

Applications of Near Infrared Radiation Spectroscopy- A Review

K. Bhavya Sri ^{*1}, G. Manusha ², D.Anil³, Mogili Sumakanth ⁴

¹Research student department of pharmaceutical analysis,
RBVRR women's college of pharmacy, Barkatpura, Hyderabad-500027, India.

²Associate Professor, Department of pharmaceutical analysis,
RBVRR women's college of pharmacy, Barkatpura, Hyderabad-500027, India.

³Research Student, 1937 Jerry Ave, Charles City, Iowa, University of Alaska Fairbanks

⁴Professor and Principal Department of pharmaceutical chemistry,
RBVRR women's college of pharmacy, Barkatpura, Hyderabad-500027, India

*bhavya.kagga@gmail.com

Abstract:

The primary experimental method used in atoms and molecular physics is called spectroscopy, and it includes observing the light that passes through or exits when an atom or molecule changes its energy level in order to ascertain what state it is in. The variation in energy between the two states of being, which determines the frequency that light is absorbed or emitted, can be used to detect interactions that disturb those energy states. Spectroscopy is divided into a number of categories based on the electromagnetic radiation's range and underlying principles. This review, we discussed, among other things, the Near Infrared Spectroscopy principle, equipment, and applications.

Keywords: Near infrared spectroscopy, Quality control, Pharmaceutical analysis, Quantitative analysis.

INTRODUCTION: ^[1]

An essential record that provides adequate details regarding the framework of a molecule is the infrared spectrum. For raw material testing, product quality control, and process monitoring, NIR spectroscopy has become increasingly popular in procedure evaluation and pharmaceutical business. The non-destructive and quick technology known as near-infrared radiation spectroscopic (NIRS) is being used more frequently to assess the quality of food. The analytical technique of near-infrared (NIR) spectroscopy has both qualitative and quantitative uses. Proteins, cellulose, fatty acids, ash-like material oil, and salt determination has been the most widely used use of NIRS. The absorption spectroscopy technique known as near infrared spectroscopy (NIRS) aids in identifying a compound's chemical make-up [Gabriele R., 2005].

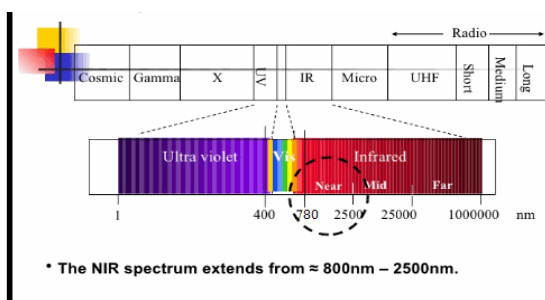


Figure 1; NIR Spectra.

TYPICAL NIR SPECTRUM

NIR Spectrum: Overtones and combinations of basic vibrational bands. [O.H. Wheeler., 1960]

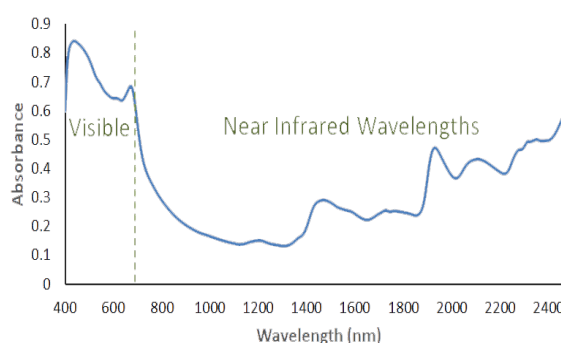


Figure 3: Near Infrared Wavelengths

Theory & Principles: ^[2] A spectroscopic technique known as near-IR (NIR) is based on molecular overtones and combinations of vibrations of CH, O-H, and N-H. A normal mode of vibration for a specific molecule involves inner atomic motions in which every atom move in phase at the same frequency but with varying amplitudes. Overtones are additional vibration transitions that correspond to these regular vibrations. The sample material interacts with NIR light (800–2500 nm). Near-infrared energy-absorbing molecules oscillate in two modes [Franklin EB., 2002]

Stretching: The Constant Change in Intraatomic Separation Around the Meridian of Bonding between each of Two Atoms is what it specifies.

Bending: It describes modifications to Bond Angle.

