Research and Selection of Sorbents for Volatile Organic Compounds (VOC) Sampling Tubes

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Abstract—Volatile organic compounds (VOCs) are common emitting substances in vehicle interior parts and materials, with a wide range of boiling points (50-250℃) and a polarity range ranging from medium to non-polar, at least 200 species. Currently, VOC sampling mainly uses Tenax TA sorbent for collection, which has good stability and is suitable for weakly polar to non-polar substances between C6-C16. But there are many types of VOC substances, and each substance has its unique characteristics. Tenax TA is not suitable for all substances, and it is not the best sorbent choice for substances with medium polarity or low carbon number. In recent years, the vehicle and indoor environmental standards are open to the selection of sorbent, no longer limiting the type of sorbent sorbent, and more sampling tubes mixed with different sorbent types have emerged to achieve the best sampling results. After summarizing the common emitted substances in the car, this article selects 6 common types of sorbents or mixed sorbents for substance collection, and searches for the optimal sorbent type for each substance.

1. Introduction

The World Health Organization (WHO, 1989) defines Total Volatile organic compound (TVOC) [1][2] as a class of organic compounds whose boiling point ranges from 50℃ to 260℃, the saturated Vapor pressure at room temperature exceeds 133.32 Pa, and they exist in the air as vapor at room temperature. According to the chemical structure of volatile organic compounds, they can be further divided into 8 categories: alkanes, aromatics, olefins, halohydrocarbons, esters, aldehydes, ketones, and other compounds.

The main sampling method for volatile organic compounds (VOCs) in China is Tenax TA packing sorbent. However, the latest testing standard GB/T18883-2022 "Standards for indoor air quality" uses Tenax TA or equivalent packing sorbent tubes for VOC sampling. In the standard ISO16000-6:2021, VOC is sampled through active sorbent, and the sorbent packing is no longer specified. Different VOC substances can be sampled using more suitable sorbents, Refer to Table 1 and Table 2.

Table 1 Foreign VOC Main Standards and Scope of Application [3][4][5][6]

NO.	Standard number	Sampling method	Test method	substances
1	ISO16000-6-2021	Solid sorbent/unlimited sorbent	Gas chromatography-mass spectrometry	VOC
2	EN ISO 16017-1-2000	Tenax TA	Gas chromatography/gas chromatography-mass spectrometry	VOC
3	EPA TO-1	Tenax TA	Gas chromatography-mass spectrometry	Non polar organic compounds with boiling points between 80 and 200℃
4	EPA TO-17	Solid sorbent/unlimited sorbent	Gas chromatography-mass spectrometry	VOC

Table 2 Main Domestic VOC Standards and Scope of Application[7][8][9][10][11][12]

NO.	Standard number	Sampling method	Test method	substances
1	HJ583-2010	Tenax TA	Gas chromatography	Benzene derivatives
2	HJ584-2010	Activated carbon	Gas chromatography	Benzene

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				derivatives
3	HJ644-2013	Stainless steel and glass materials, with an inner diameter of 6mm, equipped with Carbopack B, Carbopack C, and Carboxen 1000, with lengths of 13, 25, and 13 mm respectively	Gas chromatography-mass spectrometry	VOC
4	HJ734-2014	Combination 1 sorbent tube, equipped with Tenax GR and Carbopack B, with lengths of 30 and 25 mm, respectively. Combination 2 sorbent tubes, containing Carbopack B and Carboxen 1000, with lengths of 30 and 10 mm, respectively. Combination 3 sorbent tubes, containing Carbopack C, Carbopack B, and Carboxen 1000, with lengths of 13, 25, and 13 mm, respectively.	Thermal desorption gas chromatography-mass spectrometry	VOC
5	GB/T18883-2022	Tenax GC or Tenax TA or other sorbents	Gas chromatography	TVOC
6	HJ/T400-2007	Tenax TA	Gas chromatography-mass spectrometry	VOC

The testing of volatile organic compounds (VOC) in the vehicle industry involves a wide range of fields, including vehicle VOC (HJ/T400, ISO12219-1), vehicle interior parts and materials VOC (ISO12219-2), materials (VDA278), and evaporative pollutant emissions (hydrocarbon emissions from non fuel systems in (CHINA VI)). There are many standards involved, and the testing methods and equipment are also different. However, the main sampling method is Tenax TA sorbent sorbent, without considering the different types of sorbents applicable to different substances. Currently, research and selection are conducted on the types of sorbents for VOC substances in vehicles.

