Innovative Use of Portable Time-Domain Nuclear Magnetic Resonance Spectroscopy for Accurate In-Field Quality Analysis of Citrus Raw Juice.

Massimiliano La Colla, Marcus Semones, Wendy Coco and Nelson K Stacks
WaveGuide Corporation, Cambridge, MA, USA

Presented by Javier Aztiazarain, Ph.D. at PANIC May 19-22nd, 2025 Rockville, MD

Abstract

Harnessing the advanced capabilities of time-domain NMR (TD-NMR) spectroscopy, a portable TD-NMR device offers a precise, in-field solution for assessing citrus raw juice quality. This innovative technology has the potential to impact the profiling of some of the 1.7 million tons of global orange juice that is produced each season. Taking TD-NMR out from the confines of traditional lab environments into the open fields can provide the greatest value and maximize utility for growers and consumers. Compact and cost-effective, the portable TD-NMR device delivers the precision of conventional systems while requiring only microliters (µL) of sample. It enables accurate results within minutes—even by operators with minimal training. Early profiling of various raw fruit juices, along with analysis using a Degrees Brix (°Bx) model system, supports the use of this tool for in-field measurement of both °Bx and Titratable Acidity (TA). The device achieved accuracy up to two decimal places.

The °Bx model system, widely used by food technologists to determine sugar content in aqueous solutions, was effectively correlated with TD-NMR standards. Current measurement methods often fall short of meeting the demands of large-scale production. This portable approach enables rapid, reliable quality analysis in the field—saving time, reducing costs, and helping determine the optimal harvest window.

Objective

To demonstrate the practical application and value of portable TD-NMR spectroscopy in measuring sugar content by comparing its performance to a digital refractometer using the BRIX method. This study aims to highlight the accuracy, reliability, and advantages of TD-NMR in providing precise, in-field measurements of sugar content in citrus raw juice.

Materials & Methods

Waveguide Formµla TD-NMR

- WGC-100-1 Package
  - 3MM OD NMR Custom tubes (PN # WGC-100)
  - 3MM NMR /EPR NMR Tube caps (PN # WGC-50)
  - Cartridge (PN #WGC-10)

Milwaukee MA871 Digital BRIX/Sugar Refractometer

- Steamed distilled water for calibration (Naslow Chemical LTD)
- Water for cleaning

BRIX Calibration Solution 28% (VWR)

Sucrose S7903 (Sigma-Aldrich) & Fresh Fruit Juice

Measurement & Analysis

TD-NMR:

- NMR T2 measurement using a CPMG pulse sequence
- Single-component exponential fit
  - Outcome: A single time-constant (T2)
- Two-component bi-exponential fit
  - Outcome: Two time-constants, T2a and T2b

Refractometer:

- Brix value

Conclusions

- TD-NMR is comparable to the BRIX method in measuring sugar content in fruits and solutions, offering accurate and reliable results.
- TD-NMR provides additional insights into sample heterogeneity, allowing for a more comprehensive understanding of the sample's composition compared to traditional methods.

Attribute	Digital Sucrose Refractometer	TD-NMR
Size	√	√
Portability	√	√
Ease of Use	√	√
Sample Size	µL	15µL
Sample Process	√	√
Training	Easy to use	Easy to use
Data	BRIX value	Single exponential T2 Bi-Exponential T2a & T2b
Data Storage	Transcription to notebook after each sample	Data saved on device and sent to cloud for download
Other	Requires calibration standard run before each sample Requires cleaning after each sample	Requires 1 instrument water standard test before each day measurements Comes with carry case that fits all supplies

