Reprint from

Modern Methods of Plant Analysis
New Series, Volume 12
Essential Oils and Waxes
Edited by H. F. Linskens and J. F. Jackson

Springer-Verlag Berlin Heidelberg New York
London Paris Tokyo Hong Kong Barcelona
Budapest

Cedar Wood Oil — Analyses and Properties

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1 Introduction

Cedarwood oil is an important natural product for components used directly in fragrance compounding or as a source of raw components in the production of additional fragrance compounds. The oil is used to scent soaps, technical preparations, room sprays, disinfectants, and similar products, as a clearing agent for microscope sections, and with immersion lenses (Guenther 1952).

The price varies but has generally been about $4.50/lb. for Virginia cedarwood oil, $3.50/lb. for Texas cedarwood oil and $1.50-$1.75/lb. for Chinese cedarwood oil. The Chinese cedarwood oil, although almost identical in composition, is less valued because its fragrance is very different from the Texas and Virginia cedarwood oils. The commercial cedarwood oils are obtained from three genera of the Cupressaceae: *Juniperus* (Texas and Virginia oils); *Cupressus* (China) and *Cedrus* (Morocco, India) according to Bauer and Garbe 1985. The heartwood oils of the Cupressaceae are well known for having the same components across the family (i.e., evolutionally conserved), so the occurrence of similar oils in different genera should not be surprising.

The world production (1984) has been reviewed by Lawrence (1985), who reported the following (source and metric tons): Texas (*J. ashei* Buch.)—1400; Virginia (*J. virginiana* L., S.E. United States)—240; China (*Cupressus funebris* Endl.)—450; India (Himalaya, *Cedrus atlantica* Menetti)—20; Morocco (Atlas Mtns., *Cedrus deodora* Loud.)— 7; Kenya (*J. procera* Endl.)— no production at present.

Cedarwood oils have not been examined thoroughly or systematically. Many of the analyses of cedarwood oil were done by Runeberg (1960a-e, 1961) and associates (Pilo and Runeberg 1960; Pettersson and Runeberg 1961) (Table 1). For many years *J. ashei* was reported to contain only alpha-cedrene and cedrol (Guenther 1952; erroneously referred to as *J. mexicana* in Erdtman and Norin 1966 and Walker 1968, see Zanoni and Adams 1979 for nomenclatural discussion). However, more recently, Kitchens et al. (1971) reported beta-cedrene, thujopsene, widdrol, pseudocedrol, beta-chamigrene, prim cedrol, widdrene, isowiddrene, alpha-chamigrene, and cuparene (three isomers). Unfortunately, as is typical of most of the reports on identifications, not enough data were given by Kitchens et al. (1971) to allow confirmatory studies of their work.

Juniperus californica Carr.was reported (Pettersson and Runeberg 1961) to have cedrol as the major component (52%) of the heartwood volatile oils (Table 1) with a considerable amount of thujopsene (26%). A more recent study (Adams 1987) reported low yields (Table 1) of cedrol from two chemical races (A and B, Vasek and Scora 1967) of *J. californica.* It is presumed that Pettersson and Runeberg (1961) may have analyzed the wood of *J. occidentalis* Hooker instead of *J. californica.*

Juniperus communis L. was reported to have mostly thujopsene (37%); however, it is not clear how the author (Bredenberg 1961) arrived at these percentages

Table 1. Literature reports on the composition of the volatile wood oils of *Juniperus* species. Approximate percent concentration of key components was obtained when possible from the original literature cited

Species	[a]ACDR	BCDR	THJP	CPRN	CDRL	WDDL	Reference
J. ashei (=*J. mexicana* in part)	+	+	+	+	+	+	Guenther 1952; Windemuth 1945 Kitchens et al. 1971
J. ashei	1.8	1.6	60.4	2.8	19.0	1.1	Adams 1987
J. californica	2.6		26.0	1.0	52.0	0.2	Pettersson and Runeberg 1961
J. californica'A'	4.9	2.7	19.7	6.4	8.0	8.0	Adams 1987
J. californica'B'	3.9	1.9	18.7	4.7	9.3	9.2	Adams 1987
J. cedrus			82.4	3.7	2.2	2.6	Runeberg 1960a
J. chinensis			11.6	4.3	72.9	6.0	Pilo and Runeberg 1960
J. communis			37.0	3.0	2.0	1.0	Bredenberg 1961
J. conferta			+	+	+		Doi and Shibuya 1972
J. deppeana	16.9	3.9	14.9	3.9	26.4	1.0	Adams 1987
J. erythrocarpa	1.9	1.6	67.9	3.0	8.5	0.5	Adams 1987
J. excelsa					+		Rutowski and Vinogradova 1927
J. foetidissma	58.3				8.3	5.0	Runeberg 1961
J. horizontalis	+		+	+	+	+	Narasimhachari and von Rudloff 1961
J. monosperma	2.7	1.8	61.0	3.8	4.1	1.7	Adams 1987
J. occidentalis	+				+		Kurth and Lackey 1948
J. occidentalis var. *occidentalis*	8.8	2.6	18.9	1.5	38.9	1.6	Adams 1987
australis	3.3	1.3	20.1	1.5	38.2	1.6	Adams 1987
J. osteosperma (=*J. utahensis*)	12.7		47.8	12.5		13.5	Runeberg 1960b
J. osteosperma	4.0	1.8	40.0	2.6	13.2	1.5	Adams 1987
J. phoenicea			79.3	2.9	7.2	0.1	Runeberg 1960c
J. pinchotii	2.8	1.2	4.8	0.1	4.4	—	Adams 1987
J. procera	41.8			2.5	41.8		Pettersson and Runeberg 1961
J. recurva	3.5	0.9	5.1	1.8	49.0	16.7	Oda et al. 1977
J. semiglobosa		+			+		Goryaev et al. 1962
J. thurifera	23.3		15.5	3.9	27.1		Runeberg 1960d
J. scopulorum	4.3	2.4	57.9	6.1	6.1	3.0	Adams 1987
J. virginiana	35.0		30.0	2.0	4.0	2.0	Runeberg 1960e
J. virginiana	27.2	7.7	27.6	6.3	15.8	1.0	Adams 1987

