

Nissan Engineering Standard

N E S	VOC Test Method of Cabin Parts
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1. SCOPE

This Standard stipulates the measurement method of VOC (hereinafter referred to as the test method) for the interior parts (hereinafter referred to as parts) and materials, including cut-out parts. Method #1 is defined as the method in which VOC is measured using a part assembly unit and Method #2 is defined as the method in which VOC is measured using materials themselves or Samples cut out from production parts. The method for other parts is according to agreement between the parties concerned. In addition, these methods may be used for measuring the parts and materials other than the classification examples shown in Tables 1 and 2.

* Classification of VOC

- Aldehyde: Formaldehyde, Acetaldehyde, etc.
- VOC: Toluene, Ethylbenzene, Xylene, Styrene, P-dichlorobenzene, Tetradecane, Chlorpyrifos, Di-n-butyl phthalate, Di-2-ethylhexyl phthalate, Diazinon, Fenobucarb.

Table 1. Parts to which Method #1 is applied

Items	Parts	Details	NDS standards
(1)	CPM	INST UPR/LWR, G-BOX, CLUSTER C, CTR CONSOLE, etc.	68050NDS01, 68200NDS00, 68240NDS00, 24810NDS00
(2)	SEAT	SEAT, LEG COVER, etc.	87000NDS01
(3)	DOOR TRIM	DOOR UPR, DOOR LWR, ARM REST, etc.	80900NDS00
(4)	B/S TRIM	FIN FR PLR, CTR PLR UPR/LWR, etc.	76990NDS00
(5)	LUG TRIM	LUG SIDE UPR/LWR TRIM, CARGO COVER, etc.	76990NDS00
(6)	RR PSHELF TRIM	RR PSHELF TRIM, etc.	79910NDS00, 79910NDS010
(7)	HDLNG	HDLNG, SUNVISOR, MAP LAMP, etc.	73910NDS00, 96400NDS00
(8)	FLOOR CARPET	FLOOR CARPET, FLOOR SPCR, etc.	74902NDS00
(9)	BODYSIDE WELT	BODYSIDE WELT, WEATHER STRIP, etc.	76921NDS10
(10)	STRG WHEEL	STRG WHEEL, AIRBAG, etc.	48400NDS00, 48400NDS20
(11)	SUNROOF SHADE	SUNROOF SHADE, etc.	91200NDS00
(12)	DASH INSHLATOR	DASH INSULATOR, etc.	67900NDS00

Table 2. Examples of materials to which Method #2 is applied

Items	Examples of materials	Items	Examples of materials
(1)	Skin materials (genuine leather, cloth, a class of artificial leather)	(6)	Painted Parts
(2)	Adhesive, pressure sensitive adhesive	(7)	Materials for vehicle bodies, such as damping materials, etc.
(3)	Plastic Parts	(8)	Parts with tape applied to them
(4)	Rubber, etc.	(9)	Others (materials that emit substance of guideline 13 and are considered to influence the passenger room)
(5)	Felt, etc.		

2. EQUIPMENT

The equipment specifications applied to this test are shown in Table 3.

Table 3. Equipment and specifications

Equipments	Required performance	
	Method #1	Method #2
Test chamber	The temperature in the bag shall be increased to 40°C or 60°C within 0.5 to 2.0 hours. The time (about 5 hours) including the collection time shall be able to be maintained. Contaminated substances shall not remain in the chamber.	The temperature in the bag shall be increased to 40°C or 60°C within 0.5 to 2.0 hours. The time (about 3 hours) including the collection time shall be able to be maintained. Contaminated substances shall not remain in the chamber.
Film for test bag	Shall be made from polyvinyl fluoride (PVF) (t=0.05 mm) with a capacity of 2,000 liters.	Shall be made from polyvinyl fluoride (PVF) (t=0.05 mm) with a capacity of 10 liters
Collection valve	Shall be made from Teflon. It shall be easy to open or shut and be free from leakage. Structure to which a Teflon tube can be mounted.	Shall be made from Teflon. It shall be easy to open or shut and be free from leakage. Structure to which a Teflon tube can be mounted.
Collection pump	Shall maintain the speed, amount of collection and time shown in Table 6.	Shall maintain the speed, amount of collection and time shown in Table 6.
Welder	Shall be able to weld the opening portion of film so that a leak cannot occur.	Shall be able to weld the opening portion of film so that a leak cannot occur.
Collection tube	Aldehyde	DNPH Silica
	VOC	Tenax-TA: Porous polymer beads Tenax-GR: Blending Tenax-TA with graphite carbon
Sampling tube	Shall be made from Teflon.	Shall be made from Teflon.
Protective Tape (to protect bag against rips, tears and punctures from parts)	Shall be made from Teflon. (VOC shall not be emitted from pressure sensitive adhesives.)	Shall be made from Teflon. (VOC shall not be emitted from pressure sensitive adhesives.)
Suction pump	—	Air in the bag shall be sucked out.

