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Target with a frozen nuclear polarization for experiments at low energies

N.S. Borisov [a], V.N. Matafonov [a], A.B. Neganov [a], Yu.A. Plis [a], O.N. Shchevelev [a], Yu. A. Usov [a], I. Jánský [b], M. Rotter [b,*], B. Sedlák [b], I. Wilhelm [b], G.M. Gurevich [c], A.A. Lukhanin [d], J. Jelínek [e], A. Srnka [e], L. Skrbek [f]

[a] *Joint Institute for Nuclear Research, Laboratory of Nuclear Problems, Head Post Office, P.O. Box 79, Moscow, Russian Federation*
[b] *Faculty of Mathematics and Physics, Charles University, V Holešovičkách 2, 180 00 Prague 8, Czech Republic*
[c] *Institute for Nuclear Research, Russian Academy of Sciences, 60th October anniversary prospect 7A, 117312 Moscow, Russian Federation*
[d] *Kharkov Institute of Physics and Technology, Academy str. 1, 310108 Kharkov, Ukraine*
[e] *Institute of Physical Instrumentation, Academy of Sciences of the Czech Republic, Královopolská 147, 612 64 Brno, Czech Republic*
[f] *Institute of Physics, Academy of Sciences of the Czech Republic, 250 68 Řež, Czech Republic*

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A target with a frozen spin polarization of protons in 1,2-propanediol with a paramagnetic Cr(V) impurity is described, intended for polarization parameter studies in np-scattering at approximately 15 MeV neutron energy. The target of cylindrical shape of 2 cm diameter and 6 cm long with an initial polarization of $95 \pm 3\%$, obtainable by the dynamic polarization technique, is placed at a temperature about 20 mK in a magnetic field of 0.37 T generated by a magnetic system, which provides a large aperture for scattered particles. The relaxation time for the spin polarization in an experiment is about 1000 hours.

1. Introduction

At present in the studies of polarization phenomena at beams of many accelerators, solid-state targets with a frozen nuclear polarization are frequently used [1]. We propose a version of a target intended for polarization parameter studies in np-scattering using 15 MeV neutron beam produced by the Van de Graaff accelerator of the Charles University Nuclear centre in Prague.

The target is a complex including a stationary cryostat with a dilution refrigerator, a movable magnetic system providing a "warm" field and consisting of a superconducting solenoid and a superconducting dipole magnet with a large aperture, and electronic equipment for providing a dynamic polarization and NMR-signal detection.

In this paper the design and parameters are described of a facility produced by a large collaboration of JINR (Dubna), Nuclear Centre and Low Temperature Physics Department of Charles University (Prague), KIPT (Kharkov), INR (Moscow), IPI (Brno) and IP (Prague).

* Corresponding author. Tel +42 2 8576 2565, Fax +42 2 6641 5095.

2. Dilution refrigerator; general description

The refrigerator with a horizontal tailpiece placed stationary in a neutron beam is shown in Figs. 1 and 2. A module design was used stimulated by the idea of wider applicability of both the cryostat and the refrigerator after the conclusion the experiments in the initial set-up. As one can see from Fig. 1, the low temperature system consists of three autonomous matchable units: a cryostat (2), a ^{3}He pumping, pre-cooling and condensing unit (1), and a ^{3}He/^{4}He dilution step (3). The units are matched through rubber and indium vacuum seals, assembly and disassembly procedures being performed relatively fast. Another merit of such a design is easy access to every unit for separate tests and repairs. Furthermore, any unit could be modified without influencing other parts of the facility, provided the connecting elements are conserved. A central unit – the cryostat – can be used independently too if the holes in a helium vessel and the outer jacket are sealed by corresponding details. The cryostat and the ^{3}He pumping and condensing unit can be combined with a new dilution step, a return to the initial scheme being without difficulties. It is an authors' opinion that labour expenses and a certain complication of design are justified during adjust-

