Evaluation of the *"STUFF of Life"* (Foods, Drinks, ?, etc) by MID Infra-Red SPECTROMETRY using Direct Sampling ATR Techniques

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CHEMICAL CONCEPTS:

KEYWORDS =
Analytical Chemistry, Instrumental Analysis, Spectroscopy, Infra-Red Vibrations

The subject of Analytical Chemistry can be broken down into TWO Primary Classes: Classical ("wet") and Instrumental. While the study of the classical Titrimetric and Gravimetric analyses, along with other properties like density, pH, conductivity, ad infinitum; provides a good foundation for understanding how to "*measure*" things; it is the sophistication of Instrumental Analysis that opens up the world of "*measurement*" to Student & Teacher alike.

Each class also has a primary sub-division: Qualitative ("what" do I have?) and Quantitative ("how much" of it is there?). Again, the "classical" Qual-scheme for *inorganic* analysis is moderately useful for some applications, but QUAL for *organic* analysis requires some very fancy (and expensive; most often complex) glassware and equipment. In this vein, it is Instrumentation that has opened up and actually SIMPLIFIED the world of Qualitative determinations due to its very complexity.

Focussing on the OVERALL scheme of Instrumental Analysis, we can break down our classifications further into Analytical SPECTROSCOPY and Analytical CHROMATOGRAPHY. Literally, "*SPECTRO-SCOPY*" is the "measurement of a light spectrum" of a material based on energy range from the Electro-Magnetic Continuum"; while "*CHROMA-TO-GRAPHY*" is the "drawing of separate colors". Let's start with the techniques of SPECTROSCOPY. To further define the various wonders available to the Analytical Chemist; we need to look at the SOURCE of all this data that we generate in the evaluation of Atoms and Molecules:

There are many WAVELENGTHS of Energy in the Electromagnetic Spectrum; from the ultra-short region of X-Rays to the very long wavelengths of Microwaves. In between is a spectral region of Light called the UV-VIS (Ultra-Violet / Visible) Spectrum. These wavelengths of energy; from 190nm. beginning in the UV through 900nm. at the end of the VIS; can generate a tremendous amount of descriptive information about Atoms & Molecules. The longer wavelengths of the Infra-Red (IR) Spectrum can generate extremely selective information about almost any organic compound.

["Continuum" of the Electromagnetic Spectrum]

Gamma Radiation	X-Rays	UV	VIS	IR	Micro-Wave	Radio Waves	Sonic
< 0.01nm	*0.01-1.0nm*	*185-350nm*	*350-1000nm*	*5000 100 cm-1*	*100µm-1000µm*	*10mm-100M*	*250-1000M*

Short Wavelengths / High Frequency *[decreasing ENERGY -=>] [<=- increasing ENERGY]* Long Wavelengths / Low Frequency

Equivalents:1µm (micron) = 1000nm = 10,000cm-1 = 0.001mm = 0.000001M

DESIGN of SPECTROSCOPIC INSTRUMENTS:

By definition, ALL Spectrometers MUST have certain parts to properly be called Spectrometers, and these basic parts are:

SOURCE of Light, SAMPLE Chamber, OPTICAL System, DETECTOR and OUTPUT

Molecular Absorption (Vibrational) Spectrometers =
For a SOURCE, use a Hot Wire (Heat), build a Scanning MONO-CHROMATOR (so you can look at “one light” at a time) and use a Thermocouple Detector (which is just a real fancy Thermometer, which "measures" the heat of the IR Spectrum); and you have just designed a Scanning MID Infra-Red (IR) Spectrometer. The SAMPLE Chamber can use any “transparent” Cell or Holder to allow the SAMPLE to be scanned. This has always been a key technology for Forensics and Research, since you can "see" all kinds of materials for QUALITATIVE determination of Unknowns. It is this Mid-IR region from 4000-600cm-1 that generates the most specific data on organic molecules that is used to do Qualitative ID (Identification) and Quantitative CONCENTRATION analyses of many materials.

HOW DEMONSTRATION ADDRESSES THE CONCEPTS:

Depending on the type of Analytical Instrument being employed, we can measure the change in energy created by the ABSORPTION of specific Wavelengths of the spectrum by a Sample. The principles of ABSORPTION Spectroscopy can be applied to the components that make up Molecules. These changes in ENERGY occur because the electron shells (orbitals) of the Atoms that are parts of the Molecule can "react" with energies at certain wavelengths of energy that "match" or resonate with the energy of the electrons in those orbitals, or because the functional groups (characteristic parts: hydroxyl, carbonyl, amine, amide, etc.) of a Molecule will also "react" with some wavelengths of energy and "vibrate" from the absorption of this extra energy. We see an ABS signal because some of this optical light energy is translated in to mechanical kinetic energy.

