6 NIR
21st-Century Innovations

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6.1 A BRIEF HISTORY OF NIR SPECTROSCOPY (NIRS)

It is difficult to dispute the important role industry has played in the development of the modern-day near-infrared (NIR) spectrometer. The brief history on the development of NIRS that follows is provided to give an insight into the three-and-a-half centuries of innovation that have led to the development of the 21st-century NIRS.

6.1.1 The Development of the Spectroscope [1666–1896]

There is a long-standing and well-documented history of innovation related to spectroscopy. While the first known use of the word “spectroscopy” occurred in 1882 during a lecture by Arthur Schuster at the Royal Institution in London, England, the origins of spectroscopy predate Schuster’s lecture by at least 226 years.

The first well-documented and systematic observation and measurement of electromagnetic radiation dates to 1666, when Sir Isaac Newton recorded the separation of sunlight into bands of colors using the sun, a small circular aperture, and a prism. In a 1672 publication entitled “A New Theory about Light and Colours” [1], Newton coined the word “spectrum” [def. Latin: apparition] to describe the colored images that were hidden within the light, which danced around like ghosts with the movement of his prism.

While one could argue that Newton’s apparatus was the first spectroscope, having a source (the sun), a detector (the human eye), and a dispersive element (a prism), there are those that argue since he didn’t have a slit or bandwidth limiter, it wasn’t a spectroscope. In fact, it wasn’t until 1802, close to a century and a half later, that William H. Wollaston, through the introduction of a slit, observed the first-line spectrum consisting of discrete lines emanating from the sun [2]. As a matter of principle, one must ask, should Wollaston, the creator of the first monochromator reduced to practice, be credited with the development of the spectroscope?

Unfortunately, Wollaston did not recognize the significance of his innovation nor the observations he made using it. Twelve years later, credit for the discovery of the sun’s spectral lines went to Joseph von Fraunhofer, who it is said independently conceptualized the same innovation as Wollaston in 1814 and used it to catalog 576 narrow, dark spectral lines of the sun [3].

It is important to note that Fraunhofer’s cataloging of the lines that still bear his name today, the “Fraunhofer lines”, would never have been possible without another innovation, the diffraction grating, which he made from winding fine silver wire on a small frame.

David Rittenhouse of Philadelphia is credited with the development of the first grating [4], which was nothing more than hairs strung between two fine-pitched screws.

The world’s first spectroscope was invented in 1859, by a physicist, Gustav Robert Kirchhoff, and a chemist, Robert Wilhelm Bunsen, whose innovation led to the development of the first optical source, the Bunsen burner. Oddly enough, the Bunsen burner produced very little “spectrum”, but this is what made it ideal for Kirchhoff and Bunsen’s research, which involved the measurement of spectral emission lines from common salts that possessed “the power of developing peculiar bright lines in the spectrum of a flame into which they were introduced” [5]. Some of the first data recorded using their spectrograph is shown in Figure 6.1 to the left of the solar spectrum first observed by Fraunhofer. In principle, it is important to note that Bunsen’s burner wasn’t the source which powered the first spectroscope. The source was the flame-ionized elements that were introduced to the Bunsen burner that enabled the first spectrograph.

Using their spectroscope, Kirchhoff and Bunsen set out to acquire data that resulted in a groundbreaking publication entitled “On Chemical Analysis by Spectrum-Observations” [6]. This seminal body of work not only laid the ground works for modern-day spectroscopy but also allowed the 19th-century scientists to explain Fraunhofer’s lines – an explanation that had eluded them for half a century.

In order to perfect Kirchhoff and Bunsen’s design as shown in Figure 6.2, they collaborated with an optician Carl August von Steinheil to engineer a commercial spectroscope, which allowed...
FIGURE 6.1 An illustration of line spectra taken between 1859 and 1860 by a physicist, Gustav Robert Kirchhoff, and a chemist, Robert Wilhelm Bunsen, of six high-purity salts annexed besides the solar spectrum for comparison. Clearly shown are the solar “dark lines” first discovered by William H. Wollaston in 1802 and later cataloged by Joseph von Fraunhofer in 1814. Today, the dark lines are still referred to as “Fraunhofer lines”, a testament to the importance of the early 19th-century discovery.

FIGURE 6.2 An illustration of the apparatus used by Kirchhoff and Bunsen between 1859 and 1860 to measure the line spectra of various high-purity salts. With the addition of the Bunsen burner as a source, it is recognized as the world’s first spectroscope. The above spectroscope was the result of multiple innovations dating back to 1666 and is still being manufactured today.
contemporaries of Kirchhoff and Bunsen to discover helium on the Sun 27 years before the element was discovered on the Earth and prior to the turn of the 20th century. Steinheil and Söhne’s spectroscope as shown in Figure 6.3 latter led to the characterization and identification of several noble gases and elements that would eventually be predicted by Mendeleev’s periodic law.

6.1.2 The Discovery of NIR Radiation

It is important to note that the early spectroscope relied on the human eye for the detection of spectral lines. It wasn’t until 1800 that William Herschel noticed light passing through colored filters transferred different amounts of heat to each filter. In order to explore this phenomenon, Herschel set out to determine the “heat” energy of colors by placing a thermometer across multiple points within the sun’s color spectrum. This led to an unexpected discovery. Specifically, Herschel noticed that the largest temperatures existed beyond red, where there was no visible light that could be seen by the human eye. He went on to determine that these “heat rays” could be transmitted, reflected, and absorbed just like visible light. This portion of the electromagnetic spectrum became known as infrared (“below red”) radiation.

6.1.3 The First Spectrophotometer

It wasn’t until Leopoldo Nobili and Macedonio Melloni exploited the thermoelectric effect discovered in 1822 by Thomas Seebeck to develop the first thermomultiplier that the modern-day spectroscope began to take shape. Their seminal work ultimately led Arnold O. Beckman to conceptualize the 20th-century spectrophotometer in 1939. The effort to commercialize Beckman’s innovation took place at National Technologies Laboratories and was led by Howard H. Cary.

It is said that Bechman’s DU Spectrophotometer reduced a chemical analysis process from weeks to minutes and improved the accuracy of the result from 25% to 99.99%. Within 12 months of the release of the Model A spectrophotometer, which utilized a glass prism, the Company released a Model B which utilized a quartz prism for improved UV wavelength performance, and later, a Model C with improved resolution. In 1941, it released the Model DU, which effectively remained unchanged until 1976 when it was discontinued. Bruce Merrifield, a Nobel Laureate who received the Nobel Prize for Chemistry in 1984 for the development of an ingenious method for synthesizing chains of amino acids or polypeptides in any predetermined order, once said that
the spectrophotometer was “probably the most important instrument ever developed towards the advancement of bioscience”.