[a]ACDR = alpha-cedrene; BCDR = beta-cedrene; THJP = thujopsene; CPRN = cuparene; CDRL = cedrol; WDDL = widdrol.

Juniperus horizontalis Moench is a prostrate plant that forms mats. Due to its low wood biomass, its oil composition is primarily of academic interest. Narasimhachari and von Rudloff (1961) reported that *J. horizontalis* contained alpha-cedrene, thujopsene, cuparene, cedrol, and widdrol, but relative concentrations were not reported (Table 1). *Juniperus occidentalis* was examined by Kurth and Lackey (1948), who merely reported that the wood contained alpha-cedrene and cedrol. A more recent analysis of both varieties (Table 1; Adams 1987) showed the varieties to be high in cedrol and thujopsene.

Juniperus osteosperma (referred to as *J. utahensis* Lemm. by Runeberg 1960b) had 47.8% thujopsene, with about equal amounts of alpha-cedrene, cuparene, and widdrol (Table 1). Adams (1987) found that the taxon was high in thujopsene, but reported that cedrol was also a major component (Table 1).

Juniperus virginiana L. wood was not directly analyzed by Runeberg (1960e). Using a commercial sample of cedarwood oil said to be from *J. virginiana*, he found mostly alpha-cedrene and thujopsene with a very small amount of cedrol (4%) (Table 1). However, the commercial cedarwood oil may have been precipitated or fractionally distilled to remove cedrol because Adams (1987) stated that *J. virginiana* wood (collected from native trees in Texas) contained about 16% cedrol (Table 1). Wenninger et al. (1967) analyzed the sesquiterpene hydrocarbons of American cedarwood oil (*J. virginiana*?) and reported that the oil contained 55-65% sesquiterpene hydrocarbons, with alpha-cedrene and thujopsene as the major components. Runeberg (1960e) stated that the highest yield of oil, about 3.5% of the wood (dry wt.?), was obtained from sawmill waste from older tree (i.e., trees with a greater ratio of heartwood to sapwood). Guenther (1952) obtained only a 0.2% yield by distilling sapwood of *J. virginiana*; he noted that young trees (commonly called sap cedars) yielded less than 1% oil, compared with older trees (commonly called virgin cedars), which yielded 3.5%.

2 Sample Collection

It is assumed that the reader is not only interested in the analysis of commercial cedarwood oil, but also in investigating the cedarwood oil from trees. In general, at least five trees should be sampled (obviously ten is preferred). Unfortunately, little is known about seasonal variation of wood oils. Depending on the local customs and laws, one may be faced with only a few options in collecting wood samples. I have even resorted to visiting a local woodworking shop in Ethiopia to obtain wood sample of *J. procera*. The ideal situation is to visit a site where trees are being cut for firewood, posts, lumber, etc. and obtain wood blocks directly. Failing this, one may look for broken limbs and cut off the stump section near the stem. I have also used a drill with a large wood bit (2 cm) to obtain wood from the trunk. One must be very careful to keep track of both sap- and heartwood shavings if biomass and yields are to be determined.

If a wood block is available, a wood planer is useful to produce fresh wood shavings for steam distillation. A power drill can also be used to produce wood

chips for distillation, but care must be taken so the bit does not get hot and cause a loss of the oil. Cedarwood oil appears to be very stable in intact wood blocks, as cedarwood cut in the 1930's is still being collected and distilled near Junction, Texas, with the oil being apparently acceptable.

