



Cryostats for Electron Spin Resonance Spectroscopy

Introduction

Oxford Instruments offers a range of cryostats designed for X- and Q-band electron spin resonance/electron paramagnetic resonance (ESR/EPR) spectroscopy. The range has been designed for compatibility with commercial spectrometers.

The benefits of low temperature ESR spectroscopy include:

- An increase in signal levels from samples where relaxation times are very short at room temperature
- The ability to study phase transitions



CF935 for Q band cavities



ESR 900 for X band cavities

Cryostats for X-band cavities

Descriptions

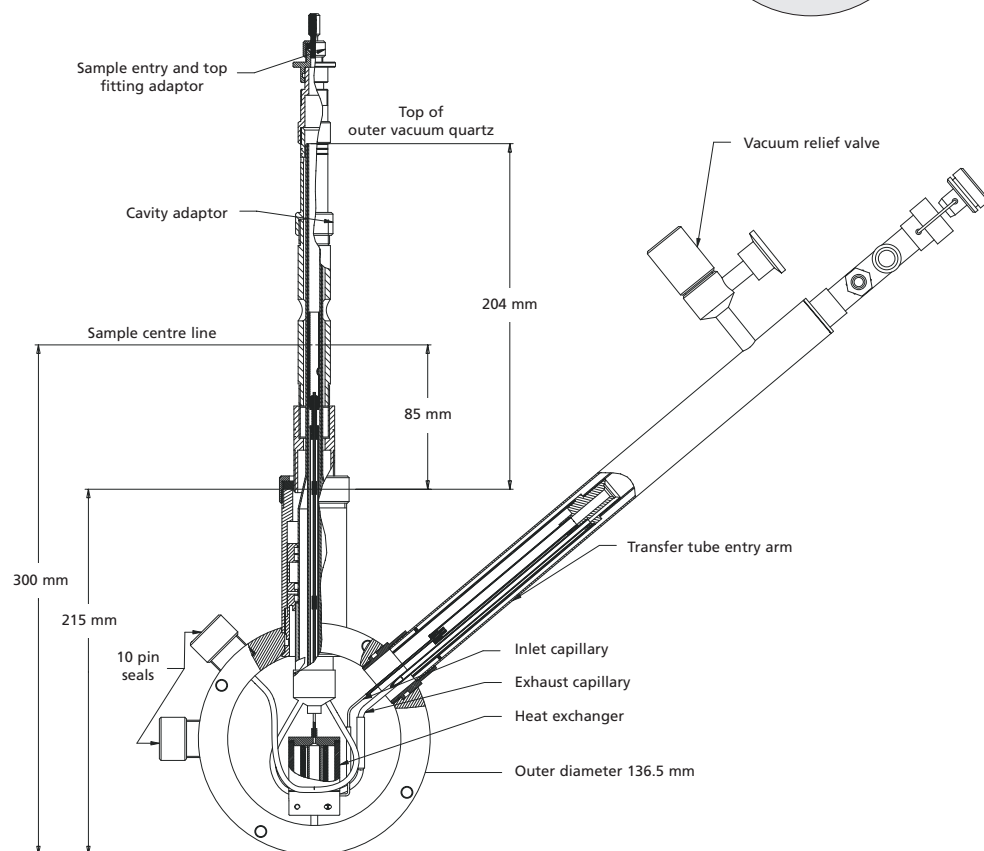
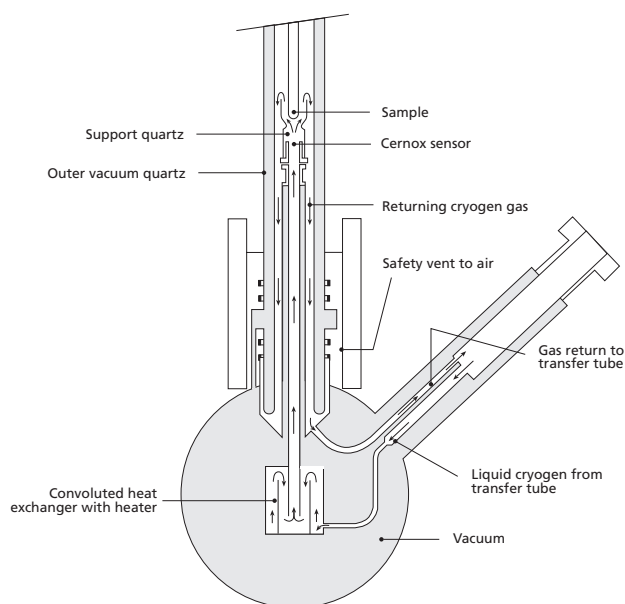
The ESR900 and ESR910 cryostats have been designed for compatibility with X-band cavities. These cryostats are top loading, continuous flow cryostats. Sample tubes are supported in the cryostat by a quartz cylinder. The sample and support quartz are contained within a double walled, vacuum insulated, quartz tube. This tube is a push-fit into the main body of the cryostat permitting easy removal for cleaning or replacement. Fittings are used to contain the quartz sections within the microwave cavity. The design allows the cooling of a sample within a room temperature spectrometer cavity.

