

The Development of Standard Methods Relating to Vehicle Interior Air Quality (VIAQ) and How to Comply With Them

Abstract

This Application Note discusses the introduction of regulations limiting the quantities of volatile and semivolatile organic compounds (VOCs and SVOCs) allowed in vehicle cabin air, and describes the evolution of harmonized, globally applicable standard methods for sampling and measuring them. The key technologies used for sampling and analysis in this area are also briefly described.

Concerns over VIAQ

Vehicle interior air quality (VIAQ) has been a topic of interest since the late 1970s, when concern over the effect of VOCs and SVOCs on the indoor environment began to be replicated in the automotive industry.

The release of VOCs and SVOCs from vehicle trim materials (including plastics, polyurethane, foam, wood, carpets, textiles, and adhesives) is the major factor causing poor VIAQ¹, and the consequent negative effect on health is the primary driver for regulations in this area. However, in China, concerns largely relate to consumers' dislike of off-odors in vehicle cabins, which has risen to be the most complained-about quality issue in that country².

Regulations relating to VIAQ

As a result of concerns over VIAQ, voluntary guidelines were developed to control acceptable levels of VOCs allowed in vehicle interiors. However, most manufacturers are now preparing to adhere to the mandatory Chinese regulation GB/T 27630³. Like its less rigorous equivalent guidelines in Japan⁴ and Korea⁵, this is government-led, in contrast to the EU and US, where limit levels have largely been defined by manufacturers or industry/regulatory bodies.

As is typical for VIAQ regulations, GB/T 27630 requires the concentration of a number of VOCs to be determined, and in this case, eight VOCs are listed (Table 1). Previously, it also stated the need for a total VOC (TVOC) concentration to be reported, and defined a strict ranking system, but it is now proposed to remove both these requirements.

Regardless of the country of manufacture, the responsibility for demonstrating conformance with specified limits for VOC levels in vehicle cabins (whether voluntary or mandatory) rests with the car manufacturer.

However, in general, a failure to meet these requirements is passed successively down the manufacturing chain to component manufacturers and raw material suppliers. This means that suppliers at all stages of the manufacturing chain need to be able to carry out testing of their products and materials to identify components causing high chemical emissions, and facilitate the development of low-emitting alternatives.

The development of standard methods relating to VIAQ

Over the years, hundreds of manufacturer-specific methods have been developed for the sampling and analysis of VOCs and SVOCs from car interiors. This proliferation of methods has become a major inconvenience for manufacturers, with regulators also challenged by the fact that the results from different protocols often cannot be meaningfully compared.

To simplify matters for the industry, the International Organization for Standardization (ISO) has been working on the development of harmonized methods through technical committee ISO/TC 146/SC 6 (Indoor Air). This has led to the release of six standards for sampling and analysis of VOCs and SVOCs from vehicle interiors and the materials used in them, with three further methods currently under development⁶. Table 2 summarizes those methods relating to the analysis of VOCs/SVOCs.

Table 1. Limit levels for VOCs defined within Chinese regulation GB/T 27630. The changes in the 2017 release also included changes to sampling conditions and reporting details.

Compound	Limit level (mg/m ³)		
	Prior to January 2016	January 2016	Since 2017
Benzene	≤0.11	≤0.06	≤0.05
Toluene	≤1.10	≤1.00	≤1.00
Xylenes	≤1.50	≤1.00	≤1.00
Ethylbenzene	≤1.50	≤1.00	≤1.00
Styrene	≤0.26	≤0.26	≤0.26
Formaldehyde	≤0.10	≤0.10	≤0.10
Acetaldehyde	≤0.05	≤0.20	≤0.20
Acrolein	≤0.05	≤0.5	≤0.5
TVOC	≤8	≤6	Removed
Ranking	A-E	A-C	Removed

Table 2. Standard ISO methods relating to sampling of VOCs and SVOCs relevant to VIAQ.