3. TEST PRODUCT

3.1 Handling before the test and storage period

Handling of the test product and storage period are shown in Table 4.

Table 4. Size of the test product, storage condition and storage period

Items	Method #1	Method #2
Size	Parts assembly (Refer to applicable parts in Table 1.)	8,000 mm ² (80 × 100 mm) is a base as an area. When the area is not secured, control the weight.
Stored period and storage condition to the test	After assembling the parts, start measuring after leaving them for 7 days in the conditions of the temperature of 20±5°C and relative humidity of 50±5%. * Remove all extra materials (protective film, packaging, etc.) before conducting the test	After manufacturing materials, start measuring after leaving them for 7 days in the conditions of the temperature of 20±5°C and relative humidity of 50±5%. * Features of materials (production date, compounding, etc.) are clearly described.
Period from the production date of each component material to the test	Plastic molding parts: Conduct the test within 3 days after the molding. Skin: Assemble within 3 days after the production. Urethane pad: Assemble within 3 days after molding (foaming). Adhesive: Assemble within 3 days after the application. Adhesive tapes: Assemble within 3 days after removing the release liner. Note: Only applies to materials containing any of the 13 guideline value substances	—

4. TEST CONDITIONS

4.1 Temperature conditions

Temperature conditions in this test are shown in Table 5. However, the temperature condition shown in Figure 1 shall be satisfied.

Table 5. Temperature conditions

	Method #1		Method #2	
	INST ASSY, RR PSHELF	Parts other than parts mentioned on the left	Materials used for INST ASSY, RR PSHELF	Materials used for parts other than parts mentioned on the left
Temperature in the bag	60±2°C	40±2°C	60±2°C	40±2°C
Retention time	4.5±0.02h	4.5±0.02h	2.0±0.02h	2.0±0.02h

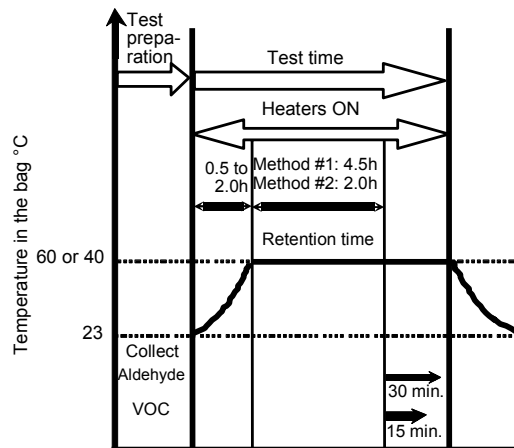


Figure 1 Temperature conditions

4.2 Collecting conditions

Collecting conditions in this test are shown in Table 6.

Table 6. Collecting conditions

Items		Aldehyde		VOC	
Collection method	Collection tube		DNPH cartridge	Tenax-GR/TA	
	Analysis		HPLC	GC-MS	
Collecting conditions	Method #1	Parts	Speed	0.8L/min.	0.2L/min.
			Collection time	30 min.	15 min.
			Amount of collection	24L	3.0L
	Method #2	Material (Cut-out parts)	Speed	1.0L/min.	0.1L/min.
			Collection time	2 min.	10 min.
			Amount of collection	2.0L	1.0L
Quantity of collection			1 piece	1 piece	

5. TEST METHOD

In this test, Method #1 shall be applied to the parts VOC and Method #2 shall be applied to the test for the materials and cut-out parts.

5.1 Method #1

5.1.1 Preparation of test

(1) Preparation of bag

If the bag cannot be obtained, it shall be made according to the following procedures (the specification shall comply with Table 3). Prepare the film (Available from film manufacturers, ex. Dupont) and the collection valve (Available from valve manufacturers, ex. AS ONE) in advance. Do not perform operation under the environment in which it is considered that VOC influences the surroundings.