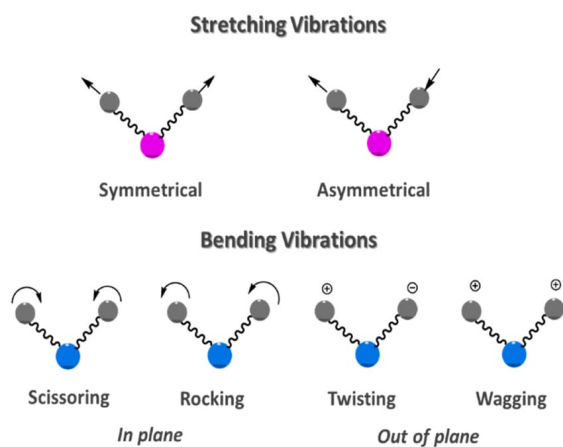


Figure 4: Stretching Vibrations

INSTRUMENTATION:^[25]

Light emitting diodes (LEDs), which are comprised of semiconductors, are the most significant source of near-infrared light for scans the wavelengths of an organic chemical. For the emission of near infrared light at a specified wavelength, gallium arsenide is utilised as a semiconductor. These don't create the radiation, which is highly beneficial and active to NIR, between 1700 and 2500 nm. [Dr. Marcel B., 2013]

Tungsten lamps (incandescent bulbs), which can generate heat up to 1100k, are also employed as light sources. These lamps produce a significant amount of NIR light in addition to visible radiation. Therefore, filters are required to reduce visible radiation, which aids in preventing unneeded sample heating. The benefits in this source are that it is inexpensive and easily accessible, and that these lamps cannot increase their output of energy as the source's voltage is raised. Since a wavelength of interest may be chosen, the current wave length selection look at can be used to assess the instrument's capabilities. LEDs emit light with a certain wavelength, although it is a broad spectrum of energy. Since LEDs and filters work well together as wavelength selectors and are inexpensive, highly effective, and portable, low-cost devices without moving parts can be made. Grates are surfaces made of metal or glass that have been carved with a lot of tiny parallel lines. Diffraction occurs when the light beam contacts the surface, dividing it into several wavelengths.

A beam splitter divides the light beam into two beams for the interferometer wavelength selection. The stationary and moving mirrors are struck by the two separated beams, which are then reflected back to the beam splitter. After being recombined, they leave an interferometer in the sample direction.

The operation of the interferometer at a particular wavelength depends on the material chosen in the beam splitter (KBr, CaF₂, or quartz).