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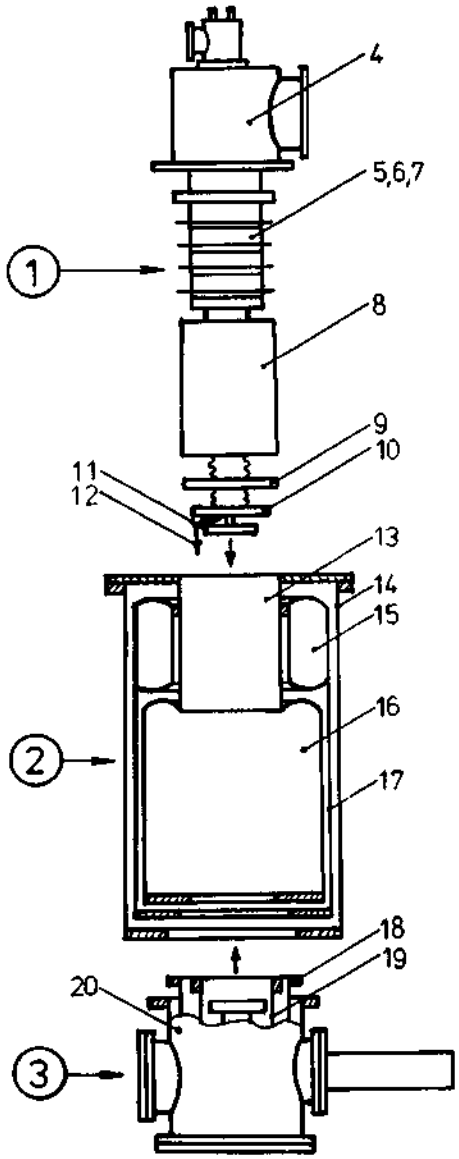

Fig. 1. Three parts of the dilution refrigerator (see text)

ment and repairs, and that they broaden the scope of the facility from an experimental point of view.

The ^{3}He pumping, pre-cooling and condensing unit (Figs. 1 and 2) consists of a cap (4), connectable to external pipelines, ^{3}He and ^{4}He pumping tubes (5,6) with gas heat exchangers (7), a ^{3}He condensing unit (8) and flanges for connection to the cryostat (9) and to the dilution step (10), and is completed by a capillary (12) for a liquid ^{3}He and a ^{3}He still cover (11).

The internal arrangement of the condensing unit is shown in Fig. 2. A pumped-out ^{4}He bath (21), which provides a temperature in the 1.1–1.3 K range depending on the heat load, is filled with ^{4}He through a vapour-cooled coil pipe (41) and an adjustable needle valve (24). ^{3}He gas cooled in an upper gas heat exchanger comes to a coil pipe (42) placed inside the ^{3}He pumping tube, condenses in a condenser (22) of sintered copper powder, and comes to the dilution step through an adjustable needle valve (23) and a constant restrictor (40).

The cryostat (Figs. 1 and 2) having a 170 mm diameter mouth (13) and a 400 mm diameter outer vacuum jacket is provided with a 17 l liquid nitrogen vessel (15) ensuring 24 h of a work without refilling. Holes are provided in the lower parts of the helium vessel (16), liquid nitrogen cooled shield (17) and outer jacket for connection of corresponding elements of the pumping unit and the dilution step. A liquid helium stock of about 19 l ensures dilution refrigerator work for 30 h without refilling.

The dilution step (Figs. 1, 2, and 3) consists of a jacket (20), a nitrogen (18) and a helium (19) shield, a ^{3}He still (26), heat exchangers (28) and (39), and a mixing chamber (31), rigidly connected by tension wires and posts. Sample loading is performed through a lock chamber (38). A microwave cavity (32) formed by a tail portion of the helium-cooled shield and a microwave choke (30) is fed through a waveguide (33). A RF-circuit coil is connected to NMR equipment by a cable (29).

Thermal contacts of the nitrogen and helium shields are made of flexible copper wire bundles (25, 27) of about 1 cm^2 cross section which are approximately 5 cm long (for each shield). The waveguide and the cable are cooled through point thermal contacts; the waveguide has contacts at 80 K and 4 K, and the RF-cable has additional contacts to the still and to the body of the main heat exchanger.

The radiation shields of the horizontal tailpiece are made removable, providing free access to the microwave cavity, the mixing chamber and the RF-circuit coil. The main horizontal heat exchanger and the still are also easily demountable.

