



NRC Nestlé Research Center



# Total Solids Determination in Dairy Products by Microwave Oven Technique

by

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# Importance of Total Solids Measurement

- **Total solids or dry matter determination is important for product release and process control.**
- **Rapid methods are required for real time measurement of production.**
- **The reference method by air drying oven is laborious and requires a laboratory.**
- **The microwave oven technique is promising due to the fast measurement. New improved instruments have entered the market like CEM Smart 5 or Sartorius MMA30.**





# CEM Smart System5™

## Components

Microwave Drying System

Electronic Balance

50 gram capacity  
> 0.1 mg sensitivity

1/4 VGA Black and White Screen

Infrared Temperature Controller

Microprocessor

Controls instrument  
Calculates results  
Able to interface with printers or computers

# Measurement Principle

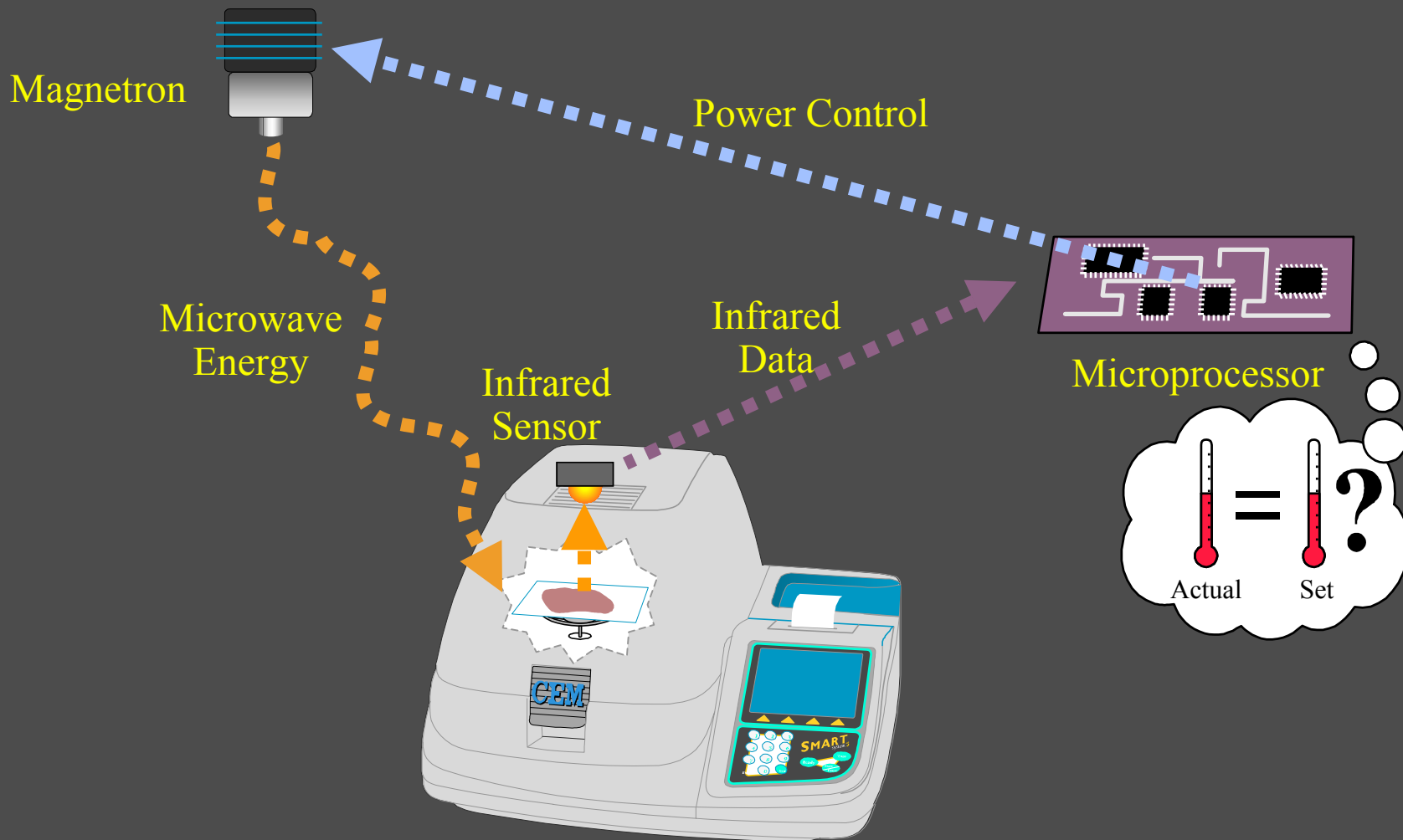
- **A sample is exposed to microwave energy, which very specifically reacts with water molecules.**
- **Water evaporates therefore very rapidly and can be principally removed in less than a minute.**
- **After evaporation of the water the sample the exposure of energy has to be stopped in order to avoid overheating of the sample and degradation of the product.**
- **The end of drying can be controlled by checking the temperature of the product or the weight loss of the balance.**



