

PATAC STANDARD

TS-BD-003

车内零部件挥发性有机化合物的测试方法——袋子法

Determination of Volatile Organic Compounds from Vehicle Interior Parts

----Tedlar Bag Method

1. Scope

The method for sampling and determination of VOCs from vehicle interior parts assembly with Tedlar bag method is specified herein.

2. References

GMW15654 GMW15600 HJ/T 400-2007

3. Determination of compounds

TVOC, benzene, methylbenzene, dimethylbenzene, ethylbenzene, styrene, formaldehyde, acetaldehyde, acrolein and acetone will be determined according to Guideline for Air Quality Assessment of Passenger Car (GBT 27630—2011).

4. Laboratory equipment

- TD-GC-MS (Thermal Desorption-Gas Chromatography-Mass Spectrometry)
- HPLC (high performance liquid chromatography)
- walk-in high and low temperature test box: air-cycling with temperature controlled within $\pm 2^{\circ}$ C
- The sampling bag with 2000L: PVF film with the thickness of 0.05mm and with at least double on/off valves;
- The bag can be repeatedly used for no more than 5 times when requirement is met for the blankness;
- The sampling valve: Teflon, can be switched on/off, no leakage of any gas, be connectable with Teflon pipe
- constant air sampling pump (to be calibrated by flow meter before being used for sampling)
- sealed-in unit for sampling bag: pedal sealer, SNS sealing strip
- Sampling tube: DNPH tube used for sampling carbonyl compound and Tenax tube for VOCs and benzene compounds
- gas flow calibrator: used to calibrate the flow of sampling pump
- Dry gas flowmeter
- Diaphragm vacuum pump
- Sampling conduit: Teflon conduit, transparent PTEF sampling pipe with outer diameter as 6mm and inner diameter as 4mm; to be heated for 6 hours in clean air at 100 °C before use.
- Connecting pipe for sampling: made of silica gel with inner diameter as 6mm and used for connecting sampling conduit and sampling pump.

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2012/5/24		
Release date: 2012/5/30	IMPL Date: 2012/5/30	
Version:001		

5 Samples

5.1 Pretreatment

When emission of VOCs is determined, the VOCs under test should be kept from absorbing any other VOCs from the environment. Therefore, the state of samples and the environmental conditions before test will be recorded in detail, such as date of production, composition, and temperature and humidity of the place where it is placed and the location.

5.1.1 Packaging, transportation and storage of determined samples

The samples should be sent to the lab for determination within 7 days after production, and will be packaged with intact aluminum foil first and then sealed with polythene bag/film before being put into the paper carton to ensure that they are kept in sealed conditions. The lab should inspect and take a picture of packaging and production date as soon as the samples arrive. In case of any disqualified packaging or samples being produced for more than 15 days, a timely notice will be given to OEM or spare parts supplier for resending samples. Where samples can not be tested within a specified period of time, they will be kept in a condition at 22±3°C and with a humidity of 50±5% for no more than 7 days (included weekends but long holidays). The name of samples, spare parts No., sizes and weight of sampled parts, materials composition, date of production, date of sample collection, date of sample making, date of test and conditions where they are kept will be recorded in detail.

5.2 Classification of sampled parts and sampling requirement

5.2.1 Vehicle interior parts assembly and sampling requirement

The spare parts under test will be tested with parts assembly as a whole and based on the principle of the side exposed to the air facing upward and all parts being spread out as much as possible.

Bags with 2000L will be used for all parts. Please refer to table 1 for detailed car interior parts assembly and sampling requirement.

