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► To cite this version:

Michel Dubus, Victoria Asensi Amoros, Stéphane Bouvet, Jean-Michel Brarda-Wieber, Isabelle Colson, et al.. Should We Discard Historical Wooden Archival Boxes?. Mieke Adriaens, Susie Bioletti and Ira Rabin. Chemical Interactions Between Cultural Artefacts and Indoor Environment, Acco, 2018, chemical interactions between cultural artefacts and indoor environment, 978-94-6344-798-0. hal-01915913

HAL Id: hal-01915913

<https://hal.science/hal-01915913>

Submitted on 8 Nov 2018

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Should We Discard Historical Wooden Archival Boxes?

Michel Dubus, Victoria Asensi Amoros, Stéphane Bouvet, Jean-Michel Brarda-Wieber, Isabelle Colson, Anne-Laurence Dupont, Agnès Lattuati-Derieux, Catherine Lavier, Éric Masson, Thi-Phuong Nguyen, Caroline Rogaume, Valentin Rottier

A total of 868 historical wooden archival boxes from the municipal and regional archives as well as from the French National Archives were studied by archaeodendrometrical techniques. The boxes were shown not to be airtight and to emit moderate concentrations of volatile organic compounds. The interactions between their content and the indoor environment were studied to investigate whether the emitted volatile organic compounds can impact the stability of the cellulosic documents and metallic artefacts they contain. Regardless of their age, results have shown that the boxes do not seem to represent a particular risk for cellulosic materials nor metallic artefacts.

1. Introduction

Since the early Middle Ages, archives have been preserved in wooden boxes, otherwise known as in French as *layette*s or *cassettes*. They were largely replaced in the twentieth century by cardboard boxes, partly because wood is known to emit volatile organic compounds (VOCs) that may degrade the documents. A number of institutions still keep them in storage or on exhibition, others in educational showcases.

Scientists from the Centre for Research on the Conservation of Collections (CRCC), the Centre for Research and Conservation of the French Museums (C2RMF), the Scientific and Technical Laboratory of the French National Library (BnF) and the Laboratory for Study and Research on Wood Material (LERMAB – ENSTIB) have been working together in collaboration with Puratech, a company specializing in air quality, to check the historical value of the boxes and their safety towards their contents. In addition, a large set of historical boxes

was studied by archaeodendrometry and dated the wood. The latter was done by the Université Pierre et Marie Curie (CNRS-URM8220), in association with the company Xylodata.

2. Experimental

2.1. Characterisation of the wooden boxes

2.1.1. Dating and wood craft

A total of 868 boxes, held in the archives of national museums (libraries and general documentation offices of the museums of France), the national archives, the municipals of Montpellier and Toulouse, the departments of the Ardennes, the Haute Marne, the Hautes-Pyrénées, the Loire-Atlantique, the Lot, the Vaucluse, the Meurthe-et-Moselle and the Puy-de-Dôme, were studied using archaeo dendrometric methods. In addition, 288 of these boxes were dated using dendrochronology.

An example of such a wooden box is shown in Figure 1. Ordered by Anne de Bretagne, Duchess of Brittany in the sixteenth century, it is among the oldest boxes in France. It shows the care taken by the craftsmen in the cutting (radial by cleavage) and the shaping of the wood (cuttings, grooves) as well as the assembly (using dovetailed joints).

