PRÉSERVER
SAUVEGARDER
VALORISER

DOCUMENTATION SUR LA CHAMBRE DE DIFFRACTION DES RAYONS X GUINIER-SIMON , HAUTE TEMPÉRATURE ENRAF-NONIUS

FICHE N° 4019

Période de fabrication : 1975-1999
Fabricant : ENRAF-NONIUS
Domaines : Chimie, Physique
Sous-domaines : Cristallographie, Chimie du solide, Cristallographie, Physique du solide
Organisme : Université de Rennes
Ville : Rennes
Modèle : Guinier- Simon
Matériaux : Papier

Description

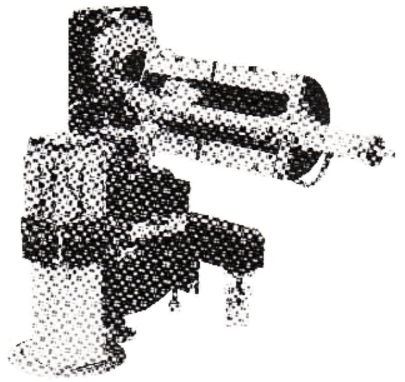
Cette documentation en français de quatre pages a été éditée par la société Enraf-Nonius. Elle présente les différents éléments de l'appareil, ses caractéristiques et performances, les opérations de maintenance ainsi que tous les systèmes de chauffage basse et haute température et leur programmation.

Utilisation

La chambre de Guinier-Simon de diffraction des rayons X servait à effectuer des études cristallographiques d'échantillon cristallin. Des études du comportement des matériaux dans ces gammes de température se révélaient très utiles notamment pour la détection de changements de phase.

High temperature experiment

The sample was investigated in a temperature range of 100°C to 500°C. After it is brought inside the camera the temperature is raised to 100° and kept constant for 1 h. Now the temperature is increased during 4 h. to 200°C and kept constant for 1 h. Hereafter it is raised to 500°C at a rate of 37.5°C/h. When 500°C was reached the temperature was kept constant for 1.5 h. after which the sample was cooled down to 100°C during 12 h.



Guinier-Simon camera for high temperature work

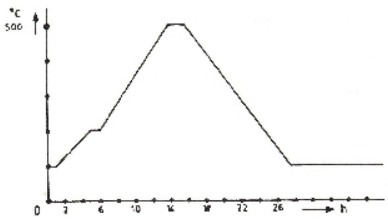
The setting for the high temperature experiment is:

Temperature regulator	4000
Span potentiometer	4

	F R Δ
Program step 1	4 // 999
2	1 99 099
3	3 62 250
4	1 99 099
5	3 94 750
6	1 66 099
7	2 83 999
8	9 // ///

/ means any value is allowed as these numbers are irrelevant.

The high temperature experiment



OPERATION REQUIREMENTS

Focus	:	horizontale line focus image 8 × 0.04 mm or 8 × 0.02 mm fine focus
Tubeshield	:	vertical, for adaption kits see below
Monochromator support bracket	:	according ACA specifications; height adjustable between 160 and 190 mm above table top.
Monochromator	:	nominal height incident beam entrance above track 100 mm. nominal focus monochromator distance 90 mm nominal focussing distance 220 mm
Camera	:	sample axis horizontal only

Mains	:	220V, 50 Hz/110V, 60 Hz 1.5 kVA
Cooling water	:	4 l/min. (high and low temperature systems)
Compressed air	:	14 l/min. for high temperature systems only
Liquid nitrogen	:	1.5 l/h independent on gas stream temperature (for low temperature systems only)
Table top	:	when the monochromator support bracket cannot be attached to the tubeshield, the table top must be stable enough to make sure that the relative monochromator focus position does not change
Film size	:	180 × 170 mm

LITERATURE

Simon, A., (1970a). J. Appl. Cryst. 3,11
Simon, A., (1970b). J. Appl. Chryst. 3,18
Simon, A., (1971). J. Appl. Chryst. 4,138
Lenné, H.-U., (1961) Z. Kristallogr. 116,190