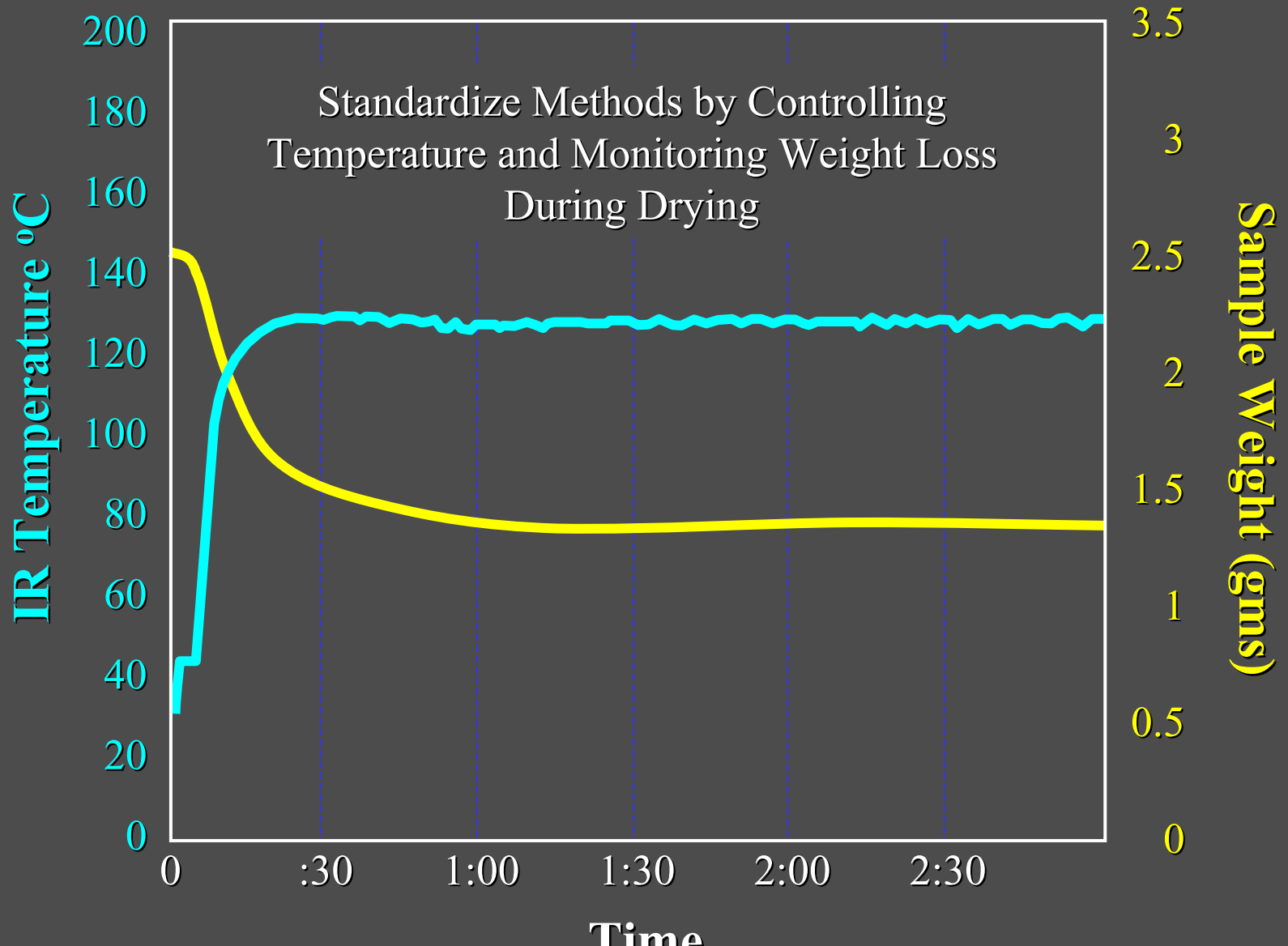
# IR Temperature Control

- **Rapid IR Temperature measurement of the sample during drying process**
- **Microwave power is automatically adjusted based on user defined temperature setpoint**
- **Rapid drying without overheating or burning the sample**

