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**Organic geochemistry of oils from Oil Spring and Florence Oil Field
near Cañon City, Colorado**

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Abstract

Oil Spring is an oil seep located approximately 6 miles north of Cañon City, Colorado. The purpose of this study is to determine whether the oil from Oil Spring and the oil from nearby Florence oil field share a common source. Bulk and molecular geochemical analyses show that the oil seep is most likely derived from a group of geochemically similar Cretaceous source rocks including the Carlile Shale, Greenhorn Limestone, Graneros Shale, and the Mowry Shale. The Florence oil is derived from the Sharon Springs Member of the Upper Cretaceous Pierre Shale.

Introduction

Oil Spring is an oil seep located along Fourmile Creek (formerly called Oil Creek) approximately 6 miles north of Cañon City, Colorado and the Florence oil field is approximately 10 miles southeast of Cañon City. Oil Spring and Florence field lie within the Cañon City-Florence basin which is located just southwest of the Denver basin. The purpose of this study is to determine whether the oil from Oil Spring and oil from Florence field share a common source. Most of the oil in the Denver basin is produced from Lower Cretaceous "D" and "J" sandstone reservoir rocks and is thought to be derived from a group of geochemically similar Cretaceous source rocks including the Carlile Shale, Greenhorn Limestone, Graneros Shale, and the Mowry Shale (Clayton and Swetland, 1980). The oil from Florence field is produced from fractured Upper Cretaceous Pierre Shale, and is believed to be derived from the Sharon Springs Member of the Pierre Shale (Swetland and Clayton, 1976; Gautier and others, 1984). The oil from Oil Spring seep has previously been suggested to be derived from Cretaceous source rocks (Clayton and others, 1984, personal communication, in Powers and others, 1984). However, the oil from Oil Spring is seeping from the Jurassic Morrison Formation, which suggests a Jurassic or older source rock.

Two oil seep samples, one oil-stained sand reported to be derived from the seep, and one oil sample from Florence field were analyzed for bulk and molecular organic composition (Table 1). One oil seep sample (97032-001) was reportedly collected in about 1983 and the other seep sample (97032-002) was collected in 1995. The oil-stained unconsolidated sand sample (97032-004 or "tourist" sample) was contained in a vial with a label claiming to be a sample of the first oil discovery in Colorado. We presume that this type of sample was sold to tourists at local gift shops. The oil samples were collected by Dr. Donald H. Kupfer, retired geologist from Cañon City, and the oil-stained sand sample was provided by Dr. Robert J. Weimer, emeritus professor from the Colorado School of Mines. The Appendix contains a verbatim description of the samples.

Analytical Procedures

Oil samples were transferred from the original plastic containers into metal cans to preclude organic contamination. Oil gravity was measured directly using hydrometers calibrated to °API and corrected for temperature. The oil from the oil-stained sand was extracted by soaking the sand in chloroform for approximately one hour at room temperature. Most of the chloroform was removed from the extract using a rotary evaporator with moderate vacuum and a water bath temperature of about 35°C. Oil concentration in the extract was determined gravimetrically on an aliquot of known volume. An aliquot of known concentration was placed in a vial and the volume was reduced by evaporation to about 1 ml under a stream of nitrogen gas at room temperature. About 2 ml of iso-octane was added to the sample and mixed with a vortex mixer on low speed. The sample was gently evaporated under a stream of nitrogen gas to about 1 ml. The iso-octane addition and evaporation step was repeated at least three times until the chloroform was completely displaced by the iso-octane. As the iso-octane replaced the

chloroform, the asphaltene fraction of the oil became insoluble and precipitated out of solution. Iso-octane was also added to the oil samples to precipitate the asphaltene fractions. All asphaltene precipitates were then removed by filtration. The maltene fractions (oil with asphaltenes removed) were separated into saturated hydrocarbon, aromatic hydrocarbon, and resin fractions by elution chromatography using alumina/silica columns and elution solvents of increasing polarity.

The C₈+ saturated and aromatic hydrocarbon fractions were analyzed with a Hewlett Packard 6890 gas chromatograph (GC) equipped with a 60m x 0.32mm fused-silica capillary column (DB-1) and a flame ionization detector. The GC temperature for the saturated hydrocarbons was programmed from 50°C to 330°C at 4.5°C/minute, and held at 330°C for 15 minutes. The aromatic hydrocarbons were analyzed under the same conditions except that the starting temperature was 40°C. Biomarker distributions were determined by analyzing combined saturated and aromatic hydrocarbon fractions by gas chromatography-mass spectrometry. A Hewlett Packard 5890 GC with a DB-1701 60 m x 0.32 mm column was directly interfaced to a VG7035 magnetic sector mass spectrometer operating with a dynamic mass resolution of 3000 (5 percent valley). Multiple ion detection was accomplished by switching the accelerating voltage at a constant magnetic field. The selected ions were m/z 191.1800 (terpanes), m/z 217.1956 (steranes), m/z 231.1174 (triaromatic steroids) and m/z 253.1956 (monoaromatic steroids). Peak identifications were based on elution time and mass spectra (Philp, 1985).

Stable carbon isotope ratios were determined for the C₁₅+ saturated and aromatic hydrocarbon fractions using a Carlo Erba elemental analyzer (EA) interfaced with a Micromass Optima continuous-flow isotope ratio mass spectrometer (IRMS). Sample aliquots were heated to approximately 1800°C in the EA quartz combustion tube filled with oxygen. The evolved CO₂ passed through chromium oxide (to complete oxidation), copper granules (reducing agent), and anhydrous (to remove water) before being swept into the IRMS with a helium carrier gas. The results are expressed in the delta (δ) notation that represents the deviation of the ¹³C/¹²C ratio in parts per thousand (per mil, or ‰) relative to the Peedee belemnite (PDB) standard.

