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TESTING WHEAT FOR PROTEIN AND MOISTURE WITH THE AUTOMATED DIGITAL ANALYZER

P. C. WILLIAMS, S. G. STEVENSON, and G. N. IRVINE,[1] Canadian Grain Commission, Grain Research Laboratory, Winnipeg, Man. R3C 3G9

ABSTRACT Cereal Chem. 55(3): 263-279

The automated digital analyzer (ADA) is a system developed for the automated analysis of up to 2,500 samples of cereal grain for protein and moisture on a routine daily basis. The system operates through near-infrared reflectance spectroscopy. Unlike other equipment of its type, the instrument is not hard wired to any particular wavelengths. Instead, the operator sets up wavelength areas within which the system is free to select the wavelengths that are most significant for the material being analyzed. An on-line digital computer performs the complicated computations involved in the calibration process. Samples are presented at an average rate of one every 9 sec, including time for scanning the ceramic standard. Sample presentation is achieved by conveyor chains, which are activated by a series of microswitches. To date, standard deviations of 0.27 with respect to Kjeldahl protein and 0.24 with respect to oven moisture have been achieved for hard red spring wheat under on-line high-volume conditions. The ADA is well suited to large-scale testing such as that involved in the protein segregation program of the Canadian Grain Commission and in cereal breeding programs.

On August 1, 1971, the Canadian government commenced the marketing of the top milling grades of hard red spring (HRS) wheat on the basis of guaranteed minimum protein levels. Wheat was segregated into subgrades at designated protein levels at terminal grain elevators at the ports of Thunder Bay, Ont.; Vancouver, B.C.; and Churchill, Man. The Canadian Grain Commission formulated the mechanism of segregation in collaboration with the University of Manitoba. Dunne and Anderson (1,2) have described the basic principles of the system. The tests on which the segregation was effected were performed in large-scale Kjeldahl laboratories in Winnipeg; Calgary, Alta.; and Thunder Bay by a semiautomated macro-Kjeldahl process (3).

The segregation program calls for testing more than 500,000 samples annually. Since June 2, 1975, all of the testing on which the program is based has been conducted at Winnipeg by means of an automated apparatus that embodies the principles of near-infrared reflectance spectroscopy (NIRS) and data reduction by digital computerization. The equipment, which is known as the automated digital analyzer (ADA) is a unique, one-of-a-kind system. The Neotec

[1]Paper No. 386 of the Canadian Grain Commission, Grain Research Laboratory, 1404-303 Main St., Winnipeg, Man. R3C 3G9.

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Corporation, Silver Spring, MD, designed and constructed the system to the Canadian Grain Commission's specifications. Briefly, the specifications stipulated: 1) a throughput of ten tests per minute, 2) a standard error of ±0.15% protein, 3) capacity to test simultaneously for protein and moisture, and 4) printout of results on a constant 13.5% moisture basis.

MATERIALS AND METHODS

Instrument Configuration

The equipment incorporates twin automatic sample presentation units, each of which carries a Neotec Model 31 sensing head. The reflectance data is stored on magnetic tape and relayed to a Data General Nova Model 1220 digital computer for processing; the results are printed on a teletype teleprinter. Figure 1 portrays the complete layout of the system, including sample presentation unit, teletype, computer, and link tape drive (Computer Operations, Inc., Bowie, MD). The second sample presentation unit is not shown.

The samples of ground wheat are presented in aluminum trays, each of which carries ten cells with special infrared-transparent glass bottoms. The glasses are sealed in place.

The Neotec sample cell cap was modified for ease of handling large numbers of

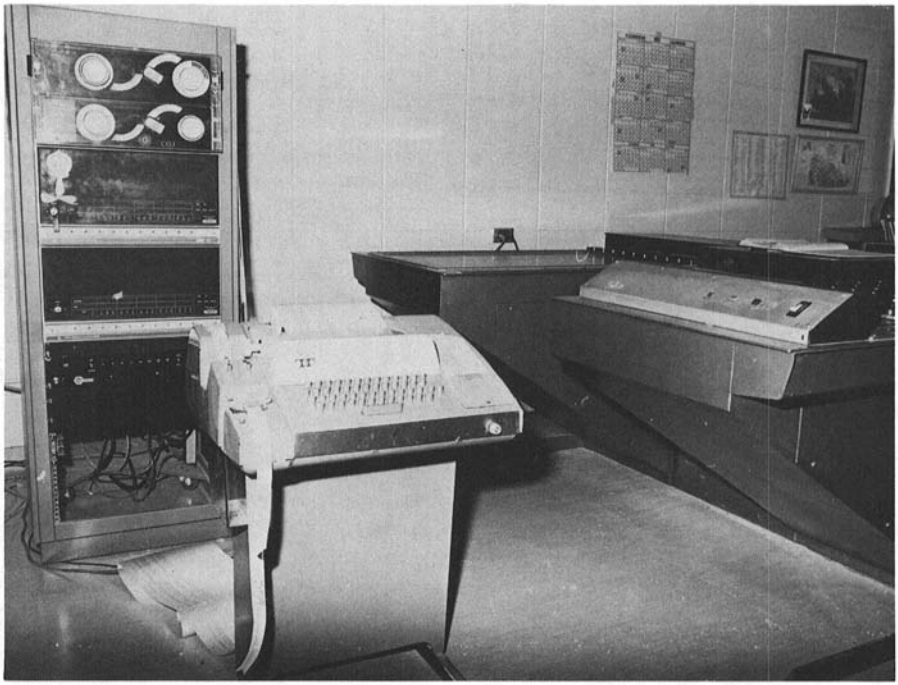

Fig. 1. General layout of ADA. R. to L.: Sample presentation unit, teletype, computer.

samples. The springs of the spring-loaded plastic compression disk were discarded, and the plastic disk cemented onto a circular piece of foam rubber, which in turn was cemented directly to the inside of the cell cap. The cell cap became both cell cover and compression disk, eliminating one operation involved in cell loading.

