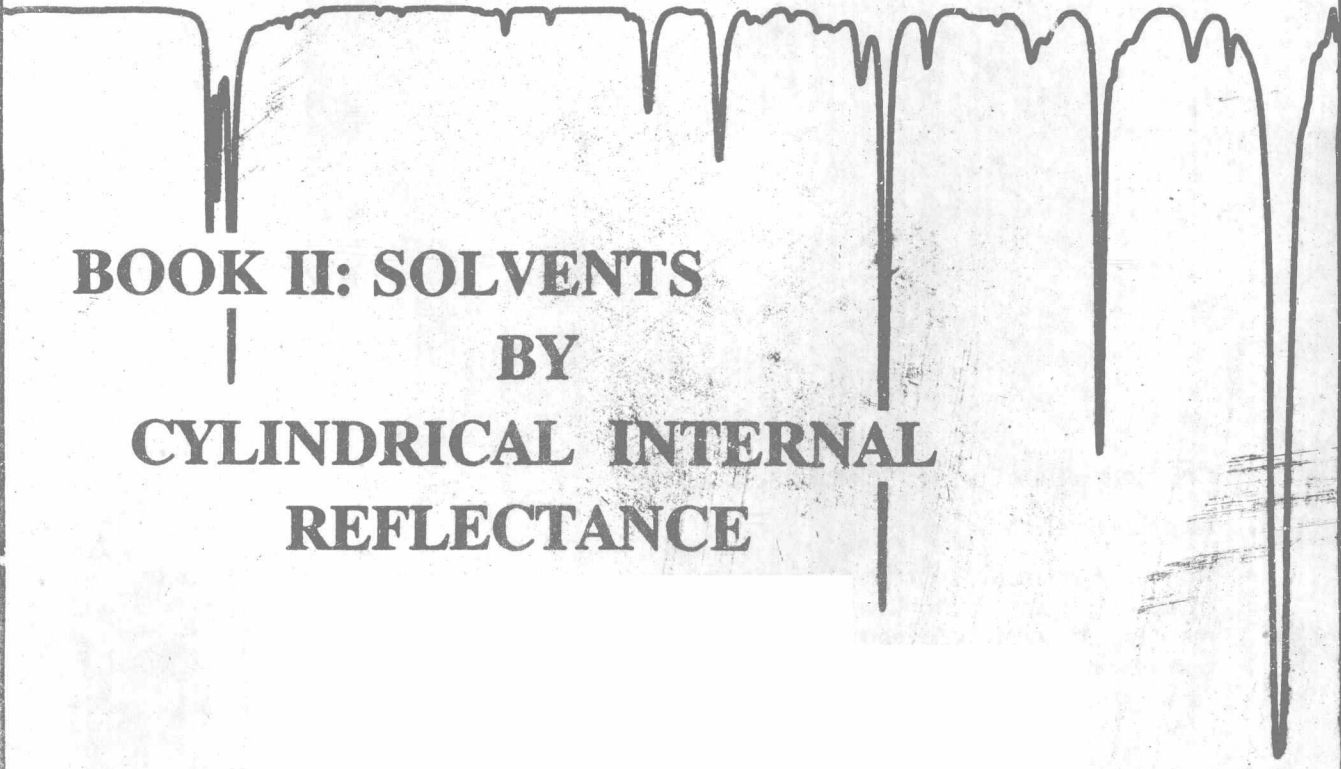


SPROUSE COLLECTION
OF
INFRARED SPECTRA

BOOK II: SOLVENTS
BY
CYLINDRICAL INTERNAL
REFLECTANCE

**SPROUSE
COLLECTION
OF
INFRARED SPECTRA**

An infrared spectrum line graph showing transmittance versus wavenumber. The graph features several sharp absorption peaks, with prominent ones in the fingerprint region (below 1500 cm⁻¹) and a strong, broad peak around 3400 cm⁻¹. The baseline is relatively flat with minor fluctuations.

BOOK II: SOLVENTS

**BY
CYLINDRICAL INTERNAL
REFLECTANCE**

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We at Sprouse Scientific Systems, Inc. would like to thank the following people whose efforts have been instrumental in the publication of this second book of the SPROUSE COLLECTION OF INFRARED SPECTRA.

