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French agency for food, environmental and occupational health & safety

Guidelines for safety assessment of lubricants used in installations for the production and distribution of water intended for human consumption

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Introduction

Water intended for human consumption (Drinking water, DW) is essential for life and health. As such, it must be continuously distributed to the population in sufficient quantities and at sufficient pressure and must meet the quality requirements set by the French Public Health Code (CSP).

When they come in contact with DW, some materials and products can adversely affect the water's organoleptic, physico-chemical or microbiological qualities and the water thereby may fail to meet the quality requirements set by the CSP.

Therefore, the marketing of materials and products designed to come into contact with DW, as well as their use in water production and distribution installations are subject to regulations.

In the case of organic materials and products, obtaining an attestation of sanitary conformity (ACS), issued by one of the laboratories authorised by the French Ministry of Health, constitutes proof of compliance with the regulatory requirements.

An ACS is issued subject to:
- the components used in the material's manufacture being included in positive lists of substances authorised by the regulations (PLs),
- the material's migration test results complying with the pass-fail criteria (PFC) defined in the regulations.

For lubricants, thus far and in the absence of a validated migration test protocol, the only requirement has been to obtain a certificate of conformity for the chemical formulation with regard to PLs (CLP), issued by one of the laboratories authorised by the French Ministry of Health, as proof of compliance with regulatory requirements[1].

Lubricants (greases and oils) are employed for three distinct purposes:
- lubrication and/or leak tightness of moving surfaces,
- jointing (leak tightness) of parts,
- easing assembly of pipes or accessories.

The purpose of this report is to specify **the conditions for obtaining an attestation of sanitary conformity (ACS) for lubricants.** These guidelines are intended to provide information on:
- the compilation of an ACS application dossier that is to be submitted to laboratories authorised by the French Ministry of Health,
- the assessment of product safety and the examination of ACS applications by the authorised laboratories.

The report specifies:
- the conditions under which the conformity of the chemical formulation of the lubricant is examined,
- the nature of the migration tests conducted, including the water contact conditions and the parameters to be analysed in the migration waters,
- the pass-fail criteria for lubricants,
- the required information has to be supplied by the applicant seeking an ACS,
- recommendations for using lubricants.

This report was presented to and validated by the CES on Water during the meetings held on 1 June and 6 July 2010.

[1] *N.B. The term 'lubricants' includes greases and oils that can be distinguished by their viscosity or their consistency and the term 'greases and lubricants' used in current regulations is inappropriate.*

Terminology

Accessories (pumps, valves, taps, etc.): finished products used in permanent facilities for the production, treatment and distribution of water intended for human consumption, but which have no water treatment function. They contain at least two types of material.

ACS: Attestation of sanitary conformity. Evidence provided by the person responsible for marketing the product that ensures that it complies with regulations in force. The ACS is issued by a laboratory authorised by the French Ministry of Health under Article R*.1321-52 of the French Public Health Code.

Assembly paste: made up of soaps, oils or greases and additives (silicon dioxide, slate, talc, mica, etc.).

Blank water: water obtained and maintained in the same conditions as the test water but which has not been put in contact with the tested material.

Composition: the respective quantities of each substance used in the manufacture of the finished product.

Constituents: list of substances used in the manufacture of the finished product.

Formulation (constituents and composition): nature and relative amount of all substances used in the manufacture of a finished product (e.g. lubricant).

Greases: organic or inorganic fatty substances, unctuous, viscous and semisolid at ambient temperature.

Jointing product: product intended to provide a seal between two parts.

Leak tightness: effective barrier between two media.

Lubricants: products placed between two moving surfaces to reduce surface-to-surface friction and/or wear. Lubricants are used to facilitate the operation of mechanical systems.

Materials:

- organic and/or inorganic compounds intended for the manufacture of structures,
- within the meaning of Article R. 1321-48 of the CSP: finished products used in installations for the production, treatment and distribution of water intended for human consumption which supply water without altering its physico-chemical or microbiological composition. These include pipes, connectors, coatings, joints, etc., irrespective of their constituent material (metal, inorganic, organic, etc.). They can be factory-made or applied on-site, particularly for manufacture, repair or reconditioning. Lubricants constitute a category of materials.

Migration: transfer of substances from the tested material into the test water.

Migration test: implementation of a test protocol to demonstrate the migration of any substances deriving from lubricants.

Mixture[2]: mixture (or solution) consisting of two or more substances.

Oakum: Material made up of 100% flax or hemp fibres cleaned and combed to separate the fibres.

Oils: organic or inorganic fatty substances, liquid at ambient temperature.

Positive reference lists (PLs): lists of chemicals authorised in the formulation of materials that come into contact with water intended for human consumption.

Soaps: fatty acid metal salts.

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[2] Definition in Regulation (EC) no. 1272/2008 of the European Parliament and of the Council of 16 December 2008 on classification, labelling and packaging of substances and mixtures, amending and repealing Directives 67/548/EEC and 1999/45/EC and amending Regulation (EC) no. 1907/2006.

Substance[2] chemical element and its compounds in a natural state or obtained through a manufacturing process, including any additive necessary for maintaining its stability and any impurity resulting from the process used, but excluding any solvent that can be separated without affecting the stability of the substance or changing its composition.

Test water: water put in contact with samples during migration tests, which must have the physico-chemical characteristics defined in the standards applicable to migration tests.

1 Background

The marketing of materials and products designed to come into contact with water intended for human consumption (DW) as well as their use in water production, treatment and distribution installations are subject to regulations.

Lubricants used in installations for the production, treatment and distribution of DW are covered by these regulations.

1.1 Regulatory context

1.1.1 Marketing of materials and products designed to come in contact with water intended for human consumption

In accordance with the provisions of the French Consumer Code, those who oversee the marketing of products must ensure that these products are appropriate for their intended use, meet the current requirements and are not likely to endanger consumer health. Article L.121-1 stipulates that "*any advertising, whatever its form, comprising claims, information or representations that are false or likely to mislead is prohibited [...]"* and Article L 212-1 specifies that "*from their initial market launch, products must fulfil the current requirements relating to public safety and health, fair trading practices and consumer protection". The person responsible for the initial launch of a product on a market is, therefore, obliged to verify that the latter conforms to current requirements [...]"*

In accordance with the provisions of Article 1321-48 of the CSP, "*materials and products marketed and intended for production, distribution and packaging installations that come in contact with water intended for human consumption must comply with the specific requirements defined by ministerial order issued by the French Ministry for Health, with the purpose of ensuring that they are not likely, in normal and foreseeable conditions of use, to present a danger for human health or lead to an alteration of the composition of water defined in reference to values set by this order […]* ».

In addition, specific provisions to be met for the major groups[3] of materials and products that come into contact with DW are defined in:

- the Ministerial order of 29 May 1997 amended [1] regarding materials and products used in permanent installations for the production, treatment and distribution of water intended for human consumption,
- Circulars no. 99/217 of 12 April 1999 and no. 2000/232 of 27 April 2000 [2], no. 2002/571 of 28 November 2002 [3] and DGS/SD7A/2006/370 of 21 August 2006 [4],
- the Guide issued by the Directorate General for Health (DGS) in March 1999 [5].

1.1.2 Use of materials and products that come into contact with water intended for human consumption

Article R. 1321-49-I of the CSP states that "*the person responsible for the production, distribution or packaging of water shall use, in new installations or in partially renovated installations, from the point of water intake to points of compliance defined in Article 1321-5, materials and products that come into contact with water intended for human consumption that comply with the provisions of Article R. 1321-48.*"

Furthermore
1) regarding natural mineral waters:

[3] *To date, the following categories of materials and products are distinguished:*
- *Metals, alloys and metallic coatings,*
- *Cementitious materials (concretes, mortars), enamels, ceramics and glass,*
- *Organic materials and products (plastic, bitumen, rubber and elastomer), particularly those that are fibre-reinforced,*
- *Accessories and subsets of accessories, consisting of at least one organic material.*

- Article L.1322-31 states that "*the provisions of Article R. 1321-49-I are applicable to natural mineral water businesses. In addition, the business must use materials in contact with natural mineral water compatible with its composition so as to prevent any chemical, physico-chemical, microbiological and organoleptic alteration of the quality of the water as found upon emergence."*
- Article L.1322-36 states that "*the materials used for packaging natural mineral water, as defined in Regulation (EC) no. 1935/2004 of the European Parliament and of the Council of 27 October 2004 on materials and products intended to come into contact with food shall be processed or manufactured and used so as to prevent alteration of its chemical, microbiological and organoleptic characteristics."*

2) regarding spring water and bottled drinking water: Article L.1321-95 states that "*materials used for packaging must satisfy the conditions laid down in Article R. 1322-36."*

1.1.3 Specific provisions regarding lubricants designed to come into contact with water intended for human consumption

Annex E of Circular no. 2000/232 of 27 April 2000 amending Circular no. 99/217 of 12 April 1999 [2] states that:

- "*in May 1999, the expert committee on Materials/Water initiated the work of assessing the level of migration into water of solvents found in certain glues. A test protocol is currently under study.*
- *It is specified that no glue, adhesive, grease or lubricant may be issued with an ACS before the protocol has been validated by the expert committee and published in the official bulletin of the Ministry of Health.*
- *However, it is strongly recommended that manufacturers of these four types of products verify their compliance with positive reference lists:*
    - *when these products are placed directly in contact with water intended for human consumption,*
    - *or when they are likely to migrate into the water, due to the way in which they are used."*

Note 3 of Circular no. 2002/571 of 25 November 2002 [3] states that "*When anaerobic and epoxy glues, adhesives, greases or lubricants are used in the accessory,*

- *if the formulation of greases and lubricants used in the accessory complies with positive lists, then the constituent solvents of these greases and lubricants do not need to be screened for in migration water, as part of the procedure for issuing the ACS for the accessory under consideration,*
- *if a substance used in the formulation of the grease or lubricant is not on the positive reference lists, then this substance will be measured using GC-MS in the migration water,*
- *for anaerobic and epoxy glues used in an accessory, the laboratory only needs to know the formulation. No migration test is necessary."*

A list of lubricants which have been issued with a CPL certificate is available on the website of the French Ministry of Health (**www.sante.gouv.fr**), under the section on "materials that come into contact with water" (via the following path: accès direct par thème / "e" / eau / eau du robinet / matériaux entrant au contact de l'eau).

