

Membrane Patch Colourimetry & i-LAB Quick Start Guide

i-LAB method for measurement of the Membrane Patch Colourimetry is an exclusive application to CleanOil LLC. It uses methods developed to measure color and calculate the delta-energy (δE) difference between a new (clean) patch and the sample patch. This is done by the application of a surface reading adapter for the i-LAB spectrometer.

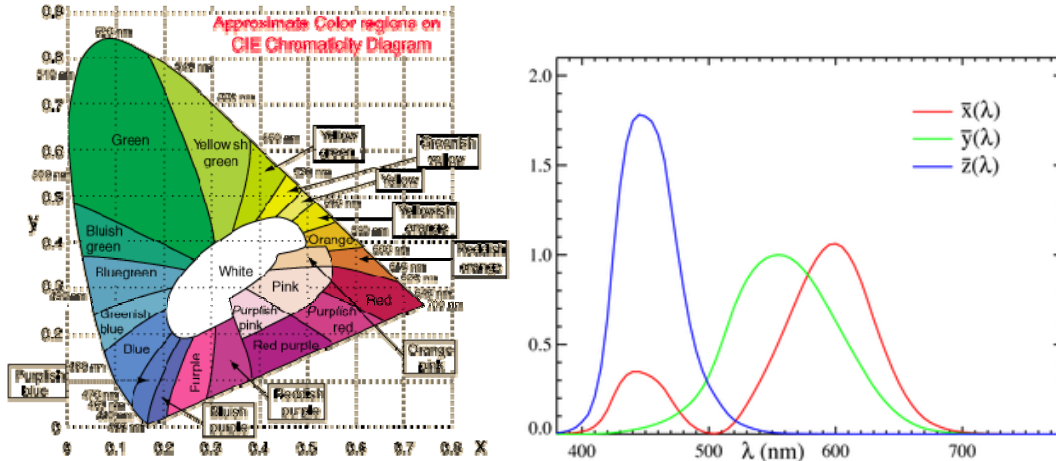
i-LAB Measurement

The iLAB has been designed with a built-in source, sampler and detection system. It also contains embedded software designed to control the spectral acquisition, storage, processing, reporting and external data output. The procedure of performing and combining these tasks is defined by a series of processing steps described below.

The i-LAB measures the visible spectrum of a sample through the use of a Light Emitting Diode (LED) light source and Linear Variable Filter (LVF) detector. The light source is a series of LEDs designed to cover the full spectral range and have a balanced intensity to produce maximum performance. There are two white lights and one blue, which are turned on at different times to develop an optimized full visible spectrum. The LVF detector is integrated with a 128 pixel CMOS array.

For the surface analyzer method, the light source is reflected down the inside of the surface adapter to illuminate the surface of the sample. This illumination has a corresponding absorption and reflection color characteristic that is recorded on the other side of the surface adapter by the detector. Similar to any other light spectral measurement, a background or reference spectrum is first obtained. This reference sample must reflect "white" light back to the detector because white light is the color of all wavelengths reflecting back. The sample being analyzed should absorb different wavelengths causing a change when compared with the reference. The sample spectrum and background spectrum are processed together to yield what is known as a transmission spectrum. This spectrum is stored and used for further processing. Overall, this means a white sample should produce a 1.0 spectrum and a black sample will produce a 0.0 spectrum. But, as with normal spectral measurements, there are intensities of white and black that can be measured between the 1.0 and 0.0 values.

This reflectance mode operates no different from a cell or transmission mode. Light is sent through the sample in the transmission mode, whereas, light is reflected to the detector in reflection mode. In both cases the sample will absorb portions of the light and allow other portions to continue to the detector. What arrives at the detector is the spectrum. For example, a result that reads red, does so for one of two reasons: 1) either the red color comes through the solution or 2) every other color is absorbed and



red is reflected to the detector.

There are numerous methods of processing spectral measurements. The simplest is visual observation of the spectrum. When intensity and wavelength can be observed, one gains significant qualification and quantification of the sample's color via visual observation.

