

Introduction

S700X - For better magnetic measurements

- Cryogen free or Liquid Helium based system
- High homogeneity 7 Tesla magnet
- Low vibration & low maintenance Pulse Tube cryocooler
- 10⁻³ EMU ultimate sensitivity
- MilliTesla field resolution and setting
- Continuous temperature range from 1.6 K to 300 K
- High temperature option to 700 K
- He-3 Insert for temperatures down to 0.3 K
- AC and DC measurements
- Transverse field
- Built-in environmental shield
- Auto de-gaussing facility
- Sample rotation facility
- Short sample scan facility
- Fast 16 bit data acquisition
- Flexible open LabVIEW software
- Real-time data access during measurement

The S700X SQUID Magnetometer is the most sensitive instrument for the measurement of magnetic properties as a function of magnetic field and temperature. Numerous different experiments may be performed with this unique instrument.

The state-of-the-art instrument is the product of more than 20 years development and is fully engineered to be robust and reliable. It is suited for both routine measurement by non-specialists and, in the right configuration, for the most advanced research on the magnetic properties of materials.

Cooling for the system is now available in either cryogen free or liquid helium based environments. Cryogenic has been the leading supplier of high field measurement systems without using liquid cryogens, and has now extended its cryogen free range to include the 7 Tesla SQUID Magnetometer. This gives great advantages to the user in terms of reduced running costs and ease of operation.

Great care has been taken to make the S700X as userfriendly as possible. The new LabVIEW software operates in an open environment that allows the user direct control of all parts of the system with real-time graphical displays of all the relevant functions. The transparent nature of the operating system greatly improves the user's understanding of the experimental set-up, as well as providing unparalleled control for the most demanding measurements.

The Superconducting Quantum Interference Device (SQUID) Magnetometer

The SQUID is the most sensitive detector of magnetic signals available, with an input noise power sensitivity of about 10⁻³⁰ Joules per root Hz. This value of energy sensitivity is 10⁸ better than any semiconductor device, such as a FET, and accounts for the instrument's greater sensitivity and its ability to resolve small magnetic signals quickly.

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The characteristic signal of a sample.

The S700X has several modes of operation. The most widely used is the measurement of total magnetic moment made by moving the sample through the pick-up coils. This method is known as the extraction method.

A second order pick-up coil is used in the S700X which is not sensitive to changes in the background field. The characteristic signal is shown in the image above.

For materials that show magnetic hysteresis it is important that the sample remains in a highly uniform field during the scan. The normal scan length is set to 40 mms, over which the field is uniform to 10^{-4} but measurements can be made with movements from 2 mm up to 130 mm.

Competitive Advantages of the Cryogenic SQUID Magnetometer

The following competitive features are provided in the basic system S700X system.

Cryogen Free Platform

The 7 Tesla Cryogenic SQUID Magnetometer (CF-S700X) can now operate without the use of liquid helium. It is offered with a Pulse Tube cryocooler which has the added benefit of low vibration and maintenance. The CF-S700X is an extremely compact and yet highly sophisticated system. It is extremely economical to run and is particularly useful for areas where liquid helium is a scarce resource.

The built-in Oscillating Mode Facility allows continuous measurements in a changing background field. The sample is oscillated vertically over a distance of few millimeters. The detected sinusoidal signal from the SQUID is proportional to the magnetic moment of the sample.

Fast Temperature Changes

The ultra light design of the sample chamber allows fast temperature changes and stabilizes quickly, because there is hardly any heat capacity to overcome other than that from the sample holder and the sample.

Any temperature within the wide range of 1.6 K to 300 K can be kept continuously.

Magnetic Shielding

Based on the experience in the field of Rock Magnetometers, the S700's shield system consists of a mumetal can and a niobium shield, which reduces the background field to as little as 0.5 mG. The system is fully shielded against external noise and changes in the background field.

