lies within the (still rather large) uncertainty range for future climate projections, so the risk cannot be ruled out.

See also

Deep-sea Drilling Results. Past Climate From Corals. Thermohaline Circulation. Water Types and Water Masses.

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ABSORBANCE SPECTROSCOPY FOR CHEMICAL SENSORS

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Introduction

Optical methods have played an important role and continue to do so in various fields of chemical analysis. Optical methods that are used in chemical analysis rely on the fact that there is an interaction of electromagnetic radiation with matter. Thus, these methods can provide a rapid and nondestructive tool for analysis. In chemical sensors, a range of wavelengths covered by optical techniques are used that can be divided into the ultraviolet (200–400 nm), the visible (400–780 nm), the near infrared (780–3000 nm), and the infrared (3–50 μm) regions. When electromagnetic radiation in the optical region interacts with matter, dispersion, absorption, diffraction, refraction, and reflection of the light can occur.

Absorbance spectroscopy has been used in conjunction with dry reagent chemistries in the development of optical chemical and biochemical sensors for a variety of applications including environmental and biomedical analyses. These sensors incorporate transducers which consist of active chemicals/biochemicals immobilized on to suitable polymeric materials and interfaced to optical fibres. The developments of transducers, which are the heart of these sensor devices, determine much of the progress in this field of chemical sensors and require a multidisciplinary team of expertise.

This article will review the practical procedures as applied to absorbance based chemical sensors and present some of the developments indicating their potential application in oceanography.

Optical Fibers

Almost all optical sensors employ optical fibers to transmit light to and from the chemical transducer. Optical fibers consist of a core of certain refractive index (n_1), surrounded by a cladding material of lower refractive index (n_2). Most optical fibers are then covered with a protective jacket that has no influence on the wave-guiding properties of the optical fiber (**Figure 1**). Incident light is transmitted through the fiber when it interacts the cladding interface at an angle greater than the critical angle, so that it is totally internally reflected at the core–cladding interface. The optical fiber light transmission characteristics are described by its numerical aperture (NA), which is directly proportional to the sine of the half angle (α) of the acceptance cone of light entering it which, in turn, is related to n_1 and n_2, as in eqn [1].

$$\mathrm{NA} = n_0 \, sin\, \alpha, \qquad \text{and} \qquad \frac{(n_1^1 - n_2^2)^{1/2}}{n_0} \qquad [1]$$

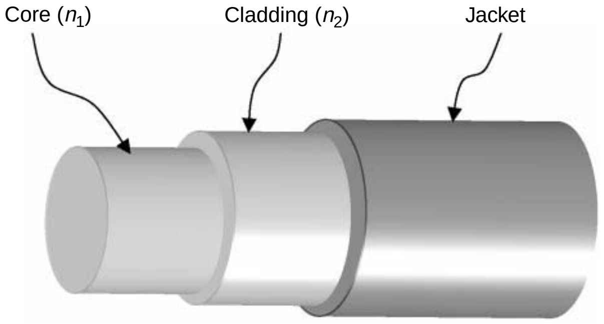

Figure 1 Schematic of an optical fibre.

where n_0 is the refractive index of the surrounding medium, e.g. air.

Basically there are three kinds of optical fibers in use for sensing purposes. These are the multimode step-index, the multimode graded-index, and the single-mode step-index fibers. Common materials used in optical fiber manufacture include plastic (poly(methyl methacrylate)), glass and quartz. Each of these materials has different light transmission characteristics and their choice depends mainly on the sensor signals and the intended application of the sensor.

A small portion of light that is transmitted through an optical fiber or a waveguide by total internal reflection extends outside core and is referred to as the evanescent wave. In an optical fibre, the evanescent wave penetrates the cladding material. The intensity (I) of the evanescent decreases exponentially with increasing distance (d) from the surface according to eqn [2].

$$I = I_0 e^{-d/d_p} \qquad [2]$$

where I_0 is the electric field intensity at the core–cladding interface and d_p, the depth of penetration, is the distance from the interface of the point at which the electric field intensity has reduced to 1/e of its value at the interface. This characteristic depth of penetration is given by eqn [3] where λ_0 is the wavelength of the propagating light. The distance d_p is typically of the order of a fraction of a wavelength of light.

