Measuring and understanding the flow properties of powders with the FT4 Powder Rheometer®
An introduction to powders

Powders are complex materials. Often perceived as just a collection of particles, there are in fact a complex mixture of solids, liquids and gases. Unlike the relatively well-characterized constituent phases from which they are derived, powders are more accurately described as a composition of solids in the form of particles suspended in a mixture of particles and gases. The surface of the powder is of particular importance. The surface characteristics of these unique particles are difficult to model and predict from first principles.

Powder behaviour

Powders exhibit many behavioural characteristics, which determine how they perform during processing and in application. These characteristics are highly dependent on the environment, and are important to understand and to be able to measure the influence of each. If a powder is likely to be fully characteristic.

1. Fluidity — some may flow well through an aperture, others may bridge, stick or flow erratically.
2. Compressibility — some are very stiff, others may experience a large change in density when consolidated.
3. Adhesiveness — powders may stick to processing equipment, others slide easily.
4. Permeability — the ease with which air can be transmitted between particles can be crucial during processing, and in some final product applications.
5. Electrostatic charge — some powders become electrostatically charged as a result of handling and processing, resulting in a change in their behaviour.
6. Hydrophobicity — most powders experience a change in behaviour if humidity or water content increases, but to varying extents.

Dynamic methodology

The FT4 employs patented technology for measuring the resistance of the powder to flow, whilst the powder is in motion. A precision ‘blade’ is rotated and moved downwards through the powder to establish a precise flow pattern. This causes many thousands of particles to interact, or flow relative to one another, and the resistance experienced by the blade represents the difficulty of this relative particle movement, or the bulk flow properties. The more difficult it is to move the blade, the more the particles resist motion and the harder it is to get the powder to flow.

Excellent reproducibility is achieved by moving the blade in a precise and repeatable way. The advanced control systems of the FT4 accurately set the rotational and vertical speeds of the blade, which defines the Holec Angle and Tip Speed.

The FT4 has proven application in all powder processing industries, including Pharmaceuticals, fine chemicals, foods, cosmetics, toners, inks, Inks, Plastics, Powder Coatings, Cement and Additive Manufacturing.

Applications extend to:

- Filtration
- Labelling compaction
- Bottle flow
- Wall granulation and three-point scale-ups
- More accurate measurement or optimisation
- Humidity effects
- Electrostatic charge
- Meting / blending
- Feeding
- Segregation
- Attrition
- Dry powder inhalers
- Calculating
- Melting
- Conveying
- Wall friction and adhesion
- Hopper design
- Compact hardness and payoff

Other methods for investigating flow rate sensitivity and agglomeration and the effect of particle size reduction / particle shape changes are also available.

Whether your objective is to optimise a formulation in a laboratory environment, predict in-process performance, understand batch differences, or to ensure the quality of raw materials or intermediates, the FT4 will provide valuable and unique information that will help you address your challenges.

Intuitive software and flexible accessories

The FT4 is supplied with fully configured software that has been written in accordance with GPPC 0112 Part II 1 parameters. The instrument measurement controls are intuitive and easy to use, guiding the operator through a wizard-style interface to ensure samples are analysed in accordance. The Data Analysis package comes with a new scientific analysis area, improving ease of use and prompt to be carried out away from the lab, and by any number of onsite users. A further application in the form of bespoke software allows process engineers, comprehensive support on all methodologies, data interpretation, calibration and automation now may be required.

A full range of accessories is available for the FT4, including sequential vacuum leaves allowing sample volumes in the range 10ml to 160ml to be analysed. In addition, the 1ml trial kit can be utilised when very limited samples sizes are available. Further accessories include compactation pistons, sieve heads, tablet inserts, an suction control unit and a universal adapter. A calibration standard powder is also available, if required. For a full list of available accessories, please contact Freeman Technology or your local representative.

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It becomes easy to see why powders cannot be described with just one or two numbers, requiring by contrast the measurement of a range of parameters to achieve a thorough understanding. Each of these properties will influence the way the powder behaves within the process environment.