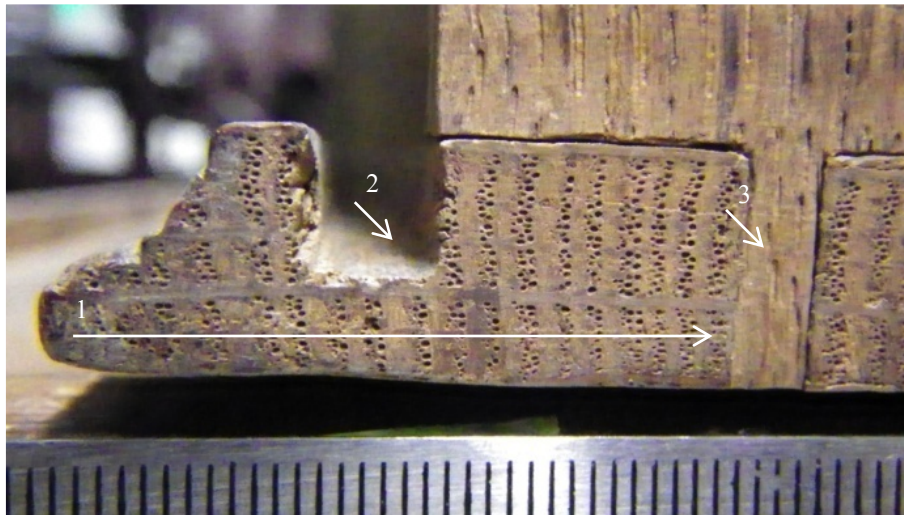


Figure 1. Detail of a box made in oak tree (*Quercus sp.*) from the treasure of the charters of the Duchy of Brittany, kept at the departmental archives of Loire-Atlantique, Nantes, France – © C. Lavier. Arrow 1 demonstrates the perfect radial cutting by cleavage; arrow 2 shows an example of a groove shaping and arrow 3 shows the assemblage by dovetail joint between the two boards.

The plant species of every board of every box was defined by microscopy. Dendrometrical examinations were performed to study the production of the boxes (cutting, shaping, and

assembly) as well as the morphology of boards (characteristics of the trees used). Dendrochronological experiments were made according to protocols set up for wooden objects and artworks, without impairing the integrity of the object, neither taking any sample, nor destructing the object [1-4]. All data obtained were analysed in order to determine the position of the wood in its tree, to precisely date the felling of every tree and to define its provenance. In all, more than 700 boards were studied by dendrochronology and 342 boards were actually dated. More than 78 000 pictures were made for the observations, measurements and use-wear analysis with a total of 280 000 analysis points of the tree rings.

2.1.2. Emission chamber tests on wood covers

A first set of measurements to determine the volatile organic compounds (VOCs), such as benzene, toluene, ethyl benzene, xylene and carbonyl compounds), were carried out in an emission chamber of 51 litres on 7 oak covers (emission areas between 0.11 and 0.22 m²), which dated between the fourteenth and twentieth century. A total of 28 passive air samplers (Radiello®*) were introduced under steady climatic conditions (25 °C / 55 % relative humidity) according to the NF EN ISO 16000-9 standard and this for 7 days (ISO 16017 2006) (Figure 2). The samples were placed in the emission chamber after 48 hours of filtered air-wash cleaning in order to avoid any contamination.



Figure 2. Set-up of the emission chamber according to the NF EN ISO 16000-9 standard.

2.1.3. Air tightness and climate performance

The air exchange rate (AER) was carried out on five wooden boxes with two different closing systems: sliding and flapping systems (Figure 3). After a week of acclimatization (temperature 21 °C and relative humidity 50 %), around 5000 ppm of CO₂ gas was injected into the boxes, after which its concentration was monitored in order to evaluate their air exchange. For each box, three to four tests were performed.

The climate performance tests were carried out using a climate chamber. Similar controlled relative humidity fluctuations (50 to 80 %) were applied to each box. The boxes were weighed at the start of the experiment and at the end of the experiment to evaluate the water mass loss or gain. Our hypothesis here was that water vapour was the main exchange between the box and its surroundings [5-10].



Figure 3. The boxes of the archives of the Lot and Hautes-Pyrénées in the climate chamber – © M. Dubus.

2.1.4. Volatile organic compounds

Six nineteenth century boxes, made of poplar wood originate from notarial archives and held in the archives of the French Hautes-Pyrénées and Lot departments, were conditioned in a 1 m³ glass chamber at a temperature of 23 °C and a relative humidity of 50 %. Radiello tubes and solid-phase micro extraction fibres (SPME)[†] were placed outside and inside these boxes for 11 days. Diffusive (passive) samplers for organic acids Swedish Environmental Research Institute (IVL) were placed in four wooden boxes in a controlled environmental chamber at a temperature of 23 °C and a relative humidity of 50 %) (Figure 3).