3 Oil Extraction

Commercially, cedarwood oil is extracted in a variety of manners ranging from home-built stills to a newly designed process by Texarome Inc., Leakey, Texas (Figs. 1, 2). Distillation times vary from 8 h or more in traditional stills, but only 30 s in the Texarome process (K. E. Harwell, pers. commun). One should bear in mind that changing the distillation time, sizes of wood chips extracted or temperature and pressure of the steam can make vast differences in the cedarwood oil composition, so that comparisons of various commercial oils may vary as much by distillery as by region or species utilized.

For laboratory use, one should be most careful about placing the wood into water and boiling out the oil. Several studies (Fischer et al. 1987; Koedam and Looman 1980; Koedam et al. 1981; Schmaus and Kubeczca 1985) have shown that plants produce acidic conditions when boiled and this leads to terpene rearrange-

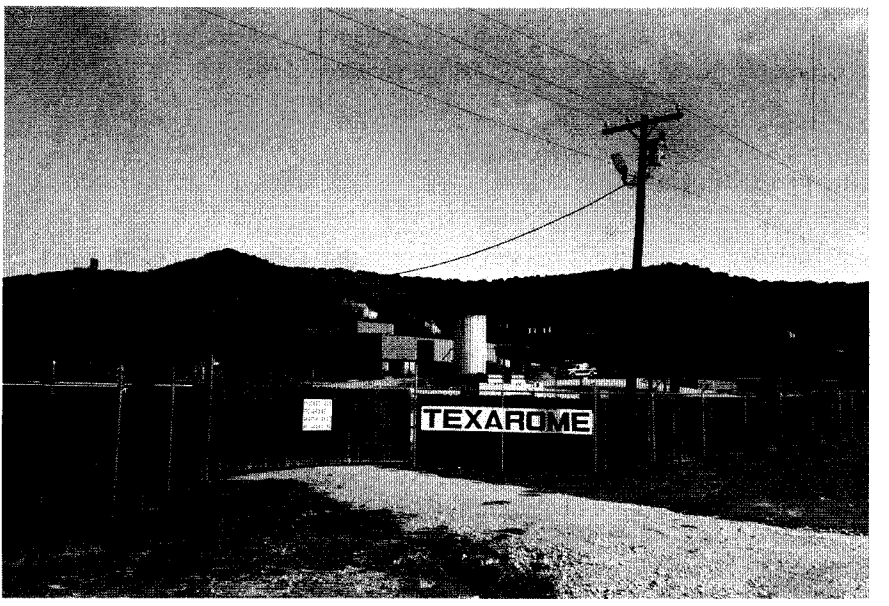

Fig. 1. Texarome's new cedarwood oil plant near Leakey, Texas. Note *Juniperus ashei* on the hillside in the background. (Photo courtesy Texarome, Inc.)

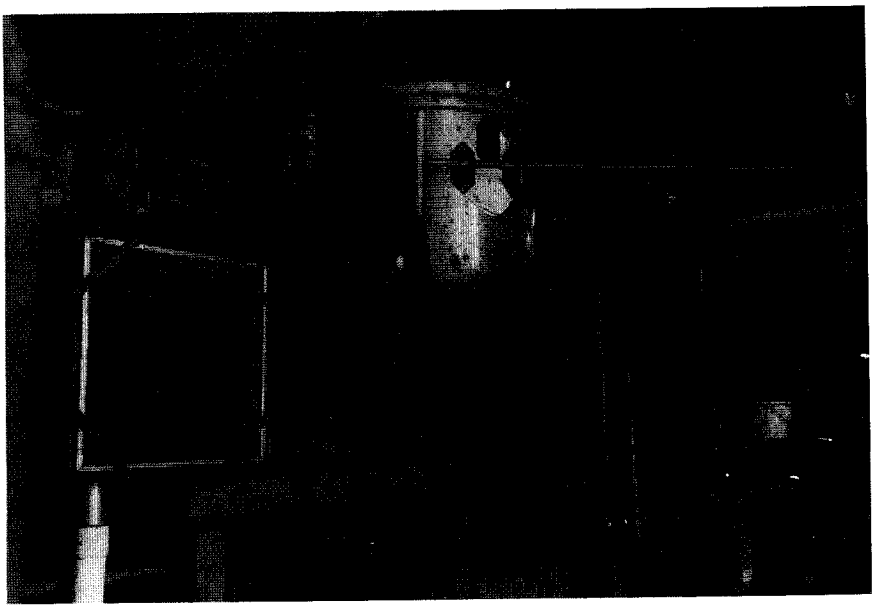

Fig. 2. Close-up of modern oil extraction equipment at Texarome, Inc. Residence time is 30 to 60 s. (Photo courtesy Texarome, Inc.)

ments and decompositions. This is shown for cedarwood oil in Fig. 3. The initial pH of the water in the boiling flask was 7.12. After 2 h boiling the wood chips directly in the flask the pH was 6.17 and the composition was quite changed (Fig. 3, upper). Steam distillation using an apparatus with the plant material suspended above the steam generator flask (Fig. 4) resulted in the chromatogram in Fig. 3 (lower). In this case, the pH of the water in the steam generator flask was 8.62 after steam distillation. The shift in the base line (Fig. 3, upper) is indicative of decomposition. Note particularly the low yield of α- and β-cedrenes (peaks 6, 7). There is a large increase in the oxygenated sesquiterpenoids (peaks 30 and upward).