The foam disks are 30 mm in diameter and 10 mm thick. Loading a 10-cell tray involves overfilling each cell with the appropriate sample, leveling all ten surfaces, and positioning the ten twist-lock caps. Vacuum suction removes residual meal. A tray of ten samples can be loaded in about 90 sec.

Chains move the trays within the sample presentation unit; a series of microswitches control the driving motors of the unit (Fig. 2). Switch No. 1 is a fail-safe mechanism. The end cell (No. 10) on each tray is inset 2 mm from the end of the tray, whereas No. 1 cell is inset 1 mm from the end. If the tray is reversed during loading, which would jam the lateral transfer mechanism and completely disrupt the sample identification, the No. 1 microswitch is activated. This shuts down the entire system, and the tray has to be removed manually. Under normal operating circumstances when the computer commands the system to access samples, the first tray moves into the sensing compartment in the direction that the arrows indicate.

When the front edge of the tray touches switch No. 2, the forward motion is arrested and lateral movement commences. Simultaneously, the front end of the tray activates switch No. 4. This alerts the computer to commence recording data for the first sample in the approaching tray.

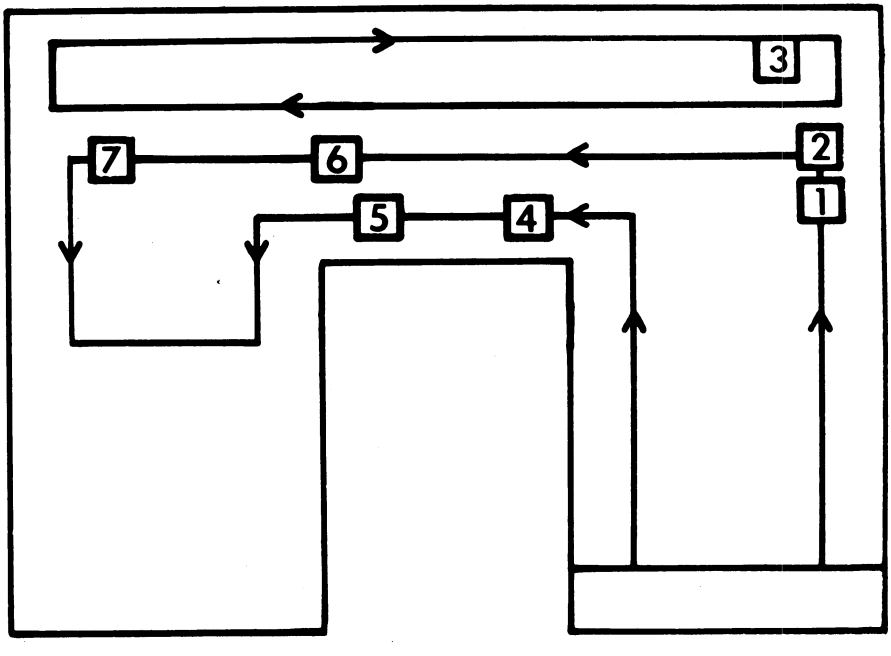

Fig. 2. Plan of ADA microswitch control.

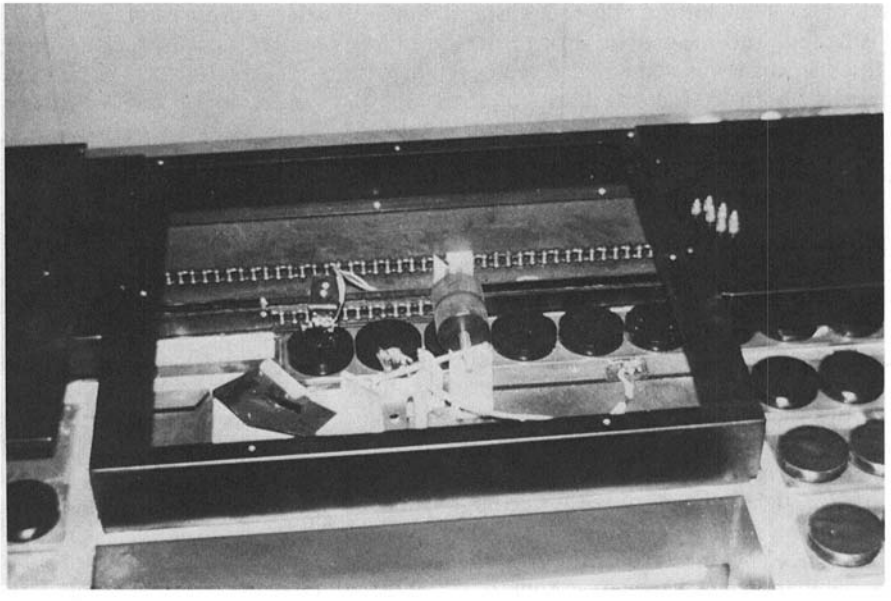

Fig. 3. Sample tray entering ADA.