CATALOGUE NUMBERS

FR553 GUINIER SIMON FOCUSSING POWDER CAMERA
for fine structure investigations of air sensitive substances, frozen liquids and condensed gasses at varying temperatures. The camera was designed and developed in co-operation with Dr. A. Simon.
Its modular design allows the user to select from a range of options to meet his particular needs.
Camera system consisting of:
- monochromator housing with beamtunnels for monochromated beam. Without Johansson monochromator (SELECT MONOCHROMATOR BLOCK FROM MONOCHROMATOR BLOCK OPTIONS) and without monochromator track systems (SELECT APPROPRIATE TRACK SYSTEM FROM TRACK OPTIONS),
- aligning slides for camera bracket,
- camera bracket including:
  * provision to set forward and back reflection geometry,
  * film cassette and cassette travel mechanism,
  * specimen centering facility,
  * specimen rotation/reciprocation motor,
  * programmer controlled beam stop,
- accessory box (1553.400),
- Guinier Simon controller including:
  * controls for filmspeed, beam stop, specimen rotation/reciprocation,
  * digital programmer for temperature programming.

AVAILABLE MODELS:

FR553L	Guinier Simon camera for temperatures between −180 and +300 degrees Celsius using pressurized LN2 containers. (Order 3500.014).
FR553H	Guinier Simon camera for temperatures between +20 and 1100 degrees Celsius.
FR553C	Guinier Simon camera for temperatures between −180 and 1100 degrees Celsius. Including low temperature system for use with pressurized LN2 containers. (Order 3500.014).

TRACK OPTIONS:

1555.921	Monochromator housing track support for Enraf-Nonius tubeshields FR551/FR561.
1555.922	Monochromator housing track support for Philips PW1316 tubeshields.

MONOCHROMATOR BLOCK OPTIONS:

1596.691	Johansson monochromator Cu.
1596.692	Johansson monochromator Mo.
1596.693	Johansson monochromator Co.
1596.694	Johansson monochromator Cr.
1596.695	Johansson monochromator Fe.

PRESSURIZED LN2 CONTAINER:

3500.014	100l LN2 storage container.
	Optional airpump for use with 0553.341.
	Optional flowmeter for use with 0553.341.
1553.030	Cross slides for fine adjustment of the camera in the direction of the monochromated beam and perpendicular to the beam. Includes adapter ring 0129.379.
0553.341	Spare heater gun.
2126.112	Spare Guinier Simon end dewar
2077.632	Spare Guinier ruler Cu.
2077.633	Spare Guinier ruler Co.
2077.634	Spare Guinier ruler Fe.
2077.635	Spare Guinier ruler Mo.
2077.636	Spare Guinier ruler Cr.
0127.570	Spare capillary fitting (min. order qty. = 10).
0127.572	Spare capillary support rod.

B.V. ENRAF-NONIUS DELFT
P.O. Box 483, 2600 AL Delft, Holland
Tel.: 015 - 56 92 30
Telex 38083
Address: Röntgenweg 1

ENRAF NONIUS DELFT

switch is provided to open and close the beam stop. In the "line" mode the shutter is opened automatically after a fixed cassette displacement. To obtain a correctly exposed primary beam reference line the shutter exposure time is continuously adjustable between 1 and 11 seconds. The combination of an adjustable primary beam exposure time and a facillity for an attenuation filter in the beam stop assembly ensures an excellent primary beam recording that can be used for accurate measurements.
In posittion "program mark" the beam stop is opened automatically at the start of each step of the temperature programmer. A diaphragm to be inserted into the shutter assembly obstructs the major part of the primary beam so that the film is marked with a small point only. The density of the marker point can be controlled by the primary beam exposure time potentiometer.

The PID temperature regulators.
The PID temperature regulator controls a thyristor unit which in its turn regulates the amount of energy dissipated in the heating element in accordance with the temperature program set by the operator. Any difference between temperature wanted (set point) and measured temperature is compensated by the feed back circuit consisting of temperature sensor, PID temperature regulator, thyristor unit and heating element.
The PID characteristic of the regulators ensure an optimal operation.
Two different PID temperature regulators are available. The range of the high temperature PID regulator is from 100°C. – 1099.9°C. (thumb wheels setting 0000 – 9999). The low temperature PID regulator's range is between −200°C. and 299°C. (thumb wheels setting 0000 – 9999). The relation between the thumb wheels setting and the temperature at the location of the sensor is given on a calibration chart. The setting accuracy is 0.1%.