1.1.4 Specific provisions regarding lubricants designed to come into contact with water intended for human consumption in other countries

Lubricants found on the French market for use in systems supplying water intended for human consumption make reference to approvals and/or certificates which most often come from European countries with acceptance schemes for materials that come into contact with water for human consumption, particularly organic materials (Germany, [6], the Netherlands [7], the United Kingdom [8]) and the United States).

In Germany, lubricants must comply with KTW[4] guideline of the Federal Environment Agency (UBA[5]). A product's compliance is verified by an approved inspection body, such as the Water Technology Centre

[4] *Kunststoffe und Trinkwasser.*

[5] *Umwelt Bundes Amt für mensch und umwelt.*

(TZW[6]) which is recognized by a certifying body, the German Technical and Scientific Association for Gas and Water (DVGW[7]), and a certificate is issued. The UBA guidelines for assessing the hygiene conditions for lubricants in contact with water intended for human consumption specifies (www.umweltbundesamt.de/wasser-e/themen/downloads/drinking-water/leitlinie_schmierstoffe.pdf):

- the PLs of the raw materials that can be used in the manufacture of lubricants,
- the recommended migration test protocols, following the Standard DIN EN 12873-2 [9],
- the pass-fail criteria (PFC) for migration tests.

(see Annex 1 for more details on migration tests and on PFC). A list of authorised lubricants is available on their website (www.umweltbundesamt.de/wasser/themen/downloads/trinkwasser/schmierstoffe_anlage4.pdf).

In the United Kingdom (England, Wales, Scotland and Northern Ireland), materials and products used in public and private drinking water supplies must be evaluated by the Drinking Water Inspectorate (DWI) and those used solely within buildings by the Water Regulations Advisory Scheme (WRAS). Although there are no PLs, the formulation is subject to certain requirements: prohibition of lead and bituminous substances derived from tar, use of food colourings if possible, etc. Food-grade lubricants can be used without additional tests; otherwise they must pass reduced migration tests according to standards BS EN 12873-2 and BS 6920 [10] (odour and flavour, enhancement of microbial growth) (see Annex 1 for more details on migration tests and PFC). A list of materials and products authorised in public and private drinking water supplies is available on the DWI website (www.www.dwi.gov.uk/drinking-water-products/approved-products/soslistcurrent.pdf, see section C.6 on Sealant & Repair Materials) and those authorised solely within buildings on the WRAS web site (www.wras.co.uk/Directory/materials_Search.asp?, see the section on Lubricants).

In the Netherlands, only lubricants with a KIWA-ATA[8] certificate are authorised and recognised by the Dutch Ministry of Housing, Spatial Planning and the Environment (VROM) as complying with the regulations in force. They are authorised without prior migration tests and only the formulation is assessed for compliance with the Dutch PLs. A list of lubricants authorised by the KIWA is available on their website (http://kiwa.nl/ATA), via the following path: “Download hier het overzicht van producten die een Kiwa-Ata hebben” / “Download overzicht produckten” and under the section “Productgroep: Glijmiddelen".

In addition, the products authorised in Germany and in the Netherlands are subject to regular inspections of the production plant (audits) by organisations in charge of issuing authorisations.

In the United States, lubricants are assessed with regard to the NSF/ANSI standard 61 [11]. The United States has not drawn up PLs but there are requirements on the formulation: for example, lead cannot be intentionally used as a substance. Furthermore, conducting migration tests is mandatory (see Annex 1 for more details on migration tests and on PFC).

1.1.5 Lubricants used in the foodstuff industries

The certificates of compliance with positive lists (CLPs) that are mandatory for lubricants used in installations for the production and distribution of water intended for human consumption should not be confused with the “food-grade certificates” that are mandatory for lubricants used in the foodstuff industries[9].

Regulations regarding materials and articles designed to come into contact with food have no specific provisions for lubricants. Nevertheless, the inertia principle defined in Article 3 of Regulation (EC) no. 1935/2004 [12] applies:

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[6] Technologiezentrum Wasser.

[7] Deutsche Vereinigung des Gas- und Wasserfaches.

[8] Kiwa Attestation of Toxicological Aspects.

[9] Additional information on materials intended to come into contact with food are available on the website of the French National Laboratory for Metrology and Testing (www.contactalimentaire.com).

"*Materials and articles, including active and intelligent materials and articles, shall be manufactured in compliance with good manufacturing practice so that, under normal or foreseeable conditions of use, they do not transfer their constituents to food in quantities which could:*

a) endanger human health,
or
b) bring about an unacceptable change in the composition of the food,
or
c) bring about a deterioration in the organoleptic characteristics thereof."

as well as the requirement of Article 16 of the Regulation (EC), to provide a declaration stipulating that the products comply with the rules applicable to them and that the appropriate documentation shall be available to demonstrate such compliance.

In general, the "food-grade certificate" specifies that the product is:

- manufactured only from substances included in PLs for materials and articles designed to come into contact with food,
- and/or complies with guidelines established by the French National Centre for the Coordination of Studies and Research on Food and Nutrition (CNERNA) in 1992 [13],
- and/or registered with the National Sanitation Foundation (NSF) as a type H1 lubricant (lubricant that can be used when there is a risk of incidental food contact)[10]. It only contains substances authorised under Title 21 of the United States Code of Federal Regulations (CFR), parts 178.3570, 178.3620 and 182, for use in lubricants with incidental food contact[11].

Moreover, the Standard NF EN ISO 21469 [14] lays down the hygiene requirements that apply to the formulation, manufacture, use and handling of lubricants which may, during manufacture or processing, incidentally come into contact with products and packaging used in the food, cosmetics, pharmaceutical, tobacco or animal feeding stuffs industries.

1.2 Technical documents

It should be noted that while these guidelines cover only the safety of lubricants, the technical quality of products and the compliance with professional practices of gluing and/or assembling pipes and connectors that require lubricants are equally important. General rules for the use of these products are defined in unified technical documents (DTUs)[12]. Thus the DTUs from the 60 series regarding 'sanitary plumbing' and, more particularly, the DTU 60.31 (NF P 41-211) [15] must be complied with.

Furthermore, it is recommended that the lubricant be chosen from the categories defined in the ISO 6743-99 standard [16], according to its intended use.

Currently, no lubricants have been issued with an ATEC technical opinion[13] nor has their production been CSTBat certified.

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[10] NSF accreditation can be verified on the following website: www.nsf.org/usda/psnclistings.asp

[11] www.fda.gov/cdrh/aboutcfr.html

[12] DTUs are documents applicable to construction work in France. They are established by the 'General Committee for Building Standards/DTU)' whose Scientific and Technical Building Centre (CSTB) provides the secretariat. They deal with products covered under 'traditional techniques' (i.e. those that have been employed over a sufficiently long period so as to provide significant experience). They provide a standardised baseline for disaster risks in construction, generally taken into account by insurers. The knowledge of and compliance with these texts contribute to the common acceptance of the provisions and methods that provide for a durable and satisfactory level of building quality and performance over time. The 'General Committee for Building Standards/DTU' is now incorporated in the French standardisation system and DTUs are therefore standards that can become mandatory regulations.

[13] ATECs are issued by a specialised group of industry experts and assessed by the CSTB. They constitute an opinion of the suitability for use of a non-traditional product (or system) intended for construction.

2 Lubricants used in installations for the production and distribution of water intended for human consumption, and risks of water contamination

Lubricants are mainly composed of the following constituents (see Annex 2, which under no circumstances constitutes a positive list of substances authorised for the manufacture of lubricants that come into contact with water intended for human consumption):

- from 60 to 90% of base oil that may be petroleum-based, synthetic or natural (vegetable or animal oil),
- from 10 to 20% of thickening agents: metallic salts of fatty acids (soaps), bentonite, polytetrafluoroethylene (PTFE), silicone dioxide, graphite, etc.
- up to 5% of additives, each used at 0.1 to 0.5%: corrosion inhibitors, antioxidants, friction modifiers, anti-foaming agents, etc.

