# PRISM REFRACTOMETRY AT THE UNIVERSITY OF ARIZONA

INTRODUCTION: Precise prism refractometry at the Optical Sciences Center at the University of Arizona is done using two instruments. A conventional Wild Model 79 Precision Spectrometer is used with various line sources to measure index in the visible spectrum at ambient temperature. Typical relative uncertainties in n are 1 part in 10^5. This instrument is also used in autocollimation mode to characterize prism angles with uncertainties of several arc-sec.

For temperatures other than ambient and for wavelengths in the ultraviolet and infrared regions, a custom-made instrument, named the Cryogenic Refractometer after its original purpose, is used. It employs a modified minimum deviation (MMD) method as described by Platt, et. al., JOSA **65**, 1264 (1975). A layout of the instrument is shown below. The basic wavelength range of the instrument extends from 300 nm to 15 μm. The temperature range over which measurements have been conducted extends from 10K to 775K. Currently measurements down to ~65K (liquid N_2 at reduced pressure) are routine. Overall measurement relative uncertainties on the order of n parts in 10^4 (k=1) are typically achieved.
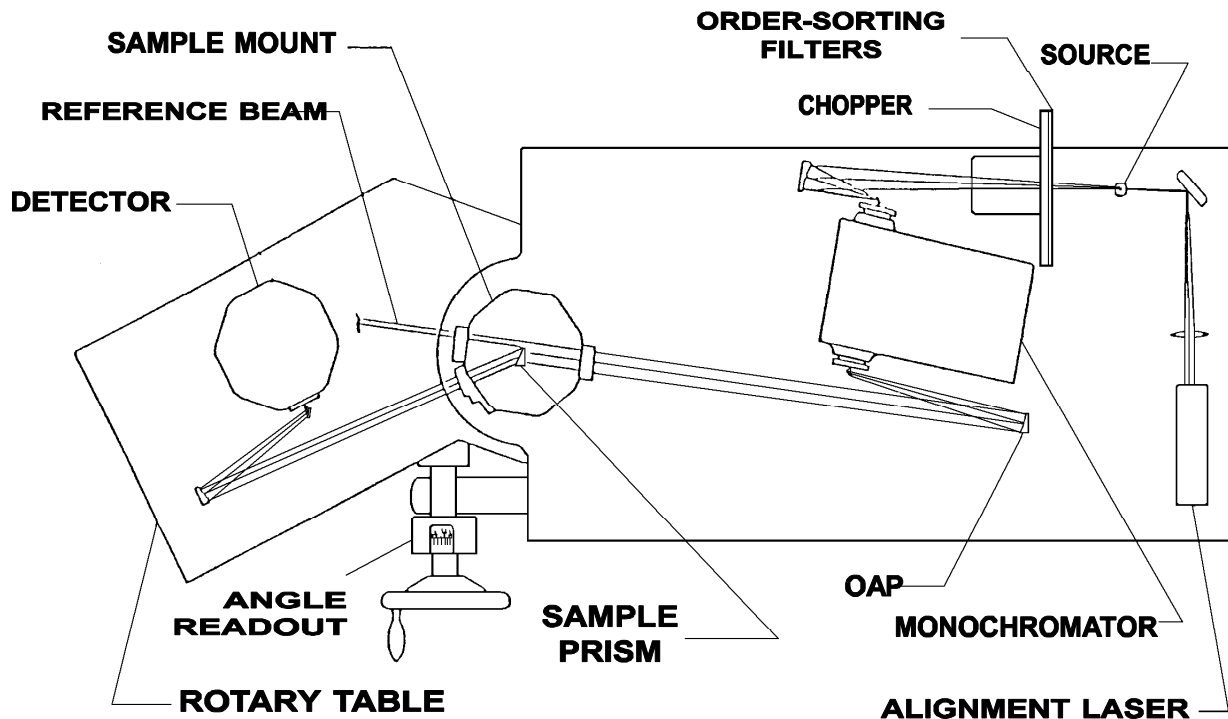
METHODOLOGY: Basic instrument alignment consists of tracing a beam from the alignment laser through the system to establish all of the appropriate centers. Then a beam expander is installed and the OAP is collimated. Detector focus is achieved by ensuring that the sample beam and the reference beam centers are identical.

For ambient temperature measurements, the sample prism in mounted on a two-axis kinematic stage in the collimated monochromatic beam coming from mirror OAP. The sample is centered over the rotary detector table. The sample prism divides the beam spatially, and the position of the centers of the reference beam and the sample beam are measured with the detector assembly mounted on the rotary table.