The lock chamber (38) is an important unit used during loading and extraction of a sample, which has a low melting temperature. The design of the lock chamber was restricted by the small distance between the elements having room and helium temperatures. As a result it is difficult to provide a lock chamber in the form of a continuous sealed channel with a reasonable thermal load to the helium shield. The problem was solved by using bellows with a flange (34), which can be disconnected from the helium shield of the dilution step.

The sample loading operation using this lock chamber is as follows. The flange (34) is sealed in with the helium shield, and after cooling the cryostat to the liquid nitrogen temperature the lock chamber channel and a cavity inside the helium shield are filled with helium gas at atmospheric pressure. Then covers (35, 36, 37) are removed using a special device, which limits air inflow to the cooled elements. After sample loading the covers are reinstalled and the helium gas is pumped out. Now the lock chamber flange can be disconnected from the helium shield, so that

the thermal coupling through the lock chamber channel is broken and the volume of the lock chamber is united with that of the cryostat vacuum jacket. The whole lock chamber is made as a single unit and can be easily disconnected from the dilution step.

3. Dilution unit; internal arrangement

During the development of the dilution unit the authors also used the idea of a module design. This most important portion of the refrigerator is illustrated in Fig. 3 and

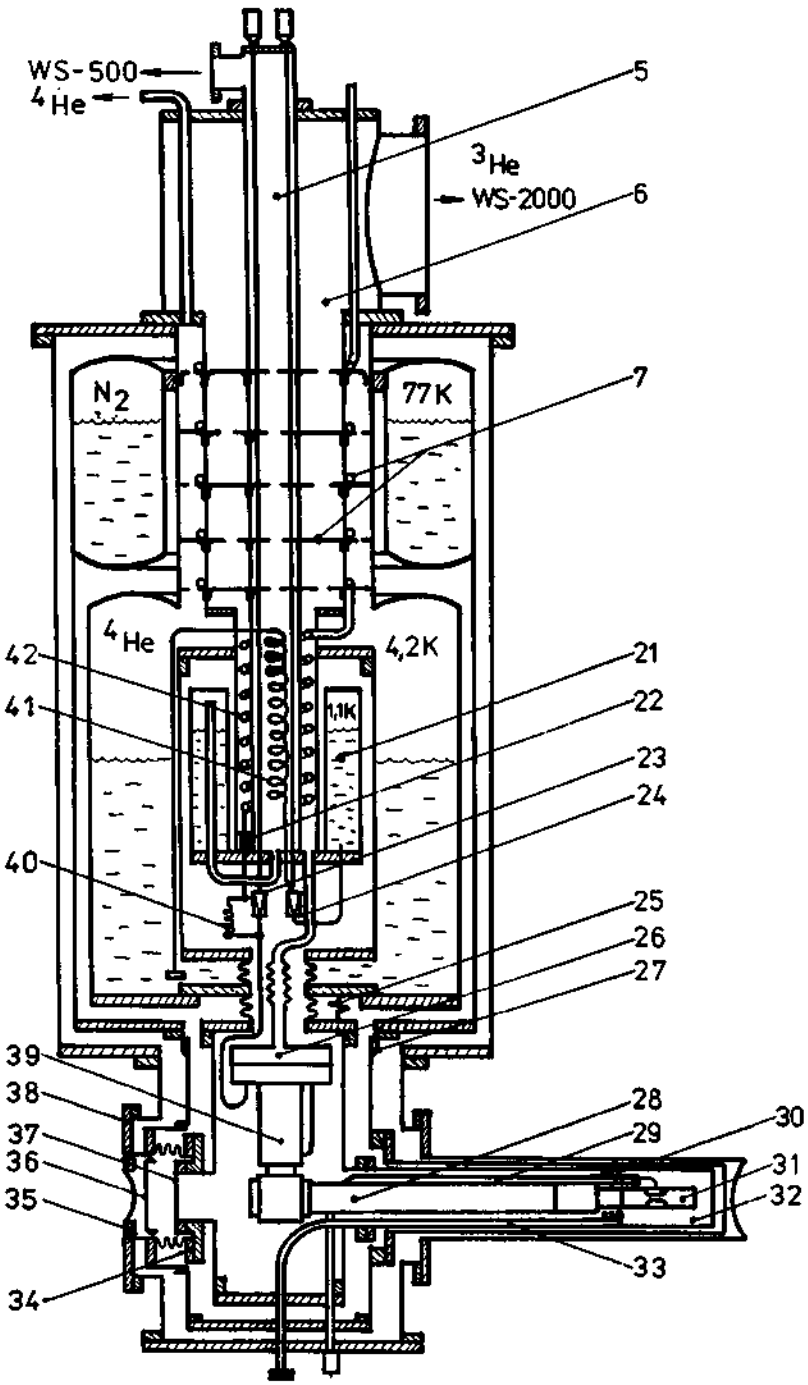

Fig. 2. Schematic view of the vertical cryostat of $^3He/^4He$ dilution refrigerator (see text for details).

consists of three easily disconnectable parts. The first part includes a still body (45), a preliminary heat exchanger (39) and a connecting assembly (57). The second part consists of a main heat exchanger (28) and a mixing chamber (31). The third part intended for sample loading and providing a solution channel is a long hollow barrel (56) of 30 mm diameter with screwed on its end a teflon ampule (53) containing a sample.