First, we would like to thank the personnel at Spectra-Tech, Inc. for their assistance and support during the preparation of this collection of infrared spectra. Spectra-Tech kindly supplied the *CIRCLE* sampling accessory used to measure the spectra presented in this book. In particular, we would like to thank John Coates for his valuable suggestions and assistance during the preparation of this collection. Also, a special thanks goes to Janet Harrold for her assistance during the final stages of the book.

Finally, we offer our thanks to the personnel at Sprouse Scientific Systems, Inc. for their continued efforts in preparing both the spectra and the final copy for the book. It is indeed a combined effort of several people to produce such a compilation of information.

D. Hansen
Editor

PREFACE

This collection of reference spectra for common organic solvents demonstrates how simple, high quality, easy to use sampling accessories can be used in conjunction with an FT-IR to routinely measure high quality infrared spectra. Of course, when an advantage is gained in one area, then there is generally always one traded-off elsewhere. While ease of sample preparation can be achieved using the *CIRCLE*, one must sacrifice direct comparisons of band intensity ratios and band shapes measured by internal reflection to those measured using conventional transmission spectroscopy. Since the *CIRCLE* is based on the principles of internal reflection, sample absorption bands have a different appearance from conventional transmission spectra because the sample pathlength is not constant for all wavelengths of light as the wavefronts propagate through the internal reflection crystal and are selectively absorbed at the sample/reflection crystal interface. Absorption bands at longer wavelengths (lower frequencies) appear stronger and bandshapes are skewed toward the lower frequencies because of longer effective sample pathlengths at greater wavelengths of light. This collection of spectra was generated for materials commonly used with the *CIRCLE* sampling technique in order to provide a collection of reference spectra that can be directly compared with those measured using the same technique. It obviously does not contain reference spectra for all compounds that can be analyzed using the *CIRCLE*, however it is an attempt to help satisfy the need for high quality reference spectra which are unique to specific sampling accessories and techniques, and have not been available in the past.

James F. Sprouse, Ph.D.
President
Sprouse Scientific Systems, Inc.

INTRODUCTION

THE CIRCLE® CELL

The Cylindrical Internal Reflectance Accessory, *CIRCLE*, is a high quality optical accessory used for performing attenuated total reflectance (ATR) measurements of all types of liquids. The basic premise of successful ATR analyses is that incident radiation must enter a material of high refractive index, penetrate a sample of lower refractive index one or more times, and exit at the reflecting interface. Traditionally, this type of analysis has been accomplished through the use of a rectangular "sandwich"-type transmission cell. However, this type of cell is subject to certain limitations. The rectangular shape of the transmission cell is not matched to the circular FTIR beam, therefore it can cause a loss of energy. Additionally, strongly absorbing liquids require a cell with a very short pathlength (typically 7 to 20 microns). A cell of these dimensions is extremely difficult to fill and clean, particularly if a viscous sample is involved.

The design of the *CIRCLE* greatly reduces all of these difficulties while still employing the basic premise of ATR. The *CIRCLE* utilizes a cylindrical internal reflection element with cone shaped ends which are well matched to the circular beam of the FT-IR instrument. Cassegrainian mirror lenses insure optimum transfer of the infrared energy to and from the cell. The percent of energy getting through the accessory without a sample present is generally 18-27%, relative to the open beam throughput of the FTIR spectrometer.

The versatility of the *CIRCLE* is demonstrated for aqueous analysis using FT-IR spectrometry. This has been a difficult task in the past, due to the highly absorbing nature of water as a solvent. As seen by the Beer-Lambert Law ($A=abc$), detectability depends upon concentration, absorptivity and pathlength. However, the presence of large obscuring bands such as the O-H stretch of alcohols, tends to obscure the presence of the smaller and weaker bands at long pathlengths. These two facts tend to specify an optimal range of pathlengths for most aqueous samples, and the pathlength of the *CIRCLE* falls within this range. The effective pathlength of the *CIRCLE* varies from 3.6 microns at the near end of the IR to 21 microns at the far end. Additionally, the pathlength is very stable, i.e. it is not affected by changes in temperature and pressure, or by the wear of the window surface, as is the transmission cell. Using a Zinc Selenide crystal, concentrations of 0.5% of moderately absorptive liquids are detectable and produce qualitative and quantitative data of superior quality in the region of 3200-750 wavenumbers. Standard triglycerol sulfide (TGS) detectors yield good signal-to-noise ratios, however, improved ratios are further achieved with mercury cadmium telluride (MCT) detectors.