$$d_p = \frac{\lambda_0}{2\pi(n_1^2 \sin^2 \alpha - n_2^2)^{1/2}} \qquad [3]$$

Optical fiber-based spectroscopic sensors can offer several advantages over conventional devices in many areas of application. Much of the advantages are imparted from the use of optical fibers, which are excellent in communicating optical signals. Optical sensors are electrically passive and immune to electromagnetic disturbances. They are geometrically flexible, corrosion resistant, and capable of being miniaturized. They are compatible with telemetry and capable of operation in remote and hostile environments. They can be low-cost, of rugged construction, and intrinsically safe. The optical fibers used in these sensors are capable of transmission of optical signals over great distances with low attenuation of optical power. Thus optical sensors are capable of measurements of samples in their dynamic environment, no matter how distant, difficult to reach, or harsh that environment is. Intrinsic safety aspects are imparted to these sensing devices by the low optical power utilized in them. Furthermore, the chemical sensing process itself is nonelectrical. With these sensors only very small sample volumes are needed for analysis that has the advantages of non-perturbation of samples in real-time monitoring applications.

However, optical sensing devices possess certain limitations, such as interference from ambient light, limited dynamic range, long response times, and nonreversibility. Many of these limitations can be eliminated or reduced by the use of appropriate instrumentation and sensing phases, and thus the sensor devices can be used advantageously in specific applications.

Spectroscopic Methods

UV/visible absorbance spectroscopy has been employed extensively in analytical chemistry. The basis for analysis here is the fact that the analyte, or an indicator reacting with an analyte, absorbs light; this phenomenon is mathematically described by the Beer–Lambert law eqn [4].

$$A = log\frac{I_0}{I_t} = \varepsilon c l \qquad [4]$$

In eqn [4], A is the absorbance, c is the concentration of the absorbing species, I_0 and I_t are the intensity of incident and transmitted light respectively, ε is the molar absorptivity of the species, and l is the optical pathlength of the absorbing species. Since I_t is measured in absorbance spectroscopy, the medium containing the analyte must be transparent.

However, if the medium is optically dense or even opaque, in which case the absorbance measurements would produce a high background, a technique that is complementary to absorptiometry (viz., reflectometry) can be employed for analytical measurements. Reflection takes place when light impinges on the interface of two media having different dielectric constants. Two distinct types of reflection are possible, which are referred to as 'specular' (or mirror-type) reflection and 'diffuse' reflection. Specular reflection occurs at the interface of a medium with no transmission through it and reflection is at the same angle as the incident light; in diffuse reflection the light penetrates the medium and subsequently reappears at the surface after partial absorption and multiple scattering within the medium. Specular reflection can be minimized or eliminated through appropriate sample preparation and optical engineering. The distribution of diffusely reflected light is rather homogeneous and largely independent of size and shape of the particles. The optical characteristics of diffuse reflectance are dependent on the composition of the system. Among several theoretical models that have been proposed for diffuse reflectance, the most widely used is the Kubelka–Munk theory. Here, it is assumed that the scattering layer is infinitely thick, which may be effectively the case with chemical transducers utilized in optical sensors. Optical sensors are devices based on measurement of light radiation; the reagent(s) and the supporting medium together are commonly referred to as the chemical transducer in these sensors.

The absolute value of reflectance (R) is related to the absorption coefficient (K) and the scattering coefficient (S) by eqn [5], where $F(R)$ is known as the Kubelka–Munk function.

$$F(R) = \frac{(1-R)^2}{2R} = \frac{K}{S} \qquad [5]$$

K can be expressed in terms of the molar absorptivity ε and the concentration c of the absorbing species (as in the Beer-Lambert law, eqn [4], as $K = \varepsilon c$. Thus, eqn [5] gives eqn [6] where $k = \varepsilon/S$, and S is assumed to be independent of concentration.

$$F(R) = \frac{(1-R)^2}{2R} = \frac{\varepsilon c}{S} = kc \qquad [6]$$

Eqn [6] is analogous to the Beer–Lambert relationship (eqn [4]) and holds true within a range of concentrations for solid solutions in which the absorber is adsorbed onto the surface of a scattering particle. The reflectance values (R) are generally evaluated relative to the reflectance of standard reference materials such as barium sulfate.

Emission spectroscopy is another technique that is commonly used in optical sensors. These involve basically fluorescence intensity and lifetime-based measurements.

Evanescent wave spectroscopy has been used widely in optical sensors. In these devices, the cladding material of the optical fiber is removed and replaced with a thin layer of the reagent phase in the sensor fabrication. The optical properties of the reagent phase can be monitored by means of the evanescent wave interaction. It is also possible to couple light from one optical fiber to another using the evanescent wave by bringing the cores of the two fibers closer together than the depth of penetration of the evanescent field. This enables optical information to be conveyed from one optical fiber to another.