For cryogenic measurements, the Dewar in which the sample is placed has fixed windows, requiring a nominal deviation angle of 30°. The equation below is used to determine the prism apex angle A to achieve the required 30° deviation angle.

$$A = \tan^{-1}\left(\frac{0.5}{n - 0.866}\right)$$

where n is the nominal (mid-band) index of refraction. The tolerance on A is $0.01A$, or 1%. For refractometry where the Dewar is not required (room- and elevated temperatures), a larger angle may be used to lower uncertainties. For visible measurements at room temperature, a larger angle may be chosen to allow use of the method of minimum deviation

The majority of our measurements are cryogenic, from ~65K to 350K. For these measurements, the prism is mounted in a copper sample holder and integrated into a custom double-chamber Dewar based upon an Infrared Laboratories design and suitable for either helium or nitrogen cooling. Included in the Dewar are two silicon diode calibrated temperature sensors and a heater for temperature control. The chamber windows are selected from coated fused silica, sapphire, CaF_2, AR-coated ZnSe and KRS-5. Wavelengths are selected using a Jarrell-Ash Model 27 275 mm spectrometer fitted with up to three gratings chosen to optimum performance in the desired spectral region. The wavelength calibration of the Jarrell-Ash spectrometer is verified using various gas discharge sources and multiple orders from a He-Ne laser at 632.8 nm. Measurements are typically made using a 250 μm exit slit, giving a spectral bandwidth (FWHM) that is dependent on the grating selected for the desired wavelength range. The bandwidths for a 250 μm exit slit are shown below.

WAVELENGTH RANGE	GRATING	FWHM
350-1000 nm	1200 lpmm	0.75 nm
1-3.6 μm	300 lpmm	3 nm
3.6-7.2 μm	150 lpmm	6 nm
7-15 μm	50 lpmm	18 nm

Sources employed are a tungsten ribbon-filament lamp from the visible out to 2.5 µm and an infrared ceramic glower for longer wavelengths. Both are operated from stabilized power supplies. The He-Ne alignment laser and a mercury discharge source are also used. Temperatures below ambient are obtained by radiative transfer from the Dewar shroud (cooled with LN$_2$) and directly via LN$_2$ in the main chamber. Temperatures between about ~65K and 77K are obtained by pumping on the main LN$_2$ reservoir. Two temperature sensors are used; (1) the feedback control sensor for the Lakeshore temperature controller is located between the heater and the test prism, and the second sensor is on the copper fixture below the test prism. The observed temperature gradient across the prism is usually less than 2K at equilibrium.

Detectors available for the system include photovoltaic Si, and InGaAs, photoconductive PbS (300K) and HgCdTe (77K). The chosen detector assembly (along with appropriate optics) is mounted behind a narrow slit on a platform on a precision calibrated rotary turntable (Société Genevoise PI-4). The swing is about 1 meter. An image of the spectrometer exit slit is formed at the detector slit.

Once the test prism is in place, the wavelength set and the temperature stabilized, the arm containing the detector system is rotated to locate the undeviated and deviated beams. The equation used to determine n using this MMD configuration is

$$ n = \frac{\sin(\alpha + \delta)}{\sin \alpha} $$

where α is the prism apex angle and δ is the deviation angle. This is similar to the equation used for minimum deviation, from which the MMD method is derived. The spatial variation of the output signal is the convolution of two narrow rectangle functions (spectrometer slit and detector slit), and appears Gaussian in shape. The nominal width of the Gaussian is 3 arc-min, and location of the peak to within 0.25 arc-min is quite difficult, especially in the presence of noise. Because of this issue, data are taken at regular (1-3 arc-min) intervals in angle around the centers of the undeviated and deviated beams. The method used to locate the "center" of the beam is to fit the data to a Gaussian using nonlinear regression techniques. Final data reduction consists of determining n and dn/dT vs. λ using conventional least-squares curve-fitting methods. The n vs. λ data can be fit to several models, including 2- and 3-term Sellmeier, Herzberger, Schott, etc. The dn/dT is usually fit with linear or parabolic equations. **All cryogenic index of refraction values reported are referred to *vacuum*;** any data taken in air has been corrected unless otherwise noted in the final measurement report.

A typical measurement report includes tables of raw and corrected data for each prism, along with graphs (corrected) for n vs. λ. Also included are graphs of n vs. T and dn/dT vs. λ. After fitting to the best model, a table shows the differences between the corrected and model data. If previous data are available in the open literature, a comparison is made between our data and the published data.

UNCERTAINTIES: There are several sources of uncertainty in these measurements. Most of them are dependent upon the value of n.

