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

freemantechnology

ASTM D7891 compliant

A universal powder tester

The FT4 Powder Rheometer was originally designed to characterise the rheology, or flow properties, of powders. This remains a primary function today, but the instrument, accessories and methodologies have been continuously developed to the point where the FT4 is now considered a universal powder tester.

In addition to the patented dynamic methodology, where a powder's resistance to flow is measured whilst the powder is in motion, the FT4 also includes a shear cell for measuring the powder's shear strength, a wall friction kit in order to quantify how the powder shears against the wall of the process equipment (in accordance with ASTM standard D7891), and also accessories for measuring bulk properties, such as density, compressibility and permeability. This range of measurement capabilities makes the FT4 a truly universal powder tester and by far the world's most versatile instrument for measuring and understanding powder behaviour.

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 Helix Angle and Tip Speed.

An introduction to powders

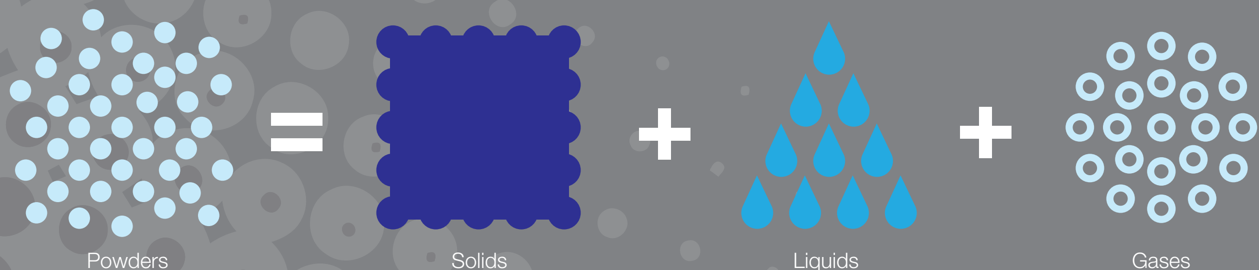
Powders are complex materials. Often perceived as just a collection of particles, they are in fact a complex mixture of solids, liquids and gases. Unlike the relatively well understood constituent phases from which they are derived, powders are more complicated materials. They are comprised of: solids in the form of particles; gas, usually in the form of air between the particles; and water, either on the surface of the particle or within its structure. The behavioural characteristics of these unique materials 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 final application. These characteristics are often independent of one another, so it is important to understand and to be able to measure the influence of each, if a powder is going to be fully characterised.

Powders exhibit the following behavioural characteristics:

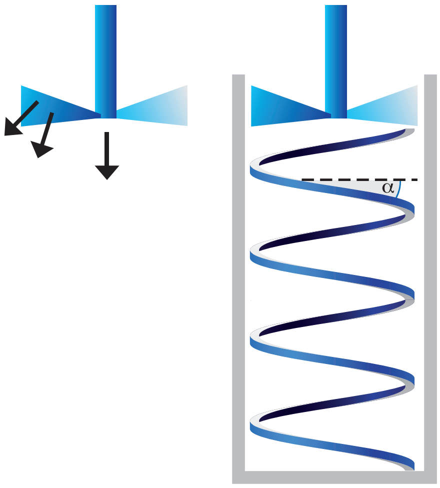
- Flowability – some may flow well through a process, others may bridge, block or flow intermittently.
- Compressibility – some are very stiff, others observe a large change in density when consolidated.
- Adhesivity - powders may stick to process equipment, others slide easily.
- Permeability – the ease with which air can be transmitted between particles can be critical during processing and in some final product applications.
- Electrostatic charge – some powders become electrostatically charged as a result of handling and processing, resulting in a change in their behaviour.
- Hydrophobicity – most powders experience a change in behaviour if humidity or water content increases, but to varying extents.
- Particle attrition – if particles are friable or weak, then mechanical stress can result in a change in size and shape, resulting in changes in powder behaviour.
- Flow rate – powders will behave differently across a range of flow (or shear) rates, influencing operations such as mixing and blending.
- Flooding – certain powders may fluidise and then retain air, resulting in fluid-like behaviour and often poor in-process control.

These examples represent just a few of the ways in which powders can demonstrate their complex 'personalities'. Moreover, these properties are frequently independent and unrelated, such that two powders can be equally compressible, but exhibit very different flowability. Other powders could flow the same, but have different permeability.