Fischer et al. (1987) discuss the fact that the original (in situ) flavor components of marjoram may be quite different from those of the commercial oils. However, if one is to work within the legal and market framework that has already been established for cedarwood oil, it seems that practical work will be forced to use steam distillation extraction. Von Rudloff (1967) examined the use of direct distillation (plant material in boiling water), a Markham-type device, and a modified Clevenger-type circulatory apparatus. He preferred the modified Clevenger-type circulatory apparatus and that is essentially what I recommend (Fig. 4). I have added ball joints so the apparatus is easier to align and the ether trap can be adjusted. Notice that the plant material is placed in the cylindrical part so that only steam comes in contact with the plant material. An external heating jacket can be added to the cylindrical part to increase the distillation efficiency if

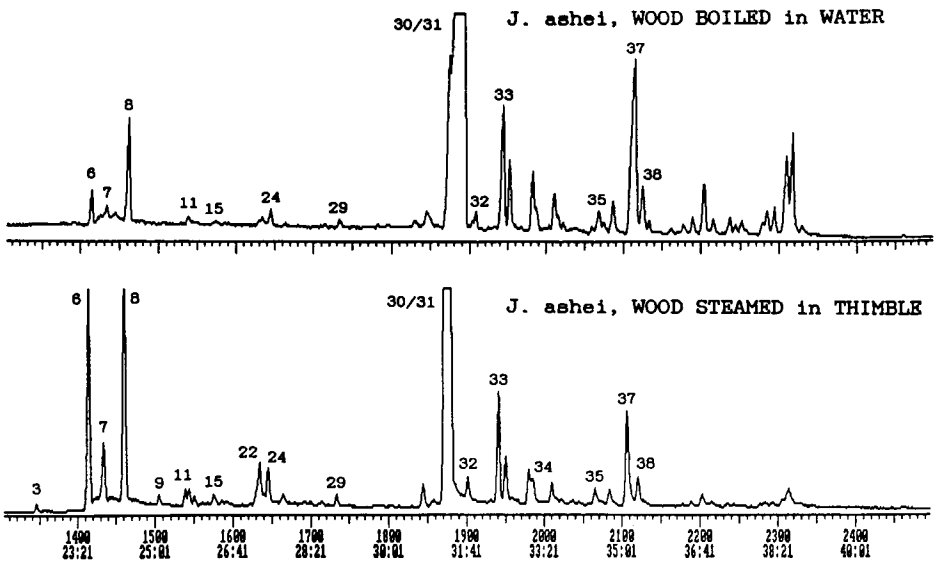

Fig. 3. Comparison of cedarwood oils obtained by hydrodistillation, boiling in water (*upper* chromatogram) and steamed in suspension (*lower* chromatogram). These and later chromatograms run on J & W DB5 silica capillary, 60–240 °C, 3°/min

desired. The condenser has also been modified so that the water jacket completely covers the ether trap area. This has resulted in much less loss of ether during distillation.

I prefer ether as the terpene trap because the ether can be evaporated by a stream of nitrogen in a hood and almost none of the terpenes are lost. Pentane could be substituted for ether. The use of hexane is discouraged because its higher boiling point results in the loss of volatile terpenes during concentration. The condenser (lower portion) should be filled with water until the water overflows into the distillation chamber. Then, the ether is placed on top of the water layer. As the distillate condenses, the oil is trapped in the ether (pentane) and the water condensate goes into the lower layer and thence back into the distillation chamber. The low density of ether allows one to trap oils that have a density greater than water. The apparatus can be run without attendance and any terpenes lost in the water are automatically volatilized as the condensate flows back into the distillation chamber.

