anses
French agency for food, environmental and occupational health & safety

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

(Solicited request no. 2007-SA-0086)
August 2010

Composition of the working group

Chairman:

Mr Yves LÉVI
Faculty of Pharmacy – University Paris Sud 11
Expert Committee (CES) on Water

Members:

Ms Christelle AUTUGELLE
CARSO – Lyon Laboratory for Health, Environment & Hygiene (LSEHL)

Mr Paul CHAMBON
CES on Water

Ms Sophie HÉRAULT
Ile de France Regional Health Agency – Yvelines Territorial Delegation
CES on Water

Mr Michel JOYEUX
Eau de Paris
CES on Water

Mr Georges MAYDATCHEVSKY
CES on Water

Mr Antoine MONTIEL
CES on Water

Ms Evelyne TRINCKQUEL
Centre for Research, Expertise and Inspection of Water in Paris (CRECEP)

French Agency for Food, Environmental and Occupational Health Safety - Food Health Directorate:

Ms Anne NOVELLI
Water risk assessment unit
Coordinator of the Working Group

People interviewed by the Working Group:

Members of the French association of glue, adhesive and sealant manufacturers (AFICAM):
Ms Jacqueline CARRARD (BOSTIK SA), Mr Thierry DELATTRE (GEB SAS) and Mr Robert RODES (HENKEL France SA).

Introduction

Water intended for human consumption (Drinking water, DW) is essential to life and health. As such, it must be distributed to the population continuously, 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 adhesives, 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].

There are several methods of joining materials together: autogenous welding, soldering, riveting, mechanical assembly and gluing. Sometimes the leak tightness of these assembly methods has to be ensured by using jointing materials, which include:

- joints, which as organic materials, must comply with Annex 3 of the Ministerial Order of 29 May 1997 as amended [1],
- lubricants, some of which are used as sealing and assembly aids. The guidelines for assessing their safety are the subject of a separate ANSES report (Solicited Request No. 2007-SA-0096),
- polytetrafluoroethylene (PTFE) tapes, which are similar to joints and which must comply with Annex 3 of the Ministerial Order of 29 May 1997 as amended [1],
- adhesives.

[1] *N.B. The term "adhesives" also includes glues, and that the term "Glues and Adhesives" used in the current regulations is inappropriate.*

The purpose of this report is to specify **the conditions for obtaining an attestation of sanitary conformity (ACS) for adhesives.** 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 application dossiers by the authorised laboratories.

The report specifies:

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

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

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 the regulations in force. It is issued by a laboratory authorised by the French Minister of Health under Article R*. 1321-52 of the French Public Health Code.

Adhesive[2]: non-metallic substance capable of joining materials by surface gluing (adhesion), with the joint having adequate internal resistance (cohesion).

Adhesive tape[1]: flexible support or backing coated with an adhesive.

Autogenous welding: welding two pieces of metal of the same type through the fusion of these pieces.

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.

Bond[1]: assembly of parts using an adhesive.

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.

Crimping: mechanical assembly which consists in folding two sheets so they are inserted into each other, the edges are then folded one upon another to form a tight seal.

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

Glue[1]: aqueous adhesive specifically designed for bonding wood and other porous substrates.

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

Leak tightness: effectiveness of a barrier between two media.

Materials:

- organic and/or mineral 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 the constituent material (metal, inorganic, organic, etc.). They can be factory-made or applied *in situ,* particularly for manufacture, repair or reconditioning. Adhesives constitute a category of materials.

Migration: the process of transfer of substances from the sample being tested into the test water.

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

Mixture[3]: mixture (or solution) consisting of two or more substances.

---

[2] Definition of French Standard NF EN 923 + A1 (May 2008): Adhesives - terms and definitions.

[3] 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.

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

Riveting: permanent joining of parts using rivets.

Soldering: assembly of metal parts using a molten filler metal which is different to the parts to be assembled.

Structural adhesive[1]: adhesive forming joints capable of bearing, in a structure, a specified load for a long given period.

Substance[3]: chemical element and its compounds, in the 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 placed in contact with the 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.

Adhesives 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 into contact with water intended for human consumption

In accordance with the provisions of the French Consumer Code, anyone responsible for 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: "*All advertising, in any form whatsoever, 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 the market is, therefore, obliged to verify that the latter conforms to current requirements […].*"

In accordance with the provisions of Article R. 1321-48 of the CSP, "*materials and products marketed and intended for production, distribution and packaging facilities 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 of 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, the specific provisions to be respected for different groups of materials and products[4] coming into contact with DW are defined in:

- the Ministerial Order of 29 May 1997, as amended [1] relating to 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 25 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 partially renovated installations, from the point of water intake to the points of compliance defined in Article R. 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) concerning natural mineral waters:

[4] *To date, the following groups of materials and products can be distinguished:*
- *metals, alloys and metallic coatings,*
- *cementitious materials (concrete, mortar), enamels, ceramics and glass,*
- *organic materials and products (plastic, bitumen, rubber and elastomer), including fibre-reinforced,*
- *accessories and subsets of accessories, consisting of at least one organic material.*

- Article R. 1322-31 of the CSP 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 the natural mineral water that are compatible with its composition so as to prevent any chemical, physico-chemical, microbiological or organoleptic alteration to the quality of the water as found upon emergence.*";
- Article R. 1322-36 of the CSP 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 articles 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) concerning spring water and packaged water made drinkable through treatment:
Article R. 1321-95 of the CSP states that: "*The materials used for packaging must satisfy the conditions laid down in Article R. 1322-36.*"

1.1.3 Specific provisions relating to adhesives designed to be used in 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 migration levels in water of solvents contained 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 this 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 now verify their compliance with the 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 the greases and lubricants used in an accessory complies with the positive lists, then the constituent solvents of these greases and lubricants will not be screened for in the migration water, under the procedure for issuing the ACS for the accessory considered,*
- *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 adhesives which have been issued with a CLP 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 theme/"e"/eau/eau du robinet/matériaux entrant au contact de l'eau).

1.1.4 Specific provisions relating to adhesives designed to be used in contact with water intended for human consumption in other countries

Adhesives 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] Netherlands [7], the United Kingdom [8]) and the United States).

In Germany, adhesives must comply with the KTW[5] guidelines of the Federal Environment Agency (UBA[6]). A product's compliance is verified by an approved inspection body, such as the Water Technology Center (TZW[7]) which is recognised by a certifying body, the German Technical and Scientific Association for Gas and Water (DVGW[8]), and a certificate is issued. The guidelines for organic materials (http://umweltbundesamt.de/wasser-e/themen/drinking-water/pruefleitlinie.htm) are used as well as those for organic coatings (http://umweltbundesamt.de/wasser-e/themen/drinking-water/beschichtungsleitlinie.htm), for polyurethane and epoxy adhesives. The formulation's compliance with the German PLs is examined, and migration tests are conducted whose protocols are based on the DIN EN 12873-2 standard [6][9][10] (see Annex 1 for more information on migration tests and PFC).

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, there are requirements on the formulation: prohibition of lead and bituminous substances derived from tar, use of food colourings if possible, etc. Moreover, it is mandatory to conduct migration tests in accordance with the BS EN 12873-2 and BS 6920 standards [11] *(*see Annex 1 for more details on migration tests and PFC). A list of materials and articles authorised in public and private drinking water supplies is available on the website of the DWI (www.dwi.gov.uk/drinking-water-products/approved-products/soslistcurrent.pdf, see Section C.6 "Sealant & Repair materials") and a list of those authorised solely within buildings can be found on the website of the WRAS (www.wras.co.uk/Directory/materials_Search.asp?, see the sections entitled "Solvent Cements" and "Sealants").

