Introducing
The MagnoMeter™ XRS
A new Instrument for Particle Characterization and Surface Analysis of Dispersions
A device that uses NMR relaxation for non-invasive routine analysis of complex solid-liquid and liquid-liquid formulations

- Samples in water, solvents and melts
- No dilution required, minimal sample preparation

Ideal for applications where speed of measurement and reliability are drivers of performance

- Research & development
- Quality control/quality assurance
- Process control labs
What is NMR relaxation?

- NMR relaxation time is a fundamental, intrinsic property of all solids and liquids.
  - Analogous to intensity of scattered light (particle sizing) and electrophoretic mobility (zeta potential).

- Relaxation time of suspensions is intermediate between that for solid and liquid.
  - Value depends on specific particle-liquid combination.
  - Determined directly using an NMR spectrometer.
Why use NMR relaxation?

“...In every industrial application, a knowledge and understanding of the molecular structure and dynamics at the particle-liquid interface is critical to improving, or optimizing, suspension and emulsion product performance.”
High resolution NMR
well-known technique for studying molecular structure and identification of compounds

Low field NMR
new technique for suspension and emulsion analysis

- high frequency needed
- expensive, complex, sophisticated operation
- intensive training
- university and analytical laboratories

- low frequency optimal
- inexpensive, simple benchtop device
- easy operation
- industrial R&D, QC/QA and Process laboratories
What can you measure?

- Dispersed (wetted) Surface Area (suspensions)
- Dispersed particle volume fraction
- Molecular weight (polymers in solution)*
- Polymer and solvent viscosity
- Particle Size (<30nm)

- Relaxation Number (liquids/suspensions/emulsions)
- Kinetic processes
  - Adsorption/desorption
  - Competitive adsorption
  - Colloidal stability
- Presence of para- and ferromagnetic impurities
- Hydroxyl (OH) number of metal oxides
- Oxygen and water content of solvents

* By calibration
Applications

The MagnoMeter can be used in an almost unlimited range of applications, and can measure samples at virtually any industrially relevant solids concentration.

- Graphene/Graphene Oxide
- Cellulose nanocrystals
- Ceramics, refractories
- Minerals, metal oxides
- Paints and inks
- Dyes
- Pharmaceutics
- Nanomedicine
- Cosmetics
- Food emulsions
- Agrochemicals
- Catalysts
- Paper pulp
- MOFs
Incoming raw materials

The MagnoMeter is a fast, simple tool for easy comparison of raw materials.

LIME: 11wt% in MeOH

ALUMINA: 37.5wt% in EtOH

Industrial raw materials are not pure → type and level of impurities depends on source and processing.
**MagnoMeter** measurements can be used as a quick estimation of product end-use performance testing

<table>
<thead>
<tr>
<th>Type</th>
<th>NMR Surface Area* (m²g⁻¹)</th>
<th>Tensile Strength** (psi)</th>
<th>Relative Abrasion**</th>
<th>Relative Road Wear**</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oil Furnace</td>
<td>129</td>
<td>3600</td>
<td>1.35</td>
<td>1.25</td>
</tr>
<tr>
<td>Oil Furnace</td>
<td>90</td>
<td>3300</td>
<td>1.25</td>
<td>1.15</td>
</tr>
<tr>
<td>Gas furnace</td>
<td>44</td>
<td>2300</td>
<td>0.56</td>
<td>0.66</td>
</tr>
<tr>
<td>Gas furnace</td>
<td>34</td>
<td>1800</td>
<td>0.48</td>
<td>0.60</td>
</tr>
</tbody>
</table>

* 10 wt% Carbon Blacks dispersed in a mixed solvent
** SBR Rubber tires
The **MagnoMeter** provides fast determination of powder wettability

Comparison of relaxation time of a hydrophobic microfine ZnO* dispersed (8wt%) in hexane and 100 Cst silicone fluid

- **Hexane**: 2100, 2050, 2000, 1950, 1900, 1850, 1800, 1750
- **Silicone**: 1200, 1100, 1000, 900, 800, 700, 600, 500

Average Relaxation Time repeatability: < 1.0%

*Obtained from three different manufacturers (USA, EU and Japan) and claimed as equivalent products: same particle size and same silane-based “coating”. 
R&D: The Best Dispersant for a Silver Metal Powder?