In 1952, Karl Norris and Wade Brant, using a modified version of the Beckman DU, set out to develop a method for detecting blood in white-shell eggs for the US Department of Agriculture to grade and ultimately sort eggs [7]. While the NIR spectrum generated by the Beckman showed an early promise for the development of a real-time sorting method, it was later found to have no correlation with egg quality. It would be close to a decade later that Karl Norris would revisit the NIR region to measure the moisture content in seeds using a Perkin-Elmer UV/Vis/NIR scanning spectrophotometer [8]. Unfortunately, the instrument he utilized struggled with highly scattering media, so Karl proceeded to develop a custom spectrometer for analyzing moisture in grains and oilseeds [9]. It can be argued that the field of NIR spectroscopy (NIRS) truly took root after Norris published the results of these efforts.

6.1.4 Twentieth-Century Innovations

Over the course of the next 35 years, NIR instrumentation evolved. The advancements made during this time period are thoroughly covered in this chapter on traditional instrumentation. While extremely important to the field of NIRS and still prevalent throughout industry, filter, scanning monochromator, Fourier transform (FT), acousto-optic tunable filter (AOTF), and diode array-based NIR platforms are all considered the 20th-century innovations and will not be reviewed within this chapter.

6.1.5 Twenty-First-Century Innovations

Typically, technological advancements are driven by either necessity or curiosity. While Plato believed that necessity was the mother of all invention, there are those that would argue it is curiosity, which has led to some of the most significant innovations in history. Steve Jobs agreed, for he once said, “Much of what I stumbled into by following my curiosity and intuition turned out to be priceless later on”. Why curiosity? Walt Disney may have hit the nail on the head when he stated, “When you’re curious, you find lots of interesting things to do”. Even Albert Einstein believed in the power of curiosity, for he once said “I have no special talents. I am only passionately curious”.

When considering the historical development of the NIR spectrometer chronicled above, it is fair to say that innovations related to NIR instrumentation have been proportionately driven by both forces, especially when one examines the developments of the past 15 years, which have led to the creation of several MEMS-based NIR platforms.

To appreciate the current state of NIRS as it relates to these new platforms, a brief review of (1) the motivation behind the past 15 years of development and (2) optical MEMS is mandated.

6.1.5.1 The Driving Force for the 21st-Century Innovations

The world is currently experiencing the Fourth Industrial Revolution, appropriately titled Industry 4.0. It is the current automation trend in manufacturing that encompasses cyber-physical systems, the Internet of Things (IoT), and cloud computing, which are the critical attributes of any “smart factory”.

Smart factories allow for a real-time process optimization, which is critical for enabling the economic viability of many consumer products. Low yields, poor quality, and reduced throughput limit the commercial practicality of many products.

The smart factory movement is so pervasive throughout industry that a special term, Industrial IoT (I₂OT), has been coined to describe the market servicing the requirements of today’s smart factories. Last year, I₂OT garnered over $200B of the $1.2T in investment that poured into the IoT sector, making I₂OT the largest segment of the IoT sector to date.
Since the mid-1980s, the NIR spectrometer has been a mainstay analytical tool for the process analytical chemist who is responsible for converting raw materials into finished goods. In support of this statement, multiple sources estimated the NIRS market is expanding at a compound annual growth rate (CAGR) approaching 10%, second only to Raman spectroscopy. While this growth rate is exciting to all instrument manufacturers, what is more exciting to NIR spectrometer manufacturers is the fact that these same sources claim that only 25% of the NIR analyzers in the marketplace are being utilized in a real-time industrial setting. In other words, over 75% of the NIR technology in the marketplace is being utilized either at-line, de-coupled from the live process, or offline within a laboratory environment, which is the antithesis of a smart factory solution. This dichotomy has created a real and apparent opportunity for NIR instrument manufacturers and has clearly been the primary driver spurring the development of MEMS-based 21st-century NIR platforms.

While the size, expense, and complexity of exiting in-line industrial solutions have received a valid criticism from industry experts, the primary factors limiting the deployment of NIR within industrial automation strategies have been twofold:

1. Existing NIR solutions require a significant manual interaction (i.e., labor) to maintain the technology in an industrial in-line or at-line setting,
2. Manufactures struggle to scale successful NIR automation strategies within their manufacturing environments due primarily to the instrument-to-instrument variability.

While industry has been investing aggressively in connected devices, cloud computing, advanced analytics, and machine learning, in parallel, NIR instrument manufacturers have invested in smaller, lower-cost, more robust, but perhaps most importantly, ultra-reliable IoT NIR platforms. Why? These manufacturers understand that at the core of an IoT solution is the connected device (i.e., the NIR spectrometer). Further, they understand that the reliability of the device is paramount to the success of any automation strategy.

### 6.1.5.2 Optical MEMS and NIR (MOEMS-NIR)

In 1965, Nathanson and Wickstrom developed what many consider to be the first MEMS device, a frequency-selective micro-electromechanical system (Figure 6.4) [10]. In Nathanson’s era, the technology was known to only a select few, but today, over half a century later, the acronym evokes visions of extremely small machines even in the minds of the common, nontechnical, consumer. What the common consumer might not know is that to be considered as a MEMS device, the microfabricated device must have both an electrical and a mechanical or nonelectrical component.

In 1993, Dr. M. Edward Motamedi coined a new term, “MOEMS” (micro-optical electromechanical systems), which he introduced during an invited talk at the SPIE Critical Reviews of Optical Science and Technology conference in San Diego (Figure 6.5) [11]. He described MOEMS as an integrated micro-optical electromechanical system.

For over a decade, Texas Instruments (TI) had been working on the development of micromirror devices. During his presentation, Dr. Motamedi reported on a laser scanner that utilized a programmable or adaptive optical element: a micromirror that would one day enable a plethora of adaptive optical applications.

The technology Dr. Motamedi spoke of would later become known as a “digital micromirror device” (DMD) and find commercial success in the consumer electronics industry, where it served as the core technology for the rear-projection TV (RPTV) market and later, the digital projector market.

Due to the breath of applications that were being developed around the DMD at the turn of the 21st century, TI appropriately branded the technology as the Digital Light Processor (DLP™), and it is fair to state that the DLP ushered in a new era for optical technologies. The era of adaptive optical systems, or more apropos, is the era of MOEMS.
FIGURE 6.4  Illustration of a frequency-selective MEMS device that is considered the first MEMS device every patent. The patent was filed on June 18, 1965.

FIGURE 6.5  Reproduction of a figure first introduced by M. Edward Motamedi at an SPIE conference in San Deigo in 1993.
Of the seven 21st-century MOEMS-based NIR platforms chronicled in Figure 6.6, it is interesting to note that the platforms leverage similar source and detector technologies. Where they differ, and where the true innovation lies, is within each platform’s monochromator. Consequently, the following reviews will focus on the monochromators in each platform and exclude any discussion of integration of the monochromator with either a source or a detector, unless the innovation is intimately coupled with one of these elements. Table 6.1 summarizes each platform’s monochromator innovation.

As shown in Figure 6.6, there was a flurry of activity in the mid-2000s that resulted in the commercialization of four MOEMS-based NIR platforms, followed by a 5-year draught of MOEMS-based NIRS innovations. Again in 2012, another wave of innovations resulted in the release of two additional inventions, followed by a third in 2014. Of particular note regarding Figure 6.6 is the fact that there have not been any notable commercial innovations since 2014. Section 6.3 will attempt to address the question of whether there appears to be another wave of technology innovations in the pipeline, which shows a commercial promise for NIRS.