In the Netherlands, only adhesives with a KIWA-ATA[9] certificate are authorised and recognised as compliant with the regulations in force by the Dutch Ministry of Housing, Spatial Planning and the Environment (VROM). They are authorised both for their mechanical properties and their safety, and in the case of the latter, without prior migration testing, with only an examination of the formulation's compliance with Dutch PLs being conducted. A list of adhesives authorised by 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 by consulting the following section "Productgroep: Lijmen".

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

In the United States, adhesives are assessed with regard to the NSF/ANSI Standard 61 [12]. The United States has not established PLs but does have requirements on the formulation: for example, lead cannot intentionally be used as a substance. Furthermore, conducting migration tests is mandatory *(*see Annex 1 for more information on migration tests and PFC).

1.1.5 Adhesives used in food packaging

The mandatory certificates of Compliance with Positive Lists (CPL) for adhesives used in permanent installations for the production and distribution of water intended for human consumption must not be confused with the mandatory "food-grade certificates" for adhesives used in food packaging[10].

[5] *Kunststoffe und Trinkwasser* [Plastics and drinking water].

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

[7] *Technologiezentrum Wasser.*

[8] *Deutsche Vereinigung des Gas- und Wasserfaches.*

[9] *KIWA Attestation of Toxicological Aspects.*

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

The regulations relating to materials and articles intended to come into contact with food do not provide specific provisions for adhesives. However, the inertia principle defined in Article 3 of Regulation (EC) No 1935/2004 [13] applies:
"*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) that a written declaration shall be provided stating that the products comply with the rules applicable to them and that appropriate documentation shall be available to demonstrate such compliance.

In general, the "food grade certificate" indicates that the product contains only substances permitted under Title 21 of the Code of Federal Regulations (CFR) of the United States, Part 175-105 on adhesives in indirect contact with food[11].

In addition, there is a European research project (2007-2010) "MIGRESIVES" (Migration From Adhesives in Food Packaging Material in Support of European Legislation and Standardisation) whose objective is to assess the risk of migration of adhesives in food, when used in packaging[12].

1.2 Technical Documents

It should be noted that while these guidelines cover only the safety of adhesives, the technical quality of products and the compliance with professional practices for gluing and/or assembly of pipes and connectors are equally important. General rules for the use of these products in construction are defined in unified technical documents (DTUs)[13].

Thus, the DTUs from Series 60 on "sanitary plumbing" and more specifically DTU 60.31 (NF P 41-211 [14]) must be complied with.

Certain solvent-based adhesives used for gluing pipes and connectors made of polyvinyl chloride (PVC) and chlorinated PVC (CPVC) are issued with technical opinions (ATECs)[14] when their production has been CSTBat certified. Obtaining a CPL certificate issued by one of the laboratories accredited by the French Ministry of Health is a mandatory criterion for the issuing of an ATEC for an adhesive intended to be used in drinking water supply and distribution systems. The symbol "*Convient pour l'eau potable*" (Suitable for drinking water) can be affixed to the adhesive's packaging. The ATECs for these adhesives then recommend a drying time of 24 hours, and 30 minutes of pressure rinsing (10 bars or 1.5 times the prescribed value if this is greater than 10 bars).

Currently, no adhesive obtained by curing a prepolymer can be issued with a technical opinion.

---

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

[12] *www.migresives.eu/project_summary.php*

[13] *DTUs are documents applicable to contracts for construction work in France. They are produced by the General Committee for Building Standards/DTU whose Scientific and Technical Building Centre (CSTB) provides the secretariat. They concern 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. 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 the DTUs are therefore standards that can be made mandatory through regulations.*

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

1.3 Opinion of AFSSA

AFSSA has issued two opinions[15] relating to the risks associated with solvent-based adhesives used to assemble pipes and connectors made of polyvinyl chloride (PVC) and chlorinated PVC (CPVC):

- Opinion no. 2001-SA-0092 relating to the request for an opinion on the relevance of the study of leaching into water of organic solvents found in plastic pipes assembled by gluing,
- Opinion no. 2002-SA-0225 on criticism expressed about comments relating to materials in hot water pipes included in Sheet 1 appended to the Circular of 22 April 2002 on the prevention of risks associated with Legionella in healthcare establishments.

These opinions:

- highlight the leaching into the water of solvents from the assembly that may continue for several weeks after gluing,
- draw attention to the risk of formation of trihalomethanes (THMs) through interaction with residual chlorine in the circulating water,
- recommend undertaking a search for alternative means of assembling PVC and CPVC pipes and connectors.

[15] *Opinions available on the ANSES website: www.anses.fr*

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

Adhesives can be classified according to their composition, properties, performance, specific application, physical state and application process [15].

These guidelines distinguish between the following two categories:

- solvent-based adhesives for pipes and connectors made of polyvinyl chloride (PVC) and chlorinated PVC (CPVC),
- adhesives obtained by curing a prepolymer, subsequently referred to as adhesives obtained by polymerisation.

2.1 Solvent-based adhesives

They are used to glue and seal the cylindrical interlocking male and female PVC pipes and connectors used exclusively for the distribution of cold water, and those made of CPVC used for the distribution of hot or cold water. They are pressure-resistant.

These products contain solvents (70-80%), additives (0-5%) and PVC (15-25%). The solvents partially dissolve the parts to be assembled and the PVC fills the spaces created. The joint is typically about 200 µm thick.

The main solvents used are: cyclohexanone (CAS No. 108-94-1), tetrahydrofuran, referred to simply as THF (CAS No. 109-99-9) and methyl ketones such as methyl ethyl ketone, referred to simply as MEK (CAS No. 78-93-3). THF and MEK are on the PLs, but not cyclohexanone.

The solvents evaporate during application and drying, are adsorbed along the inner surface of the pipes, and then spread within the mass of PVC (or CPVC). They can subsequently be leached from a surface in contact with water, whose area will then be greater than that at the initial point of gluing.

2.2 Adhesives obtained by polymerisation

Hardened by a chemical reaction, they are used to seal two threaded unions or to lock threaded elements (bolt, pin, nut, etc.). They are suitable for metal unions, as well as the sealing of other materials such as plastics, glass, ceramics, elastomers, etc.

There are several types of products depending on the chemical nature of the original monomer(s) or prepolymer(s) and their polymerisation methods. Polymerisation can be initiated by:

- anaerobic reaction: in this case, polymerisation is activated when the product is placed in a assembly isolated from the oxygen in the air, and takes place after the existing oxygen in the air has been consumed (anaerobic reaction) and in the presence of metals that catalyse the reaction,
- ultraviolet radiation (UV),
- heat,
- ambient humidity.

In addition, these products usually contain an activator and polymerisation catalysts.

2.2.1 Acrylates and cyanoacrylates

These are single-component, solvent-free products. Cyanoacrylates harden in seconds to form a thermoplastic polymer.

2.2.2 Silicones

These are single-component products containing polymethylsiloxane diluted in silanes. Polymerisation takes place rapidly, sheathing the connector. The joint remains flexible but the silicone's cohesion provides a seal under pressure of 10 to 20 bars.

2.2.3 Epoxies

These are generally two-component adhesives used when there are large spaces between the parts to be assembled. The bond is cured and is generally harder than the material to be glued. Polymerisation occurs rapidly when the two components (base and hardener) are mixed.

2.2.4 Polyurethanes

Prepolymers result from condensation between an isocyanate monomer and a hydroxide or polyol monomer. They come in the form of two components (prepolymer and activator), although technological change is tending to favour the development of single-component polyurethane adhesives because of their ease of use. The bond is cured and is generally harder than the material to be glued. Polymerisation is normally rapid.