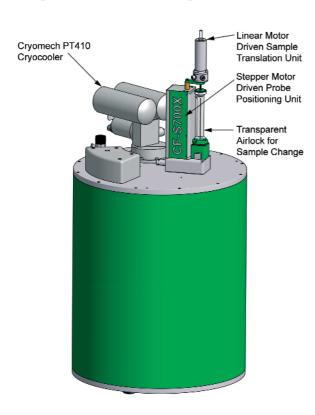
User Friendly

The data gained during a measurement are analyzed in terms of magnetic multipoles. This unique feature gives a dipole moment independent of the sample shape and is error tolerant with respect to sample position, a feature which allows inexperienced users to perform reliable measurements.



The 7 Tesla 'Croygen Free' SQUID

Magnetometer system incorporates the cryogen free, special high field superconducting magnet to 7 Tesla, the integrated variable temperature sample space of 9 mm diameter (CFVTI), cryocooler and compressor, multi-channel temperature monitor, sample temperature controller and power supply. The cooling for the magnet and sample is provided by a single cryocooler with a base temperature of less than 4 K and providing typically 1.0 Watts of cooling power at 4 K on the second stage. The current to the magnet is supplied by Cryogenic's specially designed power supply using current leads of HTS conductor. A Lakeshore model 340temperature controller controls the temperature of the sample.



Samples are top-loaded into the sample access column via an airlock and gate valve located at the top of the VTI to allow access to the centre of the temperature-controlled region. Experienced operators can typically change samples in a few minutes.

Measurements are made by the VSM method. A miniature subtle movement system vibrates the sample at typically 20 Hz by 2 mm. The signal from the moving sample is detected and measured by the SQUID. The sample displacement is also measured and controlled so that an accurate measurement of the true magnetic moment is obtained. In addition the sample can be positioned and moved over a 150 mm range with the stepper motor driven sample platform of similar design to that used on the other S700X SQUID systems.

A helium gas reservoir (50 litre NTP capacity) is supplied filled with helium gas at one atmosphere. This gas is used for the VTI. The gas used has 99.995% purity and in the event of accidental contamination, when changing samples for instance, the gas can be replaced at low cost.

No external liquid cryogens are required to cool the sample platform and make highly sensitive magnetic measurements between 1.6 K and 300 K and over a wide range of magnetic fields.

The CF-S700X is delivered as a turnkey working system tested and ready to operate on delivery.

Benefits of Cryogen Free Systems

- Long service intervals: The pulse tube cooler has long service inervals with low servicing costs.
- · Low operating costs: No liquid helium or nitrogen required for cool down or operation; there are no costs associated with storage, transport of liquids. Safety issues and training for personnel are minimised.
- Simple to use. No experience of cryogens is needed. Switch on the cooler and wait for the system to reach operating temperature in about 24 hours.
- · Compact. The absence of liquid helium and nitrogen reservoirs means that cryogen-free systems are generally more compact with easy access to the field centre.

The S700X SQUID

magnetometer consists of two main elements. The electronics and control system and the cryogenic unit described below.

The main cryogenic element consists of a variable temperature sample space insert upon which is mounted the superconducting magnet with the SQUID and magnetic detection coils.

At the top of the insert there is the sample movement system, an airlock to facilitate changing the sample and all the electrical feed-throughs for the magnetometer. The sample is mounted on a long rod with low magnetic moment which passes through a helium tight sliding seal into the sample space. Vertical translation and rotation of the sample are performed by stepper motors.

Temperature control of the sample is achieved by drawing a stream of helium gas past the sample. Liquid helium is drawn from the main helium reservoir in the cryostat and after expansion through an impedance, the gas passes through a heat exchanger which allows continuous variation of its temperature over the range of 1.6 K to 300 K. Control of the gas and sample temperature is achieved by an advanced electronic controller which measures the temperature of the gas stream to a resolution of 1 mK over the full range.

The sample space is sealed at the top with a gate valve and airlock so that samples can be changed while the system is cold without contamination of the cold space. The airlock is made of clear transparent plastic, so that the condition and position of the sample can be checked during the loading procedure just prior to lowering the sample to the measurement position.

The major components of the system are machined from solid stainless steel, which gives the S600X its excellent immunity to vibration and RF interference. The cryostat has a liquid nitrogen cooled radiation shield to provide a very low liquid helium consumption.