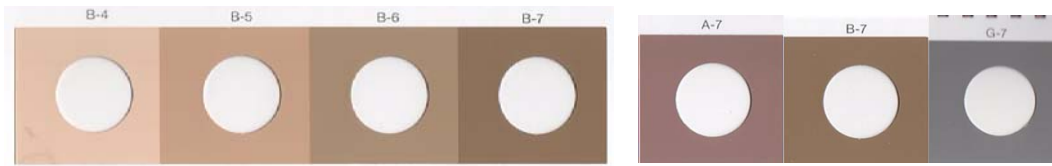
To increase the sophistication of the analyses, the observer can study one or more peak and relate this observation to the concentration of a specific component. This correlation, however, does not study the overall color of the spectrum. The CIELAB measurement of color is an attempt to reduce this color spectrum into a simpler set of numbers. The default surface method produces these numbers through a series of calculations.

Definition of Surface calculations:

Surface Colorimetric measurements can be quantified by the use of ASTM E308 parameters. This standard defines the CIELAB parameters. The parameters used to define an observed color are the three main values representing the visual color space. This color space is defined with "a*", "b*" and "L*". The a* parameter is the change in color in the red spectrum and is defined positive in the red direction and negative in the green direction. Whereas, b* is the change in color of the yellow spectrum and is positive in the yellow direction and negative in the blue direction. The L* parameter is the change in shade from white to black. As mentioned previously, these parameters are calculated against a standard white sample spectrum, such a calculation ensures that the spectrum is always compared to what the user defines as "White". These values are based off the CIE tristimulus values X, Y, and Z, which are similar to the RGB values in printing and computer screens. RGB values are not being used for determination

simply because there are no defined standards for RGB. From the visual spectrum of the sample and the reference “white” sample spectrum, the tristimulus values of the two samples can be calculated. These tristimulus values can then be used in conjunction with one another to calculate the color space parameters a^* , b^* and L^* .

Examples of CIELAB Data



	L^*	a^*	b^*
B4	82.5	7.0	15.5
B5	66.7	9.6	19.6
B6	67.3	3.9	12.3
B7	50.7	4.2	13.2
A7	72.2	5.3	1.8
B7	50.7	4.2	13.2
G7	65.8	2.7	-1.8

In this example above, we observe a steady increase in the darkening of color in the B-series with concomitant lowering of the L^* ; The a^* and b^* parameters are darkening as well. Further investigation in the A-B-G7 series makes it evident that the darkening varies as does the darkening of other colors. Even though B7 looks darker it has a higher b^* value which indicates that it is more yellow than blue. Using the same principle we can deduce that the sample is also more green than red.

i-LAB Loaded Methods

The *i-LAB* has three methods preloaded for measurement of membrane patch colorimetry. The patch should be generated as per ASTM D02 C01- WI 13070

Methods:

MPCbackg – Membrane Patch Colorimetry Background Measurement

- This method is used to measure and store the background (or clean patch) spectral data.
- It should be run before measuring the sample patch
- A clean patch should be measured periodically

MPC_dE – Membrane Patch Colorimetry Measurement

- This method which is used to measure, reports to the i-LAB screen and stores the a CIE delta energy (CIE_dE) value.
- The delta energy (CIE_dE) value is calculated against the clean patch data stored from the previous method MPCbackg.
- The CIE_dE value is based on a D65 light source and 10 Degree Observer – using the calculations in ASTM E308.
- The log data can be extracted into Excel using the program i-LAB Datalog

CIE_dE – Membrane Patch Colorimetry Measurement

- This program is identical to the MPC_dE method except that all the testing data is saved.
- Instead of just saving the CIE_dE value to the log, this method also stores the spectra and all the CIE variables to the Results Log.
- This method is typically used for further research method development.
- The log data can be extracted into Excel using the program i-LAB Datalog

Measuring Membrane Patch Colorimetry Results

Install the surface measurement adapter/head onto the i-LAB. The adapter should be installed such that the two indents fit snugly against the bracket of the i-LAB. Before each measurement, the user should confirm that the adapter is properly installed. Turn on the i-Lab and press the Menu button to bring up the Menu. Press the Center button to bring up the methods stored on the i-LAB.

Use the up-button or down-button to pick the method to be run. Press the center button to activate method.

Place the sample patch to be measured on a flat piece of aluminum foil with the color (sample) side up. Light from the measurement can be reflected through the patch material itself, therefore it is important for the user to have a consistent backing of the patches. Aluminum foil is, in this case, that consistent backing.