1. Preparation of film

Prepare two pieces with a size of 1.0 × 5.0m.

2. Oven heating of film

Put the film prepared in the size of 1.0 × 5.0m in the oven and perform oven heating in the condition of 140°C × 4h.

<Setting method of film at oven heating>

- Fold the side of 1.0 m into the width of 50 cm (Figure 2).
- Fold the side of 5.0 m into the width of 50 cm and hang the film on the shelf board of the oven using clips (Figures 3 and 4).

Ensure that the surfaces of the film do not contact each other in order to perfectly eliminate blank adhering to the film at this time. The oven used for oven heating is desired to be an exclusive facility (this is because substances adhering to the inside of the oven may contaminate the film.).

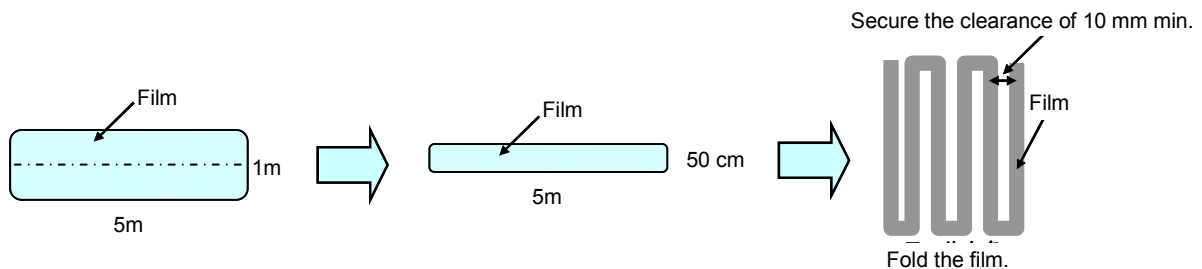


Figure 2. How to fold the bag



Figure 3. Hanging of the film



Figure 4. Putting the film in the oven

3. Sealing of film

Overlap two pieces of the film and weld the single side of the sides of 5.0m using the sealer (Figure 5). Two pieces shall be overlapped at this time so that the surfaces that were on the outside when oven heating was performed become the inside. Extend the film and fold it into the half size so that the size is 2.5m × 2.0m. Weld the single side of the sides of 2.0m and 2.5m (Figure 6). Although the width of the seal is according to agreement between the parties concerned, it shall be the width in which there is no leak. For the portion that is welded at the side of 5.0m, fold it back in the right and left direction and weld it twice because a leak is especially easy to occur at this portion (Figure 7).



Figure 5. The side of 5m is welded using the sealer.

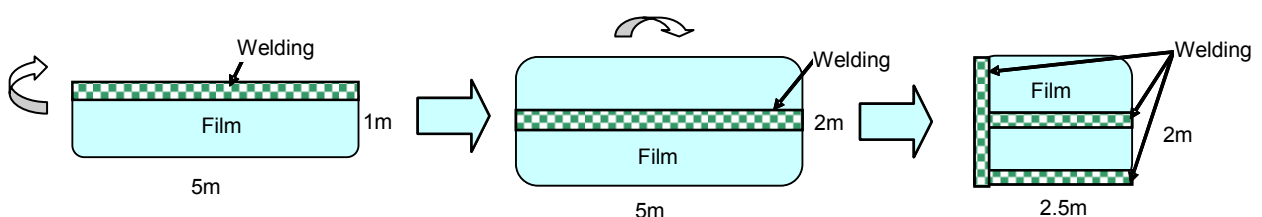


Figure 6. Flow of sealing



Figure 7. Welding is performed twice to prevent a leak.

4. Attachment of valve

Make two holes of $\Phi 6$ (Figure 8). Attach the body of the valve (Figures 9 and 10).

The rubber packing shall not be put in the inside of the bag so that volatile matter of the packing does not influence.