For example:
- Does the powder mix properly?
- Does it consolidate into one solid lump if left in storage or after vibration?
- Does it flow efficiently out of a hopper?
- Does it change its behaviour if exposed to high humidity?
- Can fill weight be accurately and consistently achieved during a filling operation?

In addition, powder properties also influence the characteristics of the finished product.

For example in relation to:
- Pharmaceutical tablet properties – powder determines weight variability, hardness, dissolution and stability.
- Powder coating – whether the powder fluidises efficiently and can be sprayed uniformly onto the panel, without agglomerating.
- Chemical manufacturing – is this powdered raw material too cohesive to mix well in our process?

Powder behaviour influences both in-process performance and the properties of the finished product.
A unique set of measured parameters

The dynamic principle of the FT4 requires that the blade rotates and moves vertically, both downwards and upwards. As a result, it will experience a resistance to rotation and a resistance to vertical movement. The FT4 measures both rotational and vertical resistances, in the form of Torque and Force, respectively. Both signals need to be measured, as it is the composite of these two signals that quantifies the powder’s total resistance to flow.

**Process diversity**

The nature of all processing environments is such that a range of conditions is unavoidable and the powder being processed will be handled under different stress regimes. In order to fully predict the powder’s in-process performance, it is essential to measure and quantify how it responds to each of these external variables.

<table>
<thead>
<tr>
<th>External variable</th>
<th>When and where</th>
<th>Effect</th>
</tr>
</thead>
</table>
| Consolidation     | Vibration / Tapping  
                    Direct pressure (hopper, IBC, keg) | Increase in particle pressure, contact area and number of contact points  
                    Reduction in air content between particles (reduced porosity) |
| Aeration          | Gravity discharge  
                    Blending  
                    Pneumatic conveying  
                    Aerosolisation | Reduction in particle pressure, contact area and number of contact points  
                    Increase in air content between particles (increased porosity) |
| Flow (shear) rate | Within powder  
                    Powder against equipment wall  
                    Mixing | Mostly non-Newtonian  
                    Greater resistance to flow at lower flow rates |
| Moisture          | Storage  
                    Processing  
                    Intentionally introduced (granulation) | Increase particle adhesion  
                    Reduces particle stiffness - more compliant but increased contact surface area  
                    Increase electrical conductivity |
| Electrostatic charge | Discharge from hopper  
                        Pneumatic conveying  
                        High shear mixing | Increase bond strength between particles  
                        Adhesion of powder to equipment |
| Storage time      | Raw materials / Intermediates | Consolidation  
                    Caking  
                    Permanently affecting downstream performance? |
Using the calculation of Work Done, it is possible to represent both resistances as a total energy, the energy required to move the blade through the powder from the top to the bottom of the powder column. However, as the blade travels through the powder the values of torque and force are constantly changing, so it is necessary to frequently calculate the energy required to move through the powder over very small distances travelled. This is the calculation of Energy Gradient, the energy measured for each millimetre of blade travel, expressed in mJ/mm.

Work Done = Energy = (Resistance x Distance travelled)

where ‘Resistance’ is the combined Torque and Force

Energy Gradient = Energy per mm of blade travel

Calculating the area under the Energy Gradient curve provides the Total Flow Energy, representing the powder’s resistance to being made to flow in a dynamic state.

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Accuracy
Excluding either Torque or Force signals would result in misleading data, as the calculated Flow Energy value would not represent the powder’s total resistance to flow.

Due to the rotational nature of the technique, approximately 90% of the total resistance is contributed from the Torque signal, with the remaining 10% from the Force component. This highlights the importance of measuring Torque as well as Force when evaluating rheological properties.

Completing the picture

In addition to the dynamic methodology, where the blade is used for measuring flow energy, the FT4 utilises other accessories and operating modes to fully characterise your powders.

Aeration
The Aeration Control Unit is a device that provides a precise air velocity to the base of the vessel containing the powder. A wide range of velocities is available and the device communicates automatically via USB with the FT4 computer.