2.2. In situ measurements into historic boxes

2.2.1. Corrosion and volatile organic compounds

A set of measurements was set up in the municipal archives of Toulouse and Montpellier, the departmental archives of Ardennes, Haute-Marne, Loire-Atlantique, Hautes-Pyrénées and Puy-de-Dôme, as well as in the archives of the national museums. Radiello® passive samplers were exposed for 10 days in the fresh air intakes, the ambient environment, and in either full or empty boxes to collect aldehydes and BTEX chemicals (benzene, toluene, ethylbenzene and xylene). In addition, Purafil type ERC[‡] silver and copper coupons were placed to assess the air quality (Figure 4). Results were interpreted according to references [11-14].



Figure 4. Exposure of Purafil coupons and Radiello samplers in one of the wooden boxes and in the ambient atmosphere at the departmental archives of Puy-de-Dôme – © M. Dubus.

2.2.2. Concentration of volatile organic compounds

Solid-phase micro extraction (SPME) coupled to gas chromatography mass spectrometry (GC-MS) allows collecting and characterizing a wide range of VOCs emitted by various materials. SPME/GC-MS is a sensitive and reliable technique. It is easy to apply and does not require material sampling. It has been successfully used to identify gaseous emissions from organic materials in artworks [15-16]. In the present study, SPME fibres were placed in three wooden boxes at the French National Archives for 15 days.

2.2.3. Cellulose degradation

The impact of the microenvironment in five wooden boxes at the French National Archives was studied. Three out of the five boxes were those that were also used for the SPME sampling reported above. The fourth one was an empty box, which was added to the test to differentiate the impact of the wood emissions from those of the box content. The fifth box was made of poplar which resembles boxes from the eighteenth century. However, it could not be studied by dendrochronology as sampling was not possible. It contained geographical maps on paper with a silk lining. Examples are shown in Figures 5 and 6.



Figure 5. Constitution box (1790), poplar, French National Archives, room 117, mezzanine, A/69 – © A.-L. Dupont.



Figure 6. Oak box, French National Archives, room 117 – © A.-L. Dupont.

Four of the above-mentioned paper samples were placed in a neutral cardboard box and were kept in stable conditions in the laboratory (23° C and 50 % relative humidity) during the same period (14 months) as the control samples.

Whatman papers no.1 (W1) and no. 40 (W40), made of 100 % cotton, were placed in the boxes for fourteen months as sensors. W1 has long fibres and a neutral pH, whereas W40 has oxidized fibres and an acidic pH. Both papers were characterized at the start and at the end of the exposure time.

Some of the W40 samples were pre-sensitised by chemical oxidation with sodium hypochlorite at concentrations of 0.13 % and 0.26 % (percentage active chlorine). This treatment induced cellulose oxidation and a random chain cleavage, and was carried out for a better appraisal of the possible modifications incurred during the exposure.

Four paper samples (W1, W40, W40-ox0.13 and W40-ox0.26) were placed in each of the five boxes, at the bottom or inserted between the archival documents.

Physicochemical tests included viscosity measurements to determine the average degree of polymerization (DP_v) and copper index measurements to determine the degree of oxidation

of the cellulose. They were performed according to the TAPPI test methods TAPPI 230-om99 and TAPPI 430 cm-99.

3. Results

3.1. Historical value

The historical value of the wooden boxes is greatly linked to the quality of their manufacturing and to their age. Some are of the same or nearly the same age as the documents they contain. The oak wood or the fir (*Abies alba*), depending on their provenance, was replaced during the last century by poplar (*Populus sp.*), which was lighter and cheaper.