Figure 8. Hole for attachment of valve



Figure 9. Attachment of valve

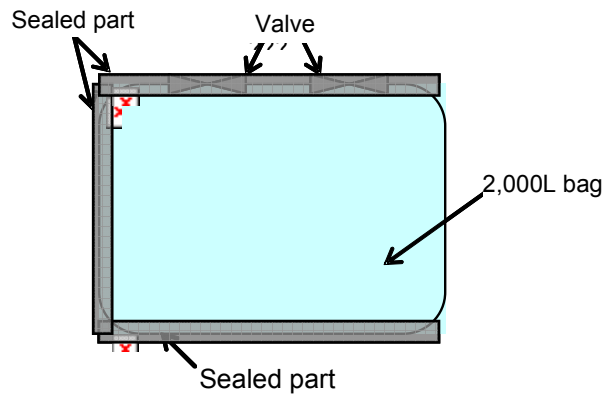


Figure 10. Pattern diagram of the bag sealed a 3 places

5. Check of sealing portion

Check whether there is poor welding in order to prevent a leak from the sealing portion.

(2) Preparation of test products

Wrap the projected section of the test product using protective tape to prevent the bag from being damaged. Prepare a second bag for the background, which will not contain a test part, but instead will be filled only with air. Media collecting air from this bag will be used as the control measurement data.

(3) Enclosing of air in the bag

Prepare the bag with a capacity of 2,000L to which the valves are sealed at 3 places for collection and are mounted (Figure 11). Place the test part in the bag and attach the Teflon tube to the collection valve. Seal 3/4 of the bag opening and start adding some of the ambient air from the test chamber (be sure to clarify the amount of air). After the air is added, seal the remaining portion as shown in Figure 12. The background bag should be prepared similarly, with no test part included inside of it (be sure to clarify the amount of air added into the background bag as well).



Figure 11. Condition in which the bag of 2,000L is prepared in the test chamber

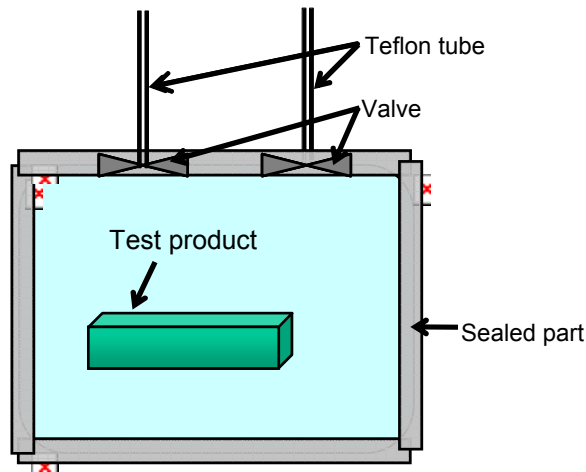


Figure 12. Pattern diagram of the bag sealed at 4 places

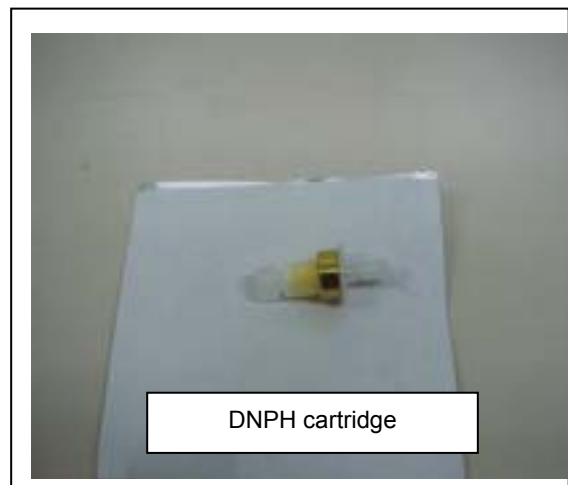
(4) Preparation of related equipment

Prepare the pump for analysis and collection tubes. Use the Tenax suction tubes for collection that is generally used for collecting the organic volatile substances (Figure 13). Where Tenax GR or Tenax TA tubes are unavailable, other suction tubes may be used. Use the DNPH cartridges that are generally used for collecting Aldehyde (Figure 14). Other equipment is acceptable if there is no problem in collecting Aldehyde and fixed quantity (Figure 15). However, collection of the background may as well be carried out at the same time when the test of the test product is carried out if the air similar to the air obtained when preparing the test product is enclosed in the bag for the background.