ESR900

A heat exchanger located in the base of the cryostat is used to regulate the cryogen temperature. A controlled flow of temperature stabilised cryogen from the heat exchanger exits a capillary within the sample support quartz that is immediately below the sample position.

A Cernox sensor located at the capillary exit is used for accurate temperature measurement. The liquid/gas flows directly over the sample before exhausting back down the inside of the vacuum insulated quartz tube.

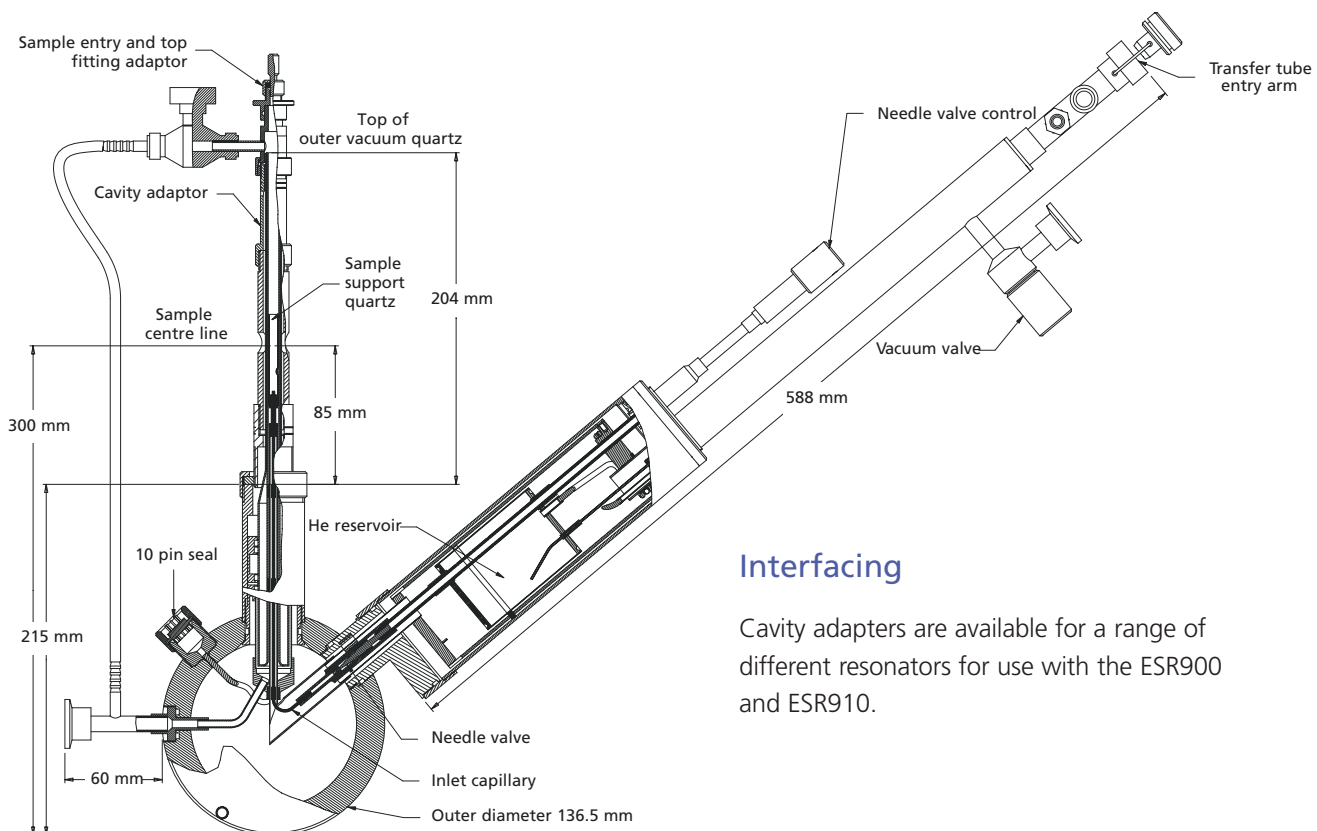
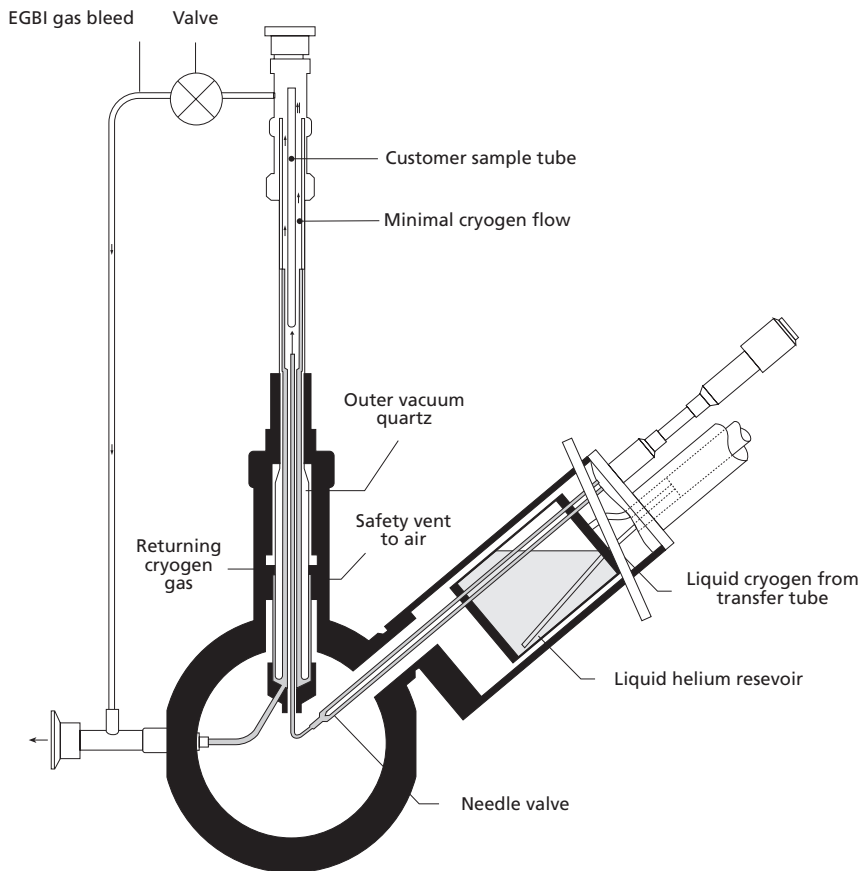
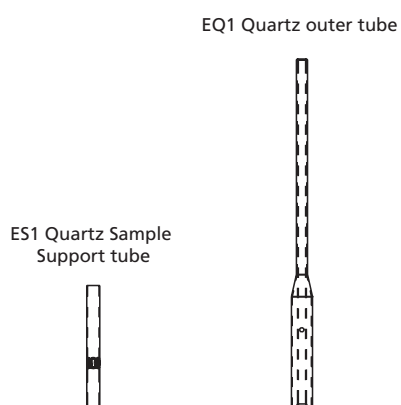
The ESR900 can be used with either liquid helium or liquid nitrogen.



ESR900 Cryostat

ESR910

A reservoir of helium located in the cryostat is used to achieve a base temperature as low as 1.9 K. The temperature is controlled using a Cernox sensor. Gas in the reservoir is returned along the transfer tube. The flow of liquid helium to the sample space is controlled by a needle valve in the base of the reservoir. The flow exhausts to a single stage rotary pump that reduces the helium vapour pressure in the sample region to provide a base temperature of 1.9 K.