The molecules likely to be released by the adhesives may cause a degradation in the water quality (organoleptic, physico-chemical), or induce microbiological and/or toxicological risks. It is therefore necessary to assess their safety.

3 Safety assessment of adhesives

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

- compliance of formulations with regard to the PLs,
- migration tests,
- common PLs and test protocols. With regard to the tests, the European Commission requested the European Committee for Standardization (CEN) to develop testing standards in the EN 12873 series.

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

- examination of the formulation of the adhesive,
- then conducting of migration tests.

The results of these assessments must then be compared with the PFC.

3.1 Examination of the formulation of the adhesive

The substances used in the formulation of adhesives must be identified and compared to the substances on PLs recommended in AFSSA's Report and Opinion of 14 September 2007[16] (solicited request no. 2006-SA-0291 [16]) relating to the positive lists of substances included in the composition of materials in contact with DWC *(*see Annex 2 which summarises the lists and the substances that can be used).

3.2 Migration tests

The protocol based on the **NF EN 12873-2** standard [10] must be implemented, along with the following additional provisions:

- The water contact test shall be performed in triplicate using three independent and parallel set-ups and three independent and parallel control pipes[17];

Preparation of the set-up

- eleven pipe pieces with ten fittings (double–sockets) shall be glued with the adhesive according to the instructions of the pipe/fittings and adhesives manufacturers, to obtain a test sample with a total length of one metre:
  - for solvent-based adhesives:
    - pipes and fittings must be PVC or CPVC and have an ACS,
    - pipes must be standardised and have a nominal external diameter of 32 (DN32)[18] and a nominal pressure of 16 (PN16)[19],
    - fittings must be standardised and have a nominal internal diameter of 32 (DN32)[20], and must include an internal stop.

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

[17] *The number of duplicates may be increased depending on the amount of water needed by the laboratories to perform the analyses on the migration water.*

[18] *External diameter around 32 mm.*

[19] *Operating pressure limit around 16 bars.*

[20] *Internal diameter around 32 mm.*

- for adhesives obtained by polymerisation:
  - pipes and fittings must comply with Annex 1 of the Ministerial Order of 29 May 1997, as amended,
  - standardised, galvanised steel pipes must have a nominal external diameter of 26.9 (DN26.9)[21] and be light-duty,
  - standardised, galvanised malleable iron fittings must have a thread at each end and a diameter of ¾ inch[22].

- the amount of adhesive used to produce the entire assembly must be determined by weighing, based on the difference in the package weight before and after use. Surplus adhesive on the outside of the pipe must be wiped off and weighed. The actual amount of adhesive used for the assembly is obtained by subtracting the amount of adhesive wiped off from the amount used;

- drying (or curing) and/or rinsing time, after assembly, shall be that recommended by the manufacturer. Failing that, a drying (or curing) time of 24 hours, and 30 minutes of rinsing shall be observed;

Performance of tests

- rinsing shall be carried out with tap water whose levels of free chlorine are less than 0.2 mg/L Cl_2, by upward flow with evacuation via the overflow system;

- tests shall be carried out both with non-chlorinated test water, and with chlorinated test water whose levels of free chlorine are equal to (1 ± 0.2) mg/L in the form of Cl_2 at a temperature of (23 ± 2)°C as specified in Section 9.1 of the NF EN 12873-2 standard.

At the end of each of the three 72h periods of stagnation[23], the migration waters from the three tests conducted at temperature T are retrieved and mixed for analysis, as well as the three controls *(*see Figure 1):

- analyses a^T_1 are performed on the mixture of the three migration waters from the tests resulting from the 1st stagnation of (72 ± 1) hours,
- analyses a^T_2 are performed on the mixture of the three migration waters from the tests resulting from the 2nd stagnation of (72 ± 1) hours,
- analyses a^T_3 are performed on the mixture of the three migration waters from the tests resulting from the 3rd stagnation of (72 ± 1) hours,
- analyses b^T_1 are performed on the mixture of the three migration waters from the controls resulting from the 1st stagnation of (72 ± 1) hours,
- analyses b^T_2 are performed on the mixture of the three migration waters from the controls resulting from the 2nd stagnation of (72 ± 1) hours,
- analyses b^T_3 are performed on the mixture of the three migration waters from the controls resulting from the 3rd stagnation of (72 ± 1) hours.

The concentration of substances measured for each stagnation period 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 mixture of migration waters from the three tests,
- b^T_n is the concentration of the substance in mg/L measured in the mixture of migration waters from the three controls.

for the following conditions:

---

[21] *External diameter around 16.9 mm*

[22] *Sleeves for 20/27 steel pipe*

[23] *For tests in hot water (temperature between 60 and 85°C), the three migration periods are 24h*

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

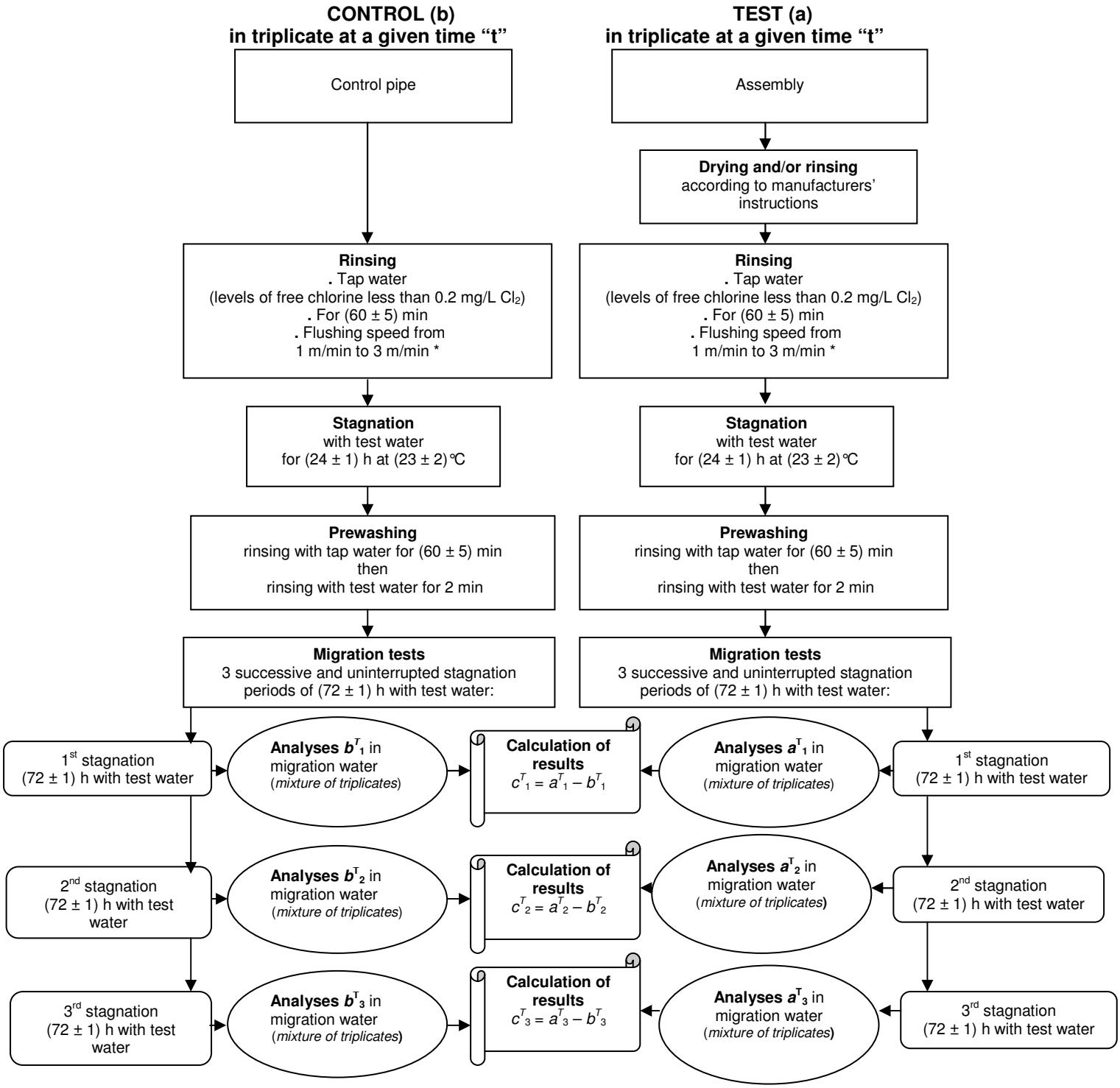
For each migration water, the migration rate M^T_n for a migrate substance may then be calculated as follows:

