

# **Near-Infrared Spectroscopy in Agriculture**

# Near-Infrared Spectroscopy in Agriculture

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# CONTENTS

Dedication . . . . .	ix
Foreword . . . . .	xi
Preface . . . . .	xiii
Contributors . . . . .	xv
Conversion Factors for SI and Non-SI Units . . . . .	xix

## SECTION A—NEAR-INFRARED SPECTROSCOPY

<b>1 Understanding and Using the Near-Infrared Spectrum as an Analytical Method . . . . .</b>	<b>3</b>
Jerry Workman, Jr. and John Shenk	
<b>2 Near-Infrared Spectrophotometers . . . . .</b>	<b>11</b>
Jerry Workman, Jr.	
<b>3 Testing and Validating Instrument Performance . . . . .</b>	<b>33</b>
W.L. Cap Munday, Charles M. Zapf, and James B. Reeves III	
<b>4 Network Design and Implementation . . . . .</b>	<b>49</b>
Phil Williams and John Antoniszyn	
<b>5 Sample Preparation . . . . .</b>	<b>75</b>
Ian Murray and Ian Cowe	

## SECTION B—CHEMOMETRICS FOR NEAR-INFRARED SPECTROSCOPY

<b>6 Mathematical Data Preprocessing . . . . .</b>	<b>115</b>
James Duckworth	
<b>7 Quantitative Analysis . . . . .</b>	<b>133</b>
Mark Westerhaus, Jerry Workman, Jr., James B. Reeves III, and Howard Mark	
<b>8 Qualitative Analysis . . . . .</b>	<b>175</b>
Richard Kramer, Jerry Workman, Jr., and James B. Reeves III	
<b>9 Standardization and Calibration Transfer . . . . .</b>	<b>207</b>
John Shenk	

**SECTION C—NIR APPLICATIONS IN ANALYSIS OF FOOD CROPS**

- 10 Analysis of Forages and Feedstuffs . . . . .** 231  
Craig A. Roberts, Jerry Stuth, and Peter Flinn
- 11 Analysis of Small Grain Crops . . . . .** 269  
Stephen R. Delwiche
- 12 Analysis of Oilseeds and Coarse Grains . . . . .** 321  
Daniel J. Dyer
- 13 Analysis of Coffee, Tea, Cocoa, Tobacco, Spices, Medicinal  
and Aromatic Plants, and Related Products . . . . .** 345  
Hartwig Schulz
- 14 Analysis of Fruits and Vegetables . . . . .** 377  
David C. Slaughter and Judy A. Abbott
- 15 Analysis of Sugarcane . . . . .** 399  
Reiji Sekiguchi, Masami Ueno, and Sumio Kawano

**SECTION D—NIR APPLICATIONS IN ANALYSIS  
OF PROCESSED FOODS**

- 16 Analysis of Cereal Food Products . . . . .** 411  
Sandra E. Kays
- 17 Analysis of Baking Products . . . . .** 439  
Christopher N.G. Scotter and Samuel J. Millar
- 18 Analysis of Beverages and Brewing Products . . . . .** 465  
Bob Dambergs, Michael Esler, and Mark Gishen
- 19 Analysis of Fats and Oils . . . . .** 487  
Ana Garrido-Varo, Juan García-Olmo, and  
Maria Dolores Pérez-Marín
- 20 Analysis of Dairy and Eggs . . . . .** 559  
Roberto Giangiacomo and Tiziana M.P. Cattaneo
- 21 Analysis of Meats . . . . .** 599  
Gerard Downey and Kjell Ivar Hildrum

**SECTION E—NIR APPLICATIONS IN ANALYSIS  
OF NON-FOOD AGRICULTURAL PRODUCTS**

<b>22</b>	<b>Analysis of Timber and Paper</b> . . . . .	635
	Laurence Schimleck and Jerry Workman, Jr.	
<b>23</b>	<b>Analysis of Animal By-Products</b> . . . . .	647
	Daniel Cozzolino and Ian Murray	
<b>24</b>	<b>Analysis of Wool</b> . . . . .	663
	Michael Hammersley and Trisha Townsend	
<b>25</b>	<b>Analysis of Cotton</b> . . . . .	671
	Joseph G. Montalvo, Jr. and Terri Von Hoven	
<b>26</b>	<b>Analysis of Soils</b> . . . . .	729
	Diane Malley, Paul Martin, and Eyal Ben-Dor	
	Subject Index. . . . .	785

## DEDICATION

*Near-Infrared Spectroscopy in Agriculture* is dedicated to Phil Williams (right) and Karl Norris (left). Collectively, they made this technology usable, especially in agricultural applications. Individually, each has his own list of contributions and achievements.