For Organic MOLECULES, as certain parts of their structure (functional groups), which have a characteristic tendency to "vibrate" when exposed to specific wavelengths of light (typically in this case the Infra-Red region); thereby generating an Absorbance Spectrum of the molecule. These Spectral PEAKS are absolute characteristics of these species, and these unique Absorbance Peaks represent the "Fingerprint" of that compound and are used for QUALITATIVE identification.

By generating Spectral SCANS for various FOODS in the MID Infra-Red Spectrum, we can see the relative levels (“concentrations”) of the BASIC Nutritional Groups… with FATS having a general ABS Peak

There are approximately 2 billion ORGANIC Compounds known so far, and almost ALL of them have Functional Groups that absorb IR radiation to give a peak. Regarding FOODS; Proteins (based on their Poly-Amide structure) absorb around ~1550cm-1, FATS (based on the Tri-Glyceride Carbonyl group) absorb around ~1730cm-1, SUGARS & Carbohydrates (from their general “Sugar Backbone” of conjugated Cyclic Hydroxyl groups) in general absorb ~1100cm-1. You can make COMPARISONS between different FOODS that are SCANNED on the IR based on the relative “Peak Height” of these known “fingerprints”.

PREPARING AND PERFORMING THE DEMONSTRATION:

Safety =

- Do NOT lick any FOOD Samples off the Sampling Cells or Accessories… since they may be made from TOXIC materials!
- Be careful using any Solvents (that may be Flammable) to clean the various Sample Cells or Accessories.
- DO NOT DROP the yellow Zinc Selenide Prism Cell ATR Crystal… it WILL break!

Equipment and Materials =

- Model M-500 Fast-Scan IR Spectrometer (from BUCK Scientific) or equivalent
- Computer loaded w/ EZ-Scan or GRAMS Spectroscopy Software or equivalent
- Prism Cell Horizontal ATR Accessory with inert ZnSe Crystal or equivalent

Supplies =

- Bottle of Mixed Methanol / Hexane / Acetone "Cleaning Solvent" (for the ATR crystal)
- Disposable plastic pipets / eyedroppers (to apply Samples to ATR crystal)
- Soft paper towels/ napkins for cleaning off ATR crystal

Sample Types =

- Fat-Free and REAL American Cheese or Mayonnaise / slap in on the Prism Cell Crystal
- Margarine, Butter and Fake-Fat “Spread” / smear it on the Prism Cell Crystal
- Ham, Salami, Turkey, Olive Loaf, Head Cheese / press it on the Prism Cell Crystal
- Pureed Cookies, Cakes, Twinkies, Kruschikies, Zeppollis, Donuts / just eat it!

Performing the Demonstration =

- Follow the Doctor DeMento Mini-Manual for the operation of the M-500 IR and GRAMS Software… of follow the Manufacturers instructions for your particular IR system.
- In general… SCAN the CLEAN, EMPTY ATR Crystal to get a “Background” Scan.
- Then… “apply” your SAMPLE to the ATR Crystal, Scan it and RATIO it to the Background to get your typical “%T” Scan.
- Compare different FOODS and evaluate specific Nutritional Groups.
- Take Alka-Seltzer or Zantac afterwards… depending on the Sample MATIRX.

Pieces of Adhesive Tape / firmly stuck against the Prism Cell Crystal

Pieces of flat, smooth Plastic / clamped firmly down on Prism Cell Crystal

OR: Anything that can be smeared between 2 Salt Plates (must be clear and water-free!)

Any powder material that can be pressed into a Potassium Bromide Pellet (dry, inorganic)

RESULTS:

Based on the principles of Infra-Red Vibrational Spectroscopy over the past 60+ years, you should see these ABSORBANCE Peaks in the highlighted region (the "fingerprint" region):

~3300 cm-1 This is a big, broad peak for **Free** WATER (H2O)
~1725 cm-1 This is the primary CARBONYL (-C=O) ABS Peak from Carboxylic Acids, Ketones, Esters (triglycerides / FATS), and Aldehydes
~1640 cm-1 This is a linear peak for HYDROXYL (-OH) of **Bound** WATER
~1550 cm-1 This is one of the POLYAMIDE (-NH-C=O) peaks found in most Proteins & Nylon-based Plastics.
~1465 cm-1 This is the "chain" METHYLENE (-CH2-) that makes up any long-chain Hydro-Carbon compound.
~1380 cm-1 This is the terminal METHYL (-CH3) absorbance from the ends of the long Hydro-Carbon chains.
~1100 cm-1 This a Carbon-Oxygen "bending" (-C-O-) Absorbance Peak that is associated with Carbohydrates and Sugar-like materials.

REFERENCES:

{coming soon!}