Results and Discussion

The bulk geochemical results are listed in Table 2. The 1983 Oil Spring sample has been moderately altered by evaporation and biodegradation as indicated by the lower gravity (20°API) and reduced relative concentration of normal alkanes (Figure 1). This is not surprising given that the oil reportedly sat in an open barrel for more than ten years (Appendix). The Oil Spring sample that was collected in 1995, and the Florence oil sample are relatively unaltered except for minor evaporation of the light normal alkanes between C₈ and C₁₅ (Figure 1). The API gravity of the Florence oil sample (30°) is similar to that reported for the field (32°) by Powers and others (1984). The extracted Tourist sample has experienced severe evaporation as indicated by the missing

hydrocarbons below C₁₃ and has experienced minor biodegradation as indicated by the lower concentration of normal alkanes and the large baseline hump also known as the unresolved complex mixture (Figure 1).

The relative degree of alteration of the seep samples is also indicated by the relative concentrations of the column chromatography fractions listed in Table 2. For example, the most degraded sample, Tourist, has the highest concentration of asphaltenes and the lowest concentration of saturated hydrocarbons. Conversely, the 1995 seep sample has the lowest concentration of asphaltenes and the highest concentration of saturated hydrocarbons.

The pristane/phytane ratios of the 1983 and 1995 samples are nearly identical (2.18 and 2.15) even though the former sample has experienced some minor biodegradation. The Tourist sample has a lower pristane/phytane ratio (1.42) that probably resulted from increased evaporative loss of the lighter isoprenoid (pristane). The seep oil values are similar to those of oils from the Lower Cretaceous "D" and "J" sandstone reservoirs in the Denver Basin. Clayton (personal communication, 1998) reports pristane/phytane values ranging from 1.86 to 2.14 and averaging 1.95 for 16 oil samples. The Florence oil has a slightly lower pristane/phytane ratio (1.65) and is similar to a Florence oil (1.6) reported by Gautier and others (1984).

The $\delta^{13}\text{C}$ saturated and aromatic hydrocarbon data of the oil seep exhibit some scatter due to alteration effects of the 1983 and Tourist samples (Table 2). We consider the 1995 seep sample to be the least altered and, therefore, the $\delta^{13}\text{C}$ data to be the best values to use for correlation purposes. The 1983 seep sample has a slightly heavy $\delta^{13}\text{C}$ saturated hydrocarbon value and a significantly light $\delta^{13}\text{C}$ aromatic hydrocarbon value when compared with the other two seep samples. The heavy saturated hydrocarbon isotope ratio may be due to biodegradation of the n-alkanes but the light aromatic hydrocarbon isotope ratio cannot be explained. The Tourist $\delta^{13}\text{C}$ values are slightly heavier than the 1995 seep sample, probably due to greater biodegradation of the Tourist sample. The Florence oil has the lightest $\delta^{13}\text{C}$ values for both saturated and aromatic hydrocarbons, and is similar in isotopic composition to another Florence oil ($\delta^{13}\text{C}$ sats = -28.58, $\delta^{13}\text{C}$ arom = -27.47) reported by Gautier and others (1984). The 0.6 per mil difference between the Florence oil and the oil seep is not large but is distinct enough to discount analytical error. The difference may be due to a slightly different source composition, slight difference in maturity, or minor alteration. Clayton and Swetland (1980) report that the $\delta^{13}\text{C}$ saturate values of the oils from the Lower Cretaceous "D" and "J" sands of the Denver Basin range from -28.0 to -29.0 per mil. Based on these data the oil seep (-28.05 per mil) and the Florence oil sample (-28.65 per mil) correlate with the Lower Cretaceous oils.

The aromatic hydrocarbon fractions of the oil seep samples appear to be relatively unaltered in the 1983 and 1995 samples and significantly evaporated in the Tourist sample as indicated by the gas chromatography data (Figure 2). The aromatic hydrocarbon composition of the Florence oil appears to be quite different with small

concentrations of naphthalenes and high concentrations of unknown compounds eluting after 50 minutes.

Biomarker compounds are relatively resistant to biodegradation and evaporation and, consequently, are useful for correlation. The biomarker mass chromatograms are shown on Figures 3 through Figure 6 with compound identifications in Table 3 and selected biomarker ratios in Table 4. The tricyclic and pentacyclic terpane (m/z 191), sterane (m/z 217), monoaromatic sterane (m/z 253), and the triaromatic sterane (m/z 231) composition of the three oil seep samples are virtually identical (Table 4). This correlation indicates that all of the bulk geochemical differences between the seep samples discussed above are caused by biodegradation, evaporation, or other alteration effects.

Although the biomarker compositions of the Florence and seep oils are similar, differences are observed upon closer inspection. The Florence oil contains two extra compounds in the m/z 191 chromatogram (Figure 3); an unidentified terpane (peak a) and oleanane (peak o). Oleanane is derived from angiosperms which first became prominent in the Late Cretaceous (Ekweozor and Udo, 1988). The Florence oil also contains relatively higher concentrations of C₂₅ tricyclic terpane (peak 3), C₂₄ tetracyclic terpane (peak 5), and norhopane (peak 8) when compared to the seep oil (Table 4). The sterane mass chromatogram (Figure 4) of the Florence oil displays a relatively higher concentration of C₂₈ steranes and a lower concentration of C₂₉ steranes when compared to the oil seep (Table 4). These data suggest that the Florence oil was derived from a source rock with relatively higher concentration of marine organic matter and a lower concentration of terrestrial organic matter than the source rock of the seep oil (Huang and Meinschein, 1979; Moldowan and others, 1985). The seep oil has a relatively higher diasterane concentration (C₂₇ R dia/C₂₇ R cholestane) which may be caused by either slightly higher thermal maturity (Seifert and Moldowan, 1978) or a more clay-rich source rock (Sieskind and others, 1979). The higher C₂₈ steranes in the Florence oil is also reflected in the triaromatic steranes (Figure 5) with a higher C₂₇ (one carbon is lost during aromatization). However, the monoaromatic sterane distribution (Figure 6) of the two oils are quite similar. The low molecular weight triaromatic steranes (C₂₀, C₂₁) exhibit higher concentration in the oil seep, suggesting higher levels of thermal maturity (Table 4).