Serial No.	Assembly type	Parts	Sampling	Volume of bag
			requirement	
1	Instrument	Instrument	With coat of	2000
	panel assembly	panel	instrument	
		assembly/decor	panel, central	
		ative parts	panel and glove	
			box included	
2	Auxiliary	Auxiliary		2000

Table 1 Car interior parts assembly under test and sampling requirement

	instrument	instrument		
	panel assembly	panel assembly		
3	Door systems	Door assembly	Front and back	2000
0		Door assembly	doors	2000
			(semi-side)	
4	Seat system	Front seat	with headrest	2000
-	Seat system	assembly	included; no	2000
		assembly	electric motor is	
			allowed; both	
			right and left	
			side are	
			acceptable;	
5	-	Back seat	Backrest,	2000
5			cushion and	2000
		assembly	headrest on	
			back seat	
6	-	Seat cover	The whole set	2000
7	-	Seat foam		2000
1		Seal Ioan	The whole set;	2000
			headrest can	
0	Deefevatere	Deefeeeembly	be excluded;	2000
8	Roof system	Roof assembly	With energy	2000
			absorbing block	
<u> </u>		Quality	included	0000
9	Flooring system	Central carpet	With energy	2000
			absorbing block	
			included, a	
40	-		whole set	0000
10		noise insulation		2000
	-	mat on the floor	The hole of	0000
11		Foot pad	The whole set	2000
12	Trunk	Side decorative		2000
	ornaments	panel		
13		Floor		2000
14	Rear window	Rear hatstand		2000
	system	assembly		
15	Column	Column	Column/seal	2000
	ornaments	ornaments	plate A, B, C	
			and D	
			(semi-side)	
16	Front wall	Front wall		2000
	sound proofing	sound proofing		
	mat	mat		
17	Steering wheel	Steering wheel	Electronic	2000
			components	

			can be excluded	
18	Seal strip	Seal strip for doors and windows	A whole set	2000
19	Air-conditioning module	Air-conditioner assembly	Plastic housing, vent, the cover for air filter net and heat insulation/buffer mat which is installed at air supply system and weighed more than 10g	2000
20		air ducting	A whole set	2000

6 Testing procedures

6.1 Rinse of blank sampling bag

6.1.1 Set the temperature of walk-in high-and-low temperature test box at 80° C, open the door and start air cycling for 30 minutes, and then close the door;

6.1.2 Cut the air sampling bag for rinse open and make the inner side facing the outside, put the bag into the box for heating for 10 to 16 hours. Then, reopen the door and replace the air for 30 minutes;

6.1.3 Turn the inner side of air sampling bag out and close the door, and then heat it up for another 1 hour;

6.1.4 Take the bag out and turn the inner side back, and seal the bag up with the sealer;

6.1.5 Pump the air out of the bag with the diaphragm vacuum pump to observe its gas-tightness (The air-tightness can also be determined by charging a certain amount of nitrogen into the bag and observing whether there is any obvious shrinkage after 2 hours);

6.1.6 To exchange the air in the bag with highly pure nitrogen by charging about 50% of high pure nitrogen as much as that of its volume and pump the air out, which will be repeated for 3 times;

6.1.7 To charge 50% highly pure nitrogen and have the bag heated up for $2h\pm10m$ in at 65°C, and then take out the benzene compounds with TENAX tube and aldehyde compounds with DNPH tube;

6.1.8 To validate the blankness with lab equipment and all substances should be lower than method detection limit.

6.1.9 6.1.1 to 6.1.8 will be repeated in case of failure to meet the requirement mentioned above.

6.2 Disposal of samples

6.2.1 Weigh the sample and take notes of its mass (W);

6.2.2 Open air sampling bag with qualified blankness test result and put the samples into it;

6.2.3 Seal the bag up with the sealer;

6.2.4 Pump the bag into a vacuum with the diaphragm vacuum pump to observe its gas-tightness (The air-tightness can also be determined by charging a certain amount of nitrogen into the bag and observing whether there is any clear shrinkage);

6.2.5 To exchange the air in the bag with highly pure nitrogen by charing about 50% of high pure nitrogen as much as that of its volume and pump the air out, which will be repeated for 3 times;

6.2.6 To charge 50% of highly pure nitrogen into the bag as much as that of its volume and take notes of the amount of charged gas;

6.2.7 Take another blank sampling bag and follow steps from 6.2.3 to 6.2.6 without putting samples into it;

6.2.8 Put two sampling bags into the sampling apparatus and heat them up for $2h\pm10$ min at the temperature of 65° C. Make sure that bags will be heated in a uniform manner;

6.2.9 To take the benzene compounds with TENAX tube and aldehyde compounds with DNPH tubes (two parallel tubes are needed).