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

Where:

- M^T_n is the migration rate for n'th migration period,
- t is the duration of the migration period in days (**3 days**),
- S/V is the surface area-to-volume ratio in dm^{-1}. Considering that the set-up consists of 20 bonds and that each one corresponds to the forming of an accumulation of adhesive around 0.5 mm thick emerging into the opening of the pipeline, the S/V ratio is estimated as:
  - 46 cm^2/L = 0.46 dm^{-1} (for solvent-based adhesives),
  - 56 cm^2/L = 0.56 dm^{-1} (for adhesives obtained by polymerisation).

Thus: $\boldsymbol{M^T_n = c^T_n}$ / **1.38 in [mg/dm^2/day]** (solvent-based adhesives)
$\boldsymbol{M^T_n = c^T_n}$ / **1.68 in [mg/dm^2/day]** (adhesives obtained by polymerisation)



** the flushing speed from 1 m/min to 3 m/min corresponds to a flow-rate of:*
- 0.58 to 1.75 L/min for PVC (or CPVC) pipes
- 0.39 to 1.17 L/min for galvanised steel pipes.

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

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

- total organic carbon (TOC) according to the NF EN 1484 standard [17],
- odour and flavour according to the NF EN 1622 standard [18],
- trihalomethanes (chloroform, bromoform, dibromochloromethane and dichlorobromomethane) according to the NF EN ISO 10301 standard [19],
- the GC-MS profile[24] according to the pr NF EN 15768 draft standard [20],
- specific screening for parameters with a quality limit or reference specified in the Ministerial Order of 11 January 2007 [21], if present in the formulation of the adhesive,
- specific screening for substances on the lists of carcinogenic, mutagenic or toxic to reproduction (CMR) substances that have been classified according to a harmonised European scheme, if present in the formulation of the adhesive.

In the absence of a European standard for assessing the ability of materials to enhance microbial growth, this criterion, although used by some Member States[25] for the assessment of organic materials, has not been included.

3.3 Pass-fail Criteria

3.3.1 Relating to the formulation

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

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

- identified substances not included in the PLs,
- commercial mixtures for which details of the specific formulation are not required because of the low percentage used 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 3).

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

However, the specific migration limits (SMLs) specified in the Pls, established for materials intended to come into contact with foodstuffs (SML_{food}), have been deemed inappropriate for adhesives even after adapting them for materials intended to come into contact with DW (SML_{water})[26], and their systematic verification is not required.

3.3.2 Relating to migration testing

The results of the migration tests for the period of stagnation "n" must be lower than or equal to the results of the migration tests for the migration period "n-1" and the results of the analysis of the migration water from the third stagnation period 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 flavour or odour perceived by the tasting panel).

---

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

[25] 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 microorganisms test (method based on the determination of oxygen consumption)
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 coatings in contact with (tap) water (method based on the determination of Adenosine Triphosphate or ATP)

[26] 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 litres of water (see AFSSA Opinion and Report No. 2006-SA-0291 [16]).

For parameters with a quality limit or reference set by the Ministerial Order of 11 January 2007, it should be considered that the quantities due to the materials must not exceed 10% of the quality requirements (limits or references) set by the aforementioned Order [21].
Note that thus far, 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 they do not exceed 10% of the regulatory quality limits and references. Given these factors and in the interests of harmonisation, ANSES proposes retaining the 10%, which confers a wider margin of safety.
In addition, a conversion factor (CF) of 0.05 is applied to determine the concentration in the test water (see Annex 3).
Given these factors, the PFC in test waters for parameters with a quality limit or reference set in the Ministerial Order of 11 January 2007, are set according to the following calculation:

PFC = 10% of the quality limit or reference / 0.05
= quality limit or reference x 2

For TOC, applying the same reasoning, the adopted PFC for test waters is 4 mg/L of C.

For THMs, applying the same reasoning, the adopted PFC for test waters is 200 µg/L.

CMR substances, if present, must not exceed 0.1 µg/L in DW except in the case of lower threshold limits set by the Ministerial Order of 11 January 2007 [21]. Considering that the amounts detected in DW due to the materials must not exceed 10% of this quality requirement, and applying a conversion factor of 0.05, the adopted PFC for test waters is 0.2 µg/L.

Concerning the GC-MS profile, for which the Ministerial Order of 11 January 2007 has not set any quality limit or reference, ANSES has assumed that the surface area on which an adhesive 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 [10] proposes to use an S/V ratio of 46 cm^2/L for solvent-based adhesives and 56 cm^2/L for adhesives obtained by polymerisation, the PFC (1 µg/L) relating to the GC-MS profile defined in DGS/VS4 Circular No. 99/217 of 12 April 1999 [2], which uses an S/V ratio of 3 cm^2/L for joints, was therefore transposed using a conversion factor of:

- 15 (46/3) for solvent-based adhesives,
- 18 (56/3) for adhesives obtained by polymerisation.

The adopted PFC for test waters are therefore:

- 15 µg/L (1 µg/L x 15) for each compound detected and quantified relative to the closest alkane for solvent-based adhesives,
- 18 µg/L (1 µg/L x 18) for each compound detected and quantified relative to the closest alkane for adhesives obtained by polymerisation.

Table I: Summary of parameters to be analysed and provisional pass-fail criteria

Parameter	Test protocol	PFC (3rd stagnation period)		Unit
		Solvent-based adhesives	Adhesives obtained by polymerisation	
Flavour[27]	NF EN 1622 [18]	< 3	< 3	threshold
Odour[27]	NF EN 1622 [18]	< 3	< 3	threshold
THMs[28]	NF EN ISO 10301 [19]	< 200 < 150	< 200 < 120	µg/L µg/dm²/day
Parameters with a quality limit or reference set in the Ministerial Order of 11 January 2007 [21][28]		< Quality limit or reference of the Ministerial Order of 11 January 2007 x 2	< Quality limit or reference of the Ministerial Order of 11 January 2007 x 2	Units of the Ministerial Order of 11 January 2007
CMR substances[28]		< 0.20 < 0.15	< 0.20 < 0.12	µg/L µg/dm²/day
TOC[28]	NF EN 1484 [17]	< 4 < 3	< 4 < 2.5	mg/L of C mg/dm²/day of C
CG-SM profile[28]	pr NF EN 15768 [20]	< 15 < 11	< 18 < 11	µg/L µg/dm²/day

Acceptability criteria are thus proposed whose relevance to adhesives which already have a certificate of Compliance with Positive Lists (CLP) must be verified (according to the protocol described in Section 3.2) before these guidelines are applied to migration tests.

The results of migration tests carried out according to other protocols are given in Annex 4 for information purposes.

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

[28] *The acceptability criterion refers to* c^T_3.

