The McGill IR Group was established in 1990 within the Department of Food Science and Agricultural Chemistry of McGill University to investigate potential applications of FTIR spectroscopy in food analysis. Our research program has been directed toward meeting the demand within the food industry for rapid and environmentally friendly analytical methods for quality control and process monitoring. In recent years, we have also been involved in the development of FTIR methods that can assist the food industry in complying with food labeling regulations. Two examples of applications falling within this category—the determination of the trans content of edible fats and oils and the analysis of alcoholic beverages—are described in this article. Both of these analyses employ SB-ATR sample-handling accessories and serve to illustrate the utility of the SB-ATR technique in the FTIR analysis of beverages and liquid or semi-liquid food products.

**Determination of trans Content of Edible Fats and Oils**

Naturally occurring double bonds in fats and oils of plant origin generally have the cis configuration. However, extensive cis \( \rightarrow \) trans isomerization may occur during industrial processing, and the levels of trans isomers in processed fats and oils can be as high as 40%. In recent years, trans fat in the diet has increasingly become of nutritional concern, particularly as a possible risk factor in cardiovascular disease [1]. In response to this concern, the U.S. FDA submitted a proposal in late 1999 to include trans fat on food nutrition labels (Figure 1). Although this proposal has not yet been adopted, it has stimulated interest in the development of accurate and rapid analytical methods for the determination of the trans content of edible fats and oils.

Infrared spectroscopy has been widely employed for more than 50 years in the fats and oils industry...
as a routine method for the determination of the *trans* content of processed fats and oils.

Traditional IR *trans* analysis is based on measurement of the intensity of the characteristic absorption of isolated (i.e., nonconjugated) *trans* bonds at 967 cm⁻¹ (Figure 2), assigned to a C—H out-of-plane deformation mode. However, the accuracy of this measurement is limited by the proximity of this band to intense oil absorption bands above 1000 cm⁻¹ that distort the spectral baseline. Furthermore, the measurement of this band is subject to interference from underlying weak absorptions of triacylglycerols, which are the predominant species making up all fats and oils. This interference is eliminated by converting the triacylglycerols to fatty acid methyl esters. In addition to this sample preparation step, the traditional IR method requires the quantitative dissolution of the sample in CS₂, a noxious and volatile solvent, to allow it to be injected into a fixed-pathlength transmission cell.

Recently, researchers at the U.S. FDA developed a new FTIR method to address the need for a rapid and convenient means of measuring the *trans* content of fats, oils, and fat-containing foods [2]. Following an international collaborative study [3], in which we participated, this method was adopted as an official method by the American Oil Chemists’ Society (AOCS) in 1999 and by the Association of Official Analytical Chemists (AOAC) in 2000. The cornerstones of the method are (i) the use of the SB-ATR sample-handling technique to allow samples to be analyzed in their neat form (i.e., without the use of a solvent) and (ii) removal of spectral interferences by ratioing the spectrum of the sample against that of an appropriate *trans*-free reference oil having a fatty acid composition similar to that of the sample [4]. The determination of *trans* content is based on the area of the 967 cm⁻¹ peak, which can be measured accurately owing to the straightening of the baseline and the elimination of underlying triacylglycerol absorptions by the ratioing procedure. The lower limit of quantitation established in the collaborative study was 5% *trans* [3]. For the analysis of fats, the SB-ATR crystal must be heated to 65 ± 2°C to ensure that samples are fully melted, and thus the excellent temperature control that can be achieved owing to the small surface area of the crystal is a key advantage of this sample-handling method.
technique. Furthermore, less than 50 µL of sample is required, making the method suitable for the analysis of small amounts of fat extracted from low-fat foods. The method is simple and convenient, requiring no sample preparation, and the total analysis time is on the order of 2-3 min/sample. Thus, it may be anticipated that it will find extensive use in the food industry if and when the proposed amendments to nutrition labeling requirements with respect to trans fat are adopted. We have also demonstrated the extension of this FTIR/SB-ATR analytical methodology to the determination of quality parameters of fats and oils [5], and this in turn should provide an impetus for the wider application of FTIR/SB-ATR analysis in the fats and oils sector.

