Table V. Sum of Deviation Squares

Equation	Sum of deviation squares
1	0.1353×10^{-8}
2	$0.126\,08 \times 10^{-7}$
3	0.1783×10^{-5}
4	$0.141\,03 \times 10^{-8}$
5	0.4860×10^{-8}
6	$0.423\,14 \times 10^{-9}$
7	0.1539×10^{-4}
8	$0.370\,66 \times 10^{-5}$

Benzene–Xylene System:

$$d^{25\,°\mathrm{C}}{}_4 = 0.8640 + 0.0696x_1 - 0.9659x_1^2 + 4.7199x_1^3 - 9.9782x_1^4 + 9.5507x_1^5 - 3.3824x_1^6 \quad (3)$$

$$n^{25\,°\mathrm{C}}{}_\mathrm{D} = 1.494\,90 - 0.000\,43x_1 + 0.000\,76x_1^2 + 0.001\,87x_1^3 \quad (4)$$

Toluene–Xylene System:

$$d^{25\,°\mathrm{C}}{}_4 = 0.8639 + 0.0016x_2 - 0.0006x_2^2 \quad (5)$$

$$n^{25\,°\mathrm{C}}{}_\mathrm{D} = 1.494\,90 - 0.001\,34x_2 + 0.002\,38x_2^2 - 0.005\,16x_2^3 + 0.002\,87x_2^4 \quad (6)$$

Benzene–Toluene–Xylene System:

$$d^{25\,°\mathrm{C}}{}_4 = 0.8662 + 0.0132x_1 - 0.0012x_2 \quad (7)$$

$$n^{25\,°\mathrm{C}}{}_\mathrm{D} = 1.494\,62 + 0.002\,01x_1 - 0.001\,17x_2 \quad (8)$$

where x_1 = mole fraction of benzene and x_2 = mole fraction of toluene.

Table V presents the sum of deviation squares according to which the reliability of the calculated approximation could be proved. Equations 7 and 8 indicate that the highest deviations occur at both extreme concentrations (at the triangle apex), what usually would be expected.

Literature Cited

(1) Battino, R., *Chem. Rev.*, **71**, 5 (1971).
(2) Chu, K. Y., Thompson, A. R., *J. Chem. Eng. Data*, **7**, 358 (1962).
(3) Himmelblau, D. M., "Process Analysis by Statistical Methods", Wiley, New York, N.Y., 1970.
(4) "International Critical Tables of Numerical Data Physics, Chemistry and Technology", Vol. III, VII, McGraw-Hill, New York, N.Y., 1933.
(5) Sumer, K. M., Thompson, A. R., *J. Chem. Eng. Data*, **12**, 489 (1967).
(6) Sumer, K. M., Thompson, A. R., *J. Chem. Eng. Data*, **13**, 30 (1968).
(7) Wood, S. E., Brusie, J. P., *J. Am. Chem. Soc.*, **65**, 1891 (1943).

Received for review October 24, 1975. Accepted April 24, 1976.

Refractive Index–Dry Substance Relationships for Commercial Corn Syrups

Anna M. Wartman,* Caroline Hagberg, and Morton A. Eliason
Augustana Research Foundation, Rock Island, Illinois 61201

A study of the relationship between refractive index and dry substance (total solids) has been made for a wide variety of commercial corn syrups (including high fructose corn syrup) and one maltodextrin. Dry substance levels from 0 to 85% and temperatures from 20 to 60 °C were covered in this study. The corn syrups and maltodextrin were produced by acid and dual conversion processes (the high fructose corn syrup process includes isomerization) and span a range of dextrose equivalent values from 12 to 95. Additional measurements to establish the dependence of the refractive index–dry substance relationship on ash content were made for ash levels from 0 to 1.5%. The results for 11 product types are presented in the form of tables and of mathematical equations for the purpose of converting refractive index measurements to dry substance contents. Ten commercial corn syrups and one maltodextrin were used.

A comprehensive study to establish accurate relationships between refractive index and dry substance content has been carried out for a wide variety of commercial corn syrups (including high fructose corn syrup) and one maltodextrin. This study included products made by acid conversion and dual conversion processes representative of products manufactured by the member companies of the Corn Refiners Association, Inc. (CRA). These products ranged in dextrose equivalent values from 12 to 95. Measurements were made for six or more dry substance levels from 15 to 85% and at temperatures of 20, 30, 45, and 60 °C for each of 11 products. Additional measurements were made on two commercial syrups which were ion exchange refined as well as on those syrups with added ash (up to 1.5% ash) to establish the dependence of the refractive index–dry substance relationships on ash content.

The resulting data for each product are available in tabular form from the Corn Refiners Association, Inc., 1001 Connecticut Avenue N.W., Washington, D.C., and have been filed with the ACS Microfilm Depository Service as supplementary tables (see paragraph at end of paper). These data have been fit by regression analysis to a suitable mathematical function for each of the 11 products. These functions have been used to generate detailed working tables which may be used for converting refractive index readings to dry substance values. Means of correcting for temperature and ash variations and for small variations in dextrose equivalent (D.E.) and/or product composition are included with these tables.

Analysis of the residuals (observed minus calculated values) indicates that the data are internally consistent and that a relatively simple mathematical model is capable of representing the relationships involved for a wide variety of product types. Careful work with sucrose and dextrose solutions and preliminary interlaboratory checks on several syrup samples served to establish the reliability and accuracy of the equipment and methods used by the investigators. Further mathematical analysis of the data reported here, aimed at establishing the relationship between saccharide composition and refractive index–dry substance relationships, will be reported elsewhere (*4*); this additional work confirms the internal consistency of the data and successfully establishes the relationship among product types.

Comparison of the results reported here with those of Cleland, Evans, Fauser, and Fetzer (*2*) shows a small, but apparently systematic, deviation at higher dry substance levels. Similar differences noted previously by member companies of the CRA, and also by Malinsky (*5*) and Graefe (*3*), prompted the present study sponsored by the Corn Refiners Association, Inc.

Table I. Preliminary Interlaboratory Dry Substance Check Analyses

Sample	Dry Substance, % (by weight, in air)			
	Augustana		CPC International Inc	
	Vacuum oven—filter aid	Karl Fischer	Vacuum oven—filter aid	Karl Fischer
High fructose corn syrup	71.17	71.20	71.11	71.03
	71.10	71.14	71.11	71.06
		71.19		71.09
		71.28		71.16
Average	71.14	71.20	71.11	71.08
42 D.E. corn syrup	80.52	80.35	80.40	80.18
	80.33	80.38	80.43	80.27
		80.31		80.31
Average	80.42	80.35	80.42	80.25
Diluted 42 D.E. corn syrup[a]	70.47	70.52	70.26	70.19
	70.43	70.50	70.34	70.27
		70.47		70.29
		70.43		70.25
Average	70.45	70.48	70.30	70.25

[a] Calculated dry substance, on the basis of 80.42% for the original syrup, was 70.37%.

Preliminary Work

Prior to the data acquisition phase of this study, refractive index measurements were made on quantitatively prepared (by weight) solutions of National Bureau of Standards sucrose and dextrose at 20 °C and at concentrations from 10 to 70% dry solids. Determinations were made on 16 samples of each sugar. The average differences in the refractive index as compared to those previously published were 0.000 036 and 0.000 071 for sucrose (*12*) and dextrose (*13*), respectively.

The validation of the equipment and experimental procedures for the determination of dry substance values took the form of concurrent measurements by this laboratory and by the research staff of CPC International Inc. The methods used were the standard vacuum oven method (*8*) and the Karl Fischer titration method (*9*). The results of this interlaboratory check work are shown in Table I.

On the basis of the agreement found here, the major data acquisition phase of the study was undertaken.

Samples

Samples of ten different commercial corn syrups and one maltodextrin were obtained from member companies of the CRA.

The following definitions were used to identify samples investigated in this study: *Corn syrup* (*glucose syrup*) is the purified concentrated aqueous solution of nutritive saccharides obtained from edible starch and having a dextrose equivalent of 20 or more. (This definition for corn syrup has been adopted by the U.S. Food and Drug Administration under Chapter 1 of Title 21 of the Code of Federal Regulations, Part 26—Nutritive Sweeteners, and parallels the definition proposed by the International Codex Alimentarius Commission.) *Maltodextrin* is a purified concentrated aqueous solution of nutritive saccharides obtained from edible starch, or the dried product derived from said solution, and having a dextrose equivalent of less than 20.

