Guide to the Corn Refiners Association, Inc. RI-DS Program

Version 4 – July, 2020

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CONTENTS

Introduction

The RI-DS Program is an Excel® -based program for personal computers using the Windows operating system which allows generation of tables relating dry substance and refractive index for corn syrups, high fructose corn syrup and blends of these products with sucrose. In conjunction with the refractive index method included in this manual, the RI-DS Program may be used to determine the dry substance of corn syrups of known composition.

Development of Dry Substance Measurement for Corn Syrups

The measurement of dry substance (DS) of commodities containing water is an integral part of trade in a wide variety of common food and industrial products. In the trade of corn syrups, high fructose corn syrups and blends of these products with sucrose, accurate knowledge of dry substance is important for both commercial consideration and for proper use of the product by the customer. Since corn sweeteners are now a substantial portion of the sweeteners used in food around the world, simple, fast and accurate means of determining dry substance are vitally important to corn refiners and the food industry.

Traditionally, dry substance of both corn sweeteners and liquid sugars were measured by use of hydrometers. For corn sweeteners, DS was reported in degrees Baume', while sucrose hydrometer readings were reported in degrees Brix. Tables relating Baume' to dry substance were first published in 1943.

These tables served the industry well, until advances in technology rendered the Baume tables incomplete. The first advance was the enzymatic hydrolysis of starch in the 1950s. By allowing for the production of diverse compositions at the same DE, the original tables became invalid. From 1965-1975, the isomerization of D-glucose to D-fructose, and their separations by chromatography on an industrial scale, increased the variety of available saccharide compositions, further eroding the validity of the tables.

Prompted by these developments, the Corn Refiners Association, Inc. sponsored three updates to the dry substance tables. In 1976, the Association sponsored an update, based on refractive index, for glucose syrup and the first high fructose corn syrup (HFCS 42). The 1980 update, again based on refractive index, developed tables for all fructose syrups and their most common blends with sucrose and invert sugar syrups. Re-measuring and reconciling previous sample types and data in terms of density and specific gravity lead to the 1984 tables. This work has been fully described in the scientific literature listed in the reference section.

The laboratory operation of refractometers for the determination of dry substance in corn syrups is described in Standard Analytical Method REFRA..01 published by Corn Refiners Association, Inc. and reprinted in this manual.

Because corn syrups are composed of mixtures of saccharides with different physical properties, accurate knowledge of composition is essential to determine dry substance using the refractive index method. Users may use the RI-DS computer program to determine refractive index-dry substance relationships, as well as commercial Baume', specific gravity and weight per gallon of syrup either by selecting from a menu of typical product compositions offered by corn refiners, or by entering customized compositional data into the program.

Downloading the RI-DS Program

1. Hardware and Software Requirements. This program has been tested with Microsoft Windows operating systems through Windows 10, and Microsoft Excel through Microsoft 365 Version MSO 16.0 (09/2019).

2. Download the program from the Corn Refiners Association, Inc. website (www.corn.org) by clicking on the link "RI-DS Program" at: www.corn.org/policies/product-safety-quality

3. Clicking on the link will automatically download a Zip file (CRA_RI-DS-Version4.Zip) to your download folder.

4. Create a new folder where you will store the RI-DS program and files. Copy the Zip program to this directory and click to expand the files in the Zip program to the new directory. Four new files will be created:

   CRA RI-DS Version4.XLS
   CRA RI-DS Version4.XLT

   CRA RI-DS Version4 (SI units).XLS
   CRA RI-DS Version4 (SI units).XLT

   The two program files (XLS) are identical except that the SI program files displays results in metric SI (Système international d'unités) units.

   Both the XLS and XLT files for each program are necessary to run the RI-DS program successfully. Make sure that they remain in the same folder on your computer. They will automatically connect to one another, and do not require you to create an association.

Starting the RI-DS Program

1. Before starting the program, have ready the following information:

   Designation of Syrup (trade name, DE description or other identification) Sulfated Ash content (Dry Basis)

   Saccharide Composition (as % of total saccharide content) Fructose content (%)
   Sucrose content (%)
   Dextrose content (%)
   Maltose (DP_2) content (%)
   Maltotriose (DP_3) content (%)
   Higher Saccharides (DP_4+) content (%)

