tube, solvent desorption, analysis by headspace GC/FID or GC/MS
- Method 3: Active sampling on an adsorbent tube, thermal desorption, analysis by GC/FID or GC/MS
- Method 4: Passive sampling on an adsorbent tube, solvent desorption, analysis by GC/FID
- Method 5: Passive sampling on an adsorbent tube, thermal desorption, analysis by GC/FID or GC/MS

Method 1 is described by 16 protocols and is based on sampling either on a low-capacity (100/50 mg) activated charcoal tube (TCAN; activated charcoal tube type NIOSH) or a higher-capacity (300/700 mg) activated charcoal tube (TCA). The validation data meet the requirements for n-butyl acetate (NBA), isobutyl acetate (IBA) and sec-butyl acetate (SBA). For SBA, note that there are no validation data with the TCA medium but that performance similar to that of the other isomers has been observed with the other protocols. In light of the above, SBA sampling on this medium can also be recommended.

When using the TCAN tube, the flow rate should be adjusted to 20 mL·min<sup>-1</sup> over 8h or to 200 mL·min<sup>-1</sup> over 15min to respectively cover 0.1 to 2\*8h-OEL (except for SBA: 0.2 to 2\*8h-OEL) and 0.1 to 2\*15min-STEEL.

When using the TCA tube, the validation data cover 0.1 to 2\*8h-OEL considering a rate of 5 L·h<sup>-1</sup> for 8h, and 0.1 to 2\*15min-STEEL for a rate of 20 L·h<sup>-1</sup> for 15min. This method has therefore been classified in Category 1A for regulatory technical control of the 8h-OEL and the 15min-STEEL, as well as for short-term exposure monitoring for each of the three butyl acetates.

Method 2 only differs from method 1 in that it analyses the headspace of the extract instead of injecting an aliquot and uses a 700/300 mg high-capacity activated charcoal tube.

Adjusting the rate to 25 mL·min<sup>-1</sup> over 8h enables the method to cover 0.1 to 2\*8h-OEL. Over a period of 15min, the method can cover 0.1 to 2\*15min-STEEL if the rate is adjusted to 800 mL·min<sup>-1</sup>, which remains within the range recommended in the protocol.

The protocol's validation data focus on IBA. However, the limit of quantification given remains indicative and the uncertainty data provided are limited. No data are available for NBA or SBA. Nevertheless, given the similarities between the isomers on the one hand and to method 1 on the other, the observed performance should be similar. Method 2 could therefore also be used to measure NBA and SBA.

This method has therefore been classified in Category 2 for regulatory technical control of the 8h-OEL and the 15min-STEEL, as well as for short-term exposure monitoring for each of the three butyl acetates.

Method 3 is covered by six protocols and has validation data for NBA and IBA based on active sampling on tubes containing Tenax TA or Chromosorb 106.

Examination of these data shows that the medium does not have a sufficient capacity to reach 2\*8h-OEL. A very low sampling flow rate of 2 mL·min<sup>-1</sup> would need to be used for 15min to cover 0.1 to 2\*15min-STEEL. Such a rate cannot currently be implemented in the field.

Therefore, this method has been classified in Category 3 for regulatory technical control of the 8h-OEL and the 15min-STEEL, as well as for short-term exposure monitoring for each of the three butyl acetates.

Method 4 is described by six protocols supplemented by another document (OSHA, 1998) presenting validation data for one of the proposed passive samplers (the SKC575-001 badge). The most complete validation data concern the SKC575-002 and 3M 3520 OVM media, for the three butyl acetates. The method is able to partially cover the range from 0.1 to 2\*8h-OEL with these badges over 4h of exposure repeated twice. It also partially covers the range from 0.1 to 2\*15min-STEEL, with preference given in this case to the higher-capacity 3M 3520 OVM badge, especially for SBA.