The temperature programmer.
The temperature programmer has 8 groups of 6 thumb wheel switches. The switches in each group are used to program one program step (= one temperature stage). A group of 6 thumb wheel switches is divided into 3 sub groups. (F R Δ)
Sub group F is one switch to program the 6 basic instructions:

preset	:	determine the start temperature
hold	:	steady temperature
run up	:	descent in temperature
run down	:	raise in temperature
wait	:	temperature as reached at the end of the previous program step remains unchanged
finish	:	same as wait instruction but the system is ready for a 'loop' command.

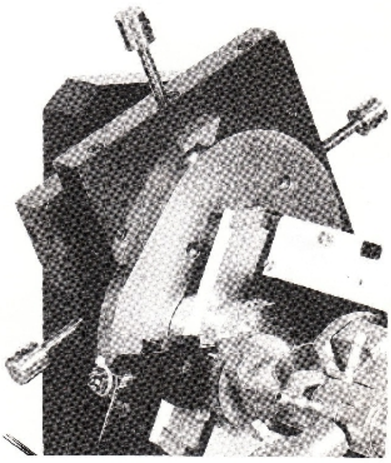
Sub group R uses two switches to select a speed between 00 and 99 counts of the program counter per hour.
Sub group (F R Δ) requires three switches to set the temperature variation between 000 and 999 counts of the program counter.

This arrangement makes it possible to program up to seven temperature stages not including the start temperature. Temperature programs with an unlimited number of temperature stages or automatic re-cycling programs with maximum of seven stages can be run by the 'loop' feature.
In position 'loop' the program automatically jumps back to the first program step when the last program step contained a 'finish' instruction.

The temperature range in which the experiment has to take place is fixed by two parameters i.e. the maximum temperature and the maximum descent in temperature. Therefore the momentary gas stream temperature during the experiment can never exceed the maximum temperature as set on the PID temperature regulator. The maximum descent in temperature (= temperature span) is set with the span potentiometer (ten turn high precision potentiometer).

The temperature program is under control of a 3 decade program counter coupled to a digital to anologue converter. The analogue output of this converter can be attenuated by the span potentiometer. In this way any temperature span (= the maximum variation of the temperature during the experiment) can be divided into 999 counts of the program counter. This feature gives an optimal temperature resolution.
LED indicators show the system's status and indicate the program step which is actually being executed.

The system has built-in protection circuits to monitor the flow of cooling water, the flow of cold nitrogen gas (LT systems); the flow of air (HT systems) and senses whether the heating elements and temperature sensors are connected to the controller. In case one of the above conditions has not been fulfilled or in case the mains and the gasflow is interrupted the controller indicates the 'reset' status. In the 'reset' status the electronics are reset, the heating element is switched off.
This prevents any damage due to freezing or excessive heating of the camera and the sample.



Camera detail with cross slides

A flat sample is only reciprocated in the beam. Preparation as flat sample may be an advantage working e.g. in back reflection position, because then more substances contributes to the diffraction.

The film cassette.
The film cassette is mounted on the focussing circle and can be rotated around its center. The maximum two theta range is about 90° for one single film setting.
Rotating the film cassette around the center of the focussing circle almost the complete two theta range can be covered by a series different exposures.
During the exposure the film can be moved at speeds between 0 and 9.9 mm/h depending on the special diffraction intensity. The diaphragm width is continously variable between 0 and 10 mm.
Secondary diaphragms are available to reduce the back ground due to scattering.

Low and high temperature work in the temperature range − 180° C / about 300° C.

For low and high temperature work in this range a dewar tube is used to lead the gas to the sample. Cold nitrogen gas, evaporated in a universal low temperature device FR 524S is used to treat the sample in the temperature range of − 180° to + 300° C.
A heating element inside the dewar tube heats the gas stream; the temperature sensor is a Pt-100 resistance element. This sensing element is part of the feedback temperature control.

High temperature work in the range between 100° C and 1100° C.

A heating element in a ceramic tube is used to heat compressed air that passes the sample.
A metal outer jacket and the sample head are flushed with water to provide an efficient cooling. A built-in thermocouple is the temperature sensing element for the feedback temperature control.