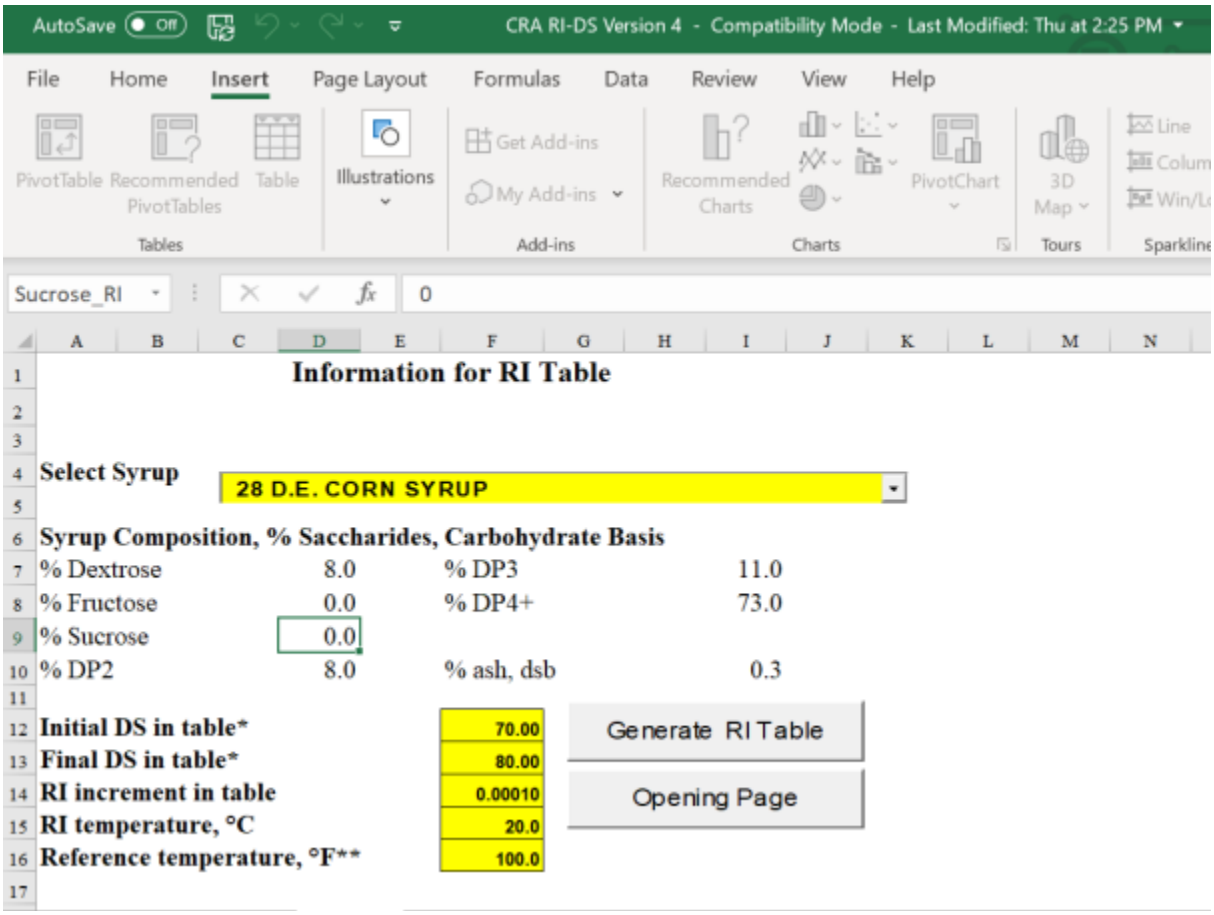
2. To start the RI-DS Program, open Excel and double-click on the .XLS file. (The .XLS file does not need to be opened.) Depending on which version of Windows/Excel is being used you will be asked to permit the he program to run macro files. Upon opening the program, you may see the screen above. The RI-DS Program will not run unless you enable macros. After you enable macros you will be taken to the title page.

3. On the title page, you will be given the option to select one of the following: RI Table, DS Table, Brix Table, Individual Calculations or Syrup Definitions.

4. Select your choice by clicking on the corresponding button, and then turn to the section of this manual with that title for further information.

Refractive Index (RI) Table

1. To use the RI Table function, click on the corresponding button on the title page of the program. This will bring you to the RI Tables sub-menu, which looks like this:



2. From this menu, you can generate RI tables of standard compositions of corn syrup, or return to the home page, by clicking the **Opening Page** tab at the bottom of the screen. To create a
RI table click on the arrow of the **Select Syrup** drop down menu. To create a RI table with the default characteristics listed in the yellow boxes, press the **Generate RI Table** button. You will see the program working, indicated by the activity in the calculation bar. After a few seconds, a table will generate. This table will have the syrup type and characteristics labeled at the top of the page. The number of tables you generate per session will determine the name of each table you generate. For example, if this is your first RI table, the program will name this CRA RI-DS Version 21.xls. Subsequent versions will be saved as CRA RI-DS Version 22.xls, CRA RI-DS Version 23.xls etc.

3. At this point, you can either print the table, save the table, or return to the opening page. To print the table, you can either click on the printer icon in the tool bar, or click on **File**,