The presented validation data meet the requirements for the three substances, in particular the stability of the sampling flow rate over periods as short as 15 minutes. However, the uncertainty data provided target concentrations outside of the range. The influence of air speed was assessed with a medium having characteristics similar to those of these two media.

This method has been classified in Category 1B for regulatory technical control of the 8h-OEL for each of the three butyl acetates with use of the SKC575-002 or 3M 3520 badge.

For regulatory technical control of the 15min-STEEL and for short-term exposure monitoring, this method has been classified:

- for NBA and IBA: in Category 1B with use of the SKC575-002 or 3M 3520 badge;
- for SBA: in Category 1B with use of the 3M 3520 badge and in Category 2 with use of the SKC575-002 badge.

Method 5 is represented by three protocols based on the use of passive tubes containing Tenax TA or Chromosorb 106.

This method has been classified in Category 3\* for regulatory technical control of the 8h-OEL due to the absence of validation data available in the protocols, whether for n-butyl acetate, isobutyl acetate or sec-butyl acetate. It has been classified in Category 3 for regulatory technical control of the 15min-STEEL and for short-term exposure monitoring, due to the time needed to obtain a stable flow rate for each of the three butyl acetates.

#### Assessment of the measurement methods for 4-aminotoluene

Three methods for the measurement of 4-aminotoluene in workplace air were identified and assessed:

- Method 1: Active sampling on filter impregnated with sulphuric acid using a GSP1 device, acetonitrile/ammonia elution, analysis by GC/MS
- Method 2: Active sampling on 2 filters impregnated with sulphuric acid using a CFC, sodium hydroxide / toluene elution, heptafluorobutyric anhydride derivatisation, analysis by GC/MS or gas chromatography – electron capture detection (GC/ECD)
- Method 3: Active sampling on XAD-7 tube, toluene elution and then heptafluorobutyric anhydride derivatisation, analysis by GC/MS or GC/ECD

Due to its vapour pressure, 4-aminotoluene is found in workplace air in vapour and particulate forms. That is why method 3, described by the IFA 8776 protocol, enabling only the gas phase to be sampled, has been classified in Category 3 for regulatory technical control of the 8h-OEL and the 15min-STEEL, as well as for short-term exposure monitoring.

Method 1, described by the DGUV 213-583 protocol, Method 1, uses a GSP1 sampling device. Although this device was adapted from the GSP3.5, considered as indicative of the inhalable fraction (ANSES, 2020b), no validation data are available for the GSP1 with regard to the inhalable fraction, whether or not this sampler is used with an impregnated filter. This sampling device has therefore been classified in Category 3\* in terms of its compliance with regard to the inhalable fraction.

Furthermore, the method is not able to reach 2 times the 8h-OEL. In the absence of data on the breakthrough volume, the analytical method and therefore the measurement method have been classified in Category 3 for regulatory technical control of the 8h-OEL.

However, for a 15min sampling, the analytical method has been validated over a range of concentrations that covers 0.1 to 2 times the 15min-STEEL. It has therefore been classified in Category 1B for regulatory technical control of the 15min-STEEL, although no information is provided regarding the influence of interferers or environmental conditions. Indeed, these parameters have hardly any influence on a 15min sampling.

Therefore, the measurement method has been classified in Category 3\*, corresponding to the overall classification (sampling + analysis), for regulatory technical control of the 15min-STEEL and for short-term exposure monitoring.

Method 2 uses a CFC sampling device, which is not compliant with regard to the inhalable fraction (ANSES, 2020b). This sampling device has therefore been classified in Category 3 in terms of its compliance with regard to the inhalable fraction.

This method has been analytically validated for a 100-120 L air sampling at 1 L·min<sup>-1</sup>, i.e. for 2h of sampling.

In light of the data on the collection efficiency determined for 100 L of air sampled at concentrations far higher than 2\*8h-OEL, it should therefore be possible to take a 4h sampling (i.e. 240 L of air) at 2\*8h-OEL with no risk of saturating the medium. The analytical method has therefore been classified in Category 1B for technical control of the 8h-OEL.