Interestingly, there is an apparent contradiction as to the relative thermal maturity of the two oils (Table 4). Several geochemical parameters suggest that the seep oil (1995) is more mature than the Florence oil including pristane/n-C₁₇, Ts/Tm, C₂₉ Ts/norhopane, diasterane/sterane, C₂₃ tricyclic/hopane and C₂₀+C₂₁ tri/triaromatic sterane ratios. Conversely, some parameters suggest that the Florence oil has about the same thermal maturity including the 20S/S+R $\alpha\alpha\alpha$ C₂₉ sterane, $\beta\beta/\beta\beta+\alpha\alpha$ C₂₉ sterane, and triaromatic/triaromatic+ monoaromatic sterane ratios.

Published biomarker data on oils in Colorado is very limited. The m/z 191 and 217 mass chromatograms of the "J" sandstone oil from Lindon Field (Clayton, 1989) are very similar to those of the seep oil. The m/z 217 mass chromatogram of the Florence oil is very similar to that of a Florence oil reported by Gautier and others (1984).

Conclusions

1. The two oil seep samples and the oil-stained sand sample (Tourist) constitute one oil family probably derived as reported from the Oil Spring near Cañon City, Colorado. Differences in composition can be explained as variations in biodegradation and/or evaporation of the parent oil after reaching the surface.
2. The oil seep composition is similar to that of the Florence oil and to other Cretaceous oils in Denver Basin, Colorado. However, minor but distinct compositional differences indicate that the seep oil is not derived from the same organic facies as the Florence oil. Swetland and Clayton (1976) also found that the Florence oil does not correlate with the Lower Cretaceous oils based on C₄ to C₇ hydrocarbon composition.
3. The oil seep is most likely derived from Lower Cretaceous source rocks as indicated by the similar $\delta^{13}\text{C}$ hydrocarbon and pristane/phytane values as the Lower Cretaceous oils in the Denver Basin, the similarity in biomarker composition with the Lower Cretaceous "J" sandstone oil from Lindon Field, and the absence of oleanane. It is less likely, but possible that the oil seep may be derived from the Sharon Springs Member of the Pierre Shale that has a relatively higher content of terrestrial organic matter; Gautier and others (1984) found that Sharon Springs has higher terrestrial organic matter in the northern-central Denver Basin (Boulder and Fort Collins, Colorado).
4. The Florence oil sample from this study correlates with another Florence oil reported by Gautier and others (1984) and with their extracts of the Sharon Springs Member of the Pierre Shale. Furthermore, the Florence oil sample in the present study contains oleanane which is consistent with an Upper Cretaceous (or younger) source rock.

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Table 1. Sample Information

Sample Identification	Job Number	Location	Latitude	Longitude	Elevation (ft)
Oil Spring 1983, K/O-1	97032-001	Sec3-T18S-R70W	N38.51340	W105.21127	5620
Oil Spring 1995, K/O-2	97032-002	Sec3-T18S-R70W	N38.51340	W105.21127	5620
Oil Spring, Tourist vial	97032-004	Sec3-T18S-R70W	N38.51340	W105.21127	5620
Fee 6, Well #370, K/O-3	97032-003	Sec21-T19S-R69W	N38.38317	W105.12302	5206

Table 2. Bulk Oil Geochemistry

Sample Identification	Grav °API	Sats wt%	Arom wt%	NSO wt%	Asph wt%	Rec wt%	Vol wt%	S/A	Pr/Ph	CPI	OEP	$\delta^{13}\text{C}$ Sats	$\delta^{13}\text{C}$ Arom
Oil Spring 1983, K/O-1	20.3	55.9	22.8	11.3	10.0	85.0	8.8	2.5	2.18	nd	nd	-27.36	-27.34
Oil Spring 1995, K/O-2	25.9	65.2	21.1	9.4	4.2	80.8	5.3	3.1	2.15	1.00	0.99	-28.05	-26.94
Oil Spring, Tourist vial	nd	35.8	13.7	15.5	34.9	93.4	0.0	2.6	1.42*	1.05	1.02	-27.69	-26.88
Fee 6, Well #370, K/O-3	30.0	74.0	20.1	5.6	0.3	93.1	10.6	3.7	1.65	1.05	1.01	-28.65	-27.59

Definitions and Comments

Grav = oil gravity in °API

Sats = normalized weight percent C₁₅₊ saturated hydrocarbons

Arom= normalized weight percent C₁₅₊ aromatic hydrocarbons

NSO = normalized weight percent C₁₅₊ N, S, O - containing hydrocarbons

Asph = normalized weight percent C₁₅₊ asphaltenes (insoluble in iso-octane)

Rec = weight percent recovered from column (non recovered fractions include lost volatiles less than C₁₅ , highly polar and high molecular weight compounds held up in the column)

Vol= weight percent volatile less than C₁₅

S/A= saturated hydrocarbons/aromatic hydrocarbons, w/w

Pr/Ph = pristane/phytane, peak height

CPI = carbon preferential index from n-C₂₅ to n-C₃₅, peak height (Hunt, 1979)

OEP= odd/even predominance centered at n-C₂₉, peak height (Scalan and Smith, 1970)

nd = no data

* = not reliable due to evaporation

Table 3. Tentative Biomarker Compound Peak Identifications

1	C ₂₃ Tricyclic terpane
2	C ₂₄ Tricyclic terpane
a	unidentified terpane
3	C ₂₅ Tricyclic terpane
4	C ₂₆ [22S] Tricyclic terpane + C ₂₆ [22R] Tricyclic terpane
5	C ₂₄ Tetracyclic terpane
6	18 α Trisnorneohopane [C ₂₇ Ts]
7	17 α Trisnorhopane [C ₂₇ Tm]
8	Norhopane [C ₂₉]
9	18 α Neonorhopane [C ₂₉]
o	Oleanane [C ₃₀]
10	Hopane [C ₃₀]
11	22S Homohopane [C ₃₁]
12	22R Homohopane [C ₃₁]
13	22S Bishomohopane [C ₃₂]
14	22R Bishomohopane [C ₃₂]
15	5 α 14 α 17 α 20R cholestane [C ₂₇]
16	5 α 14 α 17 α 20R Methylcholestane [C ₂₈]
17	5 α 14 α 17 α 20S 24-Ethylcholestane [C ₂₉]
18	5 α 14 β 17 β 20R 24-Ethylcholestane [C ₂₉]
19	5 α 14 β 17 β 20S 24-Ethylcholestane [C ₂₉]
20	5 α 14 α 17 α 20R 24-Ethylcholestane [C ₂₉] + 5 α 14 α 17 α 20S 24-n-propylcholestane [C ₃₀]
21	5 α 14 α 17 α 20R 24-n-propylcholestane [C ₃₀]

Table 4. Comparison of seep and Florence oils using biomarker data.