Determination of Alcohol Content of Distilled Liquors and Wines

For taxation purposes, alcohol content must be declared on the labels of all alcoholic beverages. As part of a joint research effort with Thermal-Lube Inc. (Pointe-Claire, Quebec, Canada) on the development of FTIR analytical methodology for the wine industry, we have investigated the determination of the alcohol content of wines as well as of distilled liquors by FTIR/SB-ATR spectroscopy. Commercially available FTIR wine analyzers are equipped with a narrow-pathlength transmission cell, and thus samples having high alcohol and/or sugar content must be diluted prior to analysis owing to their intense infrared absorption. The difficulty of injecting highly viscous samples, such as liqueurs, into a narrow-pathlength transmission cell also precludes analysis of these samples without prior dilution. Clearly, this need for quantitative dilution of certain samples is not only inconvenient but also may have a negative impact on analytical accuracy. Since SB-ATR analysis would be universally applicable to all types of alcoholic beverages, without any need for dilution, one of the objectives of our research was to ascertain whether this alternative would provide satisfactory analytical accuracy.

For the analysis of distilled liquors, a set of 22 calibration standards ranging from 0.1 to 75% (v/v) alcohol was prepared by mixing ethanol and water. The FTIR/SB-ATR spectra of three of these standards are presented in Figure 3. The calibration standards were analyzed by densitometry to obtain values for their alcohol content at the reference temperature of 15.56°C, and a calibration model for the prediction of alcohol content was obtained by partial-least-squares (PLS) regression. The calibration model was validated by comparing the alcohol contents predicted for several brands of distilled liquors to values determined by densitometry in accordance with AOAC Official Method 982.10 [6]. Typical results are tabulated in Table 1, the overall standard error of prediction (SEP) for eight different types of distilled liquors being 0.14% (v/v).

We subsequently developed PLS calibration models for the determination of various components in wines by using either wine samples preanalyzed by HPLC or “synthetic” wines, comprising ethanol/water mixtures containing fructose, glucose, glycerol, and organic acids at the levels present in

![Figure 3. FTIR/SB-ATR spectra of ethanol/water mixtures employed as standards in the development of a calibration for the prediction of the alcohol content of distilled liquors. Top: 5% ethanol; middle: 20% ethanol; bottom: 40% ethanol.](image)
dry wines. Figure 4 shows a plot of FTIR-predicted alcohol content for >100 wine samples versus the reference values provided by the Société des alcools du Québec (SAQ), the agency that regulates the sale of alcohol in the province of Quebec. The PLS calibration models derived using either real or “synthetic” wines yielded an SEP of 0.14% (v/v) for this set of >100 validation samples.

Figure 4. Plot of FTIR-predicted alcohol content of 108 wine samples versus values obtained by the reference method.

Thus, the FTIR/SB-ATR method was shown to allow for the determination of the alcohol content of both distilled liquors and wines with a high degree of accuracy. Furthermore, with the use of the Pike flow-through SB-ATR accessory shown in Figure 5, coupled to an autosampler, automated analysis of 56 samples/hr was achieved, making this method suitable for routine application.

References


Figure 5. Pike flow-through SB-ATR accessory employed for wine analysis.
Table 1
Validation of PLS Calibration Model for the Prediction of Alcohol Content in Distilled Liquors by FTIR/SB-ATR Spectroscopy

<table>
<thead>
<tr>
<th>Brand</th>
<th>Alcohol content (v/v)</th>
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<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>AOAC 982.10</td>
<td>FTIR/SB-ATR</td>
<td></td>
</tr>
<tr>
<td>Bacardi</td>
<td>40.96%</td>
<td>40.91%</td>
<td></td>
</tr>
<tr>
<td>Smirnoff</td>
<td>40.65%</td>
<td>40.54%</td>
<td></td>
</tr>
<tr>
<td>Finlandia</td>
<td>40.22%</td>
<td>40.27%</td>
<td></td>
</tr>
<tr>
<td>Canadian Club</td>
<td>40.84%</td>
<td>40.75%</td>
<td></td>
</tr>
<tr>
<td>Chivas Regal</td>
<td>40.22%</td>
<td>40.28%</td>
<td></td>
</tr>
</tbody>
</table>

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