# IR Temperature Control



# Temperature Controlled Drying







# Tested Sample Matrices

## Divers Dairy Products

27 samples, pilot plant samples

## Sweetened Condensed Milk

13 samples, partially internal collaborative study

## Non-sweetened Condensed Milk

8 samples, internal collaborative study

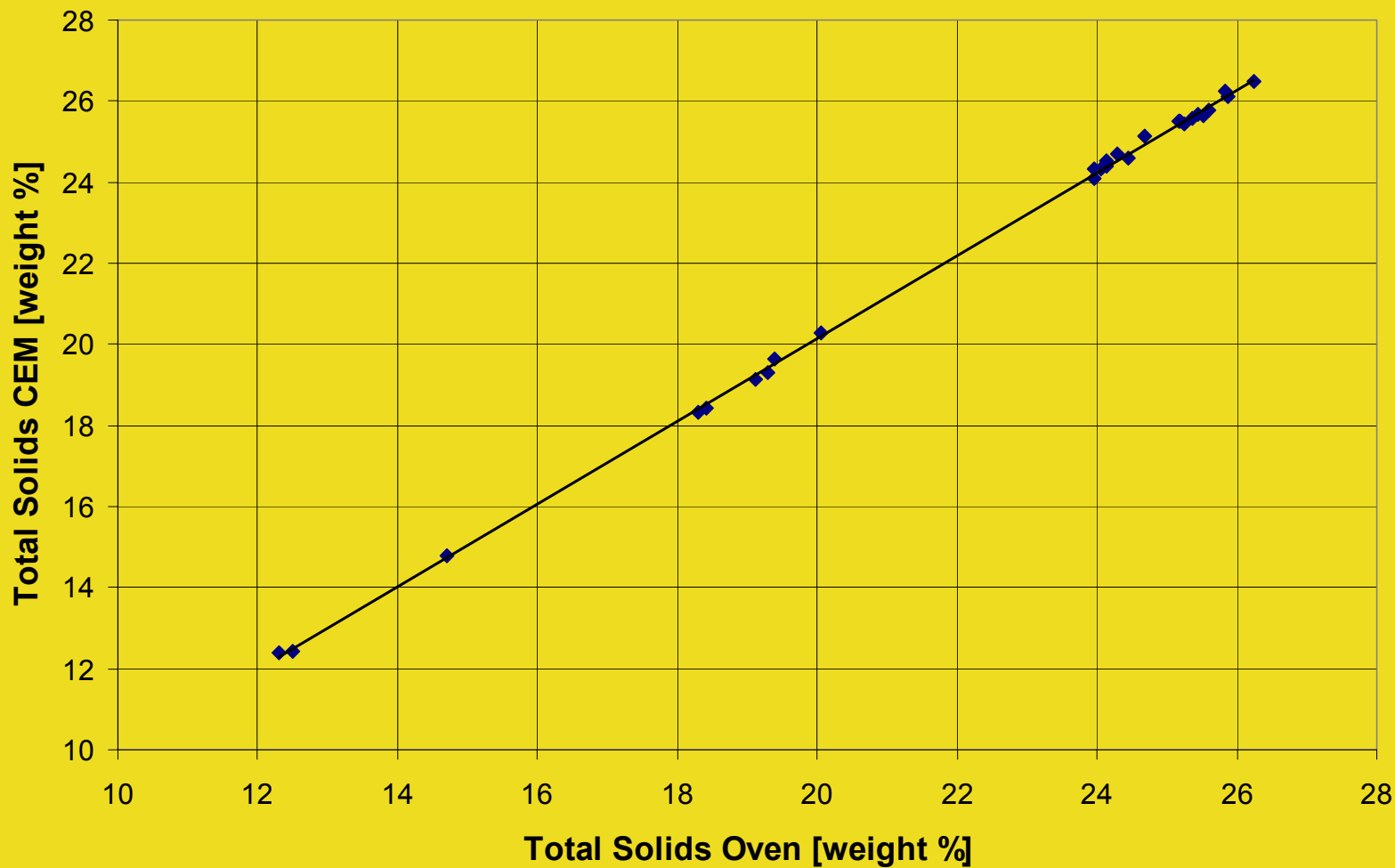
## Ice-cream Premix

21 samples, pilot plant samples

## Yogurt, Quark

4 samples for sampling and repeatability

### Total Solids by CEM Microwave Oven versus Oven Different Milk Products



Samples	Total Solids [weight %]		Difference CEM - Oven
	Oven	CEM Microwave Oven	
Premium 9 A	25.36	25.58	0.22
Premium 11 A	25.53	25.63	0.10
Assistenz 765 A	24.14	24.54	0.40
Assistenz 750 A	14.70	14.78	0.08
Bear 22 A	23.95	24.32	0.37
Premium 12 A	25.44	25.68	0.24
Reva 15 A	26.24	26.50	0.26
Reva 13 A	25.60	25.77	0.17
Imevap 2 A	24.06	24.33	0.27
Imevap 2 B	24.14	24.39	0.25
Stabilac 4 A	25.83	26.24	0.41
Free 6 A1	24.45	24.60	0.15
Hal Pep 7 A	19.40	19.65	0.25
Imevap 1 A	24.30	24.70	0.40
Sublime 12 A	25.24	25.45	0.21
Sublime 12 C	25.17	25.49	0.32
Creco 14A	18.41	18.44	0.03
Creco 15A	18.30	18.33	0.03
Creco 15C	19.11	19.12	0.01
Creco 11 A	19.29	19.29	0.00
Choc 1 A	12.50	12.43	-0.07