Sample	Analyte scope ^a	Sampling method	Method	Released
Cabin air	VOCs	Environmental chamber	ISO 12219-1	2012
Car trim	VOCs	Small sampling bag	ISO 12219-2	2012
Car trim	VOCs	Microchamber	ISO 12219-3	2012
Car trim	VOCs	Small chamber	ISO 12219-4	2013
Car trim	VOCs	Static chamber	ISO 12219-5	2014
Car trim	SVOCs	Small chamber	ISO 12219-6	2017
Car trim	VOCs	Large sampling bag	ISO/AWI 12219-9	Under development

^a Within the scope of ISO 12219, VOCs are defined as those with a boiling point above that of *n*-hexane (*n*-C₆H₁₄, b.p. 68 °C), but below that of *n*-hexadecane (*n*-C₁₆H₃₄, b.p. 287 °C); SVOCs are those boiling above the latter temperature.

However, in 2012, shortly before the release of the first of the ISO 12219 methods, it became apparent that the move for global harmonization might not be straightforward, due to the introduction of the mandatory Chinese regulation GB/T 27630³.

This regulation cites standard method HJ/T 4007 (which had been voluntary), and states the need to carry out VIAQ testing of all vehicles imported into China. This was viewed by many as a considerable barrier to trade, not only

due to the initial strict limit levels but also the logistics of shipping prototype cars to be tested in China at one of the few certified laboratories. Discussions are ongoing as to whether the regulation will allow self-certification, or whether certified laboratories outside China will be able to run the method.

A further difficulty is that, although HJ/T 400 is broadly similar to ISO 12219-1: namely, pumped sampling of cabin air within a large environmental chamber, differences in the conditions

under which sampling takes place means that the results from the two methods cannot be correlated with each other (Table 3). A similar situation arises with the widely used Japanese and Korean methods. Although not addressing this problem directly, the situation for manufacturers was somewhat alleviated in 2017 with changes to the limit levels in GB/T 27630 (Table 1), which are a result of US and EU negotiations with the Chinese regulators.

Table 3. Summary of the differences between the two key sampling methods for assessing VOC and SVOC levels in vehicle interiors (ISO 122191 and HJ/T 400), and the conditions stipulated by the forthcoming UN method. Conditions prefixed by (a) and (b) refer to separate samples acquired simultaneously.

Sampling mode	Condition	ISO 12219-1	HJ/T 400 (cited by GB/T 27630)	Forthcoming UN method
Ambient	Precondition time	1 hour	24 hours	>30 minutes
	Soak time	≥8 hours	16 hours	16 ±1 hours
	Sampling time	30 minutes	30 minutes	1) 30 minutes after 24 hours precondition; 2) 30 minutes after 16 hours soak in vehicle
	Sampling temperature	23 °C	25 ±1 °C	23.0–25.0 °C, as close as possible to 25.0 °C
	Vehicle doors	Open for preconditioning, closed for soak	Open for preconditioning, closed for soak	Closed (24 hours 30 minutes), then open (>30 minutes), then closed. Storage (near the test facility) for one day of soak time before measurement. Soak temperature as close as possible to room temperature, and between 20–30 °C.
	Sample tubes	(a) Sorbent tube (Tenax TA); (b) DNPH cartridge	(a) Sorbent tube (b) DNPH cartridge	1) Sorbent tube (Tenax TA); 2) Sorbent tube (Tenax TA) & DNPH cartridge
	Sample volume	(a) ≤3 L; (b) ≤30 L	(a) 3–6 L; (b) 3–15 L	3–6 L (sorbent tube); 12–30 L (DNPH cartridge)
Parking	Soak time	4 hours 30 minutes	Not required	four hours
	Sampling time	30 minutes		30 minutes
	Sampling temperature	23 °C; then door closed and radiator on for four hours to raise temperature to ~65 °C		25 °C
	Vehicle doors	Closed		Closed
	Sample tubes	Sorbent tube (Tenax TA)		DNPH cartridge
	Sample volume	(a) ≤3 L; (b) ≤30 L		2–30 L
	Sampling time	30 minutes		30 minutes
	Sampling temp.	A/C on, 23 °C		25 °C
Driving	Vehicle doors	Open	Not required	Open
	Sample tubes	(a) Sorbent tube (Tenax TA); (b) DNPH cartridge (b) DNPH cartridge		(a) Sorbent tube (Tenax TA);
	Sample volume	(a) ≤3 L; (b) ≤30 L		(a) 3–6 L; (b) 12–30 L

