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FROZEN SPIN POLARIZED DEUTERON TARGET 60 CM^3 IN VOLUME

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Polarization of frozen spin targets achieved by the dynamic nuclear polarization (DNP) is maintained in a comparatively low magnetic field ($B \leqslant 0.5$ T) for a long time /1/. Absence of strict requirements on the value and uniformity of the holding magnetic field opens up a new range of possible experiments.

Creation of such installations has become possible only with the use of $^3He/^4He$ dilution refrigerators, which permit one to cool substances of targets to very low temperature. On the other hand, choice of suitable target material with an optimal concentration of paramagnetic centres (needed for the DNP method) has resulted in obtaining an appropriately long nuclear spin-lattice relaxation time.

A frozen spin polarized proton target 60 cm^3 in volume was built in the Laboratory of Nuclear Problems in 1978 /2/ and successfully used in a π^--beam for measurements of the polarization parameter in a charge exchange reaction /3/. Construction of a deuteron modification of the target has nevertheless required essential improvements of the dilution refrigerator, investigations of target materials and development of apparatus for polarization measurement. This work accomplished, we have built a frozen spin polarized deuteron target, described in the present article.

1. General Description

The main part of the installation (Fig. 1) is a horizontal dilution refrigerator, which cools target material in two different operating modes: DNP process and frozen spin target operations.

The part of the refrigerator with target substance is placed in a gap of the electromagnet, its upper and lower halves being vertically movable.

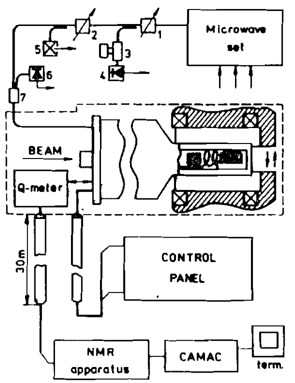

Fig. 1. Layout and block-diagram of the frozen spin target set up.

In the DNP mode both halves are closed and induction of 2.08 T, with homogeneity $2\cdot10^{-4}$ is achieved between the pole pieces to polarize the target. In the DNP mode a high cooling power up to 90 mW at a temperature about 0.3 K is employed to overcome microwave heating of target material.

After suitable nuclear polarization is achieved, the microwave irradiation is turned off and the refrigerator is transferred to the frozen spin mode, the target material being cooled down to 20 mK. At the same time the halves of the magnet move apart to provide appropriate solid angles for scattered particles. Induction of the holding field is about 0.4 T.

The design of the cryostat was described in detail in Ref. /2/. The basic design principles of the low temperature part of the dilution refrigerator will be discussed below.

The ^3He circulation system shown in Fig. 2 consists of two circuits. The first one is cascaded by WS-2000 [1], WS-500 [1], WS-150[1] Roots pumps and H-2030 [2] hermetic pump. The pumping speed about 1800 m^3/h is obtained, providing the circulation rate 20÷30 mmol/s necessary for the DNP mode. The second circuit includes BN-20000[3], WS-150 and H-2030 pumps and allows a pumping speed about 12600 m^3/h so that the 2 mmol/s circulation rate is obtained for the frozen spin mode. The pumping speed of the pump series in the ^4He precooling system is about 450 m^3/h.

The cryostat is rigidly clamped to a pipeline of the steam-oil pump by a vacuum seal flange and adjusted to the beam. Vibrations due to mechanical pump operation are damped by rubber bellows and amortisseurs, as schematically shown in Fig. 2.

Test and control facilities of the cryogenic set-up are put together in a control panel about 30 m off the experimental zone. The liquid nitrogen cooling trap purifies ^3He gas from oil fume and oil decomposition products as well as from a small amount of air penetrating through the walls of the rubber bellows.

Speer and Allen-Bradley resistors, placed in specific points of the refrigerator are applied as sensors of temperature. For temperature measurements a multichannel autorange alternating current ohmmeter has been designed /4/. The device provides continuous temperature registration in the sequence of six points, selected by an experimentator, with results being shown on the digital display and written on the paper tape of a multichannel recorder.

[1] Manufacturer: Leybold-Heraeus, GMBH KOLN.
[2] Manufacturer: Alcatel CIT, France.
[3] Steam-oil booster pump. (Osnovy vakuumnoi tekhniki, "Energiya", M., 1975).