At Nantes, 107 wooden boxes made using oak wood during the fifteenth and sixteenth centuries, can be divided into three groups. The oldest ones, which are contemporaries of Anne de Bretagne, were assembled by wide dovetails. They are 52 cm long and 30 cm wide. Their sides are 15 mm in thickness. The second group is composed of 68 boxes with thin dovetails. Their edges are decorated with mouldings. They measure between 50 and 54 cm long and between 29 and 34 cm wide. The third group contains 23 boxes which are all assembled by nails, with a round profile for their tracks. Their sides are bevelled and shaped by cleaving. They measure between 53 and 54 cm long and between 31 and 34 cm wide. These wooden boxes thus belong to the memory of the duchy and its domain.

In Avignon, 336 boxes are composed of fir and poplar with 43 whole boxes and 39 associated covers. The parchment and paper leaves, dating from the sixteenth to the eighteenth centuries, have always been preserved inside these boxes.

In Clermont-Ferrand, the boxes are composed of fir, sorb (*Sorbus sp.*), poplar and ash wood (*Fraxinus ex.*). They differ in size. The wooden boxes of the fund of Clermont's abbey, St-Alyre, may have been made during the classification and the inventory recorded in 1787-1788. A part of them was replaced in the first half of the nineteenth century (before 1860). 52 boxes of the eighteenth century and 23 of the nineteenth century are empty. Three of the eighteenth century and eleven of the nineteenth century boxes are still full. They are supplemented by two wooden boxes from the collection of the convent of the Jacobins of Clermont [18].

In Charleville-Mézières, two batches of seven and 93 boxes were not dated because of the weakness of the information obtained on their wood (poplar).

At the National Archives, a group of 92 boxes, representing 552 boards of oak and poplar, was studied but none of them were able to be dated, either because of a problem of access to the tree rings, or because of a shortage of tree rings.

The treasure of the charters of the dukes of Lorraine is preserved in the departmental archives of Meurthe-et-Moselle, in the Hôtel de la Monnaie of Nancy, since 1771. The wooden shelves were replaced by metal shelves in 1980, then by compactus in 1993 during the renovation of the Treasure Room. The wooden boxes were used until 2011. 95 boards in oak wood date from the seventeenth and the eighteenth centuries, and those in fir wood date from the nineteenth century (Figure 7).

