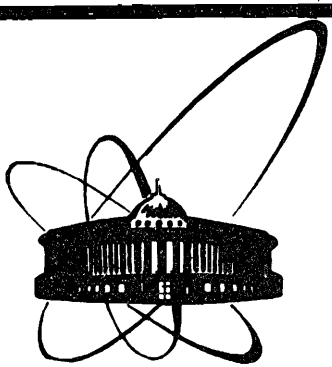

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E.I.Bunyatova, N.N.Bubnov*

SYNTHESIS OF CHROMIUM (V) COMPLEX IN DEUTERATED PROPANEDIOL FOR A TARGET WITH "FROZEN" POLARISATION OF DEUTERONS

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*EOCI Ac.Sci. USSR.

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Study of nucleon-nucleon interactions is interesting for hadron physics. Development of targets with "frozen" polarisation of hydrogen nuclei-protons - allowed solving many problems of strong interactions. Now the targets of this type have been made with polarised deuterium nuclei which are a weakly bound state of the proton and neutron.

Complex compounds of pentavalent chromium and deuterated ligands have found the widest use as an operating material for polarised deuteron targets. The most suitable technique for synthesis of such complexes is based on the reduction reaction of Cr(VI) compounds, mainly chromates and bichromates, in deuterated solvents, e.g. diols /1-11/. A sufficiently narrow line of the electron paramagnetic resonance (EPR) and practically isotropic g-factor are characteristic of the complexes like these. Their fast cooling results in glasses with homogeneous distribution of paramagnetic centres. All these properties allow a high deuteron polarisation.

Recently we have synthesised Cr(V) complexes in deuterated ethanediol /8/. The use of fully deuterated ethanediol $(CD_2OD)_2$ as the initial material allowed achieving a larger amount of deuterium in a complex which is only determined by the degree of deuteration of initially used ethanediol. The complex obtained was used as the target operating material. A 40% deuteron polarisation was achieved in a sample 60 cm^3 in volume at 0.3K in the magnetic field of 2.1T. Another important parameter of this operating material - the deuteron relaxation time which is $\sim$ 500 hours at T=20 mK and H=0.45T - allows a target to operate in the "frozen" mode, which significantly widens experimental capabilities of such polarized deuteron target /11/.

Thus the target operating material based on deuterated ethanediol comprises 17.3% by weight of deuterium. Protons only occupy 1.5% of possible hydrogen positions in this sample. Because of 4% of diol is spent for the production of the complex, the number of OD bonds is 92% of that of CD bonds.

Besides high concentrations of deuterium and high degree of nuclear polarisation, the target operating material must have the maximum

possible value of r which is the ratio of the number of polarised neutrons (or protons) to the number of all nucleons (neutrons and protons). For ethanediol r= 0.30, for propanediol following ethanediol in the homologous series of diols r=0.32. A higher concentration of neutrons in the target will allow increasing accuracy of particle scattering experiments and investigating reactions with small cross sections.

It has been also proved /1-4,9/ that dynamic polarisation of the majority of substances containing several nuclear spins leads to equality of their spin temperatures and not to equality of polarisation. Thus the deuteron polarisation obtained by the dynamic method will be of the order of 45% as compared to the proton polarisation which is close to 100%. Now the highest proton polarisation has been obtained in the Cr(V) complex compound in propanediol-1,2 $C_3H_6(OH)_2$ /2-7/.

To obtain the maximum polarisation and increase the deuterium concentration in the target operating material we have synthesised a Cr(V)-complex in fully deuterated propanediol-1,2. It should be noted that synthesis of the Cr(V) complex has been carried out in partly deuterated diol $C_3D_6(OD)_2$ so far since one considered it necessary to keep hydrogen atoms in hydroxyl groups for production of the complex /2-6/. To increase the amount of deuterium in the target material, the following method was suggested for its preparation: a high concentration of Cr(V)-complex in $C_3D_6(OH)_2$ was obtained, then this complex compound was dissolved by fully deuterated propanediol-1,2 $C_3D_6(OH)_2$ /3-6/. Ref. /12/ describes the working material of a polarised deuteron target prepared on the basis of fully deuterated propanediol by dissolving a stable Cr(V) complex - EHBA-Cr(V) - in it. We studied solutions of the analogous HMBA-Cr(V) complex by the EPR method and found out there were several different paramagnetic complexes in the solutions. The complexes were produced by the ligand exchange with the solvent /13/.

So we think that preparation of the target working material by synthesis of the Cr(V) complex in fully deuterated propanediol has advantages over the above methods.

