

	<b>Standard Operating Procedure</b> <b>Use and Calibration of Digital Refractometer</b>		<b>SOP Number</b> <b>D-714</b>	<b>Revision</b> <b>2</b>
			<b>Effective Date</b> 01/24/23	<b>Page</b> <b>Page 1 of 6</b>
<b>Written by/ Date</b> KBurns 01/20/23		<b>Reviewed by/ Date</b> SAS 01/23/23		<b>Approved by/ Date</b> SS 01/23/23
<b>Title: Quality Assurance Director</b>		<b>Title: Analytical Development Scientist</b>		<b>Title: Quality Control Director</b>

## 1.0 Purpose

This procedure provides instructions on the use and calibration of the QC Laboratory refractometer(s).

## 2.0 Scope

This procedure applies to all dietary supplement, pet product, cosmetic, and pharmaceutical raw materials and finished products used or manufactured at Ion Labs that require refractive index testing.

## 3.0 Responsibility

- 3.1 It is the responsibility of QC and Analytical Chemists to follow this procedure.
- 3.2 It is the responsibility of QC Laboratory Management to implement this procedure and to ensure that the procedure is being followed.
- 3.3 It is the responsibility of QC Laboratory Management and/or Analytical Development to keep this procedure aligned with current practices.

## 4.0 Definitions

- 4.1 **Refractometer** – An instrument for measuring the refractive index of materials.
- 4.2 **Refractive Index (RI)** – (of a material/substance) A measure of the speed of light in that substance. It is expressed as a ratio of the speed of light in vacuum relative to that in the considered medium.
- 4.3 **IPA** – Isopropyl alcohol

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- 4.4 **ETOH** – Ethanol
- 4.5 **MeOH** – Methanol
- 4.6 **ppb** – parts per billion
- 4.7 **nD** – Index of Refraction
- 4.8 **TC** – Temperature corrected
- 4.9 **BRIX** – % sucrose in aqueous solution

## **5.0 References**

- 5.1 Instruction manual, Cole-Parmer “Handheld Digital Refractometer”
- 5.2 D-714-F1, Form, Refractive Index Test Ticket
- 5.3 G-201, SOP, Calibration Program

## **6.0 Procedure**

- 6.1 Set Up - General
  - 6.1.1 Battery Installation
    - 6.1.1.1 Remove battery door by removing the screws attaching the battery door then remove the battery door.
    - 6.1.1.2 Install batteries, negative side into cavity first, positive facing out.
    - 6.1.1.3 Re-install battery door by placing the battery door back into position and affixing it with the screws
  - 6.1.2 Cleaning

- 6.1.2.1 Clean and dry sample well and measuring surface immediately after each reading. Never immerse the instrument in any liquid. When the measurement surface and well have been completely cleaned no residue should be present.
- 6.1.2.2 To properly clean the sample well and glass measurement surface use a mild soap and water solution followed by a distilled water rinse and then thoroughly dry with a soft lint and residue clean cloth or product such as Kimwipes. For oily samples IPA, ETOH, or MeOH can be used initially to remove residue followed by mild soap and a distilled water rinse.
- 6.1.2.3 To clean the refractometer's body use a soft cloth with a mild cleaning solution like window cleaner applied to it and wipe it clean. Never use any harsh cleaning agents that will damage the instrument such as acetone, N-Nimethylacetamide, cresols, lacquer thinners, benzene, gasoline, etc.
- 6.1.3 Calibration: Zero-setting (Before Use)
- 6.1.3.1 Calibration of the Cole-Palmer Hand Held Digital Refractometer should occur daily or just before use.
- 6.1.3.2 Clean the measuring surface and well as outlined under "Cleaning."
- 6.1.3.3 Apply a few drops 18.2 $\Omega$  Millipore water with TOC  $\leq$  5ppb at about 20°C to the prism.
- 6.1.3.4 Press "Zero" to start calibration
- 6.1.3.5 The unit shows the process of zero setting
- 6.1.3.6 If calibration is successful, it will show "Pass"

6.1.3.7 If calibration is not successful, it will show “Fail”

6.1.3.7.1 If calibration “Fails”, re-calibrate using a fresh water sample.

6.1.3.7.2 If calibration fails for a second time, the instrument should be re-cleaned before attempting to re-calibrate.

6.1.3.7.3 If calibration fails for a third time the instrument should be removed from use.

#### 6.1.4 Changing Scales

6.1.4.1 To change reading mode between RI and BRIX press and hold the “READ” key to toggle through those choices.

### 6.2 Sample Analysis

6.2.1 Samples will be tested at ambient conditions. Specification ranges are adjusted to allow a small variation in temperature during sample analysis.