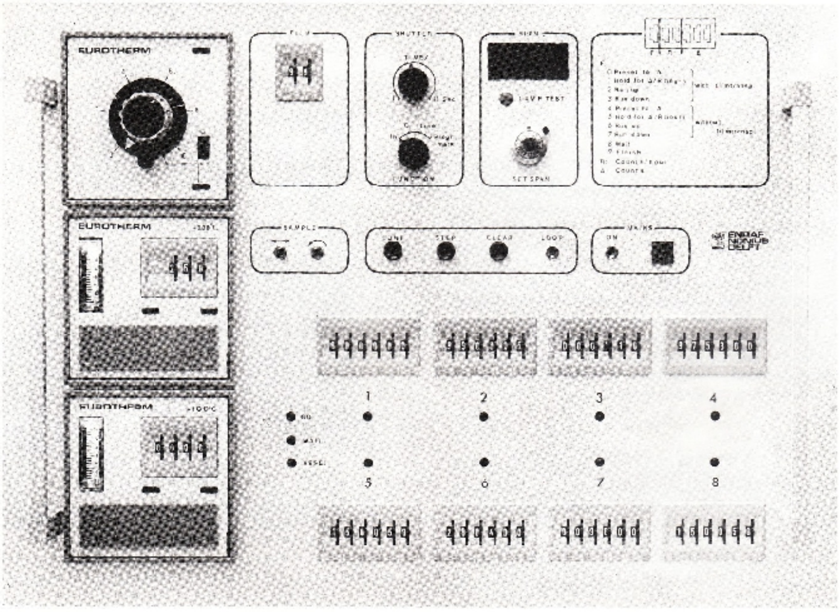
The Guinier Simon Controller contains the circuits for:

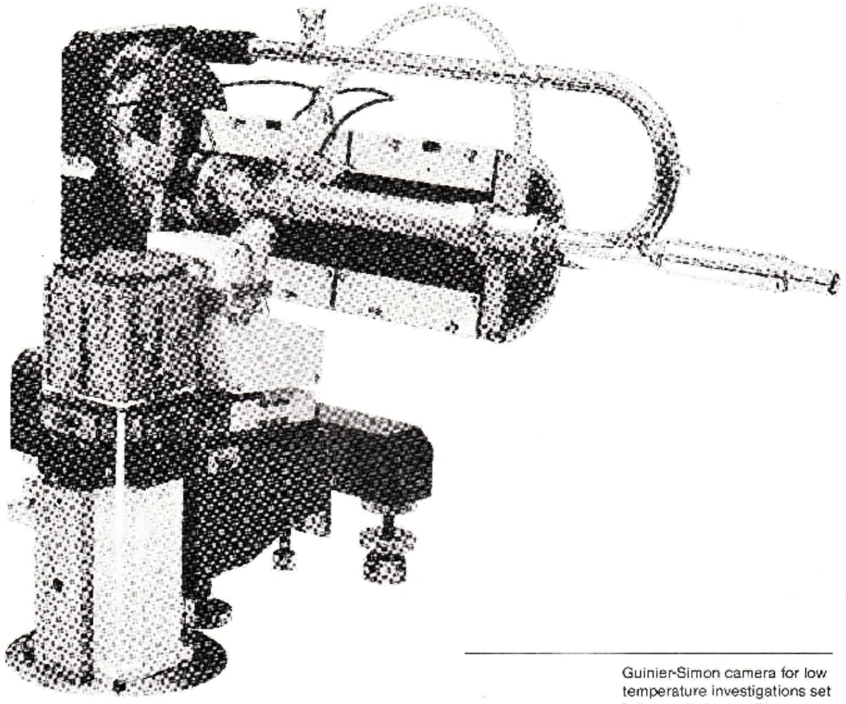
The film drive.
The film cassette is driven by a stepping motor. Two thumb wheel switches are used to select a film speed between 0 and 9.9 mm/h.

The sample drive.
Two controls are available for rotation and reciprocation of the sample. The speed and direction of rotation are fixed; the rate of reciprocation can be set with an eccenter on the camera. For capillary mounted samples both rotation and reciprocation are used. For flat samples only the reciprocating movement is used.

The beam stop.
The beam stop control has three positions; manual/line/program mark. For manual operation a

Guinier-Simon controller with PID temperature regulators for low and high temperature work.



