

# acorn area™



## Particle Surface Area

**xigo**™  
nanotools



## Introducing the Acorn Area™

The Acorn Area™ is a revolutionary instrument designed to measure the surface area of nanoparticles dispersed in a liquid. This patented nuclear magnetic resonance based method offers many advantages in comparison with conventional surface area techniques.

Measurements take about 5 minutes from start to finish. No sample preparation such as drying or degassing is required. High concentration dispersions are measured directly; without dilution. Acorn Area measurements are orders of magnitude faster than any other surface area measuring technique.

## Why Surface Area

The surface area of particles influences many aspects of product performance such as the hiding power of pigments, the activity of catalysts, the taste of food, the potency of drugs and the bioavailability of drugs. In the past, surface area measurements were made using methods such as gas adsorption, mercury porosimetry, and gravimetric analysis. These methods all have the same limitation; they are only useful for analyzing dry powders.

However, the overwhelming majority of manufactured products involve dispersions of particles in a liquid, either in the final state or at some stage of production. While dry powder methods are suitable to test incoming dry powder raw materials, they cannot provide information about the particles when dispersed in a liquid. To date this has not been possible other than to estimate the surface area from particle size measurements or to perform time-consuming adsorption isotherm or titration measurements.

## How it works

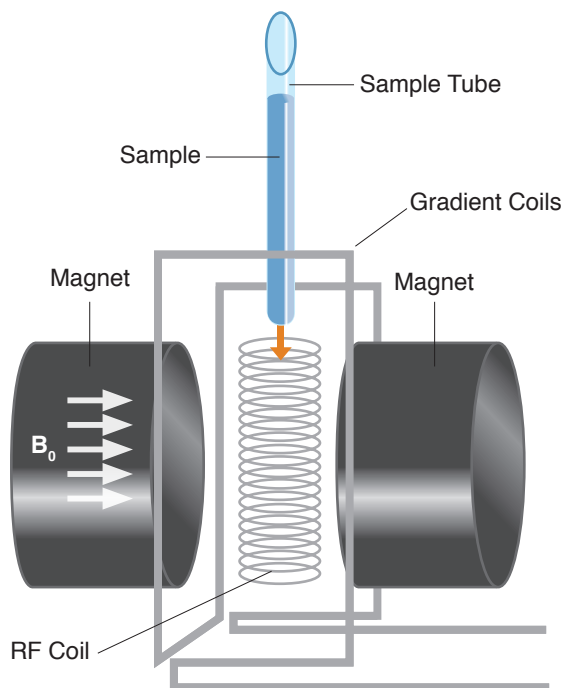
Liquid in contact with the particle surface behaves differently from that of the bulk, or “free” liquid in response to a changing magnetic field. The NMR relaxation time of liquid on the particle surface is much shorter than that of the bulk liquid. This difference can be several orders of magnitude. The relaxation time of particles dispersed in a liquid is an average of the two relaxation times, weighted by the relative amount of liquid on the particle surface and that in the free liquid. This is a direct measure of the particle surface area.

To make a measurement, place a 0.5 ml sample in a sample tube and insert it into the Acorn Area™. The sample resides within a coil located between two permanent magnets. A static, uniform magnetic field,  $B_0$ , causes the protons within the liquid to align with  $B_0$ ; this process typically takes a few seconds.

At the start of the measurement, a short radio frequency (RF) pulse excites the coil at a frequency of approximately 14 MHz. This pulse produces a large magnetic field, inducing a temporary shift in the magnetic orientation of the sample protons. When this induced field ceases, the protons of the sample fluid then realign with the static field,  $B_0$ . This realignment induces a decaying voltage in the coil, called the “free induction decay” (FID).

By using specific pulse sequences, (combinations of RF pulses of varying length and time between pulses) the sample T1 (longitudinal) and T2 (transverse) relaxation times are measured.

Although T1 and T2 are different, their shift between liquid in the bulk and liquid on the surface is similar, and both can be used to determine the surface area.