Innova 31 C	23.95	24.10	0.15
CFH 24 A	12.30	12.40	0.10
Hal-Pep 3 A1	20.06	20.28	0.22
Assistenz 751 A	24.69	25.13	0.44
Reva 12 A	25.87	26.10	0.23
Premium 7 A	25.18	25.50	0.32
Stand. Dev.	4.18	4.27	0.14
Minimum	12.30	12.40	-0.07
Maximum	26.24	26.50	0.44
Average	22.34	22.55	0.21



Creco 14 A	18.40	18.34	0.06
Creco 15 A	18.39	18.41	-0.02
Creco 15 C	19.10	19.11	-0.01
Creco 11 A	19.25	19.26	-0.01
Create 1 A	34.72	34.72	0.00
Choc 1 A	12.37	12.50	-0.13
Sublime 12 C	25.51	25.59	-0.08
Sublime 12 A	25.44	25.49	-0.05
Imeveap 2 A	24.36	24.30	0.06
Reva 15 A	26.66	26.54	0.12
Hal Pep 7 A	19.77	19.65	0.12
Free 6 A1	24.52	24.51	0.01
Stabilac 4 A	26.22	26.32	-0.10
Assistenz 765 A	24.52	24.48	0.04
Imeveap 2 B	24.41	24.40	0.01
Balance 21 A	33.53	33.57	-0.04
Premium 11 A	25.66	25.61	0.05
Premium 9 A	25.58	25.58	0.00
Reva 13 A	25.72	25.82	-0.10
Balance 19 A	33.34	33.41	-0.07
Premium 12 A	25.68	25.68	0.00
Bear 22 A	24.36	24.29	0.07
Assistenz 750 A	14.81	14.76	0.05
Innova 31 C	24.13	24.07	0.06
Cfh 24 A	12.41	12.40	0.01
Hal Pep 3 A1	20.29	20.27	0.02
Reva 12 A	26.02	26.19	-0.17
Assistenz 751 A	25.17	25.10	0.07
Premium 7 A	25.50	25.50	0.00
Balance 14 G	32.96	32.94	0.02
Balance 12 A1	27.40	27.27	0.13
Chantilly 6 A1	37.53	37.42	0.11