The term "acid conversion" refers to the treatment of starch with acid, and the term "dual conversion" refers to the sequential treatment of starch with acid and purified amylolytic enzymes.

Table II. Samples and Composition Data[a]

Sample	Dextrose equivalent	Ash sulfated % d.b.	% Saccharides, carbohydrate basis						
			DP_1	DP_2	DP_3	DP_4	DP_5	DP_6	DP_7+
1 Corn syrup AC[b]	27.3	0.40	9.2	8.7	7.8	7.1	6.5	6.3	54.4
2 Corn syrup AC	42.5	0.37	19.6	14.4	11.6	9.5	7.6	6.5	30.8
3 Corn syrup AC	54.5	0.47	30.7	17.9	12.2	9.7	6.9	5.2	17.4
4 Corn syrup DC	33.0	0.47	10.0	14.5	9.6	6.7	6.6	11.0	41.6
5 Corn syrup HM, DC	43.5	0.41	7.5	39.5	14.7	7.5	2.1	1.9	26.8
6 Corn syrup HM, DC	49.0	0.41	8.6	51.6	14.5	1.4	1.7	2.1	20.1
7 Corn syrup DC	64.5	0.56	39.2	30.5	6.6	5.3	4.1	3.3	11.0
8 Corn syrup DC	70.1	0.53	47.2	27.2	5.2	4.7	3.8	2.7	9.2
9 Corn syrup DC	95.0	0.50	92.4	3.8	0.7	0.7	0.5	0.5	1.4
10 High fructose corn syrup	(*d*)	0.04	93.1[c]	4.2	0.4	0.1	0.1	0.1	2.0
11 Maltodextrin	12.1	0.47	0.9	2.5	4.0	3.4	2.9	5.5	80.8

[a] Samples and data supplied by cooperating Member Companies of CRA. [b] AC = acid conversion, DC = dual conversion (acid–enzyme), HM = high maltose. [c] Dextrose plus fructose. [d] See text.

Table III. Interlaboratory Comparison—Original Samples Dry Substance and Refractive Index

Sample	Augustana Dry substance vacuum oven	Augustana Dry substance Karl Fischer	Augustana $n^{20}\mathrm{D}$	CPC International Inc. Dry substance vacuum oven	CPC International Inc. Dry substance Karl Fischer	CPC International Inc. $n^{20}\mathrm{D}$
1 Corn syrup AC[a]	78.09	78.16	—	78.19	78.10	—
2 Corn syrup Ac	80.37	80.22	1.49751	80.38	80.34	1.49755
Ion exchange	78.80	78.72	1.49319	78.80	78.76	1.49318
3 Corn syrup AC	80.90	80.87	1.49680	80.90	80.89	1.49688
4 Corn syrup DC	78.54	78.42	1.49464	78.55	78.44	1.49444
5 Corn syrup HM, DC	80.51	80.51	1.49810	80.49	80.58	1.49814
Ion exchange	77.84	77.67	1.49084	77.93	77.88	1.49091
6 Corn syrup HM, DC	80.62	80.54	1.49769	80.74	80.75	1.49776
7 Corn syrup DC	79.63	79.54	1.49142	79.65	79.62	1.49147
8 Corn syrup DC	82.25	82.28	1.49723	82.28	82.30	1.49742
9 Corn syrup DC	71.43	71.46	1.46558	71.49	71.51	1.46569
10 High fructose corn syrup	70.97	71.03	1.46446	70.98	71.13	1.46462
11 Maltodextrin	94.82	94.83	—	95.06	94.98	—

[a] AC = acid conversion, DC = dual conversion (acid–enzyme), HM = high maltose.

Table IV. Refractive Index Readings for Water vs. Temperature

t (°C)	nD lit. (ref 11)	nD obsd[a]	Av diff	Stand dev[a]
20	1.332 99	1.332 97	−0.000 02	0.000 024
30	1.331 94	1.331 95	0.000 01	0.000 016
45	1.329 85	1.329 88	0.000 03	0.000 028
60	1.327 25	1.327 36	0.000 11	0.000 010

[a] Based on 14 determinations at each temperature, made during the course of this work.

Table V. Coefficients for Equation 1[b]

Sample	Dry substance coefficients A_1	A_2	A_3	Temperature coefficients B_1	B_2	B_3
1 Corn syrup AC[a]	0.644 969 2	−0.001 556 1	−0.003 573 4	−0.000 004 6	0.000 123 0	−0.000 034 8
2 Corn syrup AC	0.646 333 4	−0.002 337 3	−0.001 063 8	−0.000 004 6	0.000 123 0	−0.000 034 8
3 Corn syrup AC	0.646 929 6	−0.002 363 1	−0.000 450 5	−0.000 004 6	0.000 123 0	−0.000 034 8
4 Corn syrup DC	0.645 845 0	−0.003 562 1	−0.001 054 9	−0.000 004 6	0.000 123 0	−0.000 034 8
5 Corn syrup HM, DC	0.646 007 3	−0.002 289 0	−0.001 147 7	−0.000 004 6	0.000 123 0	−0.000 034 8
6 Corn syrup HM, DC	0.646 424 4	−0.002 488 7	−0.000 808 4	−0.000 004 6	0.000 123 0	−0.000 034 8
7 Corn syrup DC	0.647 408 7	−0.001 558 8	−0.000 360 4	−0.000 004 6	0.000 123 0	−0.000 034 8
8 Corn syrup DC	0.647 687 0	−0.001 492 5	0.000 000 0	−0.000 004 6	0.000 123 0	−0.000 034 8
9 Corn syrup DC	0.648 802 2	0.001 344 6	−0.001 069 6	0.000 002 7	0.000 120 9	−0.000 031 2
10 High fructose corn syrup	0.649 299 6	0.000 585 3	−0.001 072 8	−0.000 013 0	0.000 135 3	−0.000 029 9
11 Maltodextrin	0.644 890 7	−0.005 468 9	0.000 000 0	−0.000 010 0	0.000 111 1	−0.000 015 9

[a] AC = acid conversion, DC = dual conversion (acid–enzyme), HM = high maltose. [b] Ash coefficients for all samples: C_1, 0.000 022 5; C_2, 0.000 000 0; C_3, −0.017 697 7.

The term "dual conversion" may refer also to the sequential treatment of starch with purified amylolytic enzymes.

High fructose corn syrups are those produced by a dual conversion process followed by isomerization.

Individual syrups were blended for homogeneity and divided for interlaboratory characterization studies. Samples of each were delivered to this laboratory and to laboratories of Clinton Corn Processing Company, Clinton, Iowa, A.E. Staley Manufacturing Company, Decatur, Ill., and CPC International Inc., Argo, Ill. Compositional data on the products were obtained at Clinton, Decatur, and Argo, concurrently with the refractive index and

Table VI. Corn Syrup, Acid Conversion, 28 D.E., 0.4% Ash (D.B.)

% dry substance	Refractive index 20 °C	30 °C	45 °C	60 °C
0	1.332 99	1.331 94	1.329 85	1.327 25
2	1.335 97	1.334 90	1.332 77	1.330 15
4	1.339 04	1.337 93	1.335 78	1.333 13
6	1.342 15	1.341 02	1.338 83	1.336 15
8	1.345 31	1.344 15	1.341 93	1.339 23
10	1.348 52	1.347 34	1.345 08	1.342 36
12	1.351 78	1.350 57	1.348 29	1.345 54
14	1.355 09	1.353 85	1.351 54	1.348 77
16	1.358 46	1.357 19	1.354 84	1.352 05
18	1.361 87	1.360 58	1.358 20	1.355 38
20	1.365 34	1.364 02	1.361 61	1.358 77
22	1.368 86	1.367 52	1.365 08	1.362 22
24	1.372 44	1.371 07	1.368 60	1.365 72
26	1.376 07	1.374 68	1.372 18	1.369 28
28	1.379 76	1.378 35	1.375 82	1.372 90
30	1.383 52	1.382 07	1.379 52	1.376 58
32	1.387 33	1.385 86	1.383 28	1.380 32
34	1.391 20	1.389 71	1.387 10	1.384 13
36	1.395 13	1.393 62	1.390 98	1.387 99
38	1.399 13	1.397 59	1.394 93	1.391 92
40	1.403 19	1.401 63	1.398 94	1.395 92
42	1.407 32	1.405 73	1.403 02	1.399 98
44	1.411 52	1.409 91	1.407 17	1.404 11
46	1.415 78	1.414 15	1.411 39	1.408 32
48	1.420 11	1.418 46	1.415 67	1.412 59
50	1.424 52	1.422 84	1.420 03	1.416 94
52	1.429 00	1.427 30	1.424 47	1.421 36
54	1.433 55	1.431 83	1.428 97	1.425 85
56	1.438 18	1.436 43	1.433 56	1.430 42
58	1.442 88	1.441 12	1.438 22	1.435 07
60	1.447 67	1.445 88	1.442 96	1.439 81
62	1.452 53	1.450 72	1.447 79	1.444 62
64	1.457 48	1.455 65	1.452 69	1.449 52
66	1.462 51	1.460 66	1.457 68	1.454 50
68	1.467 62	1.465 76	1.462 76	1.459 57
70	1.472 83	1.470 94	1.467 92	1.464 73

Table VII. Corn Syrup, Acid Conversion, 42 D.E., 0.4% Ash (D.B.)