Deuterated propanediol-1,2 $C_3D_6(OD)_2$ with the deuteration degree 98.3 atomic per cent and potassium bichromate $K_2Cr_2O_7$ were the initial compounds for our synthesis. $C_3D_6(OD)_2$ was analysed by the nuclear magnetic resonance method. It was established that hydrogen admixture is mainly contained in CD-groupd. The OD-group which takes part in produotion of the complex is deuterated by 99% of higher. The complex was synthesised as follows: a mixture of $C_3D_6(OD)_2$ and $K_2Cr_2O_7$ (weight

ratio 11:1) was stirred up by a magnetic stirrer at 80°C and lowered pressure in order to remove water produced in the diol oxidation reaction. In 75 minute time a Cr(V) complex compound of the necessary concentration were obtained. A detailed description of the synthesis procedure is given in Ref. /10/. The complex obtained was analysed by the EPR method. The width of the EPR line between the maximum slope points was $H=5\cdot10^{-4}$T at T=298K. This substance was tested as a target operating material where the dynamic polarisation process was investigated /9/. A 40% polarisation of deuterons was achieved. Cr(V) complexes have already been studied in Ref. /14-15/. Cr(V) complex in $C_3D_6(OD)_2$ have not been described yet. We were interested in the structure of the complex and we studied the EPR spectra. Specimens with low concentration of the complex were prepared. Beside the spectra of the deuterated complex, we also recorded for comparison spectra of Cr(V) in $C_3H_6(OH)_2$ with a low concentration of the complex. Fig. 1 shows the EPR spectra at room temperature. It was impossible to distinguish between hyperfine splitting and ligand protons even at 20-fold dilution by ethanol. Only the hyperfine interaction with nuclei of ^{53}Cr isotope (its natural concentration was 9.55%) was distinctly observed. Spectral parameters of Cr(V) complexes in non-deuterated propanediol are in good agreement with the described data /14/. In Fig. 2, one can see central parts of the spectra registered in the X-range at 77K. To find out how hyperfine splitting depends on changes in the nature of ligands, we diluted complexes with ethanol (1:20). These spectra are shown in Fig. 3. We did not observe hyperfine interaction. The table lists EPR parameters of the complexes.

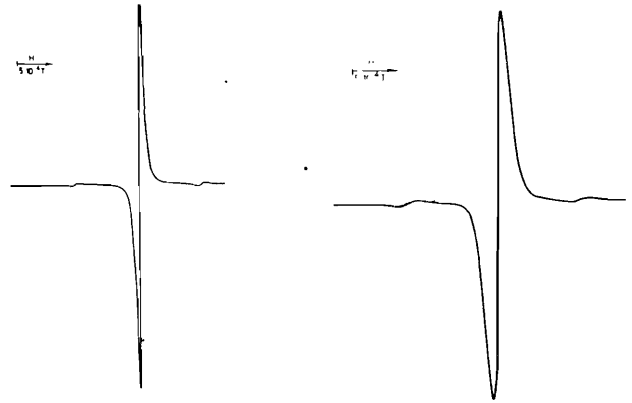

Fig. 1. EPR spectra of the Cr(V) complex at T = 298K. a - in $C_3D_6(OD)_2$; b - in $C_3H_6(OH)_2$.

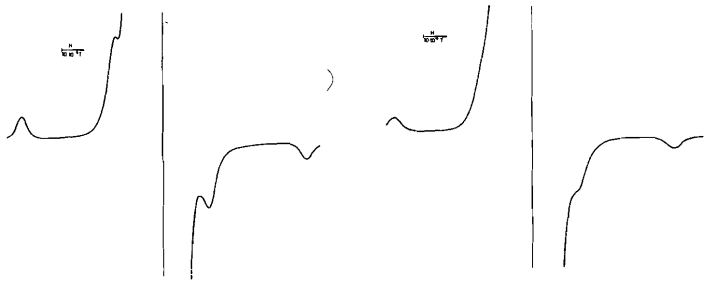

Fig. 2. Central parts of EPR spectra of Cr(V) complexes at T = 77K, a - in $C_3D_6(OD)_2$; b - in $C_3H_6(OH)_2$.

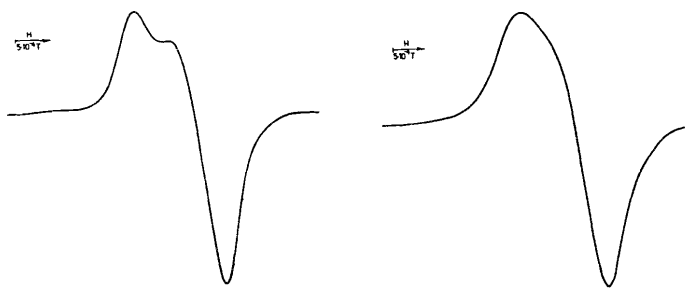

Fig. 3. EPR spectra of Cr(V) complexes at T = 77K. a - in $C_3D_6(OD)_2$; b - in $C_3H_6(OH)_2$. Solutions of the complexes are diluted with ethanol in the ratio 1:20.

The target operating material based on fully deuterated propanediol contains 19% by weight of deuterium. Protons occupy 1.7% of possible hydrogen position. To produce the complex, 4% of diol are necessary. The number of OD bonds in the target material is 60% of the number of CD bonds.