To resolve the differences between ISO 12219-1 and HJ/T 400, the matter was transferred to the United Nations, because China is not represented in the relevant body at ISO (TC 146/SC 6). This process began in June 2013 with a proposal from Korea to create a new global technical regulation (GTR) on VIAQ that would achieve consensus on testing methodology. Accordingly, an Informal Working Group on VIAQ was set up, which in July 2017 recommended new test procedures to the UN's World Forum on Harmonization of Vehicle Regulations⁸.

Developing a new method within this forum has the additional benefit of allowing the sampling and analytical conditions to be more detailed than is normally adopted in ISO standards. This is generally welcomed by manufacturers, because a highly prescriptive method improves reproducibility between results carried out by different laboratories, enabling more robust comparisons to be made. The global recognition attached to ISO standards makes it likely that, once the new UN regulation is adopted, it will be incorporated back into ISO 12219-1.

Sampling apparatus used in standard methods

The various standard methods used to assess VIAQ, or analyze VOC and SVOC emissions from component parts, all use thermal desorption (TD) to concentrate the vapors to a point where they can be detected by gas chromatography (GC), with detection by mass spectrometry (MS) or flame ionization detection (FID)⁹.

In all these cases, vapors are collected from the sampling device onto an adsorbent-packed sample tube (Figure 1). This tube is then placed into the thermal desorber, and the compounds are transferred onto a narrow, cooled focusing trap. Rapid heating of this trap in a reverse flow of carrier gas transfers the VOCs and SVOCs to the GC instrument in a narrow band. This ensures sharp peaks and high levels of sensitivity. The benefits of TD are numerous, and are discussed in more detail in Markes International's Application Note 012.

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Figure 1. 3½" x ¼" adsorbent-packed TD tubes (here shown with brass storage caps) can be used for a wide variety of applications, including the sampling of vehicle air and emissions from car-trim materials.

These TD–GC-based methods are directly applicable to five important sampling methods for VIA-related work. These are described briefly in Table 4.

It is worth pointing out a few considerations regarding these sampling methods:

- Environmental chambers and small chambers are typically used for product certification, and are often seen as the gold standard of material emissions testing due to the ability to test assemblies. However, such tests are typically lengthy, and the chambers are relatively expensive. The size of the chambers can also result in significant wall effects, whereby SVOCs stick to the sides of the chamber.

- Bag sampling is particularly popular in Asia, and although quicker than small chamber certification methods, it is also compromised by wall effects. Some concerns have also been raised about the cleaning procedures used, and chemicals emitted from the bag material itself.




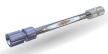
Microchambers such as Markes' Micro-Chamber/Thermal Extractor can help solve the above issues, by:

- Handling a wider analyte range than bag sampling
- Being small enough to reduce wall effects
- Allowing rapid sampling
- Improving on reproducibility in interlaboratory studies

In addition, because they use the same extraction technique, emissions data from microchambers can be correlated with that from environmental and small chambers, allowing prediction of the outcome of longer-term certification tests (see Application Note 069). As a result, microchambers are now recognized as an ideal method for emissions screening and quality control—see Application Note 073 (a peer-reviewed study by the Fraunhofer Wilhelm-Klauditz-Institute) and Application Note 093 (analysis of car trim in accordance with ISO 12219-3).

Direct desorption, although quick and useful for screening of small, homogeneous samples, has not yet been translated into a harmonized ISO method. However, the relevant standard method (VDA 278) remains popular, and details of carrying out direct desorption in accordance with this method can be found in Application Note 059.

Table 4. Sampling approaches used for monitoring VIAQ and related emissions from vehicle components and materials. All GC analyses were carried out in accordance with ISO 16000-6,¹¹ which stipulates either MS or FID for detection.