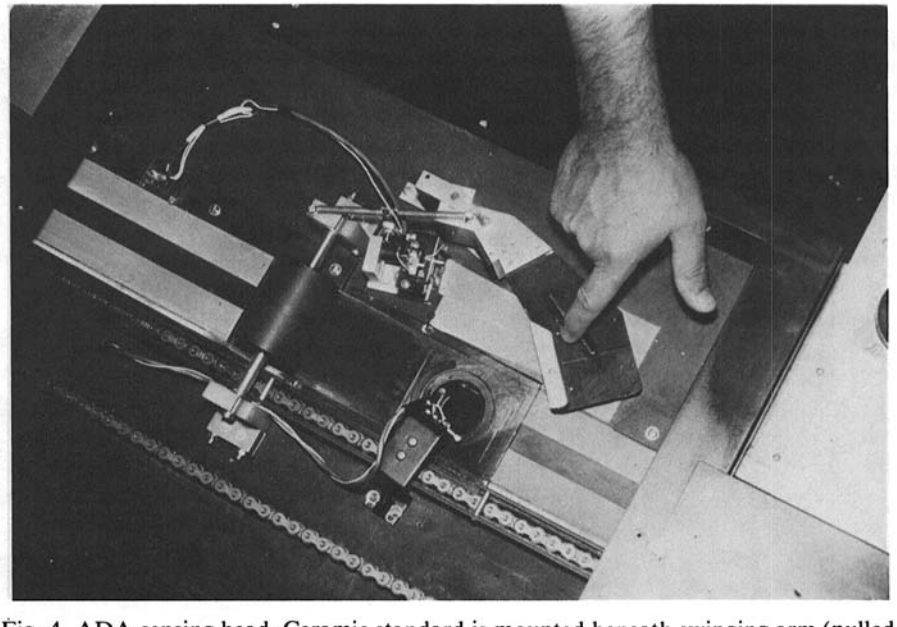

Fig. 4. ADA sensing head. Ceramic standard is mounted beneath swinging arm (pulled aside).

Switch No. 5 is mounted on a swinging arm (Fig. 3). A Coor's ceramic standard plate is attached to the underside of the swinging arm and covers the sensing head (Fig. 4). The Model 31 sensing unit is inverted to receive the reflectance signal from the bottom of the sample cells rather than from the conventional overhead position. This format, coupled with the tray design, enables fast cell loading. When the front end of the tray moves the swinging arm aside, switch No. 5 informs the computer that a sample is in position. The tilting filter of the Model 31 sensing unit rotates at 360 rpm. The cell remains in position for about 5 sec, permitting 30 readings per sample. The tray is then moved so that the second sample cell is in position over the sensing head, and the process is repeated until all ten samples have been read.

Switch No. 6 is a light-emitting diode. A beam of light is shone across the tray access area, and each individual cell interrupts it. The cells are separated by 2.5-mm gaps so that by means of switch No. 6, the computer counts the cells as they pass. After the tenth sample has passed, the tray is moved until the swinging arm replaces the standard over the sensing head. The ceramic standard is read for about 20 sec, or 120 readings, after which the tray is moved laterally until its front end impinges on switch No. 7.

Switch No. 7 simultaneously activates the ejector motor, which carries the tray out of the system, and the access motor, which brings the next tray into position. The combination of switches 4, 5, and 6 causes readings to be taken for the correct number of revolutions for each of the ten samples and for the standard. The number of trays is also counted as a result of the repeated stimulation of switch No. 4 so that the computer can tell if and when the requisite number of trays has been processed. Finally, switch No. 3 arrests lateral movement; one of the two driving lugs on the chain that is responsible for lateral movement activates it. This coincides with the arrival of the front end of a tray in the position where it activates switch No. 7.

The sensing head carries three filters. The first, or moisture, filter scans from 1.86 to 1.97 μm; the second, or protein, filter, from 2.08 to 2.19 μm; and the third, or oil, filter, from 2.20 to 2.32 μm. The signals received from sample and standard are amplified and relayed to the Nova 1220 computer. This is a 16-K, 35-bit computer that records incoming data in the form of log 1/R (apparent reflectance) values. The ADA is the first NIRS instrument ever to process reflectance data via the delta R/R or DR/R algorithm. The area of the near-infrared spectrum that each filter scans is divided arbitrarily into 100 data points, each of which corresponds to an array of wavelength; the exact dimension of the wavelength depends on the filter.

According to the principle of the tilting filter, each filter scans an area equivalent to about 5% of the designated wavelength of the filter during a single effective revolution. For example, the protein, or 2.19-μm, filter scans an area of about 1.095 μm. This means that each of the 100 data points corresponds to about 11 Å. This is an oversimplification of the true situation, since not all data points are equivalent in energy, but it illustrates the working principle of the instrumentation. At each data point, the instrument records the log 1/R value and, by the DR/R algorithm, sums the readings for a certain number of data points (in this case, five) above and below each individual point to give a delta R value. This value is then divided by the absolute reflectance at the primary point. This is the final DR/R value. Finally, the computer condenses the 300 data

points per revolution into 100 points. In this way, the total amount of data collected for the sample or the standard is smoothed to minimize the influence of system noise and is shrunk to allow the storage of data for a greater number of samples on the magnetic tape. Each tape will store data from about 500 samples.