6.2.2 Calibration: Zero-setting as per 6.1.3 should be prior to the sample analysis to ensure the greatest accuracy.

6.2.3 Before sample analysis inspect the glass measuring surface and well to be sure that there is no residue remaining from a previous sample analysis. Refer to 6.1.2 as necessary for cleaning instructions.

6.2.4 Extract the sample to be tested and apply a few drops of sample to the sample well to completely cover the glass measuring surface.

6.2.5 Allow the sample to reach the same temperature as the instrument. Failure to do so will result in inaccurate measurement.

6.2.6 Data Collection:

6.2.6.1 Press the “READ” key to take a sample measurement.

6.2.6.2 The result will be shown on screen and results will be stored for up to 60 seconds.

6.2.6.3 Press the “POWER” button to recheck previous measurements.

6.2.7 Document the results and required information on form D-714-F1Refractive Index Test Ticket.

6.2.8 Clean the prism per section 6.1.2 after each measurement

## 7.0 Specifications

7.1 RI Range: 1.3330 – 1.5400

7.2 BRIX Range: 0 – 95%

## 8.0 Alternative Instruments

8.1 If an alternative refractometer needs to be used, the manufacture operational manual will be followed for analysis.

## 9.0 Revision History

Revision	Date	Description of Changes	CCR #	By
0	06/16/16	New	16-0601	B. Johns
1	10/07/19	Revised SOP to reflect operation, calibration, and maintenance of new Cole-Parmer Refractometer. Updated D-714-F1 to reflect new changes in SOP.	19-0732	I. Garrett
2	01/20/23	Schedule review: update logo and document format. Revise responsibilities section.	CC-23-0035	K. Burris

## 10.0 Attachments

10.1 Attachment 1 – Temperature Coefficient Calculation

### Attachment 1: Temperature Coefficient Calculation

To calculate the temperature coefficient of a sample, the sample must be read in the refractive index mode without temperature correction. The sample must then be read at a second temperature. The following calculation may then be used.

$$\text{Temperature Coefficient} = (nD1 - nD2)/(T1 - T2)$$

Where:

nD1 = Refractive Index of sample at temperature 1

nD2 = Refractive Index of sample at temperature 2

T1 = Temperature of Refractive Index reading 1

T2 = Temperature of Refractive Index reading 2

The temperature coefficient is almost always a negative number. Refractive index increases as temperature decreases. Typical temperature coefficients for fluids will be from -0.0001 to -0.0004.

Once a temperature coefficient is known, the temperature corrected refractive index can be calculated from an actual refractive index reading by the following formula:

$$\text{RITC} = ((RT - AT) * TC) + RI$$

Where:

RITC = Refractive Index Temperature Corrected

RT = Reference Temperature

AT = Actual Temperature as read on instrument

TC = Temperature Coefficient of sample

RI = Actual Refractive Index as read on refractometer



**Refractive Index Test Ticket**

Form: D-714-F1

CCR No. CC-23-0035

Revision: 2

**Sample Prep**

<b>Sample Name:</b>	
<b>Sample Batch / R Number:</b>	
<b>Sample SKU / RMID</b>	
<b>Test Date:</b>	

**Equipment**

<b>Refractometer Ion ID #:</b>		
<b>Calibration Results:</b>	<input type="checkbox"/> Pass	<input type="checkbox"/> Fail
<b>Temperature (°C):</b>		
<b>Thermometer Ion ID #:</b>		
<b>Calibration Due:</b>		

**Data / Parameters**

<b>Refractive Index:</b>	
<b>Specification</b>	

Determination (circle one):                      **Pass**                      **Fail**

Comments:

Performed By/Date: \_\_\_\_\_

Reviewed By/Date: \_\_\_\_\_