Air in
Aeration test
Air can also be introduced whilst the powder is being consolidated using the vented piston. For a given air velocity and applied consolidating stress, the air pressure measured at the bottom of the powder column quantifies the resistance of the powder to transmitting air between the particles. The more resistant the bed, the greater the measured air pressure and the lower the permeability.

Air in
Permeability test

Axial compression
A “vented piston” can be applied to the top of the powder column in order to consolidate the powder under a controlled and precise normal stress.

Rotational shearing
Shear Cell and Wall Friction Modules can be attached to the FT4 and used to measure the shear strength of the powder and the wall friction between the powder and a particular wall material (in accordance with ASTM D7891). A controlled normal stress is applied, then rotation occurs to induce shearing. The greater the resistance, the higher the shear strength.

The introduction of air into the base of the powder during a dynamic test allows the Aerated Energy to be measured. This quantifies how the powder’s flow properties change as it becomes aerated, a property that is directly related to the cohesive strength of the powder.
Methodologies

The FT4 Powder Rheometer is a truly universal powder tester, with four categories of methodologies, defined as Bulk, Dynamic Flow, Shear, and Process.

**Basic Flowability** – measuring the dynamic flow properties of powders in a conditioned state.
- Flow additives
- Wet granulation end point
- Moisture content
- Attrition / Segregation
- Physical properties (particle size, shape, surface texture, etc)
- Electrostatic charging

**Aeration** – a direct measurement of a powder’s cohesive strength.
- Low stress, gravitationally induced flow
- Dosing / Mass uniformity
- Aerosolisation / DPI
- Fluidisation behaviour
- Blending / Mixing
- Segregation potential

**Shear Cell** – quantifying a powder’s shear strength in accordance with ASTM standard D7891. For understanding behaviour in hoppers, and for use in hopper design exercises. Sample sizes down to 1ml.
- Unconfined Yield Strength
- Flow function
- Cohesion
- Angle of Internal Friction

**Wall Friction** - measuring the friction acting between a powder and equipment surfaces in accordance with ASTM standard D7891. Also required for hopper design.
- Measure friction between powder and surface material
- Hopper, IBC, Punch, Die wall
- Wall friction angle for hopper design

**Consolidation** – understanding the effect of direct consolidation or vibration on powder flow properties.
- Direct pressure
- Tapped
- Understanding the effects of:
  - Transport
  - Storage
  - Processing
  - Caking

**Compressibility** – determining changes in a powder’s density as a result of a directly applied consolidating load.
- Transportation
- Storage
- Hoppers
- Kegs
- Processing
- Tablet compression
- Roller compaction
- Screw feeding
- Extrusion

**Permeability** – measuring the resistance to air flow between particles and through the powder bed.
- Tablet capping / Lamination
- Aerosolisation / DPI
- Hopper flow
- Compression
- Pneumatic transfer
A universal powder tester

The FT4 Powder Rheometer was originally designed to characterize the morphology of powders, understanding powder behaviour. A universal powder tester makes the FT4 a truly universal powder tester and by far the world’s most versatile instrument for measuring and time-saving on a minimal sample size. Accurate results allow you to optimize your process and improve your final product. The FT4 is now considered a universal powder tester.

The way these phases interact determines the behaviour of the powder.

### Powders

- **Flowability** — Some powders flow well through a process, others flow poorly.
- **Compressibility** — Some powders are very compressible, others are more rigid. Powders can be compressed or sliced like solids.
- **Adhesiveness** — Powders may stick to each other and to the surface of the apparatus in the test chamber. This can affect the results of the test.
- **Electrostatic charge** — Powders can accumulate static charges, which can affect handling and processing.
- **Hydrophobicity** — Powders can interact with liquids, but not all powders interact with water. Powders can be classified as hydrophilic or hydrophobic.
- **Particle adhesion** — Powders can be sticky and adhere to each other or to the surface of the apparatus in the test chamber.

