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Original article

Chemical components of *Choerospondias axillaris* wood by TD-GC/MS, Py-GC/MS, and TGXiaochen Yue^a, Juntao Chen^b, Yafeng Yang^a, Zhenling Liu^c, Ting Wang^{a,*}, Wanxi Peng^{a,b}^a College of Forestry, Henan Agricultural University, Zhengzhou 450002, Henan, China^b College of Materials Science and Engineering, Central South University of Forestry and Technology, Changsha 410004, China^c School of Management, Henan University of Technology, Zhengzhou 450001, China

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ABSTRACT

Choerospondias axillaris is a better fast-growing species. Its bark and fruit have the functions of anti-inflammation, detoxification, hemostasis and treatment of external burns. However, due to the lack of systematic and in-depth analysis of the chemical composition of *Choerospondias axillaris*, it is difficult to develop high value-added products, resulting in low processing efficiency and even direct abandonment. In order to improve the application value of *Choerospondias axillaris* and excavate its application in many fields, this paper takes *Choerospondias axillaris* as the research object to reveal the characteristics of volatile organic compounds and the variation rule of molecular components of jujube before and after extraction of *Choerospondias axillaris*. The extracts of *Choerospondias axillaris* were detected and identified by GC-MS and FTIR. The pyrolysis process of *Choerospondias axillaris* was characterized by TGA-DTG and Py-GC-MS. The volatile organic compounds in the ethanol extracts of *Choerospondias axillaris* were mainly alcohols, petroleum ether extracts were mainly alkanes and organic acids, and phenylethanol extracts were mainly esters. Among the three kinds of jujube extracts, nonanal, beta-caryophyllene, humus and caryophyllene oxides and other bioactive VOCs were observed. The total content of VOCs from high to low was petroleum ether extract, ethanol extract and benzene/ethanol extract. *Choerospondias axillaris* has three distinct stages of heat loss treatment: the first stage is 30–50°C, the second stage is 50–200°C, and the third stage is 200–247°C. During the heat loss treatment, three critical temperature turning points (50, 200 and 237) were observed, accompanied by significant chemical changes such as pyrolysis of macromolecules into small volatile molecules. By pyrolysis of the extracts and residues of *Choerospondias axillaris*, a large number of new components have been produced, which can be used in other industries and provide a new way for sour jujube to become a high-grade application resource.

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1. Introduction

The core of *Choerospondias axillaris* is large and very hard. Since ancient times, it has been a symbol of “wufu rimmon” (Hua et al., 2008). *Choerospondias axillaris* is one of the growth in several provinces in south China edible wild fruits, and it appropriate chooses an elevation of 300–800 m (He et al., 2003). The fruit is sweet, sour and delicious, and can be eaten directly. It can also be used to pro-

cess wine and sour jujube cake; the nucleus can be activated carbon raw material; leaves can do green manure; Bark can also be used as a raw material for tanning and tannin extracts (Doanh et al., 1996; Joshi et al., 2015). But *Choerospondias axillaris* flavour, acidity, and after harvest perishable fermentation, lose a lot of nutrients, is not conducive to fresh jujube fresh-keeping and circulation, thus has often been processing for south SuanZaoGao, wild jujube juice, zizyphus jujube wine and other products, especially south SuanZaoGao has always been the most popular food in the market (Mi et al., 2019).

Choerospondias axillaris products unique flavor, favored deeply by consumers at home and abroad, affected by food safety concerns more green food becomes the inevitable pursuit of people (Qing et al., 2005; Wang et al., 2014). However, the development of jujube is not very good due to the lack of leading processing enterprises and the lack of breeding and efficient supporting

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technologies (Li et al., 2016; Mi et al., 2015). Therefore, we should give full play to all aspects of the *Choerospondias axillaris* resource value, efforts to achieve the comprehensive utilization of resources sustainability, at the same time, the resource advantage into economic advantage, effective way of innovation, and actively develop the green industry, through thinking to guide the industrialization of ecological construction, conversion of forest products processing, extend the industrial chain. In this experiment, the analysis of TG, Py-GC/MS, TD-GC/MS and FTIR are used to analyze the molecular active components, cracking rules and cleavage products of *Choerospondias axillaris*, and to help maximize the utilization of *Choerospondias axillaris*.