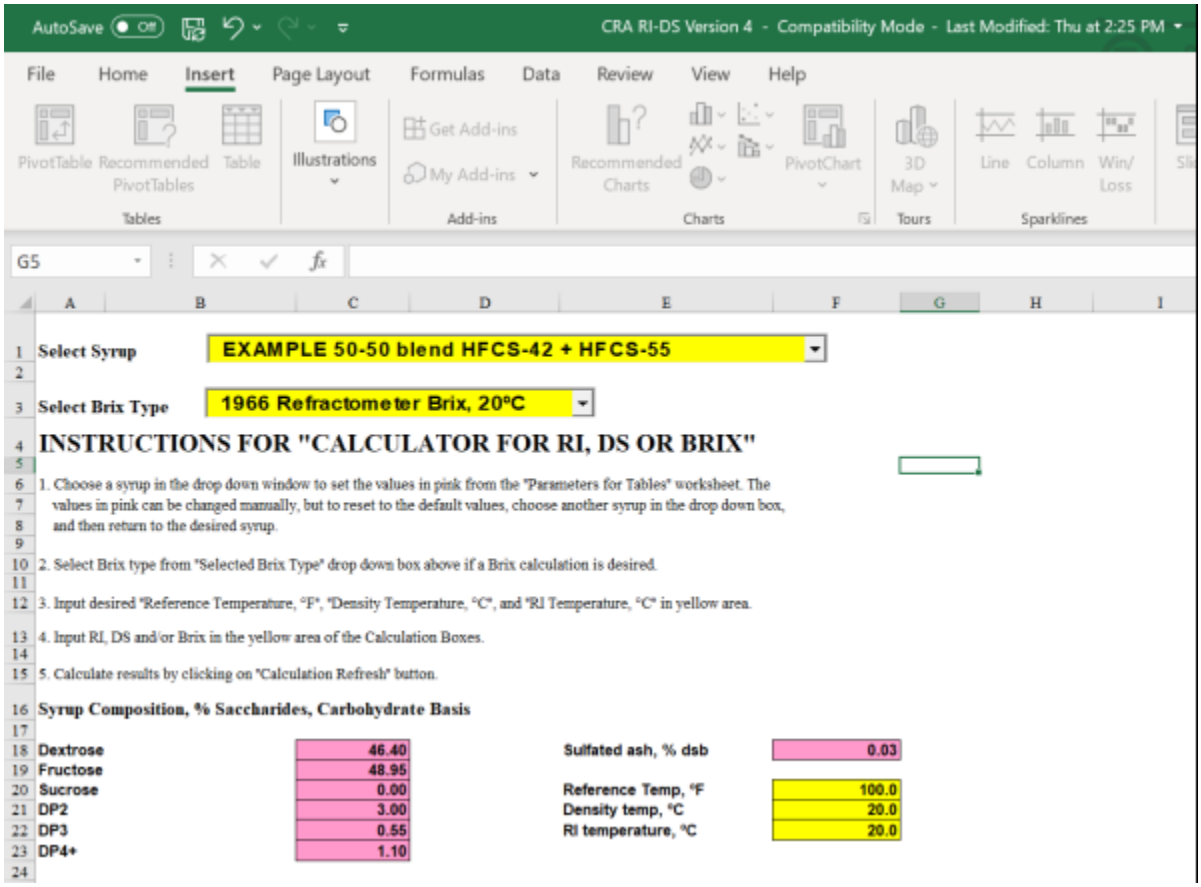
then
Print from the drop-down menu. The **Opening Page** button will not print on the table.

4. To save these files, click on the diskette icon in the toolbar, or click on **File**, then select **Save** to save with the current name and directory, or **Save As** to change the file name or target directory.

5. To return to the opening page, simply press the **Opening Page** button.

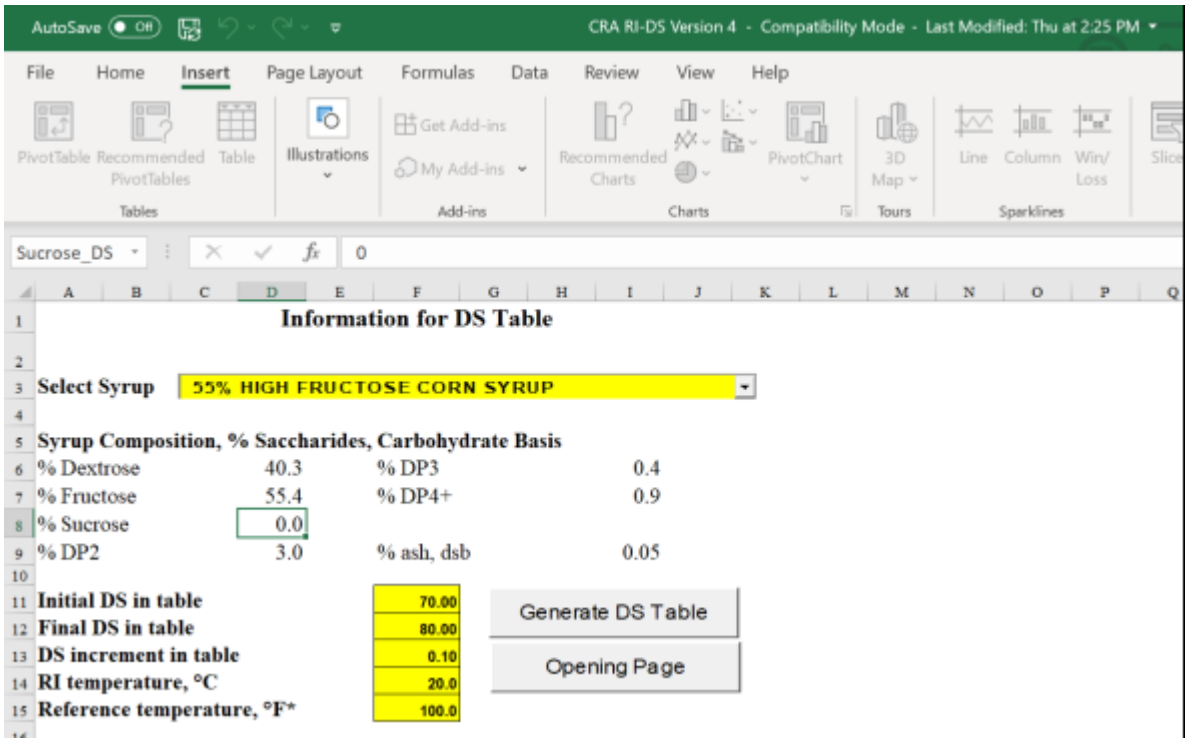
6. You can also create custom tables, you may change any of the following parameters in the yellow boxes: **Starting DS in Table, Final DS in Table, RI Increment in Table, RI Temperature, C and/or Reference Temperature, F**. This is done by selecting the yellow cell beside the parameter of your choice and re-typing the information you wish the table to reflect.

