The impact of private networks on off-flavour episodes in tap water

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ABSTRACT

Complaints registered by Veolia water agencies underlined the design and operating parameters in the private network can cause taste and odour (T&O) episodes. Three experiments were performed to elucidate the impact of the private network. Firstly, a lab-scale pilot study was designed involving four different pipes, consisting of copper, polyvinyl chloride, stainless steel and galvanized steel in which several residence times were simulated. The study was then conducted on a real private network. Finally, laboratory static experiments on tap seals were carried out. To evaluate T&O potential of drinking water samples, sensory analyses by a taste panel were performed. To detect and to quantify the molecules involved, stir bar sorptive extraction coupled with gas chromatography/mass spectrometry analyses was applied. The results showed that the residence time had a significant influence on the appearance of organic and metallic compounds. An audit of the private network identified two main critical points—corrosion and leaching—both of which are influenced by the hydraulic system. Finally, static experiments conducted on seals revealed a large quantity of polycyclic aromatic hydrocarbons leaching into water and a chemical or plastic taste according to the panel. These results suggest a huge potential impact of the private network on water quality if operating conditions are not optimal.

Key words | PAHs, private network, sanitary fittings, SBSE-GC/MS, tap water, taste and odour

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INTRODUCTION

According to an American survey (Suffet *et al.* 1993), 65% of 300 water utilities reported that the distribution system is the main source of taste and odour (T&O) problems. Three major possible causes of organoleptic contamination are the chlorine residual (40%), domestic plumbing (25%) and corrosion by-products (12%). The distribution system is composed of two parts, which are physically separated by the water meter. The pipes between the water treatment plant and the water meter constitute the public network. The home plumbing between the water meter and the taps inside the building is known as the private network (Figure 1). This latter part of the distribution system includes the pipes and their connectors, tap fittings, home water treatment devices and so on.

Private networks are composed of metallic and organic materials, and French regulations require the manufacturers to obtain a certificate of compliance for all products that come into contact with drinking water. However, this national approval system only covers the non-metallic components. The composition of any organic materials used has to comply with the national or European standard lists, which include all the substances that are authorized for use in equipment that comes into contact with drinking water. Accredited laboratories carry out a testing procedure to ensure that these materials do not impair the water quality. In particular, an organoleptic test assesses the ability of a product to impart a discernible odour or flavour to water according to migration protocol
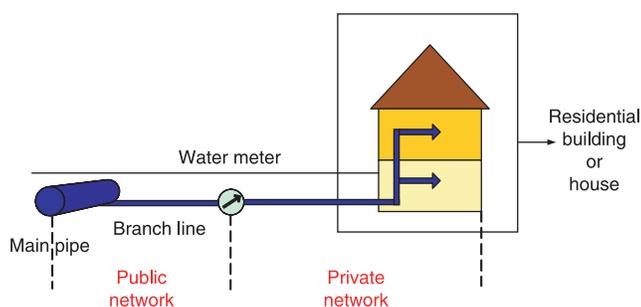
Figure 1 │ Public and private network.

described in the experimental normative standard XP P 41 250-1 (AFNOR 2001). In the case of metal equipment, only the composition has to comply with the standards lists.

Veolia water utility agencies have noted that several T&O complaints have involved the private network. That is why the study focused on the influence of components of the private network on T&O problems in tap water in order to advise consumers. Moreover, the general objective of this paper is to show the potential of domestic plumbing to alter aesthetic water quality.

MATERIALS AND METHODS

General approaches and relative procedures

Case 1: private network pilot

A loop system was designed involving the four types of pipe used (3 m in length, 2 cm in diameter), namely, galvanized steel, copper, polyvinyl chloride (PVC) and stainless steel. Before the experiment, the pipes were disinfected with a chlorine concentration of about $1\,mg\,l^{-1}$ and a contact time of 1 h, followed by an overnight cleaning phase using drinking water. Three different sampling times were simulated by means of a solenoid valve:

- one week (this experiment has been repeated seven times with this residence time)
- one night
- one weekend

This pilot unit was supplied with local distributed drinking water (Table 1).