Karl Norris was the first to demonstrate that NIR spectral data could be measured on samples such as ground grains. His real contribution was the incorporation of computers to interpret data that could predict composition. This contribution, while it appears intuitive in our time, was innovative 40 years ago, long before desktop computers existed. The coupling of computers with spectrophotometers for statistical interpretation of spectra has facilitated the subdiscipline now known as chemometrics. In addition to working with small grains, Norris worked with other agricultural products. He collaborated with coworkers at the Instrumentation Research Laboratory at the USDA, Beltsville, MD to predict moisture in soybean and fat content in milk. This work involved both design of instruments operating in the NIR region and development of software to process the data. Because of this work, as well as the applied collaboration with Williams, many people consider Karl Norris the unofficial father of modern NIR spectroscopy.

Phil Williams was the applied equivalent of Norris. He was first ever to apply NIR technology to large-scale, real-world testing of a commercial commodity. In his search for a method rapid enough to test railway carloads of wheat at the time of unloading, Williams acquired one of the first NIR instruments ever built. His commitment to analytical precision, supported by the engineers of what was then the Neotec Corporation, resulted in replacement of the traditional Kjeldahl method by an automated NIR system for the protein segregation program. Within only four years, all testing—about 600,000 samples per year—was carried out at terminal elevators. Williams' subsequent work has been aimed at resolving problems associated with application of NIR technology to grain handling, with particular emphasis on electronic grading. In the field of plant breeding, Williams has concentrated on development of calibration models for the prediction of functionality, as well as composition. Over the past 32 years his research on evaluation of new instruments has been of significant benefit to several instrument manufacturing companies.

Though Williams and Norris have both retired from their original positions, they remain active in the field of NIR spectroscopy. They continue to help jumpstart new analytical laboratories, mentor young spectroscopists, and field questions from anyone searching for an answer. As a result of their substantial contributions, they have both received prestigious awards. Today, Williams and Norris are household names, often mentioned together because of their synergistic effect in spawning, then documenting, a growing technology.



## FOREWORD

Near-infrared (NIR) spectroscopy is a remarkably versatile and robust analytical methodology. Its nondestructive nature, fast analysis time, and relative ease of use has led to the development of many applications in a broad array of agricultural fields. The evolution and widespread application of NIR spectroscopy in the past several decades is one of the great success stories in analytical technology development. From the humble beginnings of fixed-filter instruments and simple calibrations to the scanning monochromators and advanced chemometrics of today, the technology has undergone an astonishing transformation. Key to this achievement was the rapid development of the microprocessor and the advanced analytical software it made possible. Today, NIR spectroscopy is widely used to detect and quantify an almost unending list of analytes in a host of agricultural and food products.

This volume provides monographic coverage of the use of NIR spectroscopy in agriculture. It begins with a section on fundamental principles of NIR spectroscopy, including chapters on instrumentation and sample preparation. This is followed by a comprehensive section on advanced chemometrics for qualitative and quantitative NIR analysis. The remaining three sections describe NIR applications for analysis of food crops, processed foods, and other agricultural products and by-products. The chapters are authored by a who's who list of the leading experts in the design, calibration, and application of NIR spectroscopy. The book is truly international in scope with contributing authors from around the world.

Our thanks to the feasibility committee, editors, authors, reviewers, and staff that worked diligently to make this outstanding volume available. They have created an exceptional reference that will serve as a leading source and definitive authority on NIR spectroscopy in agriculture for many years to come.