The Electronics and Control System

A single rack contains all the electronics, including the Intel based computer, the pump and valves for controlling the flow of helium gas.

By incorporating all control systems into a single rack it is possible to fully integrate the design and eliminate ground loops which could disturb the system performance.

The major electronic components are all standard equipment, making service and support easier. They include the Lakeshore temperature controller, magnet power source, and the SQUID electronics. The data acquisition and control is provided by National Instrument cards which are LabVIEW compatible. Special signal conditioning and isolating circuits are used to interface between the digital cards and the more sensitive elements of the instrument.

To supplement computer control of the system the main electronic instruments have front panel indicators and controls. These allow the operator to make independent confirmation of their correct function.

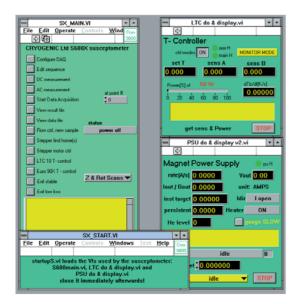
Environmental Shielding

The cryostat is fully shielded against the earth's background field and against locally generated magnetic and RF signals. An outer magnetic shield of mumetal reduces the background field in the sample space to about 0.5mG. Inside the cryostat a shield of superconducting material is used to further isolate the experimental space from external sources of magnetic interference.

The shielding factor from DC to a few kHz is 10⁸, more than sufficient to protect the measurements in all normal laboratory environments. The internal superconducting shield is placed outside the superconducting magnet so that the field does not disturb the shield, a great improvement over most other machines. The S700X has the dual advantage of the stability of a superconducting shield and the ability to make rapid measurements as a function of field.

The LabVIEW® **Operating System**

The S700X software runs under LabVIEW which is the most convenient and powerful software for instrument control, data acquisition and analysis. The software is easily understood and logically presented. Control is performed via virtual instruments with active click-on features, as well as pull-down menus. The menu structure greatly simplifies routine measurements of material properties, making the system very practical for non-specialists.



Our LabVIEW software operates in an open environment that allows the user direct control of all parts of the system with real-time data display. The transparency of the operating system greatly improves the user's control of the experimental procedure. It makes the instrument easy to control and operate as well as providing the sophisticated user with unparalleled performance and flexibility in use.

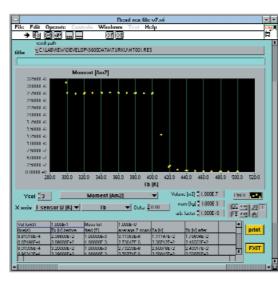
The opening display features virtual instruments for temperature and magnet field as well as a drop-down menu giving access to all the main system procedures. Measurement sequences can be created for immediate use or stored and retrieved for future use.

To enhance the performance, the operating procedures are fully configurable. For example, when changes in temperature are required it is possible to set broad limits of stability for fast measurements or fine limits where precise and accurate measurements are required.

A LabVIEW development licence is offered with each system to allow new measurement procedures to be developed as required.

Extended Temperature Range

For some material science applications it is useful to measure magnetic properties from very low temperature to above room temperature. An oven insert is offered which increases the standard temperature range. In normal operation, the standard system has a continuous temperature range of 1.6 K to 300 K. This can be increased using a heated sample stage to 450 K. For higher temperature, an oven must be used. The oven insert provides a temperature range of 200 K to 700 K.



Magnetic moment of a ferrite as a function of temperature.

Helium-3 Insert for temperatures down to 300 mK

The Cryogenic Helium-3 probe is designed as an alternative to the standard sample probe. The probe can extend the range of experimental temperatures accessible in the variable temperature sample space beyond the standard 1.6 K-300 K and can be used in either liquid helium cooled or cryogen-free systems. Using only the cooling power of the VTI and two internal temperaturecontrolled sorption pumps, the sample can be maintained at any temperature from below 300 mK to above 300 K.