Absorptiometry has been less used than techniques such as reflectance, fluorescence, or near-infrared in combination with optical fibers in sensors. However, a few examples are known to demonstrate the usefulness of this technique.

Chemical Transduction

In absorbance-based and other sensors, a chemical transducer is often used that interacts with an analyte to change its optical property. If analytes themselves possess optical characteristics, then that property can be directly measured using optical fibers and quantified to the concentrations of the analytes. Where a chemical transducer is employed, the reagent(s) is immobilized by physical (adsorption, entrapment, and electrostatic attraction) or chemical (covalent bond formation) procedures onto an inert and stable solid material (e.g., glass, silica gel, organic polymers). Physical procedures are simple and economical to carry out but they do not necessarily produce stable reagent matrices. Chemical procedures, produce the most firmly bound reagent, but several reaction steps are normally required in the synthesis or modification of a reagent and/or support material in order to realize stable chemical bond(s) between them.

The sensor response characteristics in chemical transducer-based sensors depend on the manner in which the analyte–reagent interaction takes place. In a simple system, where direct indicators are employed, the analyte concentrations can be correlated to the optical changes that occur in the reagent phase or in the product or both. The correlated sensor signal may also be dependent on the equilibrium constant of the analyte–reagent reaction. For example, pH can be measured by monitoring the changes in optical property of acid–base indicators. In many sensors, reversible reactions are preferred in the chemical transduction process because they can be used in continuous monitoring applications. The response time of these sensors (i.e., the time to reach the reaction equilibrium) is dependent on mass transfer processes. Irreversible reactions may also be employed in sensors, which can result in 'one-shot' devices. Although such sensors would be of limited merit, measurements with high sensitivity can be attained here. In certain cases, the reagent phases can be regenerated by the use of another chemical reaction and the sensor reused. For example, the sensor for aluminum based on immobilized EriochromecyanineR can be regenerated using fluoride ion solutions.

Indirect chemical reactions involving two or more reagents and/or reactions can be adapted as optical chemical transducers. Many enzyme-based reactions fall into this category.

Sensor Design and Instrumentation

A variety of transducer configurations have been utilized in sensors based on absorbance and other spectroscopic measurements. These fall into two categories: extrinsic and intrinsic sensors. In extrinsic sensors, the optical fiber acts only as a light guide between the light source and chemical sensor and between the sensor and the detector. In intrinsic sensors, the optical fiber becomes a part of the transducer. Both extrinsic and intrinsic sensor types can be differentiated by their sensing mechanisms, and they are used commonly in absorbance- and reflectance-based sensors. Some of the common sensor designs employed for absorbance and reflectance based sensors are shown in **Figure 2**.

Light is launched at one end of the optical fiber and is detected at the other end in absorbance type of sensor configurations (**Figures 2A–C**). Evanescent wave interactions are also employed in such measurements (**Figures 2B,C**). Immobilized reagent-based sensor designs are used whereby the reagent layer is placed at the distal end of the optical fiber (**Figures 2D,E**). Thin optical fiber or optical fiber bundles can be used in the design shown in **Figure 2D**. When single fibers are employed (**Figure 2E**), the source light and detected light travel through the same optical fiber, which can be discriminated either temporally or by wavelength with the aid of a beam splitter. In the sensor designs shown in **Figures 2D and 2E**, the reagent may also be immobilized directly at the tip of the optical fiber. In all cases using these designs, a reflector needed is to couple light back into the detection fiber, and the configuration is optimized with respect to a particular application. Both absorbance and reflectance can be measured with these designs. The detected optical signals are transduced into electrical signals, which are usually displayed in a meter.

