SIZEABLE PROTON POLARIZATIONS IN FROZEN ALCOHOL MIXTURES

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We present here preliminary results of a work which is part of a research programme for high proton polarizations in highly hydrogenated substances. The method of polarization was the "solid effect". The samples were ethanol-water, ethanol-methanol, and ethanol-propanol mixtures doped with porphyrexide, a free radical with a formula $(CH_3)_2CN(:O)C(:NH)NHC:NH$ [1]. The mixtures were saturated at room temperature with about 3 % by weight of porphyrexide. The experiments were carried out in a field of 25 kG, at temperatures around 1.05° K, obtained in a continuous flow cryostat [2] ; the cooling time from room temperature to liquid helium temperature was 55 minutes. The samples were contained in a rectangular holder made of copper, 3.5 x 7 x 14 mm, closed on one side by a teflon window 3 x 6 mm. This holder was located inside a 18 cm^3 copper cavity filled with helium, and connected to a 20 W carcinotron oscillating at 70 Gc/s. The polarization was measured by NMR as in the Saclay-CERN polarized targets [3], the coil being immersed in the samples and care being taken not to saturate the NMR signals.

Maximum polarizations of 35 ± 2 % were obtained for the lowest attainable temperature, with a reduced microwave power of about 500 mW. The polarization depends markedly on the concentration of the various mixtures, as shown is figures 1, 2 and 3. Figure 4 shows the melting point of ethanol-water mixtures : a correlation seems to exist between the melting point and the obtained polarizations, although we are not able now to understand why. No attempt has yet been made to observe the electronic resonance line of the frozen free radical : from the known over-all hyperfine splitting (30 gauss) and the anisotropy of the g-factor, we estimate its width to be between 60 and 90 gauss. No attempt has been

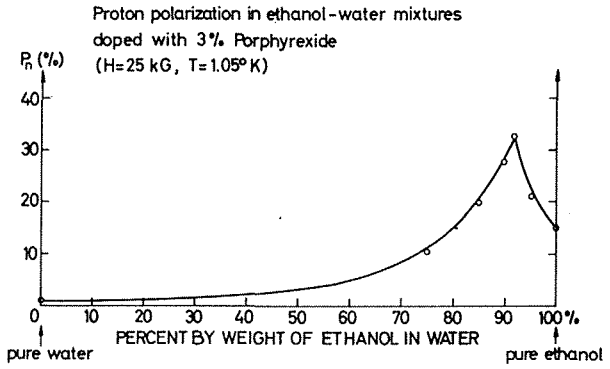

Fig. 1

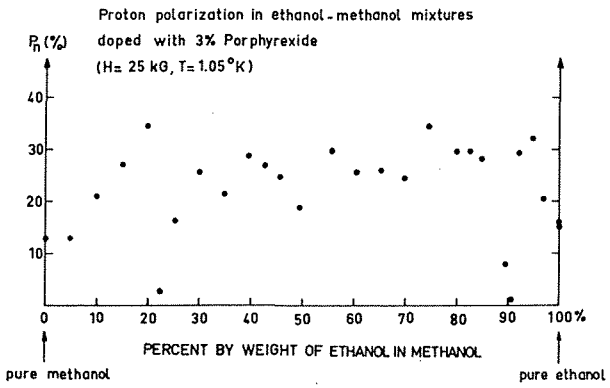

Fig. 2

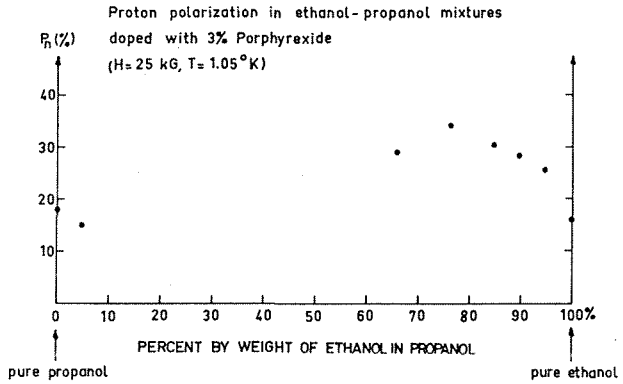

Fig. 3

made either to take off oxygen gas possibly dissolved into the samples. The nuclear relaxation times were of 3 to 7 mn in samples without free radical, were a little longer with a small amount of it, and of about 2 mn at 1.05° K with its maximum concentration. The polarization times ranged between 5 and 10 s for the optimum polarizations.