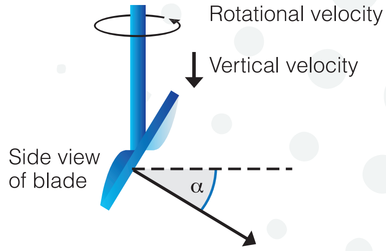


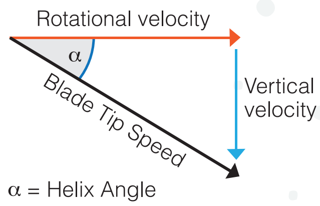
The way these phases interact determines the behaviour of the powder.

They can:
- *Behave like a single solid entity, deforming elastically and plastically*
- *Flow like liquids, when aerated / fluidised*
- *Be compressed like gases*



The blade is rotated and moves vertically, as defined by the Helix Angle and blade Tip Speed





Powder conditioning
Powders have memory, in that their behaviour and flowability will be influenced by their previous packing state. If a powder has been consolidated, a proportion of this stress will be retained after the consolidating load has been removed. Conversely, if the powder has previously been aerated, then excess air may exist within the powder. In both cases, the flow properties of the powder will be significantly influenced by the previous packing state.

This variation of stress or aeration occurs as a result of processing and handling the powder, but also during the preparation step in any measurement system. In order to address this variability, which will influence the accuracy of the measured result, the FT4 employs a unique conditioning process that prepares the sample in a homogeneous way, creating uniform low stress packing throughout the powder sample and removing any stress history or excess air prior to the measurement.

This automatic conditioning step is run before every test and is paramount if excellent repeatability is to be achieved. Conditioning reduces operator to operator variability and ensures that results generated can be accurately reproduced by a different operator, or on another instrument in a different lab.

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!

Understanding powder behaviour

The particle physical and chemical properties determine the behaviour of the powder to a large extent, but so too does the environment in which the powder is being handled (external variables).

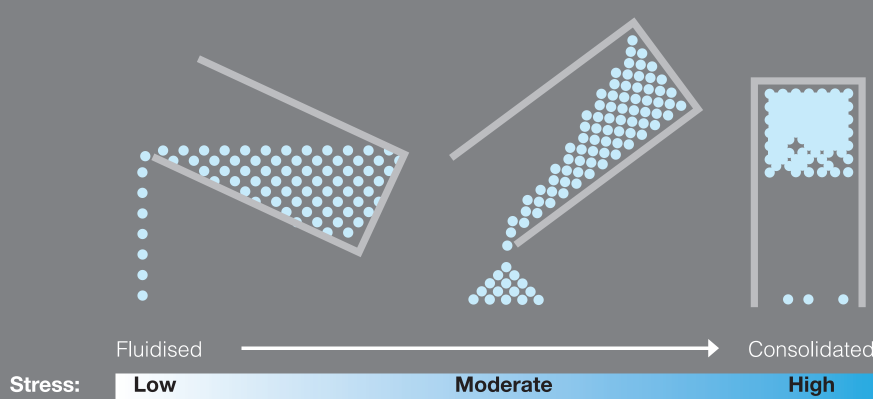
Particle properties
The particles alone are complex and rarely defined by an adequate set of descriptors. Particle size distribution has traditionally been considered, and it remains important, but in fact there are many particle properties that will influence the overall behaviour of the powder, namely:

- Particle size and distribution
- Shape
- Surface texture
- Surface area
- Density
- Cohesivity
- Adhesivity
- Elasticity
- Plasticity
- Porosity
- Potential for charge
- Hardness / Friability
- Hygroscopicity
- Amorphous content

Some of these properties can be measured directly, whilst others are more challenging. All will contribute to the way the powder behaves.

External variables
A further complicating factor is that the behaviour of the powder will depend on the environmental conditions to which it is exposed. If consolidated, its properties will be very different to if it is loosely packed or even aerated.

In each of these images, the physical and chemical properties of the particles are the same, but the way the powder flows is very different, simply as a result of changing the air content and contact stresses between the particles.



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.

Torque and Force are measured simultaneously as the blade moves down a helical path through the powder

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.

External variable	When and where	Effect
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?

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.

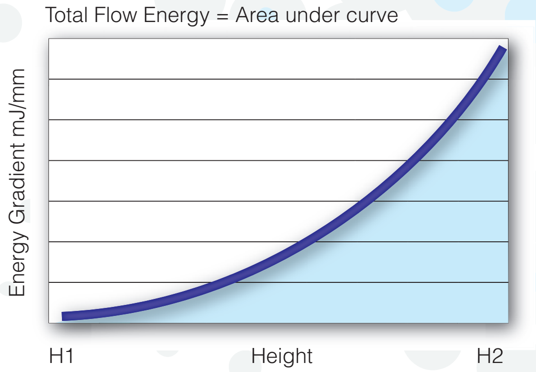
Using the calculation of Work Done, it is possible to represent both resistances as a total energy, the energy required to move the blade through the powder from the top to the bottom of the powder column. However, as the blade travels through the powder the values of torque and force are constantly 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.