Table 1 │ Characteristics of local water quality

pH	7.8
Residual chlorine	$0.3\,mg\,l^{-1}$
Temperature	25°C
Total alkalinity	$13°F = 65\,mEq\,l^{-1}$
Conductivity	283 (25°C) $\mu S\,cm^{-1}$
Cl^-	$6.1\,mg\,l^{-1}$
Mg^{2+}	$8.2\,mg\,l^{-1}$
Na^+	$13.1\,mg\,l^{-1}$
SO_4^{2-}	$10.3\,mg\,l^{-1}$
NO_3^-	$0.9\,mg\,l^{-1}$
CO_2	$2.5\,mg\,l^{-1}$
TOC	$<0.5\,mg\,l^{-1}$
BDOC	$0.1\,mg\,l^{-1}$

Case 2: real private network

The real network chosen was in office premises built in 1982. This is a space on four floors, each around 700 square metres, plus a cellar. Copper pipes were used originally, and then galvanized steel from 1982. Next to this main building, there is another office space occupying two floors and equipped with new PVC pipes. After an audit of the private network, several water intake points were selected to study the impact of various critical points such as dead end and stagnation zones on the production of T&O. Three points of reference without any identified defect were added for the follow-up campaign. Two situations (no stagnation and two days' residence time) were tested in order to take into account the employees' lifestyles within the building. The water quality is reported in Table 1.

Case 3: migration tests

Migration test procedures were designed to assess the potential for the tap fittings to impart T&O. The study began with the seals of the tap nozzles. Two tests were used to evaluate the effect of the seals on tap water: one was a static test and the other a stirring test.

Physicochemical analysis

To detect and to quantify the molecules involved, stir bar sorptive extraction (SBSE) coupled with gas chromatography/ mass spectrometry (GC/MS) analyses were used, as described

Table 2 │ Target compounds and their odour/taste threshold for case 1 and 2

Target compound	Odour/taste threshold (ng l^{-1})	References
2,4,6,-Trichloroanisole (2,4,6-TCA)	0.03	Curtis *et al.* (1972)
	0.076	Nijssen unpublished results cited in Maarse *et al.* (1988)
	0.9	Young *et al.* (1996)
	30 (45°C)	Diaz *et al.* (2005)
	10–20	Whitfield *et al.* (1985, 1987)
	50	Young *et al.* (1996)
2,4-Dichloro-6-bromoanisole (24diCl6BrA)	0.3 (25°C)	Corbi (2006)
2,6-Dichloro-4-bromoanisole (26diCl4BrA)	4 (45°C)	Diaz *et al.* (2005)
2,6-Dibromo-4-chloroanisole (26diBr4ClA)	2 (45°C)	Diaz *et al.* (2005)
	0.09–0.35 (25°C)	Corbi (2006)
2,4-Dichloro-6-bromoanisole (24diBr6ClA)	0.7 (25°C)	Corbi (2006)
2,4,6,-Tribromoanisole (246TBA)	0.008	Saxby (1985)
	0.02	Whitfield & Hill (1997)
	12	Diaz *et al.* (2005)

by Benanou *et al.* (2003). In situations corresponding to case 1 and case 2, several haloanisoles are known to impart T&O in drinking water. These compounds and their phenolic precursors were therefore targeted in the GC–MS analyses. Table 2 summarizes the T&O thresholds of haloanisoles.

For the study of the potential of plumbing materials to generate T&O issues (case 3), another family of compounds was also targeted to complete the evaluation of the organoleptic water quality. A specific extraction was performed in case 3. This consisted of exposing the product by immersing in the test water for 24 h at constant temperature. This was done first with stirring, and then under static conditions. All glassware used in this leaching protocol had been cleaned with a detergent solution, rinsed with distilled water and finally oven dried. A mix containing 39 PAHs was obtained from LGC Promochem, and used to quantify these compounds in water samples. PAHs were extracted from 100 ml water sample with a stir bar known as the Twister™. The sample was placed in a 125 ml vial, and 5 ml of ethanol and 50 μl of internal standard solution (naphthalene d8, phenanthrene d10, fluoranthene d10, benz[a]anthracene d12) were added. Adding ethanol made it possible to extract haloanisole compounds along with the PAHs. This solvent mini-mizes wall adsorption effects for the target compounds.

The instrumental gas chromatography conditions are summarized in Table 3.