Biomarker Ratio	Tourist 97032- 004	1983 seep 97032- 001	1995 seep 97032- 002	Florence 97032- 003	Comparison
C ₂₄ tricyclic/C ₂₃ tricyclic	0.78	0.71	0.68	0.67	
C ₂₅ tricyclic/C ₂₃ tricyclic	0.68	0.59	0.57	0.77	negative correlation
C ₂₄ tetracyclic/C ₂₆ tricyclic	0.47	0.42	0.45	0.62	negative correlation
C ₂₃ tricyclic/hopane	0.09	0.10	0.10	0.07	maturity difference
Ts/Tm (peak 6 / peak 7)	2.88	3.14	2.85	1.46	maturity difference
Norhopane/hopane	0.34	0.35	0.34	0.46	negative correlation
Neonorhopane/norhopane+neo.	0.44	0.46	0.44	0.32	maturity difference
Oleanane/hopane	0.03	0.04	0.04	0.11	negative correlation
C ₃₁ S/S+R homohopane	0.60	0.59	0.58	0.62	same maturity
C ₃₂ S/S+R homohopane	0.57	0.56	0.56	0.58	same maturity
C ₂₇ R dia/C ₂₇ R cholestane	0.81	0.77	0.86	0.41	negative correlation
C ₂₇ /C ₂₇ -C ₂₉ steranes	0.44	0.46	0.45	0.44	
C ₂₈ /C ₂₇ -C ₂₉ steranes	0.25	0.25	0.25	0.31	negative correlation
C ₂₉ /C ₂₇ -C ₂₉ steranes	0.31	0.28	0.29	0.24	negative correlation
C ₃₀ /C ₂₇ -C ₃₀ steranes	0.07	0.06	0.08	0.08	
S/S+R C ₂₉ ethyl cholestane	0.47	0.49	0.46	0.48	same maturity
ββ/αα C ₂₉ ethyl cholestane	0.58	0.60	0.59	0.59	same maturity
C ₂₀ +C ₂₁ tri/triaromatics	0.24	0.26	0.26	0.04	maturity difference
Tri/triaromatics + monoaromatics	0.78	0.74	0.73	0.69	same maturity
C ₂₈ tri/C ₂₈ tri +C ₂₉ monoaromatics	0.79	0.74	0.76	0.76	same maturity

Appendix. Sample descriptions that came with the samples.

Below is a verbatim description of the three “K/O” samples:

Oil Samples from Florence Basin
D.H. Kupfer, Sept. 1995.

Sample #1: K/O-1 Oil Spring, 1983?

After Beale (sp?) (of Woodland Park), leasee, died about 1983, owner, Frank Dilley, cleaned up what he found left around the Oil Spring (equipment, etc). Then, or probably somewhat later, he removed some drums of oil and plastic gallon jugs of oil to Dilley’s barn (2 miles north of the Oil Spring).

This sample is from one of the gallon jugs, and probably represent Oil Spring oil of circa 1982 +/- 2 years. The jug was 4/5 full and had a small hole above the oil line. Presumably, it has been stored in the plastic milk-type jug for over ten years and possibly was open to minor air circulation for part of that time.

Sample #2: K/O-2 Oil Spring, 1995, (27Aug)

In 1993, the shaft at the Oil Spring was filled with sand/mud with a back-hoe, and the general drainage disrupted. During the summer of 1994, no seepage was observed. In April, 1995, some new oil seemed to have seeped out around the drill-hole pipe, a few feet south of the filled shaft. On July 21, 1995, more apparent seepage was observed, and a small pit (1x1x1 feet) was dug and covered with a bucket. On August 27, the pit was ¾ full, but mainly with water (brine?). A 2-quart sample was taken and the water decanted, to the present sample (5 ounces).

Sample #3: K/O-3 Florence Oil Field, September 2, 1995
Oil from Fee 6, Well #370, SW, NW, Section 21, T19/R69.

Taken 2Sp95 from the feedpipe leading to tank (well was not being pumped at this time). Well produces ¾ to 1 bbl/dy, but having problems at the moment, so not being pumped until fixed. Kelly Black, the pumper, said that this well had not produced for about 20 years (see Washburne, below). It was recompleted and the second production started last August (1994) and it has been flowing OK since (K. Black).

It is a very rich golden brown (Black called it "green"), light, thin oil. Black says a lot of gasoline-content is typical of the field; not much variation in the oil of the field from well to well, or from north to south end (this well is north).

Washburne (USGS, 1909, page 540), lists this as a United Oil Company well; his map shows it was active in 1909, a well with gas, and an oil production over 1 million gallons (from legend).

Below is the label text of the “Tourist” vial of Oil Spring:

First Oil Discovery in Colorado

The oil in this bottle is from the seep on Oil Creek, 6 miles north of Canon City. Here in 1863 A. M. Cassidy brought in the first oil well in Colorado, only about three years after the first discovery of oil in the United States, at Titusville, Pennsylvania.