Tenax tube

Figure 13. Tenax tube



DNPH cartridge

Figure 14. DNPH cartridge

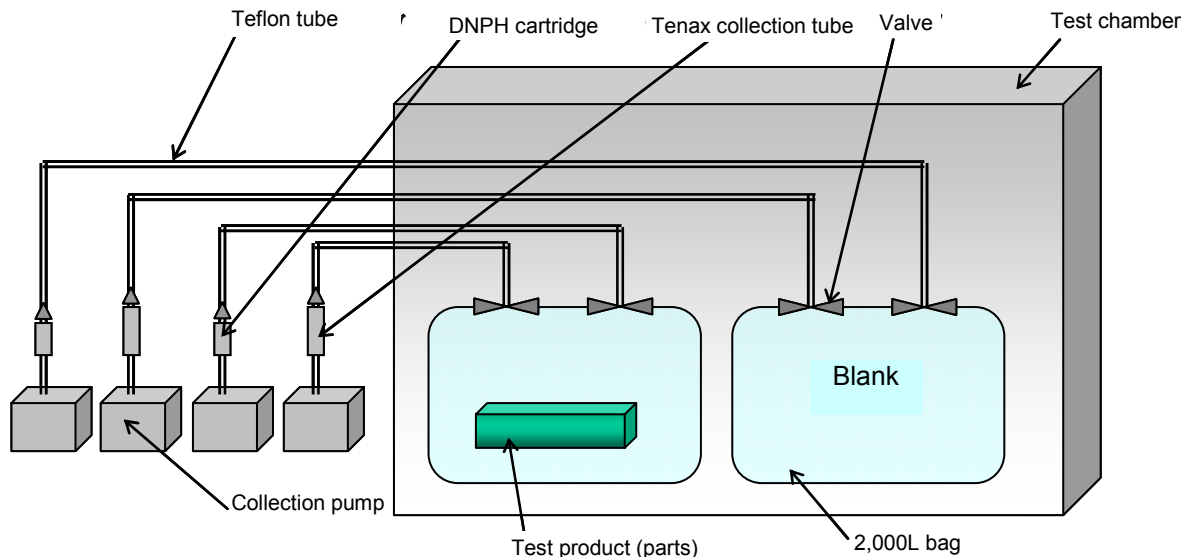


Figure 15. Pattern diagram of connection for the collection method of aldehyde and VOC of parts of Method #1

5.1.2 This test

(1) Temperature increase

The parts temperature in the bag shall be increased according to the temperature conditions in Table 5.

(2) Retention

After the temperature in the bag has increased to 40°C or 60°C, maintain it for 4.5 hours (Figure 16). For measurement of the part temperature, Use a black panel or similar thermocouple that will not interfere with the test sampling.



Figure 16. Retention condition in Method #1

(3) Collection

After 4.5 hours have elapsed, Begin pumping air from the sample bags without the collection tubes attached. The first 100 milliliters of air should be disposed of before installing the collection tubes. Then, mount the collection tubes to the pump and collect Aldehyde or VOC according to the conditions shown in Table 5.

(4) Treatment after collection

Immediately after collection, seal the Tenax tube and DNP cartridge. Store the adsorption tubes after collection in the dark cold place for the Tenax tube and in the cold storage (freezer storage is not acceptable) for the DNP cartridge and analyze them as soon as possible after collection. The time period for sample storage shall not exceed 3 days.

5.2 Method #2

5.2.1 Preparation of test

(1) Preparation of bag

If the bag is difficult to be procured, make it according to the following procedures (the specification shall comply with Table 3). Prepare the film (Available from film manufacturers, ex. Dupont) and the collection valve (Available from valve manufacturers, ex. AS ONE) as materials.

1. Preparation of film

Prepare the film with the size of 500 × 700 mm.

2. Oven heating of film

Put the film prepared in the size of 500 × 700 mm in the oven and perform oven heating in the condition of 140°C × 4h.

3. Sealing of film

Seal the film so that the size of the film becomes 500 × 350 mm.

4. Attachment of valve

Make a hole of $\Phi 6$ (Figure 8). Attach the body of the valve (Figures 9 and 10).

(2) Preparation of test products

Cut the test products to pieces with 80 × 100 mm in size. When it is inapplicable to cut to pieces with 80 × 100 mm in size, cut it with an area of 80 cm² or controlled by weight.

(3) Bag filling

Place the test products in the bag and seal the bag. Then, evacuate the air in the bag.