4 Conditions for obtaining an attestation of sanitary conformity (ACS)

The safety compliance of adhesives can only be assessed by a laboratory authorised for that purpose by the French Minister of Health (see Article R. 1321-52 of the CSP and the Ministerial Order of 18 August 2009 [22]).

The applicant must submit to the authorised laboratory a complete dossier, notably containing information relating to the formulation of the adhesive (see Annex 5).

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

For carrying out migration tests, the adhesive must be sent to the laboratory in the packaging offered for sale.

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

If the applicant intends the product to be used with hot water, testing in these conditions is mandatory. If he intends the product to be used with hot water and cold water, testing at both temperatures must be performed.

The ACS must specify:

- the temperature at which the migration tests were carried out,
- the drying and/or rinsing time after assembly and before contact with water again, as recommended by the manufacturer.

If, when an ACS is renewed, the formulation of the adhesive, verified by the authorised laboratory, is unchanged and still complies with the PLs, the ACS can be renewed for a period of five years without further migration tests being necessary.

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

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

When an adhesive is sold under various configurations (e.g. polymers of different molecular weight, etc.) using the same formulation, the migration tests must be performed on the extreme configurations, and if the migration test results are significantly different, all the product configurations must be tested.

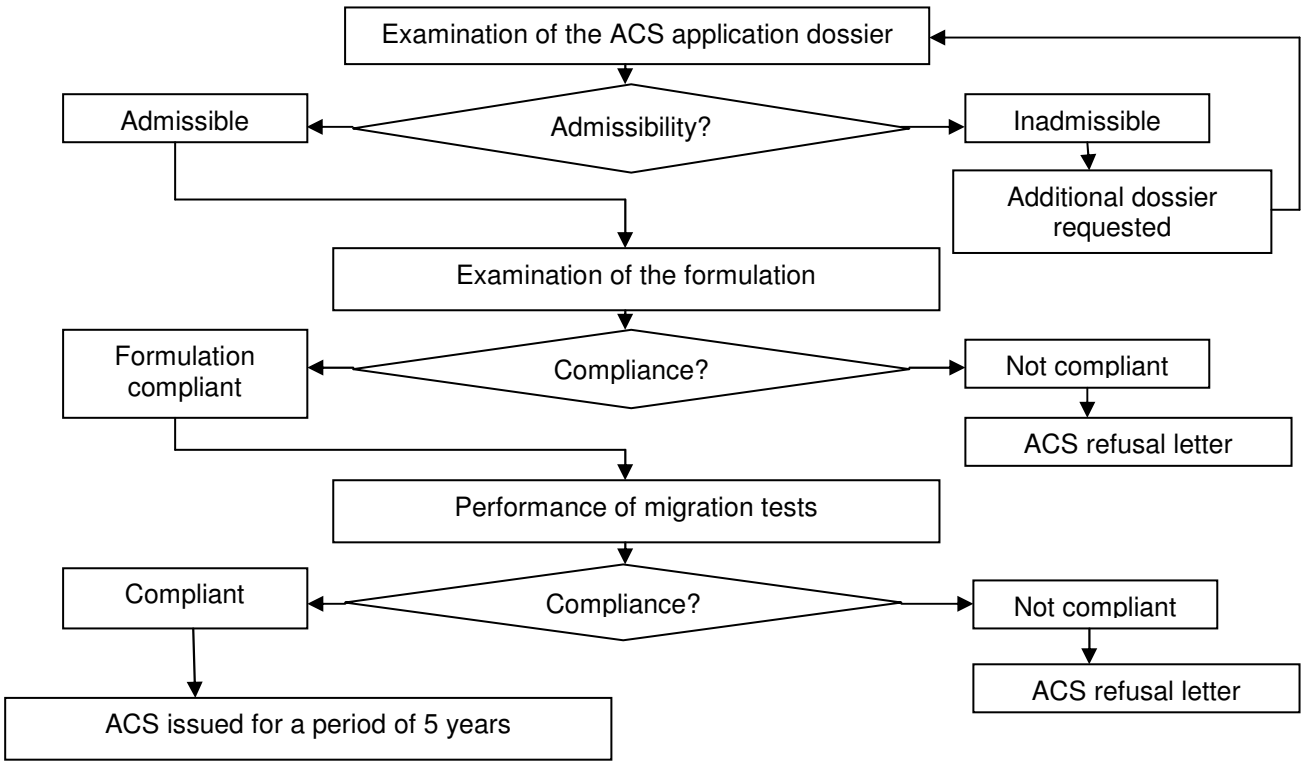


Figure 2: Decision tree for issuing an ACS

5 Conclusions

ANSES:

1) proposes that the pass-fail criteria for the migration tests defined in these guidelines be validated by tests (according to the protocol described in Section 3.2) on the different types of adhesives used in systems for the production and distribution of water intended for human consumption.

2) reiterates the importance of complying with the drying (or curing) and/or rinsing time, after assembly and before contact with water again, recommended by the manufacturer.

3) recommends that:

1. the laboratories currently authorised to verify the safety compliance of materials and articles coming into 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, for carrying out migration tests according to the protocol defined in these guidelines, and for obtaining the GC-MS profile according to the draft standard pr NF EN 15768 [20], in addition to the obligations defined in the Ministerial Order of 18 August 2009 [22];
2. each product should be subject to regular factory production inspection (audits) by an independent organisation, as is the case in other countries;
3. a sampling plan for test 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 adhesives authorised to come into contact with DW in other EU Member States differ from these guidelines, especially in 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 the migration waters, methods used to analyse 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 adhesives to date in other countries cannot be considered equivalent to each other or to the guidelines proposed in this report;
2. nevertheless, tests performed in another EU Member State could be considered on a case-by-case basis by the French laboratories authorised to issue ACSs; simplified migration tests, involving only the parameters that have not been measured, may be sufficient if the S/V ratio used is equal to or greater than that recommended in this report and if the conditions for sample preparation are comparable.

Bibliographical references, regulations and standards

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

[2] Ministerial Circulars no. 99/217 of 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 of Health no. 99/25 and no. 2000/18, respectively).

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

[4] Circular DGS/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 placed in contact with the water supply (March 1999).

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

[7] Drinking Water Decree Article 4 – Regulation of December 7, 2002, nr BWL/2002095022.

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

[9] Guideline for the Hygienic Assessment of Organic Coatings in Contact with Drinking water (http://umweltbundesamt.de/wasser-e/themen/drinking-water/beschichtungsleitlinie.htm) and TZW Guideline: Test of your anaerobic glues – TZW Karlsruhe Prüfstelle Wasser.

[10] 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.

[11] DWI Guideline: 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.

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

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

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

[15] *Matériaux & techniques* – Issue 6/7 – Special Edition - June/July 1980 – Industrial glues and adhesives

[16] 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.

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

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

[19] Standard NF EN ISO 10301 (July 1997): Water quality – Determination of highly volatile halogenated hydrocarbons – Gas-chromatographic methods.

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

[21] 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.

[22] 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.

[23] CSTB/CRECEP/Institut PASTEUR de LILLE/LHRSP (25 October 2002). Assessment of the content of organic solvents and organochlorines in water having remained in PVC pipes joined by gluing.

[24] 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)*

[25] French Scientific and Technical Centre for Building (CSTB) (26 October 2006). Research on the migration of constituents of sealants in pipe assemblies transporting water intended for human consumption. *(Study contains confidential information that is not available for consultation)*

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

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

[28] 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), pp329-37, Chelsea MI.

