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Potential Measurements for Selectivity Determinations of Polycarbonate Membranes Bounded by MgCl$_2$ Solutions

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Abstract

The emf of the cell chloride selective electrode/MgCl$_2$(N_1)/membrane/MgCl$_2$(N_2)/chloride selective electrode, containing a polycarbonate membrane, has been measured at 20, 30, 40, and 50°C. The membranes studied had pore diameters ranging from 5.0 to 0.03 μm, and bounding solutions concentrations vary from 2×10^{-4} to 10^{-1} equiv/dm^3. Apparent transport numbers of cation and permselectivities, deduced from emf values, are given. Results show that polycarbonate membranes are permselective to Mg^{2+} when the concentration of bounding solutions is low.

INTRODUCTION

The efficiency with which a membrane transports selectively any particular ionic species may be inferred (*1*) by measuring the transport number of the species in the membrane.

Membrane potentials measured using normal concentrations N_1 and N_2 on either side of the membrane may be used to derive an average transport number. If reversible electrodes with respect to the Cl$^-$ ion immersed in two chloride solutions are used, the cation transport number, t_+^{app}, is derived (*2*) from

$$dE_c = -\frac{\upsilon_+ + \upsilon_-}{\upsilon_+ \upsilon_-} \frac{RT}{F} t_+^{\text{app}} \, d \ln a \qquad (1)$$

where E_c is the emf of the membrane cell, F is the Faraday, a is the mean

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ionic activity of the electrolyte, and υ_+ and υ_- are the cation and anion stoichiometric coefficients, respectively.

The derived transport number value has been called the apparent transport number because in this type of measurement water transport has not been taken into account. This apparent value will be close to the true value when very dilute solutions are used (*1*).

In order to devise a quantitative measurement of t_+^{app}, it is necessary to bear in mind that (*3*) it is not technically possible to stir bulk solutions very vigorously against the surfaces of a thin porous membrane without exaggerating pore end effects or even pulsing solutions right through the pores. In order to include the effects of restricted stirring, it is considered that the membrane is bounded by two Nernst diffusion layers. The membrane system formed by the membrane and the two diffusion layers flanking it is simulated (*4*) by a fictitious membrane. The purpose of this paper is to study the ionic permselectivity of this membrane system, denoted ms.

Since in our experiments we have derived apparent transport numbers by varying the concentration in the two electrode chambers, from Eq. (1) we have (*5*):

$$t_+^{\mathrm{app}}(\mathrm{ms}) = \frac{\upsilon_+\upsilon_-}{\upsilon_+ + \upsilon_-} \frac{F}{RT} \left[\frac{\partial E_c}{\partial \ln a_1/a_2} \right]_{p,T,a_1} \tag{2}$$

where p is the hydrostatic pressure, a_1 and a_2 are the mean ionic activities of the solutions of concentrations N_1 and N_2 (calculated from the Stokes-Robinson hydration model (*6, 7*)), and E_c is measured by taking electrode 1 as the reference. t_+^{app} (ms) represents some average value of transport number for the complete junction (the membrane system) between the two bounding solutions.

Permselectivity of the system membrane P_s(ms) (which is the measure of the system membrane selectivity for the counterions over the co-ion) was then calculated (*8*) using the relation

$$P_s(\mathrm{ms}) = \frac{t_+^{\mathrm{app}}(\mathrm{ms}) - t_+}{1 - t_+} \tag{3}$$

where t_+ is the transport number of the cation in the free solution.

EXPERIMENTAL

Six different types of Nuclepore microporous membranes of polycarbonate have been used, with pore diameters (catalog data) of 5, 2, 0.8, 0.4, 0.1, and 0.03 μm. We shall denote them by N5, . . . , N003, respectively.

The water used in solution (double-distilled, deionized, and deaired by boiling) had a specific conductivity of the order of 10^{-6} Ω^{-1} cm^{-1}. The $MgCl_2$ (Baker Analyzed Reagent) used had a purity of 99.5%.