In our estimation, the NIR industry CAGR won’t expand by 9%–12%, as predicted by multiple industry analyst, it is in a position to expand much faster due to innovations spurred by the latest industrial revolution, Industry 4.0. Further, it is very probable that the innovations being development today, behind closed doors, will lead to a future in which NIRS is not only a mainstay technology in industry but also a pervasive technology within everyday consumer products.

### 6.2 TWENTY-FIRST-CENTURY PLATFORMS

Since each technology platform is unique, the reviews are ordered solely by the chronological date when the technology first entered the marketplace and by no other weighting. In reading the reviews that follow, one should come to appreciate that there is not a clear “leading” technology platform. It
should also be noted that each platform could have multiple commercial options, and where neces-
sary, each option will be presented.

For many technologists, this is a hard concept to grasp. How can there not be a leading technology
platform? For the applied spectroscopist, it is easy to understand how two or more competing tech-
nologies can exist in the marketplace at the same time with no clear leader. The spectroscopist
understands that commercial and/or industrial NIR applications each have their own unique set of
application-specific requirements that dictate whether a platform technology is a viable solution
to a particular application. Specifically, the spectroscopist must review the technology platform's
technical attributes or specifications, and decide which mix of attributes are a best match to their
application requirements. While this sounds difficult and potentially subjective, there are six key
technology attributes (KTAs) that can be used to assess the viability of a given platform for each
application.

The KTAs are as follows:

<table>
<thead>
<tr>
<th>Integrated Drift Compensation (IDC)</th>
<th>Unit Price (Offline/On-line):</th>
</tr>
</thead>
<tbody>
<tr>
<td>Environmental Sensitivity:</td>
<td>Dynamic Range/SNR:</td>
</tr>
<tr>
<td>Instrument Stability:</td>
<td>Spectral Range/Resolution:</td>
</tr>
</tbody>
</table>

### 6.2.1 Key Technology Attributes (KTAs)

In looking at the KTAs above, many may argue that scan rate is missing or is a critical factor in
evaluating a technology platform and they would be correct in the instances where the fast spectral
acquisition is a requirement. The screening of individual tablets produced by a tablet press in the
pharmaceutical industry would be a perfect example. Unfortunately, the scan speed is a function of
spectral range, spectral resolution, and even signal-to-noise ratio (SNR). Narrow the range, decrease
the resolution, and/or degrade the SNR requirement, and every platform can scan faster. Further,
while platforms exist that are capable of scanning even large ranges at relatively high resolution very
quickly, many of these platforms require the acquisition and subsequent averaging of multiple scans
to deliver practical SNRs. For these reasons, the scan speed has been ignored as a practical KTA for
accessing the viability of a particular platform for a given application.

To enable the reader to assess the relative strengths and potential weaknesses of a technology
platform as it relates to their specific application requirements, general values of Yes/No or Low/
Average/High have been assigned to each KTA when applicable or known.

Further, due to the plethora of instrument configurations available, even within a given platform
technology, and the impact even small changes to the spectral configuration can have on the KTAs,
the authors have elected to assign KTAs for each platform to the instrument configuration that
represents the largest spectral range the platform is capable of achieving.

A description of each KTA is as follows:

**IDC**

All NIR platforms are susceptible to source, mechanical, and electronic drift over time.
Spectroscopists have developed techniques, such as the automated sampling of a
reference material at periodic intervals to compensate for instrument drift; however, it is
possible for a platform to include the integrated drift compensation (IDC), thereby
eliminating or significantly delaying the need for automated drift correction strategies.
This can also be called “real-time or dynamic referencing”.

(Continued)
Environmental sensitivity

All NIR platforms exhibit some degree of sensitivity to changes in environmental parameters. Specifically, fluctuations in temperature, pressure, and humidity can lead to measurement inaccuracy if uncompensated. With the advent of MOEMS-based NIR platforms, it is common to encounter applications that require battery-operated systems. With batteries, the power level (i.e., the charge level) of the batteries and power quality will influence the measurement accuracy. With non-battery-powered devices, power quality impacts the measurement accuracy.

Since manufacturers have developed electronic strategies for the management of both power quality and level, and a device’s sensitivity to humidity and pressure is typically a function of industrial packaging, the end user’s calibration strategy, and the sample interface, this KTA only considers a platform’s sensitivity to environmental temperature changes. Platforms with a LOW rating exhibit minimal changes in wavelength, amplitude response, or SNR over a significant temperature range. An AVERAGE rating is given to platforms with moderate temperature sensitivities that can be corrected for through advanced calibration strategies. Platforms with a HIGH rating must be held at a constant temperature due to their inability to tolerate temperature changes.

Instrument stability

Instrument stability could also be referred to as “ruggedness or robustness” towards mechanical shock and vibration. Specifically, if a device was dropped and experienced a large, instantaneous G-force, would it still be operational and if so, would the calibration still be valid? Further, if the device was installed in a high-vibration environment, can it remain calibrated and/or operable? A platform with HIGH instrument stability has both high shock and high-vibration tolerance. A platform with AVERAGE stability would need recalibration due to abnormally high shock or vibration. A LOW rating indicates that the device would be rendered inoperable due to any reasonable shock or vibration.

Unit price (offline/in-line)

Since one could argue that each of the 21st-century innovations reviewed below was developed with price point in mind, one would think that the unit price rating for each platform would be low, but this isn’t necessarily the case as many of the platforms have expensive packaging and/or provisioning requirements if the intended use is for in-line, real-time, autonomous monitoring. Therefore, a unit price rating will be given for both offline and in-line solutions.

Dynamic range/SNR

Traditional spectroscopists define dynamic range as the difference between the largest photon flux an instrument can measure and the smallest photon flux the instrument can measure. With modern (e.g., digital) NIR spectrometers, dynamic range is defined by the ceiling of a spectrometer’s analog-to-digital converter (ADC) and the ADC’s floor, which is defined by the system’s dark current. Signal-to-noise ratio (SNR) represents an instrument’s ability to resolve small changes in flux within the system’s operable dynamic range. SNR is calculated by dividing the mean signal level by the RMS noise level. Many manufacturers will measure the instantaneous noise on the ADC in a high-flux environment to calculate SNR. However, true SNR is measured by calculating the full-spectrum shot-to-shot RMS noise across dozens of spectra. While an SNR value can be calculated for each spectral channel within the instrument’s spectral range, it is common to specify SNR for the spectral channel with the highest throughput or spectral amplitude. It is also common to specify the wavelength for which the SNR is stated.

Typically, for a given instrument configuration, an AVERAGE NIR spectrometer can measure between two and three decades of photon flux and has a true SNR between 1000 and 7000.