The measurement range covers 0.1 to 2\*15min-STEEL but most of the data (extraction efficiency, uncertainties) were obtained with a range of concentrations far higher than this range. The analytical method has therefore been classified in Category 1B for technical control of the 15min-STEEL and the of short-term exposure monitoring.

Therefore, method 2 has been classified in Category 3, corresponding to the overall classification (sampling + analysis), for regulatory technical control of the 8h-OEL and the 15min-STEEL, as well as for short-term exposure monitoring.

#### Assessment of the measurement methods for isoamyl alcohol

Two methods for the measurement of isoamyl alcohol in workplace air were identified and assessed:

- Method 1: Active sampling on an adsorbent tube, solvent desorption, analysis by GC/FID
- Method 2 : Passive sampling on an adsorbent tube or badge, solvent desorption, analysis by GC/FID

Method 1 is described by six protocols and has complete validation data meeting the requirements. It should be noted that these validation data were obtained with activated charcoal in dry air whereas high humidity is likely to reduce the trapping capacity. The



conditions described by the NIOSH 1405 protocol are able to cover the range from 0.1 to 2\*8h-OEL with an 8h sampling at the rate of 20 mL·min<sup>-1</sup> and it is possible to cover 0.1 to 2 times the 15min-STEEL with a 15min sampling at the rate of 200 mL·min<sup>-1</sup>.

This method has therefore been classified in Category 1B for regulatory technical control of the 8h-OEL and the 15min-STEEL, as well as for short-term exposure monitoring.

In the absence of essential validation data specific to isoamyl alcohol, method 2 has been classified in Category 3\* for regulatory technical control of the 8h-OEL and the 15min-STEEL, as well as for short-term exposure monitoring.

#### Assessment of the measurement methods for phosphoryl trichloride

No method for the measurement of phosphoryl trichloride in workplace air has been identified.

It is indeed difficult to measure levels of phosphoryl trichloride in air due to its reactivity with ambient atmospheric humidity.

A literature search identified a study proposing a measurement method whose principle consists of active sampling with an impinger containing an Na<sub>2</sub>CO<sub>3</sub>/NaHCO<sub>3</sub> solution, followed by an ion chromatography analysis (Zhao *et al.*, 2011). Due to primarily analytical validation data, the non-specificity of the analytical method (no distinction between phosphoryl trichloride and its hydrolysis products) and a limit of detection greater than one-tenth of the 8h-OEL and the 15min-STEEL, this method has been classified in Category 3 for regulatory technical control of the 8h-OEL and the 15min-STEEL, as well as for short-term exposure monitoring.

**Table 3 : Measurement methods identified and assessed for each substance**

Substance	Method		Protocols
	N <sup>o</sup>	Principle	References
Aniline	1	Active sampling on filter impregnated with sulphuric acid using a GSP1 device, acetonitrile/ammonia elution, analysis by GC/MS	DGUV 213-583 méthode 1 (2013)
	2	Active sampling on 2 filters impregnated with H <sub>2</sub> SO <sub>4</sub> , methanol/water+sodium hydroxide elution, analysis by HPLC/UV	HSE MDHS 75/2 (2014), INRS MétroPol M-203 (2016)
	3	Active sampling on filter impregnated with H <sub>2</sub> SO <sub>4</sub> , acetonitrile/water + dansyl chloride elution, analysis by UPLC-MS/MS	IRSST MA-363 (2019)
	4	Active sampling on filter impregnated with sulphuric acid using a CFC, ethanol elution, analysis by GC/MS	NIOSH 2017 (1998)
	5	Active sampling on silica gel tube, ethanol/H <sub>2</sub> O elution, analysis by GC/FID	NIOSH 2002 (1994) , HSE MDHS 96 (2000) , NF ISO 16200-1 (2002)
	6	Active sampling on XAD-7 tube impregnated with phosphoric acid, methanol/water + ammonium hydroxide elution, analysis by GC/FID	OSHA PV2079 (1994)
	7	Active sampling on Tenax tube, thermal desorption, analysis by GC/FID	HSE MDHS 72 (1993), HSE MDHS 104 (2016), NF EN ISO 16017-1 (2001)
	8	: Active sampling on silica gel tube, methanol + KOH elution, analysis by GC/NPD	IFA 6073 (2010)
oro met han	1	Active sampling on an adsorbent tube, solvent desorption, analysis by GC/FID	NIOSH 1001 (1994), HSE MDHS 96 (2000), NF ISO 16200-1 (2001)