Results obtained in other samples with various free radicals are given in table I. Polarization and nuclear relaxation time measu-

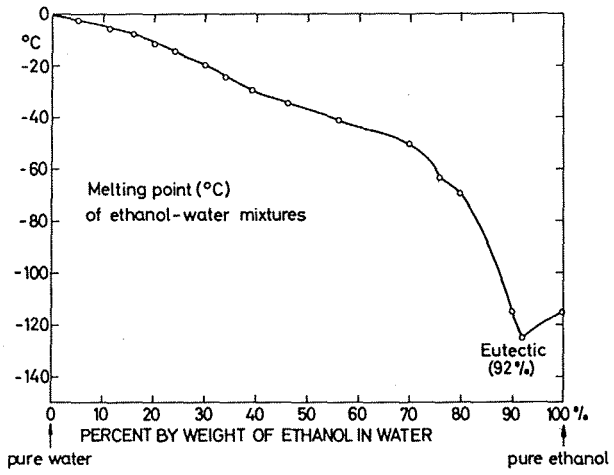

Fig. 4

Table 1

Some preliminary results on proton dynamic polarizations in organic compounds containing free radicals*)

Compound		Free radical**)														
		DPPH			PR			BPA			PB			PAC		
	***)	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3
Benzene	C_6H_6	5	2%	1					×)							
Toluene	C_7H_8	2	23%	12				4	15%	4						
m-Xylol	C_8H_{10}							4	10%	2						
Isodurol	C_9H_{12}	4	5%	1												
Tetrahydrofuran	C_4H_8O	2	4%	1												
2,5 Dimethyl-tetrahydrofuran	$C_6H_{12}O$							2	17%	1						
Diethylether	$C_4H_{10}O$	4	9%	3												
Methanol†)	CH_4O				3	13%	3									
Ethanol†)	C_2H_6O				4	16%	5									
Propanol†)	C_3H_8O				3	18%	2									
Plexiglas (solvent)	$[C_5H_8O_2]_n$	3	14%	7							10	10%	6	4	22%	8
		Chloroform									Chloroform			Benzene		
Polystyrene (solvent)	$[C_8H_8]_n$							4	20%	1				5	23%	1
								Toluene						Benzene		
Polyisobutylene (solvent)	$[C_4H_8]_n$				6	7%	1									
					Tetrahydrofuran											
Polymerized BPA									30%							

*) Magnetic field 25 kG, temperature 1.05°K.

**) DPPH = 1,1-diphenyl 2-picryl-hydrazyl; PR = Porphyrexide; BPA = 1,3-Bisdiphenylene 2-phenyl-allyl, PB = Porphyrindene; PAC = Picryl-N-amino-carbazyl.

***) Col. 1: radical concentrat. in % by weight; col. 2: max. polarizations; col. 3: No. of samples with varied concentrat.

×) With 4% BPA + 0.3% DPPH in toluene, a polarization of 20% was obtained.

†) See Figs. 1, 2 and 3 for mixtures of alcohols.

rements in frozen toluene containing DPPH are shown on figures 5 and 6, together with the results of Wagner and Haddock [4]. Figure 7 shows the peculiar behaviour of the polarization of M-Xylol versus the microwave frequency.

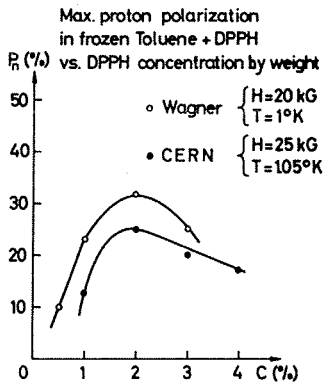

Fig. 5

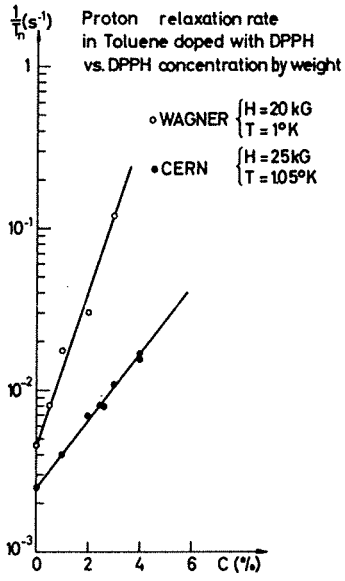

Fig. 6

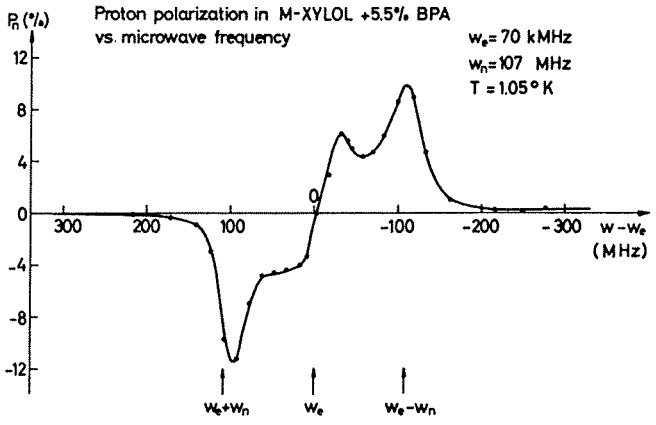

Fig. 7

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