Spectral range/resolution

As a rule, spectral range and resolution are closely coupled parameters. While it is rare for a platform to have both high spectral range and high resolution, it is possible. For the purposes of this review, any system with a range approaching one optical order (e.g., 1250–2500 nm) will be rated high. A range less than 500 nm will be rated low, and anything in between will be considered average. With respect to resolution, for the purposes of this review, resolution refers to full-width half-max (FWHM) optical resolution and not wavelength precision of the platform’s monochromator. Any platform with a FWHM resolution exceeding 0.5% of the system’s spectral range will be rated high. A platform with a FWHM resolution worse than 2% will be rated low, and anything in between will be rated average.
6.2.2 The Utility of KTAs

What follows are two examples that should serve to highlight the utility of the KTA rating system as a tool for selecting the right technology platform for a given application.

Example 1

If a client has a price-sensitive application in a relatively nontechnical field where the technology is being utilized by unskilled users to classify, not characterize, a substance in a nonhostile environment (i.e., a first responder required to check an unknown substance for drug classification), then a platform that has the following KTA ratings is a viable candidate since classification, as opposed to quantification, is all that would be required. Further, the sampling is performed manually, which allows for the frequent background sampling to correct for calibration drift or offsets due to changing environmental parameters.

Example 2

On the contrary, if the application required the characterization of the actual concentration of the unknown drug, then it is likely a different platform with the following attributes would be required.

6.2.3 Multivariate Optical Computing (MOC) Platform
The MOC has the distinguished privilege of being not only the first 21st-century NIRS platform reviewed below but also the most controversial. In fact, some would argue that the MOC: (1) isn’t a 21st-century platform since it was first conceptualized by Dr. Michael Myrick at the University of South Carolina in the late 1990s [12] and (2) doesn’t classify as a MOEMS technology. In response to the first observation, it is important to note that the MOC was not commercialized until 2005, and in response to the latter, while its true the first iterations of the technology utilized bulk optical elements and large mechanical systems, the latter iterations integrated optically encoded filters fabricated using MEMS fabrication techniques and micro-optical electromechanical elements and represent potentially the smallest and most rugged NIRS platforms available on the market today.

The downside of the MOC is it is a fit-for-purpose solution and not a traditional NIRS platform that allows for the acquisition of an NIR spectrum. Specifically, it is a compressed sensing technique that utilizes an a priori selection of discrete wavelengths within the NIR spectrum that have been encoded into a multivariate optical element (MOE) to measure a singular analyte or parameter of interest. Specifically, an MOC with a single MOE is not a multianalyte or multiparameter NIRS platform. Further, the nonrecurring engineering (NRE) expense required to develop the a priori knowledge required to calculate and ultimately encode the MOE within the MOC for the measurement of a particular parameter may outweigh the return on investment for some niche or specialty low-volume applications.

The upside of the MOC is that with a careful application development work prior to the construction of an MOE, the MOC can measure a single analyte or parameter of interest very well. Further, due to the size of the MOE, it’s possible to integrate multiple MOEs into a single MOC for the measurement of multiple parameters. In fact, the MOC as shown in Figure 6.7 includes 20 MOEs and their corresponding reference filters, each of which has been designed to measure a particular chemical or physical parameter of interest.

To understand the MOC, one must acquire a detailed understanding of the MOE that sits at the core of the MOC. Further, since MOC predictions are radiometric, it is important to note that an MOC requires the acquisition of transmission or reflectance spectra, as opposed to absorbance spectra.

6.2.3.1 The MOE
The MOE is a multilayer optical encoded interference filter designed to generate a unique signal response on a single-element photodetector, which is predicative of the parameter of interest.
Specifically, the MOE circumvents the post-chemometric analysis of a traditional NIRS platform by encoding a regression vector into an optical transmission filter, the MOE, which not only senses but optically computes in a simultaneous step, the parameter of interest.

With any MOE, the spectral response related to a variation in a parameter of interest is typically designed using alternating layers of different materials to emulate a regression vector that correlates the variation with the response function. The classical regression of the dot product with the regression vector and the sample spectrum is then performed by placing the filter directly in the optical path [15], as shown in Figure 6.8, which illustrates a simple measurement arrangement [14] of a basic MOC. The convolved output intensity of the source, sample, and MOE measured by a detector can then be directly related to a physical or chemical material property. Since a spectrum is never collected, it can be argued that an MOC is not a spectroscopic technique, but rather falls into the category of “photometric analysis”. That is not the opinion of the authors who consider MOC as one of seven commercially viable 21st-century NIRS innovations.

It must be noted that the KPAs of spectral range and resolution are not applicable (NA) to MOCs. Further, while it is possible to define an SNR for the MOC, it is more accurate to discuss the standard error of prediction (SEP), which is a chemometric term as opposed to an instrument specification, when defining the accuracy of an MOC. SEP is determined experimentally by testing a validation set of samples with known chemical or physical properties derived from standard laboratory methods against the MOC measurement, and thus, SEP can never be better than the accuracy of the standard method [14].

There are currently two approaches for generating an MOE:

1. Principle component regression (PCR);
2. Vector relaxation, which requires a correlated reference measurement.

Effectively, PCR allows for the creation of a regression vector that serves as the basis function for generating an MOE. Scaling of the basis function is arbitrary but should yield realistic transmittance/reflectance values. The second method, vector relaxation, is a regressive method. A random number generator produces a random multilayer structure, which serves as the starting point for iterating a proprietary algorithm that seeks an optimal MOE [16,17]. Interestingly, while the two approaches yield very different MOE configurations, the resulting predictions and SEPs are typically comparable. However, since the vector relaxation method results in a thinner filter, it appears to have become the preferred method for generating MOEs.
6.2.3.2  MOC KTA Summary

For a detailed review of MOCs, the reader is encouraged to read the references listed within Section 6.6.1 and prior to 2019, would have been referred to Cirtemo for more information on MOEs. However, Cirtemo was acquired by Thorlabs in May of 2019. This new division of Thorlabs still operates out of South Carolina under the name Thorlabs Spectral Works, and information on MOEs can be found on Thorlabs website.

When considering the KTAs for MOCs, the high instrument stability and low environmental sensitivity are both critical attributes for harsh environments, such as the downhole oil and gas industry, where MOCs found early commercial success.

While the unit price is listed as average for both offline and in-line implementations, this was a difficult assessment to make, as the unit price is extremely sensitive to sales volume. In fact, the high NRE associated with the design of the MOE potentially has an insignificant impact on the unit price for high-volume applications. Unfortunately, it limits the commercial viability for low-volume applications, unless the value of the information outweighs the cost of development. Again, the oil and gas industry is a good example. There aren’t a lot of downhole oil and gas tools operating in the market yet a downhole MOC still makes sense. Even when you consider the low SNR or relatively high SEPs common with MOCs, they still make sense for the downhole market. This is because the value of the information, even low-resolution information from a potentially expensive sensor, is used to develop oil field exploration strategies.

To summarize, if you are working in a harsh environment where the value of information is at a premium, or you have a high-volume application with low quantitative or simply qualitative requirements, an MOC-based NIRS may be the ideal platform for you.