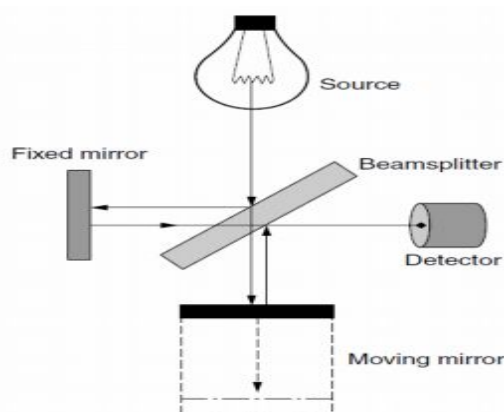


Figure 5: beam splitter interferometer

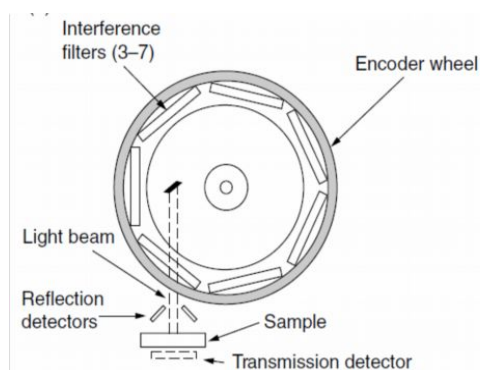


Figure 6: Flow diagram of Tilting filter

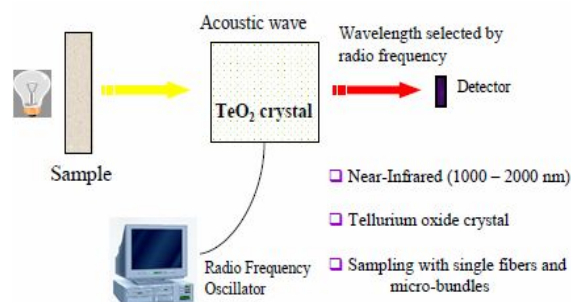


Figure 7: Flow diagram of AOTF used NIR spectroscopy

When using an AOTF (Acoustic Optic Tuneable Filter), light is focused onto a TeO₂ crystal. Using a piezoelectric material bound to the crystal, a high-frequency acoustic pulse in the vicinity of radio frequencies is linked into the crystal. Rapidly moving through the crystal, these acoustic waves interact with other broadband light to produce separate monochrome beams of light, each polarised in a distinct way. These monochromatic beams can be supplied to the sample as a supply of NIR light because they are connected using optical fibres. The fact that AOTF has no moving components, changeable intensity, and produces narrow beams are advantages. It only covers a small portion of the spectrum (1000–2000 nm), thus detecting highly absorbing substances can be challenging.

The first kind of interference filter is a tilting filter. The incidence angle of light flowing through the filter

determines the energy that is transmitted at various wavelengths. For higher accuracy, or wavelength repeatability, the filters of this were installed in an encoder wheel.

sample container quartz or glass cells for performing liquid transmission. The sizes and patterns of the cells vary depending on the instrument's design. The test sample holders are made in accordance with the instrument in terms of size and shape. Regarding solid, dry, and grained samples, the sample's holder cells might have a spherical form. For the purpose to scan the largest possible portion of the sample and to remove any uneven distribution of the sample, round cups are frequently rotated.

DETECTORS:^[23]

Wavelength range, spectrometer design features, and detector properties including photosensitivity (responsivity), noise equivalent power (NEP), and detectivity all affect the choice of detectors. When noise levels are low, photosensitivity is defined as the voltage that is output per unit of absorbed radiant at a specific wavelength. In situations where the data : noise ratio is 1, NEP estimates the amount of light. The statistic utilised to compare the effectiveness of various detectors is detectivity. The best detector should have a greater signal detectivity. The signal-to-noise ratio at a specific electrical frequency or within an 1HZ band while 1 watt of radiant energy is applied to a 1cm² active area detector is known as the detectivity. Lead sulphide detectors (PbS), Pb selenium detector (PbSe), Silicon detectors, Indium antimonide detectors (InGaAs, InSb), and Common Charge Coupled Devices (CCD) are among the detectors used in NIR spectrometers. [Brad S.,2014]

Detector	Wavelength range (nm)	Region	*Responsivity/**detectivity	Remark
PbS	1100-2500 400-2600 1100-4500	NIR UV-NIR NIR-MIR	Intermediate/intermediate	PbS 'sandwiched' with silicon photodiodes, are often used for VIS-NIR
PbSe	1100-5000	NIR-MIR	Fast/ high	The detector must be cooled with liquid nitrogen
InGaAs	700-1700	NIR NIR Raman	Fast/very high	Linear arrays high sensitivity, dynamic range, signal-to-noise performance and stability FT-NIR Diode arrays spectrometers
InSb/InAs	1000-5500	NIR MIR IR	Fast/ very high	High quality detector Detector photodiodes
CCD	800-2200	NIR	Fast/high	High performance detector Applied in cameras Diode arrays spectrometers

Table 1; Detectors used in NIR spectroscopy

Pbs Detector:

Light having ranges from one to three microns can be detected by Pbs detectors. In near infrared radiation (NIR) applications like locating O-H and C-H bonds, it is ideally suited. It enables the identification and classification of fatty acids and alcohols. Comparatively speaking, Pbs detectors are more sensitive than Pb Se detectors. Due to the ability to record the entire spectrum, equipment based on array detectors are an approach in fast moving process streams. Spectrometers based on silicon diode arrays is

readily available. Detector arrays made of indium gallium aluminium or Pbs semiconductors are available for extended NIR wavelengths. The NIR spectrometer is made for in-line quality monitoring and depends on a Pbs detector array.[J. Workman., 2001]

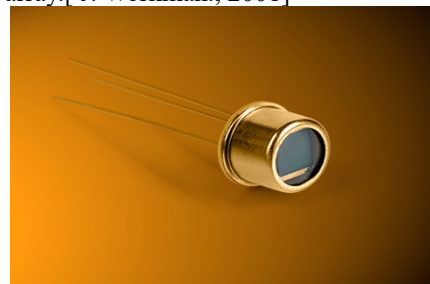


Figure 8: Pbs Detector

Pb Se Detector:

