WHAT IS AN FT-IR AND AN FT-IR SPECTROMETER?

An FT-IR Spectrometer is an instrument which acquires broadband NIR to FIR spectra. Unlike a dispersive instrument, i.e. grating monochromator or spectrograph, an FT-IR Spectrometer collects all wavelengths simultaneously. This feature, called the Multiplex or Felgett Advantage, is discussed in detail on page 3.

FT-IR Spectrometers are often simply called FT-IRs. But for the purists, an FT-IR (Fourier Transform InfraRed) is a method of obtaining infrared spectra by first collecting an interferogram of a sample signal using an interferometer, and then performing a Fourier Transform (FT) on the interferogram to obtain the spectrum. An FT-IR Spectrometer collects and digitizes the interferogram, performs the FT function, and displays the spectrum.

WHY AN FT-IR SPECTROMETER?

Choose an FT-IR over a dispersive instrument if:

- You work in the infrared
- You need high spectral resolution
- You work with weak signals
- You need to acquire your spectra quickly and with high S/N ratio
- You need high spectral accuracy

FT-IRs possess strong theoretical reasons that enable them to excel in these categories. How much of this potential advantage is realized in your application depends strongly on the instrument's design and the particulars of your measurement.

HOW DOES AN FT-IR SPECTROMETER WORK?

The Michelson Interferometer

An FT-IR is typically based on a Michelson Interferometer; an example is shown in Fig. 1. The interferometer consists of a beam splitter, a fixed mirror, and a mirror that translates back and forth, very precisely. The beam splitter is made of a special material that transmits half of the radiation striking it and reflects the other half. Radiation from the source strikes the beam splitter and separates into two beams. One beam is transmitted through the beam splitter to the fixed mirror and the second is reflected off the beam splitter to the moving mirror. The fixed and moving mirrors reflect the radiation back to the beam splitter. Again, half of this reflected radiation is transmitted and half is reflected at the beam splitter, resulting in one beam passing to the detector and the second back to the source.

Fig. 1 A Schematic of a generic Michelson interferometer.

What are OPD and ZPD?

Optical Path Difference (OPD) is the optical path difference between the beams travelling through the two arms of an interferometer. OPD is equal to the product of the physical distance travelled by the moving mirror (multiplied by 2, 4, or other multiplier which is a function of the number of reflecting elements used) and n, the index of refraction of the medium filling the interferometer arms (air, Nitrogen for purged systems, etc.). The raw FT-IR data consists of a number of (signal, OPD) pairs of values.

FT-IR has a natural reference point when the moving and fixed mirrors are the same distance from the beam splitter. This condition is called zero path difference or ZPD. The moving mirror displacement, Δ, is measured from the ZPD. In Fig. 2, the beam reflected from the moving mirror travels 2Δ further than the beam reflected from the fixed mirror. The relationship between optical path difference, and mirror displacement, Δ, is:

\[ \text{OPD} = 2\Delta n \]

The Interferogram

Interferogram is the name of the signal format acquired by an FT-IR spectrometer. It is usually significantly more complex looking than a single sinusoid, which would be expected if only a single wavelength of light was present. Fig. 3 shows the beam path of a two wavelength source; Fig. 4 is the interferogram of a broadband light source. The centerburst, the big spike in the center of Fig. 4 is a telltale signature of a broadband source. Its origin lies in the fact that all wavelengths are in-phase at the ZPD. Therefore, their contributions are all at maximum and a very strong signal is produced by the system’s detector.

As the optical path difference, OPD, grows, different wavelengths produce peak readings at different positions and, for a broadband signal, they never again reach their peaks at the same time. Thus, as you move away from centerburst, the interferogram becomes a complex looking oscillatory signal with decreasing amplitude.

The X-axis of the interferogram represents the optical path difference. Each individual spectral component contributes to this signal a single sinusoid with a frequency inversely proportional to its wavelength. This leads us to the definition of the unit of spectral measurement. The wavenumber (cm\(^{-1}\)), denoted as \(ν\), is defined as the number of full waves of a particular wavelength per cm of length (typically in vacuum; index of refraction \(n=1\)). The advantage of defining the spectrum in wavenumbers is that they are directly related to energy levels. A spectral feature at 4,000 cm\(^{-1}\) spectral location represents a transition between two molecular levels separated by twice the energy of a transition with spectral signature at 2,000 cm\(^{-1}\).