From domestic and international VOC standards and sampling methods, In some standards, the selection of VOC sorbents has gradually shown an open state. The previously designated sorbents have gradually been transformed into different substances using different sorbents for sampling, and some sampling tubes have been filled with various combinations of sorbents, such as the appearance of combination tubes 1, 2, 3, etc. in standard HJ734-2014, It can select appropriate combination solid sorbents based on specific emitting substances. Specific types of sorbents can be refer to Table 3.

Table 3 Specific Application Scope of Different sorbents[3]

Type	Sorbent	Analyte rang	Conditioning at max.(°C)	Desorption max.(°C)	Hydro-phobic	Notes
Weakly polar graphitized carbon black	Carbopack C	C8-C20	400	360	Yes	Single VOC<1ng; 40/60 Surface area 10m^2/gmesh;
	Carbotrap C		400	360	Yes	
	CarboTenax Graph 2TD		400	360	Yes	
Weakly porous polymer	Tenax TA	C6-C22	320	280	Yes	Single VOC1-2ng;Good inertness and analytical efficiency (narrow peak shape);35-60 mesh;Surface area 30m^2/g
	Tenax GR		320	280	Yes	
Weak - medium type graphitized carbon black	Carbopack B	C5-C14	400	360	Yes	Surface area 100m^2/g;
	Carbotrap		400	360	Yes	
	CarboTenax Graph 1TD		400	360	Yes	
	CarboTenax Graph 4TD		400	360	Yes	
Medium strength porous polymeric sorbent						Single VOC<10ng;Background and batch differences make sorbents rarely used
Medium to strong graphitized carbon black	Carbopack X	1,4-Butadiene Benzene	400	360	Yes	Single VOC<1ng; 40/60 mesh;
	CarboTenax Graph 5TD		400	360	Yes	Surface area 240m^2/g;
	CarboTenax Graph 4TD		400	360	Yes	Poor desorption efficiency (wider spectral band)
Carbon molecular sieve	Carboxen 1003	C2/3-C5/6	350	330	No	Single VOC<1ng; 40/60 mesh;
	SulfiCarb		350	330	No	Surface area 400-1000m^2/g;
	Carbosieve SIII		350	330	No	Poor desorption efficiency (broadened spectral band);
	Carboxen 1000		350	330	No	When the humidity is greater than 80, it affects the sorbent effect
Other (non sampling end)	Glass wool	C30			Yes	
	Stone wool				Yes	
	Glass bead				Yes	

From the types of common sorbent sorbents that there are many types of sorbents, and different sorbents are suitable for different test ranges and use temperatures. Different substances can choose appropriate sorbent types

according to their characteristics. If the composition of VOC is complex and the range of substances (temperature or polarity) is large, each sampling tube can be filled with 2-4 different sorbent types to achieve the best sampling effect.

2. Experimental Part

2.1 Instruments and reagents

2.1.1 Instrument and equipment

Thermal desorption gas chromatography-mass spectrometry (TD-GCMS); Model: Marks TD100 Agilent GC7890B MS5977B.

2.1.2 Main reagent

Dichloromethane (HPLC/UPLC grade).

2.1.3 Standard selection

According to the WTO definition of VOC, the categories include alkanes, aromatics, olefins, halohydrocarbons, esters, and other compounds (acids, alcohols, amines, etc.) (aldehydes and ketones are collected using DNPH sampling tubes and analyzed by liquid chromatography, which is not within the scope of this study). VOC standards are all pure products, and specific substances are shown in Table 5-8.