Results

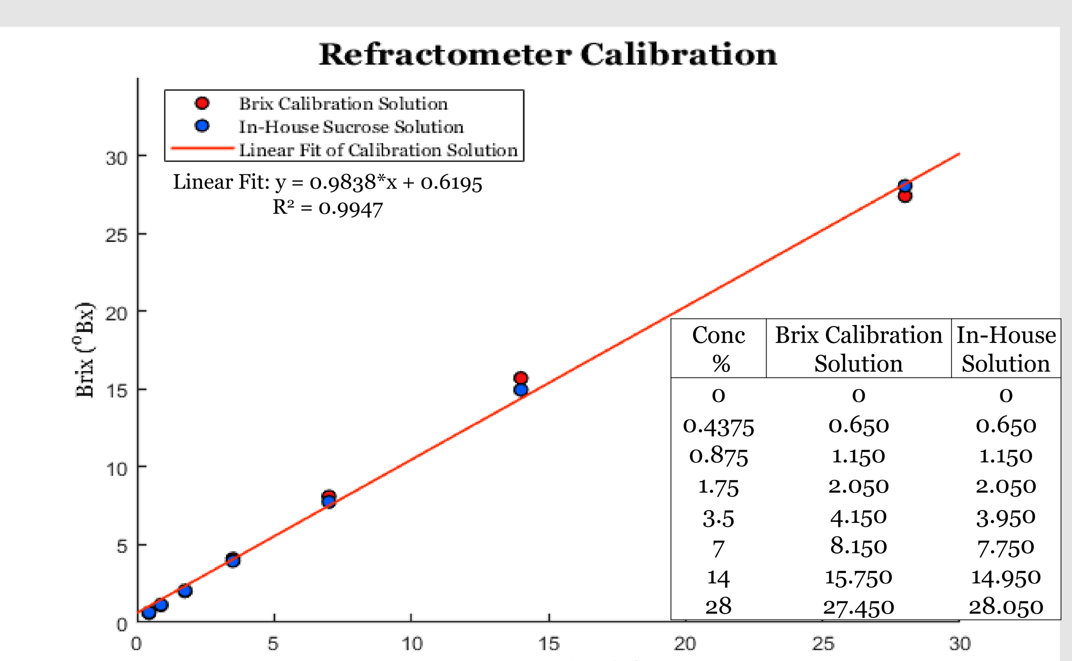

Figure1.
Digital refractometer sugar measurements using a BRIX 28% standard calibration solution and in-house prepared sucrose solution dilution ladders (weight/weight).