Place the i-LAB onto the sample where the surface adapter is only covering the colored (sample) portion. Press the action (center) button on the i-LAB to start the method's measurement. The results of the sample measurement will be displayed on the i-LAB screen when the measurement is complete. By pressing the Center-Action Button the results will be saved and you will be returned to the Method Menu. The data is stored in the log of the i-LAB by the time and date of analyses. Keeping track of this information will allow proper results storage and retrieval.

If the i-LAB is not properly placed on the patch, a Scan error “Saturation has occurred” will appear on the i-LAB screen. This message is designed to let the user know when a mistake in analysis has occurred. Re-seat the adapter and the i-LAB on the sample and repeat the analysis.

A background or reference sample should be chosen that is as pure white as possible. One should always use this same background sample for consistent sample results. The method **MPCbackg** should be used to measure this background sample before running a sample measurement. The samples are then measured using the method **MPC_dE** or **CIE_dE**. Although this instrument is stable, it is a good practice to periodically rerun the background sample to insure no instrumental changes have occurred since the last referencing.

Extracting Data from i-LAB’s Log.

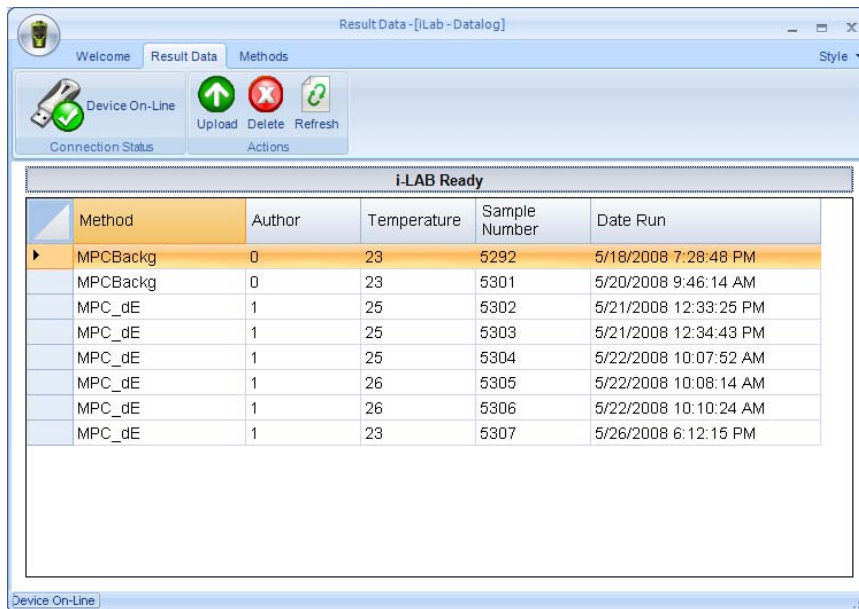
The log data from membrane patch colorimetry (MPC) measurements can be retrieved or downloaded from the i-LAB into a CSV file (read by Excel) using the supplied software “i-LAB Datalog”.

Upon opening of the software, the screen will be displayed showing an option of uploading log data or downloading a method. Clicking on the “Results Data” will activate the log upload.

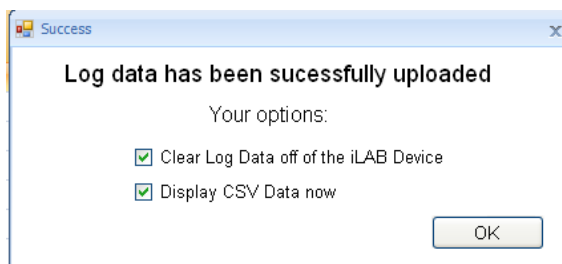


When the data upload form appears the program will load sample results into the window. This will take a minute or so depending upon the number of results on the iLAB.

After the results have been loaded, the user may choose to upload these results into a CSV file by clicking the “Upload” button.



This action will open the form below, which gives the user the options of clearing the data off the i-LAB after upload or leaving it stored. It also allows the option of displaying the CSV data just uploaded.



The uploaded data will be posted into the file:
 “My Documents\iLABResultData.CSV”

If Excel is the default CSV reader program, it will be opened to display the data.