2.2 Instrument operating conditions [9]

Gas phase conditions: chromatographic column Agilent DB-624 60m× 0.32mm×1.4μm; Column flow rate: 1mL/min Heating program: 40℃(2min) -3℃/min-92℃-5℃/min-160℃-10℃/min-260℃(10min)

Mass spectrometry conditions: voltage: 70ev; Scanning method: SCAN; Scanning range: 33-450amu; Scanning frequency: 3.5 times/second; The test results were matched based on the NIST2017 spectral library, and the qualitative and quantitative ions of each substance were confirmed.

2.3 Preparation of Standard solution

Accurately weigh 0.5000 g of each substance's single standard standard (accurate to 0.0001 g), dissolve it with dichloromethane and transfer it to a 100 mL volumetric flask for constant volume, prepare 5000 mg/L mixed standard stock solution, and store it in a refrigerator at 4℃. When using, dilute the standard stock solution with dichloromethane and prepare 1000 mg/L mixed standard solution for test and analysis.

2.4 Selection of sorbent

The selection of sorbent mainly considers medium polar and non polar substances with boiling points between (50-250)℃. The main characteristic of polar substances is hydrophilicity, and their test results are greatly affected by moisture content, making them unsuitable for sorbent sampling. Therefore, this sorbent selection mainly considers medium to non polar substances, and selects more common types of sorbents that can be purchased on the market.

Select 6 different types of monomers or combinations of sorbents based on the current standard's sorbent types and applicability. Please refer to Table 4 for specific types.

Table 4 Different types of monomers or combinations of sorbents[3][13][14][15] [16]

No.	sorbent	Analyte rang	Manufacturer	characteristics
1	Tenax TA	C5-C22	Marks Mix	Good hydrophobicity, suitable for most non-polar substances
2	Tenax GR	C5-C22	Marks Mix	
3	Combination 1 sorbent tube, equipped with Tenax GR and Carbopack B, with lengths of 30 and 25 mm, respectively	C6-C20	CNW	Good hydrophobicity, suitable for most non-polar substances
4	Combination 3 sorbent tubes, containing Carbopack C, Carbopack B, and Carboxen 1000, with lengths of 13, 25, and 13 mm, respectively	C3-C16	CNW/Camsco	For some polar and medium type substances, the sorbent is good, but the hydrophobicity is poor
5	Graphitized carbon	C5-C20	Marks Mix	Good hydrophobicity, suitable for most non-polar substances
6	Graphitized carbon mixing tube	C3-C20	Marks Mix	Combination type, wide application range

3. Results and discussion

The test material selection includes ester, alcohol ether, amine, alkane, olefin, Aromatic hydrocarbon, Cycloalkane and other material categories. Each type of substance shall be tested at least 6 times, and the test results shall be statistically significant. The test results are based on the maximum chromatographic peak area as the reference value, with the deviation being the difference between the peak area of other sorbent sorbents and the maximum value. The statistical result is (100 deviation value), and the closer the value is to 100, the smaller the deviation.

3.1 Acid ester substances

3.1.1 The chromatogram and substance list of ester substances are shown in Figure 1 and Table 5.

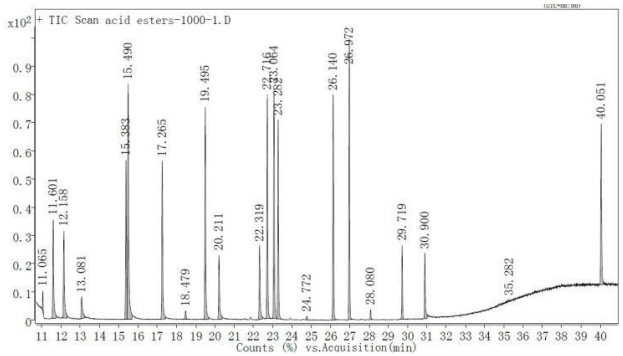

Figure 1 Chromatogram of Acid Esters

Table 5 List of 18 Common Acids Esters in Vehicles[17]