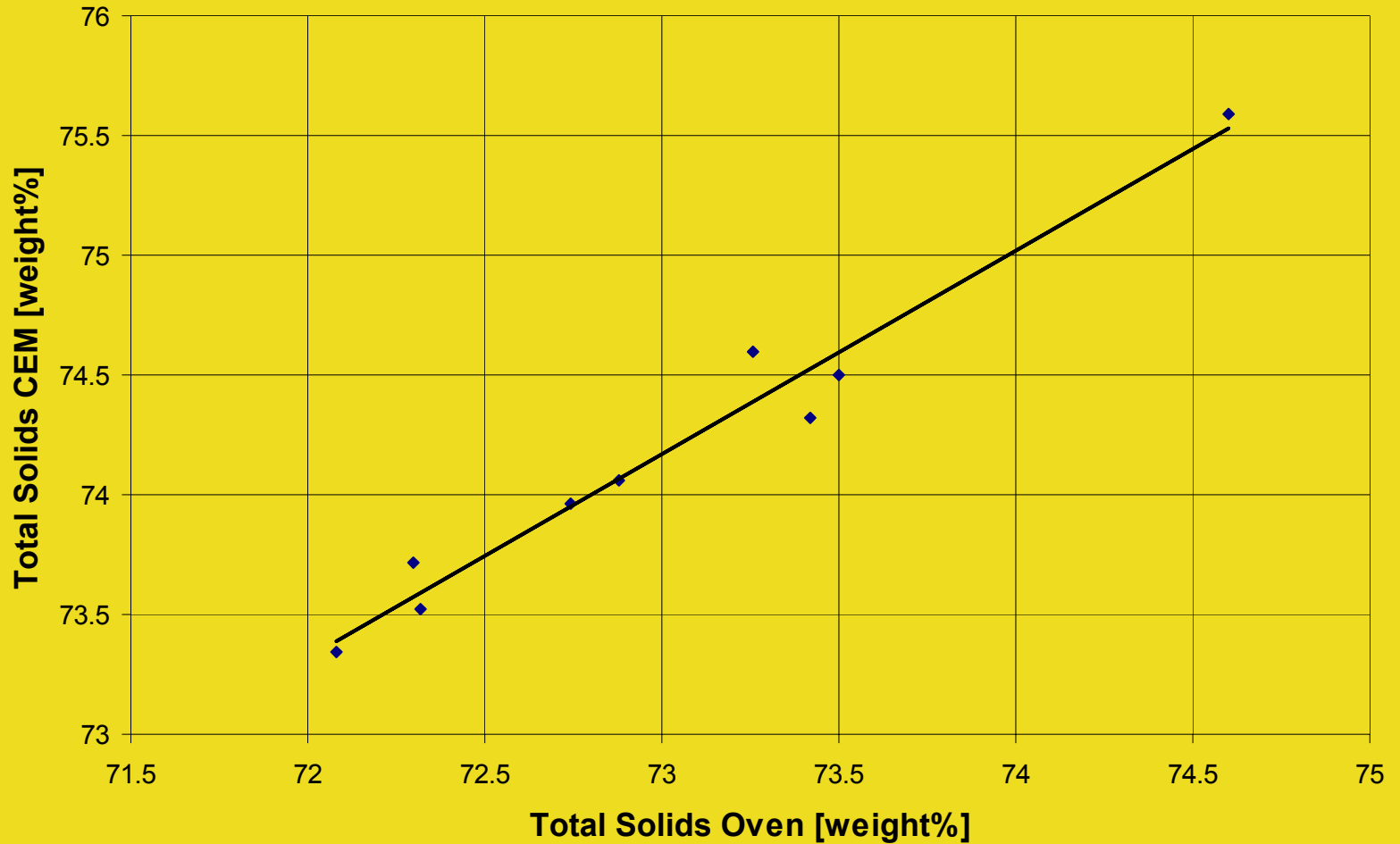
# Conclusions Divers Dairy Products

Reference method 102 +/- 2 °C, 4 hours using sand.

Repeatability is comparable to oven method (standard deviation of repeatability has been found 0.07 compared to 0.05 for oven method).

Both methods are highly correlated and give the same result. No bias is observed.

# Total Solids LCS





	CEM Microwave Oven	Oven	Difference CEM - Oven
Sample 1	73.96	72.74	1.22
Sample 2	73.72	72.30	1.42
Sample 3	74.50	73.50	1.00
Sample 4	74.32	73.42	0.90
Sample 5	73.34	72.08	1.26
Sample 6	75.59	74.60	0.99
Sample 7	74.06	72.88	1.18
Sample 8	73.52	72.32	1.20
Sample 9	74.60	73.26	1.34
Sample 10	74.51	73.32	1.19
Sample 11	75.69	74.69	1.00
Sample 12	75.25	74.19	1.06
Sample 13	74.31	73.35	0.96
Stand. Dev.	0.74	0.84	0.16
Average	74.41	73.28	1.13
Minimum	73.34	72.08	0.90
Maximum	75.69	74.69	1.42