Table

Parameters	Deuteron target	Proton target
1. Dimensions (diameter, length, volume)	d = 19.6 mm, l = 200 mm, V = 60 cm^3	
2. Target material	Ethanediol $(CD_2OD)_2$	Propanediol-1,2 $C_3H_8O_2$
3. Polarization (%)	$P_{\pm} = 37 \pm 2$	$P_+ = 90 \pm 3$ $P_- = 94 \pm 3$
4. DNP operating mode (temperature, cooling power, circulation rate, magnetic field induction)	T = 0.3 K, W = 90 mW $\dot{n} = 3 \times 10^{-2}$ mol/s, B = 2.08 T	
5. Polarizing time to reach 0.8 P_{max}	30 min	40 min
6. Polarization reversal time	90 min	120 min
7. Frozen spin target operating mode	T = 0.02 K, $\dot{n} = 2 \times 10^{-3}$ mol/s B = 0.4 T	
8. Nuclear spin-lattice relaxation time	$\tau_- = 300$ h $\tau_+ = 500$ h	$\tau_- = 800$ h $\tau_+ = 1200$ h
9. Liquid helium consumption	2.0 l/h for DNP operating mode 0.5 l/h for frozen spin target	

2. Preliminaries on DNP of Deuterium

Material for a frozen spin target must ensure both high polarization and long nuclear spin lattice relaxation time. Synthesis of

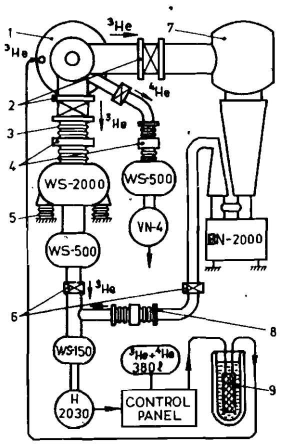

Fig. 2. Pump set.
1 - cryostat, 2 - valves of 200 mm in diameter, 3,8 - rubber bellows, 4 - intermediate weight, 5 - amortisseurs, 6 - valves - 80 mm in diameter, 7 - LN_2 (liquid nitrogen) cooled trap, 9 - LN_2 cooled trap for gas purification.

Cr(V)-complex on the basis of the fully deuterated ethanediol /5/ and propanediol-1,2 /6/ permitted us to obtain substances, which meet both requirements.

In preliminary investigations /6/ we obtained polarization of deuterons about 42% in 10 cm^3 volume samples of ethanediol and propanediol-1,2 with the concentration of Cr(V)-complex about $5 \cdot 10^{19}$ spins/cm^3. The spin lattice relaxation time was 15 h and 1 h at the temperature 54 mK and 80 mK respectively in the magnetic field 0.32 T. The results lead to a conclusion that the substances show promise as target materials although the temperature has to be lower in the frozen spin operation.

We have also established that dynamic cooling is the main DNP mechanism in the substances analyzed. This fact enables us to apply a convenient method of the polarization measurement based on the spin temperature conception /7/. The asymmetry /7/ of the line-shape deuteron magnetic resonance (DMR) spectrum may be used, i.e. it is not necessary to register a very weak DMR signal of thermal equilibrium.

In general, Cr(V)-complex in fully deuterated diols is found to be similar to the well-studied one in hydrogen diols. At present we have considerable experience in preparing these materials with an appropriate concentration, the results being reproducible.

The test results shown in the Table have been obtained for ethanediol, prepared, as usual, in the form of beads no more than 2 mm in diameter.

3. Dilution Refrigerator

The low temperature part of the dilution refrigerator is shown schematically in Fig. 3.

^3He gas is precooled to 4 K and transferred to the condenser 22 made of sintered copper powder and placed in the ^4He evaporating bath 13, volume 0.8 l, operating temperature 1.1-1.3 K. The level of liquid ^4He is regulated by a needle valve 14. Condensation pressure is controlled by a needle valve 24 and a capillary tube 23 of inner

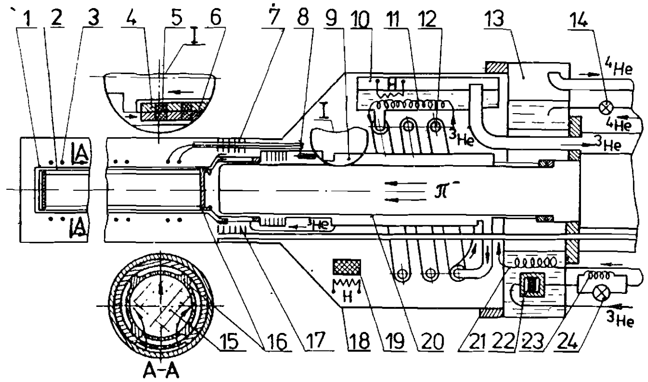

Fig. 3. The low temperature part of the dilution refrigerator.

diameter 0.2 mm and length 200 mm connected with the valve in parallel. The inpedance of the capillary is about $3 \cdot 10^9 cm^{-3}$ and additional flow is adjusted by the needle valve.