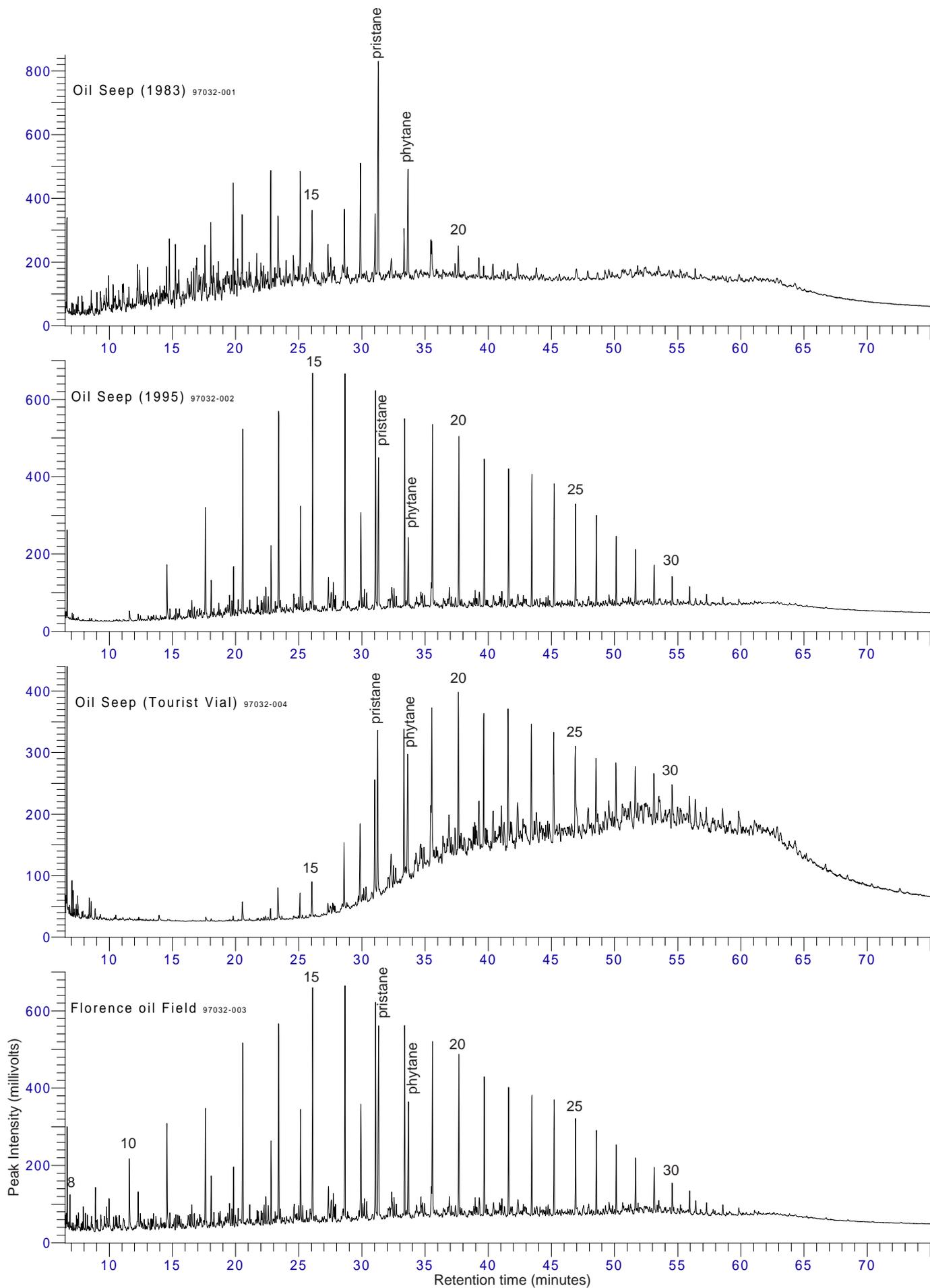


Figure 1. Gas chromatograms of saturated hydrocarbons. Numbered peaks are n-alkane carbon numbers.