Generally speaking, they can be classified according to (see Annex 3):

- their intended use (see the NF ISO 8681 [17] and ISO 6743-99 [16] standards),
- in the case of liquid lubricants, their viscosity (see the NF ISO 3448 standard [18]),
- in the case of greases, their NLGI (National Lubricating Grease Institute) penetration (see the ISO 6743-99 [16] and NF ISO 2137 [19] standards).

For the specific case of networks for the production, treatment and distribution of water intended for human consumption, this report distinguishes among the three following uses:

- for lubricating and/or ensuring leak tightness of moving surfaces,
- for ensuring leak tightness of non-moving parts,
- for easing assembly of piping or accessories.

2.1 Lubrication and/or leak tightness of moving surfaces

Lubricants likely to come into contact with water are used in various accessories; for example in:

1) pumps and turbines (including booster, circulator and pressure maintenance pumps, etc.). They are used to lubricate bearings, spindles or pistons. In this context, the lubricant is directly in contact with water over more and less large surfaces.

2) air compressors used in water treatment (ozone production, washing filters, aeration, etc.) or in the networks (water hammer control units, maintaining reservoirs under pressure, cleaning operations, etc.). The lubricant contained in compressors may be carried into the air in aerosol form.

3) valves, taps, meters, etc. The lubricant is in direct contact with water.

2.2 Leak tightness of non-moving parts

Assembly paste or impregnated oakum are used to ensure the leak tightness of parts assembled using screw threads.

2.3 Easing assembly of piping or accessories

Lubricants are used to facilitate the slippage of parts during assembly of pipes or joints in fittings. They are for temporary use and should be eliminated when the system is operational.

Molecules likely to leach from lubricants may alter water quality (organoleptic or physico-chemical quality), or even lead to microbiological or toxicological risks. Their safety must therefore be assessed.

3 Safety assessment of lubricants

In 2010, there is no harmonised European acceptance scheme for materials that come into contact with DW, unlike for materials that come into contact with food. However, European-level deliberations in recent years have led to an agreement on the need to develop a system of acceptance for organic materials based on:

- formulations that comply with PLs,
- migration tests,
- common PLs and common testing protocols. Regarding the migration tests, the European Commission requested the European Committee for Standardization (CEN) to develop testing standards in the EN 12873 series.

Assessing the safety of lubricants is therefore based on the following two complementary approaches:

- examining the formulation of the lubricant,
- then conducting migration tests.

Thereafter, results of these assessments must be compared with PFC.

3.1 Examination of the formulation of the lubricant

Substances found in the formulation of the lubricant must be identified and compared with substances included in PLs recommended in AFSSA's Report and Opinion of 14 September 2004[14] (Solicited Request no. 2006-SA-0291 [20]) regarding positive lists of substances included in the composition of materials in contact with DW (see Annex 4 summarising the lists and substances that can be used).

3.2 Migration tests

The protocol based on the Standard **NF EN 12873-2 [9]** must be implemented, along with the following additional provisions:

- the test should be conducted once;
- the lubricant to be tested is placed in a stainless steel grade 316 L "flat-bottomed tray" with a surface area of 20 cm^2 and a depth of 1 mm:
  - for greases: the tray is filled to the top with the grease to be tested and a spatula is used to scrape off the excess grease and to smooth the surface to obtain a thickness of 1 mm,
  - for oils: 1 cm^3 of oil to be tested is placed in the tray using a pipette, for a thickness of 500 µm;
- the tray is placed in a crystalliser in which test waters are carefully added and removed;
- the tests are conducted with non-chlorinated water and with chlorinated water with a free chlorine (Cl_2) concentration of 1 ± 0.2 mg/L at a temperature of 23 ± 2°C as specified in Section 9.1 of Standard NF EN 12873-2.

At the end of each of the three stagnation periods of 72 h[15], the migration water is recovered for analysis (see Figure 1).

The concentration of the measured substances for each period of migration is calculated as follows:

$$c^T_n = a^T_n - b^T_n \text{ [mg/L]}$$

where:

- c^T_n is the concentration of the measured substance in mg/L,
- a^T_n is the concentration of the substance in mg/L measured in the migration water,
- b^T_n is the concentration of the substance in mg/L measured in the blank water,

under the following conditions:

[14] *Opinion and report available on the ANSES website: www.anses.fr*

[15] *When tests are performed with hot water (temperatures between 60 and 85 °C), the three migration periods are of 24 h.*

- T is the test temperature [23 ± 2°C or other value specified in Section 9.1 of the Standard NF EN 12873-2],
- n is the sequence number of the migration period (1, 2 or 3).

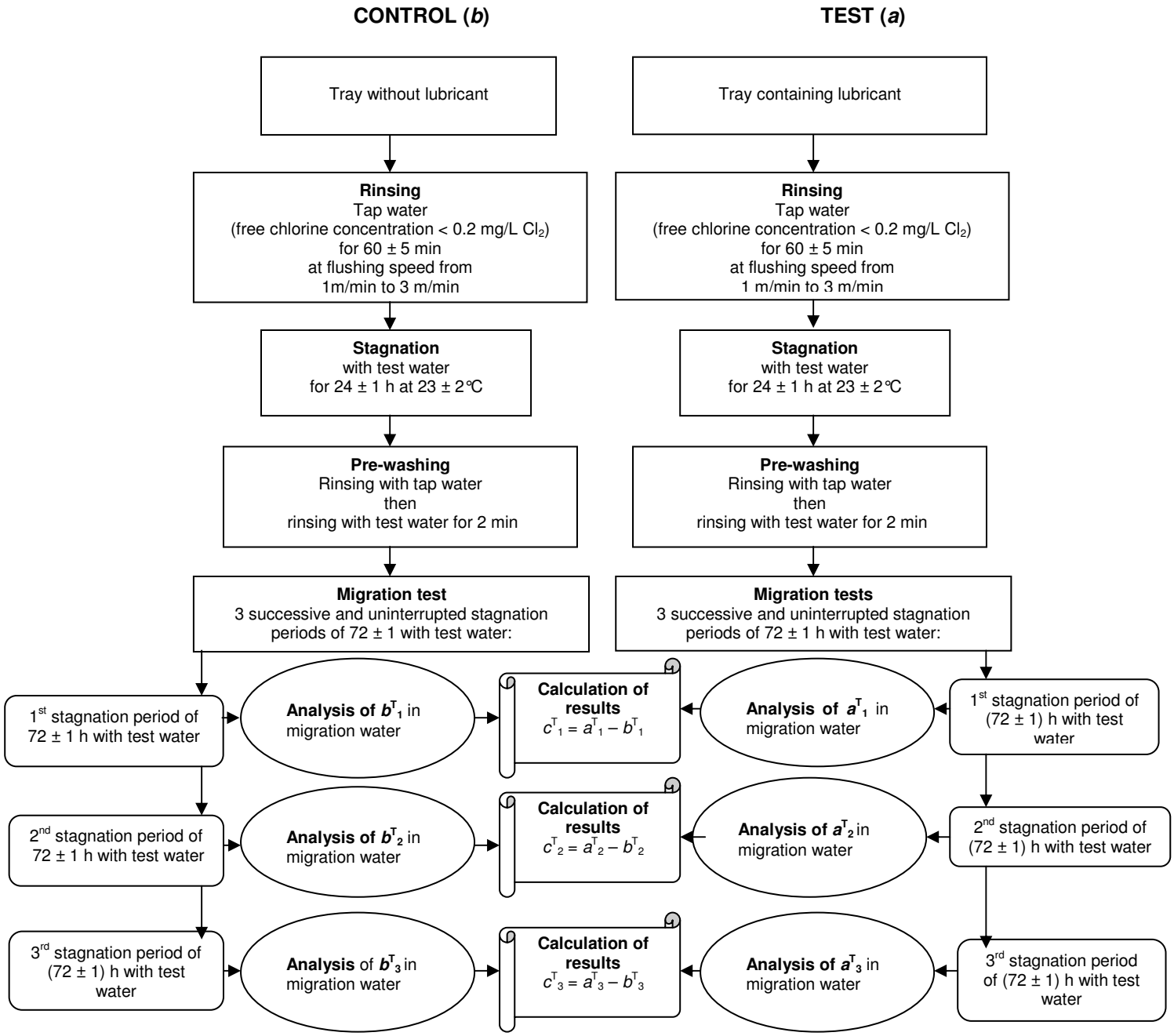
For each migration water, the migration rate M^T_n for a migrated substance can then be calculated as follows:

$$\boldsymbol{M^T n = c^T_n/(S/V \,.\, t)\ [mg/dm^2/day]}$$

where:

- M^T_n is the migration rate for n'th migration period,
- t is the duration of the migration period in days,
- S/V is the surface area-to-volume ratio in dm^{-1} estimated at 0.2 dm^{-1} (20 cm^2/L).

Thus $$\boldsymbol{M^T_n = c^T_n / 0.6\ [mg/dm^2/day]}$$



<u>Figure 1:</u> Flow diagram for migration test procedure conducted according to the NF EN 12873-2 standard
Test to be carried out at 23 ± 2°C on both non-chlorinated and chlorinated test water at 1 mg/L

The following parameters must be analysed in each of the three migration waters (t_1, t_2 and t_3):

- the total organic carbon (TOC) according to the NF EN 1484 standard [22],
- odour and flavour according to the NF EN 1622 standard [23],
- GC-MS[16] profile according to the NF EN 15768 draft standard [24],
- specific screening for parameters with a quality limit or reference specified in the Ministerial Order of 11 January 2007 [25], if present in the formulation of the lubricant,
- specific screening of substances on the lists of carcinogenic, mutagenic, toxic to reproduction (CMR) substances that have been classified according to a harmonised European scheme, if present in the lubricant.