Introduction of the ampule into the equipment cooled down to liquid nitrogen temperature is performed through the lock chamber described earlier, using a special device which permits one to seal an indium gasket in vacuum. To provide a tightening with high reliability of this very important seal the flange and the connecting assembly are warmed inside the cold equipment in vacuum practically up to room temperature. For this purpose the loading device is provided with special channels for blowing compressed air.

^{3}He still is formed during the connection of the dilution step and the pumping unit, when the cover seals the still body (45) through the indium gasket, providing a plane box of 110 mm diameter and about 100 cm^3 volume. A heater (43) in a shape of a flat spiral of 0.08 mm diameter constantan wire wounded around and glued to a 2 mm diameter enameled copper wire is placed at approximately half of the still height. For temperature measurement a 10 Ω Speer carbon resistor is mounted on the heater body which, in turn, is fixed with respect to the still body by thin stainless steel supports. Such an arrangement allows one to determine a solution level position since at drainage the heater body overheats in a stepwise manner by several degrees. Then the work position of the level above the heater can be easily calculated.

The heat exchanger (44) for cooling of returning ^{3}He flow is mounted on a still bottom. The heat exchanger is a double flat spiral of 1.6 mm diameter capillary. The heat exchanger area is 150 cm^2 and its geometric impedance is 2.8×10^8 cm^{-3}.

A preliminary heat exchanger contacting the still bottom has a 60 mm external diameter and 150 mm long body, a hollow insert (58) and a double parallel helix coil pipe (46) for ^{3}He. The total area of the heat exchanger is 750 cm^2, the cross section area of a solution channel is about 0.4 cm^2, and the impedance of a ^{3}He channel equals 2×10^8 cm^{-3}.

This design has to satisfy most completely contradictory demands of the heat exchanger in qualitatively different regimes with high and low ^{3}He circulation rates.

At a relatively low ^{3}He circulation rate, of order of 1 mmole/s, a convection in the solution and a longitudinal heat conductivity in the channels are most negative factors for the heat exchanger under consideration.

