

Permeability of anion-exchange membrane for Cl^- ions. Dialysis of hydrochloride acid in the presence of nickel chloride

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Abstract. Using a conventional two-compartment cell with stirrers the separation of an aqueous solution of HCl-NiCl_2 by an anion-exchange membrane Neosepta-AFN was investigated. The dialysis process was characterized by the permeability coefficient of the membrane towards to Cl^- ions. This quantity was determined by the numerical integration of equations, which describe the time dependence of the total concentration of Cl^- ions in compartment initially filled with stripping agent (water), combined with an optimizing procedure. The analysis of the experimental results showed that this permeability coefficient is a satisfactory characteristic for the process studied. It can be graphically correlated with the initial acid and initial salt concentrations in the compartment initially filled with acid+salt mixture.

Keywords: batch dialysis; hydrochloric acid; nickel chloride; permeability of membrane; anion-exchange membrane.

1. Introduction

The separation of mixtures by polymeric membranes is based on the fact that the individual components of the mixture are transported through the membrane at various rates. It is a consequence of differences in diffusivities or mobilities due to different interactions of the components with the membrane phase. In the case of diffusion dialysis, which uses ion-exchange membranes, it is necessary to consider beside these effects also the interactions between the species in the solution and positively charged sites inside the membrane. If an anion-exchange membrane is used for the separation of electrolytes, cations are rejected, while the transport of anions is facilitated. Hydrogen ions are not effectively rejected, because they are very small and their mobility is high not only in aqueous solutions but also in the membrane phase. This phenomenon need not be considered to be a disadvantage if the anion-exchange membrane is used in the separation of acid+salt mixtures (Kobuchi, *et al.* 1984, 1986).

An extensive study on the recovery of formic acid from pickling solution in the leather industry using diffusion dialysis and electro-electro dialysis was a subject of the communication (Akgemci, *et al.* 2004) focused on the examining of the effects of various operating variables on the process performance with an aim to find optimum operating conditions for both the processes. Xu and Yang (2001, 2003) dealt with sulfuric acid recovery from titanium white (pigment) waste liquor and the

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recovery of mixed acid ($\text{HF}+\text{HNO}_3$) from the titanium spent leaching solution using diffusion dialysis with a new series of anion exchange membranes. It was reported that $\text{HF}+\text{HNO}_3$ mixture can be separated selectively from the spent liquor. In another paper, Xu and Yang (2004) carried out experiments focused on simultaneous recovery of sulfuric acid and nickel from electrolysis spent liquor of relatively low acid concentration. The pilot experiments with surface cross-linked PPO anion-exchange membranes showed that nickel leakage can be controlled within 4%, and the recovery of acid can attain as high as 66-72%. Oh, *et al.* (2000) studied the effects of metal ions on diffusion dialysis of inorganic acids using a continuous dialyzer. It was observed that the formation of complexes affected the selectivity and flux of acid. Out of five metals investigated, Fe, Ni, Cr and Cu were reasonably rejected by the anion-exchange membrane, while Zn in the HCl solution leaked through the membrane significantly. In the paper of Wódzki and Szczepański (2001) the recovery and concentration of Zn^{2+} and Cu^{2+} ions from industrial rinse water containing a mixture of Zn^{2+} , Cu^{2+} , Mg^{2+} , K^+ and Na^+ ions were studied in a continuous membrane extraction, where sulfuric acid was used as a stripping agent for metal ions. In order to de-acidify the concentrate solution of Cu^{2+} ions, the diffusion dialysis technique was suggested. Wisniewska, *et al.* (1993) tested the processes of electrodialysis and diffusion dialysis in the separation of model mixtures containing hydrochloric acid and ferric ions. Diffusion dialysis provided acid removal of 56-67% with high retention of ferric ions - up to 98-99%. Sulfuric acid recovery from earth sulfate solutions by diffusion dialysis was a subject of the communication of Tang, *et al.* (2006). Here, the numerical analysis of the effects of operational parameters was studied. Elmidaoui, *et al.* (1991) presented results on competition between hydrochloric acid and sodium chloride across an anion-exchange membrane Neosepta-AFN. They measured the flux of each electrolyte from aqueous solutions containing two electrolytes. In all cases the flux of acid was higher than that of the salt. The results were analyzed on the basis of the Nernst-Planck equation and the Teorell-Meyer-Sievers theory.

