Gas analysis: a bit of history

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EN ISO 12460-3, commonly known as **"Gas analysis",** is one of the fastest test methods by which wood-based materials (e.g., plywood, particleboard, fiberboard, etc.) can be analyzed for formaldehyde emission.

Formaldehyde is a **carcinogenic substance** contained in the adhesives with which these panels are produced; these glues are predominantly urea resins.
For reasons related to the chemical instability of these resins, formaldehyde **is released continuously** into the environment as a result of a chemical phenomenon called hydrolysis. It goes without saying, therefore, that it has become very important to be able to **measure the emission** of formaldehyde because national laws, and not only, have set limits of exposure even for living environments.

The first country in which the problem of formaldehyde was studied in depth was Germany.
In 1980 the first classification of panels according to their formaldehyde emission value was created, according to a method called the **Chamber Method,** still used today and considered the reference method: EN 717-1. The disadvantage of this test, however, is due to its timings, which vary from a minimum of 10 to a maximum of 28 days: consequently, the panel manufacturing industry needed to develop and use a faster method to analyze productions in almost real time.
Thus, two methods correlated to EN 717-1 were born: the **Perforator test** for raw panels and the **Analysis Gas test** for coated/finished panels and plywood, whose recent changes we are going to examine in depth. Correlated means in a reciprocal relationship: if a panel complies or does not comply with the limit value according to the Chamber test, it will also do the same at the Gas analysis and vice versa.
The Gas Analysis Method was developed between 1965 and 1973 by the precursor of the European Panel Federation (EPF), i.e. the European Federation of Associations of Particleboard Manufacturers (FESYP), which published a synthesis in 1969 under the title FESYP Gas Analysis Method. In the following years the standard was improved in collaboration with the Wilhelm-Klauditz-Institute, Fraunhofer Working Group for Wood Research (WKI), leading to the birth of the first version of the standard, in 1984 and in German, published by the German standardization body DIN under the name of DIN 52 368.
Ten years later, in September 1994, CEN Technical Committee 112 published the first English version of a standard that would be called EN 717-2. This standard, with the passing of the years and the normative development, in which I am honoured to have actively participated (**standardization is one of the many activities that CATAS carries out**, sending many of its collaborators to various technical tables), will then be adopted internationally as ISO 12460-3 in 2008; the latter will lead to the publication, in 2015, of EN ISO 12460-3, recently modified at the end of 2020.

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In this article we are going to look at the **main changes** made in the latest version of this technical testing standard.
But what is the principle behind this test? Summarizing it briefly, it is an **accelerated extraction of formaldehyde from the panel**: a sample, of known dimensions and with sealed edges so that the emission can derive only from its surfaces, is placed in a closed environment heated to 60°C; a constant and controlled flow of clean air enters the test equipment and drags any formaldehyde released into washing bottles that block it and allow its analysis.
This is done for 4 hours, with an automated change of washing bottles every hour. The wash bottles are filled with a defined amount of distilled water, thus using the high solubility of formaldehyde in this solvent. This water is recovered, bringing it up to volume in special long-necked bottles called flasks; an aliquot of this water is then mixed with two reagents that, in case of the presence of formaldehyde, combine with it creating a yellow substance. The more intense this yellow is, the more formaldehyde will have been emitted by the panel: with an instrument called spectrophotometer we will measure how much energy this colored solution absorbs (absorbance) when crossed by a radiation of a specific wavelength of 412 nanometers. Thanks then to the absorbance found and the calibration lines previously created from solutions with known concentrations, we can trace the unknown concentration of formaldehyde of our panel, relating it to the surface of the sample and the duration of sampling: thus we will find a result whose unit of measurement will be mg (of formaldehyde) /m2h.

Now we come to the **2020 update of the standard**.
The basic philosophy has not changed: if in 2015 the transition from EN 717- 2 to EN ISO 12460-3 had not had any substantial modifications, the reasons that have led to the recent normative update are to be found in the desire to improve the test method, making it more in step with a market that sees commercialized panels that have a lower and lower emission of formaldehyde.
A study conducted by the WKI between 2015 and 2017, called **OptiGas 2020**, involved six European panel manufacturers and was the project drawn upon to make the changes to the standard.
The goals of the project were 3:

1. reduce the testing time;
2. increase the sensitivity of the analytical procedure;
3. to validate the use of Analysis Gas for testing raw panels.