The measurement cell (Fig. 1) is formed by the membrane holder and two symmetrical compartments (R) which contain the solutions at both sides of the membrane holder. The exposed area of the membrane in the holder is 7.5 cm^2. Each of the compartments R has an internal volume of 700 mL.

Before use, the membrane was immersed for 12 h in a 2×10^{-4} N solution. In this way the potential difference E_c was constant when the stirring (200 rpm) and the fluid circulation (100 mL/min) from either 5-L Mariotte bottle R′ was established, while the concentrations N_1 and N_2 were kept constant. In order to check the equilibrium of the hydrostatic pressure on both sides of the membrane, each half-cell was furnished with a vertical capillary tube, T. The outputs of the circulation circuit were adjusted so that the menisci of the two vertical tubes were of equal height.

The potential difference E_c was measured by two ORION-94178 solid-

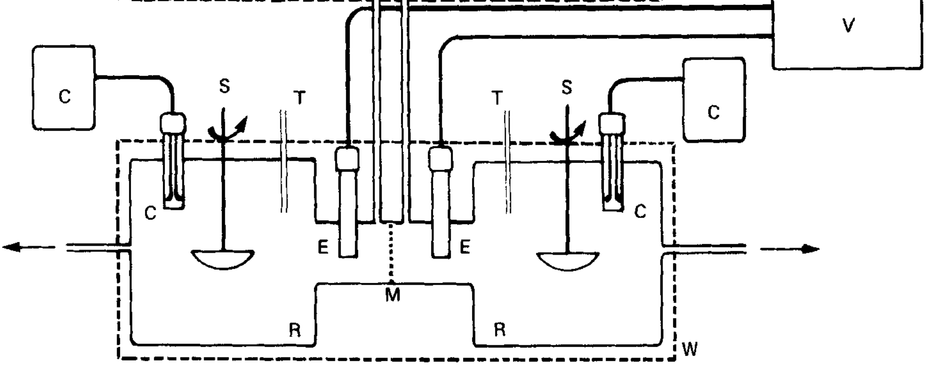

FIG. 1. Schematic representation of measurement system.

state chloride-selective electrodes, E, connected to a HP-3456A digital voltmeter, V, coupled to a HP-5150A thermal printer, I, with a timer.

All the measurements have been corrected for electrodes asymmetry. Three series of measurements were carried out with each of the membranes and at each temperature:

Series I:	$N_{1\text{fix}} = 10^{-3}$,	$2 \times 10^{-4} < N_{2\text{var}} < 10^{-3}$
Series II:	$N_{1\text{fix}} = 10^{-2}$,	$10^{-3} < N_{2\text{var}} < 10^{-2}$
Series III:	$N_{1\text{fix}} = 10^{-1}$,	$10^{-2} < N_{2\text{var}} < 10^{-1}$

The temperature control (±0.1°C) was maintained with two water thermostats, W, having a proportional temperature controller.

RESULTS AND DISCUSSION

The most representative results obtained for E_c versus ln (N_1/N_2) are reported in Figs. 2-4. They show that E_c depends on the membrane structure, the system temperature, and not only on N_1/N_2 ratio, but even on the concentration level N_1. The fitting of E_c vs ln (a_1/a_2) is a straight line for many cases; for others, a satisfactory fitting was found by means of a second degree equation.

The fitted functions $E_c = f(\ln (a_1/a_2))$, corresponding to every membrane, temperature, and series of concentration, are reported in Tables 1-3. These tables also report the number of experimental points (n) utilized for every fitting and their standard deviation (σ). The cation apparent transport numbers, t_+^{app} (ms), obtained from Eq. (2), are shown. Tables 4, 5, and 6 show the values of P_s(ms) for an N_2 value belonging to concentration Series I, II, and III, respectively.

The results of P_s(ms) show the cationic selectivity of the polycarbonate membranes. The cause of this selectivity is presumably the adsorption of the anions of solution on the pore walls (*3*).