6.2.4  Grating Light Valve (GLV) Platform

Silicon Light Machines (SLM), in Sunnyvale CA, markets and licenses Grating Light Valve (GLV™) technology [18]. The GLV is a “microprojection” technology that utilizes a programmable diffraction grating. It competes with other light valve technologies such as Digital Light Processing (DLP™) chip and liquid crystal on silicon (LCoS) chip for implementation in video projector technologies. In comparing the GLV to DLP or LCOS, it is interesting to note that the actuation of the GLV can occur in ~20 ns, which is a million times faster than LCoS, and a thousand times faster than the DLP. This high speed is attributed to the low mass and extremely small submicron displacement requirements of the GLV ribbons. Effectively, the ribbons are massless due to their small size, and since there is no physical contact between moving elements, GLV lifespans exceeding 210 billion switching cycles have been documented. At standard video rates, this translates into a 15-year lifespan under continuous operation. With mil-spec shock survivability exceeding 30,000 g and equally impressive vibration results, the GLV is an extremely robust platform technology.

6.2.4.1  The GLV

The GLV, a dynamic diffraction grating that utilizes an array of extremely small parallel rows of highly reflective microribbons suspended above an air gap over a silicon substrate, was originally conceptualized by Dr. David M. Bloom and his colleagues in the electrical engineering department at Stanford University [19]. Bloom’s array of analog-controlled reflective ribbons that when
actuated, in a concerted fashion, created a series of diffractive elements or pixels led Dr. Bloom, in 1994, to found Silicon Light Machines to commercialize the GLV in the projection industry. Within 2 years, a group from MIT, in 1996, independently developed an alternative GLV chip, in collaboration with the Defense Advanced Research Projects Agency (DARPA). The goal of the project was to develop a GLV-based device called the “polychromator”, a programmable diffraction grating that could generate synthetic chemical spectra for use as a component in a mid-infrared correlation spectrometer.

The GLV chip consists of an array of flat-parallel mirror elements. Each array of six elements represents an analog-controlled diffractive pixel capable of attenuating incident light. In the actuated state, light is diffracted if the mirror displacement is exactly one-quarter the wavelength of the incident light. In the unactuated state, light striking the mirror elements at normal incidence is completely reflected. The precise displacement to create diffraction depends on the wavelength and bandwidth of the light within each diffractive pixel, and it is important to note that generating the precise displacement is only possible due to the analog nature of the GLV. Another element of the polychromator that proved critical to the commercialization of polychromator-based device was the double-layer interlaced architecture of static and dynamic elements, where the static upper mirror elements remained flat and parallel to the substrate during actuation, enabling each diffractive pixel to induce a precise optical phase shift with significantly improved diffraction characteristics. Effectively, an incident narrow-band optical beam of light could be turned fully-on and fully-off or attenuated to any intensity in between in a consistent and reliable manner.

The DARPA-funded polychromator was first commercialized in 2000 by Polychromix for NIR and C-band telecommunications applications. The optical design was very similar to a dispersive spectrometer in that light entered from a fiber was collimated and then hit a fixed diffraction grating splitting the light along one axis into different wavelengths. Subsequently, the light was passed through intermediate optics and was focused on the polychromator, a diffractive MOEMS chip, at the near-normal incidence. The reflected light passed back through the system and was focused to another fiber. All diffracted light was trapped and prevented from traveling back through the system. Along the length of the MOEMS chip, the diffractive elements were grouped into 100 six-element diffractive pixels, each representing a discrete optical channel within the C-band. Each channel was under analog control, enabling a variable transmission, including a complete attenuation. This allowed the telecommunications industry to correct the nonuniform gain curves of erbium-doped fiber amplifiers used to boost signals in long-haul communication networks. Effectively, with analog control of each optical channel, every channel within the C-band could be acted upon so that all channels entering the communication network processed the same relative intensity.

With the knowledge above, it is easy to envision the basic system architecture of the first GLV or polychromator-based spectrometer. All Polychromix, the first company to commercialize a GLV-based spectrometer, had to do was to replace the output fiber of the C-band device with a detector. Further, it is easy to envision the system acting as a traditional monochromator that individually scans across a given wavelength range by addressing each channel independently in series, to acquire an optical spectrum. However, the polychromator was capable of much more than raster scanning through optical spectrum. It wasn’t like traditional monochromators due to the fact that the GLV had the ability to dynamically address all 100 individual optical channels simultaneously. Polychromix took an advantage of this capability to enable what later came to be known as “Digital Transform Spectroscopy” (DTS™).

Given the programmability and speed of the MEMS chip, it was possible to implement a technique similar to a Hadamard transform. Performing the Hadamard transform of this signal resulted in the calculated spectrum as shown in the bottom of the figure. The implementation required less than 10 ms for each mask such that the overall time to acquire a spectrum was ~1 second. At the time, the only competitor that could compete with the speed of Polychromix’s DTS™ spectrometer was the diode array spectrometer; however, diode arrays for the NIR spectrum were...
significantly more expensive than the single-element photodiodes required by the polychromator. But more importantly, the diode array spectrometer struggled with the elimination of stray light and temperature-sensitive dark current.

### 6.2.4.2 GLV KTA Summary

Since the DTSTM spectrometer was capable of individually modulating each diffractive pixel, which represents an individual optical channel within the dispersed optical path, the DTSTM spectrometer could effectively reject stray light and eliminate dark current effects on the measured optical spectrum. Its insensitivity to stray light was a significant design advantage that when coupled with the cost savings of its single-element detector, gave the DTSTM spectrometer a significant price-to-performance ratio relative to competitive systems.

The Polychromix spectrometer was commercialized as a small benchtop spectrometer in 2004 and later miniaturized and released as a handheld spectrometer in 2006. Polychromix’s Phazir™ was likely the first handheld commercial analyzer to incorporate an NIR spectrometer, a light source, a probe, an onboard computer, a color LCD display, and rechargeable batteries. In 2009, Polychromix designed a space-hardened version of the DTSTM spectrometer that was flown on the NASA LCROSS mission and successfully detected water on the moon. In addition to operating in space for the duration of the mission, the MEMS chip was also capable of high-temperature operation and was validated for downhole measurements in petrochemical applications. In 2010, Polychromix was purchased by Thermo Fisher Scientific and marketed by their Portable Optical Analysis division alongside Ahura and Niton, completing their handheld portfolio.

### 6.2.5 Scanning Grating (SG) Platform

<table>
<thead>
<tr>
<th>IDC:</th>
<th>No</th>
<th>Unit Price (Offline/In-line):</th>
<th>Low/High</th>
</tr>
</thead>
<tbody>
<tr>
<td>Environmental Sensitivity:</td>
<td>Unknown</td>
<td>Dynamic Range/SNR:</td>
<td>Average/Average</td>
</tr>
<tr>
<td>Instrument Stability:</td>
<td>Unknown</td>
<td>Spectral Range/Resolution:</td>
<td>High/Average</td>
</tr>
</tbody>
</table>

At the turn of the 21st century, researchers at the Fraunhofer Institute began exploring the idea of replacing the tradition tilted grating in a Czerny–Turner spectrometer with a MEMS grating structure [4,5]. Their goal was the creation of a compact, 10 mm × 10 mm × 5 mm spectrometer. By 2004, the team had filed their first patents [20,21].