HANDBOOK ORGANIZATION

This collection containing spectra for 350 solvents is arranged by chemical class. There are 13 individual classes, each separated into non-aromatic and aromatic subdivisions. This type of classification allows the user to easily compare absorption characteristics of compounds with similar functionality.

The text and spectral information for each compound are arranged on two adjacent pages, so the user has access to both the chemical and physical property information as well as the spectral information, when the book is opened to a single chemical compound. The left-hand

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page includes the chemical and physical properties associated with each compound and the right-hand page includes the *CIRCLE* cell spectrum and associated information.

For each compound, the primary name is located on the outer left-hand corner of each adjacent page for easy searching, and it is preceded by a spectral number, used as the primary identifier in the book. The spectrum number is also used as a reference for all indices presented, as well as in conjunction with software programs offered along with the book. The primary name given for each compound is either the Chemical Abstracts Service (CAS) recommended name, or its most recognizable common name that it is referred to in the open literature. Because chemists refer to common solvents by a number of names and acronyms, we have included a listing of synonyms for each compound along with the primary name. These synonyms are also listed in the alphabetical index, so that a reference spectrum for a particular compound can be easily located by any of the names. Numerical information for the solvents has also been included. This includes the Chemical Abstracts Service registry number, the NIOSH number and the Wiswesser Line Notation. The Chemical Abstracts Service Registry Number is a unique number given to each compound by the Chemical Abstracts Service, a division of the American Chemical Society. The NIOSH number, (also known as the RTECS number) is also a unique number assigned to each compound, comprised of a seven digit number preceded by two letters. This number is listed in the Registry of Toxic Effects of Chemical Substances by the National Institute for Occupational Safety and Health. The Wiswesser Line Notation is an alphanumeric notation used to denote chemical structure, originated by Chemical Information Management, Inc. of Cherry Hill, New Jersey.

Data including the molecular weight of the sample, melting point, boiling point, flash point, refractive index, density, vapor pressure and dielectric constant are also reported for most of the compounds. The molecular weight of each sample is given as the gram molecular weight. The melting and boiling points are given in degrees Celsius. A variance is provided with the melting and boiling points when the accuracy is known. When a variance is not provided, then a melting or boiling point range was not specified in our literature sources. Therefore, the variance values reported are provided for guidance only and should be confirmed through additional literature sources. For the convenience of the user, the flash point for each compound is given in degrees Celsius as well as in degrees Fahrenheit. The refractive index and densities are given at specified temperatures in degrees Celsius, and wherever possible, the values at two different temperatures are given. The vapor pressure is given in atmospheres at 25 degrees Celsius unless it is noted otherwise. The dielectric constant is also given at specified temperatures in degrees Celsius. Additional information including solubility, uses and hazard information is also provided for most of the compounds.

Located on the spectral page is the molecular structure and the empirical formula for each compound. The source of each sample is noted, centered directly above each spectrum. All samples used for measuring the spectra in this book were obtained from commercial manufacturers and are readily available from these sources.

Four indices are included in this handbook. The first is a numerical index of the compounds, listing the primary name given for each compound. An alphabetical index is second, inclusive of the many synonyms and acronyms associated with each of the primary

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names. An empirical formula index is third, listing the compounds in increasing numerical order, i.e., $C_xH_yElement_z$. A complete explanation to the use of this index is located at its preface. The Chemical Abstracts Service registry number index is presented last, and lists each compound by increasing CAS number. All information listed in these indices is represented by spectral number as well as page number.

INSTRUMENTATION

All spectra were measured on a Fourier Transform Infrared (FT-IR) Spectrometer in Sprouse Scientific's laboratory using an cylindrical internal reflectance sampling accessory. The sampling accessory was a Micro-CIRCLE Cell which is commercially available from Spectra-Tech Inc., 652 Glenbrook Road, Stamford, CT 06906. The CIRCLE uses a micro "open-boat" sample well configuration which holds 0.5 milliliter sample volume. A Zinc Selenide internal reflection crystal 3.17 millimeters in diameter by 40 millimeters in length with 45° coned ends was used for all of the measurements. The crystal typically transmitted approximately 15% of the incident energy through the ZnSe rod in a dry $N_2(g)$ purge environment. The FT-IR used for all measurements was a Digilab Model FTS-40 containing a Potassium Bromide beam splitter, HI-Intensity Globar Source and DTGS detector. Fast Fourier Transform (FFT) processing was completed using triangular apodization with a zero filling factor of two. All spectra were measured at a nominal resolution of 2 cm^{-1} from 4400 to 550 cm^{-1} in the mid-IR region. Data were collected with sufficient measurement time (signal averaging) to provide signal-to-noise ratio greater than 5,000 to 1 over the spectral transmission region of the ZnSe crystal.

