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# The Power of the IRD: Aliphatic Alcohol Identification

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## IRD Application Brief

### HP 5965B Infrared Detector

#### IRD90-2

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### IRD Productivity Profile

#### Industry

Foods and Flavors, Industrial  
Chemical, Petrochemical,  
Environmental

#### Chemicals

Dodecanol, Dodecene,  
Cyclododecane

#### Sample Matrix

Natural products, reaction  
mixtures, products, residues

#### Analysis

Structural Identification

### Introduction

While the mass spectrometer produces powerful structural information based on molecular fragmentation, often including molecular weight data, MS can be weak in the area of alcohol identification. Conversely, the infrared spectrometer is strong in this area.

This note highlights the power of the HP 5965B IRD to aid in the identification of aliphatic alcohols. While this example was chosen from the area of foods, flavors, and fragrances, knowledge of mass and infrared spectral behavior of alcohols has broad applicability in all areas where structural identification is important.

### Sample Background

Saturated primary alcohols occur widely in nature, especially in fruit, and are common in all facets of chemical, petrochemical, and phar-

maceutical industries as well. Their use in fragrances is limited since their odor is weak. Alcohols are starting materials for aldehydes and esters, which have even wider use in almost every area. Naturally occurring fatty alcohols are produced primarily by the reduction of the corresponding carboxylic acid methyl esters which come from transesterification of natural fats and oils with methanol.

### High Confidence Determination

In the further investigation of Russian coriander oil (see IRD Application Brief IRD89-4, HP 23-5954-8194) the GC peak at 32.14 minutes was examined in detail. Figure 1 is an expanded portion of the MSD's total ion chromatogram (TIC) and the IRD's total response chromatogram (TRC) along with the mass spectrum and the infrared spectrum of the peak in question. The

IRD's spectrum was obtained on about 20 nanograms of material while the MSD's was on about 4 nanograms as the combined system was configured in the series mode and about 20% of the IRD's flow cell effluent was admitted to the MSD.

The mass spectrum is not definitive, indicating cycloalkanes, olefins, alcohols, or even possibly alkyl substituted cyclopropanes. The infrared spectrum, on the other hand, is quite obviously that of a long chain saturated aliphatic alcohol, since there is no unsaturated C-H absorption above  $3000\text{ cm}^{-1}$ , the C-OH at  $3669\text{ cm}^{-1}$  is quite weak, and the C-O at  $1051$  is also weak.

To exploit the power of computerized library searching to help resolve this matter, the HP 5965B was used to perform a combined IR and MS library search. The results of this search are shown in Table 1. The 49000 entry NIST/NBS Mass Spectral Library and the 2000 entry Robertet Flavor and Fragrance Vapor Phase Infrared Library were used. The highly desired Class 1 hit was found. Both the IRD search and the PBM mass spec search found 1-dodecanol. Note that in the MS search, the Class 2 hits (in only one library) found the five matches which had a better index than the 1-dodecanol, are alkyl substituted cyclics and unsaturates. In comparing the

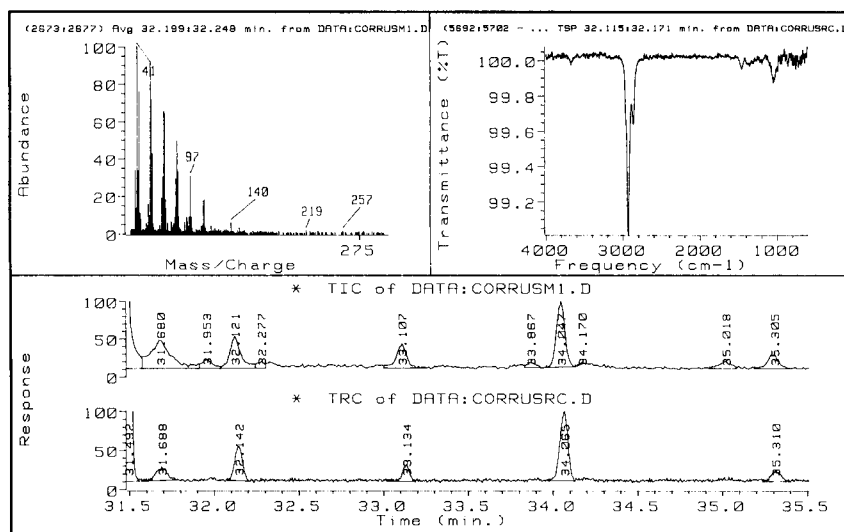


Figure 1. Russian Coriander Oil, expanded portion of chromatograms with mass and infrared spectra of peak at 32.14 minutes

