

Article

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Photographs: BI-200SM dynamic and static light scattering goniometer from Brookhaven Instruments

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Use of Brookhaven's BI-200SM dynamic and static light scattering goniometer and BI-9000AT correlator as a photon correlation spectroscopy system has helped the Bristol Colloid Centre, a commercial enterprise founded by the University of Bristol, probe the properties of a variety of complex colloidal systems. The research has important consequences for industries ranging from petrochemicals to pharmaceuticals and personal care products.

The world of microscopic particles is complex and difficult to probe. One of Bristol Colloid Centre's main concerns is the study of minute particles suspended in liquids. The team there has found that its Brookhaven photon correlation spectroscopy equipment — the BI-200SM goniometer and the BI-9000AT correlator — has helped to expand its capabilities, earning it valuable research contracts and helping to push back the boundaries of this area of work.

The BCC is part of the School of Chemistry's colloid and condensed phase sector. Recognised as a world centre of excellence in its field, the University's colloid researchers focus on areas such as the properties of colloidal dispersions, including those which conduct electricity and are important to the environment, for example interactions within colloids and concentrated colloids.

The company carries out contract research work and offers industrial training courses and specialist consultancy to clients in a wide range of markets, including agrochemical, cosmetics, food, paints and coatings, personal health care, petrochemical and pharmaceutical industries. Much of this work, carried out by its permanent staff researchers and PhD students, is on the cutting edge of the client companies' product development, and is therefore covered by strict commercial confidentiality.

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BCC research

The centre's current research is just as diverse as the sectors it serves. Current projects include an investigation into how colloidal semiconductor crystals grow and develop in complex surfactant media, which could have great importance in the design of inks and other coloured compounds. A related project is looking at the development of extremely tough inks, capable of resisting heat, solvents, corrosion and abrasion and which can be used for printing on glass, ceramics and metal surfaces. Surfactants are another interest, from the development of new surfactant molecules to the design of new shampoos that enhance the shininess of hair.

Contract research was the driving force for BCC to buy the Brookhaven instruments. Its laboratories already had equipment for light and X-ray scattering techniques, pulsed-field-gradient NMR, advanced electrokinetic methods, rheological techniques, advanced electron- and optical-microscopy with image-analysis and calorimetry. However, in the autumn of 1996, a demanding project on a series of microemulsions required the use of a high-performance photon correlation spectroscopy instrument. The conventional use of this equipment is to size particles, but the team needed to analyse the properties of microemulsions, which contain very, very small droplets of dispersed material and study their structure under different conditions.

"There are generally two goals for this," explained Dr Cheryl Flynn, one of the Centre's research scientists, "optimising formulations and characterising the microstructure of the colloid. Microemulsions contain very small species, and it's for that reason the performance of the correlator at high speeds was important," she said.

Beating the competition

BCC assessed several competitive instruments before settling on Brookhaven. The system was bought after submitting samples for competitive tender. "We sent three candidates' samples of microemulsions which were, as far as we could establish, typical of the type of system which we wanted to study and compared the data," said Dr Flynn.

Brookhaven won through because of the performance of its machines. Dr Flynn continued: "This system complements our other photon correlation spectroscopy (PCS) and angular light scattering techniques. It is a more powerful system than other PCS instruments available, in terms of laser power and correlation."

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The two instruments form a powerful and versatile suite for studying particulate systems. The BI-200SM goniometer, whose flexible design allows any laser to be used, is generally used for particle size analysis, the investigation of the structure of complex fluids, micelles, microemulsions and colloids. It allows the use of multi-angle measurements yielding more information on particles and molecules than a single-angle system. Despite this, its simple alignment is easy to learn, adjust and maintain.

The BI-9000AT correlator is equally versatile, allowing sampling times as low as 25ns and as high as 40ms, with delays ranging from 25ns to 1310s. It can process two different signals simultaneously, for example to cross-correlate two signals from different positions in a liquid or from two detector angles.

PCS analyses light scattered by the particles of an emulsion or colloid within the liquid in which they are dispersed (the continuous phase). The colloid scatters the light randomly, producing a 'noisy' signal at the spectrometer's light-sensitive detector. However, the particles are moving constantly, both because of the energy from the laser and because of Brownian motion. The intensity of the signal at the detector varies over time and the frequency of this variation is related to the size of the particles.

Stretching the capabilities

The Bristol team's research stretches the capabilities of the Brookhaven machines. "Since these instruments arrived at the centre, they have been used extensively for a wide variety of specialised research applications. They have proved particularly useful with 'difficult systems' such as small silica particles and microemulsions," said Dr Flynn. "We have used them to study both oil-continuous and water-continuous microemulsions, both in their 'native' state and when swollen with active ingredients. There is also an ongoing study of bicontinuous microemulsion phases."

Further applications have included the study of several other types of colloid under investigation at the centre; polymeric molecular species, biological complexes and inks are just three examples.

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Dr Flynn continued: "We use the system to operate in a multi-angle mode, both by analysing the correlation data and the total intensity or count rate. We have also carried out correlation and total intensity measurements simultaneously, to enable us to get a more detailed characterisation of the material under investigation. Before they bought the Brookhaven equipment, the Centre had to perform two complete series of measurements on two separate instruments to offer this type of analysis.

She concluded: "Good after-sales service is always an important consideration for us when choosing equipment. We have no major complaints about the service we have had from Brookhaven and would certainly consider it the next time we need to update instrumentation."

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Photon Correlation Spectroscopy (PCS)

The sample to be analysed, usually a colloidal dispersion, is placed in the cell of the BI-200SM goniometer where a beam of monochromatic light from a laser passes through it. Some of the incident light is scattered in all directions by the particles or droplets within the sample. In a colloidal dispersion, the particles continuously collide with the molecules of the liquid phase, causing random thermal motion called Brownian motion. This motion causes the intensity of the scattered light at any one point in space to fluctuate with time. These intensity fluctuations are caused by constructive and destructive interference of light scattered by neighbouring particles within the illuminated zone.

The time dependence of this intensity fluctuation is analysed using the BI-9000AT digital correlator, which stores the scattered intensity digitally as the number of photons in each sampling interval which can range in duration from 25 nanoseconds to 40 milliseconds. These photon measurements are used to determine the intensity autocorrelation function (ACF). Analysis of the ACF leads to the diffusion coefficient of the particles from which, via the Stokes Einstein equation and knowing the viscosity of the sample medium, the size of the particles themselves can be calculated. The ACF, initially high, diminishes to zero over the analysis period, and the exponential decay pattern of this correlation is characteristic of the diffusion coefficient of the particles. Data are usually collected over a period which can range from nanoseconds to several seconds, depending on the particle size and viscosity of the medium.

The BI-9000AT correlator software will then use one of several different approaches to numerically fit the data with calculated size distributions. A truly monodisperse sample would give rise to a single exponential decay, plotted against time, which would make calculation of the particle size distribution very straightforward. Real life, polydisperse, samples give a series of exponentials plotted against time, which require more complex calculations by the software to yield the particle size distributions.

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