When using the apparatus with finely ground or small wood chips, I have found it useful to place the ground wood into a sandwich of nylon screen (as used for window screens) and then place the elongated sandwich into the cylindrical chamber. If loose, finely ground material is placed directly into the cylindrical chamber, it will pack down and block the steam. Channels will form and the distillation will not proceed regularly. In addition, the distillate water, returning from the condenser, will accumulate on top of the plug and one faces the danger of

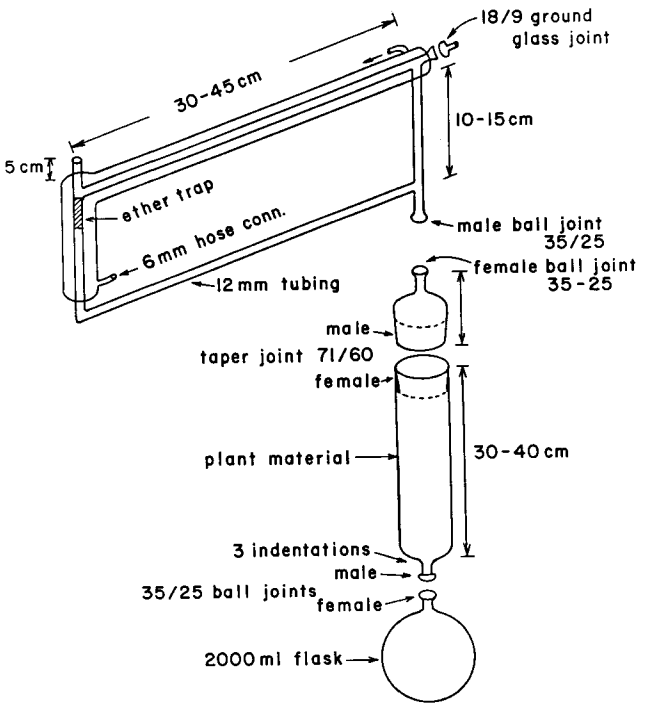

Fig. 4. Simplified Clevenger-type circulatory steam distillation apparatus recommended for cedarwood oil and general terpene extraction. Note the plant material is suspended during distillation and the oil is collected in an ether trap

running the steam generator flask to dryness. Care should be taken when handling the ether, and the entire apparatus should be placed in a well-ventilated hood when used.

The oil samples can be dried over anhydrous sodium sulfate to remove water in the ether if desired. We routinely preweigh our vials (with either compression or screw caps but in either case, using Teflon-coated caps), and evaporate the ether in a hood with nitrogen. A GC run is then used to determine the percent ether remaining in the sample and the final weight of the oil is then calculated. The samples should be stored at -20 °C or colder for long-term storage. Sealing the samples under nitrogen is also advisable for very long storage. Although decomposition of various oil samples has been mentioned to me by many colleagues, we have not experienced a problem over the past 25 years. Either our cedar and juniper oils are very stable or the aforementioned procedures mitigate decomposition. I expect that those who distill directly in water obtain oils that are quite acidic, and this may be the reason that oil decomposition is a problem. In any case, one can not assume that there will be no decomposition during long-term storage (months to years).

4 Chemical Analysis

Traditionally, cedarwood oils are defined (Walker 1968) on the basis of several physical properties: specific gravity at 15 °C (or 20 °C) 0.94-0.99; optical rotation –16 to –60°; refractive index at 20 °C 1.48-1.51; and solubility (at 20 °C) in 90 or 95% ethanol (varies with source). Although this treatment will focus on the individual chemical components, one should be aware of the practical use of the aforementioned physical properties.

4.1 Gas Chromatography

Gas chromatography has become an integral part of any essential oil analysis today. For a detailed discussion see Adams (Chap. 7, this Volume) for information on columns, carrier gases, sample injection, temperature programming and detection. All of our primary analyses are on a J & W fused silica capillary columns, DB-5, 30 m, 0.26 mm i.d., 0.25 micron coating thickness.

5 Identification

Early work on the identifications of terpenoids used component trapping from preparative GC, with subsequent liquid infrared (IR) spectral analysis for identification. The introduction of capillary columns have reduced the samples to the point that those techniques are no longer practical. The more recent development of vapor phase IR with on-the-fly analysis offers considerable promise as libraries are being compiled. However, the most practical method of identification is generally combined GC/MS or GC/MS/computer searches.

5.1 GC/MS

A large library of mass spectra is readily available from sources such as the US NBS (National Bureau of Standards, formerly the EPA/NIH data base) with thousands of spectra. Unfortunately, searches from these large data bases, with the current technology (i.e., simple matching coefficients and no retention data) do not yield reliable identifications (see Adams et al. 1979 for discussion). Although numerous papers have been written on analyses (see introduction), only the major components can be easily, unequivocally identified.

Analyses of the three major cedarwood oils are shown in Fig. 5 and a detailed list of components and retention times (on DB5) are given in Table 2. Notice that the three oils share the major components (α-cedrene, β-cedrene, thujopsene, cedrol, and widdrol). Although the minor components vary quantitatively among the oils, there is a remarkable uniformity. The off-flavor of the Chinese cedarwood oil (*C. funebris*) is apparently due to minor components.