2. Materials and methods

2.1. Materials and reagents

Choerospondias axillaris was provided from the woods in front of YiFu building of Central South University of Forestry and Technology. After working under natural conditions (40 °C), the camellia seed cake identification smashed into powder by FZ102 crusher is suitable for the plant (cape Taisite Ins).

2.2. Methods

2.2.1. *Choerospondias axillaris* extraction by solvent

Choerospondias axillaris samples were mixed with benzene/ethanol (2:1) with the solid-liquid ratio of 1:20. After immersing at room temperature for 12 h, the mixed solution were fully extracted by automatic FOSS Soxhlet Extracted apparatus (Agilent, USA) at 70 °C for 5 h.

2.2.2. TD-GC/MS analysis for *Choerospondias axillaris*

TD conditions: the initial temperature was 30 °C, retained for 1 min, raised to 100 °C at a rate of 10 °C/min, retained for 5 min, and then increased to 200 °C at a rate of 10 °C/min, not retained. The transmission line temperature is 230 °C.

GC conditions: capillary column (30 m × 250 μm × 0.25 μm). The initial temperature was 50 °C, kept 1 min, in succession, increased to 150 °C at 5 °C/min, kept 10 min, then to 250 °C at 8 °C/min, stayed 2 min; injection volume of 1 μL; no a split mode; vaporization chamber temperature was 280 °C. MS detection conditions: the program of MS was scanned over the 35–600 amu (*m/z*).

2.2.3. *Choerospondias axillaris* and its extracted residue analysis by FTIR

The FT-IR spectra of the extracted samples were obtained with a FT-IR spectrophotometer (Thermo Fisher Scientific iS10) using KBr discs containing a 1.00% finely ground sample.

2.2.4. *Choerospondias axillaris* Thermostability by TG

Thermogravimetric analysis (TGA) was conducted using a thermogravimetric analyzer (TGA Q50 V20.8 Build 34, USA), and the temperature program of TA started at 30 °C, and increased 600 °C at 10 °C/min; carrier gas is high purity nitrogen, with a flow rate of 40 mL/min.

2.2.5. Extractives and residues analysis by Py-GC/MS

PY-2020iS (Frontier Co) was used to pyrolyze 0.1 mg of residue in helium atmospheres at 50, 100, 200, and 250°Celsius (residual at 50 and 250°Celsius), respectively. Pyrolysis products (pyrolyzate) were analyzed by on-line connected GC/MS. GC/MS analysis was carried out on Agilent 5975C/6890 N (Agilent Corp., USA) using an elastic quartz capillary column DB-5MS coated with neutral phase (30 m × 0.25 mm × 0.25 μm). The temperature of the inlet is

250 °C. The temperature program of GC starts at 50 °C. The temperature rises to 300 °C at the rate of 10 °C/min, and then sprays at a ratio of 30:1. MS program scans on 35–550 amu (*m/z*) with an ionization voltage of 70 eV and an ionization current of 150 μA (EI).

3. Results and analysis

3.1. VOC components analysis of extractive from *Choerospondias axillaris*

The TD-GC/MS chromatograms of *Choerospondias axillaris* in the range of 50 °C to 250 °C were shown in Fig. 1. It can be seen that the extractives had three obvious peaks at 12.237 min, 19.610 min and 25.231 min. From Table 1, it can be found that this material is a kind of Ethanol, 2-butoxyethoxy at 12.23 min. The molecular weight of C₁₈H₃₂O₄ and the molecular weight is 312.44, and the molecular structure. The study of the properties of this property is still unknown. Also, from Table 1, it can be know that the substance present at 19.61 min was 1,2-Bezenedicarboxylic acid, bis(2-methylpropyl) ester, the chemical formula was C₁₆H₂₂O₄, and molecular structure. It's colorless transparent oily liquid, not volatile, slightly aromatic smell, flammable and toxic. Mainly as plasticizer, it can make the products have good softness, but the volatile and water extraction is bigger, so the durability is poor. At 25.21 min, through Table 1 we can know that the substance is 7, 9-di-*tert*-butyl-1-oxaspiro(4,5)deca-6,9-diene-2, 8-dione, and molecular structure. The data from TD-GC/MC can be inferred that these three unsaturated compounds should be the main components of the extracts from *Choerospondias axillaris*. However, the study on the physiological activity and pharmacological toxicity of these three compounds is not clear.