The behavior of the membrane is closely linked to its structure. The fixed anions on the pore surface exert an electrostatic influence on the ions of the electrolyte so that a diffuse electric double-layer is formed in the pores (*9*). The effective thickness of the diffuse double-layer strongly depends on the concentration of the electrolyte. By a change in the pore size, or by a change in the diffuse electric double-layer thickness, the membrane properties can change from those of a Donnan-type membrane (the co-ions are completely absent from the pores and only the cations mediate the electric contact between both solutions), to those of a permselective membrane (the co-ions penetrate into the pores and the

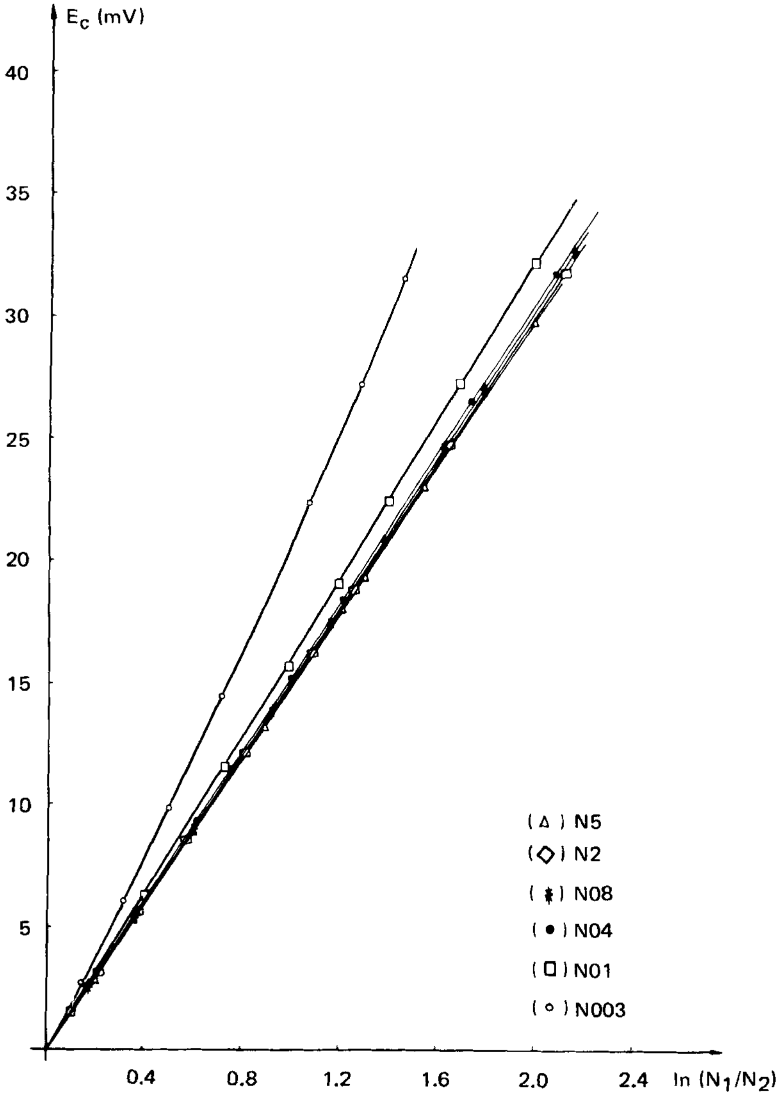

FIG. 2. E_c (mV) versus ln (N_1/N_2) at 20°C for different membranes. $N_{1\text{fix}} = 10^{-2}$, $10^{-3} < N_{2\text{var}} < 10^{-2}$.