% dry substance	Refractive index 20 °C	30 °C	45 °C	60 °C
0	1.332 99	1.331 94	1.329 85	1.327 25
2	1.335 93	1.334 85	1.332 73	1.330 10
4	1.338 95	1.337 84	1.335 69	1.333 04
6	1.342 02	1.340 89	1.338 70	1.336 02
8	1.345 14	1.343 98	1.341 76	1.339 06
10	1.348 30	1.347 12	1.344 87	1.342 14
12	1.351 52	1.350 31	1.348 03	1.345 28
14	1.354 79	1.353 55	1.351 24	1.348 46
16	1.358 11	1.356 84	1.354 50	1.351 70
18	1.361 48	1.360 19	1.357 81	1.355 00
20	1.364 90	1.363 59	1.361 18	1.358 34
22	1.368 38	1.367 04	1.364 60	1.361 75
24	1.371 91	1.370 55	1.368 08	1.365 20
26	1.375 50	1.374 11	1.371 61	1.368 72
28	1.379 14	1.377 73	1.375 20	1.372 29
30	1.382 84	1.381 40	1.378 85	1.375 92
32	1.386 60	1.385 13	1.382 56	1.379 60
34	1.390 42	1.388 93	1.386 32	1.383 35
36	1.394 29	1.392 78	1.390 15	1.387 16
38	1.398 23	1.396 69	1.394 03	1.391 03
40	1.402 22	1.400 66	1.397 98	1.394 96
42	1.406 28	1.404 70	1.401 99	1.398 96
44	1.410 40	1.408 80	1.406 07	1.403 02
46	1.414 59	1.412 96	1.410 21	1.407 14
48	1.418 84	1.417 19	1.414 41	1.411 33
50	1.423 16	1.421 49	1.418 69	1.415 59
52	1.427 55	1.425 85	1.423 03	1.419 92
54	1.432 00	1.430 28	1.427 44	1.424 32
56	1.436 53	1.434 79	1.431 92	1.428 79
58	1.441 12	1.439 36	1.436 47	1.433 33
60	1.445 79	1.444 01	1.441 10	1.437 95
62	1.450 53	1.448 73	1.445 80	1.442 64
64	1.455 34	1.453 52	1.450 57	1.447 41
66	1.460 23	1.458 39	1.455 42	1.452 25
68	1.465 20	1.463 34	1.460 35	1.457 17
70	1.470 24	1.468 36	1.465 36	1.462 18
72	1.475 37	1.473 47	1.470 45	1.467 26
74	1.480 58	1.478 66	1.475 62	1.472 43
76	1.485 87	1.483 93	1.480 88	1.477 68
78	1.491 24	1.489 29	1.486 22	1.483 01
80	1.496 70	1.494 73	1.491 64	1.488 44
82	1.502 25	1.500 26	1.497 16	1.493 95
84	1.507 88	1.505 87	1.502 76	1.499 56

dry substance measurements at this laboratory. Values of dextrose equivalent (D.E.) (*7*) and sulfated ash content (*6*), were determined as well as saccharide composition by both liquid and paper chromatography (*10*). The results of these determinations are shown in Table II.

Experimental Details

Dry Substance. The primary method for the determination of dry substance content was the vacuum oven–filter aid method. High fructose corn syrup and all corn syrups with D.E. values above 55 were dried at 70 °C; all other corn syrups were dried at 100 °C. The maltodextrin sample (a dry powder) was vacuum oven dried directly at 120 °C.

For the purpose of monitoring the vacuum oven analysis, the dry substance content for each product was also determined by the Karl Fischer titration method. The solvent system used was methanol–formamide in the ratio of 70:30 except for the maltodextrin sample when the ratio was reversed to 30:70.

Additional monitoring of the dry substance determinations was provided by CPC International Inc. Although none of these check data has been used in the calculations and tables reported here, they did suggest several recheck determinations and thus helped to minimize or eliminate possible errors. In these ways, initial dry substance values with a high probability of accuracy were obtained.

The dry substance value for each diluted samples was calculated on a weight–weight basis from the amounts of syrup and water used in the dilution process and the vacuum oven dry substance value for the original sample. The dilutions were made in polyethylene screw top bottles of 4-oz capacity. The opening was lightly greased with Silicone stopcock grease, the bottle weighed, a syrup sample quickly introduced, and the weight determined. Water of the amount needed to give the desired dry substance level was then introduced and the total weight determined. The sample was then agitated until homogeneous; several of the samples required heating for from 5 to 30 min at 65–70 °C and the maltodextrin samples required heating to 100 °C in order to achieve homogeneity. These samples were reweighed to check for possible water loss by evaporation before the final value for the dry substance of the dilution was computed. Randomly chosen samples of the diluted products were subjected to vacuum oven and Karl Fischer moisture determinations as a check on the gravimetric dilution process, with satisfactory agreement.

The agreement of the dry substance determinations and refractive index measurements on the original products, as done by this laboratory and by CPC International Inc., is shown in Table

Table VIII. Corn Syrup, Acid Conversion, 55 D.E., 0.4% Ash (D.B.)

% dry substance	Refractive index			
	20 °C	30 °C	45 °C	60 °C
0	1.332 99	1.331 94	1.329 85	1.327 25
2	1.335 90	1.334 83	1.332 71	1.330 08
4	1.338 90	1.337 80	1.335 64	1.332 99
6	1.341 95	1.340 81	1.338 63	1.335 95
8	1.345 04	1.343 88	1.341 66	1.338 96
10	1.348 18	1.346 99	1.344 74	1.342 02
12	1.351 37	1.350 16	1.347 87	1.345 13
14	1.354 61	1.353 37	1.351 06	1.348 28
16	1.357 90	1.356 63	1.354 29	1.351 49
18	1.361 24	1.359 95	1.357 57	1.354 76
20	1.364 63	1.363 31	1.360 91	1.358 07
22	1.368 07	1.366 73	1.364 30	1.361 44
24	1.371 57	1.370 20	1.367 74	1.364 86
26	1.375 12	1.373 73	1.371 23	1.368 34
28	1.378 72	1.377 31	1.374 79	1.371 87
30	1.382 38	1.380 94	1.378 39	1.375 46
32	1.386 10	1.384 63	1.382 06	1.379 10
34	1.389 87	1.388 38	1.385 78	1.382 81
36	1.393 70	1.392 18	1.389 56	1.386 57
38	1.397 58	1.396 05	1.393 39	1.390 39
40	1.401 53	1.399 97	1.397 29	1.394 27
42	1.405 54	1.403 95	1.401 25	1.398 22
44	1.409 60	1.408 00	1.405 27	1.402 22
46	1.413 73	1.412 10	1.409 35	1.406 29
48	1.417 92	1.416 27	1.413 50	1.410 42
50	1.422 18	1.420 51	1.417 71	1.414 62
52	1.426 50	1.424 80	1.421 98	1.418 88
54	1.430 88	1.429 17	1.426 32	1.423 21
56	1.435 33	1.433 60	1.430 73	1.427 61
58	1.439 85	1.438 09	1.435 21	1.432 08
60	1.444 44	1.442 66	1.439 76	1.436 61
62	1.449 10	1.447 30	1.444 37	1.441 22
64	1.453 82	1.452 01	1.449 06	1.445 90
66	1.458 62	1.456 79	1.453 82	1.450 66
68	1.463 50	1.461 64	1.458 66	1.455 49
70	1.468 44	1.466 57	1.463 57	1.460 39
72	1.473 47	1.471 57	1.468 56	1.465 37
74	1.478 57	1.476 65	1.473 62	1.470 44
76	1.483 74	1.481 81	1.478 77	1.475 58
78	1.489 00	1.487 05	1.483 99	1.480 80
80	1.494 34	1.492 37	1.489 30	1.486 10
82	1.499 76	1.497 77	1.494 69	1.491 49
84	1.505 26	1.503 26	1.500 16	1.496 97

Table IX. Corn Syrup, Dual Conversion, 32 D.E., 0.4% Ash (D.B.)