[16] *GC-MS: Gas chromatography coupled with mass spectrometry*

In the absence of a European standard for assessing the ability of the material to promote microbial growth, this criterion, although used by certain Member States[17] for assessing organic materials, has not been included for testing.

3.3 Pass-fail criteria

3.3.1 Formulation

In principle, all substances included in the formulation of lubricants must be on PLs.

A certain weight percentage of non-compliant substances may be tolerated in the formulation due to:

- identified substances that are not on the PLs,
- commercial mixtures for which the exact formulation is not required because present only in a low percentage in the formulation of the finished product.

One or more substances not included in the PLs that are present in the formulation at a concentration equal to or less than **0.5%** in weight are tolerated (see Annex 5).

In addition, the maximum residual amounts specified in the PLs must be met.

However, the specific migration limits (SML) specified in the PLs, established for materials intended to come into contact with food (SML_{food}) have been deemed inappropriate for lubricants even after adapting them for materials intended to come into contact with DW (SML_{water})[18] and it is not required that they be systematically verified.

3.3.2 Migration tests

The results from the migration tests for the period of stagnation "'n" must be lower than or equal to results from migration tests for the migration period "n-1" and results from the analysis of migration water from the third period of stagnation must meet the PFC defined below.

The PFC for the organoleptic parameters (odour and flavour thresholds) must be lower than 3 (last dilution at which there is a significant difference in odour or flavour perceived by the tasting panel).

For parameters with a quality limit or reference set by the Ministerial Order of 11 January 2007, the adopted criterion is that quantities leached by materials must not exceed 10% of the quality requirements (limits or references) set by the aforementioned Order [25].
Heretofore, the French materials assessment system has considered that these quantities must not exceed 20% of the regulatory quality limits and references, whereas the Netherlands, Germany and the United Kingdom require that these quantities not exceed 10% of the regulatory quality limits and references. Given this disparity and in the interest of aligning with other European systems, ANSES proposes to use the value of 10%, which confers a wider margin of safety.
In addition, a conversion factor (CF) of 0.1 is applied to determine the concentration in test water (see Annex 5).
Given these factors, the PFC in test waters, for parameters for which there is a quality limit or reference set in the Ministerial Order of 11 January 2007, are set according to the following calculation:

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[17] *United Kingdom: BS 6920-2.4 (2000) – Suitability of non-metallic products for use in contact with water intended for human consumption with regard to their effect on the quality of water – Methods of test – Growth of aquatic micro-organisms test.*
Germany: DVGW W270 (2007) – The growth of microorganisms on materials intended for use in drinking water systems – examination and assessment (method based on the measurement of the mass quantity of biofilm developed on the surface of the material).
Netherlands: NVN 1225 (2004) – Drinking water – Determination of the Biomass Production Potential (BPP) of plastic materials, metals and coating in contact with (tap) water.

[18] *SML_{water} = SML_{food}/20: the World Health Organization (WHO) conventionally attributes 10% of the tolerable daily intake (TDI) to contaminants in water for a daily consumption of 2 L of water (see AFSSA's Opinion and Report no. 2006-SA-0291 [22]).*

PFC = 10% quality limit or reference / 0.1
= quality limit or reference x 1

For TOC, through the same reasoning, the adopted PFC for test water is 2 mg/L of C.

CMR substances, if present, must not exceed a value of 0.1 µg/L in DW, except when lower limit values have been set in the Ministerial Order of 11 January 2007 [25]. By considering that the quantities detected in DW leached by the materials must not exceed 10% of this quality requirement and by applying a conversion factor of 0.1, the adopted PFC for test waters is 0.1 µg/L.

Regarding the GC-MS profile for which the Ministerial Order of 11 January 2007 has not set any quality limit or reference, ANSES has assumed the surface area on which a lubricant is applied can be considered to be similar to that of a joint in an assembly. Given that the protocol in section 3.2 based on the NF EN 12873-2 standard [9] proposes to use an S/V ratio of 20 cm^2/L, the PFC (1 µg/L) regarding the GC-MS profile set out in Circular DGS/VS4 no. 99/217 of 12 April 1999 [2], which uses an S/V ratio of 3 cm^2/L for joints, was transposed using a conversion factor of 6.66 (20/3).The adopted PFC for test water is therefore 6.66 µg/L (1 µg/L x 6.66), rounded down to 5 µg/L for each compound detected and quantified relative to the closest alkane.

Table I: Summary of the parameters to be measured and the pass-fail criteria

Parameter	Test protocol	PFC (3^{rd} period of stagnation)	Unit
Flavour[19]	NF EN 1622 [23]	< 3	threshold
Odour[19]	NF EN 1622 [23]	< 3	threshold
Parameters with a quality limit or reference defined in the Ministerial Order of 11 January 2007 [25][20]		< Quality limit or reference in the Ministerial Order of 11 January 2007	Units in the Ministerial Order of 11 January 2007
CMR substances[20]		< 0.1 < 0.2	µg/L µg/dm^2/day
TOC[20]	NF EN 1484 [22]	< 2 < 3.5	mg/L of C mg/dm^2/day of C
GC-MS profile[20]	draft standard NF EN 15768 [24]	< 5 < 8.3	µg/L µg/dm^2/day

The results from the migration tests performed on various lubricants according to the protocol described in section 3.2 are given in Annex 6.

[19] *The acceptability criterion refers to a^T_3.*

[20] *The acceptability criterion refers to c^T_3 and M^T_3.*

4 Conditions for obtaining an attestation of sanitary conformity

The safety compliance of lubricants can only be assessed by a laboratory authorised to do so by the French Ministry of Health (see Article R. 1321-52 of the CSP and the Ministerial Order of 18 August 2009 [26]).

The applicant must submit a complete dossier to the authorised laboratory, including, in particular, information on the formulation of the lubricant (see Annex 7).

If the laboratory finds that the product meets the PFC regarding the formulation as set out in Section 3.3.1, migration tests are then carried out. Otherwise, the lubricant is declared non-compliant and no migration test is conducted.

For carrying out migration tests, the lubricant must be sent to the laboratory as it is packaged for sale.

If migration tests indicate that the product is compliant, an ACS may be issued for a period of 5 years.

If the applicant intends that the product be used with hot water, testing in these conditions is mandatory. If the applicant intends use with both hot and cold water, testing must be carried out at both temperatures.

The ACS must specify the temperatures at which the migration tests were performed.

If, when the ACS is to be renewed, the formulation of the lubricant, verified by the authorised testing laboratory, has not been modified and still complies with positive reference lists, the ACS can be renewed for a period of 5 years without performing further migration tests.

An ACS is issued for a given formulation, and this formulation can be marketed under one or more trade names. However, only one formulation can be associated with a given trade name.

A lubricant available in bulk packaging (tubes, tubs, etc.) and in aerosol form corresponds to two different formulations and requires that two applications be filed, except when the difference only involves the aerosol propellant and this propellant is an inert gas (e.g. nitrogen).

When a lubricant is sold under different molecular configurations (e.g. polydimethysiloxanes of different molecular weight, different hydrocarbon fractions, etc.) using the same formulation, the migration tests must be performed on the extreme [molecular] configurations; if the migration test results are significantly different, all the product configurations must be tested.

