Infrared Detector

USE OF COMBINED GC/IRD/MSD IN THE CHARACTERIZATION OF SYNTHETIC BUTTER FLAVOR

Introduction

There are several types of synthetic butter flavors used today. Some contain mostly diacetyl with vanillian, various butyrates, and butyric acid. There are other types of synthetic butter flavors also. One of those was analyzed here. It surprisingly contains non of the expected smaller flavorants but higher molecular weight lactones.

Equipment

For the combined GC/IRD/MSD system, the gas chromatograph, an HP 5890 Series II was set up using a 30 meter DB-5 column. The column effluent was split at the end of the column at a 10 to 1 ratio with the bulk of the flow going to the HP 5965B IRD and the lesser amount going to the HP 5971A MSD. The details of this parallel configuration have been previously described.

Control of the GC, IRD and MSD as well as data reduction was done with G1094A software on the single PC based G1030A MS/IR ChemStation (DOS).

Results

With the combined GC/IRD/MSD high confidence qualitative analysis is possible. Using this sample of synthetic buter flavor several features of the system have been utilized, viz., library searching of infrared and mass spectra with combined reporting and selected wavenumber (functional group) chromatography.

Figure 1 is a portion of the IRD total response chromatogram (TRC). The peak at 12.18 minutes turns out to be delta dodecalactone, see Figure 2. The library search results for this peak are in Figure 3. IRD library searching allows various libraries to be chained and over 11,000 spectra were searched. Note in Figure 2 that names and structures can be annotated.

The combined IR and MS display is in Figure 4. Here the total response chromatogram and the total ion chromatogram have been software aligned for better presentation. The IR and Mass Spectra for the peak at about 12.18 minutes are also displayed.

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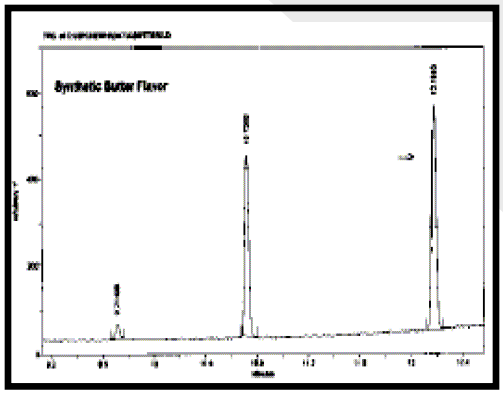