% dry substance	Refractive index			
	20 °C	30 °C	45 °C	60 °C
0	1.332 99	1.331 94	1.329 85	1.327 25
2	1.335 94	1.334 87	1.332 75	1.330 12
4	1.338 99	1.337 88	1.335 73	1.333 08
6	1.342 08	1.340 95	1.338 76	1.336 08
8	1.345 22	1.344 06	1.341 84	1.339 14
10	1.348 41	1.347 23	1.344 98	1.342 25
12	1.351 66	1.350 45	1.348 16	1.345 41
14	1.354 95	1.353 72	1.351 40	1.348 63
16	1.358 30	1.357 04	1.354 69	1.351 90
18	1.361 71	1.360 42	1.358 04	1.355 22
20	1.365 17	1.363 85	1.361 44	1.358 60
22	1.368 68	1.367 34	1.364 90	1.362 04
24	1.372 25	1.370 88	1.368 41	1.365 54
26	1.375 87	1.374 48	1.371 99	1.369 09
28	1.379 56	1.378 14	1.375 62	1.372 70
30	1.383 30	1.381 86	1.379 31	1.376 37
32	1.387 10	1.385 64	1.383 06	1.380 10
34	1.390 97	1.389 48	1.386 87	1.383 90
36	1.394 89	1.393 38	1.390 74	1.387 75
38	1.398 88	1.397 34	1.394 68	1.391 67
40	1.402 93	1.401 37	1.398 68	1.395 66
42	1.407 04	1.405 46	1.402 75	1.399 71
44	1.411 22	1.409 62	1.406 88	1.403 83
46	1.415 47	1.413 84	1.411 08	1.408 01
48	1.419 78	1.418 13	1.415 35	1.412 27
50	1.424 17	1.422 49	1.419 69	1.416 59
52	1.428 62	1.426 92	1.424 09	1.420 98
54	1.433 14	1.431 42	1.428 57	1.425 45
56	1.437 74	1.436 00	1.433 12	1.429 99
58	1.442 41	1.440 65	1.437 75	1.434 61
60	1.447 15	1.445 37	1.442 45	1.439 30
62	1.451 97	1.450 17	1.447 23	1.444 07
64	1.456 87	1.455 04	1.452 09	1.448 92
66	1.461 84	1.460 00	1.457 02	1.453 85
68	1.466 90	1.465 03	1.462 04	1.458 86
70	1.472 04	1.470 15	1.467 14	1.463 95
72	1.477 26	1.475 35	1.472 32	1.469 13
74	1.482 56	1.480 64	1.477 59	1.474 39
76	1.487 95	1.486 01	1.482 95	1.479 74
78	1.493 43	1.491 47	1.488 39	1.485 18
80	1.498 99	1.497 01	1.493 92	1.490 71
82	1.504 65	1.502 65	1.499 55	1.496 33
84	1.510 40	1.508 39	1.505 27	1.502 05

III. Further evidence bearing on the reliability of the dry substance values found and used in this report will be found in the data reduction and analysis section of this report.

Ash Content. The sulfated ash content for each of the original products was determined by the collaborating laboratories. The results are included in Table II.

The influence of ash content on the refractive index-dry substance relationship was investigated for samples 2 and 5. These 42 D.E. corn syrups, produced by acid conversion and dual conversion processes, respectively, were considered to be typical in respect to ash content of commercial products. Ash, in the form of NaCl, was added to portions of each of these syrups to bring the ash content to about 1.0 and 1.5%. In addition, ash-free samples of each syrup were prepared by ion exchange refining. Dry substance values for each of the ion-exchanged and "spiked" syrups were determined by the vacuum oven and Karl Fischer methods, dilutions prepared, and refractive index measurements made at the various temperatures.

The results of the measurements on these altered ash samples are included in the experimental data tables. All values were included in the mathematical analysis for these syrups in addition to their use in determining ash corrections.

Refractive Index. The measurements of refractive index were made with a Bausch and Lomb precision (Sugar) refractometer, with prism covering the range 1.20–1.50, using a sodium vapor lamp as light source. The precision quoted by the manufacturer is ±0.00003. The initial calibration of the instrument was made at 20 °C using the standard glass test piece provided by the manufacturer (refractive index at 20 °C = 1.464 51). Calibration checks were run routinely with distilled water and occasional rechecks were made with the glass test piece. For temperatures above 20 °C, water was used as the standard. In preliminary calibration studies, the data summarized in Table IV were obtained; these data suggest that corrections are needed at the 45 and 60 °C temperatures. Subsequently, the refractive index measurements at each temperature were corrected by adding the difference between the accepted value and the measured value for water as found on the day of that measurement.

Samples and dilutions were applied to the prism of the refractometer as quickly as possible to minimize possible evap-

Table X. Corn Syrup, High Maltose, Dual Conversion, 42 D.E., 0.4% Ash (D.B.)

% dry	Refractive index			
substance	20 °C	30 °C	45 °C	60 °C
0	1.332 99	1.331 94	1.329 85	1.327 25
2	1.335 94	1.334 86	1.332 74	1.330 11
4	1.338 97	1.337 87	1.335 72	1.333 06
6	1.342 06	1.340 93	1.338 74	1.336 06
8	1.345 19	1.344 03	1.341 81	1.339 11
10	1.348 37	1.347 18	1.344 93	1.342 20
12	1.351 60	1.350 38	1.348 10	1.345 35
14	1.354 88	1.353 64	1.351 32	1.348 55
16	1.358 21	1.356 94	1.354 60	1.351 80
18	1.361 59	1.360 30	1.357 92	1.355 11
20	1.365 02	1.363 71	1.361 30	1.358 46
22	1.368 51	1.367 17	1.364 73	1.361 88
24	1.372 05	1.370 69	1.368 22	1.365 34
26	1.375 65	1.374 26	1.371 77	1.368 87
28	1.379 30	1.377 89	1.375 37	1.372 45
30	1.383 01	1.381 57	1.379 02	1.376 09
32	1.386 78	1.385 32	1.382 74	1.379 78
34	1.390 61	1.389 12	1.386 51	1.383 54
36	1.394 49	1.392 98	1.390 35	1.387 36
38	1.398 44	1.396 90	1.394 24	1.391 24
40	1.402 44	1.400 88	1.398 20	1.395 18
42	1.406 51	1.404 93	1.402 22	1.399 18
44	1.410 64	1.409 04	1.406 30	1.403 25
46	1.414 84	1.413 21	1.410 45	1.407 39
48	1.419 10	1.417 45	1.414 67	1.411 59
50	1.423 42	1.421 75	1.418 95	1.415 86
52	1.427 82	1.426 12	1.423 30	1.420 19
54	1.432 28	1.430 56	1.427 72	1.424 60
56	1.436 81	1.435 07	1.432 21	1.429 08
58	1.441 42	1.439 66	1.436 77	1.433 63
60	1.446 09	1.444 31	1.441 40	1.438 25
62	1.450 84	1.449 04	1.446 11	1.442 95
64	1.455 66	1.453 84	1.450 89	1.447 72
66	1.460 56	1.458 72	1.455 75	1.452 58
68	1.465 53	1.463 67	1.460 69	1.457 51
70	1.470 59	1.468 71	1.465 70	1.462 52
72	1.475 72	1.473 82	1.470 80	1.467 61
74	1.480 93	1.479 02	1.475 98	1.472 78
76	1.486 23	1.484 29	1.481 24	1.478 04
78	1.491 61	1.489 66	1.486 59	1.483 39
80	1.497 08	1.495 11	1.492 02	1.488 82
82	1.502 63	1.500 64	1.497 55	1.494 34
84	1.508 28	1.506 27	1.503 16	1.499 95

Table XI. Corn Syrup, High Maltose, Dual Conversion, 50 D.E., 0.4% Ash (D.B.)

