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Application News

Fourier Transform Infrared Spectrophotometer

Identification of Caffeine in instant coffee with Fourier Transform Infrared Spectrophotometer FTIR and ATR accessory

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User Benefits

- Fast screening with IRSpirit
- Easy cleaning
- Fast analysis

■ Introduction

Infrared spectroscopy is a high effective analysis technique for the quality control of natural products. One of the almost famous products in our life might be coffee. Prepared fresh brewed from beans or from instant coffee.

Here is the approach to compare the dry-freeze instant coffee style with caffeine and one decaffeinated.

The caffeine should be in small percentage range inside of the standard instant coffee and almost zero in the decaffeinated coffee.

Fig. 1 Coffee beans, and instant coffee

Instant coffee is the result of water extraction from roasted beans. The extraction will be reduced from water by the freezing and evaporation of water. This dry-residual- the extract- is presented as powder particles commercially. Such particles/powder can be easily analyzed with ATR-spectroscopy. Infrared spectroscopy can detect the vibrations from such a natural product and the diversity it consists. The idea is to see the difference between the standard and the decaffeinated instant powder, to identify caffeine.

The regular analysis will be done with HPLC or headspace GC analysis for more details about ingredients of the coffee. Some important ingredients are chlorogenic acid, methylcafestol, free- and total carbonates and dichloromethane in decaffeinated coffee for example.

■ Measurement Conditions and Samples

Accessory

The ATR reflection technique is an easy way of sample preparation and cleaning. In this case the full diamond QATR-S was placed into the sample compartment of IRSpirit-T. IRSpirit-T is a basic laboratory infrared spectrophotometer. QATR-S is a full diamond ATR with the advantage that the beam will penetrate the sample surface by 2 µm.

Sample

For this application two instant coffee samples from same brand were compared.

Fig. 2: classical instant coffee available with and without caffeine filled from the commercial glass into the two glass bottles at left and in back at right the view to the IRSpirit-T with diamond-based QATR-S accessory.

The instant coffee was handled with care to avoid contamination by environmental water vapor. and the measurement were done immediately because the instant coffee seems to be hygroscopic. Even the fast handling could not avoid the immediately interaction of water vapor with the high surface of the instant coffee particles. This is shown in the infrared spectra from the instant coffee. The baseline of the spectra is dominated by aspects of diamond in the region of 2000 cm^{-1}, water vapor in region of 3300 cm^{-1} and 1800 to 1600 cm^{-1}. For the identification of caffeine the fingerprint region of the spectrum was used (fig. 4).

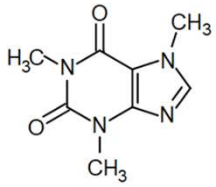

Fig. 3 chemical structure of caffeine, the molecular groups are of interest for infrared spectroscopy, ketone, different nitrogen molecules, methyl and double ring system

Infrared spectrum interpretation

The spectra of the instant coffee show high portion of cellulose. As expect it is little difference in both samples. In figure 3 these ATR infrared spectra are shown. The variation of the spectra is in the region of the water signals, these are signal ratios and additional signals. Next step to visualize the caffeine is the subtraction of decaffeinated from full caffeine instant coffee (see fig. 4).

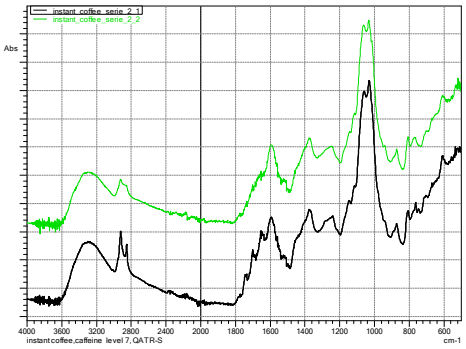

Fig. 3: ATR measurement of instant coffee with IRSpirit with QATR-S as accessory. The black line is infrared spectrum of the standard and the green line is the decaffeinated instant coffee.