7. Additionally, you can create RI Tables for custom blends of syrups, by first creating a new syrup definition in the **Syrup Definition** section of the program (see this section of the manual for further details). Once created, you can select this from the **Select Syrup** drop down menu and proceed as normal **DS Tables**.



Dry Substance (DS) Tables

1. To use the DS Table function, click on the corresponding button on the title page of the pro- gram. This will bring you to the DS Tables sub-menu, which looks like this:



2. From this menu, you can generate DS tables of standard compositions of corn syrup, or return to the home page, by clicking the **Opening Page** tab at the bottom of the screen. To create a DS table, click on the arrow of the **Select Syrup** drop down menu. To create a DS table with the default characteristics listed in the yellow boxes, press the **Generate DS Table** button. You will see the program working, indicated by the activity in the calculation bar. After a few seconds, a table will generate. This table will have the syrup type and characteristics labeled at the top of the page. The number of tables you generate per session will determine the name of each table you generate. For example, if this is your first DS table, the program will name this
CRA RI-DS Version 21.xls. Consequent versions will be saved as CRA RI-DS Version 22.xls, CRA RI-DS Version 23.xls etc.

3. At this point, you can either print the table, save the table, or return to the opening page. To print the table, you can either click on the printer icon in the tool bar, or click on **File**, then
Print from the drop-down menu. The **Opening Page** button will not print on the table.

4. To save these files, click on the diskette icon in the toolbar, or click on **File**, then select **Save** to save with the current name and directory, or **Save As** to change the file name or target directory.

5. To return to the opening page, simply press the **Opening Page** button.

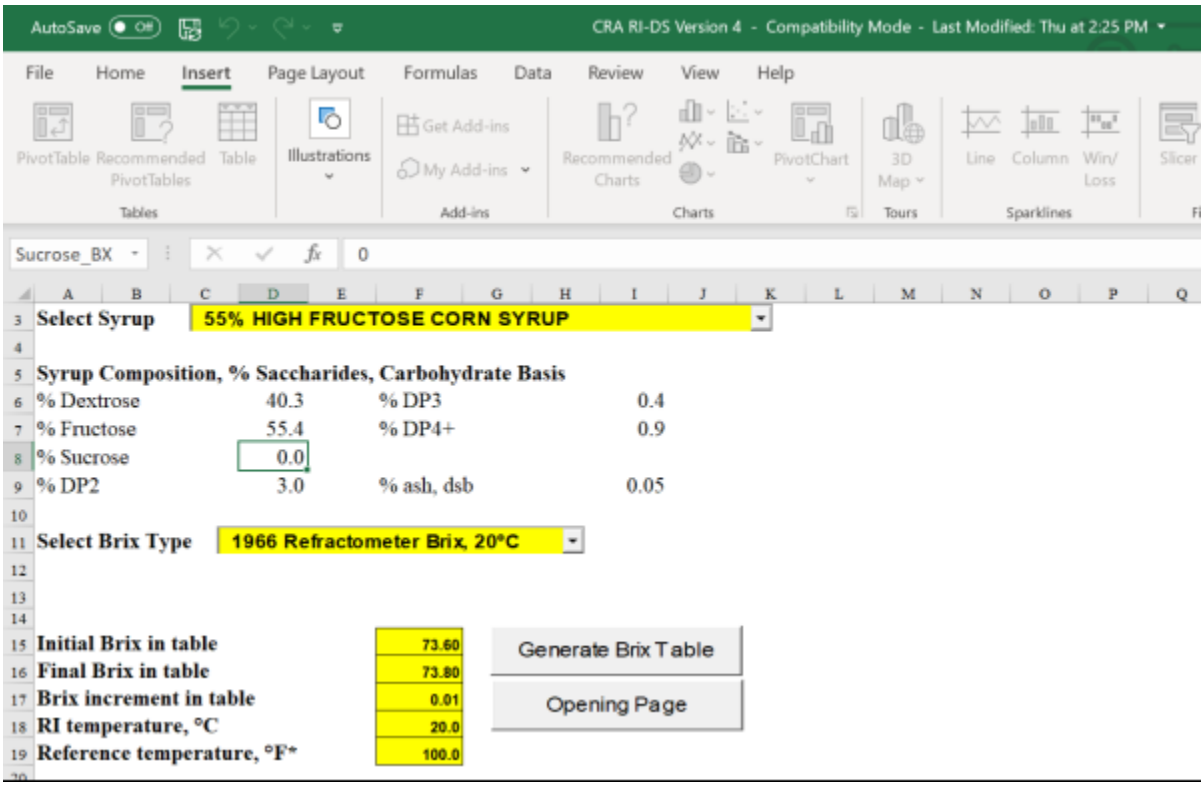
6. You can also create custom tables by changing any of the following parameters in the yellow boxes: **Initial DS in Table; Final DS in Table; DS Increment in Table; RI Temperature, C; and/or Reference Temperature, F**. This is done by selecting the yellow cell beside the parameter of your choice and entering the information you wish the program to calculate.