A typical semiconductor detectors having a resolution of between 1 and 5.2 microns is polycrystalline lead selenide (Pb Se). A particularly potent inner photoelectric effect is present in Pb Se. The resistance decreases when infrared light enters the lead selenide detector. PbSe forms a thin film with distinctive physical characteristics and a band structure that is frequently utilised for NIR applications. It is employed to find items that emit photons as thermal radiation. The initial semiconductor materials employed as detectors were lead salts like lead selenide (PbSe) and lead sulphide (PbS). For imaging applications, PbSe is used in all of its forms, including monocrystalline and polycrystalline. The photodetectors also make use of PbSe QD thin films.

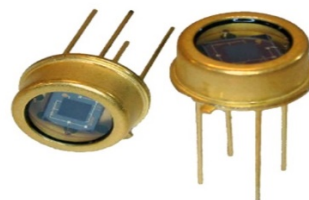


Figure 9: Pbse Detector

Indium antimonide(In Sb):

Infrared to MWIR groups, between 1 and 5.5 microns, are where indium antimonide (In sb) detector function. They are quite sensitive. Indium and antimony are combined to form the crystalline chemical known as indium antimonide (In sb). It can be used as thermal imaging cameras, infrared detectors, and other things. InSb has outstanding sensitivity and excellent stability for near-infrared radiation. Similar detectors made of other materials can't compare to InSb detectors. Materials made of inSb have an elevated infrared absorption coefficient. Photosensitive circuits in InSb material have progressed from unit semiconductors to multi-element, linear array, and area array chips. The signal circuit and sensitive device are integrated, then positioned on the focus plane. One of the important elements that affects the capability to detect efficiency of the cooler InSb detector is the functionality of the photosensitive chip.

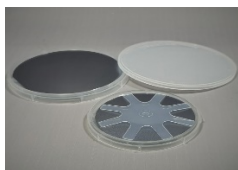


Figure 10:Indium antimonide

Indium Gallium Arsenide(In GaAs):

The most typical semiconductor used in the field of infrared spectroscopy for analysing light with ranges between 0.9 and 1.7 microns is the indium gallium arsenic (In GaAs). Gallium and arsenic combine to form gallium arsenide (GaAs). Devices including laser diodes, photovoltaic cells, and infrared emitting diodes are frequently made using it. indium gallium aluminium detector is widely utilised in the agricultural sector, medical diagnosis, surveillance of safety, and remote sensing in aerospace. High homogeneity, high stability, and a rapid detection rate are the traits of the InGaAs sensor. Because of its minimised dark current and longer integration periods, Indium gallium aluminium has a broader spectrum of applications. By producing the material on the lattice mismatched substrate, the detection range in the InGaAs sensor can be increased. Lattice mismatch, on the other hand, results in a lesser band gaps and more crystal structural flaws, leading to dark current and noise.

Charge-coupled device(CCD):

A sensitive to light integrated circuit known as a charge-coupled detector (CCD) records photographs through the conversion of photons to electrons. The image components are divided into pixels by a CCD sensor. Each pixel is transformed into an electrical charge, the strength of which is determined on the amount of light it was able to collect. A CCD is a multi-channel array of silicon-based UV, visible, and near infrared light detectors. The full spectrum is able to be detected in a single operation because it supports multi-channel operation.

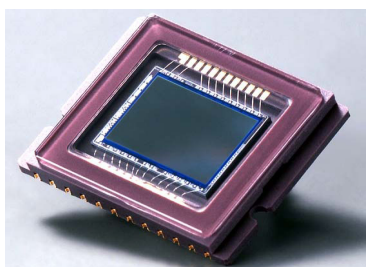


Figure 11:Charge Coupled Detector

TYPES OF NIR INSTRUMENTS:^[4]