NO.	Substance	CAS NO.	RT	Quantitativeion	Qualitativeion
1	Ethyl acetate	141-78-6	11.601	43	61,70
2	tetrahydrofuran	109-99-9	12.158	71	42,72
3	Acetic acid	64-19-7	13.086	45	60
4	Methyl methacrylate	80-62-6	15.384	69	41,100
5	N-propyl acetate	109-60-4	15.49	61	88,73
6	1, 4-dioxane	123-91-1	15.5	88	43,61
7	Sec-butyl acetate	105-46-4	17.265	87	56,43
8	Butyl acetate	123-86-4	19.495	56.1	43,73
9	Isovalerate butyrate	107-92-6	20.215	60	73
10	Butyl acrylate	503-74-2	22.314	60	87
11	Butyl propionate	141-32-2	22.716	55	73
12	Ethylene glycol ethyl ether acetate	590-01-2	23.064	75	57,87
13	Ethyl 3-ethoxy propionate	111-15-9	23.277	59.1	72,87
14	Ethylene glycol diacetate	763-69-9	26.135	117	71,101
15	Carbonate allyl ester	111-55-7	26.972	86	73,116
16	2-ethylhexanoic acid	108-32-7	29.715	57	87,102
17	2,2, 4-trimethyl-1, 3-pentanediol diacetate	149-57-5	30.9	88	73
18	Ethyl acetate	6846-50-0	40.046	71	43

3.1.2 The test results of ester substances are as follows, and the source of the results is shown in Figure 2.

a) For ester like substances, the best sorbent tube is the Graphitized carbon mixing tube with deviations within 11%, which shows the most stable performance;

b) Tenax GR sorbent has a significant deviation in high boiling point substances, with other deviations within 10%, indicating good performance;

c) The performance of graphitized carbon adsorption tube is relatively extreme, and the adsorption effect of tetrahydrofuran, acetic acid, butyric acid, isovaleric acid and 2,2, 4-trimethyl-1, 3-pentanediol diacetate is poor, but it is the adsorbent with the highest peak area and can be used in a certain range;

d) Tenax TA has poor sorbent performance for low-carbon and highly polar substances, with deviations within 15% for other substances;

e) Combination tubes 1 and 3 have poor sorbent performance for esters, but some substances with deviations within 20% can be selected for use.

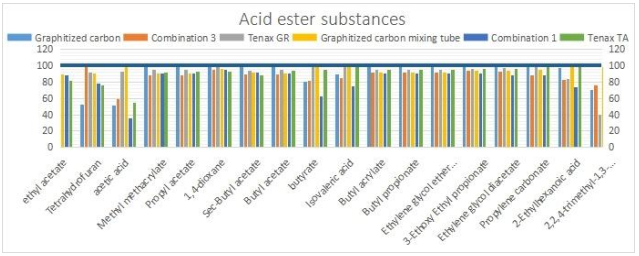

Figure 2 Sampling 100-deviation value of Acid Ester sorbents

3.2 Alcoholic Ethers and Partial Ketones

3.2.1 Chromatograms and substance lists of alcohol ethers and some ketones are shown in Figure 3 and Table 6.

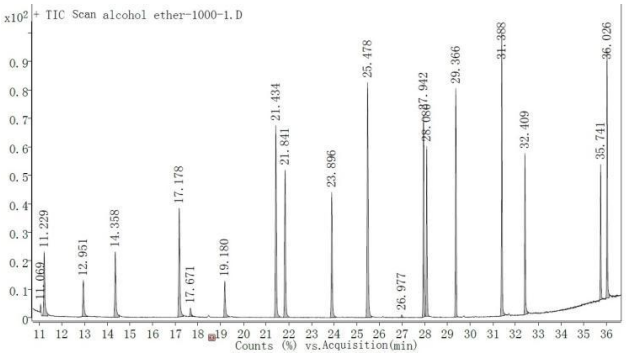

Figure 3 Chromatogram of Alcohol Ethers and Ketones

Table 6 List of 18 Common Alcohol Ether and Ketone Substances in Vehicles[17][18]