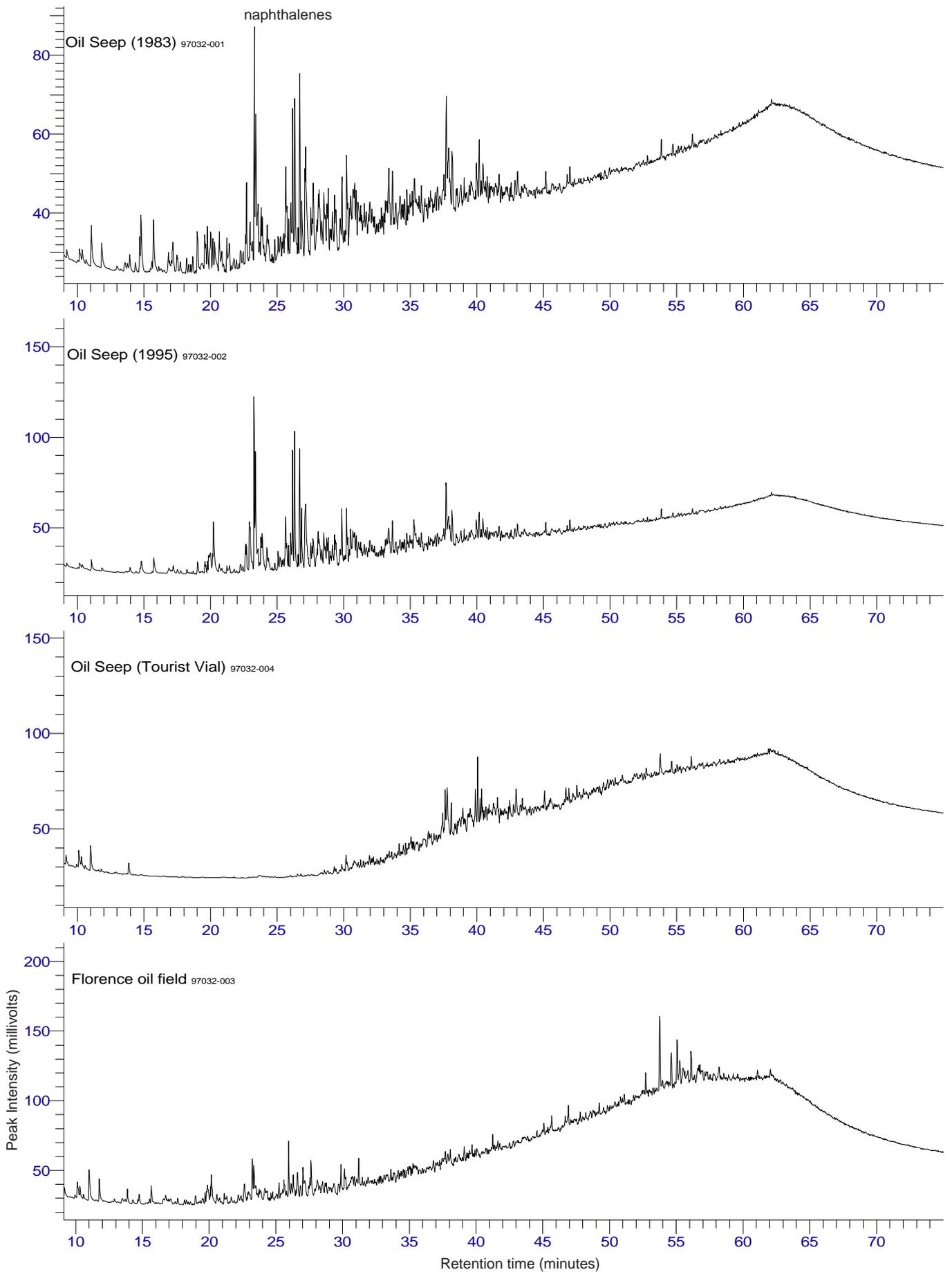


Figure 2. Gas chromatograms of aromatic hydrocarbons.

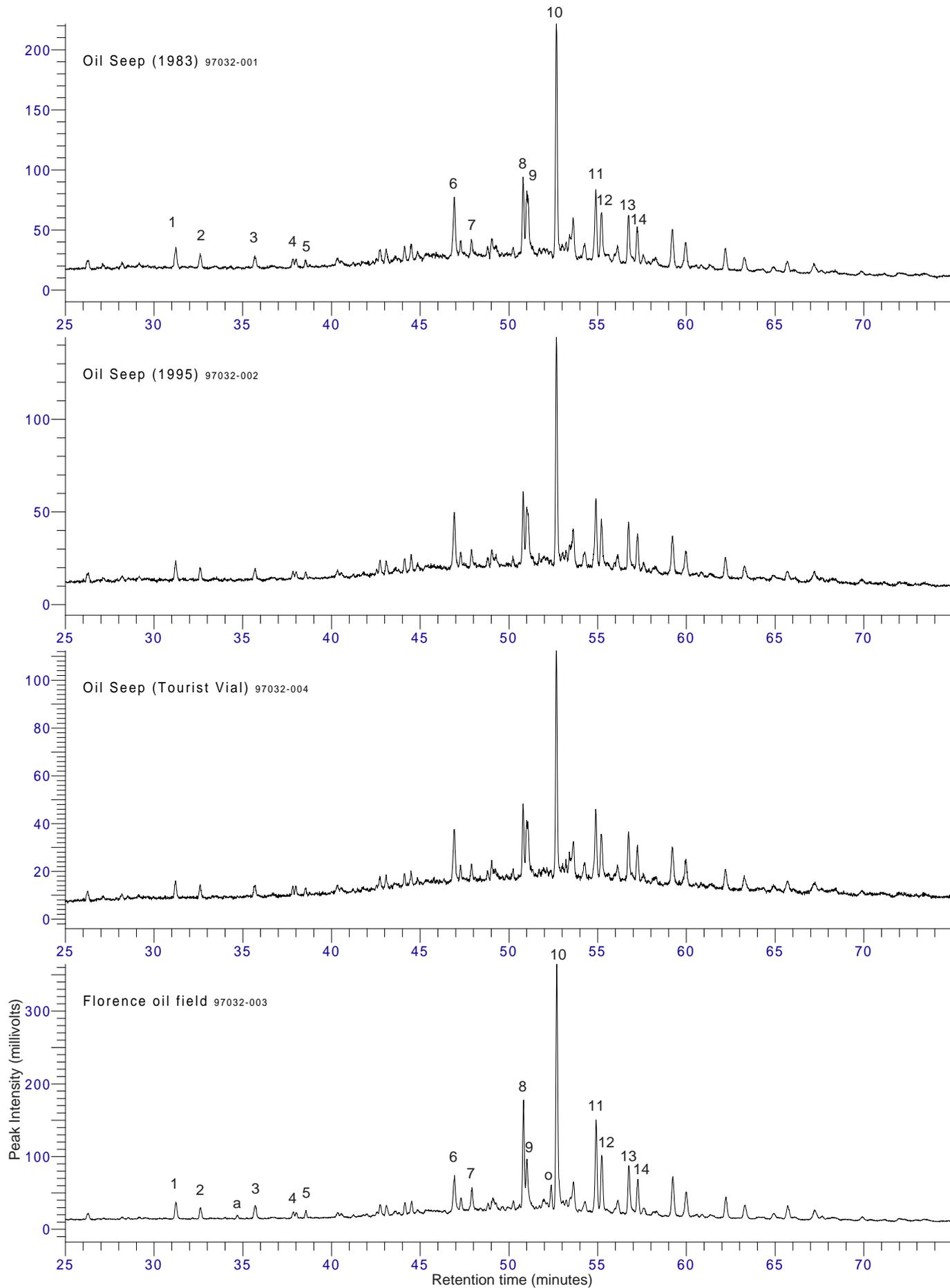


Figure 3. Mass chromatograms (m/z 191.1800) of terpanes. Peak numbers refer to Table 3.