7. You may also create DS Tables for custom blends of syrups by first creating a new syrup definition in the **Syrup Definition** section of the program (see this section of the manual for further details). Once created, you can select this from the Select Syrup drop down menu and proceed as normal.

Brix Table

1. To use the Brix Table function, click on the corresponding button on the title page of the program.

This will bring you to the Brix Table sub-menu, which looks like this:



2. From this menu, you can generate Brix tables of standard compositions of corn syrup, or return to the home page, by clicking the **Opening Page** tab at the bottom of the screen. To create a Brix table, click on the arrow of the **Select Syrup** drop down menu. In this sub-menu, you will also need to select the Brix type, by clicking on the arrow of the **Select Brix Type** drop down menu. Your choices are: 1936 Refractometer Brix, C; 1966 Refractometer Brix, C; or Hydrometer Brix, C.

3. To create a Brix table with the default characteristics listed in the yellow boxes, press the **Generate Brix Table** button. You will see the program working, indicated by the activity in the calculation bar. After a few seconds, a table will generate. This table will have the syrup type and characteristics labeled at the top of the page. The number of tables you generate per session will determine the
name of each table you generate. For example, if this is your first Brix table, the program

will name this CRA RI-DS Version 21.xls. Subsequent versions will be saved as CRA RI-DS Version 22.xls, CRA RI-DS Version 23.xls etc.

4. At this point, you can either print the table, save the table, or return to the opening page. To print the table, you can either click on the printer icon in the tool bar, or click on **File**, then **Print** from the drop-down menu. The **Opening Page** button will not print on the table.

5. To save these files, click on the diskette icon in the toolbar, or click on **File**, then select **Save** to save with the current name and directory, or **Save As** to change the file name or target directory.

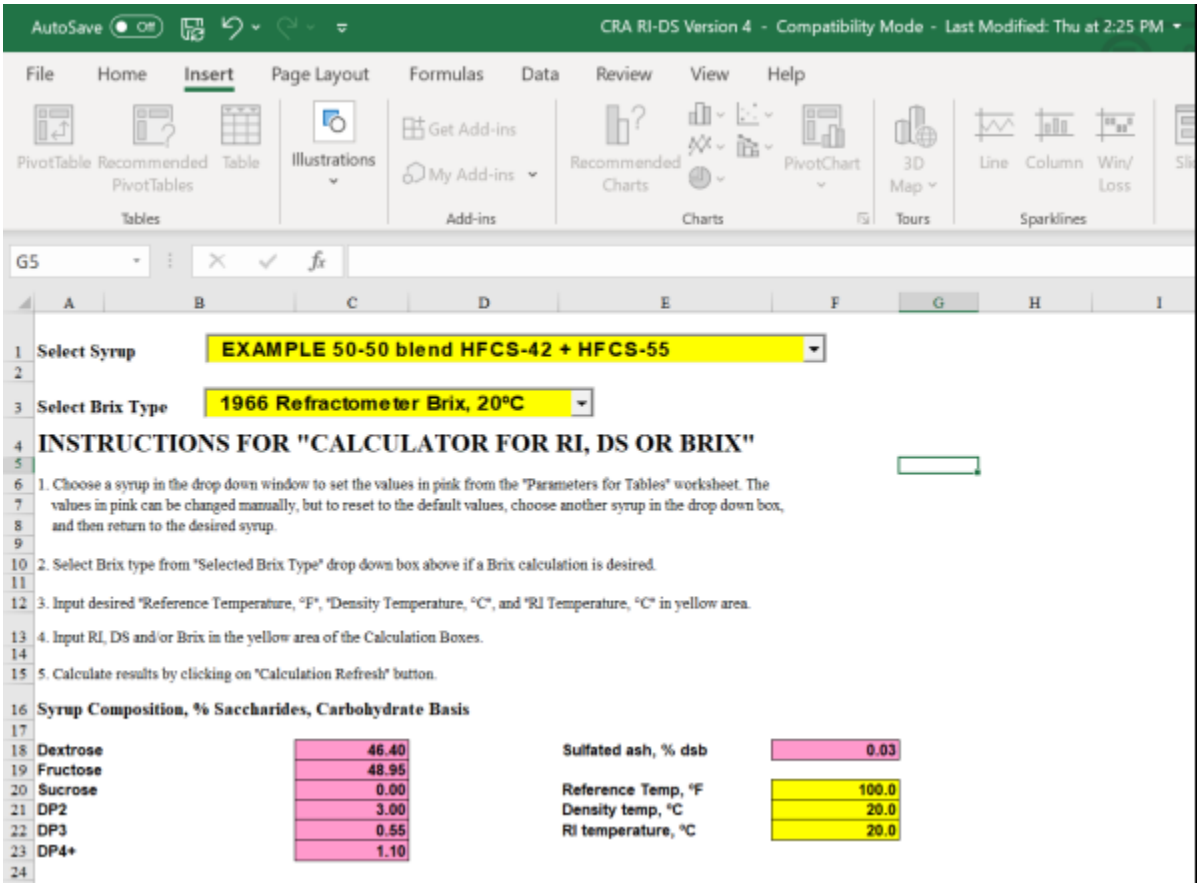
6. To return to the opening page, simply press the **Opening Page** button.