Substance	Method		Protocols
	N <sup>o</sup>	Principle	References
	2	Passive sampling, thermal desorption and analysis by GC/FID or MS	HSE MDHS 80 (1995), HSE MDHS 104 (2016), NF EN ISO 16017-2 (2003)
	3	Passive sampling, solvent desorption, analysis by GC/FID or MS	HSE MDHS 88 (1997), ISO 16200-2 (2000)
2-Phenylpropane (cumene)	1	Active sampling on an adsorbent tube, solvent desorption, analysis by GC/FID or GC/MS	<b>DFG solvent mixtures method 1 (2013)</b> , INRS MétroPol M 267 (2019), NF ISO 16200-1 (2001), NIOSH 1501 (2003), NF X43-267 (2014), <b>IRSST MA-369 (2012)</b> , OSHA PV2137 (2004), IRSST 159-1 (1990), <b>DFG solvent mixtures method 2 (2013)</b> , IFA 7733 (2005), <b>DFG solvent mixtures method 2 (1997)</b>
	2	Active sampling on an adsorbent tube, thermal desorption, analysis by GC/FID or GC/MS	NF EN ISO 16017-1 (2001), HSE MDHS 72 (1993), HSE MDHS 104 (2016), <b>DFG Solvent mixtures method 5 (1997)</b> , <b>DFG Solvent mixtures method 6 (2013)</b>
	3	Passive sampling on an adsorbent tube, thermal desorption, analysis by GC/FID	NF EN ISO 16017-2 (2003), HSE MDHS 80 (1995), HSE MDHS 104 (2016)
	4	Passive sampling on an adsorbent tube, solvent desorption, analysis by GC/FID	HSE MDHS 88 (1997), ISO 16200-2 (2000), IRSST 159-1 (1990)
n-butyl, isobutyl and sec-butyl acetates	1	Active sampling on an adsorbent tube, solvent desorption, analysis by GC/FID	<b>OSHA 1009 (2007)</b> , NIOSH 1450 (2003), HSE MDHS 96 (2000), NF ISO 16200-1 (2001), NF X 43-267 (2014), <b>INSST MTA/MA-023/A92 (1992)</b> , INRS MétroPol M 54 (2017), IRSST 274-1 (?), IRSST 249-1 (1990), IRSST-77-1 (1990), <b>IRSST MA-369 (2012)</b> , DFG solvent mixtures method 1 (1997), DFG solvent mixtures method 1 (2013), DFG solvent mixtures method 2 (1997), <b>DFG solvent mixtures method 2 (2013)</b> , IFA 7322 (2009)
	2	Active sampling on an adsorbent tube, solvent desorption, analysis by headspace GC/FID or GC/MS	<b>DFG solvent mixtures method 4 (1997)</b>
	3	Active sampling on an adsorbent tube, thermal desorption, analysis by GC/FID or GC/MS	NF EN ISO 16017-1 (2001), HSE MDH 72 (1993), <b>DFG Solvent mixtures method 5 (1997)</b> , HSE MDHS 104 (2016), <b>DFG Solvent mixtures method 6 (2013)</b>
	4	Passive sampling on an adsorbent tube, solvent desorption, analysis by GC/FID	<b>OSHA 1009 (2007)</b> , IRSST 274-1 (?), IRSST 249-1 (1990), INRS MétroPol M 351 (2017), HSE MDHS 88 (1997), ISO 16200-2 (2000), OSHA (1998)
	5	Passive sampling on an adsorbent tube, thermal desorption, analysis by GC/FID or GC/MS	NF EN ISO 16017-2 (2003), HSE MDHS 80 (1995), HSE MDHS 104 (2016)
4-Aminotoluene	1	Active sampling on filter impregnated with sulphuric acid using a GSP1 device, acetonitrile/ammonia elution, analysis by GC/MS	<b>DGUV 213-583 method 1 (2012)</b>
	2	Active sampling on 2 filters impregnated with sulphuric acid using a CFC, sodium hydroxide / toluene elution, heptafluorobutyric anhydride derivatisation, analysis by GC/MS or GC/ECD	<b>DGUV 213-583 method 2 (2019)</b> , OSHA 73 (1988)
	3	Active sampling on XAD-7 tube, elution toluene then heptafluorobutyric anhydride derivatisation, analysis by GC/MS or GC/ECD	IFA 8776 (2004)
Isoamyl alcohol	1	Active sampling on an adsorbent tube, solvent desorption, analysis by GC/FID	<b>INRS MétroPol M-85 (2016)</b> , NF X43-267 (2014), NIOSH 1402 (1994), HSE MDHS 96 (2000), NF ISO 16200-1 (2001), <b>NIOSH 1405 (2003)</b> , update de NIOSH 1402)
	2	Passive sampling, on an adsorbent tube or badge, solvent desorption, analysis by GC/FID	HSE MDHS 88 (1997), ISO 16200-2 (2000)
Phosphoryl trichloride	1	Active sampling through an impinger containing a Na <sub>2</sub> CO <sub>3</sub> /NaHCO <sub>3</sub> solution, analysis by ion chromatography	Zhao <i>et al.</i> 2011