Purpose	Sampling method	Description of analytical process	Methods
Overall VIAQ	Environmental chamber	<ul style="list-style-type: none"> Air sampled from a chamber enclosing the whole car Sampling onto a sorbent-packed TD tube Tube desorbed and vapors collected on a focusing trap Trap desorbed and vapors injected into GC 	<ul style="list-style-type: none"> ISO 12219-1 China: HJ/T 400 Japan: JASO Z125 South Korea: Article 33-3
Emissions from assembly parts	Small chambers 	<ul style="list-style-type: none"> Air sampled from a (typically) 1 m³ chamber Sampling onto a sorbent-packed TD tube Tube desorbed and vapors collected on a focusing trap Trap desorbed and vapors injected into GC 	<ul style="list-style-type: none"> ISO 16000 9 ISO 12219-4 ISO 12219-6 VDA 276 JIS A1901 BMW GS 97014-3 (Summer test) ASTM D5116-97
	Sampling bags 	<ul style="list-style-type: none"> Air sampled from bags of various sizes from 10 L to 2,000 L Sampling directly into a focusing trap Trap desorbed and vapors injected into GC 	<ul style="list-style-type: none"> Japan: JASO M902 MS300-55 (Hyundai-Kia) NES M0402 (Nissan) ATSM 0508G (Toyota) DWG 0094Z SNA 0000 (Honda) ISO 12219-2 (10 L bag) ISO 12219-9 (2000 L bag)
Emissions from components	Microchambers 	<ul style="list-style-type: none"> Air/gas sampled from micro-scale chambers (44 or 114 cm³) Sampling onto a sorbent-packed TD tube Tubes desorbed and vapors collected on a focusing trap Trap desorbed and vapors injected into GC 	<ul style="list-style-type: none"> TPJLR.52.104 (Jaguar Landrover) ISO 12219-3 ASTM D7706 GMW17082 (General Motors)¹⁰
	Direct desorption 	<ul style="list-style-type: none"> Small sample (up to ~50 mg) heated in an empty TD tube Vapors collected on a focusing trap Trap desorbed and vapors injected into GC 	VDA 278

^a Image credit: SP Technical Research Institute of Sweden. ^b Image credit: Equipco.

Conclusions

Rising awareness of the potential health effects of exposure to volatile chemicals in car cabin air, and pressure from consumers for odor-free car interiors, has driven the development of many standard methods for assessing emissions of VOCs and SVOCs.

Methodology such as ISO 12219-1 for sampling and analyzing chemicals in vehicle air has proved particularly problematic, but it is hoped that the forthcoming UN standard will, for the first time, achieve global consensus on this vital aspect of automotive monitoring. The situation for monitoring emissions from assembly parts and component materials is more straightforward, with ISO 12219-2 to -6 now widely used across the industry.

This move towards full harmonization is already benefitting the automotive industry, by making it much easier for manufacturers to sell their products globally and to develop the lower-emitting materials demanded by customers. The TD-GC/MS technology used in these harmonized methods, such as the Markes TD100-xr instrument (Figure 2), is key to achieving the robust performance needed to analyze all the chemicals and concentrations of interest. At the same time, the range of compatible sampling accessories provides manufacturers with the flexibility to deal with a wide variety of sample types and sizes.



Figure 2. Markes TD100-xr 100-tube automated thermal desorber.

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8. Documents produced by the UN's Informal Working Group on VIAQ can be obtained at: <http://wiki.unece.org/pages/viewpage.action?pageId=25266269>.
9. An exception to the use of the TD–GC approach is when needing to monitor the reactive chemical formaldehyde. In this case, vapours are collected onto cartridges packed with dinitrophenylhydrazine (DNPH), and the resulting stable hydrazone derivative is then analysed by high-performance liquid chromatography (HPLC). For reasons of convenience, a small number of less volatile aldehydes are often also monitored using this technique.
10. This method uses a nontypical procedure that is in essence a version of the direct desorption method VDA 278, but carried out in the μ -CTE.
11. ISO 16000: Indoor air. Part 6: Determination of volatile organic compounds in indoor and test chamber air by active sampling on Tenax TA sorbent, thermal desorption and gas chromatography using MS or MS-FID, International Organization for Standardization, **2011**, www.iso.org/standard/52213.html.

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