A switch interface that Neotec designed enables the system to be switched from one sample presentation unit to the other. Since its original construction, a second dedicated Nova computer has been added, which enables both sample presentation units to be used simultaneously. At the front of each sample presentation unit, a small switchboard enables the operator to access each individual batch of trays (one to ten per batch). Other switches allow the system to be halted in the event of a jam-up, and trays can be manually accessed and passed through the system for troubleshooting purposes.

Programming and Options

Nova 1220 computers carry certain programming that enables the operator to control the computer. These computers are described in detail in their operating manuals and include programs such as FILES, which prints out details of all files stored in the computer; DUPTAP, which enables the duplication of magnetic tapes; RENAME; RUBOUT; SQUASH; and EDIT. In addition, Neotec supplied certain options at the behest of the Grain Commission. These include:

GDA. This program is used to record log 1/R data for each sample and has to be used for all samples, including calibration and "unknown" samples. Calibration constants are printed each time that the program is selected. Thereafter, batches of samples may be recorded in sequence until the magnetic tape is filled. On accessing the number of trays to the computer, advance information is given if the tape is too full to receive the total of the last batch. Samples must be read in batches of ten. A minimum of one and a maximum of ten trays (100 samples) can be processed in a batch. Results are printed via the teletype. Setting a switch on the interface determines whether protein results are printed on an as-is or a constant moisture (13.5%) basis. A fail-safe mechanism to protect against reporting all results as is instead of at a 13.5% moisture basis is the inability of the GDA program to be loaded with the switch in the as-is position.

CAL. This is the calibration program that is used in conjunction with GDA. After the calibration samples are read, the standard analytic data is accessed to the computer via punched paper tape. The paper tape is generated manually at the teletype and usually takes the form of Kjeldahl protein (4) on an as-is moisture basis, together with single-stage air oven moisture values (5).

CAL is a multiple linear regression program that computes up to 6-K values (calibration constants) and an intercept (Ko) by regressing the DR/R values at each of the 100 points against the respective standard figures. The standard error of estimate and selected wavelength point are printed out alongside the K values. This enables the operator to ascertain whether the computer is searching the optimum area for its regression.

The ADA can be calibrated for oil, protein, starch, and moisture, and contains filters adequate for all four measurements. In wheat, only protein and moisture are estimated, although during the applied research done in the first year of operation, the ADA was calibrated to read oil as well as protein in rapeseed. The calibration can be done after the fact, and need not be done immediately after running the samples provided that the correct file name is accessed to comply

with the analytic data. File names can carry up to six alphanumeric characters.

LDA. This program enables the operator to recalculate the protein and moisture data for any files that are recorded on a magnetic tape by using a fresh calibration. If data for a number of samples comprising, *e.g.*, 70 batches are recorded on one or more magnetic tapes, their protein and moisture contents can be calculated after calibration by calibrating and then accessing LDA. This is useful in light of the large area of western Canada over which the daily workload is drawn. Occasionally the results of check sample analysis during the day reveal that a fresh calibration is needed due to a major change in the growing location of the samples that are newly arrived in Winnipeg. The fresh calibration is run and all preceding files recalculated, which improves the accuracy of the testing.

SELPT. This program is used to assign the area of wavelength over which the computer can search for the optimum wavelengths for the measurement of protein and moisture. The operator assigns the start and finish point for each filter. Between these two points, the computer finds the most suitable points for measurement. If the most suitable point or points lie outside the deisgnated bands, the computer tries to get as close as possible to the correct point, and in doing so, butts against the end of the band. CAL indicates the wavelength points that the computer selects. By the magnitude of the points selected, the operator can assess whether the computer has selected points within the bands assigned, and whether shifting one or more of the bands is necessary while not using up any more overall points.

Table I indicates typical wavelength points assigned to the ADA. The "wavelength points used" now represents the cumulative number of points used after the assignment of bands.

Table II illustrates a typical set of K values for protein, together with the wavelength points that the ADA selects via its multiple linear regression. In this case, the wavelengths selected for the moisture and protein filters were 18 and 80, and 51 and 78, respectively, which correspond roughly to wavelengths of 1,882 nm and 1,956 nm for moisture and 2,136 nm and 2,166 nm for protein. The oil filter selections of 84 and 96 correspond to wavelengths in the neighborhood of 2,301 nm and 2,315 nm, but the lower wavelength (point 5) occurs at the lower limit of the band assigned. If the respective band levels had been set at, *e.g.*, 69 and 88, the computer likely would have selected a point within the band. Since this is the oil filter, our experience is that shifting these bands would be unlikely to improve the efficiency of the calibration. Had it been the protein filter, *i.e.*, points 3 or 4, which indicated that further scope was necessary, a band shift likely would have improved protein prediction. When wavelength selections indicate

TABLE I
Typical Wavelength Point Selections for ADA

Filter	1		2		3	
Band	1	2	3	4	5	6
Starting number	10	87	18	65	41	72
End number	25	100	30	95	50	91
Wavelength points used	16	30	43	74	84	100

that the banding must be expanded as well as shifted, wavelength points have to be borrowed from one of the other filters. The oil filter is usually used for provision of extra wavelength points, since our research in other areas of NIRS technology has indicated that the oil filter contributes the least of the three to prediction of protein in cereals.