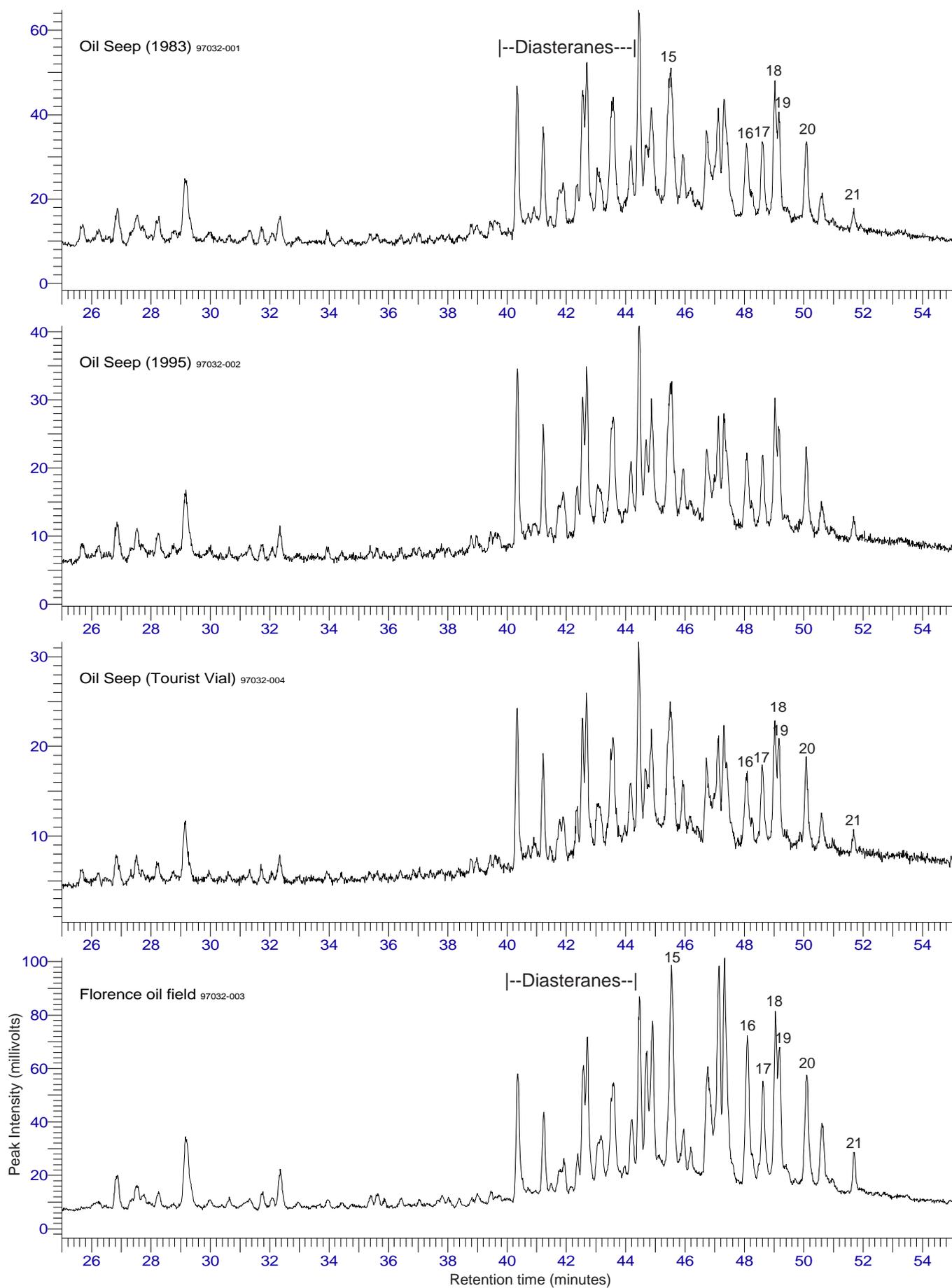


Figure 4. Mass chromatograms (m/z 217.1956) of steranes. Peak numbers refer to Table 3.