6.3 Gas trap

6.3.1 Gas trap pipe

• Please use following two kinds of trap pipes when gas will be trapped.

1) TENAX-TA trap pipe: benzene, methylbenzene, dimethylbenzene, ethylbenzene, styrene, TVOC

2) DNPH trap pipe: formaldehyde, acetaldehyde, acrolein as well as acetone

• For installation of the trap pipe, auxiliary pipe and sample, please refer to picture 1





6.3.2 Trapping conditions

Shake the sampling bag to make the gas evenly distributed before gas trapping. Then trap gas with two TENAX tubes and one DNPH tube based on trapping conditions listed in table 2. The TENAX tube will be under quantitative analysis as soon as gas is trapped. Where the trapped gas cannot be analyzed at once, both ends of tubes shall be sealed with draw-in attachment first, and then the tube will be wrapped up with aluminum foil, marked and kept in the refrigerator at $4 \sim 10^{\circ}$ C for no more than one week. The DNPH tube with trapped gas must be eluted with acetonitrile at once. In case that the tube cannot be eluted instantly, the tube will be wrapped up with aluminum foil and kept at the refrigerator for no more than one week and at the temperature of no lower than at 4° C. The solution eluted with acetonitrile will be diluted into a 5mL bottle and be analyzed as soon as possible. The solution can be kept in a refrigerator for one week.

Trapping pipe	TVOC	aldehyde compounds	
	Tenax tube	DNPH tube	
Trapped flow rate ml/min	200	800	
Trapped time min	15	15	
Trapped volume L	3	12	

Table 2 Trapping conditions

7 Analysis methods and data processing

7.1.1 TD-GC-MS will be used for analysis of benzene, methylbenzene, dimethylbenzene, ethylbenzene, styrene and TVOC. The analysis method and data processing will meet GMW15654 standard and national HJ/T 400-2007 standard.

7.1.2 Formaldehyde, acetaldehyde, acrolein as well as acetone will be analyzed with HPLC. The analysis method and data processing will meet GMW 15600 standard and national HJ/T 400-2007 standard.

8 Result calculation and expression

8.1 Benzene, methylbenzene, dimethylbenzene, ethylbenzene, styrene

 $M~(\mu g)~=~(C~/~V_2 {-} C_0 {/}~V_{02})~\times V_1$

Among which,

M: VOC of the sample (µg)

C: Compound content that has been trapped by Tenax-TA tube for testing when the sample is heated (μ g)

C₀: Compound content to be tested in the blank bag (µg)

V₁: Volume of pure nitrogen sealed in the bag (L)

V₂: Volume of pure nitrogen trapped in the bag (L)

V₀₂: Volume of pure nitrogen trapped in the blank bag (L)

8.2 TVOC

Based on the area of methylbenzene with minimum area reject value as $0.01\mu g$, all compounds whose retention time is between normal hexane and hexadecane will be

analyzed and integrated to have the peak area, which will be calculated with TIC linear equation then.

8.3 Formaldehyde, acetaldehyde, acrolein as well as acetone

$$M (\mu g) = (C/V_2 - C_0/V_{02}) \times V \times V_1$$

Among which,

M: volatile content of aldehyde ketone of each sample (µg)

C: concentration of aldehyde ketone of samples trapped by pipe for testing (mg/L)

C0: concentration of aldehyde ketone trapped by the pipe in blank bags (mg/L)

V: Volume of acetonitrile used to elute the trapping pipe (mL)

V1: Volume of pure nitrogen sealed in the bag (L)

V2: Volume of pure nitrogen trapped in the sample bag (L)

V02: Volume of pure nitrogen trapped in the blank bag (L)

9. Standard No.

The standard is quoted as TS-BD-003-2012 in SOR or drawings.

10. Issue and update

The standard is drafted by PATAC VR center in May, 2012 and issued for the first time on May 30, 2012.