**In bold** : protocols with the most complete validation data

In blue : methods recommended by the Metrology WG

## Conclusions and recommendations of the collective expert appraisal

### Conclusions

The evaluation of the reference methods applicable for the measurement of occupational exposure levels for the 9 substances to be assessed and listed in Directive (EU) 2019/1831 in light of the values established therein indicates that:

- n-butyl, isobutyl and sec-butyl acetates have a measurement method classified as category 1A for the regulatory technical control of the 8h-OEL and 15min-STEEL as well as for the monitoring of short-term exposures;
- cumene and isoamyl alcohol have a measurement method classified as category 1B for the regulatory technical control of the 8h-OEL and 15min-STEEL as well as for the monitoring of short-term exposures;
- aniline, chloromethane, 4-aminotoluene and phosphoryl trichloride do not have a validated or indicative measurement method for monitoring the OELs, or the available data were insufficient to assess the measurement methods.

### Recommendations

The table below summarises the measurement methods recommended by the Metrology WG for all substances according to their classification.

As there is no measurement method suitable for monitoring the 8h-OEL and 15min-STEEL for aniline, chloromethane, 4-aminotoluene and phosphoryl trichloride, it is recommended to develop and validate a measurement method for these substances.

Regarding aniline and 4-aminotoluene, the use of a device recommended for sampling the inhalable fraction of aerosols (cf. Anses, 2020b) and enabling the use of a filter impregnated with sulfuric acid and compatible with the analytical method described in the DGUV 213-583 method 1 protocol (method 1 for aniline and 4-aminotoluene) or the analytical method described in the DGUV 213-583 method 2 or OSHA 73 protocols (method 2 for 4-aminotoluene) should enable concentrations of these substances to be measured for comparison with the 8h-OEL or the 15min-STEEL; subject to validation.