(4) N₂ gas filling

1. Fill the bag with 4 liters of N₂ gas

2. Evacuate the gas in the bag

3. Repeat Steps 1. and 2. two additional times (such that the N₂ gas has been evacuated three times)

(5) Preparation of related equipment

Prepare the pump for collection and collection tubes. Use the Tenax suction tubes for collection that are generally used for collecting the organic volatile substances (Figure 13). Though the Tenax GR or Tenax TA is used in principle, other suction tubes are acceptable to use when these are unavailable. Use the DNPH cartridges that are generally used for collecting Aldehyde (Figure 14). Other equipment is acceptable if there is no problem in collecting Aldehyde and fixed quantity (Figure 17).

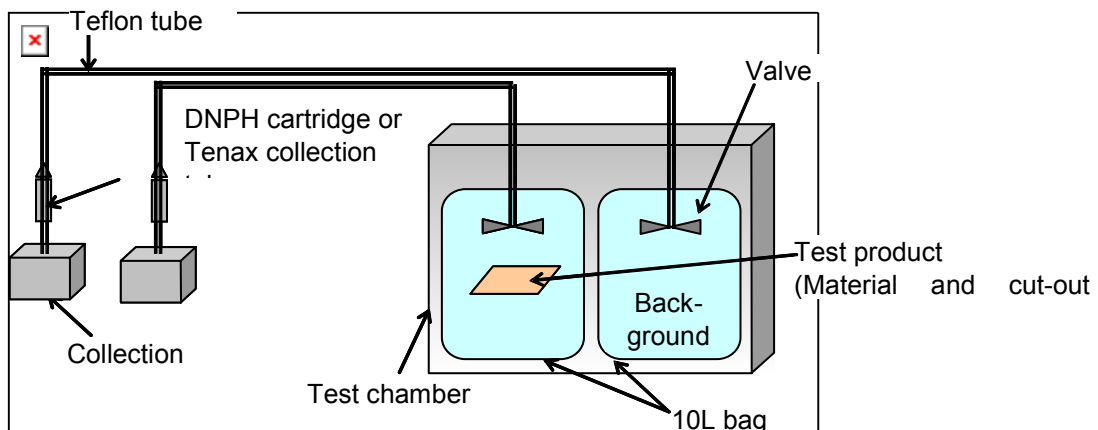


Figure 17. Pattern diagram for collection of aldehyde and VOC of materials and cut-out parts of Method #2

5.2.2 This test

(1) Temperature increase

The temperature shall be increased according to the temperature conditions in Table 5.

(2) Retention

After placing the bag in the test chamber at 40°C or 60°C keep it for 2.0 hours.

(3) Collection

After 2.0 hours have elapsed, begin pumping air from the sample bags without the collection tubes attached. The first 100 milliliters of air should be disposed of before installing the collection tubes. Then, mount the collection tubes to the pump and collect Aldehyde or VOC according to the conditions shown in Table 5. At this time, maintain the temperature in the test chamber at the specified temperature (Figure 18).



Figure 18. Method #2 Appearance in the test chamber

(4) Treatment of the collection tubes

Immediately after the collection, seal the Tenax tube and DNPH cartridge. Store the adsorption tubes after collection in the dark cold place for the Tenax tube and in the cold storage (freezer storage is not acceptable) for the DNPH cartridge and analyze them as soon as possible after collection. The time period for sample storage shall not exceed 3 days

6. ANALYSIS METHOD

The analysis method is described below. If the analysis companies other than Nissan Arc Co., Ltd. analyze, it is necessary to get analysis correlation between the company and Nissan Arc Co., Ltd. (this is because data that is measured by Nissan Arc Co., Ltd. is a base). Other methods are acceptable if correlation between this method and other methods is clarified.

<Analysis method>

- 1) Set collection tube (for HPLC, inject DNPH cartridge eluate).
- 2) Start analysis.
- 3) Analyze the obtained data.

6.1 Analysis condition

Examples are shown in Tables 7 and 8. Equipment for analysis other than those shown in Tables 7 and 8 is acceptable when a range from hexane to tetradecane can be detected.