From the results of Table 1, 16 kinds of organic compounds were detected from the extract of the experiment. Most volatile organic compounds are aldehydes and esters. For example, the substance detected at 12.42 min is 2-methylpropionate-3-heptylenyl ester. Its chemical formula is C₁₁H₂₀O₂, a colorless liquid, soluble in ethanol and oil, and generally used for food and spices. The relative content of the substance in the extractant is 3.5%. The substance detected at 15.85 min was 2,2,4-trimethyl-1,3-pentanediol. Its chemical formula is C₈H₁₈O₂, which is a white crystal, soluble in alcohols, ketones and aromatic solvents, and is difficult to dissolve in water and fatty hydrocarbon. It can produce monoester or diester with a variety of acids. Is a kind of irritant substance, mainly used in polyester manufacturing. The relative content of the substance in the extractant is 2.99%. The substance was detected in 19.6 min, 1,2-benzenedicarboxylic acid. Its chemical formula is C₁₂H₁₄O₄, it is colorless transparent oily liquid. It is mixed with ethyl alcohol ether, soluble in acetone, benzene and other organic solvents, insoluble in water. Used for cellulose acetate, it is used to improve the water resistance and elasticity of the products and give appropriate strength. Used for nitrocellulose, products can obtain excellent light resistance and toughness, and no stink. The adhesion of polyvinyl acetate can be improved (Jiang et al., 2017). Since this product is non-toxic, it can be used as the plasticizer of non-toxic adhesive for food packaging. It can also be used as the plasticizer of alkyd resin, butyl rubber and chloroprene rubber, as well as the solvent used as perfume. The relative content of the substance in the extractant is 100%. At 20.2 min, 7, 9-di-*tert*-butyl-1 was detected, with a relative amount of 20.88%, and its use is unclear.

3.2. Chemical group change characteristics of *choerospondias axillaris* and residues

FTIR is a rapid detection technology, with a high sensitivity, and it is commonly used in the identification of chemical bonds and

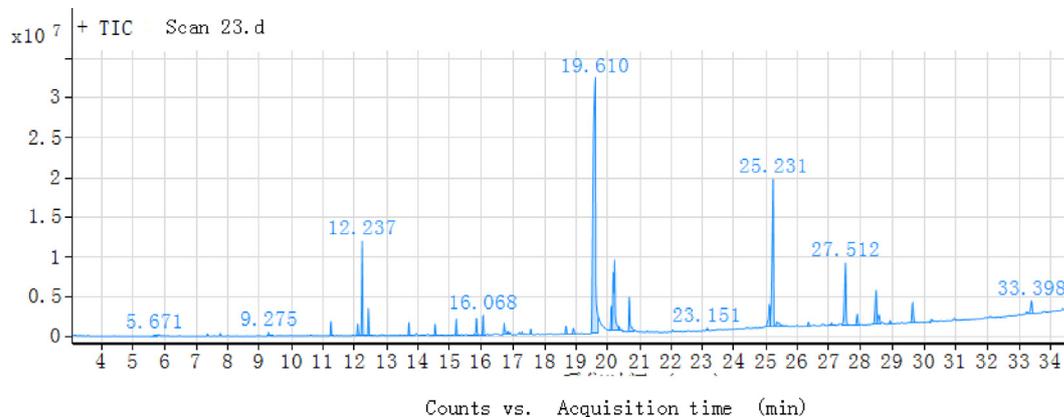


Fig. 1. The ion chromatograms of VOC from *Choerospondias axillaris* by TD-GC/MS.