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For immersion exposure, a sample was cut off each seal to obtain a surface area/volume ratio equal to 3 square centimetres in 1 litre of test water, according to the French standard XP P 40 250-1 (AFNOR 2001). The seal sample and a magnetic stirring bar were placed in a glass container with a glass lid filled to the rim with test water, excluding any headspace. The test water was mineral water that had been stored in a glass bottle. The container was maintained at 22 ± 2°C in the absence of light by placing it in a controlled temperature bath. A blank test without the seal sample was performed simultaneously. After the exposure period, the water was assessed by a taste panel and analyses of odorous compounds were also realized.

Another test under static conditions was carried out on a fresh sample using the same surface area/volume ratio. On the day that testing started, the product was rinsed using local

Table 3 │ Instrumental conditions for PAH analysis

Thermal desorption	30°C $\xrightarrow{60°C\,min^{-1}}$ 280°C (8 min)
Transfer line	315°C
Injector temperature	$-100°C \xrightarrow{12°C\,s^{-1}}$ 310°C (5 min)
Injection mode	Solvent vent: «vent flow»: 30 ml min^{-1} until 0.05 min
	«purge flow»: 20 ml min^{-1} at 5 min
Carrier gas	Helium, 1.5 ml min^{-1} constant flow
Oven program	50°C (2 min) $\xrightarrow{10°C\,min^{-1}}$ 200°C $\xrightarrow{25°C\,min^{-1}}$ 330°C (7 min)

tap water for 60 minutes, followed by three brief immersions in the test water. The product inside the container was maintained at $22 \pm 2°C$ in the same water bath for 24 h. The leachate water was retained for taste assessment and analyses of odorous compounds. This extraction procedure can be repeated using the same sample of the product until no discernible taste was detected by the panel.

Taste panel

For case 1 and 2, water samples were sent to a laboratory where they were evaluated by a trained panel according to a modified French standard procedure. In contrast to the reference protocol, which evaluates the taste and odour of a diluted sample of water, the panellists tasted the water samples undiluted.

For case 3, to evaluate the ability of seals to impart a discernible taste to tap water, the taste assessment was performed on the leachate water. In this case, the panel comprised five untrained people. The testing procedure was a modified version of French standard NF 1622 (AFNOR 1997). Some precautions were necessary before the taste assessments were started: for instance, the panellists were required to abstain from eating or smoking for at least 60 minutes before performing the odour and flavour evaluations. A triangle test was used with no forced choice. Each panellist was presented with unidentified samples of three waters in glass Erlenmeyer flasks, two of which contained mineral water, and one of which had been filled with leachate water. The panellists tried to find the leachate water sample, then to assess the intensity of any odour and flavour (scored as: very weak, weak, strong or very strong), and finally to identify the type of T&O using an odour wheel (Suffet *et al.* 1999).

RESULTS AND DISCUSSION

Case 1: private network pilot

Influence of water residence time on the release of odorous compounds

In the pilot experiment, the material/water interaction was perceived as the result of the release of metal ions. After a residence time of one week inside the four loops of pipes, iron, zinc and copper ions were found in water samples. Table 4 shows the mean values obtained during this experiment. The amount of copper released was above the guideline value (1 mg l^{-1}) but still lower than the French regulatory limit (2 mg l^{-1}). This experiment seems to assure the sanitary water quality but what about the organoleptic quality? The values originally given for the copper flavour threshold in distilled water and mineral spring water ranged from 2 to 13 mg l^{-1} (Cohen *et al.* 1960; Beguin-Bruhin *et al.* 1983; Rogers *et al.* 2004). However, more recent experiments defined a lower threshold, about 0.5–1 mg l^{-1} (Dietrich *et al.* 2004; Cuppett *et al.* 2006; Epke & Lawless 2007). Consequently, the copper release found in this experiment was around the flavour threshold, and an astringent or metallic taste (Zacarias *et al.* 2001) could be perceived in the

Table 4 │ Metallic species release with different pipe material

	Copper pipe	Galvanized steel pipe	Stainless steel pipe	French regulatory limit	French guideline value
Iron (μg l^{-1})		10	6.4		200
Zinc (mg l^{-1})		0.6			
Copper (mg l^{-1})	1.85			2	1

copper loop. A few studies have measured the flavour thresholds for iron and zinc, and have indicated values of about 0.04–3 mg l^{-1} (Cohen *et al.* 1960; Rogers *et al.* 2004) and around 20 mg l^{-1} (Rogers *et al.* 2004), respectively. The metal concentrations (Table 4) in the other pipe loops were below both the regulatory limits and the flavour thresholds. Finally, a corrosion phenomenon was assumed to affect the copper pipe used particularly in plumbing materials in French private networks. Nevertheless no taste was detected in the water samples.