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

[30] CSTB/CRECEP (1 October 2002) – Assessment of the content of organic solvents in water contained in glued PVC assemblies for different rinsing or daily drawing times. *(Study contains confidential information that is not available for consultation)*

Annex 1: Comparison of migration tests on adhesives conducted by various countries

Solvent-based adhesives

Reference	Country	Type of sample	Water contact protocol	Parameters analysed	Criteria
DIN EN 12873-2	Germany	- 20 bonds on 1m of CPVC pipe with ϕ = 32 mm - S/V = 30cm^2 for 450 ml - Drying according to manufacturer's recommendations	At 23°C (static): - Flushing with tap water for 60 minutes - Stagnation for 24 hours in test water - Prewash with tap water for 60 minutes - Stagnation for 3 x 72 hours in test water => analysis of the 3 migration waters	- TOC - Odour/flavour - Chlorine demand - Specific substances	- < 1.25 mg/(dm^2 day) (S/V = 5 dm^{-1}) - < 2 (S/V = 5 dm^{-1}) - < 1.50 mg/(dm^2 day) (S/V = 5 dm^{-1}) - Assessment on a case-by-case basis
BS EN 12873-2 and BS 6920	UK	- 1 sample for each type of water - test sample consisting of material pieces joined using the solvent cement (BS 6920-2 section 2) - S/V ≥ 1.5 dm^{-1} - Drying according to manufacturer's instructions for use (IFU)	At 23°C (static): - Rinse in tap water for 30 minutes - Stagnation: o WRAS: 24, 24, 24, 72, 24, 24 & 24 hours (BS6920-2), o Regulation 31: 3 x 72 hours (EN 12873) -Test water: non-chlorinated and chlorinated tap water (1 mg/L) => analysis: o WRAS: migration waters from the 1st and 7th stagnation o Regulation 31: migration waters from the 3 stagnations	<u>Products only used in building water systems (WRAS):</u> - Odour/flavour - Enhancement of microbial growth (BS 6920-2.4 based on the determination of dissolved oxygen consumption - MDOD) - Colour and turbidity - Metals - Cytotoxic substances <u>Products used by public water supplies (Regulation 31):</u> Reduced assessment for adhesives: - Odour/flavour - Enhancement of microbial growth (MDOD)	- odour and flavour free - <2.4 mg/L - <5 Hazen units, <0.5 FTU - <DWD limits - non toxic - odour and flavour free - <2.4 mg/L
NSF61	USA	- Bonds on pipes with ϕ = ½ in according to ASTM recommendations - Drying according to manufacturer's recommendations, or alternatively for 48 hours	At 23°C (static): - Rinse in tap water - Conditioning for 14 days in test water at pH 5, 8 and 10 (at least 10 renewals of the water) - Stagnation for 3 x 24 hours in test water at pH 5, 8 and 10 => analysis of the 3rd migration water	Relevant parameters to be screened for are determined on a case-by-case basis during the examination of the product's formulation: - Solvents - PAHs - GC-MS profile - etc…	see Annex D of the NSF/ANSI 61 document (amounts detected must not exceed 10% of the water quality requirements set by the regulations)

Reference	Type of samples	Water contact protocol	Parameters analysed	Criteria
CSTB/CRECEP/ Institut PASTEUR de LILLE/LHRSP [23]	- 4 bonds on 3x36 cm of PVC pipe with ϕ = 40 mm + 1 bond for lower female cap - Oven drying at 45°C for 48 hours	At 20°C (static): - Rinse in tap water for 30 minutes (2L/min) - Stagnation for 24 hours in test water (demineralised water, moderately mineralised water) => analysis	- Solvents - THMs - TOC - Flavour	/
CARSO XP P 41-250 [24]	- 2 bonds on 2x10 cm of PVC pipe with ϕ = 40 mm - Drying according to manufacturer's recommendations	At 20°C (static-immersion XP P 41-250): - Vigorous rinse with tap water for 60 minutes - Preconditioning for 24 hours in test water (demineralised water, moderately mineralised water) - Stagnation for 24 hours in test water (demineralised water, moderately mineralised water) => analysis	- Solvents - THMs - TOC - Flavour - GC-MS profile	/

Adhesives obtained by polymerisation

Reference	Country	Type of samples	Water contact protocol	Parameters analysed	Criteria
DIN EN 12873-2	Germany	- threads on metal or PVC pipes - S/V = 5 dm^{-1} - Drying according to manufacturer's recommendations	At 23°C (static): - Flushing with tap water for 60 minutes - Stagnation for 24 hours in test water - Prewash with tap water for 60 minutes - Stagnation for 3 x 72 hours in test water => analysis	- TOC - Odour/flavour - Specific substances	- < 1,25 mg/(dm^2 day) (S/V = 5 dm^{-1}) - < 2 (S/V = 5 dm^{-1}) - Assessment on a case-by-case basis
BS EN 12873-2 and BS 6920	UK	-1 sample for each type of water - Test sample consisting of suitable joint sealed using the adhesive - S/V ≥ 1.5 dm^{-1} - Drying according to manufacturer's instructions for use (IFU)s	At 23°C (static): - Rinse in tap water for 30 minutes - Stagnation: o WRAS: 24, 24, 24, 72, 24, 24 & 24 hours (BS6920-2), o Regulation 31: 3 x 72 hours (EN 12873) - Test water: non-chlorinated and chlorinated tap water (1 mg/L) => analysis: o WRAS: migration waters from the 1st and 7th stagnation o Regulation 31: migration waters from the 3 stagnations	<u>Products only used in building water systems (WRAS):</u> - Odour/flavour - Enhancement of microbial growth (BS 6920-2.4 based on the determination of dissolved oxygen consumption -MDOD) - Colour and turbidity - Metals - Cytotoxic substances <u>Products used by public water supplies (Regulation 31):</u> Reduced assessment for adhesives: - Odour/flavour - Enhancement of microbial growth (MDOD)	- odour and flavour free - <2.4 mg/L - <5 Hazen units, <0.5 FTU - < DWD limits - non cytotoxic - odour andflavour free - <2.4 mg/L
NSF61	USA	- Plates coated with 15 cm^2/L - Drying according to manufacturer's recommendations, or alternatively for 48 hours	At 23°C (static): - Rinse in tap water - Stagnation for 3 x 24 h in test water at pH 5, 8 and 10 => analysis of the 3rd migration water	Relevant parameters to be screened for are determined on a case-by-case basis during the examination of the product's formulation: - Solvents - PAHs - GC-MS profile - etc…	see Annexe D of the NSF/ANSI 61 document (amounts detected must not exceed 10% of the water quality requirements set by the regulations)

Reference	Type of samples	Water contact protocol	Parameters analysed	Criteria
CSTB [25]	- 6 assemblies on 4x26 cm of galvanised steel pipe DN 48.3mm + 1 assembly for lower female cap	At 20°C (static): - Rinse in tap water for 30min (150L/h) - Rinse by turning over in demineralised water - Rinse by turning over in moderately mineralised water - Stagnation for 48 hours in test water (moderately mineralised water) => analysis	- TOC - GC-MS profile	/
CARSO XP P 41-250 [24]	- 1 assembly on threaded connector - Drying according to manufacturer's recommendations	At 20°C (static immersion XP P 41-250): - Vigorous rinse with tap water for 60 minutes - Preconditioning for 24 hours in test water (demineralised water, moderately mineralised water) - Stagnation for 24 hours in test water (demineralised water, moderately mineralised water) => analysis	- Solvents - THMs - TOC - Flavour - GC-MS profile	/

Annex 2: Positive lists

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

Directives and Regulations:

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

Resolutions of the Council of Europe:

- Resolution AP (92) 2 on 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 assessed by the European Food Safety Authority as 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 foodstuffs

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 Orders and Circulars:

- Ministerial Order of 25 November 1992 on silicones.
- Ministerial Order of 9 November 1994 as amended (by the 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 articles, varnishes and coatings intended to come into contact with commodities, products and drinks for human and animal consumption, notified to the European Commission under the reference 2004/328/F[29] following AFSSA's Opinion[30] of 9 April 2002 (Solicited request no. 2001-SA-0069 [26]).
- DGCCRF memorandum no. 2003-27 of 24 March 2003 on additives to plastic materials (since 1950).