Table 1 lists a sampling of corresponding wavelength, wavenumber, frequency and energy values.

<table>
<thead>
<tr>
<th>(\nu) (cm(^{-1}))</th>
<th>(\lambda) (μm)</th>
<th>(f) (10(^{12}) Hz)</th>
<th>(E) (eV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>200</td>
<td>50</td>
<td>5.996</td>
<td>0.02479</td>
</tr>
<tr>
<td>500</td>
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<td>29.98</td>
<td>0.12398</td>
</tr>
<tr>
<td>2000</td>
<td>5</td>
<td>59.96</td>
<td>0.24797</td>
</tr>
<tr>
<td>5000</td>
<td>2</td>
<td>149.9</td>
<td>0.61922</td>
</tr>
<tr>
<td>10,000</td>
<td>1</td>
<td>299.8</td>
<td>1.23984</td>
</tr>
</tbody>
</table>
Fig. 2  Schematic representation of waves and their phases, input, output, and the two arms of the interferometer as the scan goes from zero path difference condition to OPD=λ. (a) OPD=0 case. (b) λ/4 OPD case. (c) λ/2 OPD case. (d) 3λ/4 OPD case. (e) 1λ OPD case.

Fig. 3 Two wavelength source case.

Fig. 4 Broadband source interferogram.
The Fourier Transform Algorithm

Once an interferogram is collected, it needs to be translated into a spectrum (emission, absorption, transmission, etc.). The process of conversion is through the Fast Fourier Transform algorithm. The discovery of this method by J.W. Cooley and J.W. Tukey in 1965, followed by an explosive growth of computational power at affordable prices, has been the driving force behind the market penetration of FT-IR instruments.

A number of steps are involved in calculating the spectrum. Instrumental imperfections and basic scan limitations need to be accommodated by performing phase correction and apodization steps. These electronic and optical imperfections can cause erroneous readings due to different time or phase delays of various spectral components. Apodization is used to correct for spectral leakage, artificial creation of spectral features due to the truncation of the scan at its limits (a Fourier transform of sudden transition will have a very broad spectral content).

FT-IRs are capable of high resolution because the resolution limit is simply an inverse of the achievable optical path difference, OPD. Therefore, a 2 cm OPD capable instrument, such as our MIR 8000™, can reach 0.5 cm⁻¹ resolution. Table 2 shows the relationship between resolution expressed in wavenumbers and the one expressed in nanometers, as is customary in dispersive spectroscopy.

Table 2 Resolution Values in Wavenumbers and Nanometers

<table>
<thead>
<tr>
<th>Wavelength (µm)</th>
<th>Resolution (cm⁻¹)</th>
<th>Resolution (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.2</td>
<td>1</td>
<td>0.004</td>
</tr>
<tr>
<td>0.5</td>
<td>1</td>
<td>0.025</td>
</tr>
<tr>
<td>1</td>
<td>1</td>
<td>1.0</td>
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<td>1</td>
<td>4.0</td>
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<td>5</td>
<td>1</td>
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<td>10</td>
<td>1</td>
<td>10.0</td>
</tr>
<tr>
<td>20</td>
<td>1</td>
<td>40.0</td>
</tr>
</tbody>
</table>

Advantages of FT-IR Instruments Over Dispersive Instruments

Following, we talk about three significant advantages that FT-IR instruments hold over dispersive spectrometers, but first we compare the two instruments.

Table 3 FT-IR and Dispersive Spectrometer Comparison

<table>
<thead>
<tr>
<th></th>
<th>MIR 8000™ FT-IR</th>
<th>Cornerstone™ 260 1/4 m Grating Monochromator</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wavelength Range</td>
<td>700 nm - 28 µm</td>
<td>180 nm - 24 µm</td>
</tr>
<tr>
<td>Max. Resolution</td>
<td>0.024 nm @ 700 nm</td>
<td>0.15 nm</td>
</tr>
<tr>
<td>Étendue@1 µm, 0.15 nm resolution</td>
<td>0.38</td>
<td>0.001</td>
</tr>
</tbody>
</table>
FT-IR INSTRUMENTS HAVE A SHORT WAVELENGTH LIMIT

A collimated, monochromatic light source will produce an interferogram in the form of a sinusoid, at the detector. When the light intensity goes from one maximum of the interferogram to the next maximum, the optical path difference between the two legs of the interferometer changes by exactly 1 wavelength of the incoming radiation.