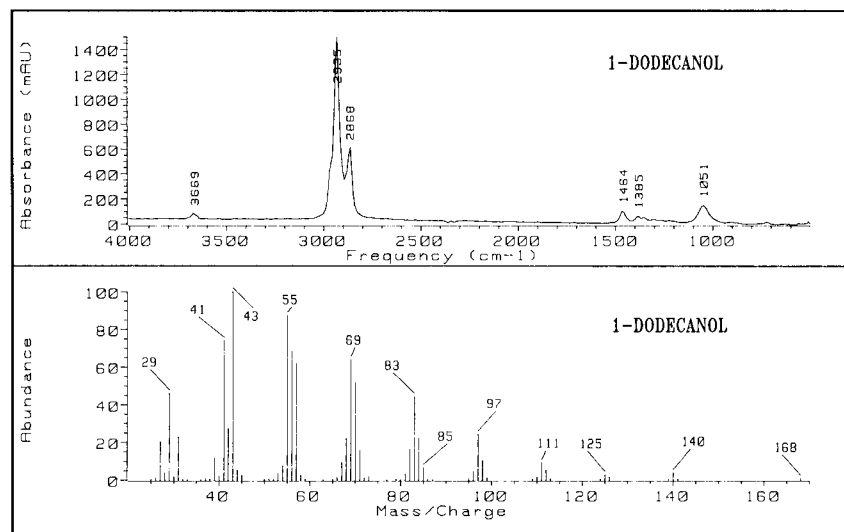


Figure 2. Infrared spectrum and mass spectrum of 1-dodecanol

Table 1. Results of combined infrared and mass spectral library searches of peak at 32.14 minutes

COMPARISON OF RESULTS FROM						
PBM Search of Library file: DATA:NBS49K.L						
ASP 32.199:32.248 min. from DATA:CORRUSM1.D						
AND						
IR Search of Library file: DATA:ROBERTET.L						
ASP 32.115:32.171 min. from DATA:CORRUSRC.D						
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Class 1 (on both lists)						
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CAS Number	PBM Qual	IR Qual	MWt	Formula	Name	
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1.	000112-53-8	86	947	186	C12H26O	1-Dodecanol
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Class 2 (in only one library)						
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CAS Number	PBM Qual	IR Qual	MWt	Formula	Name	
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2.	000294-62-2	97	---	168	C12H24	Cyclododecane
3.	000112-41-4	90	---	168	C12H24	1-Dodecene
4.	074663-85-7	90	---	168	C12H24	Cyclopropane, nonyl-
5.	001472-09-9	87	---	154	C11H22	Cyclopropane, octyl-
6.	000872-05-9	87	---	140	C10H20	1-Decene
7.	041446-67-7	86	---	196	C14H28	3-Tetadecene, (Z)-
8.	041446-66-6	86	---	196	C14H28	5-Tetadecene, (E)-
9.	021328-57-4	64	---	140	C10H20	Cyclooctane, 1,5-dimethyl-
10.	000112-72-1	58	---	214	C14H30O	1-Tetradecanol
11.	000292-74-8	58	---	112	C8H16	Cyclooctane
12.	003728-57-2	53	---	126	C9H18	Cyclopentane, 1-methyl-2-propyl-
13.	056554-78-0	53	---	272	C17H33Cl	7-Heptadecene, 1-chloro-
14.	000931-35-1	50	---	112	C6H12N2	1H-Imidazole, 2-ethyl-4,5-dihydr
15.	000930-89-2	49	---	112	C8H16	Cyclopentane, 1-ethyl-2-methyl-,
16.	061142-68-5	43	---	168	C12H24	Cyclopentane, 1-hexyl-3-methyl-
17.	019549-87-2	30	---	126	C9H18	2,4-DIMETHYL-1-HEPTENE
18.	074630-23-2	30	---	154	C11H22	2-Decene, 7-methyl-, (Z)-
19.	003664-75-3	30	---	140	C9H16O	Ethanone, 1-(2,2-dimethylcyclope
20.	001192-14-9	30	---	98	C6H10O	Cyclobutanone, 2,2-dimethyl-
21.		---	927	340	C22H44O2	1,2-DIMETHYLPROPYL HEPTADECANOAT
22.		---	927	204	C15H24	BETA-PATCHOULENE
23.	001192-14-5	---	927	312	C20H40O2	ETHYL OCTADECANOATE