According to the theory of nuclear dynamic polarisation (NDP), the final temperature achieved by nuclear spins during NDP is inversely proportional to the EPR line width of paramagnetic inclusions responsible for NDP /16/. Because of a large error in measuring the concentration of paramagnetic inclusions by the EPR method, we failed to compare the EPR line widths of complexes of the same Cr(V) concentration with deuterated and non-deuterated ligands. Ref./2/ points out that the Cr(V) complex in $C_2D_4(OD)_2$ has a narrower line than the Cr(V) complex in $C_3H_6(OH)_2$ where proton polarisation can

Table

	T(K)	$g_\perp$	$g_\parallel$	g_{iso}	$A_\perp^{53}Cr$(cm^{-1})	$A_\parallel^{53}Cr$(cm^{-1})	A_{iso}(cm^{-1})
$K_2Cr_2O_7$ + deuterated propanediol	298	-	-	1.9817*			
	77	1.9778	1.9844	1.9812**	5.9×10^{-4}	35.3×10^{-4}	15.7×10^{-4}
$K_2Cr_2O_7$ - propanediol	198	-	-	1.9796*			
	77	1.9768	1.9845	1.9793**	5.7×10^{-4}	35.6×10^{-4}	15.7×10^{-4}

* - Experimental values

** - Calculated values. $g_{iso} = \frac{1}{3}(2g_\perp + g_\parallel)$.

amount to 98-99%. It is also known that the EPR line width of the Cr(V) complex in propanediol is 10% smaller than that of the similar complex in ethanediol /2/. Consequently, the temperature of nuclear spins in the Cr(V) complex in $C_3D_6(OD)_2$ can be the lowest one, while polarisation can be at its maximum.*

Finally we note that the Cr(V) complex in fully deuterated propanediol-1,2 is now the most suitable operating material for a deuteron target with "frozen" polarisation.

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References

1. W.De Boer, M.Borghini, K.Morimoto et al. J.Low Temp.Phys., 15 (1974) 249.
2. W. De Boer, CERN, 74-11, Geneva (1974).
3. T.O.Niinikoski. Conf.Proc.Polarised Beams and Targets. Argonne (1976) 458.
4. K.Guckelsberger and F.Udo. Nucl.Instr.and Meth., 137 (1976) 415.

*In June, 1986 on the IHEP accelerator (Serpukhov) a target with a "frozen" deuteron polarization has been put into operation based on the complex described here. Maximum value for deuteron polarization was 42% for the 2.1T magnetic field.

5. M.Babou, C.Bruneton, J.Bystricky et al. Nucl.Instr.and Meth., 160 (1979) 1.
6. A.R.Cash, S.F.J.Cox, J.Dawson et al. J.Phys.E: Sci.Instr. 13 (1980) 182.
7. N.S.Borisov, E.I.Bunyatova, Yu.F.Kiselev et al. JINR 13-10257, Dubna (1976).
8. E.I.Bunyatova and N.N.Bubnov. Nucl.Instr.and Meth., 219 (1984) 297.
9. N.S.Borisov, E.I.Bunyatova, M.Yu.Liburg et al. Zh.Eksp.Teor.Fiz., 87 (1984) 2234.
10. E.I.Bunyatova and N.N.Bubnov. JINR 1-85-929, Dubna (1986).
11. N.S.Borisov, E.I.Bunyatova, M.Yu.Liburg et al. JINR 1-85-292, Dubna (1985).
12. O.Hamada, S.Hiramatsu, S.Isagawa et al. Nucl.Instr.and Meth., 189 (1981) 561.
13. S.A.Luchkina, R.M.Galimov and E.I.Bunyatova. JINR 12-82-732, Dubna (1982).
14. E.J.Derouane and T.Ouhadi. Chem.Phys.Lett., 31 (1) (1975) 70.
15. M.Mitewa et al. Inorg.Nucl.Chem.Lett., 11 (1975) 793.
16. A.Abragam and M.Goldman. Rep.Prog.Phys., 41 (1978) 395.

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Бунятова Э.И., Бубнов Н.Н. E12-86-529

Синтез комплекса Cr(V) в дейтерированном пропандиоле для мишени с "замороженной" поляризацией дейтронов

Создана дейтронная поляризованная мишень с "замороженной" поляризацией. С целью повышения содержания дейтронов в рабочем веществе и получения максимальной поляризации дейтронов синтезирован комплекс пятивалентного хрома с лигандами на основе полностью дейтерированного пропандиола - 1,2.Описан синтез и исследования полученного комплекса методом ЭПР.

Работа выполнена в Лаборатории ядерных проблем ОИЯИ.

Препринт Объединенного института ядерных исследований. Дубна 1986

Bunyatova E.I., Bubnov N.N. E12-86-529

Synthesis of Chromium (V) Complex in Deuterared Propanediol for a Target with "Frozen" Polarization of Deuterons

The deuteron polarized frozen spin target was developed. To reach higher deuteron content and maximum polarization, the chromium (V) complex with ligands on the basis of fully detuerated propanediol - 1,2 was synthesized. The synthesis and the EPR investigation is described.

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

Preprint of the Joint Institute for Nuclear Research. Dubna 1986