NO.	Substance	CAS NO.	RT	Quantitativeion	Qualitativeion
1	2, 3-butanedione	431-03-8	11.234	86	43
2	2-methyl-1-propanol	78-83-1	12.955	43.1	74,33
3	1-butanol	71-36-3	14.358	41.1	56
4	4-methyl-2-penta	108-10-1	17.178	58	43,85

	none				
5	Ethylene glycol	107-21-1	17.676	62	43
6	Propanediol	57-55-6	19.18	45.1	61
7	Dibutyl ether	142-96-1	21.434	57.1	87,41
8	Propylene glycol methyl ether acetate	108-65-6	21.836	72.1	58,87
9	Ethylene glycol butyl ether	111-76-2	23.896	57.1	87,45
10	1, 3-dichloro-2-propanol	96-23-1	25.473	79	45,59
11	Diethylene glycol monomethyl ether	111-77-3	25.482	45.1	79,59
12	1, 2-propylene glycol diacetate	623-84-7	27.939	43	87
13	2-ethyl-1-hexanol	104-76-7	28.08	57.1	70,83
14	Ethylene glycol butyl ether acetate	112-07-2	29.361	87	57,100
15	2-phenylethanol	60-12-8	31.388	91	122
16	Diethylene glycol monobutyl ether	112-34-5	32.408	45	57,75
17	Hexadiol diethyl ester	141-28-6	35.741	157.1	111,128
18	2-butyl-1-octanol	3913-02-8	36.021	57.1	71,85

3.2.2 The test results of Alcohol Ethers and Ketones are as follows, and the source of the results is shown in Figure 4.

There are highly polar substances in alcohol ether substances, and different types of sorbents have different substances used. Overall, it can be seen that:

a) For alcohols and ethers, the best comprehensive sorbents are graphitized carbon and Tenax TA, with deviations within 20% except for a few substances from 1-2;

b) Graphitized carbon mixing tube is the sorbent with the highest maximum value, but its deviation exceeds 30% in high carbon substances;

c) The other four sorbent materials have corresponding usage conditions within different substance ranges.

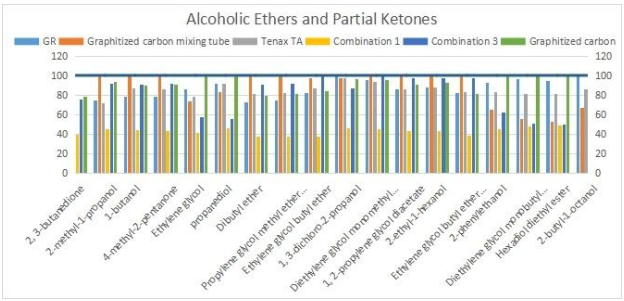

Figure 4 Sampling 100-deviation value of Alcohol Ethers and Ketones sorbents

3.3 Amine substances

3.3.1 Chromatograms and substance lists of Amine are shown in Figure 5 and Table 7.

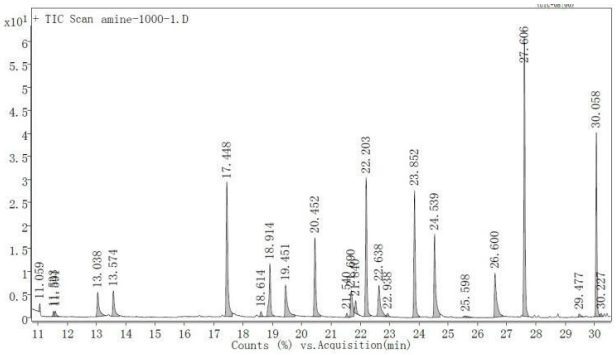

Figure 5 Chromatogram of Amine Substances

Table 7 List of 16 Common Amine Substances in Vehicles[17][18]