7. You can also create custom tables by changing any of the following parameters in the yellow boxes: **Initial Brix in Table; Final Brix in Table; Brix Increment in Table; RI Temperature, C; and/ or Reference Temperature, F**. This is done by selecting the yellow cell beside the parameter of your choice and entering the information you wish the program to calculate.

8. Additionally, you can create Brix tables for custom blends of syrups, by first creating a new syrup definition in the **Syrup Definition** section of the program (see this section of the manual for further details). Once created, you can select this from the **Select Syrup** drop down menu and proceed to generate the desired tables

Individual Calculations

1. To use the Individual Calculations function, click on the corresponding button on the title page of the program. This will bring you to the Individual Calculations sub-menu, which looks like this:



The example above shows a custom syrup consisting of equal parts of HFCS-42 and HFCS-55 (see Syrup Definition).

2. From this menu, you can generate individual calculations for standard compositions of corn syrup, or return to the home page, by clicking the **Opening Page** tab at the bottom of the screen.

3. Select a syrup type by clicking on the arrow of the **Select Syrup** drop down menu.

4. In this sub-menu, you can also select the Brix type, by clicking on the arrow of the **Select Brix Type** drop down menu, as well as change a number of different parameters, indicated by the pink and yellow cells. When entering numbers into the

different parameter cells, remember to hit **Enter** to confirm your selection. Each time you change parameters, the **Calculation Re- fresh** button will turn red, indicating that you must click it to generate new calculations. This information can be reflected as: **Calculation Using RI, Calculation using DS** or whichever Brix type you select from the Brix Type drop down menu.

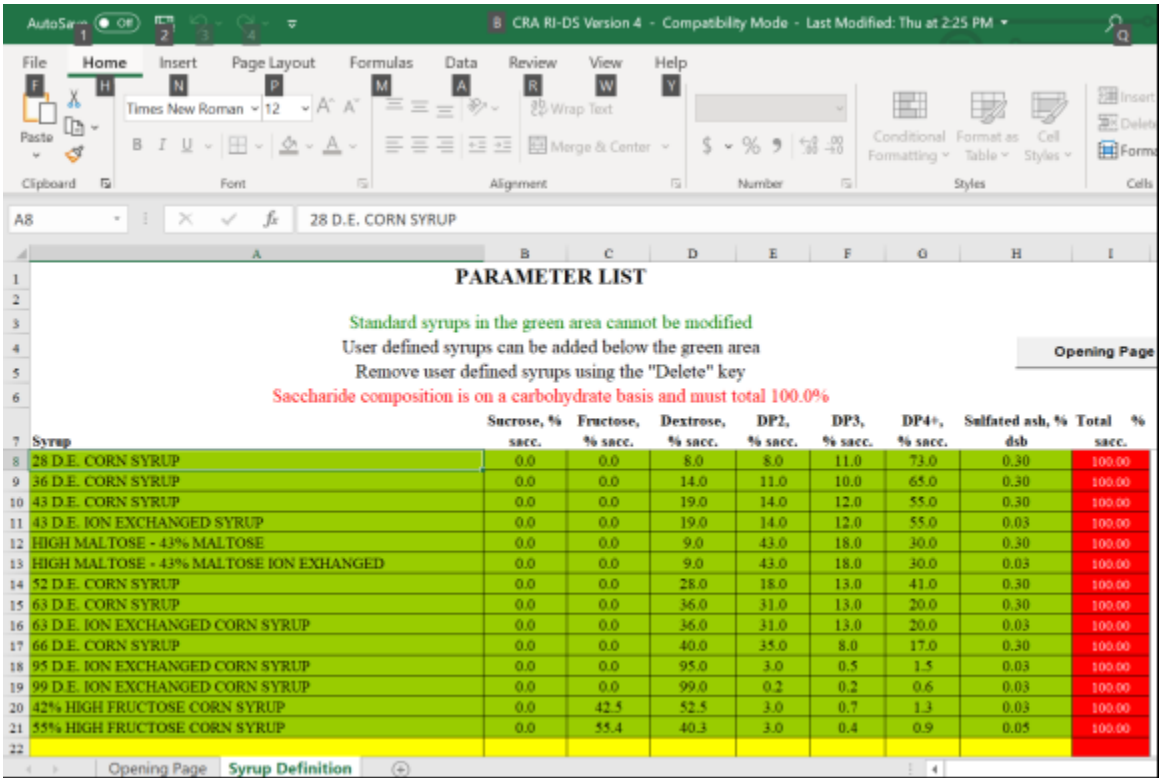
5. When changing parameters in the pink box, remember that the saccharide concentration must equal 100%. If you enter values that cause the concentration to equal more or less than 100%, you will receive an error message telling you that you must change your other parameters to compensate. If this message does not show up automatically, it will show up when you try to refresh the calculations.

6. After generating the syrup composition and/or changing parameters, you can also generate calculations by changing the **RI at 20 C, %DS** or **Brix (DS of 100% Sucrose)** by changing these parameters in the yellow cells of the corresponding boxes. Again, you will need to refresh the calculations when you finish entering your parameters.

7. Additionally, you can create individual calculations for custom blends of syrups, by first creating a new syrup definition in the Syrup Definition section of the program (see this section of the manual for further details). Once created, you can select this from the Select Syrup drop down menu and proceed as normal.