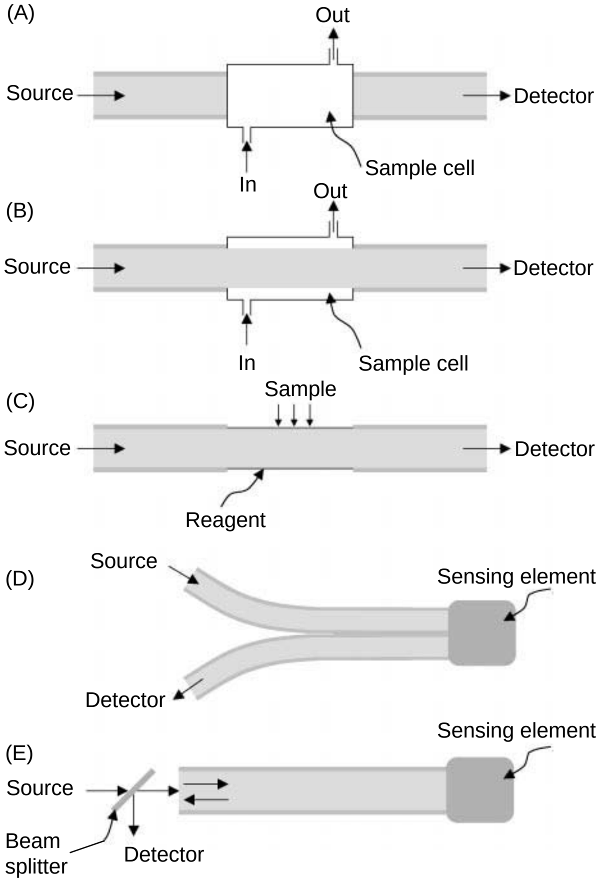

Figure 2 Optical chemical sensor designs.

In optical fiber absorptiometry or reflectometry, reference signals are employed for correcting variations in the background caused by source, detector, and also intrinsic absorption of light by optical fibers. This can be done by subtracting the blank signal from the sensor signal electronically, or by the use of two identical optical fibers, one of which is used as a reference.

Basic instrumentation employed in absorbance and reflectance based sensors requires both optical and electrical components. It involves a light source; optical fibers; a photodetector; a display; optical, electronic, and fiber optic components (modulators, amplifiers, lenses, mirrors, couplers and connectors); and monochromators or filters for selection of wavelength of light (**Figure 3**). Several types of sources, including incandescent lamps (tungsten and quartz–halogen), lasers, light-emitting diodes (LEDs), and laser diodes, have been used. Each of these types of source has its own advantages and disadvantages. The detection of light is carried out using a photocounting device, which converts optical signals into electrical signals that can be amplified electronically. Photodetectors used in optical sensors

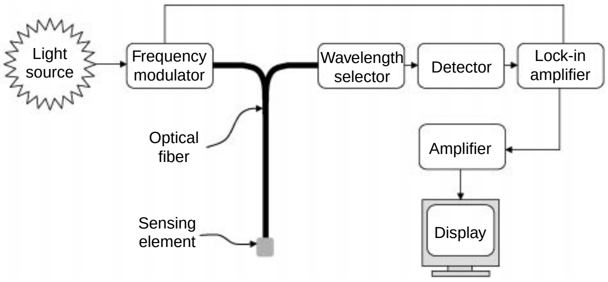

Figure 3 Schematic of Instrumentation for optical chemical sensors.

include photomultiplier tubes, p-i-n photodiodes, avalanche photodiodes, and photodiode arrays. Optical couplers and lenses are used to focus the beam of light to the optical fiber and to direct light to the detector. Filters or monochromators are used to isolate the desired wavelength for measurement. It should be emphasized that efficient coupling of light ensures the attainment of high sensitivity of sensor signals. It is also common practice to exclude extraneous light reaching the detector by suitable modulation of the light source and by synchronizing the detector to this modulation frequency so as to detect only those source signals. As mentioned earlier, instrumental drift may be eliminated or reduced by the use of a suitable referencing system.

Sensor Applications

One of the early uses of absorbance measurements for a sensor was in the technique known as oximetry (i.e., measurement of blood oxygen saturation). The measurements utilize the fact that fully oxygenated hemoglobin (i.e., oxyhemoglobin, HbO_2) and fully reduced hemoglobin have different absorption and reflectance spectra. Reflectance measurements are made at a wavelength of about 650 nm (R_1) with a reference measurement at a wavelength of about 850 nm (R_2) (isosbestic point), which are related to blood oxygen saturation expressed as $\%HbO_2$ (eqn [7]).

$$\%\mathrm{HbO}_2 = a - b\left(\frac{R_1}{R_2}\right) \quad [7]$$

In eqn [7], a and b are constants dependent on the geometry of the sensor probe.

Copper(II) ions in plating baths have been determined *in situ* using the Cu(II) absorption band at 820 nm. Two types of sensor designs have been utilized (**Figure 4**) for the determination of Cu(II) by direct absorption, resulting in inexpensive monitoring systems.