# Conclusions Sweetened Condensed Milk

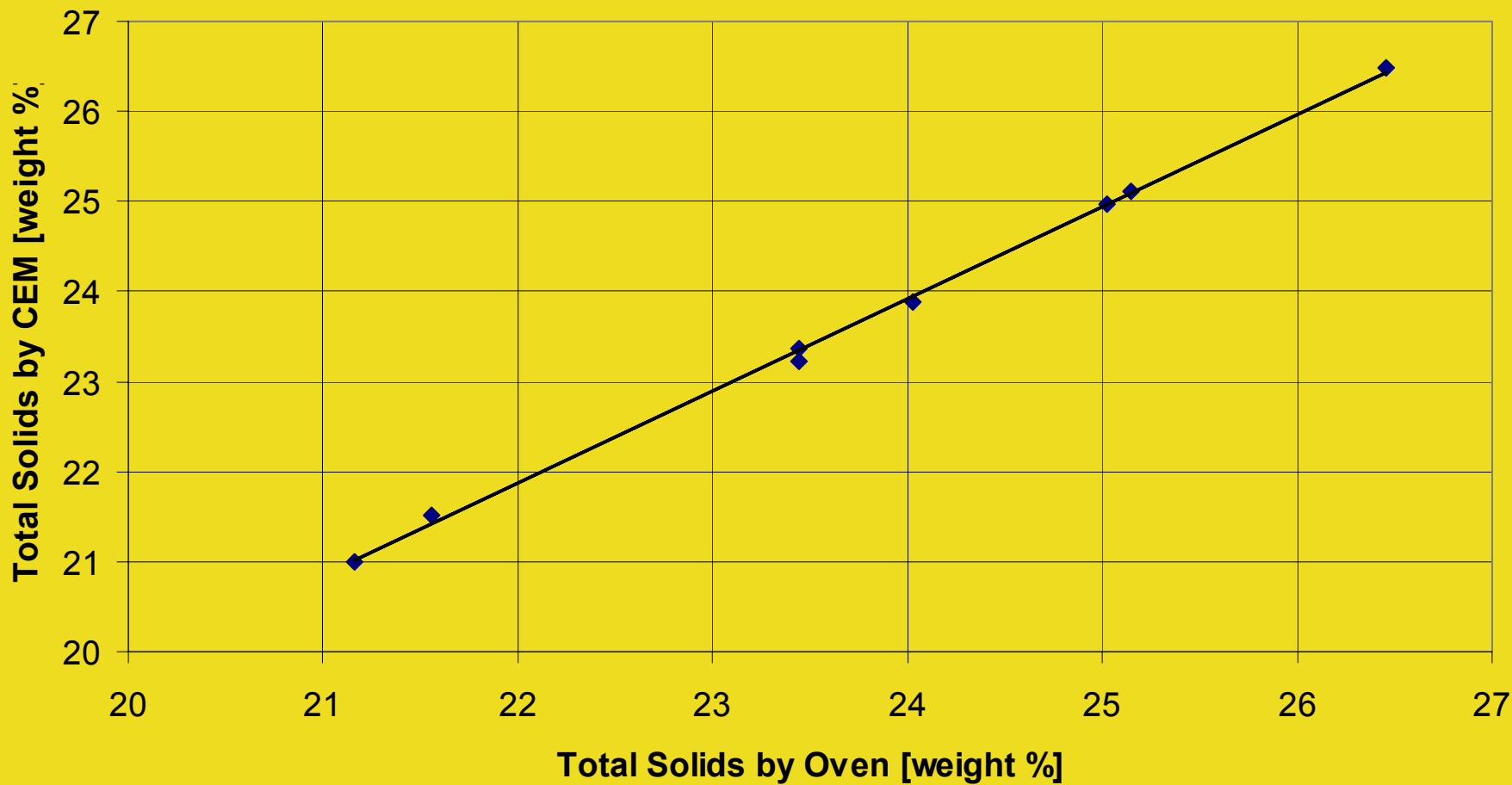
**Acceptable correlation with the reference method.**

**A significant bias of about 1 weight % has been identified.**

**The repeatability has been found significantly higher than with the reference method.  
(standard deviation of repeatability has been found 0.25 compared to 0.10 for oven method)**