% dry	Refractive index			
substance	20 °C	30 °C	45 °C	60 °C
0	1.332 99	1.331 94	1.329 85	1.327 25
2	1.335 92	1.334 84	1.332 72	1.330 09
4	1.338 94	1.337 83	1.335 68	1.333 02
6	1.342 00	1.340 87	1.338 68	1.336 00
8	1.345 11	1.343 95	1.341 73	1.339 03
10	1.348 27	1.347 08	1.344 83	1.342 11
12	1.351 48	1.350 27	1.347 98	1.345 23
14	1.354 74	1.353 50	1.351 18	1.348 41
16	1.358 05	1.356 78	1.354 44	1.351 64
18	1.361 41	1.360 12	1.357 74	1.354 92
20	1.364 82	1.363 50	1.361 10	1.358 26
22	1.368 28	1.366 94	1.364 51	1.361 65
24	1.371 80	1.370 44	1.367 97	1.365 09
26	1.375 38	1.373 99	1.371 49	1.368 59
28	1.379 01	1.377 59	1.375 07	1.372 15
30	1.382 69	1.381 25	1.378 70	1.375 76
32	1.386 43	1.384 97	1.382 39	1.379 44
34	1.390 23	1.388 74	1.386 14	1.383 17
36	1.394 09	1.392 57	1.389 94	1.386 96
38	1.398 00	1.396 47	1.393 81	1.390 81
40	1.401 98	1.400 42	1.397 74	1.394 72
42	1.406 02	1.404 44	1.401 73	1.398 69
44	1.410 12	1.408 51	1.405 78	1.402 73
46	1.414 28	1.412 65	1.409 90	1.406 83
48	1.418 51	1.416 86	1.414 08	1.411 00
50	1.422 80	1.421 13	1.418 32	1.415 23
52	1.427 16	1.425 46	1.422 64	1.419 53
54	1.431 58	1.429 86	1.427 02	1.423 90
56	1.436 07	1.434 34	1.431 47	1.428 34
58	1.440 64	1.438 88	1.435 99	1.432 85
60	1.445 27	1.443 49	1.440 58	1.437 43
62	1.449 97	1.448 17	1.445 24	1.442 09
64	1.454 75	1.452 93	1.449 98	1.446 82
66	1.459 60	1.457 76	1.454 79	1.451 62
68	1.464 52	1.462 66	1.459 68	1.456 50
70	1.469 53	1.467 65	1.464 65	1.461 46
72	1.474 61	1.472 71	1.469 69	1.466 50
74	1.479 77	1.477 85	1.474 81	1.471 62
76	1.485 01	1.483 07	1.480 02	1.476 82
78	1.490 33	1.488 37	1.485 31	1.482 11
80	1.495 73	1.493 76	1.490 68	1.487 48
82	1.501 22	1.499 23	1.496 14	1.492 94
84	1.506 80	1.504 79	1.501 69	1.498 48

oration. Plastic dropping pipets were used for this purpose when possible; Teflon covered stirring rods were used for the more viscous samples. Each sample was allowed to equilibrate to the prism temperature until three consecutive readings gave the same value. Each sample was applied to the prism three or more times.

After the temperature of the refractometer prism was established, the refractive index measurements for each of the dilutions of a particular product were made, then the next temperature was established and the refractive index for each of the dilutions measured, etc. The refractive index measurements for a single product type including all dilutions and all temperatures were made within a 2-day period to minimize the possibility of evaporation and other changes in composition. All weighing operations involving an original product were done on the same day.

Temperature Control and Measurement. The temperature of the refractometer prism was maintained through the use of a Brinkman/Lauda Model K-2/R thermostatic, external circulating water bath and monitored with calibrated A.S.T.M. No. E-1 centigrade thermometers. The thermometers used were graduated to 0.10 °C and could be read with the aid of a reading lens to ±0.02 °C without difficulty. The thermometer was mounted in a thermometer well located in the water line immediately adjacent to the inlet to the prism. This arrangement for the thermometer was necessitated by the nonavailability of thermometers (covering the temperature range of this study) for mounting in the prism itself. Experiments in which the water flow was reversed showed temperature differences ranging from 0.00° at 20 °C to a maximum of 0.05° at 60 °C. Therefore, it may be concluded that the prism temperatures could have differed from the temperature of the thermometer by no more than 0.03 °C. A temperature error of this magnitude results in a difference of refractive index of less than 0.000 01 in the worst case and is, therefore, negligible in this study.

Data Reduction

The data obtained in the experimental phase of this study were found to be expressible in the form of a mathematical function of the variables dry substance, temperature, and ash. The mathematical form of this function is:

Table XII. Corn Syrup, Dual Conversion, 63 D.E., 0.4% Ash (D.B.)

% dry substance	Refractive index			
	20 °C	30 °C	45 °C	60 °C
0	1.332 99	1.331 94	1.329 85	1.327 25
2	1.335 89	1.334 81	1.332 69	1.330 06
4	1.338 87	1.337 77	1.335 61	1.332 96
6	1.341 90	1.340 77	1.338 58	1.335 90
8	1.344 98	1.343 82	1.341 60	1.338 90
10	1.348 10	1.346 91	1.344 66	1.341 94
12	1.351 27	1.350 06	1.347 77	1.345 03
14	1.354 49	1.353 25	1.350 93	1.348 16
16	1.357 75	1.356 49	1.354 14	1.351 35
18	1.361 07	1.359 78	1.357 40	1.354 59
20	1.364 44	1.363 12	1.360 72	1.357 88
22	1.367 85	1.366 51	1.364 08	1.361 22
24	1.371 32	1.369 96	1.367 49	1.364 62
26	1.374 84	1.373 45	1.370 96	1.368 07
28	1.378 42	1.377 00	1.374 48	1.371 57
30	1.382 04	1.380 61	1.378 06	1.375 13
32	1.385 73	1.384 26	1.381 69	1.378 74
34	1.389 46	1.387 98	1.385 38	1.382 41
36	1.393 26	1.391 75	1.389 12	1.386 14
38	1.397 11	1.395 57	1.392 92	1.389 92
40	1.401 01	1.399 46	1.396 78	1.393 76
42	1.404 98	1.403 40	1.400 70	1.397 67
44	1.409 00	1.407 40	1.404 67	1.401 63
46	1.413 09	1.411 46	1.408 71	1.405 65
48	1.417 23	1.415 58	1.412 81	1.409 74
50	1.421 44	1.419 77	1.416 97	1.413 89
52	1.425 71	1.424 02	1.421 20	1.418 10
54	1.430 04	1.428 33	1.425 49	1.422 38
56	1.434 44	1.432 71	1.429 85	1.426 73
58	1.438 90	1.437 15	1.434 27	1.431 14
60	1.443 43	1.441 66	1.438 76	1.435 62
62	1.448 03	1.446 24	1.443 32	1.440 17
64	1.452 70	1.450 88	1.447 94	1.444 79
66	1.457 43	1.455 60	1.452 64	1.449 48
68	1.462 24	1.460 39	1.457 41	1.454 25
70	1.467 12	1.465 25	1.462 25	1.459 08
72	1.472 07	1.470 18	1.467 17	1.464 00
74	1.477 10	1.475 19	1.472 16	1.468 98
76	1.482 20	1.480 27	1.477 23	1.474 05
78	1.487 38	1.485 43	1.482 38	1.479 19
80	1.492 63	1.490 67	1.487 60	1.484 42
82	1.497 97	1.495 99	1.492 91	1.489 72
84	1.503 38	1.501 39	1.498 30	1.495 11

Table XIII. Corn Syrup, Dual Conversion, 70 D.E., 0.4% Ash (D.B.)