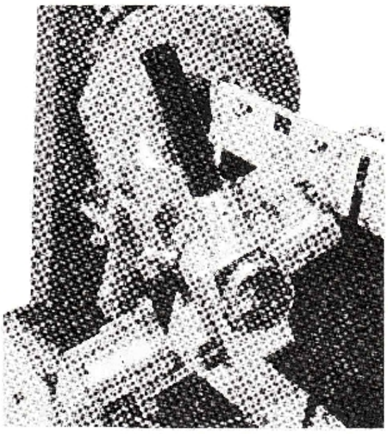


Guinier-Simon camera for low temperature investigations set in transmission position.

around the sample axis assure an easy change of inclination angle.

The sample head.
As the hot or cold gas leaves the system along the sample head, water is used to prevent icing or excessive heating of this part.
The sample that is placed in the gas stream is supported on a rod.
This rod can very conveniently be centered by a screw adjustment. The sample position can be observed using an alignment microscope with built-in illumination. Because the microscope clips over the sample head it does not obstruct the gas stream and therefore it is possible to verify the position of the sample under experimental conditions (without X-rays).

The sample.
The sample is prepared in a capillary or as a flat plate. In a capillary e.g. air sensitive substances can be investigated. In addition a cylindrical sample gives a sharp diffraction pattern of sufficient intensity. Moreover, the temperature gradient in the sample space is more favourable for accurate investigations. During the exposure the capillary is rotated and in addition an independent reciprocating movement, parallel to the line focus, is made.



The sample space with alignment microscope.

INTRODUCTION

The ENRAF-NONIUS high and low temperature Guinier camera according to Lenné is well-known and is used with much success in many laboratories everywhere in the world, where temperature dependent investigations are made in chemistry, solid state physics, crystallography, mineralogy, etc. A Guinier camera arrangement is most favourable for such sophisticated investigations, due to monochromatized radiation and its spectral purity, high resolution and the low background scattering. Structural modifications and changes depending on tempererature can very conveniently be studied, such as:

- thermal expansion and its possible anisotropy
- monotropic or allotropic alterations of modifications and phases
- chemical transformations as e.g. decompositions of water or reactions
- recrystallization of amorphous substances, etc.

The new Guinier Simon camera is based on a somewhat modified Guinier technique. It was developed and designed in co-operation with A. Simon (present address: Max Planck Institut für Festkörperforschung., Stuttgart, West Germany) of the Institute of Inorganic Chemistry at Münster, Germany, using all experience Enraf-Nonius gathered in designing and manufacturing Guiner cameras for ambient temperature and temperature dependent work.

Structural changes in solids including extremely air sensitive substances, frozen liquids and condensed gases may be studied very accurately and with proper convenience. Low and high temperature exposures may be made using one single sample setting.
A new temperature controller/programmer has been developed for this camera enabling the user to do very sophisticated work in one single run.

FEATURES OF THE GUINIER SIMON CAMERA

- highly resolving Johansson monochromator separating the k α_1 / α_2 doublet
- easy and most stable monochromator adjustment
- easy and accurate focussing procedures
- angle of radiation incidence on sample continuously variable
- transmission as well as backreflection exposures
- flat powder sample as well as cylindrical shaped sample can be investigated
- air sentitive substances are investigated filled in capillaries of glass or other material
- reciprocating sample movement during exposure: either sample translation or oscillation or both keeping the sample in aligned position
- film cassette may be rotated around the centre of the focussing circle in order to record diffraction lines in different two-theta ranges
- two-theta range for one setting of the film cassette almost 90°
- low and high temparature investigations using the gas-stream method
- very accurate temperature control and programming using thermocouple feed back
- investigations with increasing, decreasing or constant temperature characteristics
- maximum radiation protection by using beam tunnels between tubeshield and monochromator as well as between monochromator and sample space.

DESCRIPTION OF THE CAMERA SYSTEM

The major parts of the Guinier Simon camera system are:
- monochromator with radiation shield and track.
- camera with sample drive motors, cassette including cassette drive.
- low temperature unit to generate a constant flow of cold nitrogen gas, including camera dewar with built-in heating element and temperature sensor.
- high temperature unit with built-in temperature sensor.
- Guinier Simon controller containing camera controls, temperature programming unit and PID temperature regulators.

Monochromator and track

The monochromator is of the Johansson type and will separate the k α_1 / α_2 doublet of the incident radiation. The quartz monochromator crystal is set to reflect by a very sensitive micrometer/lever construction. The setting sensitivity is better than 0.01 degree Bragg angle.