Cooled in the coil pipe 21, liquid ^3He passes through the still heat exchanger 11, which consists of a helix tube, with the total surface area $10^2 cm^2$, packed at the bottom of the still. Then ^3He passes through counterflow heat exchangers 9,12, to be introduced in the mixing chamber. The central part of the mixing chamber is a perforated container 16, filled with target material 15 cooled by heat absorption due to transition of the concentrated phase to the solution.

After entering into solution, ^3He is drived by an osmotic pressure gradient along solution channels of the counterflow heat exchangers to the still 10 and then evaporated by pumping.

The annular solution channel of the heat exchanger 9 is formed by an indium sealed can 20. The latter also separates the mixing chamber from the beam channel which is used as a loading tube. Loading of the target into the refrigerator is done in the following way. The beads of target material are enclosed in the container just before its inserting into the mixing chamber. When the refrigerator is precooled down to the liquid nitrogen temperature, the container is pushed in its place. Then a special holder with six socket wrenches is inserted together with the can. The indium joint tightened, the holder is taken out, and the beam channel is sealed by a rubber O-ring seal and pumped.

In the outlet of the mixing chamber there is a cell 8 with Speer carbon resistors to measure the temperature of the solution, the thermometers being protected against microwave irradiation by a quarter-wave choke and a metallic net. The mixing chamber, the heat exchanger and the still are very important parts of the dilution refrigerator. For this reason, below we shall discuss their design features and characteristics in detail.

Mixing Chamber

The mixing chamber (Fig. 3) consists of an outer jacket 1, an intermediate shell 2 with orifices and the perforated container 16, mentioned above.

Six orifices inject liquid ^3He from below to ensure uniform dilution over the volume of the target. A solution like that is unstable against heat convection to be known as a more powerful heat-exchange mechanism compared with heat conductivity. Both the uniform dilution and the heat convection seem to act jointly to provide homogeneity of temperature and adequate cooling. Good agreement between the measured cooling power and the one calculated for ideal isothermic dilution /8/ is a criterion of validity of the conclusion.

When ^3He is injected in the mixing chamber "from below" it is necessary to maintain an optimal total amount of the concentrated phase. Both excess and deficiency of ^3He inevitably lead to deterioration of refrigerator operation. To simplify the choice of the total amount of ^3He, the container is made in such a way that there is a closed buffer volume in the upper part of the mixing chamber (see Fig. 3). The concentrated phase is collected or spent so that inevitable fluctuations of the ^3He flow are considerably smoothed.

All the details of the mixing chamber are made of teflon, outer jacket being equipped with teflon-to-brass glued joint. We have not found a superleak after twenty cooldowns at any rate. General dimensions of the mixing chamber are as follows: the container has inner diameter and length 19.6 mm and 200 mm respectively, side gaps for solution output are about 1.5 mm; the mean thickness of jecket, shell and container walls is about 0.7 mm.

Heat Exchangers

The total heat exchange surface area of the counterflow heat exchangers is about $4 \cdot 10^4$ cm^2 to obtain temperature in mixing chamber 0.02K for the frozen spin mode only.

Such a large surface is not needed for the DNP mode /2,8/. For this case it is necessary to provide transport of ^3He through canals

filled with superflow solution without essential loss of osmotic pressure. It is also an important problem to prevent turbulent flow, it being associated with geometry of the solution channel. The latter can be made in the form of either sufficient amount of canals or a single annular one of a rather large diameter to keep a suitable total cross section area of the solution part. On the other hand, in the frozen spin mode, when circulation rate becomes insufficient, too large an area of the cross section may be a reason for deterioration of the heat exchanger efficiency because of growing axial heat conductivity.

In our refrigerator two counter-flow heat exchangers are used.

The first heat exchanger 12 in Fig. 3 is a stainless steel tube 12 mm in diameter, in which seven pairs of coaxial tubes 3.5 mm and 3.0 mm in diameter are inserted. Clearences between the coaxial tubes are ^3He canals. The inner tubes and the space between coaxial pairs are filled with the solution. The length of the heat exchanger is 400 mm and the surface area is about 500 cm^2.