To determine the wavelength of the incoming radiation, we can measure the frequency \( f_i \) or period \( t_i = 1/f_i \) of the interferogram to the next maximum, the optical path difference. When the light intensity goes from one maximum of the interferogram from a pure sine wave that in turn will be considered as a mix of sinusoids. In other words, we will think that there is more than one wavelength in the incoming radiation. This behavior produces what are called "spectral artefacts".

Since the manufacture of an interferometrically accurate drive is extremely expensive, FT-IR designers added an internal reference source into the interferometer to solve the drive performance problem. A HeNe laser emits light with a wavelength which is known with a very high degree of accuracy and which does not significantly change under any circumstance. The laser beam parallels the signal path through the interferometer and produces its own interferogram at a separate detector. This signal is used as an extremely accurate measure of the interferometer displacement (optical path difference).

We can, therefore, write the following equation for a HeNe based FT-IR:

\[
\lambda_i = \lambda_r \times (f_i/f_r) \quad \text{................................} \quad (2)
\]

Where subscript \( r \) denotes HeNe reference.

We can now calculate the spectrum without extremely tight tolerances on the velocity.

This was just a theoretical example. Now let us see how the reference interferogram is actually used in the MIR 8000™. The signal from the interfering beams of the HeNe are monitored by a detector. What is observed is a sinusoidal signal. The average value is the same as you would see if the beam was not divided and interference produced. The sinusoid goes positive and negative about this value. The average signal level is called zero level. A high precision electronic circuit produces a voltage pulse when the HeNe reference sinusoid crosses zero level. By use of only positive zero crossings, the circuitry can develop one pulse per cycle of the reference interferogram, or use all zero crossings for two pulses per cycle of this interferogram. The latter case is often called oversampling. These pulses trigger the A/D converter which immediately samples the main interferogram.

There is a fundamental rule called the Nyquist Theorem which can be paraphrased to state that a sinusoid can be restored exactly from its discrete representation if it has been sampled at a frequency at least twice as high as its own frequency. If we apply this rule to the above formula we find immediately that since the minimum value of \( (f_i/f_r) \) is 2, so the minimum value of \( \lambda_i \) is twice the wavelength of the reference laser:

\[
\lambda_{\text{min}} = 633 \text{ nm} \times 2 = 1.266 \mu\text{m}
\]

With oversampling, the reference laser wavelength is effectively halved. So in this case:

\[
\lambda_{\text{min}} = (633 \text{ nm}/2)^2 = 633 \text{ nm}
\]

In practice, the FFT math runs into difficulties close to the theoretical limit. That is why we say 1.4 µm is the limiting wavelength without oversampling, and 700 nm is the limiting wavelength with oversampling.

THE RELATIONSHIP BETWEEN RESOLUTION AND DIVERGENCE

The FT-IR principle of operation is very different from that of dispersive instruments. Many aspects of this relatively new approach are counter intuitive to those of us weaned on dispersive techniques, starting of course with the funny wavenumber units that go the wrong way!

Fig. 5a shows a typical optical layout of external optics relative to a dispersive monochromator. Fig. 5b shows the same for an FT-IR spectrometer. The main optical feature of the FT-IR is that there are no focusing elements inside the instrument: it works with parallel beams. Dispersive instruments from the input slit to an output slit are self contained in the sense that major spectral characteristics do not depend very much on how you illuminate the input slit and how you collect the light after the output slit. Manipulating the light with external optics just gains or loses you sensitivity and adds or reduces stray light and aberrations.
This is not the case with FT-IRs. External optics are as important for proper functioning of the instrument as its internal parts. Fig. 6 shows in a bigger scale a simplified scanning Michelson interferometer together with a source and a detector. Suppose first that the source is a (monochromatic) point source and therefore the beam entering the interferometer (rays 1 - 1') is perfectly parallel. Exiting the interferometer it will be focused into a point on the detector surface. With motion of the scanning mirror the detector will register an interferogram - a sequence of constructive and destructive interactions between two portions of the beam in the interferometer. The further the scanning mirror is traveling, the longer the interferogram and the higher the spectral resolution that can be achieved.