Table 1

The results of TD-GC/MS of *Choerospondias axillaris*.

No.	Retention time (min)	Relative content (%)	Compound Name
1	9.275	0.64	1-(2-Butoxyethoxy)ethanol
2	11.254	1.9	Formamide, N,N-dibutyl-
3	12.086	2	2,2,4-Trimethyl-1,3-pentanediol diisobutyrate
4	12.237	12.2	Ethanol, 2-(2-butoxyethoxy)-,
5	12.426	3.5	Propanoic acid, 2-methyl-, 3-
6	13.712	1.66	Dimethyl phthalate
7	14.544	1.35	2,4-Di- <i>tert</i> -butylphenol
8	15.211	2.08	Benzene, 1,2,3-trimethoxy-5-(2-
9	15.854	2.99	2,2,4-Trimethyl-1,3-pentanediol
10	16.068	2.79	Cedrol
11	17.556	0.76	Methyl tetradecanoate
12	19.61	100	1,2-Benzenedicarboxylic acid,
13	20.114	3.74	Phthalic acid, butyl hex-3-yl ester
14	20.215	20.88	7,9-Di- <i>tert</i> -butyl-1-
15	20.681	6.4	Dibutyl phthalate
16	28.483	7.5	10,11-Dihydro-10-hydroxy-2,3,6-

Note: “-” this indicates that nothing is detected.

functional groups in compounds (Suksuwan et al., 2015; Jović et al., 2014; Yi et al., 2018). In this experiment, the changes of the *Choerospondias axillaris* and extracted residues' chemical groups were shown in Fig. 2, in the range from 4000 cm^{-1} to 400 cm^{-1} .

As shown in infrared spectroscopy of Fig. 2, it showed a total of 22 absorption peaks examined in *Choerospondias axillaris* sample. First, there was absorption peak at 3438 cm^{-1} , indicating that there was the O-H stretching vibration present, which having alcohols compound: 2-ethyl-1-Butanol. In 1741 cm^{-1} , there was a strong absorption peak, 2913 cm^{-1} and 1046 cm^{-1} also has two absorption peaks. The first strong absorption peaks are caused by C-O stretching, the last two are aldehyde group C-H stretching vibration, prove the existence of aldehydes compounds. In 1300 cm^{-1} -1000 cm^{-1} , there are two peaks, respectively, C-O-C symmetric stretching vibration (weak peak near 1140 cm^{-1} -1030 cm^{-1}) and asymmetric stretching vibration (strong peak near 1300 cm^{-1} -1150 cm^{-1}) (Adams et al., 2007; Roscini et al., 2010). In the vicinity of 3440 cm^{-1} also can often observe the weaker C=O stretching vibration frequency doubling of absorption. It can be determined that there are esters. And at 1118 cm^{-1} , it is probably ethers. There are several absorption peaks of 900 cm^{-1} to 750 cm^{-1} , which can determine the cis trans configuration of the compound. 1732 cm^{-1} -1729 cm^{-1} and 732 cm^{-1} -1503 cm^{-1} carbonyl C=O double bond stretching vibration and C-H in-plane bending vibration; 1370 cm^{-1} C-O and C-C frame vibration, 674 cm^{-1} -664 cm^{-1} C-C scale, 1510 cm^{-1} -1510 cm^{-1} near the

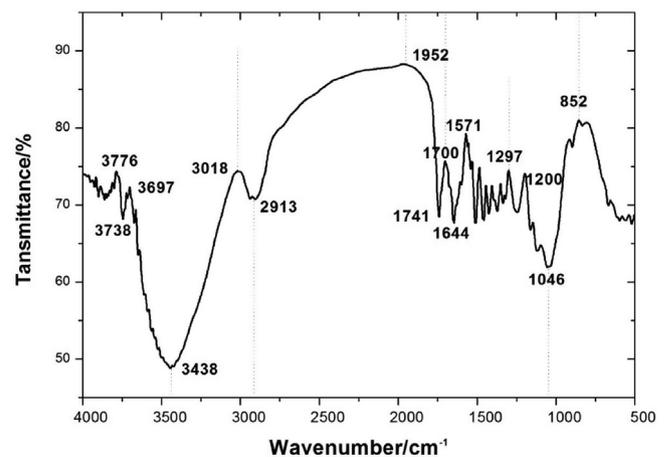


Fig. 2. Infrared spectra of VOC of benzene/ethanol extractives from *Choerospondias axillaris*.