Interfacing

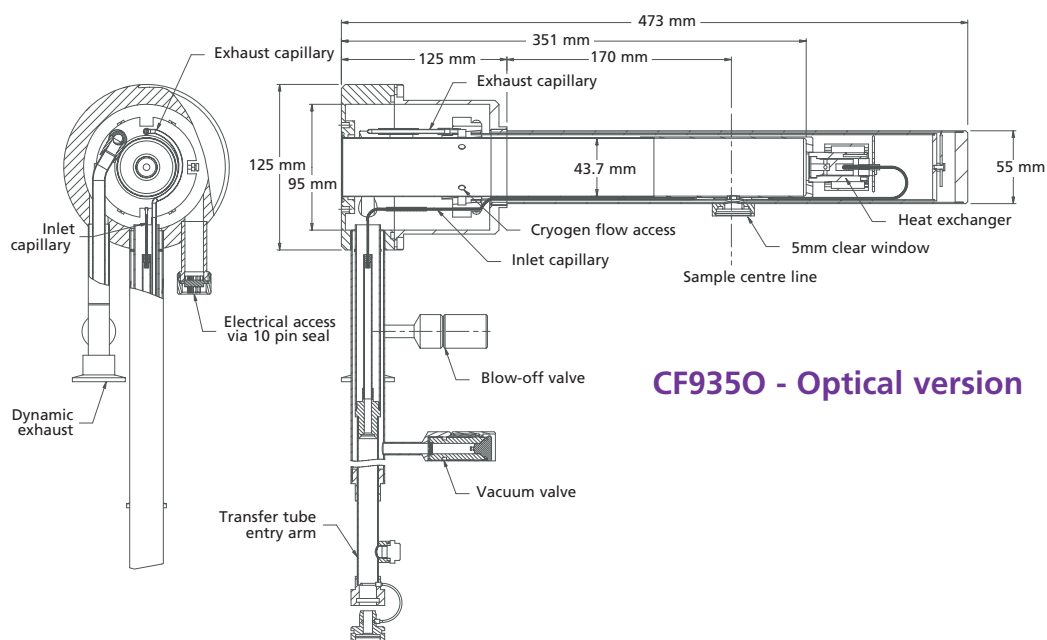
Cavity adaptors are available for a range of different resonators for use with the ESR900 and ESR910.

Cryostats for Q-band cavities

CF935P (non optical)

The CF935P cryostat immerses the entire sample and cavity in the cryogen flow. It provides experimental flexibility because it is compatible with many different cavities. The heat exchanger is fitted with a heater and Cernox temperature sensor.

The CF935P can be used with either liquid helium or liquid nitrogen.



CF9350 - Optical version

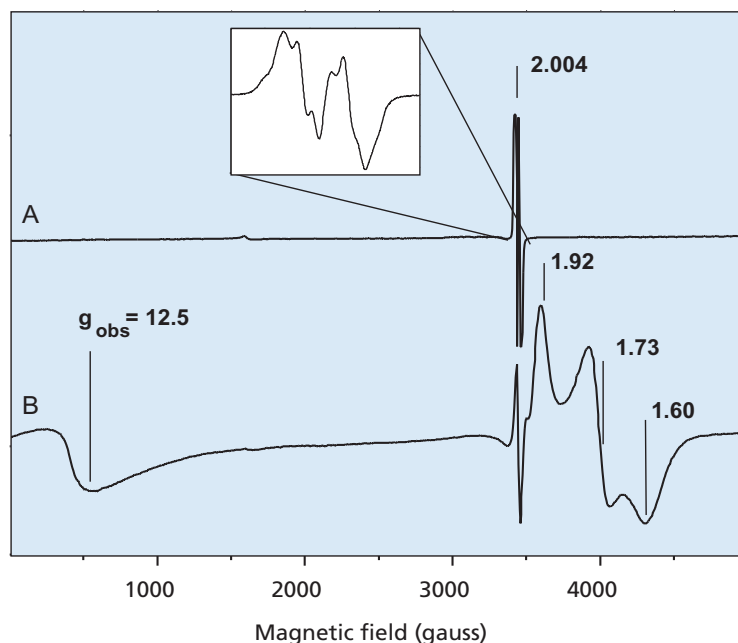
CF9350 Optical access

A version of the CF935P (the CF9350) enables optical access to the sample while ESR experiments are being carried out. It has one set of radial windows with a clear diameter of 5 mm.