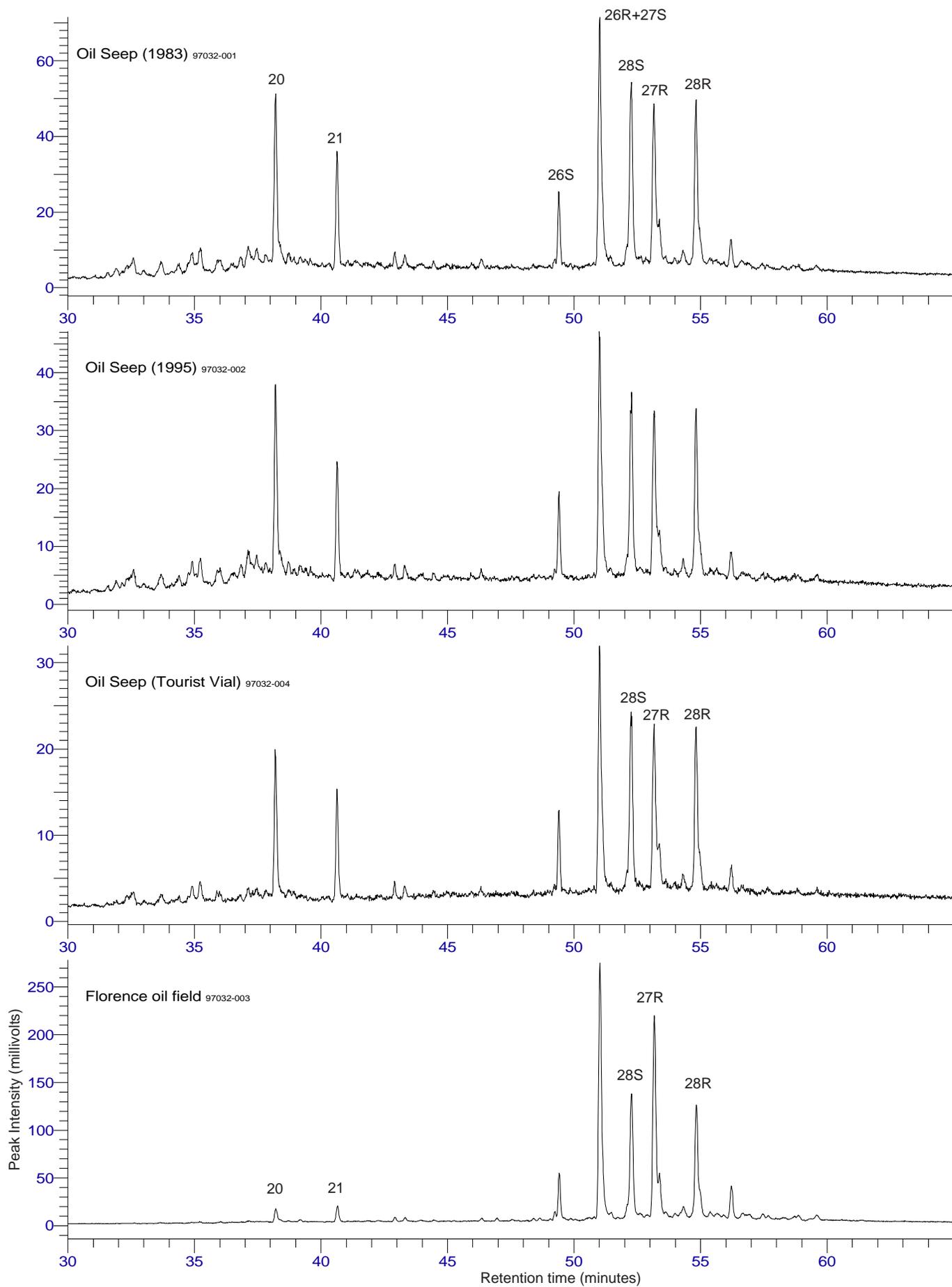


Figure 5. Mass chromatograms (m/z 231.1174) of triaromatic steranes. Peak numbers are carbon numbers.

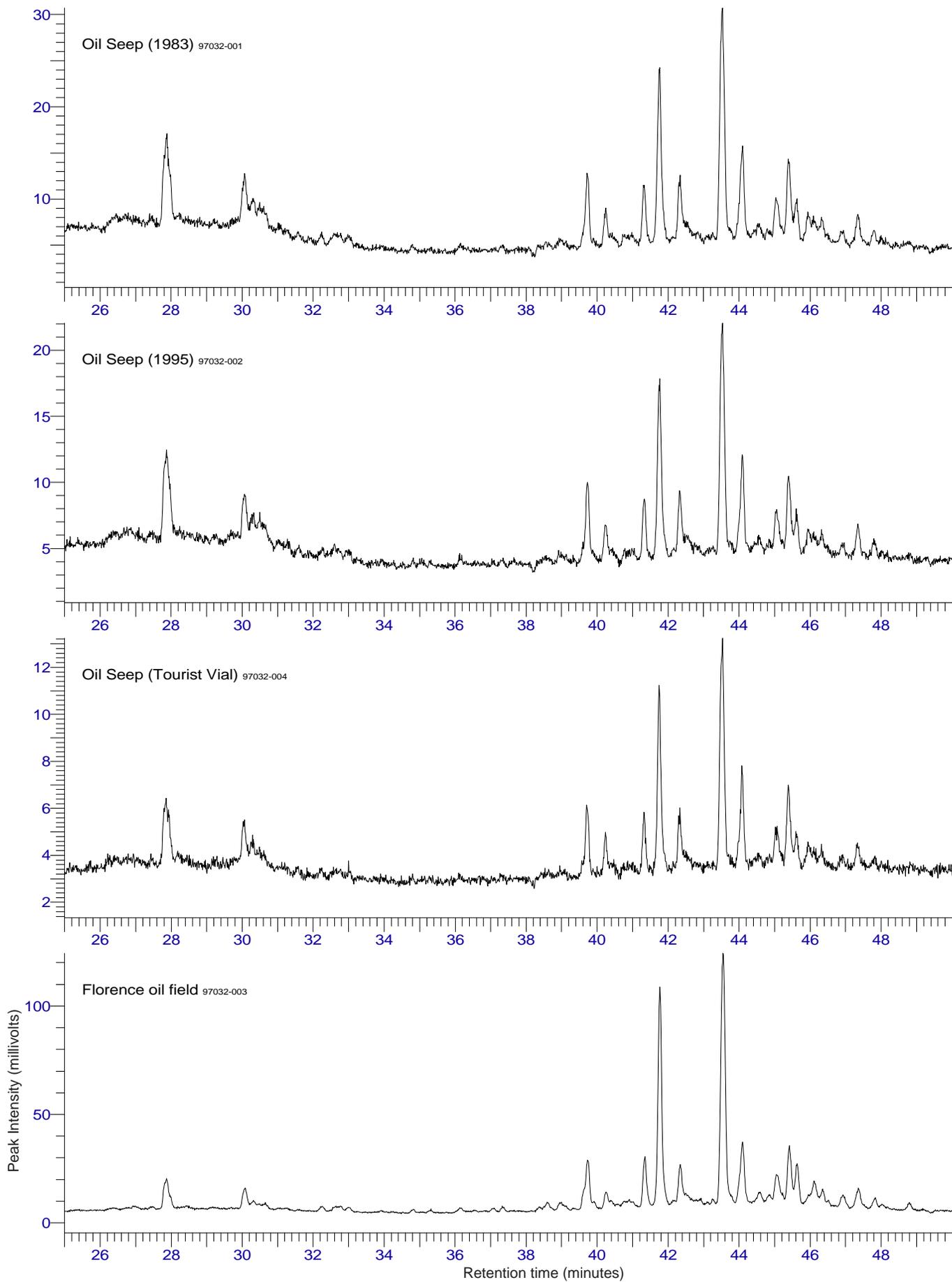


Figure 6. Mass chromatograms (m/z 253.1956) of monoaromatic steranes.