SPECTRAL FORMAT

All spectra in the book are presented in a %Transmission format using the Zinc Selenide internal reflection crystal surrounded by dry $N_2(g)$ as the reference. The %T spectrum for each sample was measured by collecting a single beam reference spectrum for the ZnSe crystal, I_{ZnSe} , without a sample in the sample boat, and storing it in the computer. The sample boat was then filled with 0.5 milliliters of sample and its single beam spectrum measured, I_{Sam} . The % Transmission spectrum for the sample was then computed according to the following equation:

$$\%T = \frac{I_{ZnSe}}{I_{Sam}} \times 100$$

After measuring the spectrum for each sample, the sample boat was cleaned by rinsing the sample well with chloroform or methylene chloride depending upon the sample, then drying with a stream of dry $N_2(g)$. For Samples which were difficult to remove from the surface of the crystal, the sample boat was submerged in an ultrasonic cleaner containing a methanol/acetone mixture for a short period of time, then the above rinsing and drying process repeated. Crystal contamination was monitored by measuring and observing the single beam reference spectrum for the crystal before each new reference was accepted for use with the next sample to be

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measured. All spectra presented in the book were plotted directly to the graphs from the digital reference spectra.

INSTRUMENT QUALITY CONTROL

The infrared spectrometer was tested for frequency calibration, photometric accuracy and baseline stability on a routine basis throughout data measurement. The following quality control methods are used as standard operating procedure in the Sprouse Scientific laboratories:

1) Wavenumber Calibration

The IUPAC (International Union of Pure and Applied Chemistry) recommended wavenumber standard material was used for the spectrometer wavenumber calibration. Two mixtures were used:

- a) A solution of 98.4 parts Indene, 0.8 parts Camphor and 0.8 parts Cyclohexanone (wt/wt) was used to calibrate the spectral range between 4400-600 cm^{-1} .
- b) A solution containing equal parts (wt/wt) of Indene, Camphor and Cyclohexanone was used to calibrate the spectral range between 600-400 cm^{-1} .

Infrared spectra of the two calibration mixtures were obtained using a 0.25mm fixed path length liquid cell. The peak positions of 50 bands were located and confirmed to the IUPAC assigned band locations. This performance check was run approximately every 30 days. Carbon dioxide reference bands were used for daily wavenumber calibration checks. Peak positions were measured at 2361.6 ± 0.2 , 2336.6 ± 0.2 and $668.5 \pm 0.2 \text{ cm}^{-1}$.

2) Instrument Performance and Stability

The performance and stability of the instrument was monitored on a daily basis, without the *CIRCLE* installed in the FT-IR by measuring the RMS noise in a 100%T baseline at selected frequencies across the spectral range. The 100%T line was measured each day by taking the ratio of one single beam spectrum to another. The 100% line was plotted between 98 and 102%T and the RMS noise level was calculated in the spectral regions of 3950-4050, 1950-2050 and 950-1050 cm^{-1} . A baseline with a maximum of 2% RMS peak-to-peak noise within these regions was used as the criterion for acceptable performance for this instrument. After the daily instrument performance checks, the *CIRCLE* was installed into the FT-IR sample compartment and the spectra measured.

3) Atmospheric Contaminants

The presence of water vapor and carbon dioxide were strictly monitored during sample scanning. The optical head was purged continuously with $\text{N}_2(\text{g})$ boiled off from liquid nitrogen in order to minimize background contamination. Extreme care was exercised while scanning of the ZnSe crystal background and sample to achieve compensation of these contaminants. Some water vapor bands are evident in the spectra despite thorough purging of the optical head with dry nitrogen. However, these background bands are generally obvious and do not interfere with the important spectral bands in the sample.