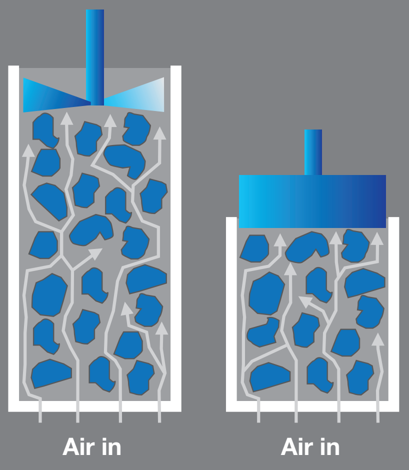
Energy Gradient is calculated directly from the measurements of Torque and Force

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.

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.

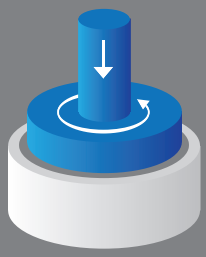


Aeration test *Permeability test*

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.

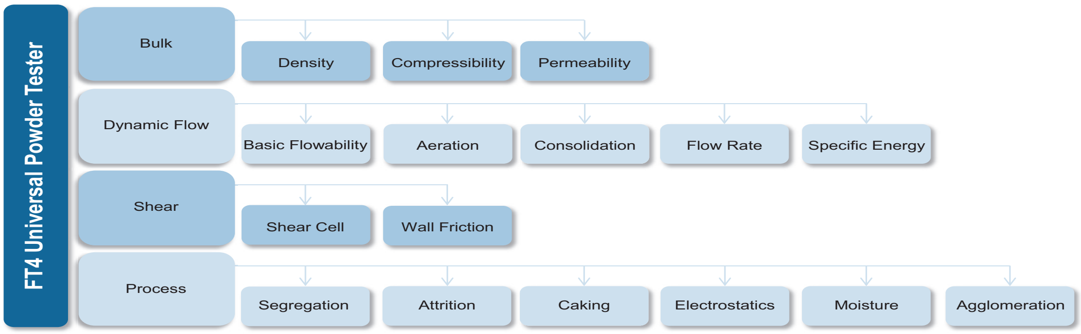
Axial compression
A 'vented piston' can be applied to the top of the powder column in order to consolidate the powder under a controlled and precise normal stress.

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.



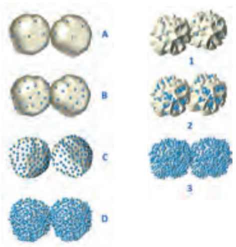
Methodologies

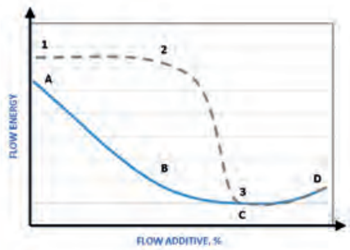
The FT4 Powder Rheometer is a truly universal powder tester, with four categories of methodologies, defined as Bulk, Dynamic Flow, Shear, and Process.



Basic flowability – *measuring the dynamic flow properties of powders in a conditioned state.*

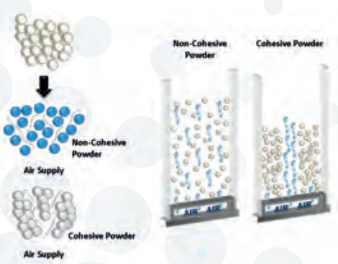
- Flow additives
- Wet granulation end point
- Moisture content
- Attrition / Segregation
- Physical properties (particle size, shape, surface texture, etc)
- Electostatic charging

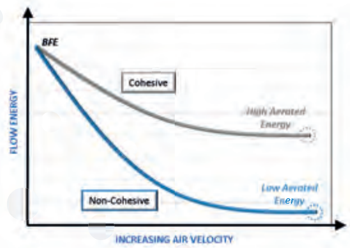




Aeration – *a direct measurement of a powder's cohesive strength.*

- Low stress, gravitationally induced flow
- Dosing / Mass uniformity
- Aerosolisation / DPI
- Fluidisation behaviour
- Blending / Mixing
- Segregation potential