Several water residence times were tested to study the influence of this parameter on the formation of organic odorous compounds. For the disinfection step, chlorine is used in local water. This can react with natural organic matter, in particular with humic or fulvic acid, to impart halophenol compounds, usually in the 2,4,6 substituted form, which are very stable. The haloanisole compounds appear after the biomethylation of halophenol compounds as a result of bacterial activity (Montiel *et al.* 1987). These odorous compounds arise during the chlorination step, and then in the distribution system. The off-flavour haloanisoles and their phenolic precursors were analysed to evaluate the organoleptic water properties inside pipe loops. In fact, water stagnation promotes formation of the haloanisoles, and an unpleasant musty or earthy taste could arise in
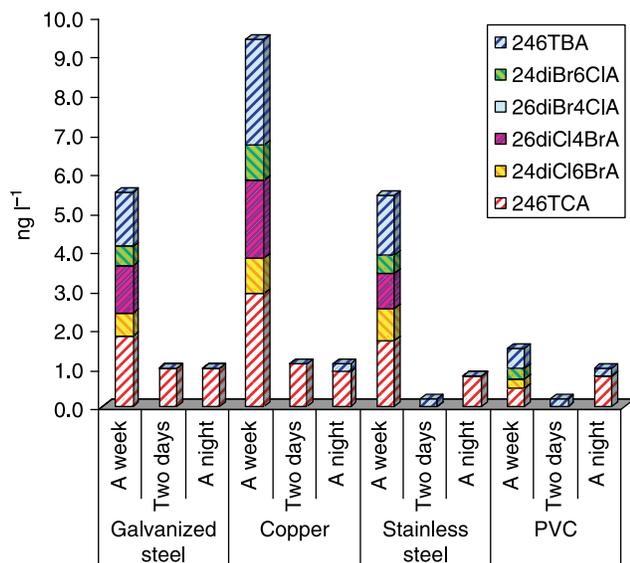
drinking water (Astier *et al.* 1995). Table 2 shows the target organic compounds for the pilot-scale study with their odorous and flavour thresholds. For all pipes, the highest concentrations of odorous compounds appeared after a water residence time of one week. Figure 2 shows that 2,4,6-trichloroanisole and 2,4,6-tribromoanisole were the dominant target chemical compounds in water samples from pipe loops, and that the copper pipe generated the greatest quantity of target haloanisoles (9 ng l^{-1}). Galvanized steel and stainless steel released equal concentrations while PVC pipes released the lowest amounts. In all pipes, the sum of haloanisoles exceeded their organoleptic thresholds (Table 2), and their phenolic precursors were also identified. In fact, the total halophenol concentration could reach 600 ng l^{-1}. This value was still below the organoleptic threshold (μg l^{-1}), but halophenol compounds could induce formation of haloanisoles as a result of biomethylation, thus leading to a musty taste in the network. In conclusion, the formation of odorous compounds depended on the kind of pipe material and increased with the water residence time in the pipe loops. The three metal pipes tested, and the copper pipe in particular, generated more haloanisoles than the PVC pipe. In this pilot study, copper displayed the highest potential for T&O formation. This finding could be attributed to leaching of the primer applied to copper pipes but more experiments are necessary to confirm this. It is intended to construct a full-scale pilot representing a private distribution system (1/1 scale) using copper material. It could be an opportunity to determine the amount of haloanisole compounds with a different source of water.

Case 2: real private network

Influence of the design and operation of the private network on odorous compound formation

With regard to corrosion, the second study conducted on a real private network confirmed that metal ions were released from galvanized steel, which supported the results obtained on the pilot-scale. In particular, a high iron concentration of around 0.4 mg l^{-1} was found at a mesh point (the water was circulating in a closed loop), and the taste panel reported a metallic flavour in water samples.

Figure 2 | Haloanisoles concentration with water residence time in pipe loop.