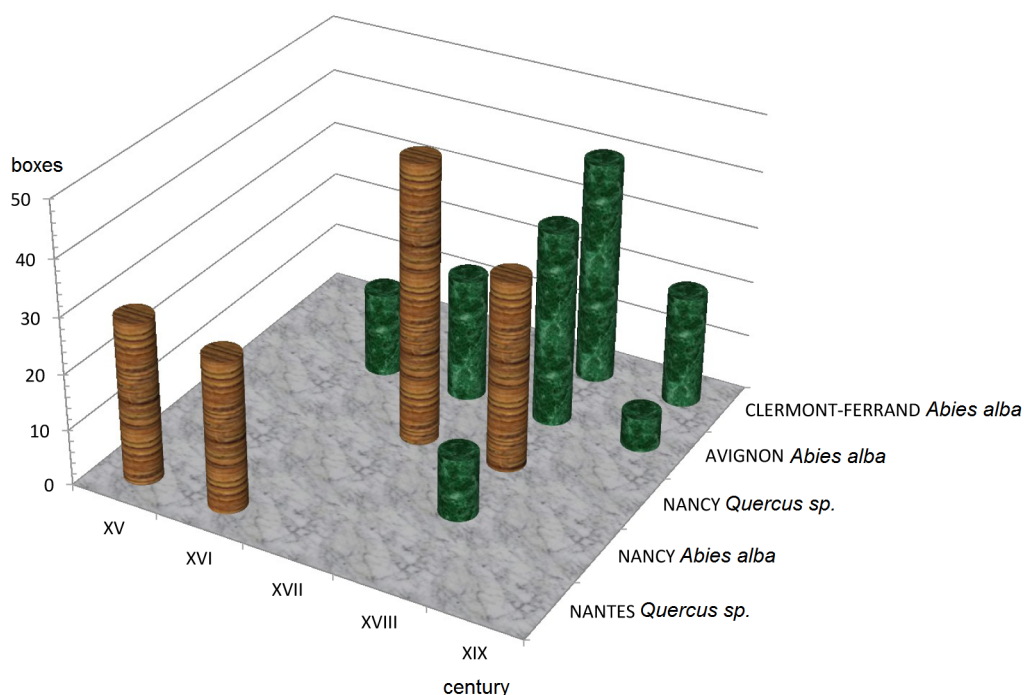


Figure 7. Distribution of species of the wooden boxes of the departmental archives at Loire-Atlantique, Meurthe-et-Moselle, Puy-de-Dôme and Vaucluse according to the age of the wood – © C. Lavier.

3.2. Air quality in the boxes

For the six boxes studied (see paragraph 3.3), holes were noticed and some of them went all the way through (cracks, gaps, exit holes...). The results for all boxes are very similar with an air exchange rate of one to two volumes per hour ($1-2 \text{ h}^{-1}$), which means they cannot be considered as airtight boxes. In comparison, a well-constructed showcase has an air exchange value between 0.02 and 0.04 h^{-1} . The air tightness seems to be better for the flapping system compared to the sliding one.

Indirect information was obtained during the test showing the climate environment in which these boxes were made and used. Indeed, while testing the boxes, it was noticed that opening and closing them is very difficult (in some case even extremely difficult) when they are acclimatized at a relative humidity of 50 %. This all becomes relatively easy at a relative

humidity of 80 %. The monitoring of the weight of the boxes and of the climate fluctuations indicates that there is always an exchange of water vapour with their surroundings. Nevertheless, in spite of their lack in airtightness, they seem to be able to protect their contents against the external daily variations of relative humidity and thereby show a certain buffering performance.

The analysis of the IVL passive samplers in the four wooden boxes indicated concentrations of the order of 51-80 $\mu\text{g}/\text{m}^3$ for formic acid and 170-189 $\mu\text{g}/\text{m}^3$ for acetic acid. It can be noted that these results are similar to those measured in archival boxes in the UK National Archives in Kew [19]. The acid concentrations measured in the ambient air, where the wooden boxes were placed for the monitoring period, were quite similar, being 64 $\mu\text{g}/\text{m}^3$ for formic acid and 166 $\mu\text{g}/\text{m}^3$ for acetic acid. These results indicate that the organic acids emissions level of the boxes is low and that the equilibrium between the interior and the exterior of the boxes was easily established due to the high air leakage and high air renewal rate of the boxes.

3.3. Air quality in the repositories

In all of the repositories surveyed, the air quality is comparable to the one found in normal indoor environments. Corrosion of Ag and Cu indicates the presence of SO_2 , O_3 , NO_2 , and HCl that could affect cellulose, organic materials and metals. Copper sulphide (Cu_2S) and copper oxide (Cu_2O), silver sulphide (Ag_2S) and chloride (AgCl) are the most frequent compounds. The data have been interpreted with reference to the recommendations of Sacchi and Muller and ISO 11844-2 standard [14]. The pollutants inside and outside the boxes are the same and their concentrations are similar. Formaldehyde, acetaldehyde, acrolein, butyraldehyde and valeraldehyde are the most abundant compounds that were identified. Formaldehyde and acetaldehyde are emitted by wood, wooden panels, solvent-based paints or cleaning agents. Acrolein is emitted by exhaust gases, heated grease, polymers manufacturing and fumes. Butyraldehyde is emitted by photocopiers, printers or organic solvents. Valeraldehyde is emitted by books and newspapers, solvent-based paints, particles boards. Toluene is emitted by paints, varnishes, glues, inks, carpets, gasoline. The correlation between the concentrations of aldehydes and toluene in ambient air and in the boxes, is equivalent, meaning that there is no particular pollution inside the boxes (see Table 1).