Syrup Definition

1. To use the Syrup Definition function, click on the corresponding button on the title page of the program. This will bring you to the Syrup Definition sub-menu, which looks like this:



Syrup	Sucrose, % sacc.	Fructose, % sacc.	Dextrose, % sacc.	DP2, % sacc.	DP3, % sacc.	DP4+, % sacc.	Sulfated ash, % dsb	Total % sacc.
28 D.E. CORN SYRUP	0.0	0.0	8.0	8.0	11.0	73.0	0.30	100.00
36 D.E. CORN SYRUP	0.0	0.0	14.0	11.0	10.0	65.0	0.30	100.00
43 D.E. CORN SYRUP	0.0	0.0	19.0	14.0	12.0	55.0	0.30	100.00
43 D.E. ION EXCHANGED SYRUP	0.0	0.0	19.0	14.0	12.0	55.0	0.03	100.00
HIGH MALTOSE - 43% MALTOSE	0.0	0.0	9.0	43.0	18.0	30.0	0.30	100.00
HIGH MALTOSE - 43% MALTOSE ION EXHANGED	0.0	0.0	9.0	43.0	18.0	30.0	0.03	100.00
52 D.E. CORN SYRUP	0.0	0.0	28.0	18.0	13.0	41.0	0.30	100.00
63 D.E. CORN SYRUP	0.0	0.0	36.0	31.0	13.0	20.0	0.30	100.00
63 D.E. ION EXCHANGED CORN SYRUP	0.0	0.0	36.0	31.0	13.0	20.0	0.03	100.00
66 D.E. CORN SYRUP	0.0	0.0	40.0	35.0	8.0	17.0	0.30	100.00
95 D.E. ION EXCHANGED CORN SYRUP	0.0	0.0	95.0	3.0	0.5	1.5	0.03	100.00
99 D.E. ION EXCHANGED CORN SYRUP	0.0	0.0	99.0	0.2	0.2	0.6	0.03	100.00
42% HIGH FRUCTOSE CORN SYRUP	0.0	42.5	52.5	3.0	0.7	1.3	0.03	100.00
55% HIGH FRUCTOSE CORN SYRUP	0.0	55.4	40.3	3.0	0.4	0.9	0.05	100.00

2. The Parameter List is equipped with 14 standard syrup compositions, indicated in the green cells, that cannot be modified. Directly following these standard compositions are several yellow cells that can be utilized to create user-defined syrups. To do this, scroll down to the yellow area and enter in a name for the syrup. Go across the table and enter in the composition of the syrup. **Note**: Saccharide composition is on a carbohydrate basis and must total 100.0%. The program will not allow for the creation of a syrup above or below that value and will give an error message, indicating that you must change your values to create the proper balance.

3. Once created, a user-defined syrup can be utilized in other parts of the program. These will be located in the **Select Syrup** drop down menu in all other parts of the program. Should you wish to delete a user-defined syrup, highlight the name and composition cells for that syrup to select the information in that row. Hit the delete button to erase the data. **Note**: The program will not allow you to select the entire row and delete, as there is formatting that is write-protected.

4. In the event that you run out of available space in the yellow area, select the bottom row, then click Insert on the toolbar. Select Row from the drop-down menu. Repeat as needed.

REFRACTIVE INDEX

REFRA..01-[1]

PRINCIPLE

The index of refraction of a substance is the ratio of the velocity of light in a vacuum to its velocity in the substance. This, in turn, is dependent on composition, concentration (e.g. dry substance) and temperature of the substance. When solids composition and temperature are known, index of refraction is a measure of dry substance (Note 1).

SCOPE

The method is applicable to corn syrup (including those containing high fructose), maltodextrin solutions, dextrose and sucrose solutions, invert sugar and blends.

SPECIAL APPARATUS

1. Refractometer: An instrument is necessary with a range of indices from 1.32 or lower to 1.53 or higher, accurate to 0.0001 unit. It should be so constructed that samples can be introduced with ease and speed, and the instrument should be easily cleaned.

   Follow the manufacturer's instructions for use of the particular instrument. Standardize using purified water and the test block supplied with the instrument; calibration with the test block or immersion (calibration) oil must be performed at the temperature specified by the supplier.

2. Water Bath: Operate at a temperature of 20° C or 45° C. It should be sufficient in size to allow circulation of water, by means of a pump, through the refractometer so that the refractometer temperature, especially the prism faces, is controlled within ± 0.2° C of that prescribed for the bath (Note 2). Certain instruments with automatic temperature control may not need water baths.

3. Light Source: Frosted incandescent bulb.

PROCEDURE

Dilute syrups are best applied with a dropper, while concentrated syrups are handled most conveniently with a fire-polished glass rod. After water bath and refractometer have reached the prescribed temperature, apply sample to the prism face, and close the prism in minimum time possible, so as to avoid sample concentration change (Note 3). Examine the optical field through refractometer observation lends; if the light and dark fields are not separated by a sharp line, remove the sample, clean and dry the prism faces, and apply a fresh sample. Read the index, estimating to the nearest 0.0001 unit, as soon as temperature equilibrium is indicated by constant readings (not more than 3 minutes) (Note 4). Prior to applying any sample, rinse the prism faces with purified water at the bath temperature (20° C or 45° C), to facilitate temperature equilibration.

CALCULATIONS

If desired, obtain the percent dry substance of the sample by referencing the CRA RI-DS calculation program located on the following webpage: https://corn.org/policies/product-safety-quality/.