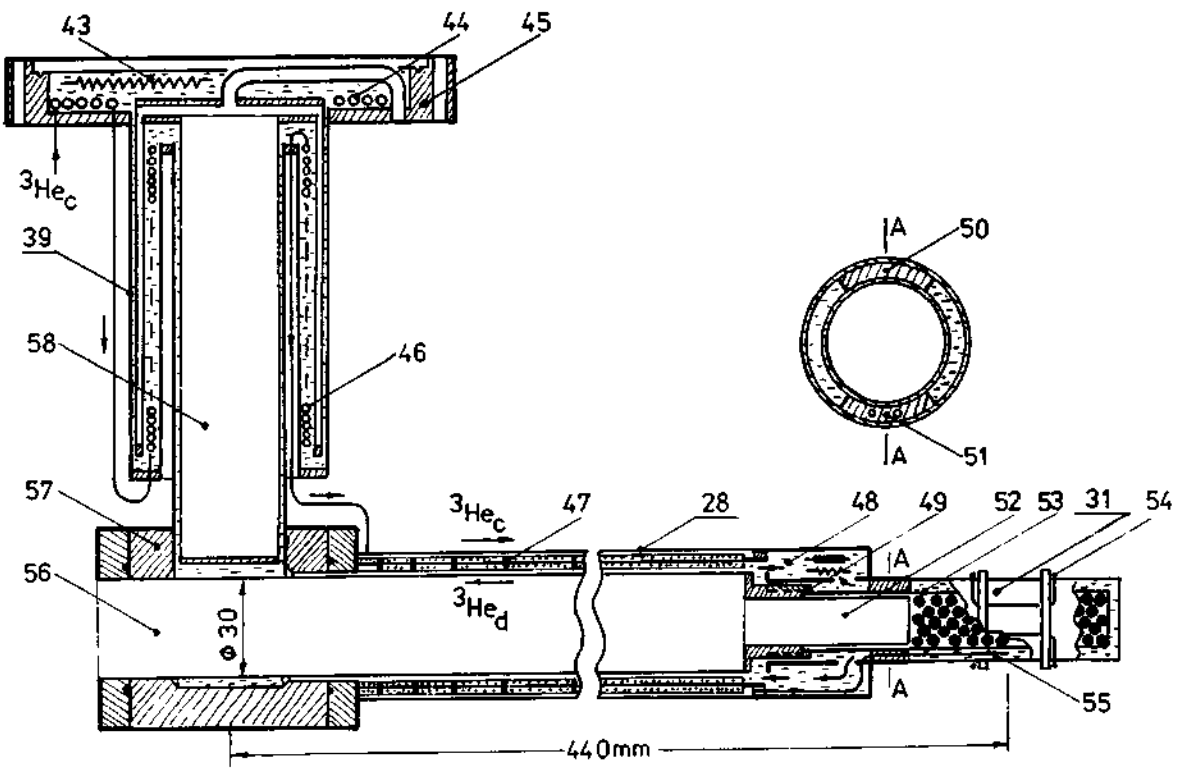

Fig. 3. Low temperature part of the dilution refrigerator (see text for details).

Since a flow in the solution at a part where coil pipes with concentrated ^{3}He are arranged is directed downwards, this design allows one to exclude the convection completely even at very low ^{3}He circulation rates. As it is known, the largest influence on heat exchanger efficiency is the thermal conductivity along the liquid ^{3}He channel and along the heat exchanging wall. In our design, as well as in Frossati-type heat exchangers, these factors are minimized. On the other hand, it is extremely difficult to exclude at least a partial influence of the thermal conductivity in the solution channel of a powerful refrigerator when it works in regimes with low ^{3}He circulation. Nevertheless, if one does not wish to cool ^{3}He in the preliminary heat exchanger below 0.16 K, then the thermal conductivity along the solution does not worsen the heat exchanger quality too much. As calculations and a direct examination show, a minimum temperature in our heat exchanger is attainable at a circulation rate of about 2 mmole/s. It should be pointed out that the choice of an annular solution channel geometry is not accidental since this geometry is preferable for obtaining both a low Reynolds factor and a small cross section area.

Connecting assembly (57) is a massive stainless steel body welded to the preliminary heat exchanger body and providing indium gasket sealing of both the main heat exchanger (28) and the barrel (56). A heater for fast removal of the ^{3}He/^{4}He mixture from the dilution step and a 220 Ω Speer resistance thermometer for temperature control are attached to the lower part of the connecting assembly.

The main heat exchanger (28), made of sintered copper powder with about 20 μm diameter grains, has a calculated area of about 4×10^4 cm^2. An annular channel for the solution between the walls of the heat exchanger and the barrel (56) has a width of about 0.5 mm. Cooled ^{3}He flows along the outer annular channel of about 0.1 mm wide. The heat exchanger design does not differ principally from that of the step heat exchanger described in ref. [2].