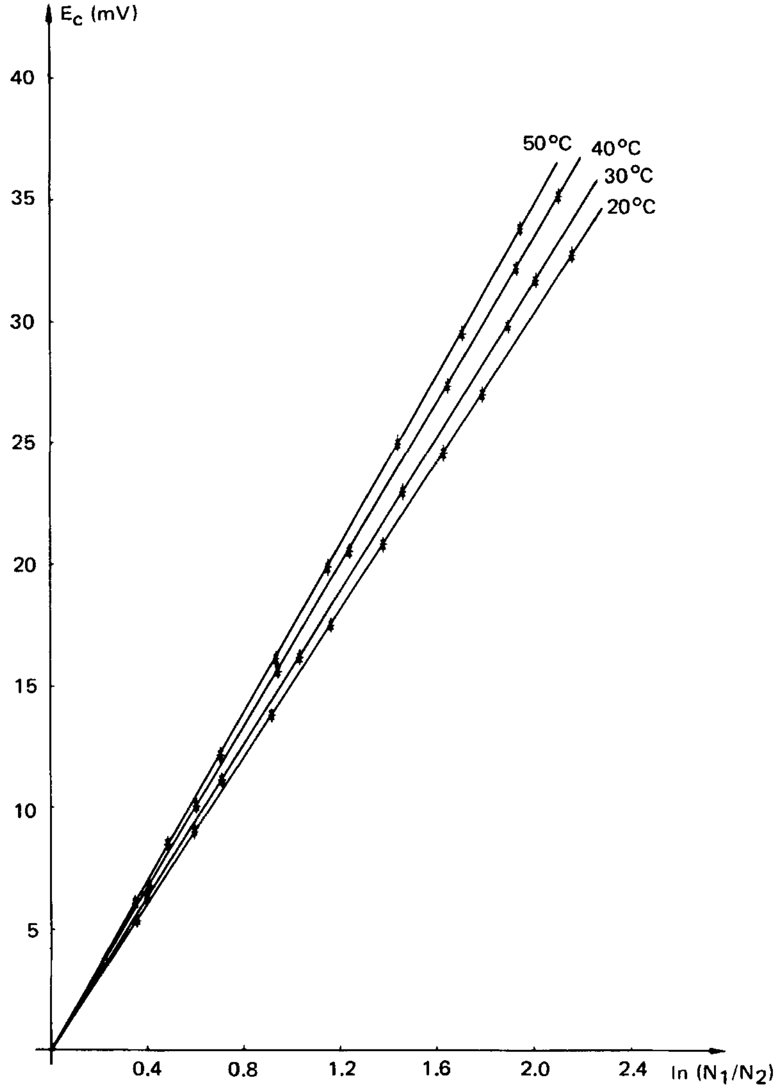

FIG. 3. E_c (mV) versus ln (N_1/N_2) for the N08 membrane at different temperatures. $N_{1\text{fix}} = 10^{-2}$, $10^{-3} < N_{2\text{var}} < 10^{-2}$.