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## PREFACE

Near-infrared (NIR) spectroscopy is fast becoming a preferred method of routine analysis, especially in agriculture. Its growing popularity is due to its accuracy and efficiency of process. As an empirical procedure, NIR spectroscopy differs from standard spectroscopy; it does not require a full understanding of the physical relationships between spectral data and chemical functional groups. Instead, NIR spectroscopy is based on mathematical relationships between spectra and reference data. Once a mathematical relationship is established, the NIR spectrophotometer collects spectra and predicts analysis, thereby expediting rapid, large-scale processing of samples. The repeatability and accuracy of predicted data are monitored through an array of blind validation protocols.

Because of its empirical approach and indirect analysis, NIR spectroscopy sometimes attracts criticism. In many ways, it is a technology comparable to cryptanalysis during World War II—it “had an aura of sorcery, but the basis was highly scientific.”<sup>1</sup> Criticism of NIR spectroscopy as a bench-top analytical tool is most common among theoretical researchers, who strain to understand the same principles they readily accept in remote sensing. Among practitioners, however, criticism is rare. In fact, practitioners have come to appreciate the inherent advantages of NIR spectroscopy, such as nondestructive sampling, reduced dependence on chemical reagents, and simultaneous quantification of constituents.

*Near-Infrared Spectroscopy in Agriculture* highlights the practical use of NIR technology during its first forty years in international agriculture. The book was requested by a feasibility committee of the American Society of Agronomy, who commissioned the work and cosponsored it with the Crop Science Society of America and the Soil Science Society of America.

*Near-Infrared Spectroscopy in Agriculture* is organized into five sections. The first two sections present the fundamentals of spectroscopy and chemometrics. These two sections were simplified as much as possible at the request of the editors because this book targets a broad scientific audience rather than a narrow group of spectroscopists and statisticians. The last three sections are applied and give this book a clear niche among other NIR publications. These applied sections detail the use of NIR analysis in crop production, food processing, and non-food agriculture. Chapters in the applied sections are generally comprehensive, and they include standardized tables of applications for ease of reference.

The editors would like to express their appreciation to all authors, reviewers, and editors of these chapters, as well as the editors of ASA, CSSA, and SSSA for their commitment and hard work in producing this book. We thank the Managing Editor, Lisa Al-Amoodi. We would also like to thank certain colleagues who helped us in the early stages of this work. Specifically, we are grateful to Ian Murray and Chris Scotter, who helped us identify international authors with various specializations in NIR application. We owe a special word of gratitude to authors Roberto Giangiacomo, Tiziana Cattaneo, Stephen Delwiche, Trish Townsend, and Michael Hammersley; these authors submitted their chapters early in the process and pro-

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<sup>1</sup> Stevenson, W. 1976. *A man called intrepid*. Harcourt, Brace, Jovanovich, New York.

vided their colleagues with excellent examples of content and composition. Finally, we are grateful to Jerry Nelson, who offered a steady stream of reliable advice through the entire publication process.

We hope this work will benefit all those interested in efficient analysis of agricultural products.

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## **Conversion Factors for SI and non-SI Units**