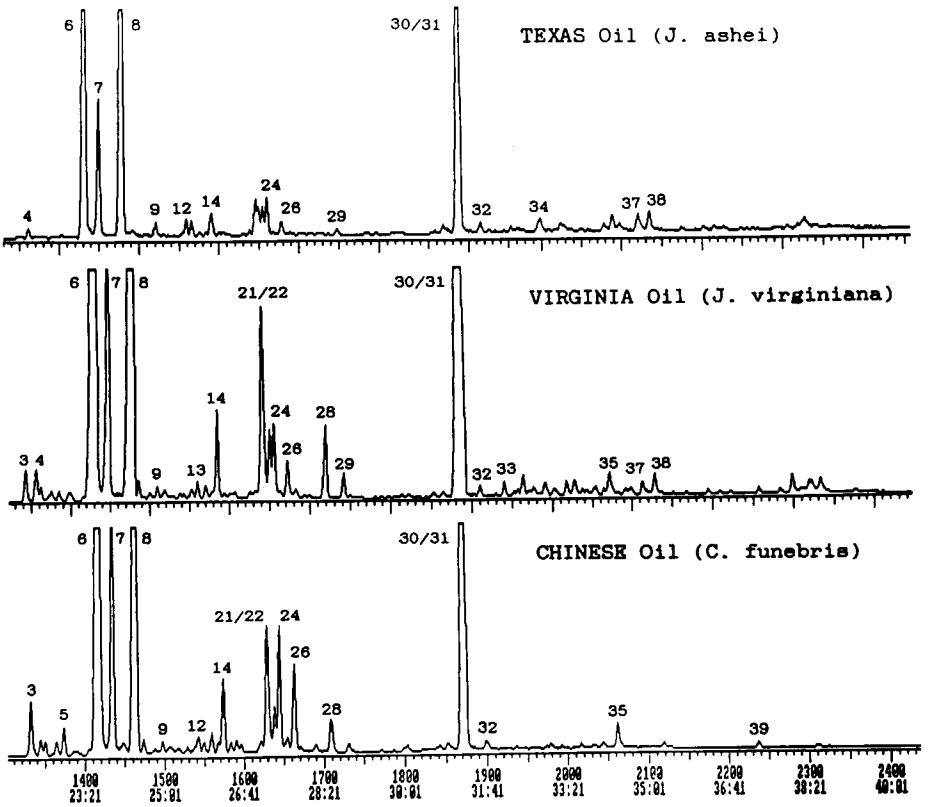

Fig. 5. Comparison of commercial cedarwood oils on a DB5 column. The *peak numbers* are identified in Table 2

Ion trap mass spectra (ITMS) for the major components are given in Figs. 6 and 7. Although the ITMS spectra are generally quite similar to quadrapole mass spectra (Adams 1989), there is a large reduction in ion 151 in both cedrol and widdrol on the ion trap. It might be noted that cedrol is very sensitive to space charging effects (overloading) and tuning on the ion trap. We use cedrol as a tuning standard on the ion trap due to its sensitivity (Adams 1989).

6 Properties

The general properties of cedarwood oils have been mentioned in the introduction. In this section, I would like to focus on several of the more unusual bioactivity properties.

Table 2. Cedarwood oil compositions from Texas (*Juniperus ashei*), Virginia (*J. virginiana*) and China (*Cupressus funebris*)

RT[a]	Compound	Texas	Virginia	China
1.	734 Camphor	0.2	—	—
2.	990 Carvacrol, methyl ether	—	—	0.7
3.	1341 Sesquiterpene	—	0.7	1.7
4.	1354 Sesquiterpene	0.3	0.7	0.5
5.	1384 Sesquiterpene	—	0.2	0.8
6.	1421 α-Cedrene	30.7	21.1	26.4
7.	1441 β-Cedrene	5.5	8.2	9.2
8.	1467 Thujopsene	25.0	21.3	29.9
9.	1507 α-Himachalene	0.5	0.2	0.2
10.	1538 cis-β-Farnesene	—	0.1	0.1
11.	1547 Thujopsadiene	0.1	—	—
12.	1551 α-Acoradiene	0.7	0.2	0.6
13.	1558 β-Acoradiene	0.6	0.3	0.3
14.	1581 β-Chamigrene	1.1	1.8	2.2
15.	1585 Γ-Himachalene	0.1	—	—
16.	1594 Γ-Curcumene	0.1	0.1	0.2
17.	1602 ar-Curcumene	0.1	0.1	0.4
18.	1608 β-Selinene	—	0.1	0.2
19.	1624 Valencene	0.1	0.1	—
20.	1631 (β-Alaskene)	0.2	0.1	0.1
21.	1633 α-Selinene + ?	1.5	3.0	3.1
22.	1643 β-Himachalene	1.4	2.1	1.4
23.	1646 (α-Chamigrene)	1.2	1.6	1.4
24.	1652 Cuparene	1.7	1.6	3.4
25.	1667 β-Bisabolene	—	—	0.4
26.	1675 α-Alaskene (=Γ-acoradiene)	0.7	0.9	2.6
27.	1701 *trans*-β-Farnesene	—	—	0.1
28.	1719 Sesquiterpene	—	1.6	1.1
29.	1739 Sesquiterpene alcohol	0.3	0.6	0.3
30.	1876 Cedrol	19.1	22.2	9.6
31.	1878 Widdrol	1.6	2.3	9.5
32.	1907 6-Isocedrol	0.4	0.2	0.1
33.	1944 Cubenol	0.2	0.1	—
34.	1966 *trans*-3-Thujopsanone	0.8	—	—
35.	2072 α-Bisabolol	0.4	0.6	0.8
36.	2085 8-Cedren-13-ol	0.9	—	—
37.	2116 Sesquiterpene alcohol	0.9	0.3	—
38.	2128 Sesquiterpene alcohol	0.8	0.6	—
39.	2246 Cedryl acetate	—	—	0.1
40.	2597 Cembrene	—	—	0.1
41.	2891 Abietadiene	—	—	0.3