Figure 1. IRD total response chromatogram of synthetic butter flavor

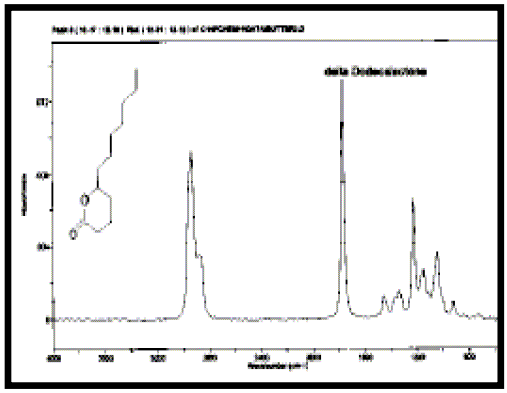

Figure 2. Infrared spectrum of peak at 12.18 minutes

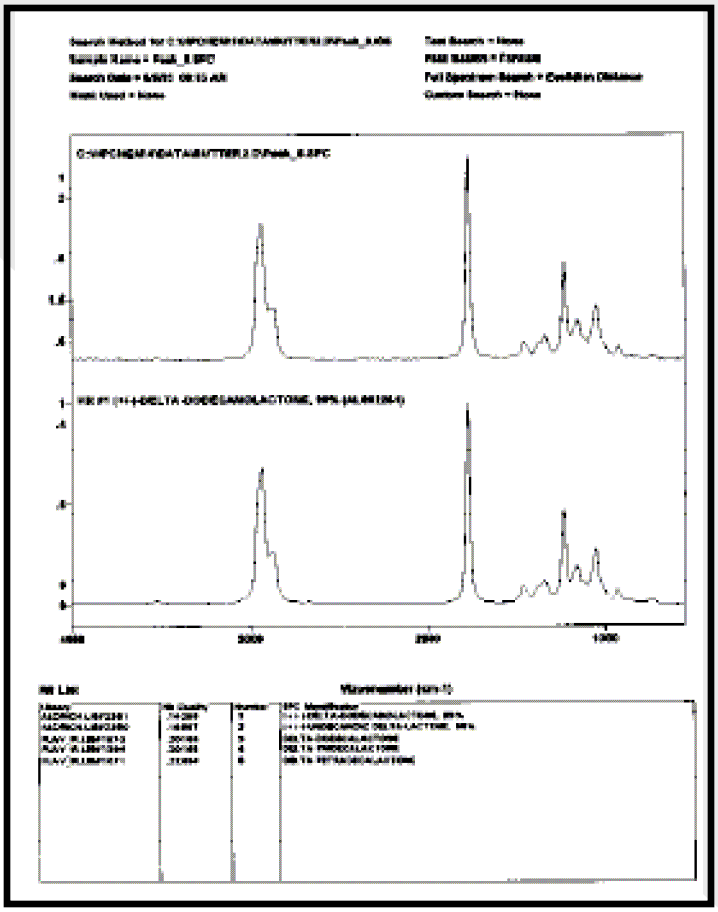

Figure 3. Results of IRD library search for peak at 12.18 minutes

Figure 5 shows the combined library search results. The same IR libraries were used as in the single search above but additionally the Wiley Library of over 130,000 entries and the NIST Library of over 75,000 entries were used for the mass spectral searches. For the peak at 10.7 minutes note that even though the names are different, the CAS Registry numbers are the same for delta decalactone. When UR and MS library searches both list the same compound, as in this case, the confidence level of identification is very high.

In Figure 6 two selected wavenumber chromatograms (SWCs) for y lactones at 1805cm-1 and delta lactones at 1770cm-1 have been calculated. These SWCs allow the TRC to be quickly screened for functional groups.

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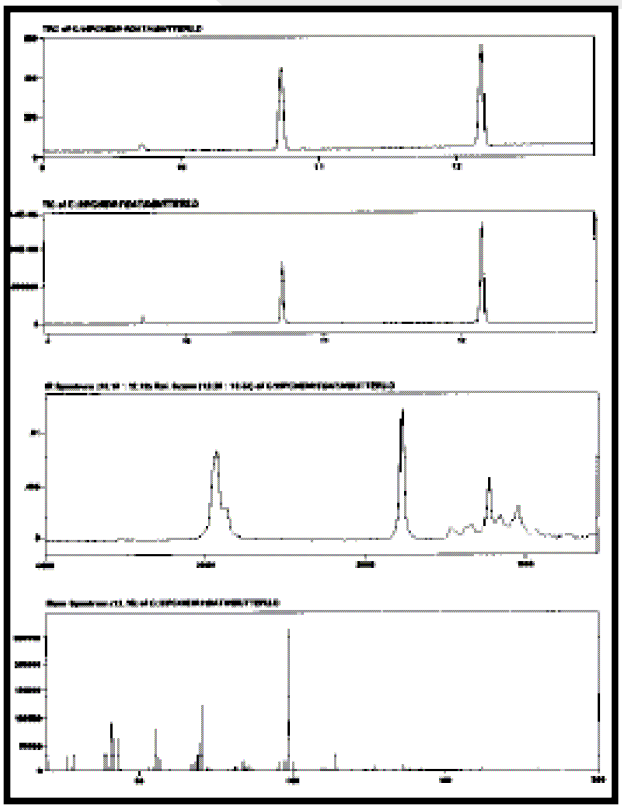