Table 7. Examples of aldehyde analysis condition

Item	Example 1	Example 2
Analyzer	HPLC (Waters make 2690 type)	HPLC (Shimadzu Seisakusho Ltd make LC-10)
Moving phase	H ₂ O/CH ₃ CN=45/55 (%)	H ₂ O/CH ₃ CN=50/50 (%)
Flow volume (cc/min.)	1 cc/min.	1 cc/min.
Gradient	CH ₃ CN (10 min.) while stabilizing for 20 minutes	H ₂ O/CH ₃ CN=20/80 (30 min.) while stabilizing for 5 minutes
Column	Zorbax ODS 4.6Φ × 250 mm	Zorbax ODS 4.6 mm (15+25 mm)
Column temperature (°C)	40°C	50°C
Detector	UV/VIS	UV/VIS
Wavelength (nm)	360 nm	365 nm
Analysis cycle	50 min./1 sample	45 min./1 sample

Table 8 Examples of VOC analysis condition

Item	Example 1	Example 2	
Analyzer	Pretreatment	Gestel Ltd-made TDSA/CIS4	TurboMatrix ATD
	Analysis	GC-MS	GC-MSD
	Split ratio	50 : 1	150 : 1
Requirements for the collection tubes	Adsorbent	Tenax-GR	Tenax-GR
	Temperature at installing/removing	280°C	270°C
	Flow volume at installing/removing	203 ml/min.	30 ml/min.
	Time of installation/removal	8 min.	10 min.
	Inlet split	—	420 ml/min.
Trap tube condition	Adsorbent	Quartz glass wool	Tenax-TA
	Trap temperature	-150°C	-30°C
	Temperature at installing/removing	300°C	270°C
	Time of installation/removal	7 min.	10 min.
	Outlet split	—	10 ml/min.
Analysis condition	Column	HP-5MS 5% Phenyl Methyl Siloxane 60 m × 0.25 mmΦ × 0.25 μm	HP-5MS 5% Phenyl Methyl Siloxane 30 m × 0.25 mmΦ × 0.25 μm
	Column temperature	40°C, 5 min. → 10°C/min. → 270°C, 7 min.	40°C, 5 min. → 10°C/min. → 270°C, 7 min.
	Detector	MSD	MSD
	Detector temperature	280°C	280°C
	Mode	SCAN	SCAN
	Mass range	25-550	40-300
	Carrier gas flow volume	1 ml/min.	1 ml/min.

6.2 Analysis result display method

Indication method of the analysis results is shown in Table 9 respectively. The analysis results of the bag for the blank ⁽¹⁾ shall be reported.

Note (1): The blank bag in Method #1 indicates the air property in the test chamber and that in Method #2 indicates the empty bag in which N₂ gas is filled.

Table 9. Analysis result display method

Title		<Example of filling in>		
Purpose		Synthetic leather A		
Sample name		Understanding of current situation of skin of synthetic leather		
Sample area (mm ²)		Skin of synthetic leather A		
Sample weight (g)		8,000 (80 × 100)		
Sample weight (g)	Before sampling	5.423g		
	After sampling	5.410g		
Supplier name		A Co., Ltd.		
Material structure		PVC + backing cloth		
Measure contents		Change of adhesive		
Model		SERENA		
Sample creation condition		Current part produced in the production line		
Sample creation date		Nov. 1, 2005		
Sample analysis date		Nov. 8, 2005		
Storage condition from sample creation to analysis		Setting at 23°C		
Condition/shape when sampling (both sides, surface)		Both sides		
Sample analysis condition		40°C × 2h		
Measuring result (μg/m ³)	Material name	BG data	Measuring result	(Measuring result) – (BG)
	Formaldehyde	15.6	16.1	0.4
	Acetaldehyde	23.3	65.5	42.2
	Toluene	4.1	167.2	163.1
	Ethylbenzene	0.8	60.5	59.6
	Xylene	1.1	179.6	178.5
	Styrene	0.3	8.5	8.3
	p-dichlorobenzene	0.4	2.1	1.8
	Tetradecane	0.0	6.1	6.1
	Chlorpyrifos	—	—	—
	Di-n-butyl phthalate	—	—	—
	Di-2-ethylhexyl phthalate	—	—	—
	Diazinon	—	—	—
Fenobucarb	—	—	—	
TVOC	558.1	8,750.2	8,192.1	

6.3 Handling of analysis result

The result of Method #1 may be predicted not needed based on the result obtained in Method #2. For example, the result of Method #1 may be predicted not needed by converting the result obtained in Method #2 using a specific factor (it is necessary to get correlation between Method #1 and #2 in advance.).