The output results format for the MPC_dE method data is:

STD_DEV	METHOD NAME	TEMPERATURE	SAMPLE NUMBER	DATE RUN	TIME RUN
24.09348	MPC_dE	24	173	5/31/2008	5:07 PM

The column labeled “STD_DEV” is the “CIE delta energy” result. This is from the i-LAB variable used to originally store this calculated variable.

The output results format for the CIE_dE method data is considerably more complex, since all the data used in the calculation is stored. This method reports the data as:

XSPECTRUM_UDF	YSPECTRUM_UDF	USER_VAR1	USER_VAR2	AREA	Y_PEAK	USER_VAR1
400	1.4504	90.20621	97.73578	99.12942	-4.307985	139.4608
401	1.4409					
402	1.4291					
403	1.4157					
404	1.3991					
405	1.3802					
406	1.3621					
407	1.3388					
408	1.317					
409	1.2885					
410	1.2566					

Continuation of Row 1

Y_VALUE	USER_VAR1	USER_VAR2	Y_MAX
-19.81577	91.49918	96.62742	98.693

Continuation of Row 1

Y_MAX	USER_VAR1	Y_MAX	STD_DEV
-0.0899434	103.4062	0.1294613	20.39104

Continuation of Row 1

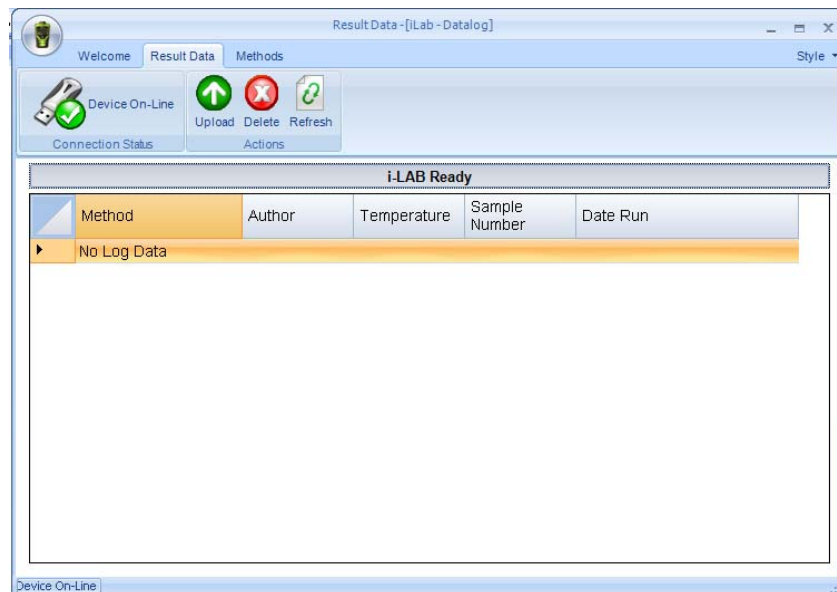
METHOD NAME	TEMPERATURE	SAMPLE NUMBER	DATE RUN	TIME RUN
CIE_dE	25	173	5/31/2008	5:20 PM

The column definitions for these results are:

- XSPECTRUM_UDF = X-spectral values for the sample spectrum (nm)
- YSPECTRUM_UDF = Y-spectral values for the sample spectrum (transmittance)
- USER_VAR1 = CIE X Sample
- USER_VAR2 = CIE Y Sample
- AREA = CIE L* Sample
- Y_PEAK = CIE a* Sample
- USER_VAR1 = CIE Z Sample

- $Y_VALUE = CIE\ b * Sample$
- $USER_VAR1 = CIE\ X\ Blank$
- $USER_VAR2 = CIE\ Y\ Blank$
- $Y_MAX = CIE\ L * Blank$
- $Y_MAX = CIE\ a * Blank$
- $USER_VAR1 = CIE\ Z\ Blank$
- $Y_MAX = CIE\ b * Blank$
- $STD_DEV = CIE\ \text{delta Energy}$
- $METHOD\ NAME = \text{name of the method use to generate this data}$
- $TEMPERATURE = \text{temperature of the i-LAB during the test measurement}$
- $SAMPLE\ NUMBER = \text{chronological sample number}$
- $DATE\ RUN = \text{date sample was run}$
- $TIME\ RUN = \text{time sample was run}$