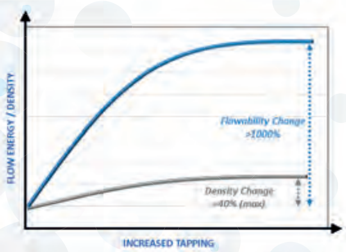


Consolidation – *understanding the effect of direct consolidation or vibration on powder flow properties.*

- Direct pressure
- Tapped

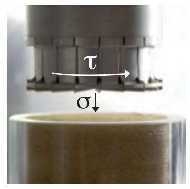
Understanding the effects of:

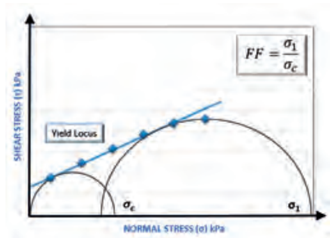
- Transport
- Storage
- Processing
- Caking



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.*

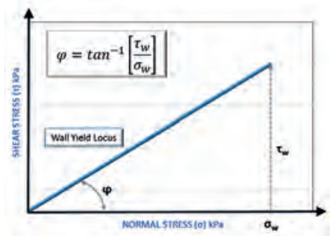
- Unconfined Yield Strength
- Flow function
- Cohesion
- Angle of Internal Friction





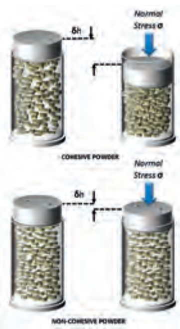
Wall Friction - *measuring the friction acting between a powder and equipment surfaces in accordance with ASTM standard D7891. Also required for hopper design.*

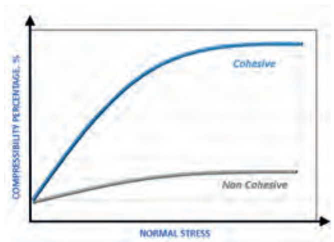
- Measure friction between powder and surface material
  - Hopper, IBC, Punch, Die wall
- Wall friction angle for hopper design



Compressibility – *determining changes in a powder's density as a result of a directly applied consolidating load.*

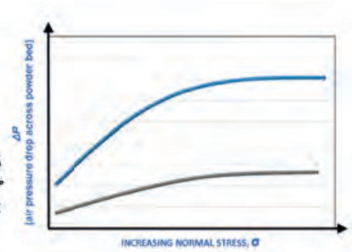
- Transportation
- Storage
  - Hoppers
  - Kegs
- Processing
  - Tablet compression
  - Roller compaction
  - Screw feeding
  - Extrusion





Permeability – *measuring the resistance to air flow between particles and through the powder bed.*

- Tablet capping / Lamination
- Aerosolisation / DPI
- Hopper flow
- Compression
- Pneumatic transfer



Proven applications

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

Applications extend to:

- Filling
- Tablet compression
- Hopper flow
- Wet granulation end point and scale up
- Flow additive selection and 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 in a development 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 CFR21 Part 11 guidelines. The Powder Rheometer control software is intuitive and easy to use, guiding the operator through a wizard style interface to ensure sample analysis is uncomplicated. The Data Analysis package comes with a site licence allowing data interpretation and reporting to be carried out away from the lab, and by any number of onsite users. A further application in the form of Support Documents provides on-instrument, comprehensive support on all methodologies, data interpretation, calibration and additional help that may be required.

A full range of accessories is available for the FT4, including standard vessel sizes allowing sample volumes in the range 10ml to 160ml to be analysed. In addition, the 1ml Shear Cell can be selected when only very limited samples sizes are available. Further accessories include compaction pistons, shear heads, wall friction kits, an aeration control unit and a uniaxial tester. A calibration standard powder is also available, if required. For a full list of available accessories, please contact Freeman Technology or your local representative.

Freeman Technology has over 15 years' experience in the design of powder characterisation instrumentation and in powder processing applications. So working with Freeman Technology means more than simply purchasing an instrument. Thanks to our expertise and know-how, we provide FT4 Powder Rheometer customers around the world with extensive and 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
- EN61326: 1997 + A2:2001
- ASTM D7891

Certificates of conformity available on request.

PERFORMANCE: -

Force	+/- 50N maximum 0.0001N resolution
Torque	+/- 900mNm maximum 0.02mNm resolution
Vertical travel	185mm
Rotor speed	120 rpm maximum
Axial speed	30 mm/sec maximum
Residual energy level in air	< 2mJ

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 freemantechnology

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