Apex angle measurement: This measurement on the Wild 79 spectrometer has a typical absolute uncertainty σ_{AA} of 2 arc-sec, contributing a systematic uncertainty in the index determination which is given by:

$$\frac{\sigma_n}{n} = \sigma_{AA}\left[-3.95 \times 10^{-6} + 4.41 \times 10^{-6}n - 6.82 \times 10^{-7}n^2\right]$$

For example, for a material with a nominal n of 2.5 and a measurement uncertainty of 2 arc-sec, the relative uncertainty in n is 0.000028. The following graph shows the translation from a 1 arc-second uncertainty in the prism apex angle to the relative uncertainty in the refractive index.

APEX ANGLE UNCERTAINTY
(for 1 arc-sec)
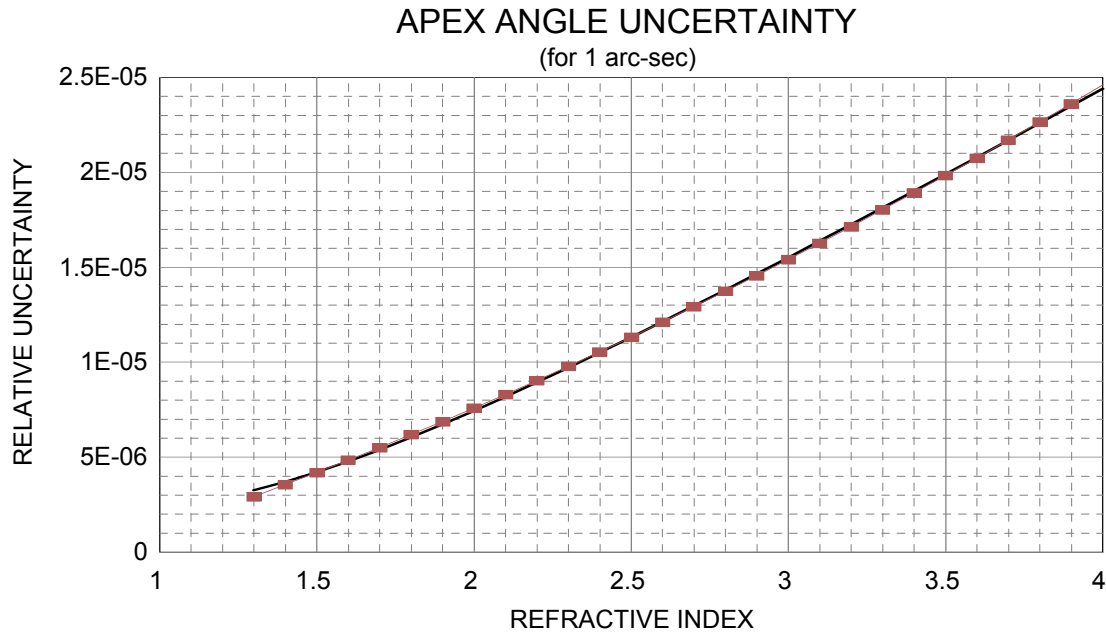
Autocollimation alignment: The retroreflected beam can be set within about 50 μm (one-fifth of the exit slit width), introducing a prism misalignment of 0.8 arc-minutes. This misalignment introduces a random uncertainty of 0.00008. It is slightly dependent upon n per the following equation:

$$\frac{\sigma_n}{n} = \sigma_{AC}\left[0.0002167 - 8.78 \times 10^{-5}n + 2.356 \times 10^{-5}n^2 - 2.27 \times 10^{-6}n^3\right]$$

This equation is for a misalignment of 0.8 arc-minute.

Stability of autocollimation alignment: When the temperature of the chamber is changed, the test prism has a tendency to shift and lose its initial alignment. A separate He-Ne laser is mounted external to the Dewar and directed to the entrance face of the test prism. The reflected beam travels to a target on the laboratory wall

at a distance of about 6 meters. The prism position can then be maintained during the measurements to within 0.4 arc-min, giving a random uncertainty of 0.00009. The appropriate graph is the same as the previous one.

Deviation angle measurement: Using the curve fitting technique described above, the estimated uncertainty in a single angle measurement is 12 arc-sec. The impact on index is dependent upon the nominal n as follows:

$$\frac{\sigma_n}{n} = \sqrt{2}\sigma_{DA}\left[-3.97\times10^{-6}+4.95\times10^{-6}n-6.25\times10^{-7}n^2\right]$$

Since there are two independent measurements (one for the undeviated beam and one for the deviated beam), the combined random uncertainty (the √2 term above) is 0.000076 at n = 2.5.

Temperature uncertainty: The uncertainty in the prism temperature is related to the recorded temperature of the mount (2 sensors), the thermal conductivity and diffusivity of the prism material along the settling time, and temperature gradients in the prism material due to conduction, convection and radiation. We usually allow a settling time of at least 10 minutes to ensure equilibrium. The temperature sensor calibration is within 0.5K for temperatures between 65 and 300K, increasing to 5K at 500K. The impact of this random and systematic uncertainty on index is dependent upon the dn/dT of the prism material and must be evaluated separately for each material.