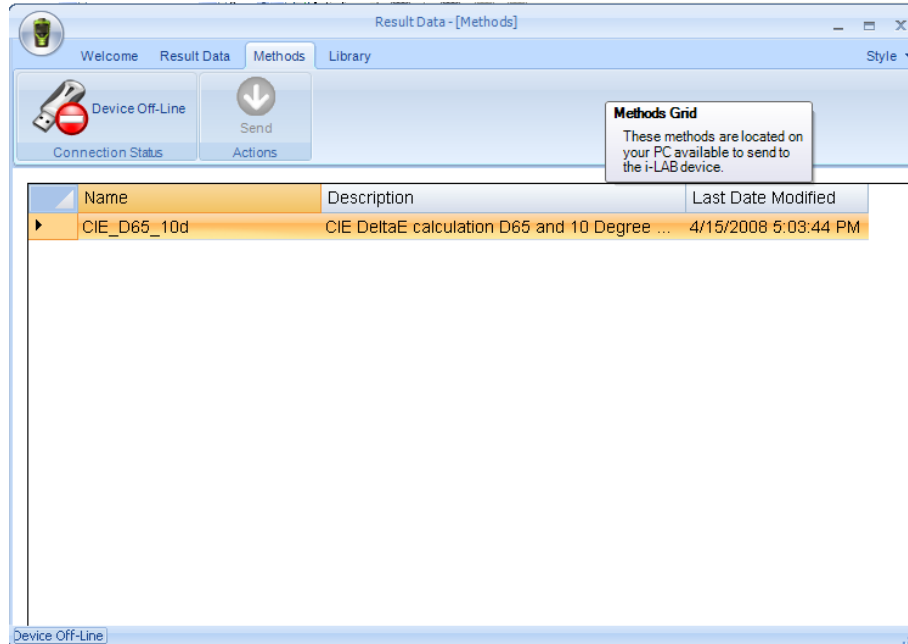
If the data has been cleared from the i-LAB or there is no data currently stored in the i-LAB's log the program will display that no data is in the log.



Updates or Upgrades

If an update for the i-LAB Datalog software exists and the user's computer has internet access, there is an auto-update function built-in the software. This auto-update function will check for any updates and notify the user when the program is first opened of this update and that it is available to be loaded.

Method or i-LAB firmware is also capable of being updated if newer revision becomes available. This includes the addition of new methods for i-LAB.



Interpretation of CIE_dE data for the Membrane Patch Colorimetry

There are four ranges of Varnish Potential Rating Severity:

1. **Normal range:** This indicates that there are low levels of the precursors that lead to soft contaminants (varnish).
2. **Monitor range:** This means the production of varnish within the system could be approaching soon and the oil condition should be watched. Typically samples in this range will not show varnish problems already, however they may start forming varnish when the lubricant is cooled to an ambient temperature. If the antioxidants have been depleted, the formations of varnish precursors are forming at a reasonably high rate. As a result, it would be expected that the condition of this lubricant would deteriorate quickly.
3. **Abnormal range:** This means that there is an increased level of soft contaminants due to oil degradation. These soft contaminants will form varnish in tight clearance zones or cooler spots in the system. The oil may also cause servo valves to stick or seize causing a unit trip, especially after a shut-down when the unit is being brought back online. Inspect filters and reservoir (when possible) for signs of varnish formation and bearings for temperature increases. Pay particular attention to Last Chance Filters and pencil strainers before the hydraulic circuit's valve blocks.

4. **Critical range:** This means that there is an increased level of soft contaminants due to oil degradation. These soft contaminants will form varnish in tight clearance zones or cooler spots in the system. The oil may also cause servo valves to stick or seize causing a unit trip, especially after a shut-down and the unit is being brought back online.

These ranges are defined by the color on the patch. The more color, or higher the CIE delta energy values, the high the varnish potential. A general range has been developed by CleanOil for these four categories:

Normal Range: ≤ 15 CIE_dE

Monitor: 15 to 30 CIE_dE

Abnormal: 30 to 40 CIE_dE

Critical: ≥ 40 CIE_dE