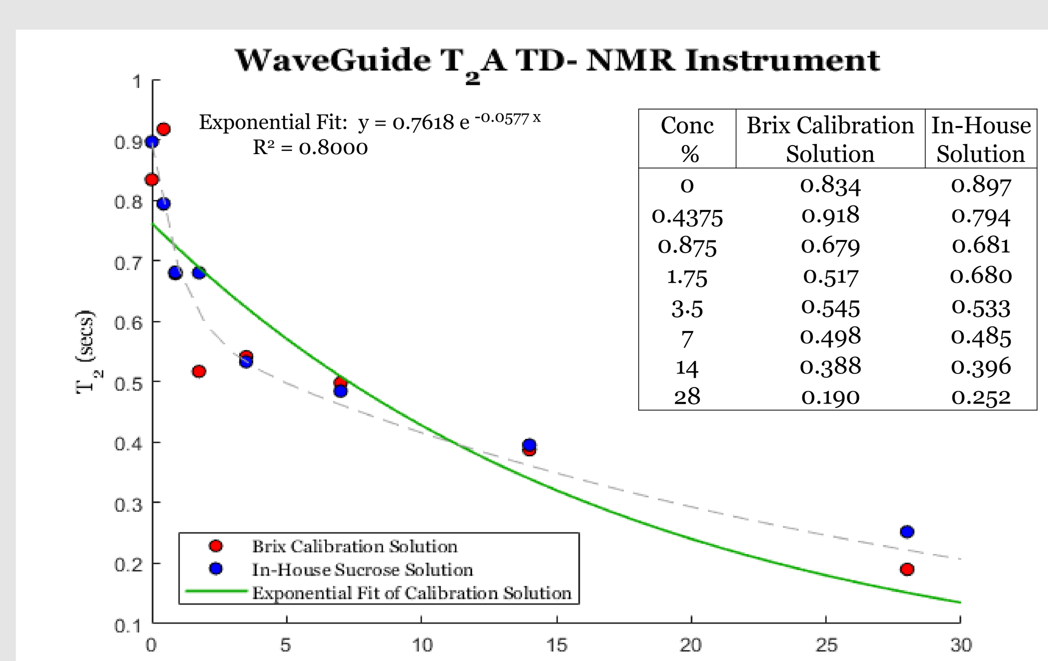

Figure3.
TD-NMR Bi-Exponential Model T2a

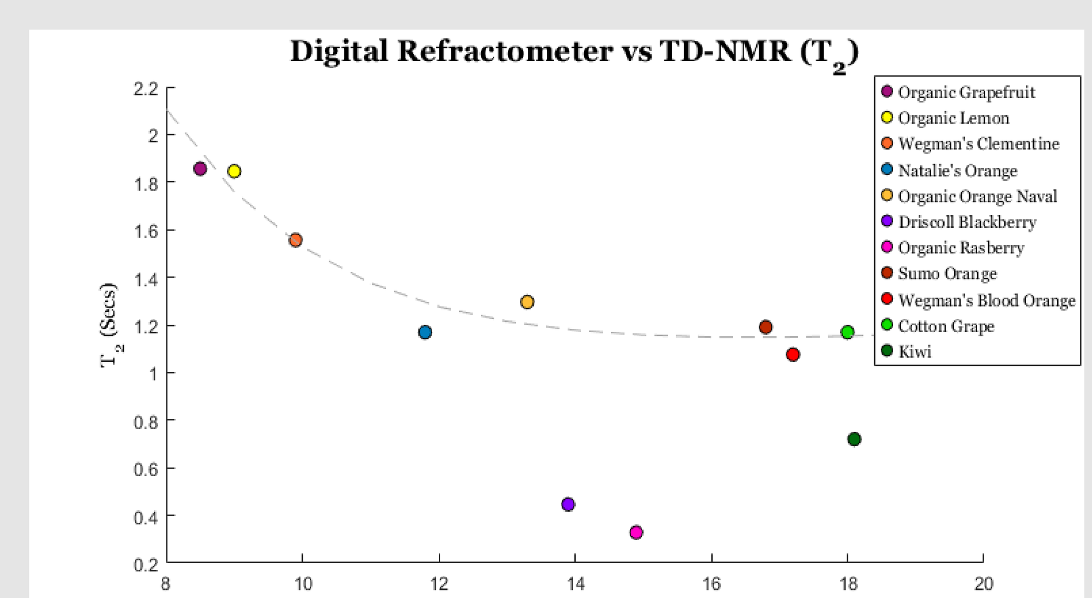

Figure 5.
TD-NMR (T2) and Digital Refractometer field testing of raw juice

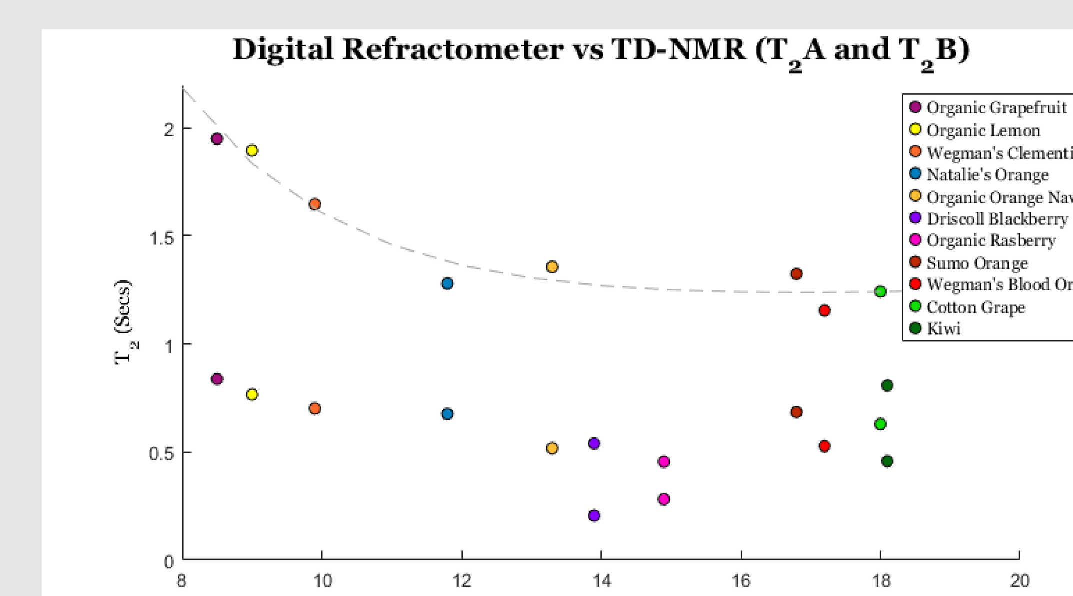

Figure 6.