Diffusion dialysis can be used not only in the separation of mixtures containing inorganic acids - as shown above - but also in the separation of organic acids from their salts (Narebska and Staniszewski 1997, 2008).

The experiments analyzed in our earlier paper (Palatý and Žáková 2007) revealed that an anion-exchange membrane Neosepta-AFN (ASTOM Corporation, Japan) is a good separator for an $\text{HCl}+\text{NiCl}_2$ mixture, because the partial flux of nickel chloride is very low, i.e. below 3.2%. For that reason and with respect to the high importance of the $\text{HCl}+\text{NiCl}_2$ system in the industrial practice, the aim of this communication is to present another attempt in the data analysis, which is based on the quantitative description of permeation of Cl^- ions through an anion-exchange membrane. For that purpose, the experimental data obtained formerly (Palatý and Žáková 2007) are used.

2. Theory

Consider a two-compartment cell with an anion-exchange membrane, which is depicted in Fig. 1. If the concentration of Cl^- ions in compartment I (left) is higher than that in compartment II (right), the Cl^- ions are crossing the membrane from left to right. This transport can be quantified by the permeability coefficient of the membrane towards Cl^- ions, P_{Cl^-} , which is interrelated with the flux of Cl^- ions by the following equation

$$J_{\text{Cl}^-} = P_{\text{Cl}^-} ([\text{Cl}^-]_{\text{I}}^I + (-[\text{Cl}^-]_{\text{I}}^{\text{II}})) \quad (1)$$

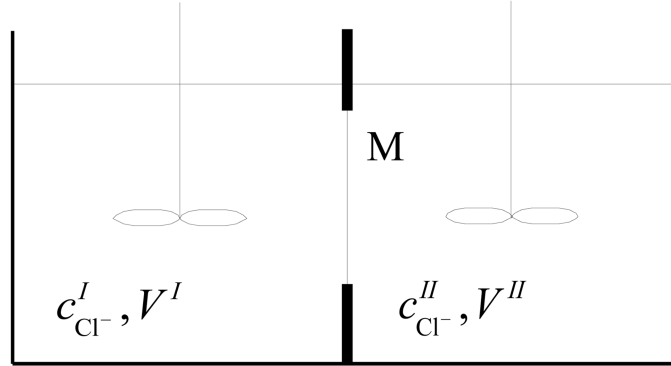


Fig. 1 Scheme of dialysis cell

(Note: If the effect of liquid films on both the sides of the membrane can be neglected, then the concentrations of Cl^- ions in liquid at the solution/membrane interface are equal to those in the bulk solutions, i.e. $[\text{Cl}^-]_{if}^k = [\text{Cl}^-]^k$ ($k=I, II$)).

The flux of Cl^- ions can easily be determined from the change in the concentration of Cl^- ions in compartment I or II, i.e.

$$J_{\text{Cl}^-} = -\frac{1}{A} \frac{dn_{\text{Cl}^-}^I}{d\tau} = \frac{1}{A} \frac{dn_{\text{Cl}^-}^{II}}{d\tau} = -\frac{V^I d[\text{Cl}^-]^I}{A d\tau} - \frac{[\text{Cl}^-]^I dV^I}{A d\tau} = \frac{V^{II} d[\text{Cl}^-]^{II}}{A d\tau} + \frac{[\text{Cl}^-]^{II} dV^{II}}{A d\tau} \quad (2)$$