NO.	Substance	CAS NO.	RT	Quantitative ion	Qualitative ion
1	Triethylamine	121-44-8	13.56	86.1	101,28
2	Pyridine	110-86-1	17.444	79	52
3	2-butanone oxime	96-29-7	18.914	87.1	42,58
4	Morpholine	110-91-8	19.441	57.1	87
5	N,N-dimethylformamide	68-12-2	20.452	73	44
6	Cyclohexylamine	108-91-8	21.686	56.1	99,43
7	N-ethylmorpholine	100-74-3	22.198	100.1	115
8	Dimethylacetamide	127-19-5	23.852	87.1	44,72
9	N-butylamine	109-73-9	24.534	86.1	44,129
10	1-methylimidazole	616-47-7	26.6	82	54
11	Aniline	62-53-3	27.606	93	66
12	Bis (dimethylaminoethyl) ether	3033-62-3	29.473	86	56
13	Methylpyrrolidone	872-50-4	30.058	99	44
14	Ethyl pyrrolidone	2687-91-4	31.601	98	113,70
15	Benzothiazole	95-16-9	33.855	135	108
16	Caprolactam	105-60-2	35.301	113.1	55,85

3.3.2 The test results of Amine Substances are as follows, and the source of the results is shown in Figure 6.

Amine substances contain highly polar substances, and there is a significant deviation in the sorbent effect between different sorbents. For amine substances:

a) The best comprehensive sorbents are graphitized carbon and Tenax TA, with deviations within 20% except for 1-2 individual substances;

b) The deviation of the Graphitized carbon mixing tube is within 20%, and this sorbent can be used according to different substances;

c) The other three sorbent materials have relatively poor performance, and it is recommended to use them as appropriate.

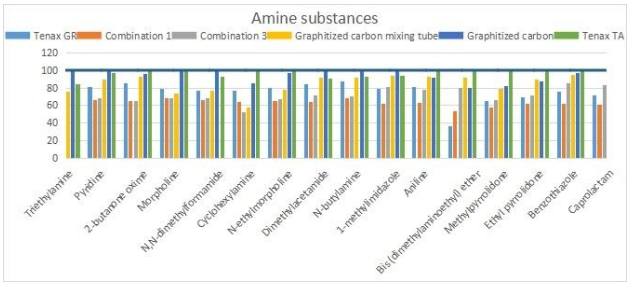

Figure 6 Sampling test results of amine sorbent

3.4 Alkanes, Cycloalkane, olefins, Aromatic hydrocarbon and other substances

3.4.1 Chromatogram and list of alkanes, Cycloalkane, olefins, Aromatic hydrocarbon and other substances are shown in Figure 7 and Table 8

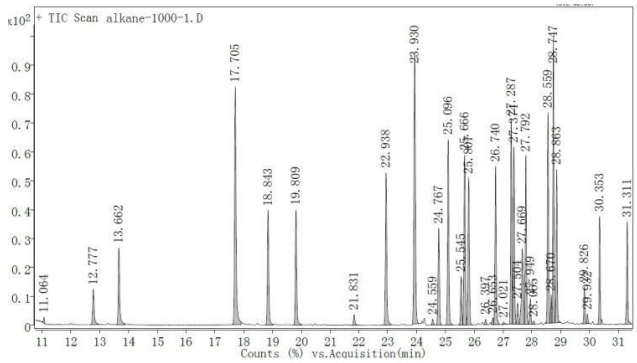

Figure 7 Chromatogram of Alkanes, Cycloalkane, Olefins, Aromatic hydrocarbon and Other Substance

Table 8 List of Common Alkanes, Cycloalkane, Olefins, Aromatic hydrocarbon and Other Substances in Vehicles[16][17][18][19]