Scanning spectrophotometers, Fourier transform spectroscopy, Acoustic Optical Tuneable Filter spectrophotometers, and Photo Diode Array spectrophotometers are examples of NIR equipment. The light that is projected onto the diffraction grating in scanning spectrophotometers comes from an optical fibre. The grating separates the electromagnetic radiation into its individual wavelengths. A small group of wavelengths

are permitted through the small opening by mechanically rotating the grating. Transmitted light enters the slit and is directed towards the detector. On the other hand, light is transmitted direct to the sample in post-dispersive equipment. The fibre optic cable's reflected light is sent to a grating for dispersion before it goes through a slit in front of a detector. Michaelson interferometer is the foundation of the Fourier transform spectrophotometer. Acoustic Optical Tuneable Filters serve as the foundation for spectrophotometers. To analyse the entire sequence of wavelengths, photo diode array spectrophotometers set the detector at the proper distance from the diffraction grating. Only silicon PDAs below 1000nm can be used. The PDA is now more advantageous for NIR analysis due to the more recent accessibility of InGaAs detector material.

These detectors can detect light between 900 and 2200 nm. This spectrophotometer makes use of optical interference filters. These filters only let through light with a specific wavelength range [Lin H., 2009]

SCANNING SPECTROPHOTOMETER^[16]

Characteristic Group Absorption Regions:

More information is accessible from a compound's near-infrared spectrum than is typically provided by its electronic spectrum. In this method, some groups absorb typically within a predetermined range. The position of the shift in absorption for a certain group may alter as the molecule's structure changes. In basic circumstances, bond lengths and bond angles can be determined using the force constant that causes the absorption peaks. Numerous questions, such as which groups are found in the compound, what surroundings are influencing it, or what kind of carbon skeleton is there in the molecule, spring to mind once the near infrared spectrum of an unknown compound is scanned. The distinctive groupings absorb light at a specific frequency. In order to identify molecules and anticipate the existence of functional groups, this technique is highly helpful. and characterise the group absorption zone in the NIR.

ADVANTAGES:

Since NIR optical routes across liquid samples may be millimetres or even centimetres long instead of microns, NIR was able to resolve some of the challenging sampling approaches characteristic of IR after the development of fibre optics, powerful light sources, and sensitive detectors. There is no need for sample dilution, sample preparation, or matrices like mineral oil or KBr because NIR radiation penetrates the sample with a considerably deeper depth. so as to reduce sample damage and errors during sample preparation. [Romero-Torres., 2010]

DISADVANTAGES:

Process NIR suffers from the properties of its spectra, which, in contrast to IR spectra, are frequently made up of large, overlapping peaks. No structural elucidation was achieved, and analysing wetted samples was not advised. [Romero-Torres., 2010]

APPLICATIONS:^[19]

In the petrochemical sector, it is helpful. In the food sector, NIR spectroscopy is frequently employed. It is a commonly used instrument for quality control in the petrochemical, chemical, polymer, and food sectors for raw materials. For the analysis of hydrogen bonding, hydration, and molecular self-association, NIR spectroscopy is highly helpful. Process control and observation. Raw material identification and qualification.[Jones JA et al., 1993]

CONCLUSION:

Due to its applicability, we came to the conclusion that this near-infrared spectroscopy has been one of the best methods.

REFERENCES :