In real life, point sources as well as purely parallel beams, do not exist. A finite size source produces a fan of parallel beams inside the interferometer. A marginal beam, 2 - 2', of this fan is shown in Fig. 6. This beam will be focused at some distance from the center of the detector. With motion of the scanning mirror the detector will register an interferogram - a sequence of constructive and destructive interactions between two portions of the beam in the interferometer. The further the scanning mirror is traveling, the longer the interferogram and the higher the spectral resolution that can be achieved.

Both f and \( A_L \) are expressed in the same units, e.g. m, m\(^2\), or mm, mm\(^2\). We can use F/# instead of focal length, \( \Omega = \pi/(4(F/#)^2) \text{ sr} \) ........................................ (5)

The product of solid angle and area of an image at a plane where the solid angle originates is called by various names, optical extent, geometrical extent or \( \text{é} \text{tendue.} \) (Often, the term throughput is used instead of \( \text{é} \text{tendue.} \)) \( \text{é} \text{tendue} \) determines the “radiation capacity” of an optical system. The fundamental law of optics states that any optical system can be characterized by an optical extent/\( \text{é} \text{tendue}/\text{throughput} \) which stays constant through all optical transformations:

\[
G = A_\Omega = \text{constant} \ ................. (7)
\]

Where:
\[
\alpha_{max} = \text{the maximum divergence half angle (in radians)}
\]
\[
\sigma_{max} = \text{the maximum wavenumber in the spectrum}
\]
\[
\Delta \sigma = \text{spectral resolution}
\]

Note: In real life, point sources as well as purely parallel beams do not exist. A finite size source produces a fan of parallel beams inside the interferometer. A marginal beam, 2 - 2', of this fan is shown in Fig. 6. This beam will be focused at some distance from the center of the detector. With motion of the scanning mirror the detector will register an interferogram - a sequence of constructive and destructive interactions between two portions of the beam in the interferometer. The further the scanning mirror is traveling, the longer the interferogram and the higher the spectral resolution that can be achieved.

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\[
G = A_\Omega = \text{constant} \ ................. (7)
\]
The relevance of this is that every optical system has something that sets or limits the value of \( G \), and it is often the detector section. Knowing what part that is and improving it as best as possible is fruitful. Working to increase the \( G \) value for another part of the system is a waste of time, but a very common waste of time.

In what follows, we consider the étendue of the MIR 8000™. In general, we like to start by calculating what is the largest étendue we can tolerate to get the resolution we need. If the étendue of the instrument, including source and detector, is larger than that value, then we have to limit it.

Let's determine the resolution limit on étendue for the MIR 8000™. We know that it has an aperture of 1.25 inches (31.75 mm). We can also find a maximum allowed divergence angle of a beam propagating through it according to a maximum wavenumber in a spectrum and required resolution. From this, we can find the maximum solid angle of the fan of rays by making use of equation (6). Thus, we will find the étendue of the interferometer:

\[ G_{\text{intfr}} = 2.5 \times 10^3 \frac{[\Delta \sigma]}{\sigma_{\text{max}}} \text{mm}^2 \text{sr} \] .................(8)

When collecting spectra with wavelengths longer than 2 \( \mu \)m, \( \sigma_{\text{max}} = 5,000 \text{ cm}^{-1} \), and \( \Delta \sigma = 0.5 \text{ cm}^{-1} \),

\[ G_{\text{intfr}} = 0.25 \text{ mm}^2 \text{sr} \]

Detector Optics

Now let us consider auxiliary optics; first, on the detector side. Suppose that the allowed acceptance angle is filled freely with light. Continuing the conditions cited in the example above, we want to collect this light and squeeze it onto the smallest possible detector, since smaller detectors have better noise characteristics. To do this, we will take a very fast lens with F/# = 1. Then according to (5) the solid angle at the focal spot will be:

\[ \Omega = 0.79 \text{ sr} \]

and useful detector diameter,

\[ D = 2x \sqrt{\frac{0.25}{\pi \times 0.79}} \] .................(9)

or

\[ D = 0.6 \text{ mm} \]

Table 4 shows some other detector diameters useful at different resolutions and wavelength ranges. Similar relations apply to the source side. If we optimize the system for high resolution, we miss the opportunity to pump in a lot more radiation at lower resolutions.