Their first challenge involved the development of an efficient grating structure. Initially, they etched lines and spaces in a silicon substrate, creating rectangular grating structures. Unfortunately, their rectangular gratings had extremely low efficiency (4%–5%), which were unsuitable for the real-world applications. Using anisotropic wet etching, a common MOEMS fabrication process, the team was able to achieve grating efficiencies in the 40%–50% range over a broad wavelength range. Figure 6.9 shows the surface electron microscopic (SEM) images of three grating structures: a simple V-grove (top), a sinusoidal structure realized by glass filling of V-grooves followed by metallization (middle), and lastly, a tilted crystal sphere geometry that could serve as a master for blaze-grating creation [22]. Fortunately, the simple V-groove met the developmental goals of a high-efficiency grating capable of operation of the spectral range of 900–2500 nm [23].

### 6.2.5.1 The Scanning Grating

Realization of a viable grating structure upon a scanning MEMS element allowed for the creation of a miniaturized spectrometer with a configuration resembling the classical Czerny–Turner system. Figure 6.10 compares the traditional configuration with the MEMS scanning grating
configuration. After entering the entrance slit (S1) proximal to the distal end of an optical fiber, the incoming light is collimated via a high-precision micromachined static mirror (M1). The collimated light is then incident on the scanning grating where it is diffracted and subsequently captured by a second high-precision refocusing mirror (M2). M2 captures the diffracted light of
a given wavelength interval that is correlated with the grating’s active position, focusing onto a
detector behind the exit slit (S2). It must be noted that extreme care had to be taken in the design
of the system to account for even-order aberrations, resulting in complex surfaces for both M1 and
M2 [23].

The scanning grating assembly utilizes an electrostatic resonant drive (ESD) that is co-fabricated
with the grating assembly in a single MOEMS unit in order to scan the grating. The ESD oscil-
lates the grating at unique resonances that can be physically modeled as a mass-spring system. The
grating size determines the range of deflection angle and the optimal resonance frequency. As an
example, a larger grating area allows for a high number of ruled lines, leading to a higher overall
throughput. The larger mass of the larger grating results in a lower resonance frequency, allowing
for longer integration times at the detector and ultimately, improved SNRs. The resonant working
principle results in high-stability scanning, and the correlation of the wavelength scale with the
magnitude of the deflection [23,24].

Figure 6.11 shows an exploded view of a scanning grating spectrometer module. Clearly visible
are the ultra-precision micromachined routing optics [25]. The microfabrication process allows for
the integration of the following items within a single monolithic device:

- The entrance and exit slits;
- The grating plate;
- The springs attached to the grating plate;
- The comb structures of the ESD;
- The position measurement transducers.

6.2.5.2 Scanning Grating KTA Summary
The SG platform of Figure 6.11, with associated control and signal processing electronics, is com-
mmercially available from HiperScan in a variety of configurations. Unfortunately, the authors were
6.2.6 Micro-Mirror Array (DLP™) Platform

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
<th>Spectral Range/Resolution:</th>
<th>Dynamic Range/SNR:</th>
<th>Unit Price (Offline/In-line):</th>
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</thead>
<tbody>
<tr>
<td>IDC:</td>
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<td>High/High</td>
<td>High/High</td>
<td>Average/Average</td>
</tr>
<tr>
<td>Environmental Sensitivity</td>
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<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Instrument Stability</td>
<td>High</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The micromirror array, a MOEMS component consisting of millions of hinged microscopic aluminum mirrors on a silicon chip, was developed by Dr. Larry Hornbeck of Texas Instruments (TI) in 1987, after struggling for close to a decade to perfect an analog-controlled MEMS mirror. Over the course of the next 15 years, Dr. Hornbeck and his colleagues at TI were awarded a series of patents that formed the foundation for micromirror array technology and established the practical methods for manufacturing high-density arrays of micromirrors on integrated circuits. US patent no. 4,710,732 entitled “Spatial Light Modulator and Method”, which was granted in 1987, is considered the seminal patent to potentially the most successful MOEMS technology developed to date. The secret to Hornbeck’s success and the successful commercialization of the micromirror array was a move from analog to digital control and the addition of a mechanical hard stop to control the micromirror’s displacement, creating a bistable mirror and enabling the repeated and consistent actuation of millions of mirrors in concert [27].

Six years later, in 1993, TI formed its Digital Imaging Division, which was tasked with commercializing Dr. Hornbeck’s invention. While it is true, the digital projection industry was the first to capitalize on the micromirror array as a core technology, the television (TV) industry soon followed with Mitsubishi’s release in 2001 of the RPTV. Eventually, TI’s Digital Imaging Division rebranded the micromirror array as a digital light processing chip or DLP® chip, and the future of DLP® technology was established.

Unfortunately, with the release of the first LED TV by Sony in 2005, by 2008, Mitsubishi was the only remaining manufacturer of RPTVs, and by 2012, they too ceased production. Fortunately, with billions invested in the development of the DLP® chip and moreover, the availability of a proven technology and established supply chain, at the turn of the 21st century, researchers around the world began looking into other opportunities for the core technology [28].

The DLP® chip (Figure 6.12), with its tiny digitally controlled mirrors tilting thousands of times a second, has led to the creation of advanced 3D imaging systems, tiny pico projectors embedded in cell phones, in addition to the state-of-the-art digital cinema projectors. Further, the DLP® chip has found its way into a variety of non-display applications such as optical switching, holographic data storage, stereolithography, programmable lighting, hyperspectral or multipoint imaging, and programmable spectroscopy [29].

While the term “programmable spectrometer” was likely coined to describe the grating light valve [GLV] spectrometer, reviewed above, it is fair to say it has become synonymous with the DLP spectrometer thanks to TI and the spectroscopy group inside TI that was created in 2011 to facilitate the development and commercialization of DLP-based spectral products.

The terminology stuck because the programmable nature of the DLP® technology allowed for the development of new and/or advanced spectral methods previously not possible with conventional spectrometers [30,31].
6.2.6.1 The DLP

In 2006, Christian et al. [32] conceptualized a spectrometer built around a DLP™ chip that could route and encode spectrally dispersed light between a sample and the reference path within a NIR spectrometer designed to go downhole to characterize oil and gas within a reservoir. The spectrometer had to be capable of operating over a 200°C temperature range in a high shock and vibration environment. Further, the spectrometer had to be capable of mitigating both temperature- and time-dependent measurement (i.e., calibration) drift (Figure 6.13).

The DLP platform was chosen due to its ability to not only route light repeatedly and accurately between two independent light paths but also encode the routed light at frequencies well above the electromagnetic frequencies common within an oil and gas operating environment. Further, due to the natural variability in the composition of crude oil, the device required a relatively large dynamic range and high SNR.