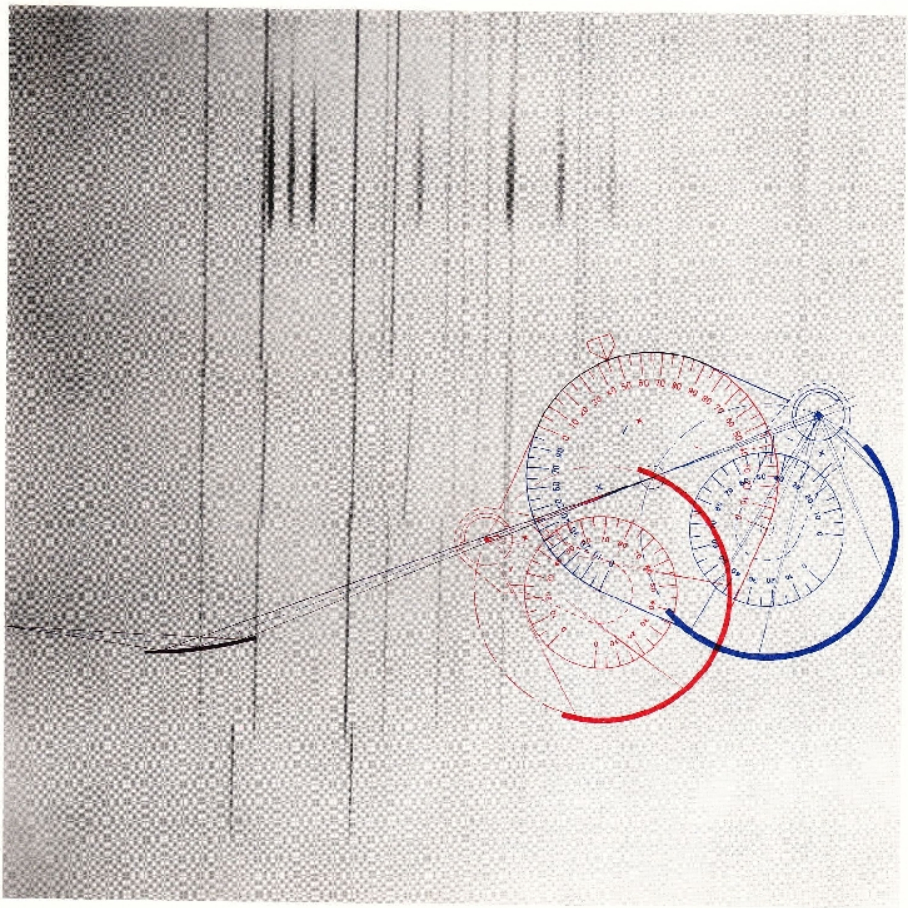
The monochromator crystal can easily be replaced for various radiation use. The monochromator housing fits on an ACA accepted track system. The Enraf-Nonius FR 555 support bracket assures a stable base for the monochromator. The support bracket fits to the Enraf-Nonius FR 551/561 tubeshields. A special monochromator support bracket adaptable to the Philips PW 1316 tubeshield is available. Adaptions to tubeshields of other make will be made available on user's request.

Camera

The sample head and film cassette assemblies are mounted on a support that can be adjusted around the monochromatized beam focussing point. The sample is adjusted in the beam by a rotation around the center of the focussing circle. The film cassette can move on the circumference of the focussing circle. The beamstop can be rotated around the sample axis. Scales around the focussing point, the center of the focussing circle and