The order of point assignment is of fundamental importance and is selected by a stepwise multiple linear regression program at the Neotec spectrocomputer facility. Readings are taken for 50 HRS wheat samples that the Grain Commission supplies and the range from 18,000 to 24,000 Å is scanned. A total of six wavelength points are assigned, two to each of the three filters of the tilting filter system. Points 1 and 2 are assigned to the moisture filter, 3 and 4 to the protein, and 5 and 6 to the oil filters. Wavelengths were originally selected by the stepwise regression program in the order 4, 1, 6, 3, 2, 5 for protein and 2, 4, 6, 1, 3, 5 for moisture.

The order in which the ADA uses wavelengths is assigned via the SELPT program. Zero values are allotted to wavelengths for constituents for which no analysis is to be done, usually oil and starch. Although the CAL program selects 6-K values for each constituent, the operator has the option of using less. The pattern of the K values illustrates the possible usefulness of each K value. The most important constant is the first, which should appear as the largest, and is usually over 1,000. If the last point or two are of a high order of magnitude, *e.g.*, 600–1,000, it is likely that they have been influenced by electronic noise in the system rather than by true log 1/R signal. When this occurs, even though they often appreciably lower the standard error of estimate, they should be eliminated since they are unlikely to add to the efficiency of predicting unknowns. This is frequently the case for moisture calibrations. Table III illustrates such a calibration. In this case, the last point would be eliminated.

CLETAP. This program clears the magnetic tape of all accumulated data and enables the reuse of tapes. A magnetic tape usually lasts for about six months before it becomes unusable. Symptoms are difficulties in calling in the program, which means that the entire programming needs to be rebootstrapped in, and rapid exhaustion after accumulation of only a few samples.

TABLE II
Typical Data for ADA Protein Calibration[a]

K	K Value	SEE[b]	Wavelengths Selected by ADA[c]	Calibration Band Sequence
0	5.353	...	...	...
1	−1189.0	0.2870	78	4
2	198.4	0.2456	18	1
3	646.7	0.2125	96	6
4	105.0	0.2010	51	3
5	95.94	0.1970	80	2
6	313.1	0.1930	84	5

[a]ADA protein = 13.98, Kjeldahl protein = 13.97, SD, 0.223.
[b]SEE = standard error of estimate (calibration).
[c]Arbitrary numbers corresponding to specific wavelengths in range, 1,860–2,320 nm.

BASICA. This option enables the computer to be used as a computer separate from the ADA, with programs written in BASIC language. Extensive use is made of the BASIC programming in statistical analysis of check sample data, checking the accuracy of calibrations against standard laboratory data, calibration of the 37 portable NIRS instruments that the Grain Commission uses, general statistical analysis, and program writing and debugging in BASIC. To date, about 20 BASIC programs are in use on the ADA, extending from simple moisture calculation programs to stepwise multiple linear regression.

Calibration

The ADA is calibrated every day. A set of 50 fresh samples selected to provide about seven samples at each of seven steps from 10 through 16% are analyzed for protein by the Kjeldahl process after the ADA has been used for their original selection. These samples are also tested for single-stage air-oven moisture. Recommended AACC methods are used (4,5), with slight modification of the Kjeldahl process to accommodate the automation. A punched paper data tape is prepared the night before calibration, *i.e.*, on the same day as the analysis. For calibration, the trays are loaded and read as for normal samples, and the system is calibrated via the CAL program.

The next 50 unknown, or fresh, samples are analyzed by ADA and standard methods, and the results subjected to statistical analysis. If the results indicate that the new calibration is superior to the existing calibration, the new calibration is used and the day's results are recalculated using LDA. If the new calibration is not superior, the original calibration persists. Some calibrations last several weeks.

Calibrations can be stored as punched paper tapes, and one of the exercises done earlier was to analyze the 50 check samples on a constant tape, which was used every day for several months. As may be expected, the overall standard deviation was higher than that given by using updated calibrations, but on certain occasions for several days, the constant calibration gave results that were quite adequate for routine purposes. This indicated that the samples analyzed that day as check samples conformed to the samples used in the original calibration.

TABLE III
ADA Calibration Printout With Typical Illustration of Redundant K_6 Value

K	K Value	SEE[a]	Wavelength Point	Calibration Band Sequence
0	11.761	...	...	...
1	−1204.6	0.3172	78	4
2	209.7	0.2601	14	1
3	587.2	0.2211	96	6
4	111.4	0.2088	51	3
5	386.1	0.1904	29	2
6	1409.4	0.1462	87	5

[a]SEE = standard error of estimate (calibration).

Factors Affecting Accuracy and Precision of ADA

Two main factors affect day-to-day performance of the ADA. These are grinding and the uniformity of the ground wheat presented for analysis, and changes in the location from which the samples originate.

Grinding. The Canadian Grain Commission routinely uses four types of grinders. These are the Hobart 2040 coffee grinder, which is now obsolete but is still used for grinding most wheat and barley for Kjeldahl testing; the Cyclotec grinder, which is used for sample preparation for all NIRS analysis; the Krups Model 75 macerator/grinder, which is used to pulverize fibrous crops such as oats for Kjeldahl testing; and the Buhler laboratory grinder, which is used for the routine grinding of cereals for the second stage of the AACC two-stage air-oven moisture test.

The ADA was calibrated with 50 samples of wheat ground on each of the four above-mentioned grinders. Each of the four series of ground samples were then analyzed on each of the four calibrations. The results are summarized in Table IV. Overall, the Cyclotec grinds provided the lowest standard deviations, with the Krups showing the greatest variability. The finer ground samples gave low ADA results when analyzed on calibrations based on coarser material, and vice versa. This subject is covered in detail elsewhere (6).