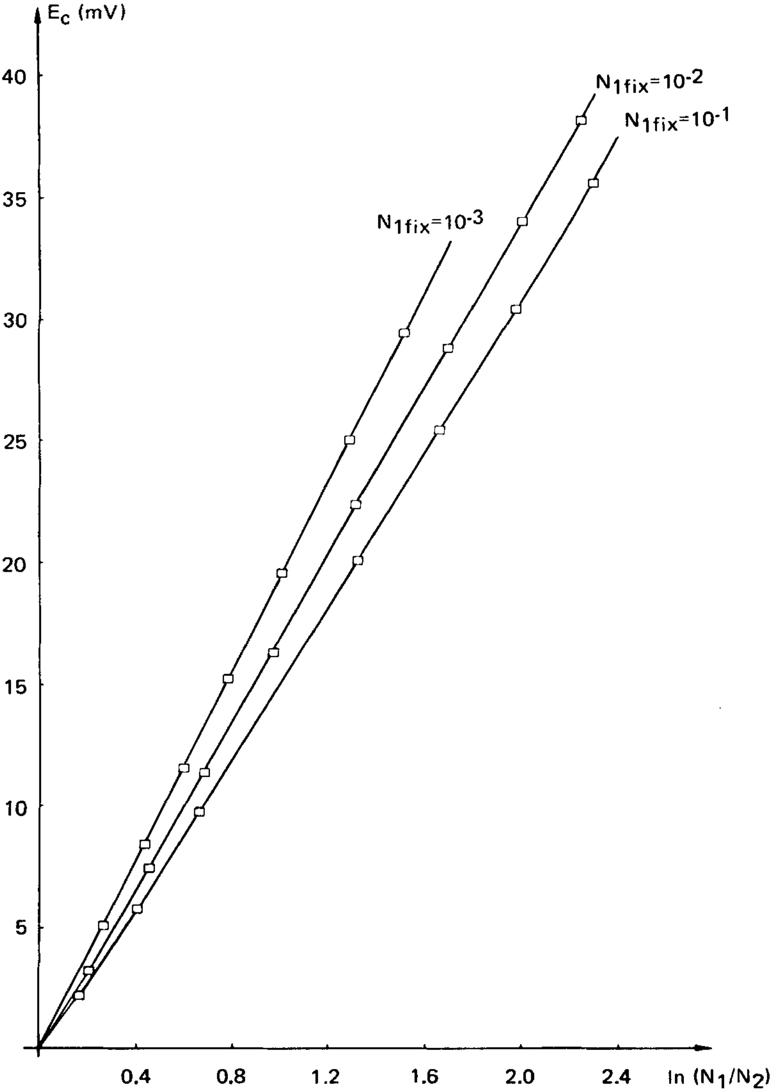

FIG. 4. E_c (mV) versus ln (N_1/N_2) at 30°C for three series of concentrations. Results are for the N01 membrane.

TABLE 1

Results Obtained for E_c (mV) and t_+^{app}(ms) at Different Temperatures. $N_{1fix} = 10^{-3}$, $2 \times 10^{-4} < N_{2var} < 10^{-3}$. Here, $x = \ln(a_1/a_2)$

	20°C	30°C	40°C	50°C
N5	$E_c = 15.39x$ $n = 6; \sigma = 0.07$ t_+^{app}(ms) = 0.406	$E_c = 16.18x$ $n = 6; \sigma = 0.08$ t_+^{app}(ms) = 0.413	$E_c = 16.90x$ $n = 6; \sigma = 0.09$ t_+^{app}(ms) = 0.421	$E_c = 17.28x$ $n = 5; \sigma = 0.06$ t_+^{app}(ms) = 0.417
N2	$E_c = 15.70x$ $n = 6; \sigma = 0.08$ t_+^{app}(ms) = 0.415	$E_c = 16.18x$ $n = 6; \sigma = 0.08$ t_+^{app}(ms) = 0.413	$E_c = 16.64x$ $n = 6; \sigma = 0.14$ t_+^{app}(ms) = 0.415	$E_c = 17.46x$ $n = 6; \sigma = 0.09$ t_+^{app}(ms) = 0.422
N08	$E_c = 16.04x$ $n = 5; \sigma = 0.11$ t_+^{app}(ms) = 0.427	$E_c = 16.77x$ $n = 5; \sigma = 0.10$ t_+^{app}(ms) = 0.432	$E_c = 17.37x$ $n = 6; \sigma = 0.07$ t_+^{app}(ms) = 0.433	$E_c = 18.14x$ $n = 5; \sigma = 0.06$ t_+^{app}(ms) = 0.438
N04	$E_c = 16.31x$ $n = 7; \sigma = 0.06$ t_+^{app}(ms) = 0.434	$E_c = 16.75x$ $n = 6; \sigma = 0.08$ t_+^{app}(ms) = 0.431	$E_c = 17.60x$ $n = 6; \sigma = 0.22$ t_+^{app}(ms) = 0.438	$E_c = 18.06x$ $n = 6; \sigma = 0.12$ t_+^{app}(ms) = 0.436
N01	$E_c = 18.70x$ $n = 6; \sigma = 0.17$ t_+^{app}(ms) = 0.497	$E_c = 19.55x$ $n = 7; \sigma = 0.08$ t_+^{app}(ms) = 0.503	$E_c = 20.56x$ $n = 6; \sigma = 0.16$ t_+^{app}(ms) = 0.512	$E_c = 20.93x$ $n = 7; \sigma = 0.25$ t_+^{app}(ms) = 0.505
N003	$E_c = 0.17x^2 + 29.02x$ $n = 7; \sigma = 0.04$ t_+^{app}(ms) = $0.10x + 0.770$	$E_c = 0.24x^2 + 29.81x$ $n = 7; \sigma = 0.04$ t_+^{app}(ms) = $0.012x + 0.765$	$E_c = 0.30x^2 + 30.20x$ $n = 7; \sigma = 0.08$ t_+^{app}(ms) = $0.015x + 0.750$	$E_c = 0.020x^2 + 31.61x$ $n = 5; \sigma = 0.10$ t_+^{app}(ms) = 0.761