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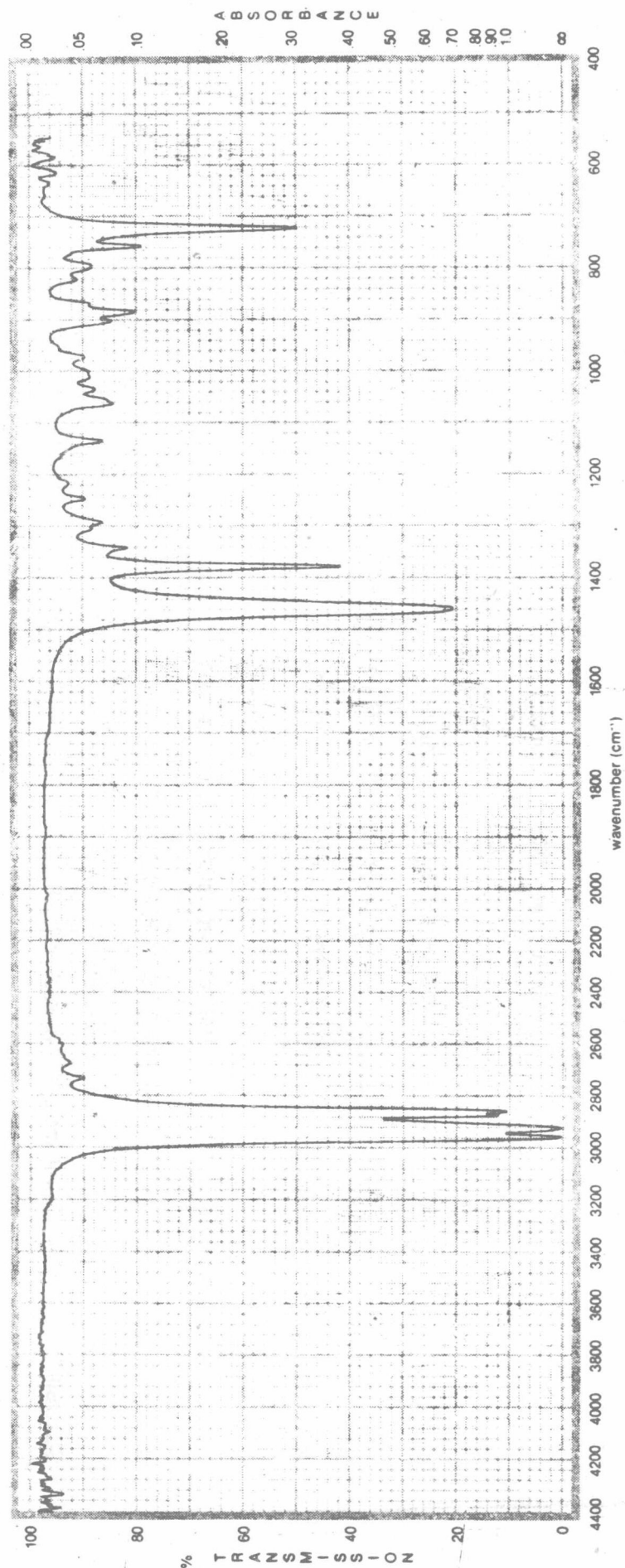
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1 **HEXANE (99 + %)****SYNONYMS:** n-HEXANE**CAS #:** 110-54-3**NIOSH #:** MN9275000**WLN:** 6H**PHYSICAL PROPERTIES****MOLECULAR WT:** 86.18**MELTING POINT:** -95°C**BOILING POINT:** 69°C**FLASH POINT:** -22.0°C/-7.6°F**REFRACTIVE INDEX:** 1.3749/20°C, 1.3723/25°C**DENSITY:** 0.6594/20°C; 0.6548/25°C**VAPOR PRESSURE:** 100/15.8°C**DIELECTRIC CONSTANT:** 1.89/20°C; 1.88/25°C**SOLUBILITY:** miscible with ether, chloroform, alcohol; insoluble in H₂O**COMMENTS:** Colorless, distinct odor. Major component of petroleum ether. Extractant for soy beans, peanuts, cottonseed and linseed oils and rendering of meat scraps. Used in refractive index determination of minerals.**RESPIRATORY IRRITANT, NARCOTIC (in high concentrations)**

SOURCE: J.T. Baker Chemical Co.



GENERAL FORMULA: C_6H_{14}



1 1
HEXANE (99 + %)



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