The Cyclotec grinder was selected as the most reliable grinder from the aspect of test results. Careful daily checks include visual inspection of grinders and ground material and biweekly analysis of particle size. This ensures that the five Cyclotec grinders in constant use with the ADA program are producing grinds of a sufficiently consistent mean particle size and particle size distribution to comply with the levels of accuracy and precision necessary for the segregation program.

TABLE IV
Influence of Grinding of HRS Wheat on Accuracy of Subsequent Analysis of Carlot Samples Using ADA

Calibration Samples	Ground On	Hobart 2040	Krups 75	Cyclotec 1.0 mm Screen	Buhler
Hobart-ground carlot	ADA protein[a]	13.69	13.75	12.98	13.77
	Kjeldahl protein	13.67	13.67	13.67	13.67
	SD[b]	0.232	0.524	0.384	0.361
Krups-ground carlot	ADA protein	13.80	13.74	13.62	14.87
	Kjeldahl protein	13.75	13.75	13.75	13.75
	SD	1.230	0.325	0.907	0.710
Cyclotec-ground carlot	ADA protein	14.10	13.77	13.77	14.99
	Kjeldahl protein	13.74	13.74	13.74	13.74
	SD	0.278	0.303	0.226	0.272
Buhler-ground carlot	ADA protein	13.74	13.99	12.70	13.50
	Kjeldahl protein	13.55	13.55	13.55	13.55
	SD	0.351	0.559	0.352	0.418

[a]13.5% Moisture basis.
[b]SD = standard deviation of the difference.

Growing environment or location. Growing environment and growing season have both been found to affect accuracy of NIRS analysis of wheat for protein (7). In the case of the ADA, daily calibration reduces the influence of growing season to a minimum. The samples used in the segregation program, however, arrive in Winnipeg by the hundreds every day and are compiled at country, or primary, elevators.

The shipping program is under the control of the Canadian Wheat Board, which keeps a running inventory of the volume of wheat in the three prairie provinces and estimates of grade and protein from the annual fall surveys of the new crop that the Canadian Grain Commission conducts. The provinces are divided into 49 shipping blocks, the boundaries of which follow main and spur railroad lines. Shipping from several blocks simultaneously is customary to maintain volume and freight car turnaround. As an area becomes depleted in the grade or protein level of wheat required, the shipping areas are changed by the wheat board. This results in changes in the nature of the wheat arriving in Winnipeg due to sometimes profound changes in the growing environment.

Figure 5 illultrates the soil zones of the wheat-growing area of Canada. The brown area has consistently provided the wheat with the highest protein content. On the other hand, wheat is grown in Alberta over a wide range of soil types, and this is reflected in the fact that the protein content of Alberta wheat shows more variation than that of the other provinces.

Based on the environmental study reported earlier (7), samples of HRS wheat were selected from the new crop survey material to represent 12 of the wheat board shipping blocks. These blocks were selected to cover the maximum range in variability in soil type, with replicates of the four chief soil zones: the brown earth, the dark brown earth, the brown-black transition earth, and the black earth zones. The ADA was calibrated with sets of 50 samples drawn from each zone, and 100 random carlot samples were analyzed on each calibration. Typical

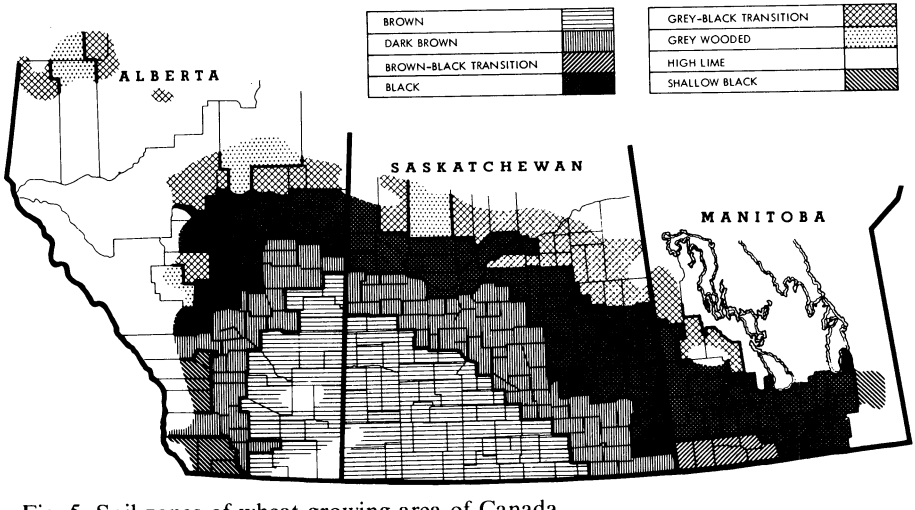

Fig. 5. Soil zones of wheat-growing area of Canada.

results are included in Table V. The origin of the samples used in calibration clearly could affect the day-to-day accuracy of analysis. On the other hand, a combination calibration made up of samples representative of all of the blocks gave a higher standard error of estimate during calibration but a more accurate analysis of the random carlots combined with a lower standard deviation of the difference. In practice, ensuring that the samples used in calibration are selected to give as wide a coverage as possible of the growing area resolves the point of origin effect.

RESULTS

The ADA is an operational system and not a research tool. It was purchased to streamline the testing aspect of the segregation program and to reduce the monumental volume of expensive Kjeldahl testing. In 1972, the commission ran well over 600,000 Kjeldahl tests, each of which was matched by a moisture test done with the model 919 meter (8). The results of the switch over to the ADA in 1975 can best be assessed in terms of its effects on the overall efficiency of the segregation program from the aspects of accuracy, precision, throughput, and maintenance and operational costs.