TABLE 2

Results Obtained for E_c (mV) and t_+^{app}(ms) at Different Temperatures. $N_{1\mathrm{fix}} = 10^{-2}$, $10^{-3} < N_{2\mathrm{var}} < 10^{-2}$. Here, $x = \ln (a_1/a_2)$

	20°C	30°C	40°C	50°C
N5	$E_c = 14.94x$ $n = 8$; $\sigma = 0.06$ $t_+^{\mathrm{app}}(\mathrm{ms}) = 0.394$	$E_c = 15.75x$ $n = 8$; $\sigma = 0.06$ $t_+^{\mathrm{app}}(\mathrm{ms}) = 0.402$	$E_c = 16.36x$ $n = 9$; $\sigma = 0.10$ $t_+^{\mathrm{app}}(\mathrm{ms}) = 0.404$	$E_c = 17.29x$ $n = 7$; $\sigma = 0.04$ $t_+^{\mathrm{app}}(\mathrm{ms}) = 0.414$
N2	$E_c = 15.02x$ $n = 8$; $\sigma = 0.05$ $t_+^{\mathrm{app}}(\mathrm{ms}) = 0.397$	$E_c = 15.75x$ $n = 8$; $\sigma = 0.06$ $t_+^{\mathrm{app}}(\mathrm{ms}) = 0.402$	$E_c = 16.40x$ $n = 8$; $\sigma = 0.05$ $t_+^{\mathrm{app}}(\mathrm{ms}) = 0.405$	$E_c = 17.18x$ $n = 8$; $\sigma = 0.07$ $t_+^{\mathrm{app}}(\mathrm{ms}) = 0.412$
N08	$E_c = 15.10x$ $n = 9$; $\sigma = 0.04$ $t_+^{\mathrm{app}}(\mathrm{ms}) = 0.399$	$E_c = 15.86x$ $n = 9$; $\sigma = 0.06$ $t_+^{\mathrm{app}}(\mathrm{ms}) = 0.405$	$E_c = 16.77x$ $n = 8$; $\sigma = 0.04$ $t_+^{\mathrm{app}}(\mathrm{ms}) = 0.415$	$E_c = 17.38x$ $n = 8$; $\sigma = 0.06$ $t_+^{\mathrm{app}}(\mathrm{ms}) = 0.416$
N04	$E_c = 15.16x$ $n = 8$; $\sigma = 0.02$ $t_+^{\mathrm{app}}(\mathrm{ms}) = 0.400$	$E_c = 15.88x$ $n = 8$; $\sigma = 0.06$ $t_+^{\mathrm{app}}(\mathrm{ms}) = 0.406$	$E_c = 16.85x$ $n = 7$; $\sigma = 0.05$ $t_+^{\mathrm{app}}(\mathrm{ms}) = 0.417$	$E_c = 17.47x$ $n = 6$; $\sigma = 0.06$ $t_+^{\mathrm{app}}(\mathrm{ms}) = 0.419$
N01	$E_c = 16.28x$ $n = 8$; $\sigma = 0.06$ $t_+^{\mathrm{app}}(\mathrm{ms}) = 0.430$	$E_c = 16.82x$ $n = 8$; $\sigma = 0.10$ $t_+^{\mathrm{app}}(\mathrm{ms}) = 0.429$	$E_c = 17.81x$ $n = 8$; $\sigma = 0.09$ $t_+^{\mathrm{app}}(\mathrm{ms}) = 0.440$	$E_c = 18.42x$ $n = 10$; $\sigma = 0.10$ $t_+^{\mathrm{app}}(\mathrm{ms}) = 0.441$
N003	$E_c = 1.49x^2 + 19.32x$ $n = 10$; $\sigma = 0.07$ $t_+^{\mathrm{app}}(\mathrm{ms}) = 0.079x + 0.510$	$E_c = 1.47x^2 + 20.38x$ $n = 8$; $\sigma = 0.05$ $t_+^{\mathrm{app}}(\mathrm{ms}) = 0.075x + 0.520$	$E_c = 14.9x^2 + 21.88x$ $n = 7$; $\sigma = 0.03$ $t_+^{\mathrm{app}}(\mathrm{ms}) = 0.074x + 0.541$	$E_c = 1.10x^2 + 23.62x$ $n = 7$; $\sigma = 0.06$ $t_+^{\mathrm{app}}(\mathrm{ms}) = 0.053x + 0.566$