TD-NMR (T2a and T2b) and Digital Refractometer field testing of raw juice

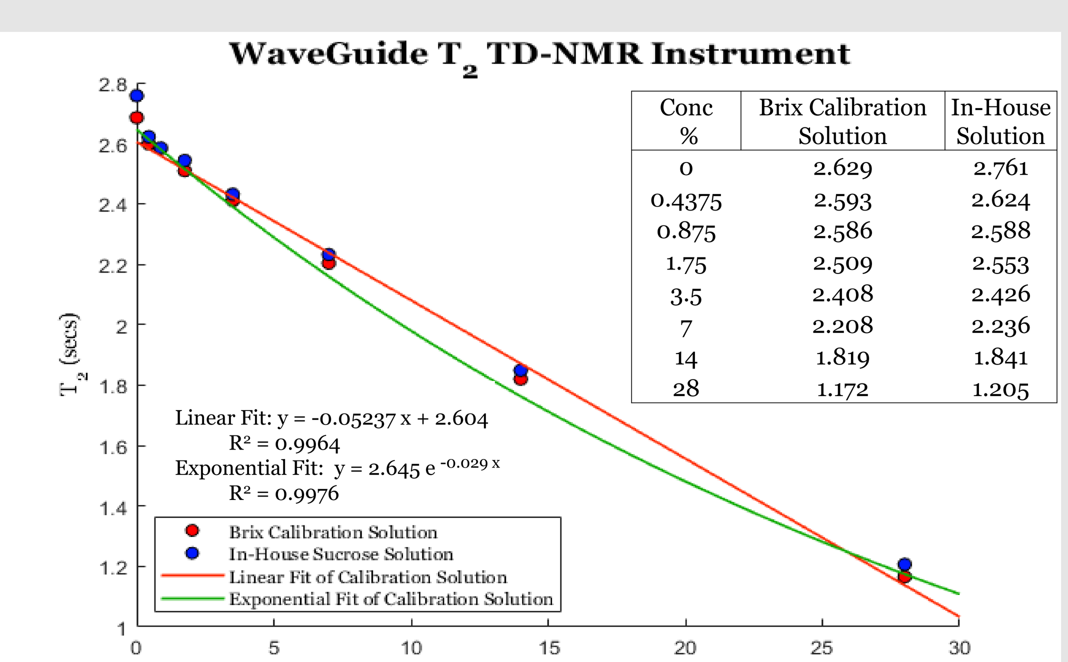

Figure 2.
TD-NMR sugar measurements using a BRIX 28% standard calibration solution and in-house prepared sucrose solution dilution ladders (weight/weight).