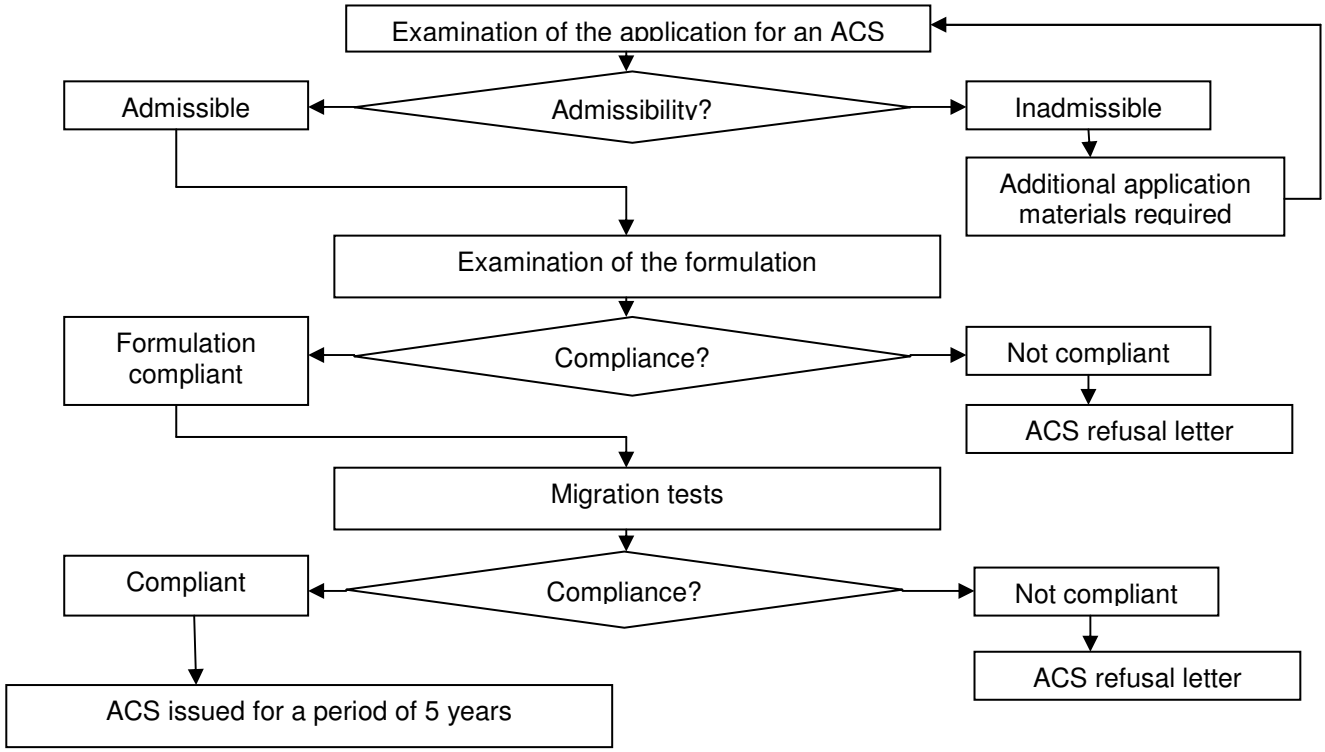

Figure 2: Decision tree for issuing an ACS

5 Recommendations for using lubricants

Lubricants used in pumps and turbines, as well as in valves, taps and meters, are likely to enter into direct contact with water and therefore must be used parsimoniously.

Regarding air compressors, it is recommended to use oil-free compressors or to expect effective retention system of lubricants.

Oakum impregnated, as packaged, are easily contaminated during operation and constitute a surface favourable to microbial growth in water production and distribution systems. Oakum can be advantageously replaced with Teflon tape that can be considered to be joints and come under the same regulations as joints, or with thread-locking fluid whose use falls under the guidelines regarding adhesives.

The lubricants used for easing the assembly of piping using sealing rings are only for temporary use; it is recommended to preferentially use water-soluble products that can be eliminated by rinsing after assembly has been completed.

6 Conclusions

Lubricants, certified or not for conformity with positive reference lists were tested using the present guidelines. The obtained results show:

1. the discriminating power of odour and flavour thresholds; measuring these parameters along with TOC can thus be used in the initial analyses of migration tests;
2. the usefulness of carrying out migration tests with both chlorinated and non-chlorinated water with regard to the differences in the results.

ANSES recommends that:

1. laboratories currently authorised for verifying the safety compliance of materials and products in contact with DW be accredited by the French Accreditation Committee (COFRAC), or by any other equivalent European accreditation body signatory to the multilateral agreement of the European Co-operation for Accreditation, to carry out migration tests using the protocol defined in the present guidelines and to carry out a GC-MS profile following the pr NF EN 15768 draft standard [24] in addition to the obligations defined in the Ministerial Order of 18 August 2009 [26];
2. each product should be subject to regular factory production audits by an independent organisation, as is the case in other countries;
3. a sampling plan for tests be carried out by an independent organisation, with spot checks of product samples in the factory.

Moreover, ANSES notes that the conditions for approval and/or certification of lubricants authorised to come into contact with DW in other EU Member States differ from the present guidelines, particularly in terms of the conditions for examining the formulation (PLs) and for conducting the migration test protocols (number of samples tested, surface/volume ratio used, sample preparation, parameters measured in migration water, methods for analysing these parameters, and interpretation of results including the PFC).

Given this situation, ANSES deems that, with regard to the potential recognition of tests and/or authorisations obtained in another EU Member State:

1. the authorisation procedures for lubricants to date in other countries cannot be considered to be equivalent, neither to each other nor to the guidelines proposed in this report;
2. nevertheless, the tests carried out in another EU Member State can be considered on a case-by-case basis in French laboratories authorised to issue ACSs; simplified migration tests, involving only parameters that have not been measured may be sufficient if the S/V ratio used is equal to or greater than that recommended in the present report and if the conditions of sample preparation are comparable.

Bibliographic references, standards and regulations

[1] Ministerial Order of 29 May 1997 relating to materials and articles used in permanent installations for the production, treatment and distribution of water intended for human consumption amended by Ministerial Orders of 24 June 24 1998, 13 January 2000, 22 August 2002 and 16 September 2004 (published in the Official Journal of 1 June 1997, 25 August 1998, 21 January 2000, 3 September 2002 and 23 October 2004, respectively).

[2] Ministerial Circulars no. 99/217 12 April 1999 and no. 2000/232 of 27 April 2000 relating to materials used in permanent facilities for the distribution of water intended for human consumption (published in the Official Bulletin of the Ministry for Health no. 99/25 and 2000/18, respectively).

[3] Ministerial Circular no. 2002/571 of 25 November 2002 relating to the procedures for verifying the safety compliance of accessories and subsets of accessories, consisting of at least one organic component that comes into contact with water intended for human consumption.

[4] Circular DGS/4/SD7A/2006/370 of 21 August 2006 relating to evidence of the safety compliance of fibre-reinforced organic materials and finished products that come into contact with water intended for human consumption, excluding natural mineral water.

[5] DGS Practical Guide to the compilation of dossiers relating to the safety compliance of materials that come into contact with the water supply (March 1999).

[6] Guideline for the Hygienic Assessment of Organic Materials in Contact with Drinking water (KTW guidelines) (http://umweltbundesamt.de/wasser-e/themen/drinking-water/pruefleitlinie.htm).

[7] Drinking Water Decree Article 4 – Regulation of 7 December 2002, no. BWL/2002095022.

[8] DWI Guideline: The approval scheme for products used in contact with water intended for human consumption (http://www.dwi.gov.uk/drinking-water-products/index.htm).

[9] Standard NF EN 12873-2 (June 2005) Influence of materials on water intended for human consumption – Influence due to migration – Part 2: Test method for non-metallic and non-cementitious site-applied materials.

[10] DWI Guidelines: The approval scheme for products used in contact with water intended for human consumption:
- Advice Sheet 1 – Overview of the application process and general requirements
- Advice Sheet 8 Regulation 31(4) (b) – The approval and use of products with a small surface area in contact with water (lubricants, solvent cements, adhesives…).
And
BS 6920 (2000): Suitability of non-metallic products for use in contact with water intended for human consumption with regard to their effect on the quality of the water.

[11] NSF/ANSI Standard 61 (2008): NSF International Standard / American National Standard / Drinking Water System Components – Health Effects.

[12] Regulation (EC) no. 1935/2004 of the European Parliament and of the Council of 27 October 2004 concerning materials and articles intended to come into contact with food and repealing Directives 80/590/EEC and 89/109/EEC.

[13] CNRS-CNERVA (1992) – *Les lubrifiants dans les industries agro-alimentaires : Guide de choix et d'utilisation* – TEC & DOC LAVOISIER.

[14] Standard NF EN ISO 21469 (July 2006) Safety of machines – Lubricants with incidental product contact – Hygiene requirements.

[15] French Standard NF P 41-211 - Reference DTU 60.31 (May 2007: Building works – Unplasticised polyvinyl chloride piping installations: cold water under pressure – Specifications.

[16] International Standard ISO 6743-99 (August 2002): Lubricants, industrial oils and related products (Class L) – Classification – Part 99: General.

[17] International Standard NF ISO 8681 (August 1987): Petroleum products and lubricants – Method of classification system – Definition of classes.

[18] French Standard NF ISO 3448 (December 1993): Industrial liquid lubricants – ISO Classification according to viscosity.

[19] French Standard NF ISO 2137 (March 2008): Petroleum products and lubricants – Determination of cone penetration of lubricating greases and petroleum jelly.

[20] AFSSA Opinion and Report no. 2006-SA-0291 (September 2007) relating to positive lists of substances included in the composition of materials in contact with water intended for human consumption.

[21] CARSO – Lyon Laboratory for Health, Environment & Hygiene (5 March 2007). Study on the chemical composition of glues, adhesives, greases and lubricants used in facilities for the production and distribution of water, coming into contact with water intended for human consumption. *(Study contains confidential information that is not available for consultation)*

[22] Standard NF EN 1484 (July 1997): Water analysis – Guidelines for the determination of total organic carbon (TOC) and dissolved organic carbon (DOC).

[23] Standard NF EN 1622 (October 2006): Water quality – Water quality – Determination of the threshold odour number (TON) and threshold flavour number (TFN).

[24] Draft Standard pr NF EN 15768: GC-MS identification of water-leachable organic substances from materials in contact with water intended for human consumption.

[25] Ministerial Order of 11 January 2007 relating to the quality limits and references for raw water and water intended for human consumption mentioned in Articles R. 1321-2, R. 1321-3, R. 1321-7 and R. 1321-38 of the French Public Health Code.

[26] Ministerial Order of 18 August 2009 relating to the conditions for authorising laboratories in application of Article R*. 1321-52 of the French Public Health Code.