benzene ring of vibration, etc., corresponding to a variety of oxygen-containing organic compounds, such as phenols, aldehydes, acids, ketones and other substances. In summary, *Choerospondias axillaris* mainly contains fat hydrocarbon and aromatic structure and various kinds of oxygen-containing functional groups. In addition, it can be concluded that organic solvent extraction does not made compound groups of *Choerospondias axillaris* changed.

3.3. Volatility characteristic of *Choerospondias axillaris* by thermostability analysis

Changes in *Choerospondias axillaris* from 30 °C to 250 °C are shown in Fig. 3. There are three distinct phases of heat loss willow cheilophila: the first phase of the initial temperature is 30 to 50 °C, the change of weight from 3.455906 mg to 3.455906 mg, this part of the quality loss is one of the most rapidly. At this point, the DTG curve also declines from the maximum value, indicating that the TG content decreases at a slower rate (Elbaz et al., 2015). In the second phase, from 50 °C to 200 °C, this one phase curve flattens the quality, there are fewer. This part of mass loss should be caused by evaporation or volatilization of small amount of water. At this point DTG is also leveling off, basically staying the same. The third part is from 200 °C to the end of 247 °C, quality change from 3.178024 mg to 3.109858 mg. The DTG curves of this part of the rise, this is because the decreased from 200 °C to TG quality rate begin to ascend (Cuetos et al., 2009; Jia et al., 2012; Paama et al.,

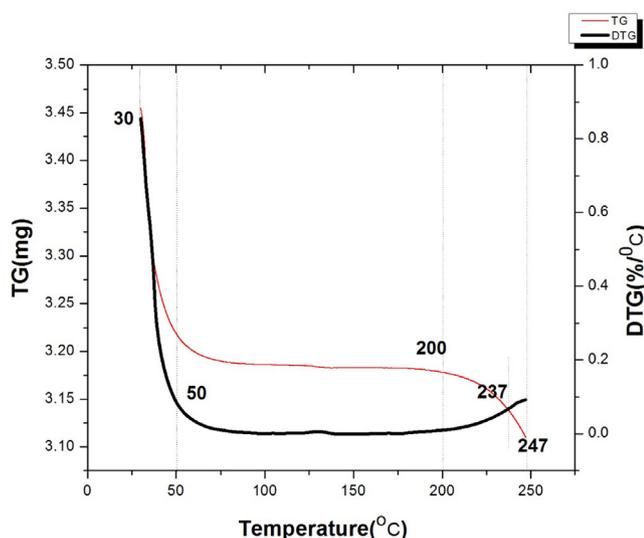


Fig. 3. Thermogravimetric curve of *Choerospondias axillaris*.

2000). According to the DTG curve, the order of mass loss rate was observed as follows: the first, the third and the second stages. There are two critical temperature turning points in the whole heating process. At these temperatures, the mass of *Choerospondias axillaris* changes dramatically, which may be caused by chemical changes, such as the breakdown of macromolecules into smaller molecules that are more volatile (Sasca et al., 2010).

3.4. Component characteristics of pyrolyzates from extractive

The total ion chromatograms of the samples studied by Py-GC-MS are shown in Fig. 4. And the relative content of each component has been counted by area normalization. The MS data is analyzed by using the NIST standard MS map and publicly published books and papers, and then identify each component (Ribechini et al., 2009; Bo et al., 2015; Khabbaz et al., 2015). And the analytical results of the six samples were listed in Tables 2.