% dry substance	Refractive index			
	20 °C	30 °C	45 °C	60 °C
0	1.332 99	1.331 94	1.329 85	1.327 25
2	1.335 88	1.334 80	1.332 68	1.330 05
4	1.338 85	1.337 74	1.335 59	1.332 93
6	1.341 86	1.340 73	1.338 54	1.335 87
8	1.344 92	1.343 77	1.341 55	1.338 84
10	1.348 03	1.346 85	1.344 59	1.341 87
12	1.351 19	1.349 97	1.347 69	1.344 94
14	1.354 39	1.353 15	1.350 84	1.348 07
16	1.357 64	1.356 38	1.354 03	1.351 24
18	1.360 94	1.359 65	1.357 28	1.354 46
20	1.364 29	1.362 97	1.360 57	1.357 73
22	1.367 69	1.366 35	1.363 91	1.361 06
24	1.371 14	1.369 77	1.367 31	1.364 44
26	1.374 64	1.373 25	1.370 76	1.367 86
28	1.378 19	1.376 78	1.374 26	1.371 35
30	1.381 80	1.380 36	1.377 81	1.374 88
32	1.385 46	1.383 99	1.381 42	1.378 47
34	1.389 17	1.387 68	1.385 08	1.382 12
36	1.392 94	1.391 43	1.388 80	1.385 82
38	1.396 76	1.395 23	1.392 58	1.389 58
40	1.400 64	1.399 09	1.396 41	1.393 39
42	1.404 58	1.403 00	1.400 30	1.397 27
44	1.408 57	1.406 97	1.404 25	1.401 20
46	1.412 63	1.411 00	1.408 25	1.405 20
48	1.416 74	1.415 09	1.412 32	1.409 25
50	1.420 91	1.419 24	1.416 45	1.413 37
52	1.425 14	1.423 45	1.420 64	1.417 54
54	1.429 44	1.427 73	1.424 89	1.421 79
56	1.433 80	1.432 07	1.429 21	1.426 09
58	1.438 22	1.436 47	1.433 59	1.430 46
60	1.442 71	1.440 93	1.438 04	1.434 90
62	1.447 26	1.445 47	1.442 55	1.439 41
64	1.451 88	1.450 07	1.447 13	1.443 98
66	1.456 56	1.454 73	1.451 78	1.448 62
68	1.461 32	1.459 47	1.456 50	1.453 34
70	1.466 14	1.464 27	1.461 29	1.458 12
72	1.471 04	1.469 15	1.466 15	1.462 98
74	1.476 01	1.474 10	1.471 08	1.467 91
76	1.481 05	1.479 12	1.476 09	1.472 91
78	1.486 16	1.484 22	1.481 17	1.477 99
80	1.491 35	1.489 39	1.486 33	1.483 15
82	1.496 62	1.494 64	1.491 57	1.488 39
84	1.501 96	1.499 97	1.496 88	1.493 70

$$\frac{1}{n_D} = \frac{1}{n_{D,H_2O}}(1 + 0.027\,770\,1s)(1 - s) + (A_1 + A_2s + A_3s^2)s + B_1 + (B_2s + B_3s^2)(t - 20) + C_1 + (C_2 + C_3s)(su/100) \qquad (1)$$

Where n_D = refractive index of the solution at temperature t(°C); n_{D,H_2O} = refractive index of water at temperature t, °C; s = percent dry substance/100; u = sulfated ash, percent dry basis.

A_1, A_2, and A_3 are coefficients characteristic of the particular syrup; B_1, B_2, and B_3 are temperature correction coefficients (the same for syrups 1–8 and different for each of samples 9, 10, and 11). C_1, C_2, and C_3 are ash correction coefficients determined by the experiments on syrups 2 and 5 (commercial syrups, ion-exchanged syrups, and "spiked" syrups) and used with all of the samples. The initial form of this function was suggested by work of Barber (*1*) on the density of sucrose solutions.

The fitting of the experimental data to this functional form was carried out in a sequential and iterative series of computations. In the first stage the temperature correction coefficients B_1, B_2, and B_3 were calculated by the use of a stepwise multiple linear regression program on an IBM 1130 computer. Here the necessity of using different coefficients for samples 9 through 11 was discovered, and these coefficients were obtained. These permitted the correction of measurements for any temperature to 20 °C and thus made possible the use of all of the data for a given sample and its dilutions in stage two. This second stage was the calculation of the coefficients A_1, A_2, and A_3, which express the dependence of refractive index on dry substance for a given product. Again, the computer and stepwise multiple linear regression analysis were used to generate a set of coefficients for each of the 11 products. Up to this point, no corrections were possible for ash content, so the A coefficients were merely provisional. An analysis of the residuals (observed minus calculated values) indicated a few outliers. Reexamination of the data for these points revealed a few minor errors; when these were corrected and the A coefficients recalculated, a good fit with satisfactorily small residuals was obtained for each product.

The third stage of computation involved the development of

Table XIV. Corn Syrup, Dual Conversion, 95 D.E., 0.5% Ash (D.B.)

% dry substance	Refractive index 20 °C	30 °C	45 °C	60 °C
0	1.332 99	1.331 94	1.329 85	1.327 25
2	1.335 82	1.334 75	1.332 63	1.330 00
4	1.338 74	1.337 64	1.335 49	1.332 84
6	1.341 71	1.340 58	1.338 40	1.335 72
8	1.344 72	1.343 56	1.341 35	1.338 65
10	1.347 76	1.346 58	1.344 34	1.341 62
12	1.350 86	1.349 65	1.347 37	1.344 63
14	1.353 99	1.352 76	1.350 45	1.347 69
16	1.357 17	1.355 91	1.353 58	1.350 79
18	1.360 39	1.359 11	1.356 75	1.353 94
20	1.363 66	1.362 36	1.359 96	1.357 14
22	1.366 98	1.365 65	1.363 22	1.360 38
24	1.370 34	1.368 98	1.366 53	1.363 67
26	1.373 75	1.372 37	1.369 89	1.367 01
28	1.377 21	1.375 80	1.373 29	1.370 39
30	1.380 71	1.379 28	1.376 75	1.373 83
32	1.384 27	1.382 81	1.380 25	1.377 32
34	1.387 87	1.386 40	1.383 81	1.380 86
36	1.391 53	1.390 03	1.387 42	1.384 45
38	1.395 24	1.393 71	1.391 08	1.388 09
40	1.399 00	1.397 45	1.394 79	1.391 79
42	1.402 81	1.401 24	1.398 55	1.395 54
44	1.406 68	1.405 08	1.402 37	1.399 35
46	1.410 60	1.408 98	1.406 25	1.403 21
48	1.414 58	1.412 94	1.410 18	1.407 13
50	1.418 61	1.416 95	1.414 17	1.411 10
52	1.422 70	1.421 02	1.418 22	1.415 14
54	1.426 85	1.425 15	1.422 33	1.419 23
56	1.431 06	1.429 34	1.426 49	1.423 39
58	1.435 33	1.433 59	1.430 72	1.427 61
60	1.439 66	1.437 90	1.435 01	1.431 89
62	1.444 05	1.442 27	1.439 36	1.436 23
64	1.448 51	1.446 70	1.443 78	1.440 64
66	1.453 03	1.451 20	1.448 26	1.445 11
68	1.457 61	1.455 77	1.452 80	1.449 65
70	1.462 26	1.460 40	1.457 42	1.454 26
72	1.466 98	1.465 10	1.462 10	1.458 93
74	1.471 77	1.469 86	1.466 85	1.463 68

Table XV. High Fructose Corn Syrup, 94 (Dextrose + Fructose) (D.B.), 0.05% Ash (D.B.)