Figure 4. Combined IRD and MSD chromatograms and spectra for peak at 12.18 min

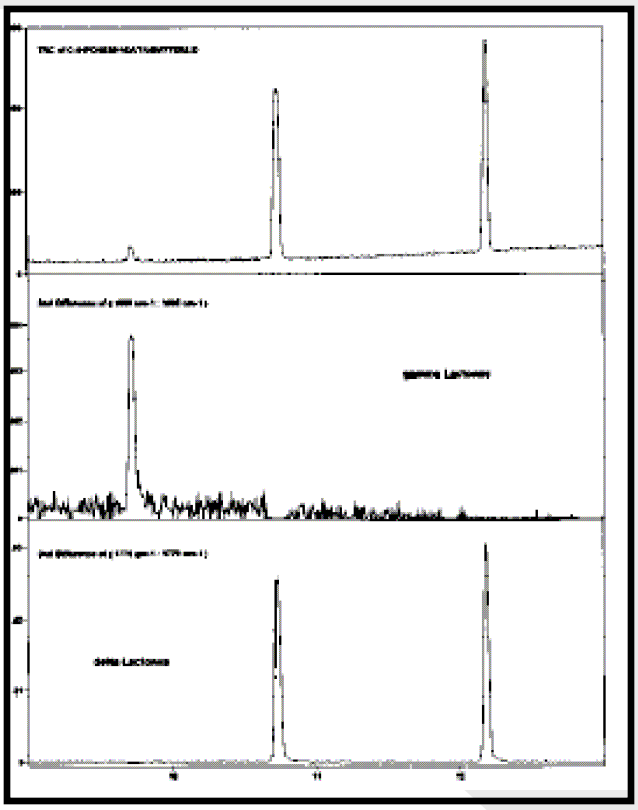

Figure 6. IRD Total Response Chromatogram and Selected Wavenumber Chromatograms for Lactones

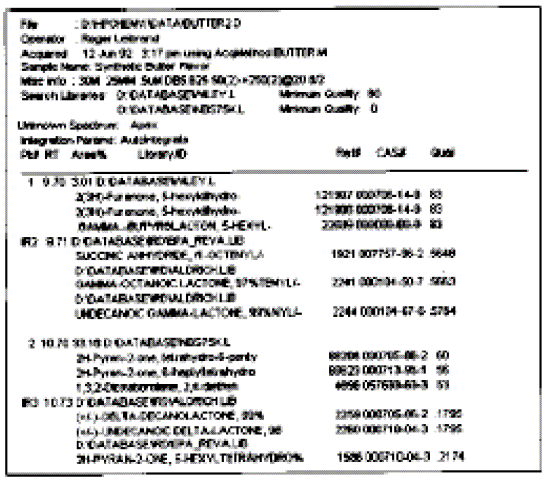

Figure 5. Combined IRD and MSD library search results for peak at 12.18 minutes

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Conclusion

As seen with this analysis of a synthetic butter flavor, the combined GC/IRD/MSD provides a powerful tool for the characterization of flavor components using library searching and selected wavenumber chromatography

Conditions

Gas Chromatograph
Column: 30m x 0.25mm x .5um DB-5
Carrier Gas: Helium at 180kPa, 1mL/min
Oven: 50°C (2 min) at 250°C at 20°C/min with 2 min hold
Injection Port: 250°C
Sample Injection: 1ul, split 20 to 1

IRD Parameters
Light Pipe: 260°C
Transfer Lines: 280°C
Sweep Gas: Nitrogen 110 kPa inlet, 100 kPa outlet
Scan Parameters: 8cm-1 resolution, 2 coads, 3 scans/second
Detector: Wide Band MCT, 4000-550cm-1

MSD Parameters
Mass Range: 10 to 310 daltons
Scan Parameters: 2 a/d samples, 1.4 scans/second stored

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