Table 1 Continued

----- Class 3 (in both libraries, but on only one list) -----						
CAS Number	PBM Qual	IR Qual	MWT	Formula	Name	
24. 000112-30-1	---	950	158	C10H22O	DECANOL	
25. 000112-42-5	---	949	172	C11H24O	UNDECANOL	
26. 000628-97-7	---	929	284	C18H36O2	ETHYL HEXADECANOATE	
27. 041114-00-5	---	928	270	C17H34O2	ETHYL PENTADECANOATE	
28. 014010-23-2	---	928	298	C19H38O2	ETHYL HEPTADECANOATE	
29. 000124-06-1	---	927	256	C16H32O2	ETHYL TETRADECANOATE	

is not the top quality hit but when the two very different kinds of spectral information are combined and analyzed, the assignment of 1-dodecanol is of very high confidence.

### Conclusion

The combined IRD/MSD system has been shown to be a powerful analytical tool in the identification of aliphatic alcohols with combined library searching using the NIST/NBS Mass Spectral Library and the Robertet Flavor and Fragrance Infrared Library. The combined instrument provides a higher confidence result than either the IR or MS technique alone.

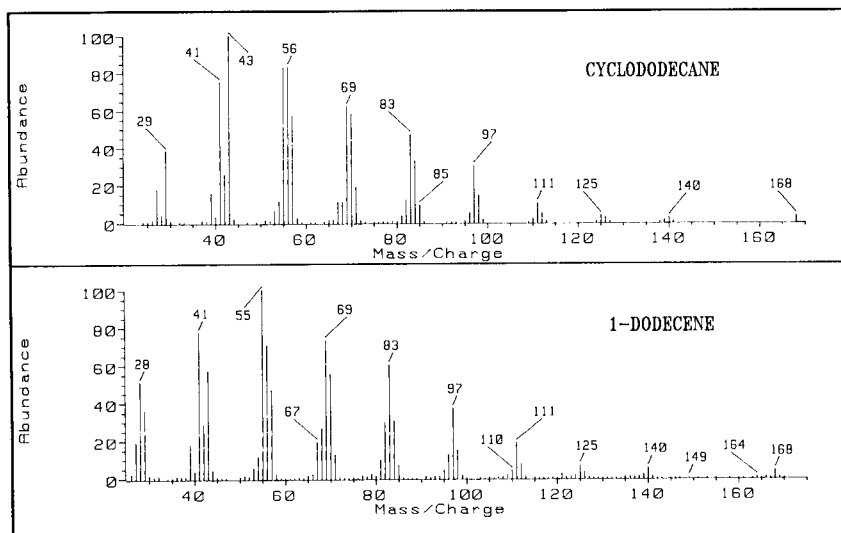


Figure 3. Mass spectra of cyclododecane and 1-dodecene

mass spectra of 1-dodecanol, cyclododecane, and 1-dodecene (see Figures 2 and 3) it is easy to see why there is difficulty in differentiating these compounds. The 1-dodecanol, the number sixth ranked hit with a match quality of 72, is the only alcohol that appears in

the top ten. On the other hand, the top three IR hits, in Class 1 and Class 3, are all alcohols. Due to the nature of their IR spectra it is not surprising that their match qualities are all very similar. In both the case of the IR and MS searches the Class 1 match

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## **Conditions**

### **Gas chromatograph**

*Column:* 50m x 0.32 mm id  
HP-1 (methyl silicone), 0.52  
micrometer film

*Carrier gas:* Helium @ 25 psi;  
2.0 mL/min

*Oven:* 40°C (2 min) to 120°C at  
3°C/min, 120°C to 200°C at  
5°C/min, hold 6.33 min

### **IRD parameters**

*Light pipe:* 250°C

*Transfer lines:* 260°C

*Optical resolution:* 8 cm<sup>-1</sup>, wide  
band MCT detector

*Scan rate:* 3 scans/sec

### **MSD parameters**

*Mass range:* 33-300 daltons

*Scan rate:* 1.6 scans/sec  
(4 samples)