Copper powder is sintered on both the outer and the inner surfaces of a thin walled cupro-nickel tube of 36 mm diameter. These surfaces are preliminary galvanically covered by a thin copper layer. The sintering was performed in hydrogen atmosphere at 820°C in a special stainless steel matrix. The heat exchanger was sectioned by piles of thin stainless steel rings (47), which were inserted during a filling of the matrix by the copper powder. This technique provides isolated sections without additional treatment after the sintering. Isolation in the pile is ensured by a large number of successively connected Kapitza resistances between the rings.

For a more effective use the total heat exchanger area is distributed unevenly between the sections. As calculations show, even at a small number of the sections it is possible to ensure, using optimum area distribution, that the efficiency of the step heat exchanger will in practice not differ from that of an ideal continuous heat exchanger. In our case the section size increases about 15 times from the beginning to the end of the heat exchanger.

The arrangement of the mixing chamber and the measuring cell is shown in Fig. 3. ^{3}He cooled in the heat exchangers is fed through two 1.5 mm diameter capillaries (55) under a perforated 20 mm diameter ampule (53) containing a sample. Points of the ^{3}He feed are disposed at 1/4 and 3/4 of the ampule length. A lower annular sector (51) in the mixing chamber is intended for fastening of the ^{3}He capillaries, an upper sector provides a certain stock of ^{3}He in the upper part of the mixing chamber. Outside the mixing chamber teflon body (31) having a diameter of 27 mm a RF-circuit coil is placed. A flow of ^{3}He quasiparticles goes through the side annular channels, as shown in Fig. 3 (AA cross section). Then the flow is directed through windows, covered by a mesh against microwave irradiation, to the measuring cell (48) where a set of carbon resistance thermometers and an additional heater are placed, and further to the heat exchanger channel. A bottom of a spout of a hollow cylinder (52), upper and lower annular sectors, and a microwave choke (Fig. 2) form the back wall of the microwave cavity. A portion of the microwave power penetrates the side channels for the solution as far as the ampule threaded plug (49), and also through the annular clearance between the microwave choke and the mixing chamber (Fig. 2).

4. ^{3}He and ^{4}He pumping system; refrigerator control equipment

^{3}He circulation in the dynamic polarization mode ($\sim 10^{-2}$ mole/s) and during an experimental run ($\sim 2 \times 10^{-3}$ mole/s) is produced by a set of pumps WS-2000, WS-250 (Leybold) and H-2030 (Alcatel). ^{4}He pumping from the condensing bath is produced by pumps WS-500 and H-2060. An additional sealed pump D16B (Leybold) is used for ^{3}He/^{4}He mixture filling and removing operations.

As preparation to the work, vacuum tests and initial cooling are performed using vacuum-gas equipment disposed in the immediate vicinity of the target.

Helium cooling and filling of liquid helium, as well as control of all cryogenic equipment of the target, including the dilution refrigerator, are performed using a remote control panel placed in a control room.

A special multichannel automatic ohmmeter [3] is used for temperature measurements of various target units with the help of 15 carbon resistors of Speer, Allen-Breadley and TVO types. The control circuit of the device mounted on the remote panel is connected by an optically decoupled cable to a detecting circuit placed near the refrigerator. The device allows one to measure resistances of any six chosen carbon resistors in an automatic mode according to an operator program. Information is displayed on a digital display and recorded at the tape of a multichannel recorder.

This allows one to monitor slow processes and to file information about the refrigerator work. The accuracy of the temperature measurement is 0.5% at an input power to the thermometers in the 10^{-13}–10^{-14} W range (depending on the resistance).

Liquid helium levels in all the cryostats of the target are controlled by superconducting level meters. For a reliable filling of the condensing bath by liquid helium, distant control of the needle valve of this bath is used.

5. Magnetic system

The target magnetic system consists of two superconducting magnets with a "warm" field, intended respectively for the dynamic polarization mode and experimental runs in the neutron beam.