### Total Solids by CEM Microwave Oven versus Oven Non-sweetened condensed Milk





	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7	Sample 8
CEM 1	25.02	23.41	21.50	25.22	21.18	23.40	24.07	26.43
CEM 2	25.02	23.41	21.51	25.22	21.14	23.48	24.02	26.40
CEM 3	24.90	23.41	21.55	25.13	21.15	23.45	24.01	26.47
CEM 4	25.02	23.45	21.62	25.13	21.14	23.46	24.01	26.42
CEM 5	25.04	23.43	21.56	25.12	21.14	23.41	24.09	26.47
CEM 6	25.08	23.53	21.61	25.16	21.23	23.42	23.99	26.50
CEM 7	25.02	23.49	21.59	25.14	21.21	23.44	23.97	26.49
CEM 8	25.05	23.48	21.50	25.14	21.15	23.43	24.06	26.49
CEM 9	25.03	23.45	21.57	25.14	21.09	23.50	24.04	26.46
CEM 10	25.01	23.38	21.58	25.12	21.18	23.41	24.01	26.47
Average	25.02	23.44	21.56	25.15	21.16	23.44	24.03	26.46
Stand. Dev.	0.046	0.045	0.041	0.038	0.040	0.033	0.037	0.033
cv %	0.18	0.19	0.19	0.15	0.180	0.14	0.15	0.12
Oven 4h. 102°C	24.97	23.36	21.52	25.11	20.99	23.23	23.88	26.48

Repeatability data of non-sweetened condensed milk by CEM microwave oven



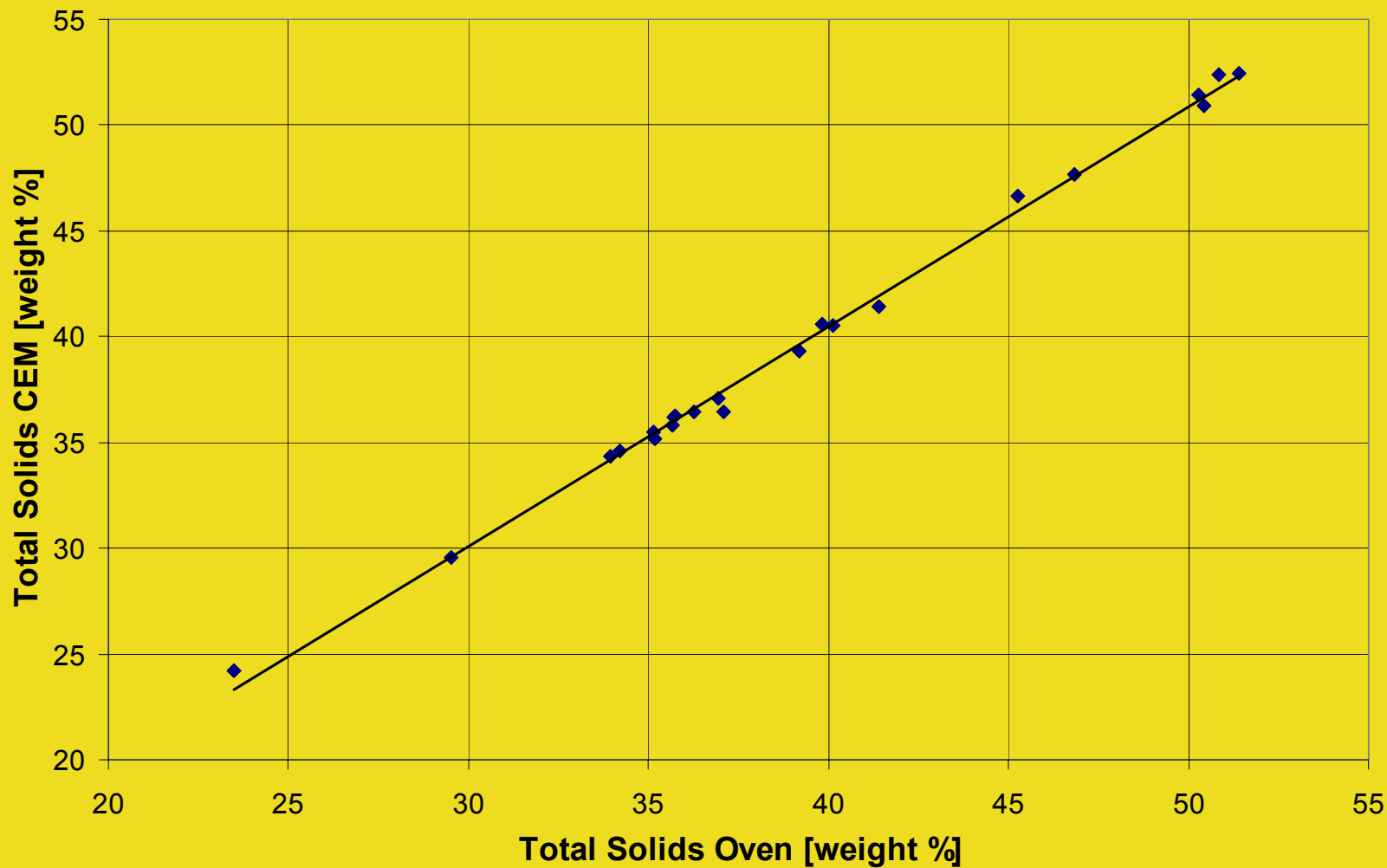
# Conclusions Non-sweetened Condensed Milk