% dry substance	Refractive index 20 °C	30 °C	45 °C	60 °C
0	1.332 99	1.331 94	1.329 85	1.327 25
2	1.335 83	1.334 75	1.332 62	1.329 99
4	1.338 74	1.337 63	1.335 46	1.332 79
6	1.341 69	1.340 54	1.338 33	1.335 64
8	1.344 68	1.343 50	1.341 25	1.338 53
10	1.347 71	1.346 50	1.344 22	1.341 46
12	1.350 79	1.349 55	1.347 23	1.344 44
14	1.353 91	1.352 64	1.350 28	1.347 46
16	1.357 08	1.355 78	1.353 38	1.350 53
18	1.360 29	1.358 96	1.356 52	1.353 64
20	1.363 55	1.362 18	1.359 71	1.356 80
22	1.366 85	1.365 46	1.362 95	1.360 01
24	1.370 20	1.368 78	1.366 23	1.363 27
26	1.373 61	1.372 15	1.369 57	1.366 58
28	1.377 06	1.375 57	1.372 95	1.369 93
30	1.380 55	1.379 04	1.376 38	1.373 34
32	1.384 10	1.382 56	1.379 87	1.376 80
34	1.387 71	1.386 13	1.383 40	1.380 31
36	1.391 36	1.389 75	1.386 99	1.383 87
38	1.395 06	1.393 43	1.390 63	1.387 48
40	1.398 82	1.397 16	1.394 32	1.391 15
42	1.402 63	1.400 94	1.398 07	1.394 87
44	1.406 50	1.404 78	1.401 87	1.398 65
46	1.410 42	1.408 67	1.405 73	1.402 49
48	1.414 40	1.412 62	1.409 65	1.406 38
50	1.418 44	1.416 63	1.413 63	1.410 34
52	1.422 54	1.420 70	1.417 66	1.414 35
54	1.426 69	1.424 82	1.421 75	1.418 42
56	1.430 91	1.429 01	1.425 91	1.422 55
58	1.435 19	1.433 26	1.430 12	1.426 75
60	1.439 52	1.437 57	1.434 40	1.431 00
62	1.443 93	1.441 94	1.438 75	1.435 33
64	1.448 39	1.446 38	1.443 15	1.439 71
66	1.452 93	1.450 89	1.447 63	1.444 17
68	1.457 52	1.455 46	1.452 17	1.448 69
70	1.462 19	1.460 10	1.456 77	1.453 28
72	1.466 92	1.464 80	1.461 45	1.457 93
74	1.471 73	1.469 58	1.466 20	1.462 66
76	1.476 60	1.474 43	1.471 02	1.467 46

the ash correction coefficients. Once again the computer and the regression analysis program were used to calculate the necessary coefficients from the data obtained with ash variations. With these in hand, the optimal set of A coefficients were recalculated on the basis of data corrected to zero ash.

Attempts to correlate the dry substance–refractive index relationships with D.E. showed that D.E. alone is not adequate for representing the differences between products of widely differing compositions. A model based on the saccharide compositional data as expressed in Table II (including D.E.) was developed and found to successfully represent the data for all of these products. A full description of this model is to be pub-

Table XVI. Maltodextrin, 12 D.E., 0.4% Ash (D.B.)

% dry substance	Refractive index 20 °C	30 °C	45 °C	60 °C
0	1.332 99	1.331 94	1.329 85	1.327 25
2	1.335 99	1.334 92	1.332 80	1.330 18
4	1.339 07	1.337 97	1.335 83	1.333 19
6	1.342 20	1.341 08	1.338 91	1.336 25
8	1.345 38	1.344 24	1.342 04	1.339 36
10	1.348 62	1.347 45	1.345 23	1.342 53
12	1.351 91	1.350 72	1.348 47	1.345 75
14	1.355 26	1.354 05	1.351 76	1.349 02
16	1.358 66	1.357 42	1.355 11	1.352 36
18	1.362 12	1.360 86	1.358 52	1.355 75
20	1.365 64	1.364 35	1.361 99	1.359 19
22	1.369 21	1.367 90	1.365 51	1.362 69
24	1.372 84	1.371 51	1.369 09	1.366 26
26	1.376 53	1.375 17	1.372 73	1.369 88
28	1.380 28	1.378 90	1.376 43	1.373 56
30	1.384 10	1.382 69	1.380 19	1.377 30
32	1.387 97	1.386 54	1.384 01	1.381 10
34	1.391 91	1.390 45	1.387 90	1.384 97
36	1.395 91	1.394 43	1.391 84	1.388 90
38	1.399 97	1.398 47	1.395 86	1.392 90
40	1.404 10	1.402 57	1.399 93	1.396 96
42	1.408 30	1.406 74	1.404 08	1.401 08
44	1.412 56	1.410 98	1.408 29	1.405 28
46	1.416 90	1.415 29	1.412 57	1.409 54
48	1.421 30	1.419 67	1.416 92	1.413 87
50	1.425 77	1.424 11	1.421 34	1.418 27
52	1.430 31	1.428 63	1.425 83	1.422 74
54	1.434 93	1.433 22	1.430 39	1.427 29

Table XVII. Factors for Approximate Calculation of Refractive Index at Temperatures from 10 to 70 °C[a]

t, °C	Water	28 to 95 D.E. syrups	High fructose corn syrup	Malto-dextrin
10	0.000 716	0.002 260	0.002 590	0.002 300
12	0.000 605	0.001 808	0.002 072	0.001 840
14	0.000 477	0.001 356	0.001 554	0.001 380
16	0.000 333	0.000 904	0.001 036	0.000 920
18	0.000 174	0.000 452	0.000 518	0.000 460
20	0.000 000	0.000 000	0.000 000	0.000 000
22	−0.000 187	−0.000 452	−0.000 518	−0.000 460
24	−0.000 389	−0.000 904	−0.001 036	−0.000 920
26	−0.000 603	−0.001 356	−0.001 554	−0.001 380
28	−0.000 829	−0.001 808	−0.002 072	−0.001 840
30	−0.001 068	−0.002 260	−0.002 590	−0.002 300
32	−0.001 318	−0.002 712	−0.003 108	−0.002 760
34	−0.001 579	−0.003 164	−0.003 625	−0.003 220
36	−0.001 850	−0.003 616	−0.004 144	−0.003 680
38	−0.002 133	−0.004 068	−0.004 662	−0.004 140
40	−0.002 425	−0.004 520	−0.005 180	−0.004 600
42	−0.002 727	−0.004 972	−0.005 698	−0.005 060
44	−0.003 039	−0.005 424	−0.006 216	−0.005 520
46	−0.003 360	−0.005 876	−0.006 734	−0.005 980
48	−0.003 690	−0.006 328	−0.007 251	−0.006 440
50	−0.004 029	−0.006 780	−0.007 770	−0.006 900
52	−0.004 377	−0.007 232	−0.008 288	−0.007 360
54	−0.004 734	−0.007 684	−0.008 806	−0.007 820
56	−0.005 099	−0.008 136	−0.009 324	−0.008 280
58	−0.005 472	−0.008 588	−0.009 842	−0.008 740
60	−0.005 853	−0.009 040	−0.010 360	−0.009 200
62	−0.006 331	−0.009 492	−0.010 878	−0.009 660
64	−0.006 760	−0.009 944	−0.011 396	−0.010 120
66	−0.007 168	−0.010 396	−0.011 914	−0.010 580
68	−0.007 586	−0.010 848	−0.012 432	−0.011 040
70	−0.008 035	−0.011 300	−0.012 949	−0.011 500

[a] The approximate refractive index change with temperature can be represented as the sum of the water effect and the solids effect: $n\mathrm{D}^t = n\mathrm{D}^{20} + F_\mathrm{w}(1 - s) + F_\mathrm{s}(s)$ where s = percent dry substance/100, F_w = water factor from table, F_s = solids factor from table, appropriate to the product type. Exact refractive index at any temperature can be calculated from eq 1, but this is a tedious procedure. This table is presented as a convenient alternative.

Table XVIII. Ash and D.E. Corrections

% dry substance	Change in n_d for an increase of: 1% Ash	1 D.E.
2	0.000 000	−0.000 001
4	0.000 000	−0.000 003
6	0.000 001	−0.000 005
8	0.000 002	−0.000 007
10	0.000 003	−0.000 010
12	0.000 004	−0.000 012
14	0.000 006	−0.000 015
16	0.000 008	−0.000 017
18	0.000 010	−0.000 020
20	0.000 013	−0.000 023
22	0.000 016	−0.000 026
24	0.000 019	−0.000 029
26	0.000 022	−0.000 033
28	0.000 026	−0.000 036
30	0.000 030	−0.000 040
32	0.000 034	−0.000 044
34	0.000 039	−0.000 048
36	0.000 044	−0.000 052
38	0.000 049	−0.000 057
40	0.000 055	−0.000 061
42	0.000 061	−0.000 066
44	0.000 068	−0.000 071
46	0.000 074	−0.000 076
48	0.000 082	−0.000 081
50	0.000 089	−0.000 087
52	0.000 097	−0.000 093
54	0.000 105	−0.000 099
56	0.000 114	−0.000 105
58	0.000 123	−0.000 112
60	0.000 133	−0.000 118
62	0.000 143	−0.000 125
64	0.000 153	−0.000 132
66	0.000 164	−0.000 140
68	0.000 175	−0.000 147
70	0.000 187	−0.000 155
72	0.000 199	−0.000 163
74	0.000 212	−0.000 172
76	0.000 225	−0.000 181
78	0.000 239	−0.000 190
80	0.000 253	−0.000 199
82	0.000 268	−0.000 208
84	0.000 283	−0.000 218

lished elsewhere (*4*). On the basis of this model a few of the original dry substance values appeared to be slightly out of line; these were rechecked experimentally and the averages of the original and the later work used in a repeat computation with the composition model. This led to excellent agreement between calculated and observed values for the entire set of data, covering temperature, ash, and dry substance variation over the complete range of compositions. While this model and computation are beyond the scope of this report, they are mentioned here because of their use in suggesting questionable points for further work and because they indicate the internal consistency of these data.