(Note: In Eq. (2) the pseudo-steady state is supposed, i.e. $J_{\text{Cl}^-}^I = J_{\text{Cl}^-}^{II} = J_{\text{Cl}^-,M} = J_{\text{Cl}^-}$.)
A combination of Eqs. (1) and (2) leads to Eqs. (3) and (4)

$$\frac{d[\text{Cl}^-]^I}{d\tau} = -\frac{A}{V^I} P_{\text{Cl}^-} ([\text{Cl}^-]^I - [\text{Cl}^-]^{II}) - \frac{[\text{Cl}^-]^I dV^I}{V^I d\tau} \quad (3)$$

$$\frac{d[\text{Cl}^-]^{II}}{d\tau} = \frac{A}{V^{II}} P_{\text{Cl}^-} ([\text{Cl}^-]^I - [\text{Cl}^-]^{II}) - \frac{[\text{Cl}^-]^{II} dV^{II}}{V^{II} d\tau} \quad (4)$$

Using the substitution

$$\frac{d[\text{Cl}^-]^k}{d\tau} = \frac{d[\text{Cl}^-]^k dc_{\text{Cl}^-}^k}{dc_{\text{Cl}^-}^k d\tau} \quad k=I, II \quad (5)$$

one can obtain Eqs. (6) and (7), which describe the time dependence of the total concentration of Cl^- ions in compartment I and II

$$\frac{dc_{\text{Cl}^-}^I}{d\tau} = -\frac{\frac{A}{V^I} P_{\text{Cl}^-} ([\text{Cl}^-]^I - [\text{Cl}^-]^{II}) + \frac{[\text{Cl}^-]^I dV^I}{V^I d\tau}}{\frac{d[\text{Cl}^-]^I}{dc_{\text{Cl}^-}^I}} \quad (6)$$

$$\frac{dc_{\text{Cl}^-}^{\text{II}}}{d\tau} = \frac{\frac{A}{V^{\text{II}}} P_{\text{Cl}^-} ([\text{Cl}^-]^{\text{I}} - [\text{Cl}^-]^{\text{II}}) - \frac{[\text{Cl}^-]^{\text{II}}}{V^{\text{I}}} \frac{dV^{\text{II}}}{d\tau}}{\frac{d[\text{Cl}^-]^{\text{II}}}{dc_{\text{Cl}^-}^{\text{II}}}} \quad (7)$$

The initial conditions for Eqs. (6) and (7) are

$$\tau = 0 \quad c_{\text{Cl}^-}^{\text{I}} = c_{\text{Cl}^-}^{\text{I}},_0 \quad c_{\text{Cl}^-}^{\text{II}} = c_{\text{Cl}^-}^{\text{II}},_0 = 0 \quad (8)$$

The proper utilization of Eqs. (6) and (7) depends on the system investigated. Generally, Eqs. (6) and (7) can be numerically integrated, and if this procedure is combined with a suitable optimizing procedure, then it is possible to determine the permeability coefficient, P_{Cl^-} .

3. Experimental

The experimental set-up and the experiments are described in detail elsewhere (Palatý and Žáková 2007). In all the experiments, an anion-exchange membrane Neosepta-AFN (ASTOM Corporation, Japan) was used. The conditions, under which the experimental data were obtained, are summarized in Table 1.

4. Data treatment and discussion

In the case of HCl/membrane/H₂O system, the total and actual concentrations of Cl[−] ions are equal to the hydrochloric acid concentration because hydrochloric acid is completely dissociated. The derivative $d[\text{Cl}^-]^k/dc_{\text{Cl}^-}^k$ ($k=\text{I}, \text{II}$) in Eqs. (6) and (7) is equal to 1. Thus, the permeability coefficient for Cl[−] ions is equal to that for hydrochloric acid ($P_{\text{Cl}^-} = P_{\text{HCl}}$). It is in the range from approx. 0.84×10^{-6} to $2.4 \times 10^{-6} \text{ m s}^{-1}$. Its dependence upon the initial acid concentration in the compartment initially filled with hydrochloric acid is given elsewhere (Palatý and Žáková 2007).