TABLE 3

Results Obtained for E_c (mV) and t_+^{app}(ms) at Different Temperatures. $N_{1fix} = 10^{-1}$, $10^{-2} < N_{2var} < 10^{-1}$. Here, $x = \ln(a_1/a_2)$

	20°C	30°C	40°C	50°C
N5	$E_c = 0.23x^2 + 14.06x$ $n = 8$; $\sigma = 0.03$ $t_+^{app}(ms) = 0.012x + 0.371$	$E_c = 0.21x^2 + 14.88x$ $n = 10$; $\sigma = 0.06$ $t_+^{app}(ms) = 0.011x + 0.380$	$E_c = 0.24x^2 + 15.45x$ $n = 9$; $\sigma = 0.04$ $t_+^{app}(ms) = 0.012x + 0.382$	$E_c = 16.39x$ $n = 7$; $\sigma = 0.08$ $t_+^{app}(ms) = 0.393$
N2	$E_c = 0.15x^2 + 14.23x$ $n = 8$; $\sigma = 0.04$ $t_+^{app}(ms) = 0.0079x + 0.376$	$E_c = 0.18x^2 + 14.90x$ $n = 8$; $\sigma = 0.04$ $t_+^{app}(ms) = 0.0092x + 0.380$	$E_c = 0.13x^2 + 15.69x$ $n = 10$; $\sigma = 0.05$ $t_+^{app}(ms) = 0.0064x + 0.388$	$E_c = 0.30x^2 + 16.08x$ $n = 9$; $\sigma = 0.08$ $t_+^{app}(ms) = 0.014x + 0.385$
N08	$E_c = 0.18x^2 + 14.10x$ $n = 8$; $\sigma = 0.04$ $t_+^{app}(ms) = 0.0095x + 0.372$	$E_c = 0.18x^2 + 14.88x$ $n = 7$; $\sigma = 0.04$ $t_+^{app}(ms) = 0.0092x + 0.380$	$E_c = 0.29x^2 + 15.41x$ $n = 10$; $\sigma = 0.09$ $t_+^{app}(ms) = 0.014x + 0.381$	$E_c = 0.10x^2 + 16.51x$ $n = 8$; $\sigma = 0.05$ $t_+^{app}(ms) = 0.005x + 0.396$
N04	$E_c = 0.23x^2 + 14.11x$ $n = 7$; $\sigma = 0.06$ $t_+^{app}(ms) = 0.012x + 0.373$	$E_c = 0.17x^2 + 14.93x$ $n = 8$; $\sigma = 0.04$ $t_+^{app}(ms) = 0.0087x + 0.381$	$E_c = 0.10x^2 + 15.83x$ $n = 8$; $\sigma = 0.07$ $t_+^{app}(ms) = 0.0049x + 0.391$	$E_c = 0.25x^2 + 16.21x$ $n = 8$; $\sigma = 0.07$ $t_+^{app}(ms) = 0.012x + 0.388$
N01	$E_c = 0.17x^2 + 14.34x$ $n = 8$; $\sigma = 0.06$ $t_+^{app}(ms) = 0.0090x + 0.379$	$E_c = 0.22x^2 + 14.95x$ $n = 8$; $\sigma = 0.03$ $t_+^{app}(ms) = 0.011x + 0.382$	$E_c = 0.49x^2 + 15.05x$ $n = 10$; $\sigma = 0.10$ $t_+^{app}(ms) = 0.024x + 0.372$	$E_c = 0.12x^2 + 16.63x$ $n = 8$; $\sigma = 0.05$ $t_+^{app}(ms) = 0.0058x + 0.399$
N003	$E_c = 0.94x^2 + 14.41x$ $n = 9$; $\sigma = 0.04$ $t_+^{app}(ms) = 0.050x + 0.380$	$E_c = 0.87x^2 + 15.42x$ $n = 8$; $\sigma = 0.03$ $t_+^{app}(ms) = 0.044x + 0.394$	$E_c = 1.24x^2 + 15.75x$ $n = 8$; $\sigma = 0.09$ $t_+^{app}(ms) = 0.061x + 0.389$	$E_c = 0.72x^2 + 17.10x$ $n = 10$; $\sigma = 0.03$ $t_+^{app}(ms) = 0.035x + 0.410$