[a]Compounds are listed in order of their retention times (RT) on a J β W DB5 capillary column. Compounds in parenthesis are tentatively identified.

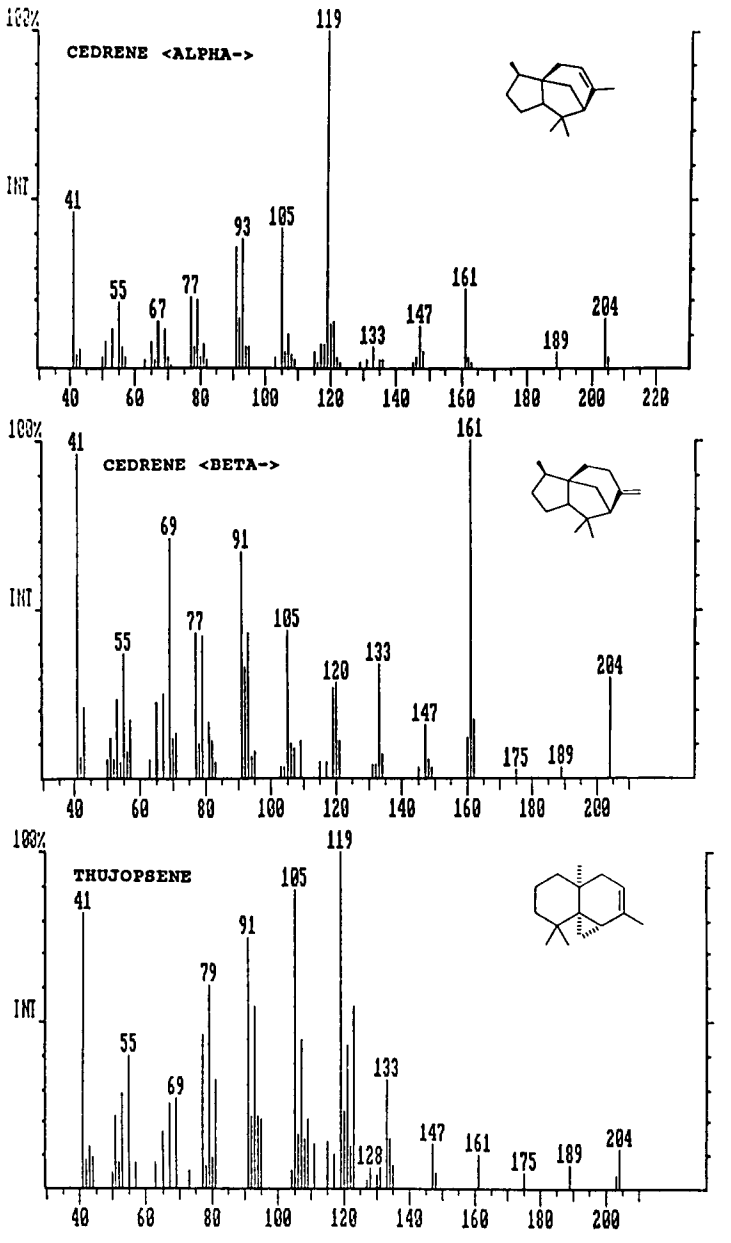

Fig. 6. Ion trap mass spectra of α-cedrene, β-cedrene, and thujopsene (*cis*)