What can we do in this situation? We do not have the luxury of using a different detector for each resolution. For general use, we can choose one detector which corresponds to a reasonably high but not necessarily the highest resolution. 4 \( \text{ cm}^{-1} \) is a popular choice for this, because 4 \( \text{ cm}^{-1} \) resolution is adaptable for condensed phase work. What if subsequently we need a higher resolution? There are a couple of ways to handle this eventuality. One way is to increase the focal length of the detector’s fore optics. Longer focus means higher F/#, lower throughput and a higher allowed resolution. It means, of course, a radiation loss also.

Another way is to use an aperture (Jacquinot Stop) to increase the F/#, by decreasing the effective source size; this reduces the spot size on the detector.

Source Optics

The source with its optics will typically present a beam with étendue greater than the required étendue of the interferometer. We have seen that the étendue of the instrument is usually limited by the desired resolution or detector size and optics.

<table>
<thead>
<tr>
<th>( \lambda )</th>
<th>( \Delta \sigma )</th>
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<th>1</th>
<th>2</th>
<th>4</th>
<th>8</th>
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</thead>
<tbody>
<tr>
<td>0.7</td>
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<td>1.5</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>0.6</td>
<td>0.9</td>
<td>1.3</td>
<td>1.8</td>
<td>2.5</td>
<td></td>
</tr>
<tr>
<td>4</td>
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<td>1.3</td>
<td>1.8</td>
<td>2.5</td>
<td>3.6</td>
<td></td>
</tr>
</tbody>
</table>

Table 4 Maximum Detector Diameter (mm), at F/1, vs Shortest Wavelength and Resolution
MORE CONSIDERATIONS ON OPTICAL ELEMENTS FOR FT-IR

Parabolic Mirrors

Most FT-IR instruments use off-axis parabolic mirrors for collimating and focusing light external to the interferometer. These gold coated mirrors are very broadband, from 0.7 to 10 microns they reflect more than 98% (see the reflectance curves on page 10), and it stays in this range up to 25 microns (bear in mind that for wavelengths shorter than 0.6 micron, gold is a bad reflector; its reflectivity drops abruptly to less than 40%). An important feature of mirrors in general also is that they do not have any dispersion; there is no chromatic aberration so the focal spot stays at the same place for any wavelength. They do have monochromatic aberrations.

Parabolae are devices ideally suited for collimating light from small sources and conversely for tightly focusing collimated beams of radiation. They are, however, limited to this purpose. They cannot be used for imaging of larger objects.

Light from a point source placed in a focus of a parabola (Fig. 8) will be transformed after reflection into an ideally parallel beam. Accordingly, a parallel incoming beam will be focused into a tiny focal spot. This is true for any section of the parabola. So, an off-axis section of the paraboloidal mirror can be cut out for convenience (see Fig. 9).

\[
\text{EFL} = 2f
\]

Fig. 8 Light from a point source placed at the focus of a parabola.

Fig. 9 Section of off-axis parabolic mirror.

The arrangement shown in Fig. 9 is described as a 90° off-axis mirror since the ray striking the center of the aperture and parallel to the main axis turns exactly at 90° and comes into the focal point. The distance from the point on the surface of the parabola at the center of the aperture, to the focal point, is called effective focal length (EFL) and it is exactly two times the focal length of the parabola.

\[
\text{EFL} = 2f
\]

F/numbers of off-axis parabolic mirrors can reach very low values; F/1 or even less is practical. If a finite size source, instead of a point source, is placed in the focal point of the parabola, the reflected beam will not be ideally parallel any more. It will have some angular divergence according to the angular size of the source. On top of that it will suffer significant aberrations. Accordingly, a parallel incoming beam will be focused into, not a spot, but a blurred spot.
It is important to analyze how the angular divergence of a beam turns into a blur spot in a parabola focus. We created, with an optical design software package, the optical schematic of MIR 8000™ with an F/1 parabolic mirror at the output. The effective focal length of the mirror is 20 mm. We traced rays with different divergence through the system and watched for the focal spot size.