In the full boxes held in the national museums archives and in the archives of the department of Lot, this correlation is not observed; this is probably due to the content which emits additional VOC's. The patterns of VOC's distributions inside 3 boxes held in the French national archives are similar. The VOC's identified are very varied, the most abundant are: straight-chains alkanes, BTEX (benzene, toluene, ethyl benzene, xylene), PAH (polycyclic aromatic

hydrocarbons) and phthalates; aldehydes and straight-chain saturated carboxylic acids, furfural, vanillin, which are characteristic of wood and paper degradation; cinnamic and benzenic derivatives which are indicators of beeswax. These last VOC's are detected in boxes which contain wax seals.

Table 1. Aldehydes and toluene concentrations in the atmosphere, in the boxes and in the fresh air, at the archives of Haute-Marne, Hautes-Pyrénées, Loire-Atlantique, Lot and Montpellier. At the archives of the National museums and of the Lot, additional VOC probably come from the content itself.

| | Ambient-box correlation coefficient | Remarks on air quality |
|---|-------------------------------------|---|
| Haute-Marne | 0,99 (empty box) | Valeraldehyde emitted by new books or magazines, solvents or particle board: 225 $\mu\text{g}/\text{m}^3$ in ambient compared to 0 $\mu\text{g}/\text{m}^3$ in fresh air. Some alkanes, derivative aromatic (trimethylbenzene, methylisobutylcetone, probably from recent paintings). |
| Ardennes | 0,97 (empty box) | High concentration of gaseous pollutants (equivalent to a hydrogen sulphide level higher than 10 ppb). |
| Loire-Atlantique | 0,91 (empty box) | Concentration of butyraldehyde higher than average but identical in ambient and fresh air intake (80 $\mu\text{g}/\text{m}^3$), hence no pollution indoor excepted for cyclic and aliphatic hydrocarbons with butanol and dodecane which may be derived from white spirit, glues, waxes, wood varnishes, carpets, carpets. In the box, glycol ethers (1-methoxy 2-propanol, 1-ethoxy 2-propanol), methyl methacrylate and furfural. |
| Archives of the French National museums | 0,62 (full box) | Acrolein, propionaldehyde, butanol, methyl methacrylate, butyl acetate and furfural in the can (30 $\mu\text{g}/\text{m}^3$) than in the atmosphere (10 $\mu\text{g}/\text{m}^3$). |
| Montpellier | 0,98 (empty box) | Internal pollution by sulfur. Valeraldehyde more concentrated in the box that was oiled (104 $\mu\text{g}/\text{m}^3$) than in the new air intake (40 $\mu\text{g}/\text{m}^3$) and in the environment (73 $\mu\text{g}/\text{m}^3$). In the environment, cyclic and aliphatic hydrocarbons with compounds that may come from white spirit, glues, waxes, wood varnishes, carpets, carpets (butanol, nonane, decane and dodecane). |
| Toulouse | - | Significant pollution outdoors. Cyclic and aliphatic hydrocarbons indoors, from white spirit, glues, waxes, wood varnishes, carpets, carpets. |
| Hautes-Pyrénées | 0,98 (empty box) 0,97 (full box) | Acrolein concentration higher than at other sites. In the atmosphere, furfural and butyl acetate, not detected in the box. |
| Lot | 1,00 (empty box) 0,30 (full box) | - |
| Puy-de-Dôme | 0,93 (empty box) | - |

3.4. Degradation of the reference papers

Being a hygroscopic material, paper is subjected to sorption and desorption cycles of humidity and gaseous pollutants, depending on the ambient thermohygrometric parameters [20-21]. The impact of pollutants on paper, whether these are outdoors or indoors generated, is still poorly understood. Recent research showed that formic acid is more aggressive towards paper-based collections than acetic acid, whereas aldehydes (formaldehyde, acetaldehyde, hexanal, furfural) have no deleterious effect [22].