Table 5 | T&O evaluation after 24-h immersion exposure in mineral water with stirring

Type of seal	Taste/odour	Odour descriptor	Taste descriptor
Blank	No (4/5)		
NBR	No (5/5)		
EPDM	No (4/5)		
FPM	Yes (3/5)	Chemical	Mouth feel (metallic, astringent)
Unknown 1	Yes (5/5)	Chemical/hydrocarbon, phenol, plastic	Mouth feel (not identified)
Unknown 2	No (5/5)		
Fibre	No (4/5)		

Despite the fact that the main pipes in the private network were made of copper, only one case of significant copper release ($0.5\,mg\,l^{-1}$) was detected at a dead end, and no taste was perceived by the panel.

The critical points, such as dead ends or mesh points, caused the formation of haloanisoles at concentrations ranging from $0.18\,ng\,l^{-1}$ to $0.54\,ng\,l^{-1}$ (sum of haloanisoles) and bacterial growth. Moreover, the concentration of odorous compounds increased with the residence time, which was consistent with the results obtained in the pilot-scale study. Phenolic precursors were found at concentrations ranging from 8 to $39\,ng\,l^{-1}$ (sum of halophenols) in the network, and also identified at the outlet from the water treatment plant. Nonetheless, no off-flavour was identified by the panel.

A new construction near the main building made it possible to monitor the priming of new PVC pipes. Contrary to the regulations, no disinfection procedure had been applied. The data revealed a quantity of phenolic compounds ($93\,ng\,l^{-1}$) that was higher than the concentrations found at critical points (dead end and mesh point) in the main building after two days' residence time. Consequently, there was a considerable risk of haloanisoles formation and a high concentration of haloanisoles ($2.9\,ng\,l^{-1}$) was indeed detected in the PVC pipe. Moreover, the taste panellists determined a strong plastic flavour in the water samples. A release of chemical substances, in particular of plasticizers used in the composition of PVC or lubricants used for the PVC connection, could explain this off-flavour episode. An American study (Wiesenthal & Suffet 2007) investigated leaching from PVC and its joint solvents, of primer and cement into a drinking water system. These experiments showed that PVC primers and cement alone or applied to

PVC pipes led to a glue/varnish odour. GC–MS analysis of water samples from the new building revealed the presence of benzothiazole and its derivatives and of ketones, probably arising from adhesives used to connect the pipes. However, one month after the priming, the halophenol and haloanisole concentrations fell quickly as the pipe aged, to $26.6\,ng\,l^{-1}$ and $0.29\,ng\,l^{-1}$, respectively. The intensity of the plastic flavour had also decreased but a 'bad taste' persisted.

In conclusion, critical points such as mesh points or dead ends promoted the production of metallic and organic odorous compounds. Moreover, failure to follow the proper procedure when priming new PVC pipes led to a strong plastic taste in consumers' tap water. The next step in the study is to evaluate the leaching of various components of private network parts and to investigate their influence on organoleptic water quality.

Case 3: migration tests

The ability of seals to impart T&O in drinking water

The first component studied was the seal of the tap nozzle. According to an approved French laboratory, the materials usually used for seals are nitrile butadiene rubber (NBR) and ethylene–propylene diene rubber (EPDM). Fluorinated rubber (FPM), fibre and two unknown materials were also used in the study. All seal samples were tested at the same time and Table 5 shows that only the 'unknown 1' and the FPM seal samples generated a distinct T&O under these critical conditions. The intensity of T&O was not evaluated, but the panellists reported a low intensity T&O for the FPM seal, and a very strong T&O for the 'unknown 1' seal. For both these seals, the panellists identified a chemical odour

Table 6 │ T&O evaluation after static migration test in mineral water and treated water

	Type of seal	Taste/odour	Odour descriptor	Taste descriptor
Mineral water	Blank 1	No (5/5)		
	Unknown 1	Yes (4/5) very strong	Sulfurous	Bitter, mouth feel (not identified)
	Unknown 2	No (3/5)		
	Blank 2	Yes (3/5) weak	Earthy	
	Fibre	No (3/5)		
	EPDM	Yes (3/5) weak	Earthy	Bitter, metallic
Local distributed water	Blank 1	No (5/5)		
	Unknown 1	Yes (5/5) strong	Chemical/hydrocarbon, rubber	Bitter, mouth feel (metallic)
	Unknown 2	Yes (3/5) weak	Flowery	Acid
	Blank 2	No (3/5)		
	Fibre	Yes (3/5) very weak	Earthy	Bitter, metallic
	EPDM	Yes (3/5) very weak	Earthy	Bitter, acid

and, although it was difficult to match a descriptor for the taste, a mouth feel was perceived. Therefore, in this first flavour assessment in mineral water, the 'unknown 1' seal was the most likely to lead to an off-flavour episode which could lead to complaints of plastic, hydrocarbon or chemical tastes, because all the panellists detected a bad taste.