Accuracy

The simplest way to illustrate the accuracy of the ADA is by means of the cargo lot distribution study. During the loading of wheat onto ships, the shipping belts are sampled continuously by automatic Woodside samplers. Every 30 min, a subsample is withdrawn to check the grade and protein content of the loading. These samples may represent anywhere from 10 to 300 tonnes, and are referred to as cargo lot samples. The samples are returned to Winnipeg where they are tested for protein by the Kjeldahl process. This has been the practice ever since segregation commenced.

Since June 1975, all of the wheat binned in terminal elevators has been segregated on the basis of ADA testing. A study of the cargo lot protein distribution at comparable periods up to and since June 1975 will therefore indicate the efficacy of the ADA segregation as compared with the pre-June 1975, 100% Kjeldahl era. Table VI summarizes the quarterly cargo lot

TABLE V
Influence of Geographic Source of Origin of HRS Wheat Samples Used in Calibration on Accuracy of Subsequent Analysis of Random Carlot Samples by ADA

Calibration Samples From Area	Mean ADA Protein[a]	Mean Kjeldahl Protein[a]	RMSD[b]
13	13.08	13.16	0.324
77	12.54	13.16	0.445
84	12.58	13.16	0.458
Composite	13.14	13.16	0.268

[a]13.5% Moisture basis.
[b]RMSD = root mean square deviation between ADA and Kjeldahl data.

TABLE VI
Efficiency of Segregation of Wheat by Protein Content as Assessed by Cargo Lot Distribution (August Through October)

		Thunder Bay						Vancouver			
		1 CW[a]			2 CW[b]			1 CW[a]			
Year	Protein Level	Protein	SD[c]	N[d]	Protein	SD[c]	N[d]	Protein	SD[c]	N[d]	2 CW[b]
1973	12.5	12.80	0.25	1,177	12.77	0.23	624	12.75	0.24	313	No segregation
	13.5	13.65	0.21	2,226	13.59	0.21	531	13.65	0.23	748	Up to 1974 inclusive
1974	12.5	12.88	0.24	675	12.84	0.22	618	12.72	0.18	36	
	13.5	13.71	0.21	560	13.63	0.20	318	13.63	0.24	364	
1975	12.5	12.88	0.30	390	12.83	0.27	720	12.79	0.27	200	
	13.5	13.74	0.24	673	13.75	0.23	643	13.68	0.28	364	
1976	12.5	12.64	0.26	931	12.81	0.27	943	12.75	0.29	186	
	13.5	13.63	0.27	1,307	13.70	0.25	750	13.64	0.28	251	

[a]1 CW = grade No. 1 Canada Western.
[b]2 CW = grade No. 2 Canada Western.
[c]SD = standard deviation.
[d]N = number of cargo lots.

distribution for the August to October periods of 1973 through 1976. This includes two years of segregation before and after ADA. Clearly, introduction of the automated NIRS testing has not materially affected efficiency of the segregation program. Daily spot comparisons between ADA and Kjeldahl have an overall root mean square deviation of ±0.25%. This does not conform to the original specifications of 0.15%, but is satisfactory for the operation of the segregation program.

The ADA was purchased primarily for analysis of wheat. It has been used, however, for analysis of certain other commodities for purposes of applied research. These include rapeseed, durum wheat, durum semolina, barley, and chickpeas. The results of testing these commodities were practically identical with those obtained with the GQA Models 31 and 41, but the throughput was significantly higher. For screening purposes in breeding programs, the ADA affords the opportunity of screening thousands of samples in a short time. A set of 574 durum wheats and more than 300 bread wheats of several types (hard and soft) were screened for protein in one afternoon using a durum calibration and an all-purpose calibration, both of which formed part of the afternoon's work. The calibrations were completed at the end of the afternoon, and all of the results were calculated using LDA.

Precision

The precision of all of the protein testing that the Canadian Grain Commission has done is assessed by testing secret check samples of the respective grains. In the case of HRS wheat, a bulk sample of 1 tonne of wheat is procured, thoroughly blended by a large-scale mechanical end-over-end blender, divided into 25-kg sublots, and placed in cans in cold storage. Each individual can is sampled and tested in quadruplicate for Kjeldahl protein and oven moisture. "Outlying" cans are reblended until the entire series of cans has a standard deviation of ±0.10 to 0.12% protein (13.5% moisture basis).

The sample is designated as the annual protein check (PC) and is further identified by year, *e.g.*, the current sample is PC 77. Subsamples of the PC are disguised as normal samples and interpolated into the system. This checks the entire system, including sampling, moisture testing, sample preparation, protein testing, and documentation. The results are compiled as a report on the protein-testing operation on a weekly, monthly, and annual basis. Table VII depicts a typical report on the test procedures. Daily checks consist of grinding and blending a 0.5-kg sample to give a bulk ground sample of wheat. This minimizes sampling and sample preparation error. The standard error per test is generally lower than that of the secret checks. The purpose of the daily check is to permit the laboratory supervisor to detect progressive or sudden changes in performance.