[27] AFSSA: Opinion (9 April 2002) on the draft Ministerial Order on the colouring of plastic materials and articles, varnishes and coatings intended to come into contact with commodities, products and beverages for human and animal consumption.

[28] AFSSA: Report (May 2005): Threshold of toxicological concern for analysis of the health risk of chemical substances in foods.

[29] Rulis A.M. (1986): *De minimis* and the threshold of regulation. In: Food protection technology, current and projected technologies for food protection – Recommendations and Implementations. Felix CW (Ed), pp 329-37, Chelsea MI.

[30] Draft report from the Working Group of the Four Member States (4MS): France, United Kingdom, Germany, Netherlands (2010): Positive Lists for Organic Materials.

[31] CARSO – Lyon Laboratory for Health, Environment & Hygiene (12 May 2010). Migration tests on lubricants used in contact with water intended for human consumption. Protocol described in the ANSES draft guidelines for the health assessment of lubricants. Study funded by the Directorate General for Health.

Annex 1: Comparison of migration tests on lubricants conducted in other countries

Reference	Country	Type of sample	Water contact protocol	Parameters analysed	Criteria
NF EN 12873-1 NF EN 12873-2	Germany	Plates coated with a thickness of 0.2 to 5 dm^{-1} (from 20 to 500 cm^2/L)	At 23°C (static): - 60 min flushing with tap water - 24 h stagnation in test water - 50 min pre-wash with tap water - 3 x 72 h stagnation in test water => analysis of all 3 migration waters	- TOC - Odour/flavour	- Decreasing values. - After the 3rd migration: . TOC < 1.25 mg/(dm^2 day) (S/V = 5 dm^{-1}), . Odour/flavour threshold < 2 (S/V = 0.2 dm^{-1}) - The specific limits of substance migration must be met (in a migration test or by modelling)
BS EN 12873-2 and BS 6920	UK	-1 sample for each type of water - S/V ≥ 1.5 dm^{-1} - Drying conditions according to manufacturer's recommendations	At 23 °C (static): - 30 min flushing with tap water - Stagnation: o WRAS: 24, 24, 24, 72, 24, 24 & 24 h (BS6920-2), o Regulation 31: 3 x 72 h (EN 12873) -Test water: Non-chlorinated tap water and chlorinated tap water (1 mg/L) => analyses: o WRAS: migration waters from the 1st and the 7th stagnation o Regulation 31: migration waters from all 3 stagnation periods	<u>Products only used in building water systems:</u> - Odour/flavour - Enhancement of microbial growth (BS 6920-2.4 based on determination of dissolved oxygen consumption) - Colour and turbidity - metals - Cytotoxic substances <u>Products used by public water supplies (Regulation 31):</u> Simplified assessment for adhesives: - Odour/flavour - Enhancement of microbial growth	At final leachates: -odour and flavour free - <2.4 mg/L - <5 Hazen units, <0.5 FTU - <DWD limits - free from cytotoxicity -odour- and flavour-free - <2.4 mg/L
NSF61	USA	- Plates coated with 15 cm^2/L and 1 mm lubricant thickness - Drying according to manufacturer's recommendations or, alternatively 48 h	At 23°C (static): - Rinsing with tap water - 3 x 24 h stagnation in test water at pH 5, 8 and 10 => analyses on the 3rd period of migration water	The appropriate parameters to measure are determined on a case-by-case basis upon examination of the product's formulation: - Solvents - PAHs - GC-MS profile - etc.	*see* Annex D of the NSF/ANSI document [11] (the quantities detected must not exceed 10% of the water quality requirements set by the regulations)

Annex 2: Examples of lubricant components

(This annex is provided for information purposes and is based on the CNERVA guide [13] and the CARSO report [21].)

Lubricating base oils:

- Mineral (petroleum-based) base oils
  These base oils are complex mixtures of liquid hydrocarbon fractions obtained from the distillation of crude oil and subsequent application of various refining processes to decrease the concentration of aromatic hydrocarbons.
  Base oils for industrial lubricants are obtained from different hydrocarbon fractions after removing asphaltenes and certain aromatic and paraffinic compounds with high freezing points. They are composed of a liquid mixture of aromatic, paraffinic and naphthenic hydrocarbons.
  Oils used in the food, pharmaceutical and cosmetic industries are called white oils, vaseline oil, paraffin oil or liquid paraffin. They are obtained after extensive refinement and their concentration in aromatic hydrocarbons is very low.

- Synthetic lubricating base oils
  Petrochemical-based lubricants: poly-alpha olefins. These derivatives are obtained by polymerisation or co-polymerisation of *alpha*-olefins, ethylene, propylene, butane-1, octane-1, decene-1 and sometimes butane-2.
  Other lubricating base oils can be directly synthesised:
  - Alkylbenzenes, of which there are two types:
    - by-products from the manufacture of dodecylbenzene,
    - products obtained by a reaction of light alpha-olefins with benzene and its methylated homologues.
  - Oxygenated compounds:
    - esters,
    - polyalkylene glycols and derivatives.
  - Polyphenyl ethers: these products are mainly used as coolant fluids.
  - Fluorinated polyethers.
  - Polydimethylsiloxanes.

- Natural plant- or animal-based lubricating base oils

Lubricating base oils from animal sources: tallow oils composed mainly of triolein, lard oils composed of a mixture of triolein and stearin, suets composed of fatty acid glycerides, primarily stearic, oleic and palmitic acids.
Vegetable oils: These oils are mainly mixtures of oleic, linoleic and ricinoleic glycerides.
Derivatives of natural fatty substances: these oils are obtained by hydrogenation or polymerisation by air oxidation of thickening agents or by reaction with a strong base (metallic salts of fatty acids or soaps, bentonite, polytetrafluoroethylene, silicon dioxide, etc.) and additives (corrosion inhibitors, antioxidants, friction modifiers, anti-foaming agents, etc.).

Thickening agents
Fatty acid metal salts (soaps), bentonite, polytetrafluoroethylene (PTFE), silicon dioxide, graphite, etc.

Additives
Greases and lubricants have numerous additives. The main ones are:
- Gelling agents that are used to obtain grease from lubricating bases.
- Pour point depressants that enhance lubricant flow performance at low temperatures (high molecular mass polymethacrylates),
- Viscosity index improvers that decrease the magnitude of change in viscosity with temperature (polymers or co-polymers of methacrylates, olefins, diolefins with styrene and its alkylated derivatives),

- Anti-foaming agents that prevent the formation of persistent foam during use (organic polymers, organosilicone polymers),
- Antioxidants that inhibit oxidation reactions (phenols, amines, dithiophosphates or dithiocarbamates),
- Anti-wear and extreme pressure additives that reduce the friction coefficient and wear (molybdene bisulphide, graphite, polytetrafluoroethylene),
- Emulsifiers that promote the formation of emulsions with water. These can be:
  - anionic: alkylaryl sulphonates and alkyl sulphates of alkaline metals, alkali metal and amine carboxylates,
  - cationic: fatty amine acetates, hydrochlorides and hydroxides,
  - amphoteric: amino-acid salts,
  - non-ionic: condensates of ethylene oxides of organic substances with a free hydrogen (alcohols, acids, phenols, amines, sorbitan esters).
- Other additives may be included: stickiness improvers, friction modifiers, fungicides, bactericides, corrosion and rust inhibitors, passivating agents, dyes, fluorescent indicators, etc.

Annex 3: Classification of lubricants

General classification system, which applies to petroleum products, lubricants and related products (see Standards NF ISO 8681 [17] and ISO 6743-99[16])

The classification system is based on assigning a prefix letter that characterises the major classes of petroleum products:

Class	Designation
F	Fuels
S	Solvents and feedstock for the chemical industry
L	**Lubricants, industrial oils and related products**
W	Waxes and paraffins
B	Bitumens

In the L class, 18 product families have been defined, according to the type of application:

Product family letter code	Application
A	Total loss system
B	Mould release
C	Gears
D	Compressors
E	Oils for internal combustion engines
F	Spindle bearings, bearings, clutches
G	Slideways
H	Hydraulic systems
M	Metal working
N	Electrical insulation
P	Pneumatic tools
Q	Coolant fluids
R	Corrosion preventives
T	Turbines
U	Heat treatment
X	Greases
Y	Other applications
Z	Steam cylinders

Classes of viscosity for liquid lubricants (see the NF ISO 3448 Standard [18]),

Liquid lubricants can be classified according to their kinematic viscosity at 40°C. This classification defines 20 viscosity classes from 2 mm^2/s to 3200 mm^2/s at 40°C. Each kinematic viscosity class is designated by the closest whole number to the median kinematic viscosity value, expressed in mm^2/s, of the class at 40°C. The tolerated limits for each class correspond to the median value ± 10%.

ISO viscosity class	Median kinematic viscosity cSt at 40.0 °C
ISO VG 2	2.2
ISO VG 3	3.2
ISO VG 5	4.6
ISO VG 7	6.8
ISO VG 10	10
ISO VG 15	15
ISO VG 22	22
ISO VG 32	32
ISO VG 46	46
ISO VG 68	68
ISO VG 100	100
ISO VG 150	150
ISO VG 220	220
ISO VG 320	320
ISO VG 460	460
ISO VG 680	680
ISO VG 1 000	1 000
ISO VG 1 500	1 500
ISO VG 2 200	2 200
ISO VG 3 200	3 200

NLGI (National Lubricating Grease Institute) grease penetration numbers (see Standards ISO 6743-99 [16] and NF ISO 2137 [21]).