The other heat exchanger consists of three coaxial tubes 500 mm in length. The middle thin-walled cupro-nickel tube 6 is coated outside and inside with sintered copper powder in the form of annular sections 4, as is shown in Fig. 3. Each section is 2 mm thick and 10 mm wide, the intervals between them being filled with teflon rings 5 2 mm wide. The estimated total surface area of the heat exchange is about $4 \cdot 10^4$ cm^2.

Still

The principal function of the still is efficient evaporation of ^3He. In the DNP mode heat power about 1 W must be emitted in the solution. For this reason the still heater must have as large surface as possible.

In our case a net-work heater is distributed over the still volume. It is assembled of components made of copper capillary tubes 2 mm in diameter with constantan wire glued inside. Their outer side is coated with sintered copper powder.

The still temperature is measured by a carbon resistor immersed in the solution. No measures are taken against superfluid film, penetrating the pumping pipe, because the flow speed is high enough in each mode.

Operation

The carbon resistors placed in specific points of the refrigerator for temperature measurement were calibrated in the following way. In the region up to 0.5 K it was done in a special set. In the lower temperature region the calibration was carried out by CMN susceptibility (with the magnetic field being absent) and by means of the proton magnetic resonance in the magnetic field of the DNP mode value. In the latter case we placed a distributed heater and propanediol beads in the mixing chamber. The beads were prepared as usual target material but strongly concetrated to have a conveniently short relaxation time. It also permitted us to choose optimal operating modes, which prevented overheating of the species, and to calibrate the microwave power.

The cooling power $\dot{Q}$ measured versus the mixing chamber temperature T at different temperatures of the still is shown in Fig. 4. The experimental points are seen to obey the curves, calculated for a continuous heat exchanger /8/, rather well up to 0.3K. At a higher temperature the experimental data deviate from the calculations. This fact may be explained by the turbulent flow, occuring in the solution channel of the heat exchanger. As a result, osmotic pressure reduced in the still, which leads to increase of ^{4}He concentration in the circulating gas mixture. This is qualitatively confirmed by direct measurements.

Nevertheless, we can achieve the cooling power of 90 mW at 0.3K, which is enough for the DNP operation of the 60 cm^3 target. The limit temperature obtained in the refrigerator is 16 mK for the magnetic field 0.45 T, and 14.7 mK without magnetic field. In the frozen spin target mode we obtain 20 mK solution temperature at $2 \cdot 10^{-3}$ mol/sec circulation rate for the heat loading from charged particles beam of $(3 \div 4) \cdot 10^5$ s^{-1}.

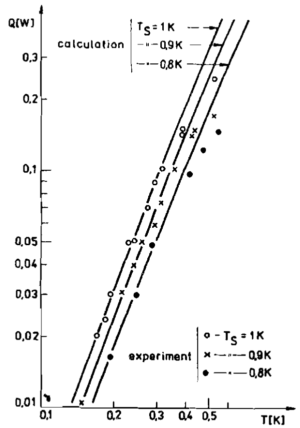

Fig. 4. The cooling power of the dilution refrigerator.

4. DNP and Polarization Measurement Apparatus

The microwave circuit is shown in the upper part of Fig. 1. The oscillator (diffraction-radiation generator) feeds the microwave system through the attenuator 1. For frequency measurement a wavemeter 3 and a detector diod 4 are used. The microwave power introduced in the

cryostat is measured by a bolometer 5 and controlled by a calibrated attenuator 2. There is a circular wave guide inside the refrigerator to reduce heating due to microwave attenuation. In view of this, two rectangular-to-round transitions are employed. One of them (7) is included as shown in Fig. 1 and the other is placed just in front of the wave-guide joint with the cavity. The latter is formed by the walls of the tail part of the 1 K-shell 18 and the choke 17 in Fig.3. The reflected microwave power level is measured by a diod 6. A phase-sensitive detector allows elimination of electron spin resonance signals, which essentially facilitates finding of DNP frequencies.

For deuteron target polarization measurement a Q-meter with a phase automatic frequency control (PAFC) of a measuring circuit is used /9,10/. The parallel circuit consists of a coil 3 (in Fig. 3), a coaxial cable 7 and a controlled capacitor at the Q-meter input. The coil and the coaxial cable are the same as for proton polarization measurement.