According to the results of Py-GC-MS analysis, 50 peaks were detected in Table 2. The results show that the content of more substances are as follows: hydroxy-Acetic acid (7.46%), Carbamic acid-monoammonium salt (5.65%), Ammonium acetate (4.02%), 2 (5H)-Furanone (6.26%), 2-Methoxy-4-vinylphenol (5.14%), *trans*-Isoeugenol (3.52%), Ammonium acetate (4.02%), Propanoic acid, 2-oxo- methyl ester (3.14%), 1-hydroxy-2-Propanone (2.94%),

N-Butyl-*tert*-butylamine (2.53%), *p*-Cresol (3.08%), 1,2,4-Trimethoxybenzene (2.57%). The main components of these detected compounds are esters, acids, phenols, anthraquinones and ketones. By analyzing the main functions and functions of different compounds, *Choerospondias axillaris* can be more effectively and fully utilized and exerted.

4. Conclusion and discussion

Many extracts were extracted in benzene/ethanol extract, mainly used as plasticizer, but from the extraction of saliva extract-butanol, carbamic acid phenol and sheen – three oxygen radicals, its function is not clear. In addition, a variety of useful substances, such as benzene, methyl ketone, 2 (5 h) – Furanone, hexanal, ammonium acetate, 2-Propanone, propionic acid, their function is different, in the actual processing and utilization, can according to different functions, the production of various kinds of useful products and making fully effective use of resources. FTIR results further confirmed the organization structure of *Choerospondias axillaris* and extracting of residue is consistent. It suggests that the sample of *Choerospondias axillaris* contains esters, aldehydes, alcohols, ether, etc., and display, organic solvent extraction no organized with *Choerospondias axillaris* change significantly.

There are three obvious stages of thermal loss. During the whole process of thermal gravity, the DTG curve is in the first decline, then the latter tends to be gentle, and finally the trend is improved. There are two key temperature tipping points of 50 °Celsius and 200 °Celsius. At these temperatures, the quality of the salivary bacteria changes significantly, such as the decomposition of large molecules into small molecules that are more volatile. The main pyrolysis products include ester, acid, alcohol, aldehyde, ether, enyl acid, ammonium, anhydride, phenol, ketone, furan and heterocyclic compounds. There is a big difference between the properties and functions of matter. Therefore, the application prospect is also very different.

Through the pyrolysis law and pyrolysis products of *Choerospondias axillaris*, the new pathway of *Choerospondias axillaris* resource utilization and the application of *Choerospondias axillaris* in other fields are revealed, which provides a scientific basis for the maximum resource utilization of *Choerospondias axillaris*.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

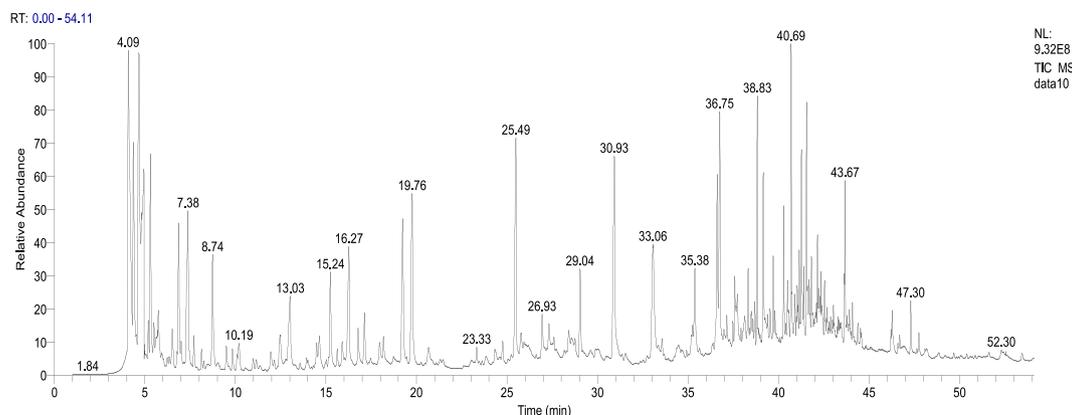


Fig. 4. The total ion flow diagram of *Choerospondias axillaris* by Py-GC/MS.