Example of application for the ESR 900 cryostat:

The Department of Molecular Biosciences at the University of Oslo has been using an ESR cryostat for the study of structural and functional properties of enzymes with biologically active metal centres. In particular, this department has been interested in the enzyme ribonucleotide reductase, studying stable and transient paramagnetic states of its diiron centre and different amino acid radicals, such as tyrosine, cysteine, and tryptophan.

The use of an ESR cryostat is invaluable, especially for the study of metal centres, where the temperature based fine tuning of electron spin distribution between different energy levels is a crucial factor.



Results and analysis courtesy of Dr Matthias Kolberg, Department of Molecular Biosciences, University of Oslo, Norway.

X Band (9.65 GHz) EPR spectra of the diiron/tyrosyl radical protein R2 of ribonucleotide reductase from a mouse at different oxidation states.

Spectrum A: Active diferric R2 where only the tyrosyl radical is detected.

Spectrum B: Chemically reduced forms of R2.

The ferric-ferrous form (FeII/FeIII) and the diferrous form have g- values (1.92, 1.73, 1.60) and 12.5, respectively.

Conditions: (A) 15 K, 100 μ W. (B) 4 K, 20 mW.

Recorded on a Bruker Elexsys E560 spectrometer using a Bruker ER 4116DM dual mode resonator with an Oxford Instruments ESR 900 helium flow cryostat.

System Components	ESR900	ESR910	CF935
Cryostat	ESR900	ESR910	CF935P or CF935O
ESR910 gas bleed valve		EGB1	
Flexible gas shielded transfer tube	LLT600/13	LLT600/13	LLT600/13
Storage vessel adapter	SV12	SV12	SV12
ESR outer quartz	EQ1	EQ1	
ESR sample support quartz	ES1	ES1	
Cavity adapters for Bruker EN801 resonator	CAB	CAB	
Temperature controller	ITC503	ITC503	ITC503
3m cryostat cable – 10 pin connector	CC1	CC1	CC1
Gas flow controller with flowmeters	VC41	VC31	VC41
Gas flow pump	One GF4	Two GF4	One GF4

Specifications	ESR900	ESR910	CF935P	CF935O
Temperature range (K)	2.6 – 300 using an EPS40	1.9 – 300 using EPS40 + GF4 pump	1.9 – 400 using an EPS40	1.9 – 300 using an EPS40
	3.4 – 300 using a GF4	3.0 – 300 using two GF4 pumps	3.3 – 400 using a GF4	3.2 – 300 using a GF4
Temperature stability (K)	±0.1	±0.1	±0.1	±0.1
Cool down time (mins)	30	30	60	60
Cooldown consumption (litres)	2	2	2.5	2.5
Helium consumption at 4.2 K (litres/hr)	1.1	1.6	1.2	1.2
Sample change time (mins)	~5	~5	~1	~1
Temperature range with LN 2 (K)	80 - 300		80 - 400	80 - 300
Sample space diameter (mm)			43	43
Tail outer diameter (mm)			55	55

Optional Items	
HD30	30 litre helium dewar
EG1	ESR goniometer
STC4	ESR sample top cap
CAV	Cavity adapters for Varian E231 (for ESR900 and ESR910)
LLT650/13	Automated flexible gas shielded transfer tube
ITC502	Temperature controller
SCI	Three channel upgrade interface for ITC502/ITC503
CCI	3 m cryostat cable - 10 pin connector
EPS40	40 m ³ /hr helium pumping system
HVP4	High vacuum pumping system
NSA	Nitrogen side arm
ESRT	Temperature sensor for the ESR 900 and 910 Calibrated Cernox sensor Fitted in a 4 mm diameter sample tube Temperature range 1.9 K-300 K

Note: All specifications refer to a standard system using a LLT transfer tube.

Oxford Instruments offers a wide range of accessories, including windows, vacuum pumps, samples holders, guaranteed to be compatible with your original Oxford Instruments product.



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