If one envisions the light within the spectrometer dispersed across the DLP™ chip such that columns are wavelengths and rows represent the intensity, an understanding of how the DLP spectrometer works begins to take shape. By turning one or more columns at a time, it is possible to scan through wavelengths, effectively capturing an optical spectrum. By turning one column at a time, resolution is maximized but throughput and scan time are impacted. By attenuating rows, which

**FIGURE 6.12** A rendering of TI’s DLP™ Chip, which is considered an adaptive optical element. The chip shown consists of over a million tiny mirrors aligned in columns and rows. The mirror diagonals are approximately 8 times smaller than the average diameter of a human hair and can move between two stable positions at kHz rates. The mirrors are so small; they are effectively massless, allowing the chip to be considered a solid-state component.

**FIGURE 6.13** A ray trace of an Offner relay DLP-based NIR spectrometer first conceptualized by Christian et al. [32]. The component at the far right of the ray trace is a DLP™ chip. On the far left were three input fibers. A center fiber for bringing light into the relay and two fibers on opposite sides of the input fiber for receiving light that had been encoded and routed by the DLP™ chip.
represent amplitude, throughput is impacted, as is dynamic range. The beauty of the DLP is in its flexibility, its tunability, or better stated, its programmability. Next, imagine being able to modulate a predetermined series of columns and rows to deliver a specific number of photons within a limited spectral range to a sample and then lock-in on the modulated signal after it is interacted with that sample. All of this and more is possible with a DLP-based spectrometer. In fact, the programmable nature of the DLP™ chip allows for other forms of encoding such as Hadamard encoding, enabling the DLP spectrometer to continually operate under a high-flux condition, maximizing SNR across the full spectral range of the spectrometer, and maximizing dynamic range. The importance of the above capabilities is likely apparent to a spectroscopist, but what might not be apparent is:

1. The importance of the DLP’s ability to route light accurately and repeatedly between two independent light paths. In the case of the chip as shown in Figure 6.13, the two light paths are separated by ±12° normal to the DLP surface. The innovation of Christian et al. in 2006 centered around the realization that either of the two independent states could be utilized for the internal full spectrum drift and/or environmental correction;

2. The programmability of the DLP, which allows instrument manufacturers, for the first time, to clone NIRS analyzers. Since NIRS instruments require complex multivariate methods to produce rational outputs, the importance of spectral clones with respect to the scaling of applications within an industrial environment should not be underestimated;

3. The DLP platform’s ability to only scan the wavelengths of interest, allowing for extremely fast methods if an application can be reduced to just a handful of relevant wavelengths.

While DLP-based NIRS can be purchased direct from multiple vendors, Neolitics, Inc., a company in the process automation industry, retains the rights to using the traditional off-state of the DLP for IDC.

### 6.2.6.2 DLP KTA Summary

To the authors’ knowledge, the DLP platform is the only platform capable of offering true full-spectrum integrated drift correction or IDC. Further, the low environmental sensitivity and high instrument stability are attractive KTAAs for industrial clients or systems integrators. The average offline unit price may not work for low-end, low-cost applications but the average unit price for in-line process control applications is very attractive. With the high dynamic range, spectral range, and SNR achievable with the DLP platform, it could be the right technology for your application. The only consideration is the average resolution, but like diode array-based spectrometers, with the DLP spectrometer, you can sacrifice a range for resolution.

### 6.2.7 Linear Variable Filter (LVF) Platform

| IDC: No | Unit Price (Offline/In-line): Low/High |
| Environmental Sensitivity: High | Dynamic Range/SNR: Average/Low |
| Instrument Stability: High | Spectral Range/Resolution: Average/Average |

The LVF platform, like the MOE, is not technically a MOEMS device in the strictest sense, since it is difficult to identify an element with mechanical functionality. It is, however, enabled by microfabrication techniques, and MEMS developers have come to agree that MEMS devices with static micromechanical features are still MEMS devices [33]. Armed with this understanding, the LVF,
with its array of photodiodes placed with micron mechanical precision and further, optically filtered with nanometer accuracy using microfabrication techniques, is without a doubt, a MOEMS platform technology.

6.2.7.1 The LVF

After decades of demonstrated success of filtered photodiodes in the field of photometric analysis, it is difficult to understand why the LVF spectrometer wasn’t commercially available prior to 2012 [34]. The answer to this question can be found by examining the complexity of the linear variable filter (LVF), also known as the “linear variable bandpass filter” (LVBF) or the “linear variable optical filter” (LVOF).

As shown in Figure 6.14, the basic architecture of the LVF consists of an optical substrate upon which a first dielectric mirror stack, a precision-tapered optical cavity, and a second dielectric mirror stack are layered. Effectively, the wedge separation between the dielectric layers creates a Fabry–Perot (FP) cavity with variable thickness laterally, enabling a linearly progressive series of wavelength-selective FP filters. The relatively modern ability to create such precision coatings makes this platform a powerful approach for designing the fit-for-purpose LVF platforms.

The LVF element can be considered equivalent to a Fabry–Perot interferometer (FPI), except spatial coordinates as opposed to temporal coordinates define the passband.

To create a spectrometer, the LVF must be precisely integrated with a linear detector array. For NIRS, the preferred detector is an InGaAs linear photodiode array. While the LVF design can limit the spectral range and resolution of the integrated platform, the physical specifications of the detector array typically determine the spectral resolution and spectral range of the LVF platform. Table 6.2 summarizes the operational specifications of the Viavi MicroNIR 1700 ES for two different InGaAs photodiode arrays: a standard range detector array that covers 950–1650 nm and an extended range detector array that covers 1150–2150 nm. An image of the LVF platform specified by Table 6.2 can be found in Figure 6.15 [35–37].

6.2.7.2 LVF KTA Summary

Figure 6.15 clearly illustrates an attractive attribute of the LVF platform, its size. Clearly visible in the image are the two integrated sources that allow the MicroNIR 1700ES to perform reflectance spectroscopy. It should be noted, however, that a platform capable of transmission spectroscopy is also available. The package shown, which is solid state in nature, is a complete NIRS system with integrated electronics.

While the LVF itself is extremely robust with respect to shock and vibration, unfortunately, InGaAs photodiode arrays are sensitive to temperature fluctuations and moreover, struggle with dynamic range and SNR at elevated temperatures. In fact, the recommended maximum operating
temperature for an InGaAs photodiode array is 50°C. To mitigate the influence of environmental temperature on the performance of InGaAs photodiode arrays, manufacturers integrated single- or dual-stage thermoelectric coolers into the InGaAs package but doing so adds to the cost of the arrays and increases the electrical power requirements of the overall platform. In all, you have an application within a relatively low and stable thermal environment, and if you are concerned about shock and vibration or size, the LVF platform may be a viable option.