The polarizing magnet is a superconducting solenoid which is being put on the horizontal tailpiece of the refrigerator in the dynamic polarization mode, producing in the target volume a 2.7 T horizontal field with a uniformity no worse than 10^{-4}. A solenoid coil of 114 mm outer diameter, 75.2 mm internal diameter and 293 mm long contains 24400 turns of 0.4 mm diameter NbTi cable. A field correction is produced with the help of an internal projection of the coil having a length of 137.5 mm and a diameter of 78.4 mm. The internal diameter of a hole in the horizontal portion of the cryostat is 62 mm. The liquid helium stock in the vertical part of the cryostat is about 4 l which allows solenoid work for 12 h. For convenience of the work and for economical use of the liquid helium the solenoid coil is provided with a superconducting switch. The solenoid produces a field of 3 T at a current of 30 A.

The holding magnet is a superconducting dipole with a vertical field, placed stationary at the beamline, as shown in Fig. 4. The cryostat of the dipole is divided into two parts connected rigidly by two tubes, through which the helium vessel of the lower coil communicates with the upper main helium vessel. The distance between the upper and lower parts is 380 mm at a cryostat diameter of 400 mm, so that apertures are $\pm 50°$ in the vertical plane and 360° in the horizontal plane (neglecting the connecting tubes). The coils having an inner diameter of 255 mm, an outer diameter of 295 mm and a height of 150 mm contain 2155 turns of 0.8 mm diameter NbTi cable each. At a distance between coil centers of 500 mm and a current of 100 A a field as high as 0.25 T with a uniformity of about 1% is achieved within the target volume. 16 l liquid helium stock over the upper coil ensures magnet work for about 32 h. To decrease the liquid helium consumption the magnet is provided with a superconducting switch.

Manipulations with the magnetic fields in various modes are illustrated in Fig. 4. After achievement of a high nuclear polarization, using the dynamic technique, in a highly uniform 2.7 T field (Fig. 4.1) the solenoid current is decreased until a field induction of about 0.25 T is achieved. After that the field of the holding magnet is

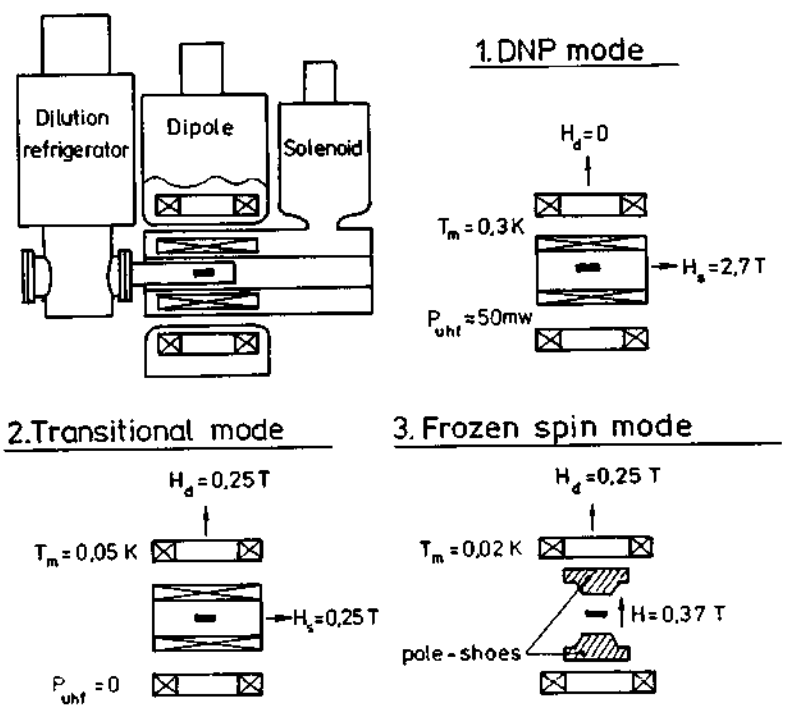

Fig. 4. Magnetic field configurations in different working modes of the target

increased up to the same value of about 0.25 T (Fig. 4.2). Then the solenoid field is decreased to zero, and the solenoid is moved firstly along the beam axis and further in a transverse direction to remove it from the vicinity of the target.