NO.	Substance	CAS NO.	RT	Quantitativeion	Qualitativeion
1	Cyclohexane	110-82-7	12.777	84.1	56,69
2	1-heptene	592-76-7	13.667	56.1	56,71
3	1-octene	111-66-0	17.695	55.1	70
4	Toluene	108-88-3	17.71	91	65
5	2,4-Dimethylheptane	2213-23-2	18.847	43.1	85,57
6	2,4-Dimethyl-1-heptene	19549-87-2	19.809	70.1	43,55
7	O-xylene	95-47-6	22.938	91.1	106
8	Alpha pinene	80-56-8	23.92	93.1	105
9	Isopropyl benzene	98-82-8	23.94	105	120
10	Octamethylcyclotetrasiloxane	556-67-2	24.771	281	73,155
11	Propyl benzene	103-65-1	25.096	91	120
12	2,2,4,6,6-pentamethylheptane	13475-82-6	25.666	57.1	41,71
13	Beta-pinene	127-91-3	25.807	93.1	69
14	Carene	13466-78-9	26.74	93.1	77
15	Butylcyclohexane	1678-93-9	27.287	83.1	55,170
16	Limonene	138-86-3	27.378	68.1	93,79
17	1,2,3-trimethylbenzene	526-73-8	27.79	105.1	105,120
18	Naphthane	91-17-8	28.559	138.1	67,82
19	Phenol	108-95-2	28.67	94	66
20	Undecane	1120-21-4	28.747	57.1	43,71
21	Indene	95-13-6	28.863	115	89
22	Azodiisobutyronitrile	78-67-1	29.826	69.1	41,54
23	1,2,4,5-tetramethylbenzene	95-93-2	30.353	119.1	134
24	Triethyl phosphate	78-40-0	31.286	155	99
25	2,6-dimethylphenol	576-26-1	31.311	107	122
26	2-methylnaphthalene	91-57-6	34.977	141.9	
27	Dimethyl phthalate	131-11-3	38.038	163	77,194
28	2,6-di-tert-butyl-4-methylphenol	128-27-0	38.14	205.1	220
29	2,4-di-tert-butylphenol	96-76-4	38.638	191.1	206,57

3.4.2 The test results of alkanes, Cycloalkane, olefins, Aromatic hydrocarbon and other substances are as follows, and the source of the results is shown in Figure 8, 9.

Alkanes, alkenes and Aromatic hydrocarbon are mainly non-polar substances with boiling points between 50-250℃, and there are many types of sorbents that can be used:

a) For alkanes, olefins and Aromatic hydrocarbon, the best sorbent in comprehensive effect is graphitized carbon, Tenax TA, and mixed sampling tube 1. Except for 1-2 individual substances, other deviations are within 20%;

b) Most of the other three sampling tubes (boiling points between 100 and 200℃) have good sorbent effects, while only some substances have poor sorbent effects. They can be used according to specific substances for reference.

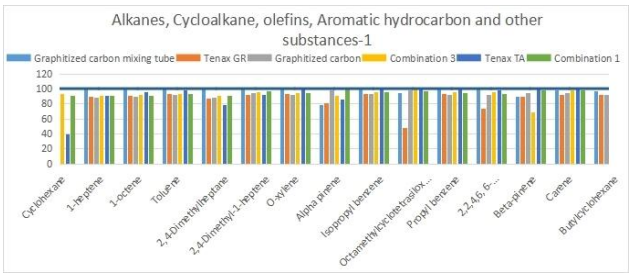

Fig.8 Sampling Test Results of sorbents for Alkanes, Cycloalkane, Olefins, Aromatic hydrocarbon and Other Substances-1

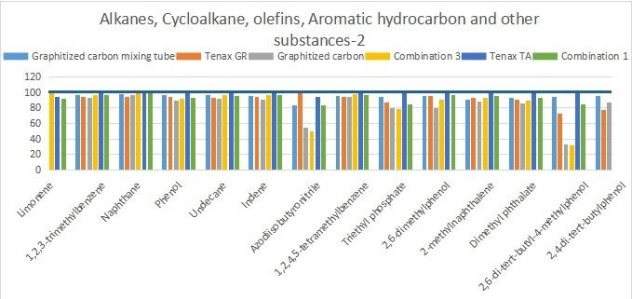

Fig.9 Sampling Test Results of sorbents for Alkanes, Cycloalkane, Olefins, Aromatic hydrocarbon and Other Substances-2

4. Conclusion

From the Conclusion, it can be seen that for substances with strong or medium polarity between boiling points (50-200)℃, Graphitized carbon mixing tube is the most effective sorbent, and its sorbent effect is the best; For substances with boiling points between 100 and 250℃, graphitized carbon and Tenax TA with weaker polarity have better effects; During the specific use process, appropriate sorbent types can be selected based on relevant substances. Achieving optimal testing results.

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