Results comparable to reference method by sand solids oven method (102+/-2 °C, 4 h). Reference value has been determined by collaborative study within Nestlé.

Repeatability almost as good as reference method.

No bias detected.

### Total Solids by CEM Microwave Oven versus Oven Icecream Premix





# Conclusions Ice Cream Premix

**Reference method 102 +/- 2 °C, 4 hours using sand.**

**Repeatability is higher than for oven method (standard deviation of repeatability has been found 0.12 compared to 0.05 for oven method).**

**Both methods are highly correlated. A bias is observed for samples with higher total solids and fat content.**

	HIRZ nature	LC1 Nestlé vanille	HIRZ banane	Magerquark
<b>CEM 1</b>	14.40	23.98	24.57	16.18
<b>CEM 2</b>	14.65	23.90	24.60	15.99
<b>CEM 3</b>	14.50	24.08	24.51	16.07
<b>CEM 4</b>	14.43	24.12	24.52	16.12
<b>CEM 5</b>	14.55	23.85	24.68	16.03
<b>CEM 6</b>	14.42	23.86	24.91	15.87
<b>Average</b>	14.49	23.96	24.63	16.04
<b>Difference</b>	0.095	0.11	0.15	0.11
<b>CV %</b>	0.66	0.47	0.60	0.58
<b>Power</b>	100%	100%	100%	100%
<b>Température</b>	100°C	100°C	100°C	100°C



# Some experimental Considerations

- **A good distribution of the sample is important.**
- **The sample should penetrate well the filter pad. Everything which facilitates the evaporation improves the final result (dilution, sample size, etc.)**
- **Homogeneity of the samples is essential (Grinding, mixing or homogenization might be required).**
- **Degradation should be prevented by proper temperature selection (indication oven conditions)**



# Overall Study Results

	<b>SD(r)</b>	<b>BIAS</b>
<b>LNS</b>	<b>0.05 %</b>	
<b>LCS</b>	<b>0.25 %</b>	<b>ca. 1 %</b>
<b>Ice cream premix</b>	<b>0.15 %</b>	<b>ca. 0.5 %</b>
<b>Dairy Products</b>	<b>0.10 %</b>	<b>ca. 0.2 %</b>
<b>Yogurt</b>	<b>0.15 %</b>	
<b>Quark</b>	<b>0.15 %</b>	





# Advantages of CEM Microwave

**Provides usable information, quickly**

- **Rapid (1-3 minutes)**
- **Precise (up to 0.1%)**
- **Accurate (up to 0.1%)**

**Standardized methods on temperature**

**Rugged enough for the process area**

**Easy to operate**

- **Automatic Weighing and Calculating**
- **Reduced sample handling**
- **Less operator error**

**Very Reliable**

**AOAC methods for some products**



# Conclusions on Analytical Data

**Repeatability is better with lower total solids content and lower fat content.**

**Observed Bias increases with increase of total solids content.**

**The performance of the new microwave ovens (CEM, Sartorius) is strongly improved compared to older models. For lower total solids the performance approaches the oven sand solids method.**



# Overall Conclusions

- **It is important to establish the performance of instrumentation on different products and under real conditions.**
- **The choice of instrumentation needs to take into account the instrument performance and the specification of the product.**
- **For a wide range of products the microwave oven technique can replace the traditional oven technique.**
- **To optimize applications a good understanding of the instrument is required.**