The NLGI classifies greases according to their consistency, which is characterised by the penetration of greases worked 60 cycles. The NLGI classification has nine grades or consistency indices, with each grade corresponding to a given interval of worked penetration:

NLGI class	Penetration of greases worked 60 cycles at 25°C (x 0.1 mm)
000	445 - 475
00	400 - 430
0	355 - 385
1	310 - 340
2	265 - 295
3	220 - 250
4	175 - 205
5	130 - 160
6	85 - 115

Annex 4: Positive lists

European reference lists for materials and articles intended to come into contact with food

Directives and Regulations:

- Directive 2002/72/EC of 6 August 2002 amended [Directives 2004/1/EC, 2004/19/EC, 2005/79/EC, 2007/19/EC and 2008/39/EC and Regulation (EC) no. 975/2009] on monomers, other starting substances and additives for plastic materials.
- Directive 78/142/EEC on vinyl chloride.
- Regulation (EC) no. 1895/2005 on epoxy derivatives (EGDAB/EGDFB/EGON).

Resolutions of the Council of Europe:

- Resolution AP (92) 2 on the control of aids to polymerisation that initiate and directly influence the formation of polymers, provided that the maximum quantities of starting substances used are less than 1% w/v.
- Resolution AP (2004) 1 on coatings (parts A and C).
- Resolution AP (2004) 3 on ion exchange resins (list 1).
- Resolution AP (2004) 4 on rubber products (substances classified from 0 to 4).
- Resolution AP (2004) 5 on silicones (list 1).

Substances evaluated by the European Food Safety Authority on substances found in food contact materials (substances classified from 0 to 4 by SCF/EFSA).

French reference lists for materials and articles intended to come into contact with food

Texts transposing the Directives:

- Ministerial Order of 2 January 2003 (monomers and additives), as amended (by the Ministerial Orders of 29 March 2005, 9 August 2005, 19 October 2006, 25 April 2008 and 19 November 2008) referring to the Ministerial Decree of 30 January 1984 (vinyl chloride) [transposition of Directive 2002/72/EC as amended]
- Ministerial Order of 30 January 1984 (vinyl chloride) [transposition of Directive 78/142/EEC].

Other Ministerial Decrees and Circulars:

- Ministerial Order of 25 November 1992 on silicones.
- Ministerial Order of 9 November 1994 as amended (by Ministerial Orders of 9 August 2005 and 19 October 2006) on elastomers and rubbers [Resolution AP (2004) 4 on rubber products (substances classified from 0 to 4)].
- Ministerial Order of 4 November 1993 on celluloses.
- Draft Ministerial Order on the colouring of plastic materials and objects, varnishes and coatings intended to come into contact with commodities, products and beverages for human and animal consumption, notified to the European Commission under the reference no. 2004/328/F[21] following AFSSA's Opinion[22] of 9 April 2002 (Solicited request no. 2001-SA-0069 [27]).
- DGCCRF memorandum no. 2003-27 of 24 March 2003 on additives in plastic materials (since 1950).

Substances authorised for materials and articles intended to come into contact with water intended for human consumption (favourable AFSSA opinions)

- **(Solicited Request no. 2002-SA-0095)** Diethylmethylbenzenediamine (CAS: 68479-98-1).
- **(Solicited Request no. 2003-SA-0275)** 5-vinylnorborn-2-ene (CAS: 3048-64-4).
- **(Solicited Request no. 2004-SA-0373)** 2-phenyl-2-imidazoline (CAS: 936-49-2).
- **(Solicited Request no. 2006-SA-0288)** 2-octyl-2H-isothiazole-3-one (CAS: 26530-20-1).

[21] *http://ec.europa.eu/enterprise/tris/index_en.htm*

[22] *Opinion available on the ANSES website: www.anses.fr*

Annex 5: Justification of the tolerated weight percentages of non-compliance in the formulation

The migration of a substance found in a material into the water with which it is in contact can be assessed by calculation following the rules defined in the DGS Guide of March 1999 [5]:

1 - Calculation method:

The amount of substance that can migrate corresponds to the "wettable" part of the material, which will depend on the material's chemical nature.

So if

- **t** (in metres) is the thickness of the "wettable" material,
- **S** (in m^2) is the surface area of the material,
- **d** (in kg/m^3) is the density of the material,
- **p** (in % m/m) is the weight percentage of the substance in the material,

then the mass **m** (in kg) of this substance likely to migrate into the water is

$$\mathbf{m = \frac{S \times t \times d \times p}{100}}$$

- If the whole substance migrated all at once, its concentration in the water would be

$$\mathbf{C1\ (kg/m^3) = \frac{S \times t \times d \times p}{100 \times V}} \quad \text{or} \quad \mathbf{C1\ (\mu g/L) = S/V \times t \times d \times p \times 10^4}$$

where **V** is the volume (in m^3) of water in contact with the material.

- If there was no mass migration of the substance, and migration occurred gradually over 100 days, which is more realistic, its concentration in the water would be

$$\mathbf{C2\ (\mu g/L) = S/V \times t \times d \times p \times 10^2.}$$

Therefore the weight percentage of a substance in a material corresponding to a concentration C2 in a given water would be

$$\mathbf{p\ (\%) = \frac{C2 \times 10^{-2}}{t \times d \times S/V}}$$

This equation is used to calculate the maximum weight percentage of a substance (p) in a lubricant in order that its migration (C2) be lower than the value adopted for the PFC.

2 - Calculation applied to lubricants:

AFSSA's report of May 2005 [30] set a threshold of toxicological concern (TTC) of **1.5 µg per person and per day**. This threshold was established by considering that one-third of the daily intake comes from solid food (0.5 µg per person and per day) and two-thirds from beverages (1 µg per person and per day) [31]. Therefore, for a daily consumption of 2 L of water, the TTC corresponds to a maximum value in DW of **0.5 µg/L**.

The ongoing work of the group of four Member States (4MS: Germany, the Netherlands, the United Kingdom and France) proposes conversion factors (CFs) to determine the real impact of materials on the quality of DW with regard to the concentrations found in migration tests. Accordingly, the surface/volume ratio and the stagnation times used for migration tests according to the NF EN 12873-2 standard do not reflect the reality of a water distribution system [30].

The CFs are established under the following assumption:

$$FC = F_g \times F_o$$

where

- F_g is the realistic S/V ratio,
- F_o is the assumed residence time of the water in the system:
  - 4 days in the main public distribution systems (Ø ≥ 300 mm),
  - 2 days in the secondary public distribution systems (80 mm ≤ Ø < 300 mm),
  - 0.5 days in plumbing systems (Ø < 80 mm).

Based on the above assumptions, a conversion factor was defined for lubricants. With regard to the possible uses of lubricants, easing pipe assembly in plumbing systems inside buildings was used as the worst case on a linear metre of piping where there are three connectors and thus six lubrication points; the real S/V ratio was estimated at 14 cm^2/L and the residence time at 0.5 days.

Let

	S/V (cm^2/L)	**Residence time (day)**
Real case	14	0.5
Migration test	20	3

A conversion factor (CF) makes it possible to convert from a real case to a migration test as defined in this report and corresponds to **CF = 14 x 0.5 / 20 x 3 = 0.1.**

$$\mathbf{C2_{(in\ test\ water)} = C2_{(tap\ water)} / 0.1}$$

thus a $C2_{(tap\ water)}$ of 0.5 µg/L corresponds to a $C2_{(in\ test\ water)}$ of 5 µg/L

The following data and assumptions were used to apply the calculation mentioned in Section 1:

	GREASE	**OIL**
Density of the lubricant (d)[23]	900 kg/m^3	900 kg/m^3
Thickness of the applied lubricant	0.001 m	0.0005 m
Thickness of the associated wetted lubricant (10%[24]) (t)	0.0001 m	0.00005 m
Surface/volume ratio (S/V)	2 m^{-1}	2 m^{-1}
$C2 = C2_{(in\ test\ water)}$	5 µg/L	5 µg/L

=> Maximum weight percentage of a substance in a **grease** so that less than 5 µg/L (p) would migrate into DW: **0.28%**

=> Maximum weight percentage of a substance in an **oil** so that less than 5 µg/L (p) would migrate into DW: **0.55%**

Thus, one or more substances not included on the positive reference lists that are used in the formulation whose total content is less than or equal to 0.5% in weight are tolerated as they are not expected to migrate beyond the adopted PFC.

[23] *Weight commonly observed in lubricants' technical notices.*

[24] *The basic assumption is that the thickness of the lubricant involved in the exchange with water cannot exceed 10% with regard to migration values obtained on TOC in the CARSO study [23].*

Annex 6: Results of migration tests

Four lubricants were tested following the protocol based on the NF EN 12873-2 standard defined in Section 3.2 [31]:

- product A: a silicone-based grease issued with a certificate of compliance with positive reference lists (CPL)
- product B: a hydrocarbon-based grease that has not been issued with a CPL certificate,
- product C: a silicone-based oil issued with a CPL certificate,
- product D: a synthetic oil (1-decene homopolymer) issued with a CPL certificate.