**Table 4: Measurement methods recommended by the Metrology WG**

Identification of the substance		Principle of the recommended method	Implementation protocols (References)	Classification for regulatory technical control		Classification for short-term exposure monitoring	Additional information
Name	CAS number			8h- OEL	15min-STEEL		
Aniline	62-53-3	No recommended method in workplace air					The use of a device recommended for sampling the inhalable fraction of aerosols (cf. Anses, 2020b) and enabling the use of an impregnated filter compatible with the analytical method described in the DGVU 213-583 method 1 protocol should enable aniline concentrations to be measured for comparison with the 8h-OEL or the 15min-STEEL ; subject to validation
Chloromethane	74-87-3	No recommended method in workplace air					/
2-Phenylpropane (cumene)	98-82-8	Active sampling on sorbent tube Solvent desorption Analysis by GC/FID or GC/MS	<p><b>DFG solvent mixtures method 1 (2013), INRS MétroPol M 267 (2019), NF ISO 16200-1 (2001), NF X 43-267 (2014), NIOSH 1501 (2003), IRSST MA-369 (2012), OSHA PV2137 (2004), IRSST 159-1 (1990)</b></p> <p><b>DFG solvent mixtures method 2 (2013), IFA 7733 (2005), DFG solvent mixtures method 2 (1997)</b></p>		1B		Users should be aware that CS <sub>2</sub> used for desorption (alone or in mixture with other solvents) is classified as toxic to reproduction, Category 2.

Identification of the substance		Principle of the recommended method	Implementation protocols (References)	Classification for regulatory technical control		Classification for short-term exposure monitoring	Additional information
Name	CAS number			8h- OEL	15min-STEL		
n-Butyl acetate	123-86-4	Active sampling on activated charcoal tube Solvent desorption Analysis by GC/FID	OSHA 1009, NIOSH 1450, INSHT MTA/MA-023/A92, IRSST MA-369, HSE MDHS 96, NF ISO 16200-1, NF X 43-267, INRS MétroPol M 54, , <b>INSST MTA/MA-023/A92</b> , IRSST 274-1, IRSST 249-1, IRSST-77-1, DFG solvent mixtures method 1 (1997 & 2013), <b>DFG solvent mixtures method 2 (1997 et 2013)</b> , IFA 7322	1A		Users should be aware that CS <sub>2</sub> used for desorption (alone or in mixture with other solvents) is classified as toxic to reproduction, Category 2.	
Isobutyl acetate	110-19-0						
sec-Butyl acetate	105-46-4			Passive sampling on activated charcoal badge (SKC575-002 and 3M 3520 OVM) Solvent desorption Analysis by GC/FID	OSHA 1009		1B

Identification of the substance		Principle of the recommended method	Implementation protocols (References)	Classification for regulatory technical control		Classification for short-term exposure monitoring	Additional information
Name	CAS number			8h- OEL	15min-STEL		
4-Aminotoluene	106-49-0	No recommended method in workplace air					The use of a device recommended for sampling the inhalable fraction of aerosols (cf. Anses, 2020b) and enabling the use of an impregnated filter compatible with the analytical method described in the DGUV 213-583 method 1 protocol or the analytical method described in the DGUV 213-583 method 2 or OSHA 73 protocols should enable 4,aminotoluene concentrations to be measured for comparison with the 8h-OEL or the 15min-STEL ; subject to validation
Isoamyl alcohol	123-51-3	Active sampling on activated charcoal tube Solvent desorption Analysis by GC/FID	<b>INRS MétroPol M-85 (2016)</b> , NF X43-267 (2014), NIOSH 1402 (1994), HSE MDHS 96 (2000), NF ISO 16200-1 (2001), <b>NIOSH 1405 (2003)</b>		1B		Users should be aware that CS <sub>2</sub> used with dichloromethane or 2-propanol for desorption is classified as toxic to reproduction, Category 2.
Phosphoryl trichloride	10025-87-3	No recommended method in workplace air					/

*In bold : protocols with the most complete validation data*  
 (1) For sec-butyl acetate: sampling only on badge 3M 3520

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