Table 2
The components of *Choerospondias axillaris* pyrolyzates by Py-GC/MS.

NO.	Retention time (min)	Relative content (%)	Compound Name
1	4.09	5.65	Carbamic acid, monoammonium salt
2	4.36	4.33	Acetic anhydride
3	4.67	7.46	hydroxy-Acetic acid
4	4.81	1.56	Acetic acid
5	4.95	4.02	Ammonium acetate
6	5.19	0.54	2-Butenal
7	5.31	2.94	1-hydroxy-2-Propanone
8	5.5	0.53	1,4,2,5 Cyclohexanetetrol
9	5.7	0.70	Furan, 2,5-dimethyl-
10	5.74	0.67	Tolycaine
11	6.86	2.02	1,2-Ethanediol, monoacetate
12	7.38	3.14	Propanoic acid, 2-oxo- methyl ester
13	8.74	2.01	Furfural
14	10.19	0.50	4-Hexen-2-one
15	12.46	0.47	2(5H)-Furanone
16	12.47	6.26	2(5H)-Furanone
17	13.03	2.03	2-Cyclopenten-1-one, 2-hydroxy-
18	14.64	0.64	2-Propenamide, N-(aminocarbonyl)-
19	15.24	1.65	Phenol
20	16.27	2.53	N-Butyl-tert-butylamine
21	16.78	0.65	1,3,5-Cycloheptatriene, 1-methoxy-
22	17.14	0.84	1,2-Cyclopentanedione, 3-methyl-
23	18.18	0.50	Phenol, 2-methyl-
24	19.24	3.08	p-Cresol
25	19.76	3.61	Phenol, 2-methoxy-
26	20.67	0.66	6-Oxa-bicyclo[3.1.0]hexan-3-ol
27	25.49	3.99	Creosol
28	29.04	1.29	Phenol, 4-ethyl-2-methoxy-
29	30.93	5.14	2-Methoxy-4-vinylphenol
30	33.06	3.90	Phenol, 2,6-dimethoxy-
31	33.34	0.46	Phenol, 2,6-dimethoxy-
32	35.38	1.59	Phenol, 2-methoxy-4-(1-propenyl)-, (Z)-
33	36.62	2.57	1,2,4-Trimethoxybenzene
34	36.75	3.52	trans-Isoeugenol
35	37.58	0.81	Bicyclo[5.3.0]decane, 2-methylene-5-(1-methylvinyl)-8-methyl-
36	37.72	0.76	à-Guaiene
37	38.32	0.77	5-tert-Butylpyrogallol
38	38.83	2.40	Benzene, 1,2,3-trimethoxy-5-(2-propenyl)-
39	39.16	1.93	3',5'-Dimethoxyacetophenone
40	40.28	0.92	.gamma.-eudesmol
41	40.69	2.63	2-Naphthalenemethanol, decahydro-à, à,4a-trimethyl-8-methylene-, [2R-(2à,4aà,8aà)]-
42	41.14	0.49	Phenol, 2,6-dimethoxy-4-(2-propenyl)-
43	41.26	1.74	Phenol, 2,6-dimethoxy-4-(2-propenyl)-
44	41.55	2.04	6-Isopropenyl-4,8a-dimethyl-1,2,3,5,6,7,8,8a-octahydro-naphthalen-2-ol
45	41.69	0.64	Ethanone, 1-(4-hydroxy-3,5-dimethoxyphenyl)-
46	41.82	0.62	Diepicedrene-1-oxide
47	42.15	0.56	6-Isopropenyl-4,8a-dimethyl-1,2,3,5,6,7,8,8a-octahydro-naphthalen-2-ol
48	43.67	1.11	Tricyclo[4.4.0.0(2,7)]dec-8-ene-3-methanol, à,à,6,8-tetramethyl-, stereoisomer
49	46.28	0.56	cis-Vaccenic acid
50	47.3	0.61	Benzene, 1,1'-(1-methylethylidene)bis[4-methoxy-

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