TABLE 4
Results Obtained for P_s(ms) at Different Temperatures. $N_{1\text{fix}} = 10^{-3}$, ln $(a_1/a_2) = 0$

	20°C	30°C	40°C	50°C
N5	<0.02	<0.02	<0.02	<0.02
N2	<0.02	<0.02	<0.02	<0.02
N08	0.05 ± 0.02	0.04 ± 0.02	0.04 ± 0.02	0.04 ± 0.02
N04	0.06 ± 0.02	0.04 ± 0.02	0.05 ± 0.02	0.04 ± 0.02
N01	0.17 ± 0.02	0.16 ± 0.02	0.18 ± 0.02	0.16 ± 0.02
N003	0.62 ± 0.02	0.60 ± 0.02	0.58 ± 0.02	0.59 ± 0.02

membrane acquires the properties of a liquid junction influenced by the presence of fixed ions on the walls of the pores), and finally, to those of a liquid junction (the inside of the pore is an electroneutral mixture of counterions and co-ions, and the membrane has degenerated to a mere diaphragm with an internal liquid junction). This double-layer model explains our results:

The selectivity of the membrane systems made up of the membranes N5, N2, N08, and N04 is negligible. This is because the selectivity determining parameter (ratio of pore radius to Debye length) is very large. Differences are more significant for lower bounding solution concentration and for membranes of lower pore size.

The results of P_s(ms) for N01 and N003 membrane systems clearly indicate that those membranes are permselectives for Mg^{2+} cations. Permselectivity falls with increasing concentration because with an increase in external electrolyte concentration, more and more co-ions enter the membrane pores and the membrane thus loses its selectivity.

TABLE 5
Results Obtained for P_s(ms) at Different Temperatures. $N_{1\text{fix}} = 10^{-2}$, ln $(a_1/a_2) = 1$

	20°C	30°C	40°C	50°C
N5	<0.02	<0.02	<0.02	<0.02
N2	<0.02	<0.02	<0.02	<0.02
N08	<0.02	<0.02	<0.02	<0.02
N04	<0.02	<0.02	<0.02	<0.02
N01	0.07 ± 0.02	0.05 ± 0.02	0.06 ± 0.02	0.06 ± 0.02
N003	0.33 ± 0.02	0.33 ± 0.02	0.35 ± 0.02	0.35 ± 0.02

TABLE 6
Results Obtained for P_s(ms) at Different Temperatures. $N_{1\text{fix}} = 10^{-1}$, ln $(a_1/a_2) = 1$

	20°C	30°C	40°C	50°C
N5	<0.02	<0.02	<0.02	<0.02
N2	<0.02	<0.02	<0.02	<0.02
N08	<0.02	<0.02	<0.02	<0.02
N04	<0.02	<0.02	<0.02	<0.02
N01	<0.02	<0.02	<0.02	<0.02
N003	0.10 ± 0.02	0.09 ± 0.2	0.11 ± 0.02	0.10 ± 0.02

The variation of Debye length is not very important from 20 to 50°C (*10*); thus, a variation of selectivity when the temperature increases is not observed due to the influence of experimental errors.

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