To increase the holding field for making the nuclear relaxation time longer a soft steel core is used to be introduced into the dipole gap. As a result the magnetic field increases approximately to 0.37 T without an essential decrease of the magnet aperture during an experimental run (Fig. 4.3).

The rate of the field manipulations is limited mostly by the permissible heat load of the refrigerator whose temperature was held within 0.05–0.1 K with a polarization loss of no more than 2%. A complete transition from the pumping mode to the experimental run takes usually about one hour.

6. Dynamic polarization and target polarization measurement

The main work with the target begins after loading of the ampule with a sample into the mixing chamber, as was described in section 2.

After this operation is finished complex vacuum tests of the refrigerator at about 80 K are carried out. Then the helium cooling and the filling of the refrigerator by liquid helium are performed, which usually takes about 4 h. $^3He/^4He$ mixture condensation and refrigerator starting take about 2 h. As a rule simultaneously with these operations cooling of the solenoid and its filling by liquid helium are carried out.

After introducing a magnetic field into the solenoid the mode of the thermal equilibrium signal measurement is started at a temperature of about 1.05 K in a field of 2.7 T. For this purpose the liquid mixture composition and the circulation rate are chosen in such a way that phase separation of the mixture, intensive cooling or temperature variations could not take place. The absence of a temperature difference between the mixing chamber and the target substance is extremely important in this mode.

1.2-propanediol with a paramagnetic Cr(V) impurity having a spin concentration of 1.5×10^{20} cm^{-3} (it was kindly provided by E.I. Bunyatova [4]) or with a stable HMBA complex [5] were used as a target substance.

The target polarization measurement is carried out using a Q-meter with an automatic phase control of resonance frequency of an input series-tuned circuit similar to one described in ref. [6]. The operating frequency of the Q-meter is about 114 MHz. A coil of the circuit contains two turns of silvered wire, whose axis is perpendicular to axes of the sample ampule and the beam. To connect the circuit to the Q-meter a specially prepared coaxial cable with a low heat conductivity was used. The cable was made of polished bronze wire of 0.5 mm diameter with tightly wound teflon ribbon, which was placed into a thin-walled neusilber tube and drawn through a die. As was indicated in section 2, to cool the cable in vacuum four thermal contacts are used. The connector of the cable is placed on a lower flange of the refrigerator cryostat. The electric length of the whole cable equals $\lambda/2$. A gated analog integrator is used to average NMR signals. The total error of the polarization determination does not exceed 3%.

After measurement of the thermal equilibrium signal the refrigerator is put into a large thermal load mode for irradiation of the target with microwave power. An ATT diode generator with an output power of 200 mW at a frequency about 75 GHz is used for the dynamic building up of polarization. The thermal load of the refrigerator in this mode equals 30–40 mW at a temperature 0.3–0.4 K in the mixing chamber and a ^3He circulation rate about 10 mmole/s. The time demanded to achieve 80% polarization is about one hour. Maximum values of the polarization obtainable at a longer building up time equal 93% and 98% for positive and negative polarizations respectively.

After the measurement of polarization is obtained the whole target complex is put into work mode in the neutron beam, as was described in section 5. The target temperature in the experimental working conditions is about 20 mK at a circulation rate of 2 mmole/s. A duration of one continuous experimental run at a given sign of a polarization equals about 10–12 h. Polarization degradation during this period is insignificant since the nuclear spin relaxation time in a magnetic field of 0.37 T is about 1000 h.

Table 1
Main parameters of the target

Target dimensions	
Volume	20 cm^3
Length	6 cm
Diameter	2 cm (cylindrical shape)
Target material	$C_3H_8O_2$ – 1.2-propanediol
Target mass	15 g
Cr(V) paramagnetic center concentration	1.5×10^{20} cm^{-3}
Maximum polarization P_{max}	+93%; −98%
Relaxation time (at 0.37 T and 20 mK)	positive: 1000 h, negative: 300 h
Time necessary for $P = 0.8P_{max}$ buildup	under 1 h
Total time for preparation the target from the beginning of cooling to an experimental run	24 h
Total consumption of liquid helium in the frozen mode	1.2 l/h

In conclusion, the main target parameters characterizing its quality are presented in Table 1.

Acknowledgements

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