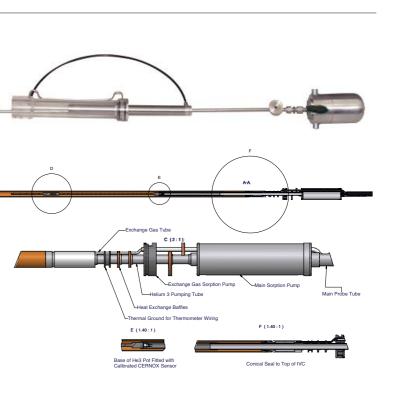
The miniaturised Helium-3 pot fits inside the 8 mm susceptometer VTI and a high-purity silver cold finger is used to position the sample in the magnet bore. A working volume of 0.9 cc of Helium-3 is sufficient to maintain temperatures down to 300 mK for up to 12 hours.

Resistivity Measurement Option

The Resistivity system is configured for resistance and Hall voltage measurements in the range microOhm to megaOhm. The resistance is measured by a high quality Keithley digital voltmeter and current source and extensions of the range of impedances are offered as required. The applied voltage can be dynamically reversed in order to subtract thermal potentials, and the measurement averaged. For each measurement the current is set at a low level and increased to provide an adequate measurement voltage. The maximum current, power or voltage on the sample can be preset to avoid sample heating and/or damage.



As each measurement is performed the magnetic field and temperature are varied according to a pre-arranged sequence. Different sequences can be stored and recalled by command, which generally simplifies operating procedures making the system easy to use without a detailed knowledge of the system software and hardware.



AC Susceptibility Option

An AC measurement option is available as a complement to the standard DC method to study the magnetic susceptibility of materials directly. We offer the facility to perform direct susceptibility measurements in AC magnetic fields from 10^{-2} to 500 Hz. The magnet is fitted with a separate coil for the AC field, providing up to 5 Gauss and driven by the main system electronics.

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The AC option window.

To make accurate measurements of the complex AC susceptibility of the sample, it is important to eliminate the instrument response time. The S700X software performs this function by moving the sample between the pick-up coils, making two measurements so that instrument errors are removed from the results. This feature increases the sensitivity and accuracy of both the in-phase and outof-phase response. It represents another example of the flexibility and sophistication of the S700X software.

Ultra Low Field Options

Many measurements are required in fields as close to zero field as practical. Since all superconducting magnets exhibit remanence, a small magnetic field will be left after an excursion to high field even when there is no current in the magnet. Use of a special degaussing program allows the remanent field to be reduced from its typical normal value of about 0.7 mT down to less than 0.1 mT. If the magnet has previously been to high field then the remanent magnetism can be removed either by heating the magnet or by going through a degaussing procedure.

For controlled measurements at the lowest fields it is convenient to apply a magnetic field with higher resolution. By adding a precision 4-quadrant low current source (500 mA) to the main power supply, very low fields in the region \pm -50 mT (500 G) can be obtained with a high degree of certainty and avoiding a significant remenant field in the magnet.

The low field option covers the range of ± 50 mT. It allows the user to set the field in this range with the precision of 1 micro Tesla (=0.01G). True Zero field (±0.001 G), assuming the system is shielded from the earth's field, can be achieved by warming the magnet above the critical temperature and subsequent cool-down. Zero field to within 1 Gauss can be achieved using the degauss procedure.

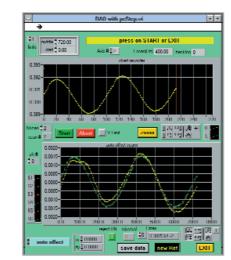
Vibrating Sample Measurement Options

Magnetic moment can be measured with a Vibrating Sample Magnetometer (VSM) which moves the sample between two pick-up coils at a frequency of 0.1 to 100Hz. Sensitivity is better than 10⁻⁸ emu with a suitable averaging time.

First order gradient with pick-up coil is used at the input to the SQUID together with a sample drive mechanism at the top of the cryostat. The VSM drive mechanism has been specifically developed by Cryogenic Ltd. The sample movement can be up to 10mm and it is measured using a moving coil detector.

The device and detection currents are built around the PAR phase sensitive lock-in amplifier.