According to Table 6, the static experiment confirmed the potential of the 'unknown 1' seal to impair the organoleptic water properties. In the mineral water test, four out of five panellists found a strong T&O, the mouth feel was again detected by the panel, but in contrast to the first test, the odour descriptor changed. Without stirring, the response of panellists could be different because the concentration of compounds was lower. Whereas no T&O had been identified in the first test, this static experiment did detect a weak T&O for the EPDM sample seal, although the panellists thought that the leachate water was fit for consumption. The static migration protocol was carried out using locally distributed water in order to allow for the influence of chlorinated water on the T&O assessment. In this case, both of the unknown seal samples imparted a discernible taste to the tap water. All the panellists detected a strong chemical, hydrocarbon or rubber odour and a bitter or metallic taste for 'unknown 1' seal sample. In contrast to the first experiment, the 'unknown 2' seal sample gave a flowery odour, and an acid taste in water. It is possible that this untrained panel was becoming more sensitive as a

result of practice. New static migration tests are therefore necessary to confirm these findings.

A large quantity of polycyclic aromatic hydrocarbons was released into the leachate water after both the stirring test and the static test for the 'unknown 1' seal. Several PAHs were quantified (Table 7), some in concentrations as high as 1 or $2\,\mu g\,l^{-1}$. Nevertheless, the concentration of PAHs was lower in the static test.

Table 7 │ Quantification of polycyclic aromatic hydrocarbons in mineral water after the migration procedure (seal 'unknown 1')

Compound	After the stirring test (ng l⁻¹)	After the static test (ng l⁻¹)
Naphthalene	42	17
Σ Methyl naphthalene	170	43
Σ Dimethyl naphthalene	551	93
Acenaphthylene	124	19
Acenaphthene	171	21
Trimethyl naphthalene	141	14
Fluorene	1,421	102
Phenanthrene	2,264	113
Anthracene	742	31
Methyl phenanthrene	206	12
Methyl anthracene	981	50
Dimethyl phenanthrene	33	0
Fluoranthene	407	20
Pyrene	298	13
Methyl fluoranthene	22	6

CONCLUSION

Three points emerged during these studies: First, metal corrosion impaired the organoleptic water quality, and a metallic or astringent taste could appear in the consumer's tap water. The kind of pipe used in the private network also had an influence on the organic odorous compounds (halophenols and haloanisoles) released into the tap water. The risk of organoleptic degradation depends on the nature of the pipe material:

1. Galvanized steel is subject to corrosion. Indeed, a metallic taste was linked to the release of iron ions in case 2.
2. Copper is also subject to corrosion: once again this leads to a metallic taste, but also seems to promote haloanisoles formation, which imparts a musty/earthy taste to the drinking water, as in case 1. However, more investigation is necessary to elucidate this second point.
3. For PVC pipes, it is necessary to be careful when connecting pipes. Joining products, such as adhesives, used to connect PVC pipes, seem to be a source of off-flavour episodes described as a taste of plastic.

Second, the water residence time inside the pipe had an impact on the concentration of odorous compounds. The concentrations increase with the water residence time. Actually, the more critical water residence time tested was one week. In this case, the metal ions release and the formation of odorous compounds was greatest. Even if one night is the current critical water residence time in a private network, the desired one should be as short as possible with a quick flush before drawing water to drink.

Third, design parameters (mesh points, dead ends, etc.) and operating parameters (water residence time) must be optimized to limit the organoleptic problems in private networks.

Thanks to the panel, the ability of tap nozzle seals to influence off-flavour production was determined. Nevertheless, the assessment of T&O after migration experiments has made it clear that the panellists found it very difficult to choose descriptors to characterize taste or odour. Migration test results showed that some seals generated T&O. These off-flavours could be described as tastes of chemical, hydrocarbon, plastic or rubber. According to the GC–MS analysis, PAHs can be one of the reasons for this T&O production. Other tests are necessary to determine the organoleptic thresholds, and the kinetics of leaching. It is intended to continue the study by applying the same migration protocols to other components of private networks.

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