The four products were purchased in a store that supplies water production and distribution professionals (valve manufacturers, plumbers, etc.).

Measure of total organic carbon (TOC):

Table II: Results on TOC (NF EN 1484)

Product	b^t_1 (mg/L of C)	a^t_1 (mg/L of C)	M^t_1 (mg/dm^2/ day of C)	b^t_2 (mg/L of C)	a^t_2 (mg/L of C)	M^t_2 (mg/ dm^2/day of C)	b^t_3 (mg/L of C)	a^t_3 (mg/L of C)	M^t_3 (mg/dm^2/ day of C)
A (0 mg/L)[25]	0.2	1.6	2.3	< 0.2	0.4	0.5	< 0.2	0.4	0.5
A (1 mg/L)[26]	0.2	1.0	1.3	< 0.2	0.6	0.8	< 0.2	0.4	0.5
B (0 mg/L)	0.2	1.0	1.3	< 0.2	0.6	0.8	< 0.2	0.8	1.1
B (1 mg/L)	0.2	1.6	2.3	< 0.2	0.8	1.1	< 0.2	0.6	0.8
C (0 mg/L)	0.2	0.8	1.0	< 0.2	0.4	0.5	< 0.2	0.4	0.5
C (1 mg/L)	0.2	1.6	2.3	< 0.2	1.0	1.5	< 0.2	0.4	0.5
D (0 mg/L)	0.2	0.4	0.3	< 0.2	0.4	0.5	< 0.2	0.4	0.5
D (1 mg/L)	0.2	0.4	0.3	< 0.2	0.4	0.5	< 0.2	0.4	0.5

Non-negligible differences in the results were occasionally observed in the two test waters (non-chlorinated water and chlorinated water). For all of the products, the PFC set at 2 mg/L C (corresponding to 3.5 mg/dm^2/day) was met in test waters from the 3rd stagnation period.

Measurements of odour and flavour:

Measurements were performed by the same four tasters to obtain the most consistent results possible. Taste tests were carried out with the demineralised water used in the migration tests as the reference water.

[25] *(0 mg/L) Tests carried out with non-chlorinated water.*

[26] *(1 mg/L) Tests carried out using chlorinated water with an active chlorine content of (1 ± 0,2) mg/L in the form of Cl_2.*

Table III: Results on flavour (NF EN 1622)

Product	b^t_1	a^t_1 (threshold)	M^t_1	b^t_2	a^t_2 (threshold)	M^t_2	b^t_3	a^t_3 (threshold)	M^t_3
A (0 mg/L)	1	2 (Plastic)	/	1	1	/	1	1	/
A (1 mg/L)	1	2 (Plastic)	/	1	1	/	1	1	/
B (0 mg/L)	1	5 (Hydrocarbon)	/	1	5 (Hydrocarbon)	/	1	5 (Hydrocarbon)	/
B (1 mg/L)	1	7 (Hydrocarbon)	/	1	7 (Hydrocarbon)	/	1	7 (Hydrocarbon)	/
C (0 mg/L)	1	3 (Plastic)	/	1	3 (Plastic)	/	1	3 (Plastic)	/
C (1 mg/L)	1	2 (Plastic)	/	1	2 (Plastic)	/	1	1	/
D (0 mg/L)	1	3 (Hydrocarbon)	/	1	3 (Hydrocarbon)	/	1	3 (Hydrocarbon)	/
D (1 mg/L)	1	2 (Hydrocarbon)	/	1	2 (Hydrocarbon)	/	1	2 (Hydrocarbon)	/

Table IV: Results on odour (NF EN 1622)

Product	b^t_1	a^t_1 (threshold)	M^t_1	b^t_2	a^t_2 (threshold)	M^t_2	b^t_3	a^t_3 threshold	M^t_3
A (0 mg/L)	1	2 (Plastic)	/	1	1	/	1	1	/
A (1 mg/L)	1	2 (Plastic)	/	1	1	/	1	1	/
B (0 mg/L)	1	5 (Hydrocarbon)	/	1	5 (Hydrocarbon)	/	1	5 (Hydrocarbon)	/
B (1 mg/L)	1	5 (Hydrocarbon)	/	1	5 (Hydrocarbon)	/	1	5 (Hydrocarbon)	/
C (0 mg/L)	1	2 (Plastic)	/	1	2 (Plastic)	/	1	2 (Plastic)	/
C (1 mg/L)	1	2 (Plastic)	/	1	2 (Plastic)	/	1	1	/
D (0 mg/L)	1	2 (Hydrocarbon)	/	1	2 (Hydrocarbon)	/	1	2 (Hydrocarbon)	/
D (1 mg/L)	1	2 (Hydrocarbon)	/	1	2 (Hydrocarbon)	/	1	2 (Hydrocarbon)	/

Product B did not meet the adopted PFC (threshold < 3) for neither odour nor flavour after the 3rd stagnation period for neither chlorinated nor non-chlorinated water. Products C and D did not meet the adopted PFC (threshold < 3) for flavour after the 3rd stagnation period for non-chlorinated water.

Results for the GC-MS profile:

Table V: Results regarding the GC-MS profile (with respect to NF EN 16768)

Product	b^t_1 (µg/L)	a^t_1 (µg/L)	M^t_1 (µg/dm²/j)	b^t_2 (µg/L)	a^t_2 (µg/L)	M^t_2 (µg/dm²/day)	b^t_3 (µg/L)	a^t_3 (µg/L)	M^t_3 (µg/dm²/day)
A (0 mg/L)	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
A (1 mg/L)	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
B (0 mg/L)	N.D.	Diphenylamine*: 580 R.T. (min)***: 33.70: N.D.	966 N.D.	N.D.	434 4.2	723 7	N.D.	308 3.8	513 6.3
B (1 mg/L)	N.D.	Diphenylamine*: 450 R.T. (min)*** 25.11: 9.1 3-chlorodiphenylamine**: 17.6 Benzamine,2,6-dichloro-N-phenyl**: 6.5 R.T. (min)*** 28.25: 5.3 R.T. (min)*** 28.98: 1.6 R.T. (min)***: 29.57: N.D. R.T. (min)***: 33.70: N.D.	750 15 29 11 9 3 N.D. N.D.	N.D.	341 4.5 10.3 10.1 5.9 1.8 11.0 3.1	568 7.5 17.1 16.8 9.8 3.0 18.3 5.2	N.D.	295 1.9 7.8 13.9 6.7 5.9 12.7 3.6	491 3.1 13 23.1 11.1 9.8 21.1 6
C (0 mg/L)	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
C (1 mg/L)	N.D.	R.T. (min)*** 29.35: 11.4 R.T. (min)*** 30.01: 3.4	19 5.7	N.D.	8.2 2.1	13.7 3.5	N.D.	6.3 1.3	10.5 2.2
D (0 mg/L)	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
D (1 mg/L)	N.D.	R.T. (min)*** 19.44: 18 R.T. (min)*** 19.65: 10.8	30 18	N.D.	14.1 6.9	23.5 11.5	N.D.	10.8 5.2	18 8.7

**Compound identified and quantified with respect to its calibration standard.*
*** Compound identified and partially quantified with respect to the alkane having the closest retention time.*
**** Compound not identified and partially quantified with respect to the alkane having the closest retention time.*

Non-negligible differences in the results were occasionally observed in both test waters (chlorinated and non-chlorinated water). For three of the four products (B, C and D), the PFC (5 µg/L corresponding to 8.3 µg/dm²/day) was not met after the 3rd stagnation period.

Annex 7: Documents required in the application dossier for an attestation of sanitary conformity (ACS)

The application dossier must include the following documents:

1. Name and address of the applicant (permanent address in the European Community);
2. Name and address of the manufacturer of the lubricant, if different from the applicant;
3. Name and, if applicable, the trade names of the lubricant;
4. The type of lubricant for which the application is being made;
5. The safety data sheet and the technical instructions for using the product;
6. A specimen of the proposed label;
7. The formulation (composition and constituents) of the product:
    a. the chemical names of the substances used in the product formulation [chemical name specified in Annex 1 of Directive 65/548/EEC or according to the nomenclature of the IUPAC (International Union of Pure and Applied Chemistry) and CAS (Chemical Abstracts Service)],
    b. the CAS number and EC number (EINECS or ELINCS) of the substances,
    c. concentrations of the substances;
8. If the formulation uses one or more mixtures (or commercial products), the following must be specified for each of them:
    a. the exact trade name,
    b. the contact information of the supplier (address, telephone, contact person);
9. In the event that the product has already received authorisation in an EU Member State or in a Member State which is a contracting party to the Agreement establishing the European Economic Area:
    a. a copy of these authorisations,
    b. translation in French or in English of the dossier submitted in the Member State that granted authorisation, including the full migration test report, when available. This report must include:
        1. the number of samples tested,
        2. the S/V ratio that was used,
        3. how samples were prepared,
        4. the parameters screened for in migration waters and their analysis methods,
        5. the results of analyses and PFC;
    c. The references of the scientific organisation(s) that carried out the analyses and/or the tests along with certificates of its (their) technical competence (as a minimum, accreditation for the parameters measured).