- Gabriele R, Near-infrared spectroscopy and imaging: Basic principles and pharmaceutical applications, *Advanced Drug Delivery Reviews*, Volume 57, 2005, Page No. 1109–1143.
- Franklin EB, Theory and principles of near infrared spectroscopy, *Spectroscopy Europe*, Volume 14, 2002, Page No.12-18.
- Lin H., Ying Y. Theory and application of near infrared spectroscopy A review. *Food Qual.* 2009
- Dr. Marcel B, Use of NIR spectroscopy and multivariate process spectra calibration methodology for pharmaceutical solid samples analysis, *Journal of pharmaceutics*, Volume 1, 2013, Page No.56-64.
- O.H. Wheeler, Near Infrared Spectra. A neglected field of spectral study, *J. Chem. Education*, 1960, 37, 234-236.
- R.F. Goddu, and D.A. Delker, "Spectra-Structure Correlations for the Near-Infrared Region, *Anal. Chem.* 1960, 32, 140-141.
- L. Weyer and S.-C. Lo, "Spectra-Structure Correlations in the Near-infrared," In *Handbook of Vibrational Spectroscopy*, Volume 3, Wiley, U.K., 2002, pp. 1817-1837.
- J. Workman, *Handbook of Organic Compounds: NIR, IR, Raman, and UV-Vis Spectra Featuring Polymers and Surfactants*, Volume 1, Academic Press, 2001.
- Brad S, The current state of near infrared spectroscopy application in the pharmaceutical industry, *Journal of near infrared Spectroscopy*, Volume 22, 2014, Page No.153- 156.
- <http://www.science.uva.nl/onderwijs/thesis/centraal/files/f120518228.pdf>. Received on: 15-12, 2010.
- http://www.aapspharmaceutica.com/inside/discussion_groups/socal/imagespdfs/Romero-TorresMar2010.pdf Received on: 15-12-2010.
- Sanchez FC, Massart DL, Dive SS and Hailey PA, The role of NIR spectroscopy in the measurement of pharmaceutical manufacture, *Fresenius Journal of Analysis chemistry*, Volume 352, 1995, Page No.771-778
- Cozzolino D. An overview of the application of near infrared spectroscopy *Appl. Rev.* 2015;50:859–867.
- W. Kaye, Near-infrared spectroscopy; A review. I. Spectral identification and analytical applications, *Spectrochimica Acta* 1954, 6, 257-287.
- G.D. Schrieve, G.G. Melish, and A.H. Ullman, "The Herschel-Infrared--A Useful Part of the Spectrum," *Appl. Spectrosc.* 1991, 45, 711-714.
- D. Burns, and E. Ciurczak, *Handbook of Near-Infrared Analysis* 2nd Edition, Marcel-Dekker, Inc. New York, 2001.
- H. Mark and J. Workman, *Statistics in Spectroscopy* 2nd Edition, Elsevier, Amsterdam, 2003.
- H. Martens and T. Naes, *Multivariate Calibration*, John Wiley and Sons, Chichester, 1992.
- Jones JA, Last IR, MacDonald BF and Prebble KA, Application of near- infrared reflectance analysis in the pharmaceutical industry, *Journal of Pharmaceutical and Biomedical Analysis*, Volume 11, 1993, Page No.1227– 1231.
- Blanco M and Villar A, Development and validation of a method for the polymorphic analysis of pharmaceutical preparations using near infrared spectroscopy, *Journal of Pharmaceutical Science*, Volume 92, 2003, Page No.823– 830.
- Blanco M, Eustaquio A, Gonzalez JM, and Serrano D, API determination by NIR Spectroscopy Across pharmaceutical production process, *Journal of Pharmaceutical and Biomedical Analysis*, Volume 22, 2000, Page No.139–148.
- Alba E, Paul G, Roger DJ, Anthony CM and Andrew DT, Quantification of Paracetamol in intact tablets using nearinfrared transmittance spectroscopy Analyst, *International journal of Chem Tech Research*, Volume 123, 1998, Page No.2303–2306
- Roggo Y, Chaus P, Hauron L, Mammoz CL, Edmond A and Jent N, A review of near infrared spectroscopy and chemometrics in pharmaceutical technologies. *Journal of Pharmaceutical and Biomedical Analysis*, Volume 44, 2007, Page No.683-700.
- Neville WB, Jee RD, Anthony CM and Smith MR, Application of transmission near-infrared spectroscopy to uniformity of content testing of intact steroid tablets Analyst, *International Journal of Pharmaceutics*, Volume 126, 2001, Page No.2207–2211.
- E. Stark, K. Luchter, and M. Margoshes, "Near-Infrared Analysis (NIRA): A Technology for Quantitative and Qualitative Analysis," *Appl. Spectrosc. Reviews* 1986, 22(4), 335-399.
- G.D. Schrieve, G.G. Melish, and A.H. Ullman, "The Herschel-Infrared--A Useful Part of the Spectrum," *Appl. Spectrosc.* 1991, 45, 711-714.
- K.B. Whetsel, "Near-Infrared Spectrophotometry," *Appl. Spectrosc. Reviews* 1968, 2(1), 1-67.
- B. Osborne and T. Fearn, *Practical NIR Spectroscopy with Applications in Food and Beverage Analysis* 2nd Edition, John Wiley & Sons, Inc., New York, 1993.
- P. Williams and K. Norris, (Eds.), *Near-Infrared Technology in the Agricultural and Food Industries* 2nd Edition, American Association of Cereal Chemists, St. Paul, Minnesota, USA, 2001.
- Maric L and Homseka I, Contribution to the Application of Near-Infrared Spectroscopy Volume 78, 2010, Page No.574.