The MagnoMeter allows rapid determination of the suitability and efficacy of dispersants.

<table>
<thead>
<tr>
<th>Dispersant type</th>
<th>Relaxation Time (ms)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Texanol</td>
<td>266</td>
</tr>
<tr>
<td>A</td>
<td>170</td>
</tr>
<tr>
<td>B</td>
<td>132</td>
</tr>
<tr>
<td>C</td>
<td>123</td>
</tr>
<tr>
<td>D</td>
<td>162</td>
</tr>
<tr>
<td>E</td>
<td>94</td>
</tr>
</tbody>
</table>

A: No dispersant  
B: Zephrym PD 2206  
C: Hypermer B210  
D: Crodafos M915A  
E: Hypermer KD1

60wt% silver flake in texanol; 2wt% dispersant

Average Relaxation Time repeatability: < 1.0%
R&D: Dispersion of a Blue Dye

The MagnoMeter is a fast tool to aid in optimizing the formulation and preparation of any suspension.

<table>
<thead>
<tr>
<th>Surfactant (trade name)</th>
<th>Surface Area (m²g⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>None</td>
<td>15.4</td>
</tr>
<tr>
<td>Fluorad</td>
<td>16.0</td>
</tr>
<tr>
<td>Natrol 42</td>
<td>18.5</td>
</tr>
<tr>
<td>Lomar D</td>
<td>35.5</td>
</tr>
<tr>
<td>Aerosol AOT</td>
<td>42.0</td>
</tr>
<tr>
<td>Ultravon W</td>
<td>46.2</td>
</tr>
</tbody>
</table>

Aqueous blue (carboxy) dye suspension: 20% wt/wt; pH10
 QC: An API Product

MagnoMeter measurements are non-destructive samples can be stored and re-analyzed

Relaxation time of batches of paliperidone palmitate

Pass batches after 3 months aging at 40°C

Target: Invega
MagnoMeter measurements are very fast allowing for rapid QC of dispersions

<table>
<thead>
<tr>
<th>Pigment</th>
<th>Solids Wt%</th>
<th>Surface Area (m²/gm)</th>
<th>Pass/Fail</th>
</tr>
</thead>
<tbody>
<tr>
<td>Y1A</td>
<td>12.9</td>
<td>83</td>
<td>Y</td>
</tr>
<tr>
<td>Y1B</td>
<td>13.4</td>
<td>60</td>
<td>N</td>
</tr>
<tr>
<td>Y4C</td>
<td>10.8</td>
<td>30</td>
<td>Y</td>
</tr>
<tr>
<td>Y4D</td>
<td>10.0</td>
<td>26</td>
<td>N</td>
</tr>
<tr>
<td>M1A</td>
<td>18.3</td>
<td>90</td>
<td>Y</td>
</tr>
<tr>
<td>M1B</td>
<td>18.5</td>
<td>31</td>
<td>N</td>
</tr>
<tr>
<td>C1E</td>
<td>15.6</td>
<td>56</td>
<td>Y</td>
</tr>
<tr>
<td>C1F</td>
<td>15.3</td>
<td>46</td>
<td>N</td>
</tr>
</tbody>
</table>
**QA: Lotions**

*MagnoMeter measurements can be used to rapidly screen commercial products*

Comparison of six commercial Lotions

<table>
<thead>
<tr>
<th>Product*</th>
<th>Cost ($/kg)</th>
<th>Short T$_2$ (ms)</th>
<th>Oil Phase %</th>
<th>Long T$_2$ (ms)</th>
<th>Water Phase %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Jergens</td>
<td>57</td>
<td>278.1</td>
<td>30.0</td>
<td>540.7</td>
<td>70.0</td>
</tr>
<tr>
<td>Generic</td>
<td>16</td>
<td>80.2</td>
<td>21.3</td>
<td>301.7</td>
<td>78.7</td>
</tr>
<tr>
<td>Aveeno</td>
<td>51</td>
<td>147.7</td>
<td>53.1</td>
<td>337.1</td>
<td>46.9</td>
</tr>
<tr>
<td>Generic</td>
<td>38</td>
<td>255.7</td>
<td>43.3</td>
<td>497.0</td>
<td>56.7</td>
</tr>
<tr>
<td>Gold Bond</td>
<td>26</td>
<td>47.1</td>
<td>24.0</td>
<td>244.1</td>
<td>76.0</td>
</tr>
<tr>
<td>Generic</td>
<td>21</td>
<td>76.9</td>
<td>47.6</td>
<td>251.0</td>
<td>52.4</td>
</tr>
</tbody>
</table>