Substances authorised for materials and articles designed 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-isothiazol-3-one (CAS: 26530-20-1).

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

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

Annex 3: Justification of the tolerated weight percentages of non-compliance in the formulation

The migration of a substance found in a material to the water with which it is in contact can be assessed by calculation according to 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 "wettened" 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 substance migrated all at once, its concentration in the water would be:

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

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

- If there was no mass migration of the substance all at once, 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 last equation is used to calculate the maximum weight percentage of a substance (p) in an adhesive in order that its migration (C2) is lower than the value adopted for the PFC.

2 - Calculation applied to adhesives

AFSSA's report of May 2005 [27] sets a threshold of toxicological concern (TTC) of **1.5 µg per person per day.** This threshold was established by considering that one third of daily intake comes from solid foods (0.5 µg per person per day) and two thirds from beverages (1 µg per person per day) [28]. Thus, for a daily consumption of 2 litres 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, Netherlands, United Kingdom and France, proposes conversion factors (CFs) to determine the actual impact of materials on the quality of DW in terms of concentrations found during the migration tests. Indeed, the surface/volume ratio and stagnation time used for the migration tests according to the EN 12873 standard do not reflect the reality of a water distribution system [29].

The CFs are set according to the following assumption:

$$CF = F_g \times F_o$$

where:

- F_g is the S/V ratio that reflects reality,
- F_o is the assumed residence time of the water in the system:
    1. Four days in the main public distribution systems (Ø ≥ 300 mm),
    2. Two days in the secondary public distribution systems (80 mm ≤ Ø < 300 mm),
    3. 0.5 days in plumbing systems (Ø < 80 mm).

Based on the above assumptions, a conversion factor has been defined for adhesives. Considering that glued assemblies are mainly found in plumbing systems inside buildings and that, in the worst case, on a linear metre of piping there are 3 connectors corresponding to 6 gluing points, the S/V ratio has been estimated at 14 and 17 cm^2/L for a residence time of 0.5 days.

i.e.:

	S/V (cm^2/L)	**Residence time (day)**
Actual case:		
- **Solvent-based adhesive**	14	0.5
- **Adhesive obtained by polymerisation**	17	
Migration tests:		
- **Solvent-based adhesive**	46	3
- **Adhesive obtained by polymerisation**	56	

The conversion factor (CF), used to change from the reality to the migration tests as defined in this report, corresponds to:

- **CF =** 14 x 0.5 / 46 x 3 = **0.05** (Solvent-based adhesive)
- **CF =** 17 x 0.5 / 56 x 3 = **0.05** (Adhesive obtained by polymerisation)

$$C2_{(\text{in test water})} = C2_{(\text{in tap water})} / CF$$

thus a $C2_{(\text{in tap water})}$ = 0.5 µg/L corresponds to a $C2_{(\text{in test water})}$ = 10 µg/L.

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

	Solvent-based adhesive	**Adhesive obtained by polymerisation**
Density of the adhesive (d)[31]	900 kg/m^3	1100 kg/m^3
Thickness of the applied adhesive[32]	0.0006 m	0.0003 m
Thickness of the corresponding wetted adhesive (10%[33]) (t)	0.00006 m	0.00003 m
Surface/volume (S/V) ratio	4.6 m^{-1}	5.6 m^{-1}
$C2 = C2_{(\text{in test water})}$	10 µg/L	10 µg/L

[31] *Mass commonly observed in the technical instructions for adhesives.*

[32] *Filling thickness in the interstitial space between the pipe and the connector.*

[33] *The basic assumption is that the thickness of the adhesive involved in the exchange with the water cannot exceed 10% with regard to the migration values obtained for the TOC parameter in the 2007 [24] and 2006 [25] studies.*

=> Maximum weight percentage of a substance in a **solvent-based adhesive** in order that migration is less than 10 µg/L (p): **0.402%**

=> Maximum weight percentage of a substance in an **adhesive obtained by polymerisation** in order that migration is less than 10 µg/L (p): **0.541%**

Thus, one or more substances not included on the positive reference lists that are used in the formulation and 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.

Annex 4: Results of migration tests

Solvent-based adhesives

The migration tests performed, even if they were not performed in accordance with these guidelines, all showed the release of solvents in large quantities:

2007 study [24]

The migration tests carried out following the recommendations of the XP P 41-250 standards are described in Annex 1 and the results (Table II) show that a drying time of more than two months was needed before solvent migration was no longer observed.

Table II: Results of migration tests for a solvent-based adhesive for PVC with an ATEC from the CSTB but no CLP certificate

		Drying time						
		1 hour	**6 hours**	**24 hours**	**7 days**	**14 days**	**1 month**	**2 months**
Adhesive mass (in g/2L)		0.1663	0.1301	0.1165	0.1085	0.1419	0.1260	0.1269
GC-MS profile (in µg/L)	∑ of unidentified peaks	5.6	4.2	1.6	0.5	0.7	4.6	0.8
	p,p'dioctyldiphenylamine (CAS: 26603-23-6)						0.7	
TOC (in mg/L of C)		0.5	0.5	0.5	0.9	0.3	0.2	0.2
Odour (threshold)		1.5 (solvent)	1.5 (solvent)	None	None	None	None	None
Flavour (threshold)		2 (solvent)	1.5 (solvent)	1.5 (bitter)	None	None	None	None
Ethylbenzene (in µg/L)		1.9	1.5	1.1		0.57		
Xylene (m + p) (in µg/L)		3	6.5	4.4	2.2	2.2	1.1	
Xylene (o) (in µg/L)		3.1	2.3	1.5	0.7	0.84		
Cyclohexanone (in µg/L)		1117	825	414	143	29	26	24

2002 study [23]

The migration tests carried out are described in Annex 1 and the results (Table III) show that a drying time of more than two months was needed before solvent migration was no longer observed:

Table III: Results of migration tests for a solvent-based adhesive for PVC

			Drying time						
		Control	**1 day**	**2 days**	**5 days**	**10 days**	**15 days**	**1 month**	**2 months**
Adhesive mass (about 10 g)									
THF (in µg/L) *Average of the values obtained*		290	21000	12900	7650	*Not useable, pipes with leaks*	1740	1990	1620
MEK (in µg/L) *Average of the values obtained*		40	4910	2930	1600	*Not useable, pipes with leaks*		580	630
Flavour (threshold) *Average of the values obtained*		6	30	25	22.5	*Not useable, pipes with leaks*	18.5	16	11
Chloroform (in µg/L)	**1 mg/L Cl_2** *(24 hours of stagnation)*	3.8	7.7						
	20 mg/L Cl_2 *(24 hours of stagnation)*	6.2	7.6						
	40 mg/L Cl_2 *(24 hours of stagnation)*	6.4	8.0						
	100 mg/L Cl_2 *(6 hours of stagnation)*	5.2	7.2						

In addition, studies [30] have shown that the rinsing time has little effect on the migration of solvents into the water.

Glues and adhesives obtained by polymerisation

2007 study [24]

The migration tests carried out following the recommendations of the XP P 41-250 standards are described in Annex 1 and the results (Tables IV) show that a drying time of two months was needed before a significant reduction in the migration of substances was observed, even though manufacturers state that pipes may be placed in contact with water immediately after gluing.