Transverse Moment Facility with Sample Rotation



Some crystal structures exhibit anisotropic magnetic

to a magnetic moment perpendicular to the vertical

amplified by a second SQUID circuit. The output is

anisotropic moment may be measured and studied.

applied magnetic field. This transverse moment is

characteristics. To allow these to be measured the S700X

can be fitted with a signal detection circuit, sensitive only

recorded as the sample is rotated around the vertical axis

by a computer controlled stepper motor. In this way the

Transverse moment window

The fluxgate measures and profiles fields up to 20 G with a resolution of 10^{-5} G. For some applications it is very important to verify the magnet field at very low applied fields. Additionally, the fluxgate can also profile the magnet along its entire length. This facility is recommended with the ultra low field option.

Hall Probe Field Measurement Facility

The Hall probe is offered to enable the magnetic field to be measured at higher fields. It allows fields from 0.1 G to 10⁵ G to be profiled and measured, complimenting the fluxgate. For high accuracy, the zero offset must be referenced.

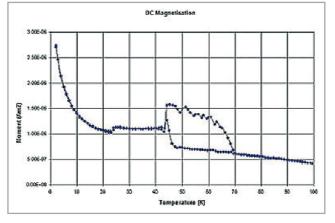
Dynamic Range Extender

The S700X is an exceptionally sensitive instrument with the result that strongly magnetised materials can only be studied if prepared in minute samples. For greater convenience Cryogenic offers a dynamic range extender which reduces the input signal by a factor of 500, allowing strongly magnetised materials to be measured conveniently in the same instrument as those of very low magnetic moment.. With this option the standard measurement range (10⁶ total dynamic range) from 10⁻¹¹ to 10⁻⁵ Am² can be extended to measure signals as large as $5 \ge 10^{-3}$ Am² (5 emu).

Fluxgate Field Measurement System

Special Measurement Options:

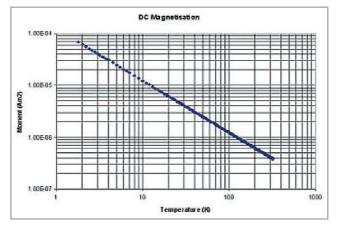
Cryogenic prides itself in being able to keep its clients at the forefront of research using the most advanced technology. As such we are always prepared to consider supplying other special options. Transverse magnetic field coils, optical and microwave illumination of the sample are some examples of the special options that can be provided. Further requests are always welcome.



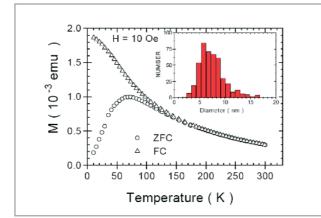
The measurement consists of two parts:

1. Measurement from 2 K to 100 K at 1 T after zero field cooling (lower curve)

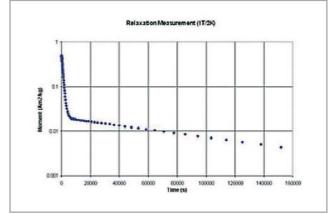
2. Measurement from 100 K to 2 K at 1Tesla (upper curve)



DC Magnetisation of GdSO4 at 1 T from 1.8 K to 325 K

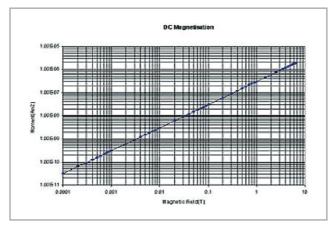


Total magnetic moment of the sample, measured in ZFC and FC procedures. The insert shows the size distribution of the particles deduced from transmission electron microscopy.



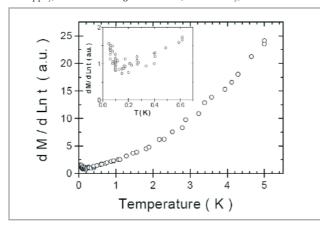
Example of a Relaxation Measurement:

The sample was cooled down to 2 K in zero field, the first measurement was made after a field change of 1 T. The intervals in time between measurements increase in 'pseudo logarithmic' steps. This example demonstrates continuous operation at temperatures below 4.2 K.