### Solids

- **Flowability** — Some solids flow well through a process, others flow poorly.
- **Compressibility** — Some solids are very compressible, others are more rigid. Solids can be compressed or sliced like solids.
- **Adhesiveness** — Solids may stick to each other and to the surface of the apparatus in the test chamber. This can affect the results of the test.
- **Electrostatic charge** — Solids can accumulate static charges, which can affect handling and processing.
- **Hydrophobicity** — Solids can interact with liquids, but not all solids interact with water. Solids can be classified as hydrophilic or hydrophobic.
- **Particle adhesion** — Solids can be sticky and adhere to each other or to the surface of the apparatus in the test chamber.

### Liquids

- **Flowability** — Liquids flow easily through a process.
- **Compressibility** — Liquids are not compressible.
- **Adhesiveness** — Liquids do not stick to each other or to the surface of the apparatus in the test chamber.
- **Electrostatic charge** — Liquids do not accumulate static charges.
- **Hydrophobicity** — Liquids do not interact with solids.
- **Particle adhesion** — Liquids do not stick to each other or to the surface of the apparatus in the test chamber.

### Gases

- **Flowability** — Gases flow easily through a process.
- **Compressibility** — Gases are compressible.
- **Adhesiveness** — Gases do not stick to each other or to the surface of the apparatus in the test chamber.
- **Electrostatic charge** — Gases do not accumulate static charges.
- **Hydrophobicity** — Gases do not interact with solids.
- **Particle adhesion** — Gases do not stick to each other or to the surface of the apparatus in the test chamber.

### Dynamic methodology

The FT4 employs patented technology for measuring the resistance of the powder to flow, whilst the powder is in motion. A precision ‘blade’ is rotated and moved downwards through the powder to establish a precise flow pattern. This causes thousands of particles to rearrange or flow relative to one another, and the resistance experienced by the blade represents the difficulty of this relative particle movement, or the bulk flow properties. The more difficult it is to move the blade, the more the powder resists motion and the harder it is to get it to flow.

Excellent reproducibility is achieved by moving the blade in a precise and repeatable way. The advanced control systems of the FT4 accurately set the rotational and vertical speeds of the blade, which defines the Holo Angle and Tip Speed.

### Proven applications

The FT4 has proven application in all powder processing industries, including Pharmaceuticals, Fine Chemicals, Foods, Cosmetics, Toners, Reagents, Laminars, Plastics, Powder Coatings, Cement, and Additive Manufacturing.

### Applications extend to:

- **Flowing**
- **Labelled compression**
- **Hopper flow**
- **Wall granulation and blend scale up**
- **More advanced scale up or optimisation**
- **Humidity effects**
- **Electrostatic charge**
- **Mixing / blending**
- **Feeding**
- **Segregation**
- **Attrition**
- **Dry powder inhalers**
- **Caking**
- **Milling**
- **Conveying**
- **Wall friction and adhesion**
- **Hopper design**
- **Compact hardness and payoff**

### Other methods for investigating flow rate sensitivity, agglomeration and the effect of particle size reduction / particle shape changes are also available.

Whether your objective is to optimise a formulation or process, whether your sample size is small or large, or whether you are starting from fresh or need to re-characterise powders, the FT4 will provide valuable and unique information that will help you address your challenges.

### Intuitive software and flexible accessories

The FT4 is supplied with fully configured software that has been written in accordance with CFR21 Part 11 parameters. The powerful measurement control software is intuitive and easy to use, guiding the operator through a wizard style interface to ensure samples are tested in accordance with the test method. The Data Analysis package comes with a user-friendly software configuration and reporting to be carried out away from the lab, and by any number of on-site users. A further application in the form of 'BagPack' continually processes on instrument, comprehensive support on all methodologies, data interpretation, calibration and accessories now may be required.