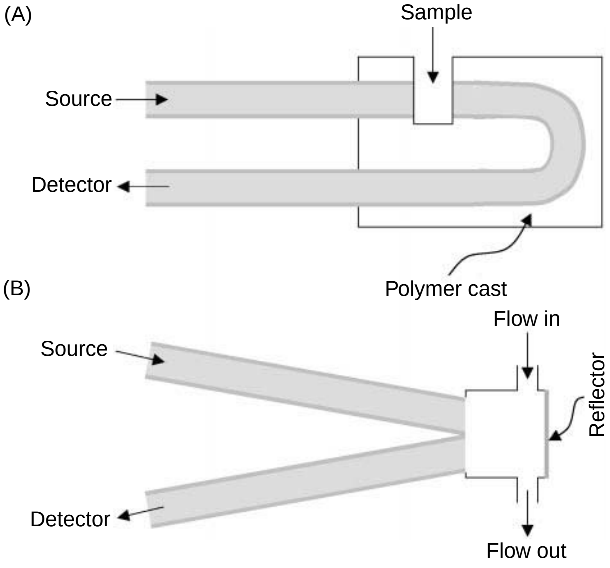

Figure 4 Optical sensor designs for measurement of Cu(II) ions.

Air pollutants have also been measured using absorbance spectroscopy and optical fibers. A nitrogen dioxide sensor has been constructed using optical fibers and measurement of absorbance of the gas at 0.5 μm using an argon-ion laser source. Real-time measurement in the lower parts per million (ppm) concentration range has been conducted at remote locations that are 20 km away. A similar device has been used for the measurement of methane at low concentrations by recording absorbance at 1.33 μm.

A number of pH sensors have been developed based on the use of hydrogen-permeable membranes that encapsulate immobilized colorimetric indicators. These devices utilize the measurement of light intensity at a particular wavelength relating to the absorption of the indicator together with a similar measurement at a reference wavelength insensitive to pH changes, such as the isosbestic point. For example, the pH sensor based on absorbance measurements of phenol red indicator immobilized by covalent bonding on to polyacrylamide microspheres of 5–10 μm diameter is reversible. The probe design is similar to that shown in **Figures 2D** and **2E** which consists of the immobilized dye together with polystyrene microspheres of about 1 μm size to scatter light. The particles are packed into an ion-permeable cellulose dialysis tube with two plastic optical fibers inserted into it. Light intensity measurements made at a wavelength of 560 nm (I_{560}) with a reference signal measurement at 600 nm (I_{600}) provide the signal S, which is the ratio I_{560}/I_{600}. S is related to pH by eqn [8], where k is an optical constant of the system, D is the optical density of the sensor (at $\lambda = 560\,\text{nm}$) when the dye is totally in the base form, and $\Delta = \text{pH} - \text{p}K_{\text{In}}$, where K_{In} is the indicator constant.

$$S = k \times 10\left[-\frac{D}{10^{-\Delta} + 1} \right] \qquad [8]$$

The shape of the response curve of S versus pH is sigmoidal with a linear range around the physiological pH range and a precision of ± 0.01 pH unit. Like other types of pH sensors (e.g. potentiometric), optical pH sensors are also sensitive to temperature changes that can be compensated using the knowledge on the variations of pK_{In} as a function of temperature.

The sensor based on reflectance measurements has a response function similar to that in eqn [8], and is given by eqn [9], where Q is given by eqn [10].

$$R = 1 + Q - \sqrt{(1 + Q)^2 - 1} \qquad [9]$$

where

$$Q = \frac{F(R_a) \times 10^{-\Delta} + F(R_b)}{10^{-\Delta} + 1} \qquad [10]$$

$F(R_{\text{a}})$ and $F(R_{\text{b}})$ are the Kubelka–Munk functions for limiting reflectances of acid and base forms of the indicator, respectively.

Sensors for many metal ions such as Pb^{2+}, Cd^{2+}, Fe^{3+}, Hg^{2+}, Al^{3+}, K^{+}, Ca^{2+}, etc., have been developed that are based on the use of immobilized metal-ion-selective reagents interfaced to optical fibers. These sensors rely on the fact that the metal ion (M) reacts with the immobilized reagent (R) to form a metal complex (MR), accompanied by either an enhancement or change of color of the immobilized reagent. This change can be correlated to metal ion concentrations. Reflectance measurements are employed in many sensors, since reagents have been immobilized on opaque materials. These reactions are pH dependent because most of the reagents used are of acid–base type indicators, and thus a strict pH control of the medium is necessary for effective sensing reactions. The metal ion–reagent interaction is also governed by the formation constant of MR (K_{f}); the higher the K_{f}, the more sensitive the measurement will be with the optical sensor. In such cases, the sensors are normally irreversible and the sensing phases may be regenerated by the use of external reactions such as the use of another complexing agent to remove the metal from MR or the use of a different pH of the medium, usually lower than that used in the formation of MR. Sensors for anions such as F^{-}, S^{2-}, CN^{-}, etc., have been developed similarly using suitable immobilized reagents and spectroscopic techniques.