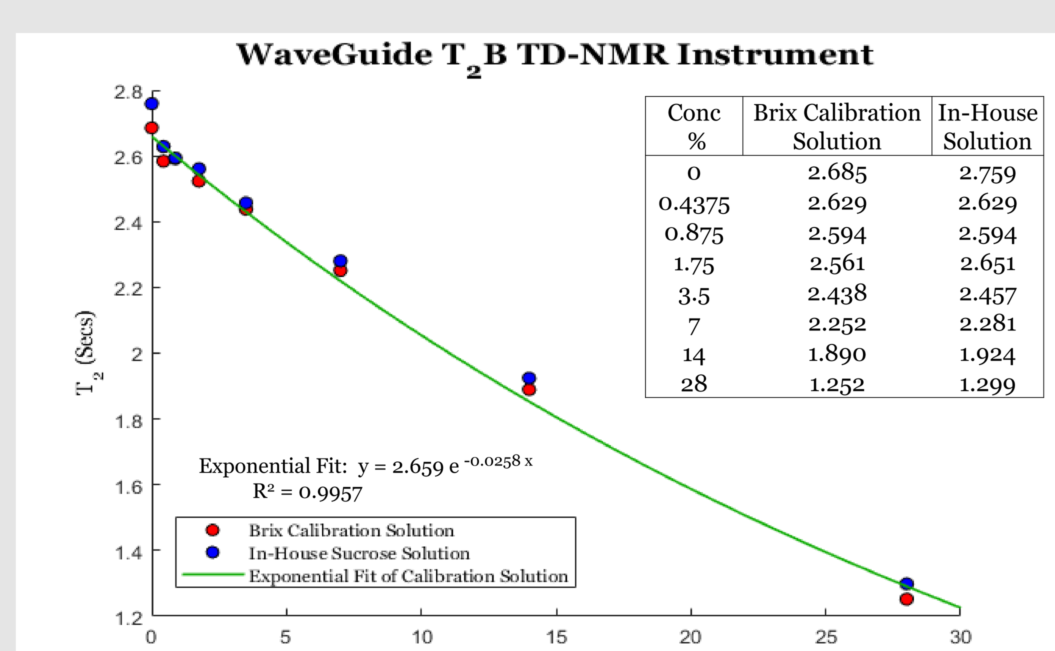

Figure 4.
TD-NMR Bi-Exponential Model T2b

Fruit (n=5)	Digital Refractometer °Bx		WaveGuide TD-NMR T2		T2a		T2b	
	Avg.	St. Dev.	Avg.	St. Dev.	Avg.	St. Dev.	Avg.	St. Dev.
Organic Grapefruit	8.5	0.000	1.855	0.006	1.950	0.010	0.838	0.020
Organic lemon	9	0.000	1.845	0.002	1.896	0.007	0.766	0.048
Wegman's clementine	9.9	0.000	1.556	0.025	1.647	0.044	0.701	0.021
Natalie's orange	11.8	0.000	1.169	0.008	1.280	0.018	0.676	0.009
Organic orange naval	13.3	0.000	1.296	0.008	1.357	0.018	0.517	0.024
Driscoll blackberry	13.9	0.000	0.446	0.003	0.539	0.003	0.205	0.002
Organic raspberry	14.9	0.000	0.328	0.005	0.455	0.010	0.281	0.005
Sumo orange	16.8	0.000	1.190	0.010	1.325	0.020	0.685	0.004
Wegman's blood orange	17.2	0.000	1.075	0.024	1.155	0.029	0.527	0.047
Cotton grape	18	0.000	1.169	0.001	1.243	0.004	0.629	0.024
Kiwi	18.1	0.000	0.720	0.023	0.808	0.052	0.457	0.035

Table 1. Table shows raw measurement data obtained from portable TD-NMR and digital refractometer devices. BRIX values represent sugar content (%) as measured by refractometry; corresponding TD-NMR values reflect T_2 relaxation times correlated with sugar concentration.