# Conversion Factors for SI and non-SI Units

To convert Column 1 into Column 2, multiply by	Column 1 SI Unit	Column 2 non-SI Units	To convert Column 2 into Column 1, multiply by
	<b>Length</b>		
0.621	Kilometer, km ( $10^3$ m)	mile, mi	1.609
1.094	meter, m	yard, yd	0.914
3.28	meter, m	foot, ft	0.304
1.0	micrometer, $\mu\text{m}$ ( $10^{-6}$ m)	micron, $\mu$	1.0
$3.94 \times 10^{-2}$	millimeter, mm ( $10^{-3}$ m)	inch, in	25.4
10	nanometer, nm ( $10^{-9}$ m)	Angstrom, $\text{\AA}$	0.1
	<b>Area</b>		
2.47	hectare, ha	acre	0.405
247	square kilometer, $\text{km}^2$ ( $10^3$ m) <sup>2</sup>	acre	$4.05 \times 10^{-3}$
0.386	square kilometer, $\text{km}^2$ ( $10^3$ m) <sup>2</sup>	square mile, mi <sup>2</sup>	2.590
$2.47 \times 10^{-4}$	square meter, m <sup>2</sup>	acre	$4.05 \times 10^3$
10.76	square meter, m <sup>2</sup>	square foot, ft <sup>2</sup>	$9.29 \times 10^{-2}$
$1.55 \times 10^{-3}$	square millimeter, $\text{mm}^2$ ( $10^{-3}$ m) <sup>2</sup>	square inch, in <sup>2</sup>	645
	<b>Volume</b>		
$9.73 \times 10^{-3}$	cubic meter, m <sup>3</sup>	acre-inch	102.8
35.3	cubic meter, m <sup>3</sup>	cubic foot, ft <sup>3</sup>	$2.83 \times 10^{-2}$
$6.10 \times 10^4$	cubic meter, m <sup>3</sup>	cubic inch, in <sup>3</sup>	$1.64 \times 10^{-5}$
$2.84 \times 10^{-2}$	liter, L ( $10^{-3}$ m <sup>3</sup> )	bushel, bu	35.24
1.057	liter, L ( $10^{-3}$ m <sup>3</sup> )	quart (liquid), qt	0.946
$3.53 \times 10^{-2}$	liter, L ( $10^{-3}$ m <sup>3</sup> )	cubic foot, ft <sup>3</sup>	28.3
0.265	liter, L ( $10^{-3}$ m <sup>3</sup> )	gallon	3.78
33.78	liter, L ( $10^{-3}$ m <sup>3</sup> )	ounce (fluid), oz	$2.96 \times 10^{-2}$
2.11	liter, L ( $10^{-3}$ m <sup>3</sup> )	pint (fluid), pt	0.473

<b>Mass</b>			
gram, g ( $10^{-3}$ kg)	pound, lb	454	
gram, g ( $10^{-3}$ kg)	ounce (avdp), oz	28.4	
kilogram, kg	pound, lb	0.454	
kilogram, kg	quintal (metric), q	100	
kilogram, kg	ton (2000 lb), ton	907	
megagram, Mg (tonne)	ton (U.S.), ton	0.907	
tonne, t	ton (U.S.), ton	0.907	
<b>Yield and Rate</b>			
0.893	kilogram per hectare, kg ha <sup>-1</sup>	1.12	
$7.77 \times 10^{-2}$	kilogram per cubic meter, kg m <sup>-3</sup>	12.87	
$1.49 \times 10^{-2}$	kilogram per hectare, kg ha <sup>-1</sup>	67.19	
$1.59 \times 10^{-2}$	kilogram per hectare, kg ha <sup>-1</sup>	62.71	
$1.86 \times 10^{-2}$	kilogram per hectare, kg ha <sup>-1</sup>	53.75	
0.107	liter per hectare, L ha <sup>-1</sup>	9.35	
893	tonne per hectare, t ha <sup>-1</sup>	$1.12 \times 10^{-3}$	
893	megagram per hectare, Mg ha <sup>-1</sup>	$1.12 \times 10^{-3}$	
0.446	megagram per hectare, Mg ha <sup>-1</sup>	2.24	
2.24	meter per second, m s <sup>-1</sup>	0.447	
<b>Specific Surface</b>			
10	square meter per kilogram, m <sup>2</sup> kg <sup>-1</sup>	0.1	
1000	square meter per kilogram, m <sup>2</sup> kg <sup>-1</sup>	0.001	
<b>Density</b>			
1.00	megagram per cubic meter, Mg m <sup>-3</sup>	1.00	
<b>Pressure</b>			
9.90	megapascal, MPa ( $10^6$ Pa)	0.101	
10	megapascal, MPa ( $10^6$ Pa)	0.1	
$2.09 \times 10^{-2}$	pascal, Pa	47.9	
$1.45 \times 10^{-4}$	pascal, Pa	$6.90 \times 10^3$	

(continued on next page)