Wavelength setting: After calibration with higher orders of a He-Ne laser, the wavelength uncertainty associated with the grating spectrometer is a maximum of 0.05% of the wavelength setting. The impact of this random and systematic uncertainty on index is dependent upon the dn/dλ of the prism material and must be evaluated for each material. The effect of changing the spectrometer bandpass (via slit width) is estimated to be too small to be significant.

Prism positioning: The location of the prism within the sample holder and the positioning of the sample holder with respect to the detector arm axis is another source of uncertainty. There are three axes of rotation and three orthogonal translation possibilities. The rotations are all adjustable at any time, and their effects are included in the autocollimation alignment. Two of the translations have been determined to have no measurable effect on the measured index. Translation along the optical axis has an effect, determined to be significant, requiring positioning to within 2 mm to keep the uncertainty in index less than 0.00005.

Effect of window wedge: When windows are required, they contribute an additional uncertainty due to their inevitable wedge. We have developed an *in situ* procedure to minimize this effect, rotating the windows in their mounts until their residual deviation contributions are in a plane perpendicular to the deviation plane of the test prism. The residual horizontal deviation is a systematic uncertainty estimated to be on the order of 0.2 arc-min. The impact on index is dependent upon the

nominal n. Since there are two independent measurements (one for the undeviated beam and one for the deviated beam), the combined systematic uncertainty is 0.00019 at n = 2.5.

Temperature and pressure corrections: Room-temperature and higher measurements are made in air, whereas cryogenic measurements are made in an evacuated dewar. Corrections must be made to translate from one condition to the other.

Correction of n vs. temperature to standard temperature (293K = 20°C)

Determine (from published data or actual measurement) the temperature coefficient of index of refraction (dn/dT) at or near the standard temperature and at the desired wavelength. Then the following equation is used.

$$n_{293} = n_{meas} + \left(\frac{dn}{dT} \right)(T_{meas} - 293)$$

Correction of n from air to vacuum

Edlen[1] gives an equation for the index of refraction of air as a function of temperature, pressure and wavelength:

$$n_{tp} = 1 + (n-1)_s \times \frac{0.00138823p}{(1+0.003671t)}$$

where p is pressure (Torr), t is temperature (Celsius) and $(n\text{-}1)_s$ is the spectral refractivity of air under standard conditions. At sea level, the term $(n\text{-}1)_s$ is 0.00028 in the visible portion of the spectrum and 0.00027 in the infrared. The average atmospheric pressure in Tucson (el. 762 meters) is 686 Torr. Temperatures in the laboratory typically range from 20°C to 25°C. The refractive index of air for the range of measurement conditions is shown in the table below:

LOCATION	TEMP (Celsius)	PRESSURE (Torr)	n_{AIR}
Sea level	20	765	1.000277
Tucson (762 m)	20	686	1.000248
Tucson (762 m)	22.5	686	1.000246
Tucson (762 m)	25	686	1.000244

To obtain the absolute refractive index from measurements made in air, the observed values must be multiplied by the above values of n_{air}. The residual systematic uncertainty introduced is 0.000005.

The estimated absolute total uncertainty (k = 1) of these measurements for nominal n = 2.5 and n = 4 are given in Table 1.

Table 1. Typical index of refraction absolute uncertainties (k = 1)

ERROR SOURCE	ERROR (n=2.5)	ERROR (n=4)
Apex angle measurement	0.000057	0.000192
Autocollimation alignment	0.000087	0.000078
Stability of autocollimation	0.000087	0.000078
Deviation angle measurement	0.000076	0.000102
Temperature uncertainty	0.000020	0.000020
Wavelength setting	0.000060	0.000060
Prism positioning	0.000050	0.000050
Effect of window wedge	0.000192	0.000406
Pressure correction	0.000050	0.000050
RSS TOTAL	**0.000265**	**0.000436**

For measurements made in air (without the window wedge uncertainty) on a prism with a nominal index of 4, the overall rss uncertainty is 0.00016. On a recent measurement of Schott SF6, we observed uncertainties of less than 0.0001 at room temperature in vacuum (in the Dewar) when compared to the published values from Schott, indicating that these estimates may be conservative.

It is more difficult to estimate the uncertainty in $\Delta n/\Delta T$. The relative uncertainties that we have observed as a result of multiple readings over temperature are on the order of 0.05 of the calculated value for $\Delta n/\Delta T$.

1. B. Edlen, "*The Refractive Index of Air*," Metrologia 2, 71 (1966).

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