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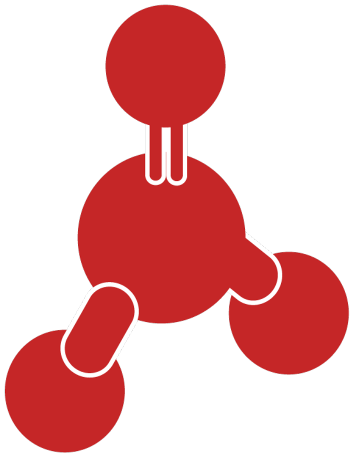
The request of the industrial production components to evaluate the shortening of the testing time brought several discussions to the working table of the technical standardization committee.
The debate that generated the highest tension was between those who wanted to make optional the reduction of the testing time to 3 hours (instead of the planned 4) and those who wanted it mandatory.
After several discussions, the conservative line of optionality prevailed, for two main reasons: the first is that gaining an hour is equivalent to a 25% reduction in time, but does not bring particularly significant advantages since, in a work shift, it is possible to do a maximum of 2 tests in series; the second reason concerned some types of high-thickness panels, where the accuracy of the result over 3 hours was lower than over 4 hours, since the panel heats up in much longer times than a thin one.



When performing laboratory analysis, one of the ways to know if the instrument we are using is not literally "going on the fritz" is to use a certified reference material, i.e. a material for which a third party guarantees one of its characteristics (amount present of a certain substance, for example).
The lack of a certified reference material to perform formaldehyde tests, undoubtedly, has not facilitated the goal of improving the sensitivity of the method, as we have had to rely on many repeated tests on the same panels. Unfortunately, formaldehyde emission results on the same matrices have some intrinsic variability: think about the fact that some panels can have an emission difference of up to 20% depending on the sampling area of the tested sample.
This difficulty did not prevent, however, the **definition of new options for the execution of the test**, for example, using less water in the washing bottles (thus concentrating the formaldehyde in a smaller volume) and even replacing the solvent in the same bottles, moving from distilled water to a solution where the reagents responsible for the yellow coloration were already present, the so-called mixed reagent, to avoid further dilutions.
Lastly, I cannot fail to mention that with this update, even though we are talking about a technical aspect that will only be understood by those in the industry, **a procedure has been defined for the conditioning of materials used for coating panels**, such as *finish* papers. Conditioning, in this case, is nothing more than a short period of maintenance of the material under test in pre-established conditions of temperature and humidity (7 days at 20±2°C and 65±5% relative humidity) in order to stabilize the conditions and improve the accuracy of the test.

I would like to end on a personal note.
I have personally experienced the development of this standard through my participation in the technical tables, but before that as a laboratory technician who has performed thousands of tests and literally put his hands inside the instruments and on the materials. We often run the risk of performing a test in an automatic way and, from a regulatory point of view, we risk letting a standard remain essentially unchanged without thinking about how it can be improved.
Instead, I have discovered that, **as the market and its products evolve, so does standardization**: certainly the timing is not and will never be as fast as the industrial ones, but all these years have taught me how important it is to sit at the tables of standardization, create links with people who, like you but in other countries, support the importance of providing the right tools to the people who have to use them, which in the first instance are the test laboratories and panel manufacturers, but in the end affect the lives of all of us, and do you know why?

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Because most of the furniture that is now in our homes is built with panels that have been tested with this standard as well.
Who would have guessed it: progress is sometimes made not only by simply lowering a legal limit for formaldehyde emission, but also by providing the best methods and tools to measure it.

Bibliography

1. DIN 52 368
2. EN 717-2, varie versioni
3. EN ISO 12460-3, varie versioni
4. EN 717-1
5. OptiGas 2020 Project https://www.wki.fraunhofer.de/en/departments/qa/ research-and-development/ research-projects/OptiGas2020-gas-analysis- formaldehyde-wood-based-materials.html

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