An optical sensor that has been developed for the determination of pesticides involves the use of more than one type of transduction system. Here, an enzyme and a pH indicator are co-immobilized on suitable polymeric material. In the absence of pesticides, the reaction of the immobilized enzyme with its specific substrate will be accompanied by a change in pH that can then be measured by the pH sensor using absorbance or reflectance. In the presence of the pesticides, however, the substrate/immobilized enzyme reaction is inhibited and the degree of inhibition, transduced in the pH sensor, can be correlated to the pesticide concentration. Many toxic metal ions also behave in a manner similar to pesticides, and hence optical sensors for metal ions have been developed based on this sensing principle. Analytical samples are prepared in a buffered medium for each sensor.

Many sensors for gases and vapours such as ammonia, carbon dioxide, humidity, hydrogen cyanide, hydrogen sulfide, etc., have been that developed are also based on the use of analyte-specific immobilized reagents and measurements of absorbance or reflectance. In some of the sensor designs used (**Figures 2D,E**), a membrane is employed to contain the sensing reagent and this, sometimes, aids the selectivity of the sensing device to a particular gas analyte.

Conclusions

As described above, many types of optical sensor designs have been studied for a variety of analytes using different types of tranduction reactions in the development of absorbance- and reflectance-based sensors. A current trend in this development is to construct multianalyte sensing systems based on the use of single or a few reagent phases, together with the employment of appropriate signal processing techniques such as pattern recognition and artificial neural networks. Most of the optical sensors described above can be designed for use in oceanographic measurements in the analysis of heavy metal ions, dissolved gases and other species.

Applications of absorbance based sensors to ocean sciences have yet to demonstrate its potential. Most of the applications published describe ‘proof-of-concept’ studies with the sensors and with very little or no practical demonstration in such areas. Some of the recent studies in this area have been focused on the monitoring of dissolved CO_2 in seawater using absorbance and fluorescence measurements with transducers that incorporate pH sensitive indicators. The use of infrared absorption measured through the evanescent waves in optical fibers for sub-sea monitoring of organic compounds (at parts per million levels) has also been demonstrated. However, there is substantial interest in new sensors for oceanographic applications including monitoring of nutrients and pollutants, and optical sensors have great potential here. The devices are clearly attractive in concept and require expertise from several scientific disciplines including analytical chemistry, polymer chemistry, environmental chemistry, fiber-optics and opto-electronics.

See also

Fluorometry for Chemical Sensing. Inherent Optical Properties and Irradiance. Wet Chemical Analysers.

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ABYSSAL CURRENTS

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Introduction

Historically the term ‘abyss’ characterizes the dark, apparently bottomless ocean under extreme static pressure far beyond coastal and shelf areas. Today this ancient definition remains still rather unfocused in earth sciences. Geographers, marine biologists, and geologists use abyss for deep-sea regions with water depths exceeding 1000 or 4000 m. In physical oceanography a widely accepted definition of the abyss denotes the water column that ranges from the base of the main thermocline down to the seabed.

The main thermocline itself – occasionally also called warm-water sphere – extends laterally between the polar frontal zones of both hemispheres. In contrast to deep strata the surface exposition of the thermocline allows the direct exchange of heat, substances, and kinetic energy with the atmosphere. This wind-driven part of the water column and its fluctuations are consequently an immediate subject of weather and climatic conditions. The base of the thermocline at about 1000–1200 m represents the lower boundary of the warm-water sphere with temperatures well $>5°C$.

The abyss or cold-water sphere below, is clearly colder. Below 2000 m potential temperatures $<4°C$ are found virtually everywhere. Below 4000 m values of 0–2°C are more characteristic. Until the advent of modern self-recording instrumentation abyssal currents were believed to be very slow ($<2\,cm\,s^{-1}$) and negligible in comparison with rather vigorous and variable surface currents (sometimes $>100\,cm\,s^{-1}$). Only subsurface passages in rises and ridges that subdivide ocean basins (**Figure 1**) seemed to allow for more energetic deep interior currents funneling through gaps and channels.

Until a few decades ago practically all knowledge about abyssal currents had to be inferred from the