Finally, the entire set of experimental data was reanalyzed in the manner already described. The final values of the coefficients to be used with eq 1 are given in Table V. The supplementary tables include both the experimental values and values calculated with eq 1 along with the corresponding residuals. Of the total of 433 points listed in the data tables, only four have residuals greater than 0.000 20 and only 32 have residuals greater than 0.000 10. The residuals for the 0.0% dry substance entries are noteworthy in that these experimental values were *not* included in the regression analysis that led to the coefficients used with eq 1. Thus, these calculated values represent an extrapolation from the data on actual product solutions, and the small size of these residuals (0.000 02–0.000 05) is an indication of the quality of the experimental points and of the curve fitting procedures.

Dextrose Equivalent Corrections. Corrections for small D.E. variations (on the order of ±2 D.E.) within a product type are possible and can be simply expressed as a correction in the form:

$$n_\mathrm{D}(\text{corrected}) - n_\mathrm{D}(\text{original}) = K[\text{D.E.(new)} - \text{D.E.(original)}] \quad (2)$$

Here K, the proportionality constant, is the same for all products but is dependent on the dry substance of the sample. Analysis of the data for the products with D.E. values ranging from 12 to 95, at eight dry substance levels (as calculated from eq 1) yielded the expression for K given below.

$$K \times 10^5 = -8.895s - 12.5361s^2 - 9.36838s^3 \quad (3)$$

Results

The raw data of the experimental work are given in the supplementary tables. Subsequent data reduction and curve fitting yielded eq 1 and the coefficients given in Table V, and provided the calculated values and residuals for all samples analyzed. The equation and appropriate coefficients have been used to generate the more convenient Tables VI–XVI. These tables represent commercial products in terms of D.E. (where appropriate) and ash levels, and are much more detailed in their expression of the dry substance dependence of refractive index.

Table XV for high fructose corn syrup was calculated in a similar manner even though the product is ordinarily not characterized by D.E. Sample 10 contained 93.1% (dry basis) dextrose plus fructose, and the observed D.E. was 94.1. Table XV represents a product containing 94.0% (dry basis) dextrose plus fructose, and has a calculated D.E. value of 94.6.

Correction factors for small variations in temperature, ash, and D.E. are given in Tables XVII and XVIII. The refractive index and dry substance values in Tables VI through XVI, higher than the experimental data originally obtained, were extrapolated within reasonable limits using eq 1 and the coefficients listed in Table V. Interpolation for intermediate values of dry substance should be easy and obvious.

Conclusions

Careful experimental work and extensive data analysis have been combined to produce accurate and useful tables, detailing the relationship between refractive index and dry substance content for a wide range of commercial corn syrups and maltodextrin. With these tables, measurements of refractive index may be used to obtain the dry substance content of corn syrups and maltodextrin solutions with speed, precision, and accuracy comparable to that of the vacuum oven–filter aid method.

Acknowledgments

The financial support, the aid in obtaining samples, and the coordination of this work with that of the collaborating laboratories by the Corn Refiners Association Inc. is gratefully acknowledged. Mr. R. J. Smith, Mr. E. A. Schleichert, and Mr. F. A. Kurtz, of CPC International Inc., were particularly helpful with advice and encouragement during the course of this work.

Literature Cited

(1) Barber, E. J., "Calculation of the Density and Viscosity of Sucrose Solutions", Union Carbide Corp., Nuclear Division, Oak Ridge Gaseous Diffusion Plant, Oak Ridge, Tenn., June 24, 1965 (K-1635).
(2) Cleland, J. E., Evans, J. W., Fauser, E. E., Fetzer, W. R., *Ind. Eng. Chem., Anal. Ed.*, **16**, 161–165 (1944).
(3) Graefe, G., *Die Stärke*, **13**, 402–404 (1961).
(4) Kurtz, F. A., et al., to be submitted for publication.
(5) Malinsky, V., *Die Stärke*, **11**, 63–67 (1959).
(6) Method E-6, Standard Analytical Methods of the Member Companies of the Corn Refiners Association, Inc., 1001 Connecticut Avenue N.W., Washington, D.C.
(7) Method E-26, *ibid.*
(8) Method E-42, *ibid.*
(9) Method E-46, *ibid.*
(10) Method E-62, *ibid.*
(11) Tilton, L. W., Taylor, J. K., *J. Res. Natl. Bur. Stand.* **20**, 419–477 (1938).
(12) Thoburn, J. M., *Int. Sugar J.*, **66**, 205–207 (1966).
(13) Zerban, F. W., Martin, M., *J. Assoc. Off. Agric. Chem.*, **27**, 295 (1944).

Received for review October 28, 1975. Accepted June 14, 1976.

Supplementary Material Available: Experimental data and calculated residuals on the refractive index of ten different commercial corn syrups and one maltodextrin at 20, 30, 45, and 60 °C (14 pages). Ordering information is given on any current masthead page.

Thermodynamics of the System H_2O–NaH_2PO_4–H_3PO_4

Robert F. Platford
Canada Centre for Inland Waters, Burlington, Ontario, Canada L7R 4A6

The excess free energies of mixing for the system H_2O–NaH_2PO_4–H_3PO_4 have been determined from vapor pressure measurements at 273.15 and 298.15 K. They are the same at the two temperatures, and, in common with other phosphate mixtures studied to date, are negative.

Mixtures of orthophosphoric acid with simple water-soluble salts are important models for phosphate fertilizers. Furthermore, such systems are of considerable theoretical interest because of the atypical behavior they exhibit which results from hydrolysis of the orthophosphate anion. The existence in phosphate solutions of the dimer $(H_2PO_4)_2^{2-}$ has been demonstrated by free energy measurements made in this laboratory (*9*), and, in view of the recent interest shown in phosphates in mixed-salt solutions (*5, 8*), it seems appropriate to have data on a mixture of a simple phosphate salt with phosphoric acid. The solubility diagram for the aqueous system NaH_2PO_4–H_3PO_4 has been reported on (*4*) but, although water activities also are plotted in the diagram, they are unfortunately not given with sufficient precision to permit the calculation of free energies. For this reason, I have redetermined the water activities in this system.

Experimental Section

The method used was that in the previous papers in this series (*1, 2, 6, 7*) (I neglected to refer to ref 2 in ref 7; in the region of overlap our results for $\phi(H_3PO_4)$ agree to within 0.01.) and in addition to the 298 K measurements, a further few measurements were made at 273 K. The isopiestic concentrations are given in Table I, with the phosphoric acid treated as a 1–1 electrolyte. A power series in I, the ionic strength of each mixture, was fitted to the data, using the expression:

$$(2y_A + k_B y_B)\phi - 2y_A\phi_A{}^0 - k_B y_B \phi_B{}^0 = y_A y_B(b_{01}I + b_{02}I^2) \qquad (1)$$

where k_B, a constant characteristic of the electrolyte, is 2. As explained in the discussion, the data were recalculated after treating the H_3PO_4 as, next, a 1–2 electrolyte ($k_B = 1$) and, finally, a 1–3 electrolyte ($k_B = \frac{2}{3}$). This, of course, necessitated a recalculation of the y and ϕ values in each case. The b coefficients in eq 1 are related to the excess Gibbs energy of mixing in the system $\Delta_m G^E$ through Friedman's g_0 function (*2*)

$$b_{01} + \tfrac{1}{2}b_{02} = g_0 = \Delta_m G^E/(RTI^2 y_A y_B) \qquad (2)$$

The values for the mixtures are plotted in Figure 1 for the three