## Exclusive Software

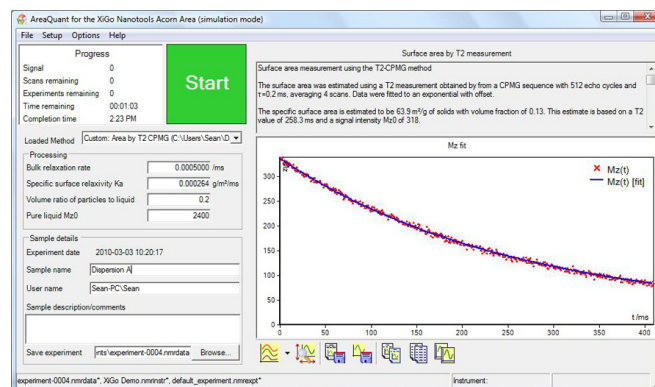
AreaQuant™, the Acorn Area software, measures T1 using an inversion recovery pulse sequence, and measures T2 using the CPMG pulse sequencing method. The resulting data are analyzed to determine the relative proportion of bound and free liquid and the corresponding particle surface area.

There are no assumptions about the sample particle size (distribution) or shape used to determine surface area; the wetted area is measured directly. The user inputs the particle dispersion weight concentration, the liquid and particle density. The particle-to-liquid volume ratio (used in the calculation of surface area) is automatically determined.

There are two Operational Modes in the software setup: Standard (or QC) Mode and Advanced (or R&D) Mode. The former is the mode used to measure surface area. In R&D mode, the instrument behaves like a traditional NMR spectrometer capable of a wide range of low-resolution NMR measurements.

Measurements with the Acorn Area are simple and easy. Dispersions can be measured non-invasively, without dilution. The upper limit in concentration is essentially unlimited; the lower limit is about 1-2%.

Using this automatic mode, AreaQuant then determines all the measuring



parameters needed such as the amplitude and duration of the pulses, the number of repetitions, etc. without operator intervention. This ensures measurements are made with the highest resolution and repeatability without a great deal of operator training.

To simplify method development, the software also features an automatic test parameter file configuration based on an estimated T1 or T2 time input by the user.

Once a set of parameters are defined, operation of the Acorn Area is simple: just enter the operator name and sample information - the software does the rest!

AreaQuant™ operates on virtually all Windows Platforms, including XP®, Vista®, and Windows® 7. Data files are saved in XML format, easily opened with Microsoft Excel or Open Office. Reports are saved in RTF format. Graphs may be copied to the clipboard or saved in JPG or PNG formats. To make sure you are using the most up to date software available, updates may be downloaded and installed via the Internet with a single click of the mouse.

# Applications

## Pharmaceuticals

The wetted area of the active pharmaceutical ingredient (API) available to react with stomach acids controls the rate that the product will relieve acid indigestion. Two formulations may have the same weight percentage of API but may provide a different therapeutic benefit owing to a difference in the API surface area. This relates to the concept of bioequivalence of drugs, an important issue in the debate between brand and generic pharmaceutical companies.

## Cosmetics

Titanium dioxide is an important ingredient in many sunscreen products. UV radiation attenuation is directly related to the surface area of the  $\text{TiO}_2$  particles. Samples having the same particle equivalent spherical diameter may have dissimilar shape and morphology, resulting in vastly different levels of UV protection. Such differences are detectable via surface area measurement of the wet suspensions. Small variations in dispersion particle aggregation are easily detected in surface area measurements. Thus, the Acorn Area can be used to monitor the quality of the dispersion.

## Electronics

The rate at which a battery produces energy is related to the total amount of surface wetted by the reactants in the paste. Any surface not wetted will not contribute to energy generation.

As energy storage becomes more and more important, the relationship between dispersion wetted surface area and product performance becomes critical.



## Industry standards

### Comparison with Surface Area using the BET

Particle size measurements from different techniques do not always agree. Each technique provides a different sensitivity to the various size fractions within a particulate suspension. In addition, the various techniques use different methods to convert the raw data, i.e., diffracted light, light fluctuations, acoustic attenuation, etc., into a particle size.

A similar situation exists when comparing surface area measured using the Acorn Area and gas adsorption (BET). The Acorn Area measures the total wetted surface area of the particles in a liquid, while the BET technique measures the amount of gas adsorbed on the surface of dry particles. If the wetted area and the area available for gas adsorption are the same, then the two techniques should produce the same number. In practice, however, differences in sample preparation between wet and dry states may yield samples in which the wetted area and the dry area are dissimilar, resulting in disparate values for the measured surface area.