Tables IV: Results of migration tests for two adhesives obtained by polymerisation with no CLP certificate

		Drying time						
		1 hour	**6 hours**	**24 hours**	**7 days**	**14 days**	**1 month**	**2 months**
Mass of adhesive no. 1 (in g/2L)		0.1778	0.1000	0.0954	0.1181	0.1065	0.1057	0.1051
GC-MS profile (in µg/L)	∑ of unidentified peaks	122	114.5	50.2	31.8	2.5	3.7	40.3
	2-hydroxyethyl methacrylate (CAS: 868-77-9)	10.5	6.0	3.4	0.4			
	Acetophenone (CAS: 98-86-2)	5.5	2.5	0.5				
	Isoquinoline (CAS: 119-65-3)	0.7						
	1,2,3,4-tetrahydroquinoline (CAS: 635-46-1)	1.2						
	Bis(2-ethylhexyl) phthalate (CAS: 117-81-7)	2.1	2.3	1.4				
TOC (in mg/L of C)		0.7	0.4	0.4	0.4	0.3	0.2	0.2
Odour (threshold)		None	None	None	None	None	None	None
Flavour (threshold)		None	None	None	1.5 (bitter)	None	None	None

		Drying time						
		1 hour	**6 hours**	**24 hours**	**7 days**	**14 days**	**1 month**	**2 months**
Mass of adhesive no. 2 (in g/2L)		0.1180	0.1000	0.1000	0.1122	0.1149	0.0904	0.0919
GC-MS profile (in µg/L)	∑ of unidentified peaks	177.2	162.7	160.3	49.7	133.4	35.9	6.9
	Cyclododecane (CAS: 294-62-2)				2.0	0.8	0.6	
	Heneicosane (CAS: 629-94-7)	1.0	1.5					
	2 propanoic acid, 2 methyl, oxybis (2,1 ethane diyloxy - 2,1-ethanediyl) ester (CAS: 109-17-1)	44.5	42	48	7.4	62	1.6	
	2 propanoic acid, 2 methyl, oxybis (2,1 ethane diyloxy - 2,1-ethanediyl) ester (CAS: 109-17-1)	5.7	5.9	11		16		
TOC (in mg/L of C)		0.4	0.5	0.5	0.6	1.2	0.3	0.3
Odour (threshold)		None	None	None	None	None	None	None
Flavour (threshold)		None	None	None	2 (bitter)	1.5 (fat)	None	None

2006 study [25]

The results (Tables V) of the migration tests carried out following the recommendations described in Annex 1 show that the migration of substances does not always reduce as a function of the drying time.

Tables V: Results of the migration tests for three adhesives obtained by polymerisation with no CLP certificate

		Drying time						
		Control	**2 days**	**4 days**	**10 days**	**18 days**	**27 days**	**34 days**
Mass of adhesive no. 1 (in g/1.5L)			?	5.17	5.73	8.18	14.02	?
GC-MS profile (in µg/L) only substances with the highest concentrations	Peak no. 1 unidentified		197	101	116	149	161	78
	Peak no. 2 unidentified		566	278	349	465	496	245
	Peak no. 3 unidentified		737	308	465	579	636	340
	Peak no. 4 unidentified		543	222	325	321	404	216
	Peak no. 5 unidentified		366	153	235	198	303	158
	Peak no. 6 unidentified		202	81	136	97	173	90
TOC (in mg/L of C)		2	17	10	13	10	15	11

		Drying time						
		Control	**2 days**	**4 days**	**10 days**	**18 days**	**27 days**	**34 days**
Mass of adhesive no. 2 (in g/1.5L)			7.2	6.58	6.41	7.46	5.82	5.71
GC-MS profile (in µg/L) only substances with the highest concentrations	4- methylbenzyl (CAS: 2431-00-7)			180.7	84.1	98.6	59.3	
	Formamide N(2,4 dimethylphenyl) (CAS: 60397-77-5)			11.3	11.1	14.4	22.9	
	Triethylene glycol dimethacrylate (CAS: 109-16-0)			25.0	16.5	14.4	9.3	
	Triethylene glycol dimethacrylate (CAS: 109-16-0)			652	327	224.6	59.1	
	Bis(2-butoxyethyl) phthalate (CAS: 117-83-9)			456	559	490	542	
TOC (in mg/L of C)		3.8	7.4	15.9	16.1	12.5	19.1	16.3

		Drying time						
		Control	**2 days**	**4 days**	**10 days**	**18 days**	**31 days**	**35.5 days**
Mass of adhesive no. 3 (in g/1.5L)			4.85	4.36	4.14	4.39	4.07	4.85
GC-MS profile (in µg/L) only substances with the highest concentrations	Acetophenone (CAS: 98-86-2)			27.8	33.1	41.4	86.4	118.9
	Benzene methanol alpha, alpha dimethyl (CAS: 617-94-7)			165.2	189.7	208.9	358.5	370.4
	NN'-methylenebis metacrylamide (CAS: 2359-15-1)			109.5	69.9	59.8	197.8	138.8
	Unidentified peak			93.2	108.4	97.9	127.6	140.7
	Unidentified peak			90.9	105.2	96.6	134.1	163.9
	Triethylene glycol dimethacrylate (CAS: 109-16-0)			214.8	250.1	411.6	620.6	563.9
	Triethylene glycol dimethacrylate (CAS: 109-16-0)			676.6	502.2	317.8	1007.6	894.4
	Triethylene glycol dimethacrylate (CAS: 109-16-0)			430.6	270.7	171.7	571.9	583.1
	Triethylene glycol dimethacrylate (CAS: 109-16-0)			271.3	173.7	198.7	313.8	335.3
	Hexanoic acid, 2-ethyl-, oxybis(2,1-ethanediyloxy-2,1-ethanediyl)ester (CAS: 18268-70-7)			25.6	16.7	29.6	40.9	37.7
	Triethylene glycol dimethacrylate (CAS: 109-16-0)			88.6	109.9	69.6	198.2	240.1
	Triethylene glycol dimethacrylate (CAS: 109-16-0)			54.8	35.5	43.9	129.4	188.2
TOC (in mg/L of C)		2.4	17	13	17	20	18	19

When applying for an ACS, the adhesive manufacturer must specify the drying (or curing) and/or rinsing time necessary before contact with water again. These times must be complied with before the migration tests themselves are conducted. However, while for the construction of new buildings contact with water may take place several months after gluing, this is rarely the case with renovations, when contact with water takes place after only a few hours of drying. Thus, even if some solvent-based adhesives obtain an ACS, it may be difficult for the user to comply with the drying time recommended by the manufacturer and stated on the ACS.

Annex 5: 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 adhesive, if different from the applicant;
3. Trade name(s) of the adhesive;
4. The type of adhesive for which the application is being made;
5. The safety data sheet and technical instructions for using the product;
6. A specimen of the proposed label;
7. The product formulation (composition and constituents):
    a. the chemical names of substances used in the product formulation [chemical name specified in Annex I to 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. CAS number and EC number (EINECS or ELINCS) of the substances,
    c. concentration of the substances;
8. Where 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. contact information of supplier (address, telephone, contact person);
9. Where the product has obtained authorisations 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 English of the dossier submitted in the authorising Member State, including the full migration test report when available. This report must include:
        - the number of samples tested,
        - the S/V ratio used,
        - the conditions of sample preparation,
        - the parameters screened for in the migration waters and their methods of analysis,
        - analysis results and PFC;
    c. references of the scientific organisation(s) that performed the analyses and/or tests as well as evidence of its (their) technical competence (as a minimum, accreditation for the parameters measured).