** Composition of each product pair is similar
Data suggests that product performance behavior will differ**
Lotions: Rheology data*

* TA Rheometer Model AR 1000

MagnoMeter measurements correlate well with emulsion viscoelastic characteristics

<table>
<thead>
<tr>
<th>Product</th>
<th>Viscosity (Pa.s)</th>
<th>Yield (Pa)</th>
<th>Short $T_2$ (ms)</th>
<th>Long $T_2$ (ms)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Jergens</td>
<td>93,640</td>
<td>20</td>
<td>80</td>
<td>302</td>
</tr>
<tr>
<td>Generic</td>
<td>66,790</td>
<td>10</td>
<td>278</td>
<td>541</td>
</tr>
<tr>
<td>Aveeno</td>
<td>280,170</td>
<td>100</td>
<td>256</td>
<td>497</td>
</tr>
<tr>
<td>Generic</td>
<td>450,040</td>
<td>170</td>
<td>148</td>
<td>337</td>
</tr>
<tr>
<td>Gold Bond</td>
<td>233,700</td>
<td>60</td>
<td>77</td>
<td>251</td>
</tr>
<tr>
<td>Generic</td>
<td>280,400</td>
<td>110</td>
<td>47</td>
<td>244</td>
</tr>
</tbody>
</table>

For each emulsion pair: (a) as Viscosity increases, the long $T_2$ is seen to decrease
(b) as Yield increases, the short $T_2$ is seen to decrease
Processing: Graphene Oxide

MagnoMeter measurements are very fast, and processing can be followed in almost real-time.

Two sets of GO process material: slurries in water at 3wt%.

Average Relaxation Time repeatability: < 1.0%
Processing: Zinc Oxide

MagnoMeter measurements do not require dilution – samples can be taken directly from mill for analysis.

Two sets of ZnO process material: slurries in C\textsubscript{12}-C\textsubscript{14} alkyl benzoate.

<table>
<thead>
<tr>
<th>No dispersant: 52% solids</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.5% PHSA: 60% solids</td>
</tr>
</tbody>
</table>
Direct MagnoMeter measurements on 30% ceramic millbase slurry trends with BET (gas adsorption) data.

Surface Area (m^2g^-1) vs. Milling Time (hours) for 30mm milling media.

MagnoMeter Measurements take minutes, not hours – and with no sample preparation.
What does the MagnoMeter provide?

- Direct information about the extent and nature of any particle-liquid interface → suspensions and emulsions
  - any type of particle, and any liquid – including mixtures
  - exceptionally wide concentration (0.01% to 90+%)  
  - no dilution required, minimal sample preparation
  - small samples (typically 0.1mL; as little as 200μL)

- Complementary information to traditional particle characterization techniques

- Intelligence that is not possible with traditional techniques
Summary

Advantages of the MagnoMeter

General
- Any colloidal-size suspension – virtually all liquids
- Measure locally with data manipulation at remote terminal
  - Ideal for controlled or hazardous environments
- Multiple pod sensors facilitate sampling at different locations
- Frequency lock
- Measure mixture homogeneity

Options and Extensions
- Large diameter NMR tube
  - Viscous liquids, concentrated slurries/emulsions
- MRI methods (imaging) in specific samples
- Can be adapted for use with an auto-sampler
  - Multiple sample analysis
- Can be adapted for flow-through operation
In Conclusion

The MagnoMeter finds use from Fundamentals to End-Use Performance

- **Research & Development**
  - Basic formulation of products: reproducibility, stability (coagulation/flocculation), settling and sedimentation, shelf life

- **Quality Control**
  - Release of raw materials, batch-to-batch reproducibility of final product

- **Quality Assurance**
  - Enable release of complex systems which can currently only be characterized by the raw materials used rather than the manufactured product

- **Process Management**
  - Follow and monitor milling and manufacture processes in almost real-time
THANK YOU!

For more information, to send samples, to arrange a demonstration at your facility, or to speak to a technical applications specialist, please contact:

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