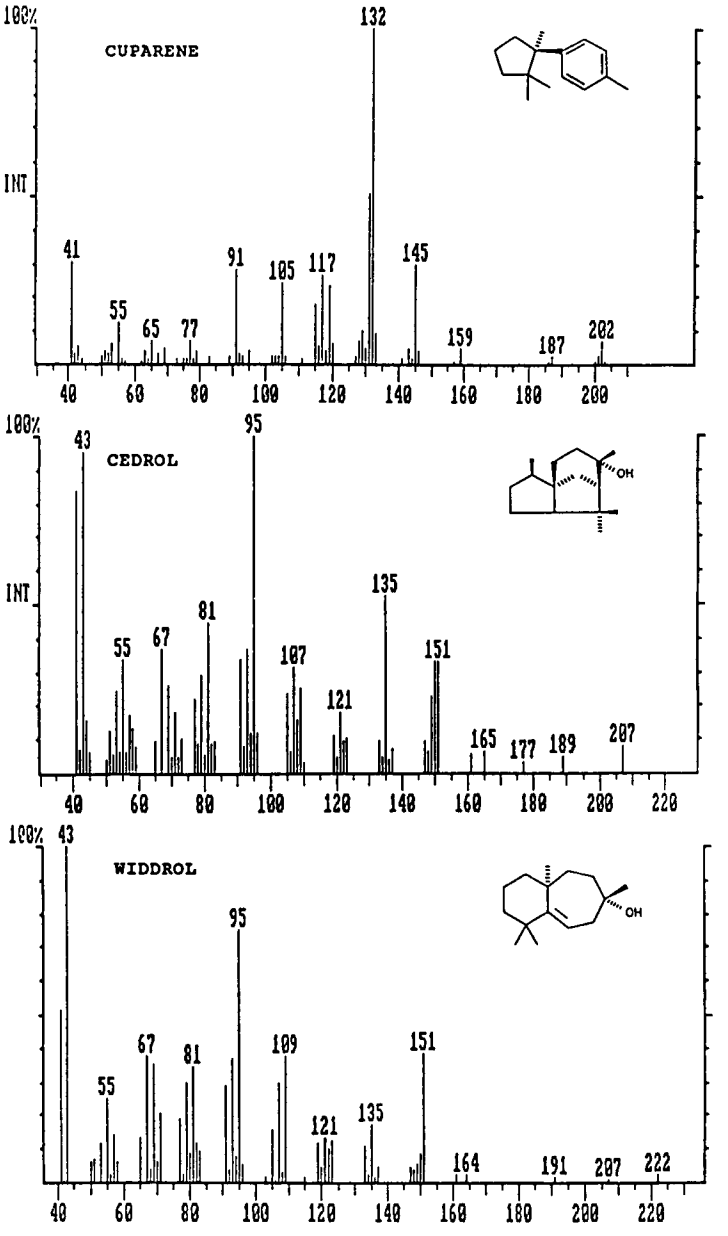

Fig. 7. Ion trap mass spectra of cuparene, cedrol, and widdrol

6.1 Antimicrobial Activities

Hexane and methanol extracts of heartwood, bark/sapwood, and leaves of 12 taxa of *Juniperus* from the United States were assayed for anti-fungal and anti-bacterial activities (Clark et al. 1990). The hexane extracts of the heartwood (which contains the cedarwood oil) of several junipers appear comparable in antibacterial activity to streptomycin. No anti-fungal activities comparable to amphotericin B were found in either the hexane or methanol extracts of heartwood. Additional research is needed to isolate and determine the anti-bacterial components.

6.2 Insecticidal Activities

Oda et al. (1977) examined the insecticidal activities of several extracts of the heartwood of *Juniperus recurva* from Nepal. The insecticidal activities were found in the steam volatile fraction(i.e., cedarwood oil). Detailed examination of individual components revealed the following LD_{50} μg/mosquito: α-cedrene— 33.5; β-cedrene— not active; thujopsene,— 4.5; acoradiene— not active; β-chamigrene— not active; curparene— not active; 8, 14-cedranoxide— 10.7; 8-cedren-13-al— not active; cedrol— 21.2; widdrol— not active; 8-cedren-13-ol acetate— not active; 8-cedren-13-ol- 6.6; 8S,13- and 14-cedrane-diols— not active. Clearly the most insecticidal components were thujopsene and 8-cedren-13-ol. Again, additional research is warranted on both *Juniperus* and *Cupressus* (and other Cupressaceae species) for natural insecticidal compounds to replace chlorinated pesticides of current usage.

6.3 Termiticidal Activities

The control of termites is a world wide problem. Current preservatives use arsenic, and chlorinated and copper-based products, all of which are toxic to humans and/or carcinogenic. Carter (1976) found that termites (*Reticulitermes flavipes*) could not survive on sawdust from *Juniperus virginiana* or on filter paper treated with a pentane extract (cedarwood oil) of *J. virginiana* sawdust.

Subsequently, Adams et al. (1988) found extremely high termiticidal activities in the heartwood sawdust from all 12 of the United States junipers examined. Hexane extracts of the heartwoods revealed that treated paper showed termiticidal activities for seven of the taxa. Additional research is continuing (McDaniel and Adams in prep.) to determine if the extracts are anti-feedants and/or toxic to termites. Because the junipers are used for posts in the United States, it is obvious that wood preservatives are in the wood. These same kinds of observations about wood rotting should be used to select promising species for additional termiticidal (and wood rotting) tests around the world (particularly in the Cupressaceae).

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