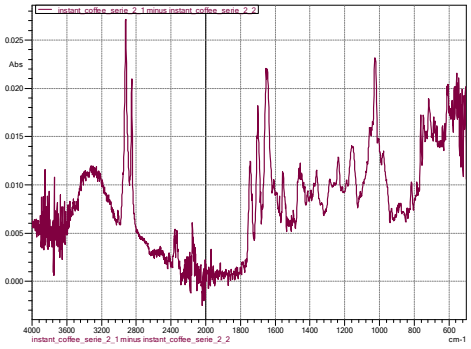

Fig. 4: Difference spectrum from the two sorts of instant coffee from same brand, the graph y-axis is in 0.025mAbs scale

The difference is dominated by cellulose substructures and fat. So, the subtraction goes on using a natural oil. (fig. 5) This spectrum was used for a library search. (Fig. 6)

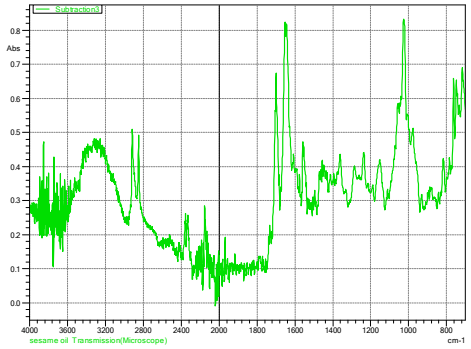

Fig. 5: Difference spectrum minus a sesame oil, as place holder for a natural oil.

Infrared spectrum identification

The absorbance values of this difference spectrum are under 0.025 mAbs for the caffeine in that coffee. For the library search such spectrum is normalized. The search found from three different libraries the match for the caffeine in leading position of the hitlist.

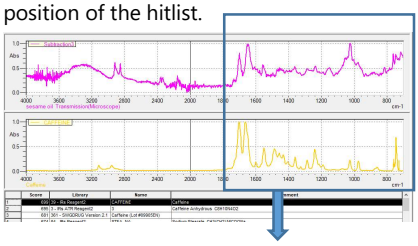

Fig. 6: Library search result for the difference spectrum as result from subtraction procedure from instant coffee, match from three different libraries is caffeine.: Wavenumber list and spectral interpretation for caffeine in an ATR infrared spectrum, see table 1

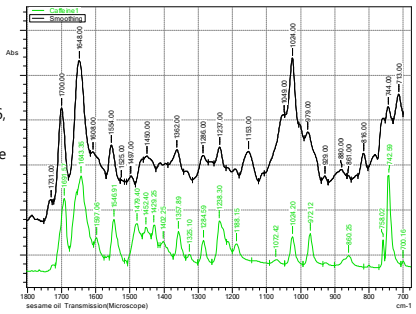

Fig. 7: Zoom from Fig.6, blue frame, Peak pick of the fingerprint range for pure caffeine (green) and for difference spectrum (black), peak interpretation is in table 1.

Caffeine in instant coffee Wavenumber[cm^{-1}]	Pure caffeine Wavenumber [cm^{-1}]	Molecular groups of caffeine
744.00	742.59	-C-H oop ring
861.00	860.25	-C-H bending
979.00	972.12	C=C stretching
1024.00	1024.20	Stretching C-O, C-N
1237.00	1238.30	ν(C-N)
1362.00	1357.89	ν(C-N)
1450.00	1452.40	-CH3, C-H bending
1554.00	1546.91	ν(C-N) stretch
1648.00	1643.35	-C=O, C=N
1700.00	1691.57	ν(C=O, C=N)

Table 1: Wavenumber list and spectral interpretation for caffeine in an ATR infrared spectrum

■ Discussion

It is possible to establish a method for the analysis of caffeine in instant coffee with single reflection FTIR-ATR technique. The precision of the instrument is high so that signals in milli absorbance units can be shown after diverse mathematical treatments of the infrared spectrum of a complex natural mix as it is present in natural good like food is. Such subtraction as it was used here is possible because the superposition – the addition of all infrared spectra which are component in the food- is valid. The peak table result supports the identification. Once found such minor components in a mix, a quantitative determination should be as well possible.

■ Instrumentation

Instruments	: IRSpirit™-T (KBr window plate) Fourier transform infrared spectrophotometer QATR-S single reflection diamond-based ATR Libraries from Shimadzu, and SWGDrug, free library
Resolution	: 4 cm^{-1}
Accumulation	: 40 times
Apodization function	: Happ-Genzel
Detector	: DLATGS

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