NOTES AND PRECAUTIONS

1. The RI-DS tables on the CRA website relate refractive index to dry substance for commercial corn syrups, high fructose corn syrups and selected blends. They were developed based on investigations by Augustana Research Foundation (Rock Island, Illinois), with the financial support of the Corn Refiners Association, Inc. See: Anna M. Wartman, Caroline Hagberg and Morton A. Eliason, Journal of Chemical Engineering Data, Vol. 25, No. 3, July 1980, pages 277-282. See also: Frank A. Kurtz and Morton A. Eliason, Journal of Chemical and Engineering Data, Vol. 24, No. 1, January 1979, pages 44-45. Tables relating refractive index and concentration for pure dextrose solution have been reported by F. W. Zerban and M. Martin, Journal of the Association of Official Agricultural Chemists, Vol. 27, 1944, page 295. Similar data for solutions of pure fructose, pure maltose and pure sucrose are given in the following articles: U. S. Department of Commerce, National Bureau of Standards, Circular No. C440, Table 129, R.C. Weast, Handbook of Chemistry and Physics, 53rd Edition, Chemical Rubber Company, Cleveland, Ohio, 1972, page D-196; and International Sugar Journal, Vol. 39, 1937, page 225.

2. If the refractometer temperature is lower than room temperature, there is a tendency for the prism to fog, especially at high relative humidities. Also, thick viscous syrups present difficulties which are best overcome at higher temperatures where their viscosities are sharply decreased.

   Hose connections between the water bath and refractometer must be the shortest length possible to avoid temperature differences. Hose insulation is recommended. A 1° C temperature discrepancy corresponds to a dry substance discrepancy of about 0.1%.

3. For accurate measurements, particularly with warm samples, speed in application of the sample and closing the prism is imperative. No more than 2-3 seconds should be consumed in this operation. Also, water used to rinse the prism faces should be at the measuring temperature specified.

4. When analyzing freshly prepared solutions of crystalline sugars, mutarefraction equilibrium must be attained before accurate refractive index values can be obtained. Equilibration can be hastened by heating and is obtained with the optical rotation stabilizes. In addition, solutions of pure sugars, and syrups exhibiting a crystallization tendency, must be completely free of all crystalline materials because they prohibit accurate refractive index measurement.

5. The refractive index/dry substance relationship will vary with composition. A computer program, RI-DS, which will produce tables based upon user-supplied saccharide compositions, is available on the Corn Refiners Association, Inc. website (see Calculations). Tables suited for the needs of particular end-users of these products may be produced, e.g., tables contained in Quality Guidelines and Analytical Procedures Bibliography for "Bottlers" High Fructose Corn Syrup 42 and 55, International Society of Beverage Technologists (www.bevtech.org). For discussion of the mathematical models necessary for construction of tables for products of different composition, see J. Chem. Eng. Data, No. 1, January 1979, pp. 44-45, J. Chem Eng. Data, Vol. 25, No. 3, July 1980, pp. 277-282 and J. Agr. Food Chem., Vol. 32, 1984, pp. 974-979.

METHOD HISTORY

Corn Syrup, Refractive Index (E-54), Date of Acceptance 5-07-1956, Revised 10-9-2009.

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2. Hebeda, Ronald E. and Fred W. Schenck. Starch Hydrolysis Products: Worldwide Technology, Production and Applications. VCH Publishers, Inc. New York, NY. 1992. pp. 377-378.

3. Kurtz, F.A. and M. A. Eliason. *Journal of Chemical and Engineering Data*. 1979. 24, pp. 44-45.

4. Maxwell, J. L., F. A. Kurtz and B. J. Strelka. *Journal of Agricultural and Food Chemistry*. 1984. 32, pp. 974-979.

5. Wartman, A. M., A. J. Bridges and M. A. Eliason. *Journal of Chemical and Engineering Data*. 1980. 25, pp. 277-282.

6. Wartman, A. M., C. Hagberg and M. A. Eliason. *Journal of Chemical and Engineering Data*. 1976. 21, pp. 459-468.

7. Wartman, A. M., T. D. Spawn and M. A. Eliason. *Journal of Agricultural and Food Chemistry*. 1984. 32, pp. 971-974.

Acknowledgments

The original RI-DS program was created by Dr. Ki Park, formerly of Cargill, Incorporated, as a C-compiled stand-alone MS-DOS program based on the mathematical models developed at Augustana Research Foundation. Subsequent work showed the need to reconcile the program's physical data which was completed with the assistance of Dr. John Rasche, formerly of A. E. Staley Manufacturing Company (now Tate & Lyle Americas).

Thomas Gagnon, formerly of Tate & Lyle Americas, was instrumental in the creation of the Windows-based Microsoft Excel ®-based program. CRA would also like to extend a special thanks to Jerry Geske, formerly of Tate and Lyle Americas, for his assistance with the program's development and data reconciliation.

The development of the extensive physical data, mathematical relationships and computer program which are represented in the original RI-DS program provided the foundation for the update and was made possible by a wide variety of personnel in the corn refining industry and academia. CRA would like to thank the following their work on the original data: Anna Wartman; Robert J. Smith; Sharon Purvis; Earl Engel; Frank Kurtz; Raffaele Bernetti; Kenny Brobst; Larry Fitt; and Ki Park.