Fig. 10 shows a graph of the diameter of the focal spot vs. angular divergence of the beam propagating through the interferometer.

The limit on divergence angle in the interferometer we found from formula (3) (page 5), at the smallest possible $\Delta \sigma$ which is 0.5 cm$^{-1}$ and the highest possible $\sigma$ which is 14,000 cm$^{-1}$, is 0.006 rad. The graph shows that the diameter of the focal spot which corresponds to this value is about 0.5 mm. (The rough estimate, of the same value made with the formulae on the preceding pages, gives a value of 0.4 mm). With increasing divergence of the beam, the diameter of the focal spot also increases, as we see, but it has some limit between 1.5 and 2 mm. The reason for this is that the interferometer itself is blocking high angle rays and they cannot reach the parabola. The maximum value of angle of rays that can get through the interferometer is 0.06 - 0.07 rad. This is exactly the region where the curve in Fig. 10 starts to flatten out.

Fig. 11 shows the energy distribution in the focal plane of the off axis reflector for beams of different divergence. This shows the increasing impact of aberrations as the “field of view” of the parabola is increased.

Lenses

Despite universality and wide usage of off-axis parabolic mirrors in FT-IR spectroscopy, they have certain disadvantages. They are pretty difficult to align; each reflection turns the beam through 90°, and this may make the system bulky. At low F/#, i.e. large fields of view (high étendue), they suffer from significant aberrations.

In many applications, especially in the Near IR, lenses could be a good choice. Fig. 12 shows the energy distribution in the focal spot of a CaF$_2$ lens having about the same focal length and F/# as the parabolic mirror considered earlier.
When using lenses, you need to consider the lens material. We recommend the use of CaF$_2$ lenses in the whole range where the CaF$_2$ beam splitter is applicable. In the very Near IR, up to 3 µm, fused silica lenses are fine, though the water absorption bands can cause some loss with lenses that are not "IR grade". They are somewhat cheaper than CaF$_2$ lenses. A wide variety of materials are available for the Mid IR. You usually have a choice among performance, expense, durability, birefringence, etc. The hygroscopic nature of some materials can be a big problem. NaCl windows and KBr are two such popular materials. Some materials are transparent in the visible and others not; this can be a plus if you are trying to align in the visible, or a negative when you would prefer the material to act as a filter.

A popular rugged and transparent material which is used for manufacturing lenses is ZnSe. It has, however, a very high index of refraction that pushes reflectance losses to relatively high levels: up to 30%. Anti-reflection coatings can help, but at further expense, and reduction of the spectral range.

A second issue is dispersion of the lens material. Lenses are definitely good for limited wavelength range applications. For example, the sensitivity range of an InGaAs detector is from 800 to 1700 nm. Using a lens should not pose a major problem, though we do see some dispersion in our labs with fused silica lenses over this range; i.e. you can axially move the lens to optimize the long wavelength or short wavelength signal. For a wider wavelength range you should position the detector at the shortest focal length position, in other words, in the position of minimum spot size for the shortest wavelength, since usually system efficiency is the lowest, there.

These examples show us that the auxiliary optics for an interferometer must be carefully chosen and arranged. Poor choices of components will lead to lack of resolution or unnecessary system throughput limitations.
**Fig. 5** Typical near normal incidence reflectance of freshly deposited Al, AlMgF$_2$, AlSiO, and Au.

**Fig. 6** Typical near normal incidence reflectance of Rhodium, Enhanced Rhodium, and Silver with Dielectric overcoat.

**TECH NOTE**

All metal reflectors deteriorate slowly in polluted atmosphere. Cumulative exposure to intense ultraviolet radiation also affects performance; overheating of the reflecting surface will destroy the reflector.

The enhanced rhodium coating has been optimized for high performance in the ultraviolet, a wide range of angles of incidence and longevity. It is the most efficient and durable coating available for ellipsoidal reflectors. The AlMgF$_2$ coating has been optimized for performance in the near UV.