A full range of accessories is available for the FT4, including sample vessels allowing sample volumes in the range 1ml to 160ml to be analysed. In addition, the FT4 has a wide range of accessories to ensure the quality of raw materials or intermediates, whether your objective is to optimise a formulation or process, whether your sample size is small or large, or whether you are starting from fresh or need to re-characterise powders, the FT4 will provide valuable and unique information that will help you address your challenges.

### Support Documents

Further accessories include compaction pistons, shear heads, pellet from kilos, an adhesion control unit and a particlesubmitter. A calibration standard powder is also available, if required. For a full list of available accessories, please contact Freeman Technology or your local representative.

### Comprehensive support on all methodologies, data interpretation, calibration and additional help that may be required.

In addition, the FT4 can be used to work with real-world experience. Ongoing consultation and applications support, based on real-world experience.
FT4 Specifications

**SYSTEM:**

FT4 Powder Rheometer intended for use in a laboratory environment for measuring the rheological properties of powders, pastes and semi-solids. Complies with the following EMC specifications and ASTM International standards:
- EN61000-3-2:2001
- EN61000-3-3: 1995
- ASTM D7891

Certificates of conformity available on request.

**PERFORMANCE:**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Force</td>
<td>+/- 50N maximum</td>
</tr>
<tr>
<td>Torque</td>
<td>0.0001N resolution +/- 900mNm maximum</td>
</tr>
<tr>
<td>Vertical travel</td>
<td>185mm</td>
</tr>
<tr>
<td>Rotor speed</td>
<td>120 rpm maximum</td>
</tr>
<tr>
<td>Axial speed</td>
<td>30 mm/sec maximum</td>
</tr>
<tr>
<td>Residual energy level in air</td>
<td>&lt; 2mJ</td>
</tr>
</tbody>
</table>

**COMPUTER SPECIFICATION:**

The instrument incorporates an integrated high specification processor and operates on a Microsoft Windows Embedded operating system. It has built in networking capability and a universal serial bus to provide serial daisy chaining of all automated accessories.

**CONSTRUCTION:**

Working zone: 316 stainless steel
Contact parts: 316 stainless steel Borosilicate glass Delrin and Peek plastics

**DIMENSIONS:**

Main instrument 306 x 306 x 760mm high

**WEIGHTS:**

Main instrument 22kg net

**POWER REQUIREMENTS:**

Supply voltage range: 90 to 264VAC
Input current range: 1.6A at 120VAC 0.8A at 230VAC
Input frequency range: 47Hz to 63Hz

**ENVIRONMENTAL CONDITIONS:**

Humidity range 20-80% non-condensing
Temperature range (operating) 10°C to 40°C
Temperature range (storage) 0°C to 50°C

**VESSELS:**

Precision bore, borosilicate glass tube.
Standard sizes:
- 25mm x 10ml Split Vessel
- 25mm x 25ml Split Vessel
- 25mm x 35ml Vessel
- 50mm x 85ml Split Vessel
- 50mm x 160ml Split Vessel
- 50mm x 260ml Vessel
- 62mm x 137ml Split Vessel
- 62mm x 240ml Split Vessel
- 62mm x 400ml Vessel

**BLADES:**

Hardened stainless steel.
Standard sizes:
- 23.5mm diameter x 6mm wide
- 48.0mm diameter x 10mm wide
- 60.0mm diameter x 10mm wide

**CALIBRATION KIT:**

Force, torque, height, carriage velocity and spindle speed are configured for calibration.

Calibration fixtures, weights and height gauges are supplied as part of the calibration kit.

A calibration log is automatically kept of the current and all previous calibrations.

**ACCESSORIES:**

25mm Accessories Kit
50mm Accessories Kit
62mm Accessories Kit
Aeration Control Kit
24mm Shear Cell
48mm Shear Cell
24mm Wall Friction Kit
48mm Wall Friction Kit
1ml Shear Cell

**SOFTWARE:**

All Control and Data Acquisition Software is supplied and configured with the instrument and includes Microsoft Office.
Measuring and understanding the flow properties of powders with the FT4 Powder Rheometer®