DC Magnetisation of NIST Aluminium reference from 0.1 mT to 6.5 T at 300 K

Measurement results from customer's laboratory. 'Anomalies in the relaxation of small magnetic particles at very low temperatures'. R. Sappey, E. Vincent and J. Hammann, CEA Saclay, France.



Magnetic viscosity as a function of temperature.

Specifications

| DESCRIPTION | CRYOGEN FREE S700X | LIQUID HELIUM S700X |
|---|--|--|
| Field range: | ±7 Tesla | ±7 Tesla |
| Field stability: Long term: | 0.1ppm/hr | 0.1ppm/hr |
| Central field uniformity over ±2cm: | 0.01% | 0.01% |
| Field set resolution (16 bit) Standard range: Low field option: | 0.11 mT 10 ⁻⁷ T | 0.11 mT 10 ⁻⁷ T |
| Maximum Remenant field: | \sim 0.7 mT (7 Gauss) | \sim 0.7 mT (7 Gauss) |
| Maximum current: | 80 Amps | 80 Amps |
| Maximum sample size: | < 9 mm | < 9 mm |
| DC magnetisation (1,2 and 3 axes) Differential sensitivity: | 1x10 [*] EMU 5-10 ⁻⁷ in 7 T | 1x10 ⁻⁸ EMU 5-10 ⁻⁷ in 7 T |
| Range of measurement Standard Extended | 10 ⁸ to 10-2 EMU 10 ⁸ to 5 EMU | 10 ⁻⁸ to 10 ⁻² EMU 10 ⁻⁸ to 5 EMU |
| AC Susceptibility (1,2 and 3 axes): | 0.01 Hz to 500 Hz | 0.01 Hz to 500 Hz |
| Frequency range: Sensitivity: | 1x10* EMU in 1 T 3x10 ⁻⁷ EMU in 7 T | 1x10* EMU in 1 T 3x10 ⁻⁷ EMU in 7 T |
| Temperature Range Standard Extended | 1.6 K to 300 K 0.3 K to 700 K | 1.6 K to 300 K 0.3 K to 700 K |
| Temperature calibration Number of sensors Accuracy of calibration | 2 0.3% | 2 0.3% |
| Temperature stability At sample At sample At sample | 2 mK @ 10 K 3 mK @ 100 K 10 mK @ 300 K | 2 mK @ 10 K 3 mK @ 100 K 10 mK @ 300 K |
| Temperature resolution | 1 mK all temperatures | 1 mK all temperatures |
| Temperature spatial variation in sample region: At 20K At 100K At 100K | ±5 K over 4 cm long ±25 mK over 4 cm long ±80 mK over 12 cm long | ±5 K over 4 cm long ±25 mK over 4 cm long ±80 mK over 12 cm long |
| Rate of temperature change (inc. stabilisation): ±5K ±100K ±295K | 5 mins 20 mins 30 mins | 5 mins 20 mins 30 mins |
| Cooling method | Pulse Tube Cryocooler | Liquid Helium with 50 litre helium capacity and consumption of 2-4 litres per day. |

Cryocooler specifications for the Cryogen Free S700X (Cryocooler & cold head)

We offer the PT410 pulse-tube cryocooler for this application. This unit is quieter than the GM alternative cryocooler, has a longer service interval, lower servicing costs and has a faster cool-down from room temperature.

| PULSE-TUBE CRYOCOOLER: CRYOMECH PT410 | | | | | |
|---------------------------------------|--------------------------------------|--|--|--|--|
| Cooling powers at 50 Hz | 40 W @ 45 K, 1.0 W @ 4.2 K | | | | |
| Base temperature | <4 K | | | | |
| Maintenance interval | 20,000 hrs (Adsorber change only) | | | | |
| Mains power requirement | 8 KVA, 3-phase | | | | |
| Water cooling | 8 L/min | | | | |
| Ambient temperature | 7°C to 38°C | | | | |
| Compressor dimensions | W 580 mm, L 530 mm, H 660 mm, 119 Kg | | | | |