# Conversion Factors for SI and non-SI Units

To convert Column 1 into Column 2, multiply by	Column 1 SI Unit	Column 2 non-SI Units	To convert Column 2 into Column 1, multiply by
		<b>Temperature</b>	
1.00 (K - 273) (9/5 °C) + 32	kelvin, K Celsius, °C	Celsius, °C Fahrenheit, °F	1.00 (°C + 273) 5/9 (°F - 32)
9.52 × 10 <sup>-4</sup> 0.239 10 <sup>7</sup> 0.735 2.387 × 10 <sup>-5</sup> 10 <sup>5</sup> 1.43 × 10 <sup>-3</sup>	joule, J joule, J joule, J joule, J joule per square meter, J m <sup>-2</sup> newton, N watt per square meter, W m <sup>-2</sup>	British thermal unit, Btu calorie, cal erg foot-pound calorie per square centimeter (langley) dyne calorie per square centimeter minute (irradiance), cal cm <sup>-2</sup> min <sup>-1</sup>	1.05 × 10 <sup>3</sup> 4.19 10 <sup>-7</sup> 1.36 4.19 × 10 <sup>4</sup> 10 <sup>-5</sup> 698
		<b>Energy, Work, Quantity of Heat</b>	
		<b>Transpiration and Photosynthesis</b>	
3.60 × 10 <sup>-2</sup>	milligram per square meter second, mg m <sup>-2</sup> s <sup>-1</sup>	gram per square decimeter hour, g dm <sup>-2</sup> h <sup>-1</sup>	27.8
5.56 × 10 <sup>-3</sup>	milligram (H <sub>2</sub> O) per square meter second, mg m <sup>-2</sup> s <sup>-1</sup>	micromole (H <sub>2</sub> O) per square centi- meter second, μmol cm <sup>-2</sup> s <sup>-1</sup>	180
10 <sup>-4</sup>	milligram per square meter second, mg m <sup>-2</sup> s <sup>-1</sup>	milligram per square centimeter second, mg cm <sup>-2</sup> s <sup>-1</sup>	10 <sup>4</sup>
35.97	milligram per square meter second, mg m <sup>-2</sup> s <sup>-1</sup>	milligram per square decimeter hour, mg dm <sup>-2</sup> h <sup>-1</sup>	2.78 × 10 <sup>-2</sup>
		<b>Plane Angle</b>	
57.3	radian, rad	degrees (angle), °	1.75 × 10 <sup>-2</sup>

**Electrical Conductivity, Electricity, and Magnetism**

10	siemen per meter, S m <sup>-1</sup>	millimho per centimeter, mmho cm <sup>-1</sup>	0.1
10 <sup>4</sup>	tesla, T	gauss, G	10 <sup>-4</sup>

**Water Measurement**

9.73 × 10 <sup>-3</sup>	cubic meter, m <sup>3</sup>	acre-inch, acre-in	102.8
9.81 × 10 <sup>-3</sup>	cubic meter per hour, m <sup>3</sup> h <sup>-1</sup>	cubic foot per second, ft <sup>3</sup> s <sup>-1</sup>	101.9
4.40	cubic meter per hour, m <sup>3</sup> h <sup>-1</sup>	U.S. gallon per minute, gal min <sup>-1</sup>	0.227
8.11	hectare meter, ha m	acre-foot, acre-ft	0.123
97.28	hectare meter, ha m	acre-inch, acre-in	1.03 × 10 <sup>-2</sup>
8.1 × 10 <sup>-2</sup>	hectare centimeter, ha cm	acre-foot, acre-ft	12.33

**Concentrations**

1	centimole per kilogram, cmol kg <sup>-1</sup>	milliequivalent per 100 grams, meq 100 g <sup>-1</sup>	1
0.1	gram per kilogram, g kg <sup>-1</sup>	percent, %	10
1	milligram per kilogram, mg kg <sup>-1</sup>	parts per million, ppm	1

**Radioactivity**

2.7 × 10 <sup>-11</sup>	becquerel, Bq	curie, Ci	3.7 × 10 <sup>10</sup>
2.7 × 10 <sup>-2</sup>	becquerel per kilogram, Bq kg <sup>-1</sup>	picocurie per gram, pCi g <sup>-1</sup>	37
100	gray, Gy (absorbed dose)	rad, rd	0.01
100	sievert, Sv (equivalent dose)	rem (roentgen equivalent man)	0.01

**Plant Nutrient Conversion**

<i>Elemental</i>		<i>Oxide</i>	
2.29	P	P <sub>2</sub> O <sub>5</sub>	0.437
1.20	K	K <sub>2</sub> O	0.830
1.39	Ca	CaO	0.715
1.66	Mg	MgO	0.602