### Validation of the Acorn Area Technique

Klebesol 30HB 25K (Rohm & Haas) is a nanoparticulate silica slurry used in electronics as a polishing compound for silicon wafers. These silica particles are spherical; the dispersion is highly monodisperse and is delivered as a 30% by weight slurry.

The surface area of these particles reported by the manufacturer (measured by an unspecified titration method) was  $120\text{m}^2/\text{g}$ . Estimation of the surface area based on a particle diameter determined from dynamic light scattering was  $123\text{ m}^2/\text{g}$ . Direct measurement by the Acorn Area, on the 30% by weight slurry, yielded  $121\text{ m}^2/\text{g}$ .

Measurements with the Acorn Area on this concentrated dispersion took less than 5 minutes. In contrast, gas adsorption would have required separation of the particles from the liquid by drying, then degassing the surface - a process that can take several hours. Such sample preparation can cause particle aggregation, leading to an inaccurate determination of surface area. The titration method is non-routine, requires sensitive instrumentation and skilled operators. Calculation from particle size is only possible because the material is spherical and monodisperse. Measurement by dynamic light scattering also requires substantial dilution of the dispersion. This is not always a straightforward exercise and can lead to particle aggregation.



## Support

The Acorn Area has no moving parts and is designed using state-of-the-art microprocessor technology; system failures are highly unlikely. However, should it be necessary to service your unit we will Priority ship overnight any replacement parts to keep down-time to an absolute minimum. All parts are user replaceable.

Our service technicians can operate the Acorn Area remotely through a web session, allowing us to diagnose the problem, and in some cases, fix the problem remotely.

### The Acorn Area Advantage:

- **Low cost**
- **Measurements without dilution**
- **No sample preparation**
- **Results in minutes**
- **Compact**
- **No moving parts**
- **Suitable for QC or R&D**
- **Simple to use**

### Specifications:

**Interface:** PC or PDA via USB

**Power:** 100-240 VAC 50/60 Hz, 3 amps

**L x W x H:** 240 x 360 x 160 mm

**Wt.:** 7 kg

## Contact us!

Is the Acorn Area suitable for your application? Call to discuss it with our technical staff. Send us a sample. We will be happy to make a measurement. If you would like a demonstration, we can arrange to visit your facility or demonstrate the Acorn Area via the Internet. See for yourself the incredible cost-performance benefits that the Acorn Area provides.

For more information, please contact:

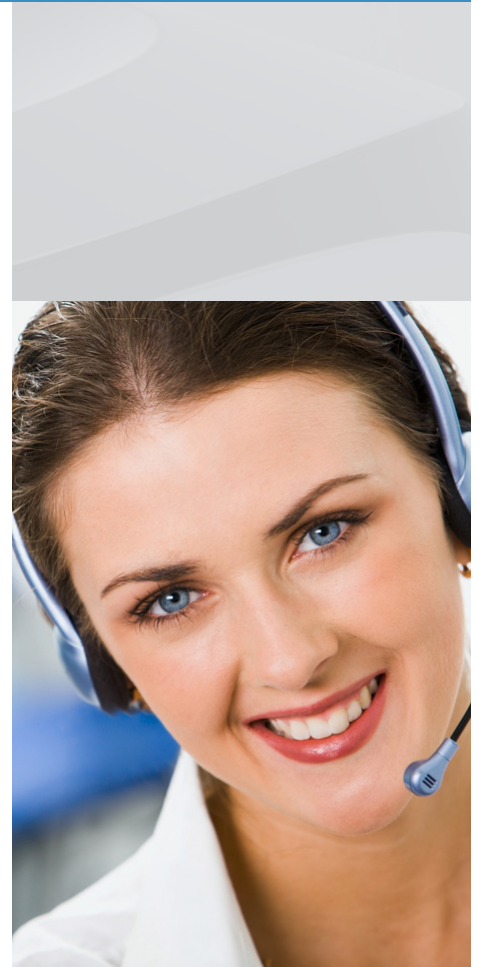
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