At the end of the 14 months exposure period in the boxes, most papers (whether or not pre-oxidized) did not show any significant decrease in the degree of polymerization (DPv) nor any additional oxidation.

However, in two boxes, the highly pre-oxidized papers W40-0.26 showed a polymerization (DPv) drop of 23 % and 8 %, respectively, compared to the reference sample kept as control in the neutral cardboard box. The two boxes, their content, or their environment thus showed a significant degradation potential for paper. The repository ambient air is likely not the cause of this degradation since the other boxes that were in the same environment (same room) did not induce degradation in the reference papers. It is worth noting that the two 'aggressive' boxes were both made of poplar and that they had a varied materials content, paper-based for the most part, but also a certain number of wax seals in one of the boxes, and silk used as paper lining material in the other.

It is difficult at this stage to relate the decrease the degree of polymerization of cellulose to the VOCs measured in the boxes. It is thought that cross contamination between emissive materials and the paper may be the cause, some organic acids may have been produced besides formic and acetic. Despite the short exposure period, these results indicate a low deterioration potential of the old wooden boxes for paper based materials and evidence possible problems of cross contamination between vicinal mixed materials. Using sorbents in the mixed content boxes would possibly help to lower their deterioration potential.

4. Conclusions

Irrespective of their age, wooden archival boxes present no danger neither to the paper nor to the metals, on the one hand because they are not airtight, on the other hand because they emit little volatile organic compounds. The boxes offer good protection against dust and water. They were not infested by insects.

New passive samplers for organic acids have been tested. A methodology to assess the impact of pollutants on the paper collections was applied in true scale.

The dating method by dendrochronology has put the undeniable historical value of numerous boxes in to focus. For example, in Brittany and in Lorraine, where accurate and precise craftsmen made custom-made *layettes* to keep the treasures of the charters. Their know-how, until now underestimated, even perhaps ignored, is unique in Europe.

The species of wood used vary over time and in geography: oak wood in Nantes in the fifteenth and sixteenth centuries, as well as in Lorraine in the eighteenth and nineteenth centuries and fir wood in Avignon from the sixteenth to the nineteenth century, as well as in Lorraine and Puy-de-Dôme during the eighteenth and nineteenth centuries.

In Nantes, Clermont, Montpellier, Tarbes, the boxes have not been used for several years, but they are still carefully stored. Whereas in Cahors, Avignon, Châlons-en-Champagne, Charleville-Mézières or Toulouse, the boxes are still in use.

5. Acknowledgements

This study is dedicated to our friend and colleague John Havermans (Netherlands Organization for Applied Scientific Research) who passed away on Sunday 12 March 2017.

6. References

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* Radiello® is a radial diffusion passive sampler made up of a diffusive element in which a cartridge is inserted, all mounted on a support. The type of diffusive element and cartridge depends on the pollutants to be analyzed. The diffusion of the compounds depends on the thickness of the membrane and of its porosity, hence higher or lower flow rates. The compounds are desorbed by adding a solvent (CS₂) to be analyzed by gas chromatography.

† The principle of this technique is based on the adsorption of volatile compounds onto a fused silica fiber coated with a polymer phase inserted into a support about twenty centimeters in length. The fiber is placed close to the emissive material for a predetermined period of time ranging from a few hours to several days. The fiber on which the volatiles are adsorbed is then inserted into the injector of a chromatograph and the compounds are thermally desorbed, separated and then characterized by GC / MS.

‡ Silver and copper corrosion coupons (CCC) are used to assess the air quality in low corrosive environments in museums, archives and libraries. The corrosion products depend on the pollutants, and the corrosion rate depends on the level of contamination. After one month, the coupons were sent to the Purafil laboratories to be electrochemically analyzed. The report contains the photography of the coupon, the thickness of the corrosion film and the classes of corrosion. Most often, copper sulfide (Cu₂S) and oxides (Cu₂O), silver sulfide (Ag₂S) and silver chloride (AgCl) are identified. A data base for museums and archives is used as a benchmark for comparison.