To obtain a DMR-spectrum, a Q-meter oscillator frequency sweep of 400 kHz with a 5 ms duration is applied. The central frequency is 13.6 MHz. The PAFC - system allows us to diminish baseline shifts, caused by mechanical vibrations of the measuring circuit, by a factor of about 30. The preliminary treatment of DMR signals is executed with CAMAC units controlled by the crate controller based on the I8080 microprocessor /11/. The Q-meter output signal passes through the calibrated attenuator and the amplifier and then comes to the analog-to-digital converter input /12/. Conversions are initiated by the strobe-generator (up to 256 strobes per sweep) after the microcomputer transfers the "ready" signal. Each digital equivalent of the analog voltage is stored in the buffer memory. At the sweep end the microcomputer receives data from the buffer memory and adds them to its own memory. When the given number of sweeps is executed (up to 65536), the result is averaged and may be output to the graphic display, the line printer and stored by the cassette tape recorder.

The software (written in the assembler language) allows controlling CAMAC modules (including interfaces of peripheral devices) correcting the baseline, calculating signal areas, etc.

The digitized DMR spectrum is introduced in the computer to calculate deuteron polarization. At first the experimental spectrum is transformed into a corresponding absorption line-shape, as described in Ref. 13. Then an iteration procedure /6/ is applied to fit the R parameter, which is equal to asymmetry ratio of the DMR spectrum and related to the polarization value through a simple formula.

5. Basic Characteristics

It takes usually 10 h to cool the refrigerator from room temperature to 4K, and load the target material. All the next stages of putting the set-up into operation up to beginning of the experiment are shown in Fig. 5.

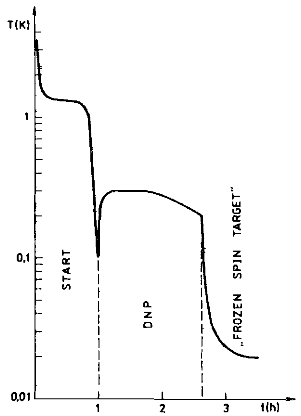

Fig. 5. Typical time curve of the target cooling from the beginning of ^3He-^4He gas mixture condensation.

After developing the deuteron polarized target, we have essentially improved the proton target parameters compared with those earlier reported /2/. The basic characteristics of the frozen spin target, which is used at the IHEP 70 GeV accelerator (Serpukhov) are summarized in the Table.

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Борисов Н.С. и др. E1-86-75
Замороженная мишень поляризованных ядер дейтерия объемом 60 см^3

Описана мишень с "замороженными спинами" поляризованных ядер дейтерия длиной 200 мм, 19,6 мм в диаметре и массой 44 г., охлаждаемая рефрижератором растворения большой мощности. Дано краткое описание низкотемпературной части рефрижератора. Более подробно обсуждаются вопросы, связанные с устройством камеры растворения, теплообменников и испарительной ванны. Рефрижератор способен поддерживать температуру 0,3К при тепловой нагрузке 90 мВт; самая низкая температура, получаемая в камере растворения,составляет 0,014К. Приводится общее описание аппаратуры и процедуры измерения поляризации. В качестве вещества мишени используется полностью дейтерированный этандиол с синтезированным комплексом Cr(V). Получены следующие результаты: поляризация ± 0,37 ± 0,02, время спин-решеточной релаксации отрицательной и положительной поляризаций 300 ч и 500 ч, соответственно, при температуре 0,02К и в магнитном поле 0,4 Т.

Работа выполнена в Лаборатории ядерных проблем ОИЯИ.

Препринт Объединенного института ядерных исследований. Дубна 1986

Borisov N.S. et al. E1-86-75
Frozen Spin Polarized Deuteron Target 60 cm^3 in Volume

A "frozen spin" polarized deuteron target (200 mm in lenght, 19.6 mm in diameter and 44 g in weight) cooled by the high cooling power dilution refrigerator is described. The low temperature part and operation of the refrigerator is outlined. The discussion of the mixing chamber, the heat exchangers and the still is somewhat more detailed. The dilution refrigerator produces 90 mW cooling power at 0.3K temperature; the lowest temperature that can be obtained is 0.014K. The general description of the apparatus and the procedure of the polarization measurement is made. Fully deuterated ethanediol with Cr(V)-complex synthesized is used as the target material. The following results are obtained: polarization is ± 0.37 ± 0.02, spin-lattice relaxation time of negative and positive polarization is 300 and 500 h, respectively, at 0.02K tempearture and 0.4 T magnetic field.

The investigation has been performed at the Laboratory of Nuclear Problems, JINR.

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