### 6.2.8 Fabry–Perot Interferometer (FPI) Platform

In terms of size, it’s difficult to compete with today’s FPI NIR platform. There are two ways to integrate an FPI to form the monochromator: (1) integrated with the photodetector and (2) integrated with the light source. While both are available commercially, a MEMS-FPI integrated with

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**TABLE 6.2 VIAVI MicroNIR specifications**

<table>
<thead>
<tr>
<th>Specification</th>
<th>Standard: 950–1650 nm</th>
<th>Extended: 1150–2150 nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Detector array</td>
<td>Standard: 6.25 nm</td>
<td>Extended: 8 nm</td>
</tr>
<tr>
<td>Geometric resolution (pixel size)</td>
<td>1.25% of center wavelength</td>
<td>@ 1000 nm, resolution=12.5 nm</td>
</tr>
<tr>
<td>Optical resolution (spectroscopy)</td>
<td>@ 2000 nm, resolution=25 nm</td>
<td></td>
</tr>
<tr>
<td>Wavelength accuracy</td>
<td>&lt;3 nm (NIST SRM-2036)</td>
<td></td>
</tr>
<tr>
<td>Wavelength repeatability</td>
<td>&lt;1 nm (NIST SRM-2036)</td>
<td></td>
</tr>
</tbody>
</table>

**FIGURE 6.15** MicroNIR 1700ES from VIAVI. Graphic provided courtesy of VIAVI Solution, Inc.
a photosensor is the most common approach. This section will examine both options, realizing that the limitations and advantages of the FPI are not dependent on where integration occurs. Regarding size, fully integrated FPI NIR modules, inclusive of electronics, are commercially available in the range of 25 mm³. Further, OEM modules without associated electronics can be packaged in TO-5 cans.

6.2.8.1 The FPI

Figure 6.16 shows an exploded view of the FPI integrated with a photodetector, which for the NIR spectral region is typically an InGaAs pin photodiode (Hamamatsu MEMS-FPI technical note). The FPI consists of an air gap between upper and lower mirror structures. The air gap is defined by physical spacers, also shown in this figure. The application of an electrostatic voltage to the upper mirror membrane changes the gap shifting the transmission wavelength of the assembly. Increasing the applied voltage reduces the gap, blue shifting the transmission. The voltage step size corresponds to the spectral resolution. The FPI substrate is silicon, and the mirrors are common multilayer dielectric coatings (silicone dioxide, silicon nitride, and poly-silicon). Since the FPI element is voltage-driven, the device is electrostatically sensitive. The FPI element may also be driven with piezoelectric actuators. Piezo-driven systems have significantly larger apertures [38,39].

In this configuration, the FPI module requires an axial collimated illumination of the assembly. The bandpass filter sets the wavelength range of the assembly and supplies high out of band rejection. The FPI filter allows for scanning across the wavelength range within the pass band. Typically, these types of FPI modules have limited bandwidths (250–450 nm) and 12–28 nm measurement resolution. Consequently, several assemblies are necessary to span the full NIR region.

Since the FPI platform supplies the scanning capability for the spectrometer, it is important to understand how scanning occurs and the underlying control required. FPI scan rates can be very fast, in the 50–100 μs range; however, averaging is needed at fast scan rates to improve SNR. Further,
both bandwidth and spectral resolution are impacted by the scan rate. Longer scan periods minimize
the need for averaging multiple scans, with similar SNR levels and improved optical resolution.

The allowed wavelength range is determined by the maximum deflection of the FPI. Since
the filter response is nonlinear and temperature-dependent, suppliers of FPI assemblies provide a
detailed information on the drive voltage requirements as a function of temperature.

In addition, the upper mirror is sensitive to both shock and vibration. In a laboratory environ-
ment, these sensitivities are less problematic because the instruments typically experience a minimal
thermal variation or shock and vibration. However, in process applications, care must be taken with
respect to packaging, and complex electronic control systems are required to make the FPI a viable
platform. For example, a careful characterization of the wavelength response versus temperature gen-
erates an additional correction factor that changes the applied voltage profile to keep the transmission
wavelength constant and eliminate the thermal drift. Typically, this is accomplished with on-board
thermal measurement either in the control electronics or in the FPI module. When properly charac-
terized and controlled, the FPI assembly keeps center wavelength across the temperature range of
interest to ±1.4 nm, which is acceptable for many NIR applications. Further, it must be noted that
if the temperature range is sufficiently wide, the tunable spectral range of an FPI could be limited.

As noted above, a second integration point for an FPI is at the light source (Figure 6.17). This
creates a variable wavelength light source, a predispersive NIR spectrometer, with the photode-
tection occurring post-sample interaction. The widest application of this approach is for optical
coherence tomography (OCT) [40,41].

Commercially, the FPI is integrated with either a superluminescent LED (Axsun IntegraSpec
XL) [42] or a solid-state laser (Axsun IntegraSpec2) [43]. The laser-based approach is by far the
most common. Both these light sources have limited bandwidth ranges resulting in a spectrometer
with high resolution (~1 nm) or a narrow spectral range. The commercial FPI light source is a com-
plex module and needs a bit of unpacking [44].

First, a discussion of light sources is made. Solid-state light sources are small in size and offer
brightness levels 1000 times or greater than traditional broadband light sources. The small sizes
make an integration on micro-optical benches significantly simpler. Additionally, both options have
small coherent beams, minimizing the need for beam-shaping optics. The FPI in this assembly has
all the same requirements for scanning control as described above. However, the light source itself
sets the scanning range, not an integrated filter on the FPI. Two added source control elements
are integrated on the optical bench: real-time wavelength referencing and real-time source power
referencing. Axsun has branded this integrated module as WARM (wavelength and amplitude reference module). The entire module or micro-optical bench is environmentally controlled allowing the better overall operational control, minimizing the environmental fluctuations, and enhancing the output stability. Optical coupling between integrated elements uses free-space micro-optics. Wavelength referencing occurs via the fringe pattern analysis, supplying exact wavelength values under changing environmental conditions.

### 6.2.8.2 FPI KTA Summary

From the review above, it can be seen that there are a variety of FPI platforms available within the market for NIRS. Further, depending on the application requirements, there may be more than one implementation that is acceptable. If size is a concern and the application environment is relatively benign, FPI could be the right solution. Just keep in mind that FPI platforms have a limited spectral range.

With this said, it can be argued that Axsun’s Warm module with its amplitude reference arm is a form of IDC, allowing WARM-enabled micro-benches to maintain a 24-hour baseline stability on the order of ±0.5%, across the operational temperature range of 0°C–55°C.

### 6.3 THE FUTURE OF NIR & I2O T

The authors are of the opinion that the future of NIR is bright, pun-intended. We envision a future where MOEMS-based NIR platforms are small, easily installed by a client, and require virtually no maintenance after installation. A future where the TOC of an in-line NIR I2OT strategy is literally the cost of the technology itself, the CAPEX, and the traditional OPEX associated with the deployment of the technology is virtually zero. In this future, due to the ease of use and affordability of MOEMS-based NIR platforms, the distributed process monitoring is enabled, which subsequently enables distributed process analytics (DPA). Distributed units acting in concert to optimize throughput and yield, ensure quality, and enhance safety will usher in a new era in process visualization and optimization, a new era for smart factories. In our opinion, the NIR industry is not expanding by an estimated 9%–12% CAGR. It is expanding much, much faster.

### REFERENCES


ADDITIONAL READING