ENRAF NONIUS DELFT

Guinier Simon camera FR 553



553 SEP 83 vW

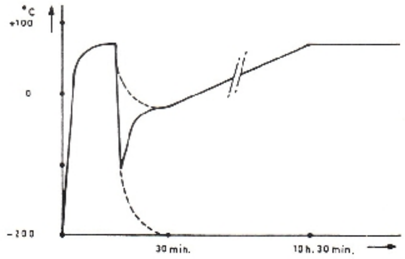
TWO EXAMPLES SHOW THE VERSATILITY OF THE TEMPERATURE PROGRAMMER

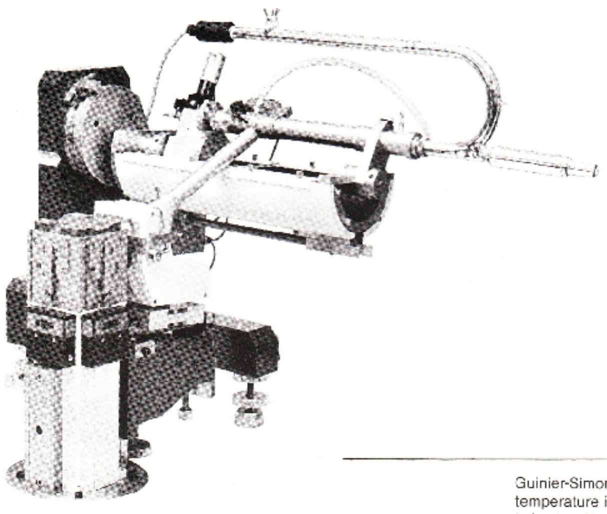
Low temperature experiment

Before making the exposure the sample was treated inside the camera. The temperature treatment before the exposure starts at minimum temperature (about −180°C). After this the temperature is raised as fast as possible to +65°C.
To make sure that the sample temperature is actually +65°C the gas stream temperature is kept constant for e.g. 15 minutes. Now the temperature must be lowered as fast as possible to −20°C. This can be done by programming an overshoot to −200°C and a hold for 2 minutes after which a jump to −20°C and a 15 minutes hold are progammed.
The preparation of the sample is ready now and the exposure can start. During 10 hours the temperature is raised from −20°C to +65°C. After the 10h. exposure the sample temperature is held constant at +65°C.
The setting for the low temperature experiment is:

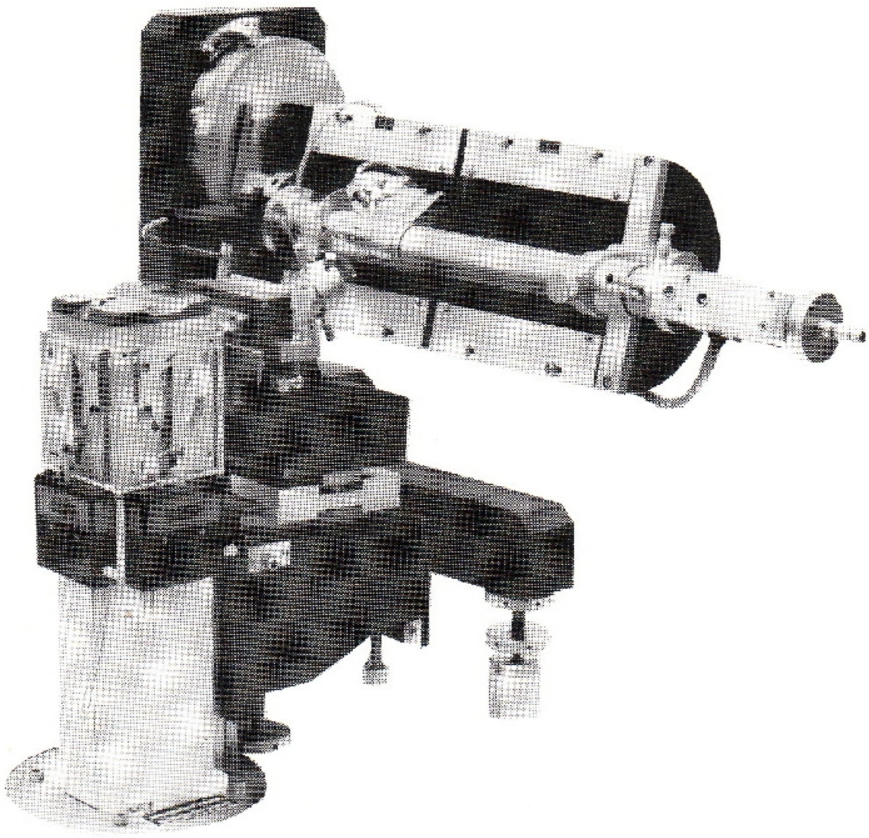
Temperature regulator		5300
Span potentiometer		10
		F R Δ
Program step	1	4 // 000
	2	5 60 015
	3	4 // 530
	4	5 60 002
	5	4 // 170
	6	5 60 015
	7	3 17 170
	8	9 // ///

The low temperature experiment





Guinier-Simon camera for low temperature investigations set in back reflection geometry.



Guinier-Simon camera for high
temperature work