In general, the ADA is slightly less precise than the Winnipeg Kjeldahl laboratory. By effecting certain improvements in the cell-stopping mechanism, replacing the original Teflon standard with Coor's ceramic, and continuously updating the calibration, the precision of the ADA secret checks has been improved from an average of about 0.29 in 1974 to 0.21 in 1976. In certain instances, the ADA is more consistent than is the Kjeldahl process in that the ADA secret check precision is quite stable (about 0.19–0.23), whereas the Kjeldahl laboratory is occasionally subject to a day or two of high standard error.

The overall precision of the ADA is entirely compatible with the segregation program.

Throughput

Throughput was tested by subjecting the system to continuous workload for 7 hr per day over a five-day period. A total of 12,300 tests were processed in the five-day experiment. Five Cyclotec grinders were each used to prepare more than 500 samples per day, a crew of six was occupied in loading and cleaning trays, and two more people operated the ADA and documented the results so that a total of 13 people were able to test the volume stated for protein and moisture in five days. To process the same volume in Winnipeg by Kjeldahl and electric moisture meter 13 days and a total staff of 13 would be required. In other words, the ADA required 65 man-days to complete work that would require 169 man-days by standard methods. In addition, the ADA uses no chemicals and a negligible amount of power, whereas the Kjeldahl laboratory would use more than 0.5 ton of chemicals, more than 100,000 gal of water, and 10,500 kw hr of electricity to accomplish the same workload.

The actual time for testing a tray of ten samples is about 70 sec, which is slightly

TABLE VII
Typical Report on Test Procedures

	Daily Check			Secret Check			
Date	Mean	SD[a]	N[b]	Mean	SD[a]	N[b]	Volume
				Kjeldahl			
May 16	12.89	0.051	30	12.73	0.098	20	672
May 17	12.85	0.065	19	12.72	0.080	10	648
May 18	12.86	0.061	34	12.76	0.169	10	432
May 19	12.89	0.046	15	12.70	0.078	20	648
May 20	12.88	0.031	20	12.79	0.155	20	528
Week	12.88	0.055	118	12.74	0.122	80	2,928
Month	12.88	0.060	320	12.73	0.134	210	10,104
Year	12.90	0.056	1677	12.82	0.154	899	57,312
				ADA			
May 16	13.02	0.278	79	12.89	0.218	10	2,060
May 17	12.97	0.212	76	13.07	0.216	10	2,000
May 18	12.87	0.268	81	12.94	0.158	10	2,230
May 19	12.95	0.278	66	12.97	0.183	10	1,900
May 20	12.84	0.311	55	12.60	0.298	10	1,650
Week	12.94	0.272	357	12.89	0.264	50	9,840
Month	12.95	0.230	885	12.94	0.253	151	25,430
Year	12.91	0.214	3509	12.93	0.219	878	102,650

[a]SD = standard deviation.
[b]N = number of tests.

less than the required ten tests per minute. Accessing data from time to time, however, is nececcary for identification. Tape changeovers occur every 500 samples, which reduces the work throughput. Nevertheless, a daily workload of 2,500 samples can be achieved consistently under normal labor rules. Occasionally, overtime shifts have to be run to reduce massive workloads that accumulate over holiday weekends in the summer months. On one such occasion, more than 4,000 samples were processed in a 12-hr period.

Maintenance and Operational Costs

Since its installation during 1974, the ADA has processed well over 700,000 samples, or nearly 1,500,000 individual tests. Over the three-year period, it has been remarkably free of downtime beyond replacing a light source about every 2,000 hr. Most of the problems that were encountered concern periodic variability in precision from batch to batch and from tray to tray. Reloading the program usually resolved these problems. The cost per test for 1976 amounted to $.74 for 296,000 samples, including labor, rent, auxiliary supplies, power, and maintenance. The cost of Kjeldahl testing in the Winnipeg laboratory for the same period was $1.26 per test.

Future Improvements

Future improvements projected for the ADA involve mainly data processing. The number of points scanned is to be extended to 600. Recent work at the USDA, Beltsville, MD, has verified that the use of a reference wavelength by which all DR/R readings are divided significantly improves the precision of the results so that this option will be added to the programming. The readout will probably be modified to include a variance inflation factor, which will simplify the decision to eliminate K values. A 600-point dump feature will enable the operator to study the absorbances over the area that the system scans. The number of filters will probably be increased to six. This will increase the range scanned, which will improve the accuracy of testing. Finally, a small storage display oscilloscope may be added, which will enable the operator to study the DR/R trace visually as well as via the printout. These modifications will update the ADA almost to the status of a spectrocomputer.

Use of reference wavelengths has been shown to reduce significantly sensitivity to variations in particle size and moisture content, two of the main sources of error in NIRS testing for protein. The reference wavelengths would be incorporated in such a manner that only three wavelengths will be used as test wavelengths, but each will work in association with its own reference wavelength.

SUMMARY

The ADA is a contracted system built for testing large numbers of samples of ground grain for protein and moisture by NIRS. It has the capacity to process more than 2,500 samples in a normal 7-hr working shift, with an accuracy and precision adequate for the sophisticated program for segregating wheat into protein levels for marketing. It uses no chemicals and minimal amounts of power.

Semiskilled employees are necessary for routine daily calibration and operation of the equipment. This has resulted in a dramatic reduction in the cost

of operation of the Canadian Grain Commission segregation program without causing any marked reduction in efficiency of the system.

The forte of the ADA lies in automation of large-scale testing of cereals and pulses. Segregation systems and breeding programs such as those at grain processing companies and major international agricultural research centers could significantly improve the efficiency of their operations by means of instrumentation of this type.

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