4.1. NiCl₂/membrane/H₂O system

According to Kotrlý and Šůcha (1985), the following species exist in aqueous solution of NiCl₂:

Table 1 Experimental conditions

Variable	Value or range	Unit
Initial acid concentration in compartment I	0.2–3.0	kmol m ^{−3}
Initial salt concentration in compartment I	0.1–1.0	kmol m ^{−3}
Initial liquid volume in compartment I	1.0×10^{-3}	m ³
Initial liquid volume in compartment II	1.0×10^{-3}	m ³
Membrane area	62.2×10^{-4}	m ²
Temperature	20.0±0.5	°C

Cl^- , Ni^{2+} and NiCl^+ . The concentration of Cl^- ions and the derivative $d[\text{Cl}^-]^k/dc_{\text{Cl}^-}^k$ ($k=\text{I, II}$), which are needed in the calculation of the permeability coefficient, can be found by solving the set of equations describing the ionic equilibrium ($\text{Ni}^{2+}+\text{Cl}^- \leftrightarrow \text{NiCl}^+$) (Eq. (9)), electro-neutrality condition (Eq. (10)) and balance of chloride ion (Eq. (11))

$$\frac{[\text{NiCl}^+]}{[\text{Ni}^{2+}][\text{Cl}^-]} - \beta_1 = 0 \quad (9)$$

$$2[\text{Ni}^{2+}] + [\text{NiCl}^+] - [\text{Cl}^-] = 0 \quad (10)$$

$$[\text{NiCl}^+] + [\text{Cl}^-] - c_{\text{Cl}^-} = 0 \quad (11)$$

(Note that $c_{\text{Cl}^-} = 2c_{\text{NiCl}_2}$)

Solving the set of Eqs. (9)-(11), one can find that

$$[\text{Cl}^-] = \frac{-(2 - \beta_1 c_{\text{Cl}^-}) + \sqrt{(2 - \beta_1 c_{\text{Cl}^-})^2 + 16\beta_1 c_{\text{Cl}^-}}}{4\beta_1} \quad (12)$$

and

$$\frac{d[\text{Cl}^-]}{dc_{\text{Cl}^-}} = \frac{2 + \beta_1 [\text{Cl}^-]}{2 + \beta_1 (4[\text{Cl}^-] - c_{\text{Cl}^-})} \quad (13)$$

In order to determine the permeability coefficient of the membrane for Cl^- , the ordinary differential Eq. (7) was numerically solved - the 4th order Runge-Kutta method was used. This integration was followed by the optimizing procedure searching for a minimum of the objective function (14) - for this purpose, the Golden section search was used.

$$F(P_{\text{Cl}^-}) = \sum_{i=1}^n \left(\frac{c_{\text{NiCl}_2, \text{exp}}^{II, i} - c_{\text{NiCl}_2, \text{calc}}^{II, i}}{c_{\text{NiCl}_2, \text{exp}}^{II, i}} \right)^2 \quad (14)$$

(Note: n is the number of the experimental points in one time series)

In Fig. 2 the permeability coefficient for Cl^- ions is plotted versus the initial nickel chloride concentration in compartment I. It is evident that the permeability of the Neosepta-AFN membrane for Cl^- ions in the case of dialysis of aqueous solutions of NiCl_2 exhibits very low values, i.e. in the range from approx 0.2×10^{-8} to $2.75 \times 10^{-8} \text{ m s}^{-1}$. Moreover, it is about two orders of magnitude lower than that for hydrochloric acid.

As the transport characteristics of the solution/membrane system are usually referred to the initial component concentration in compartment I, we modified the procedure given above in such a way that the objective function (14) was considered a function of the coefficients a_0 , a_1 and a_2 of the polynomial (15) used for the approximation of the dependence $P_{\text{Cl}^-} = f(c_{\text{NiCl}_2, 0}^I)$.

$$P_{\text{Cl}^-} = a_0 + a_1 c_{\text{NiCl}_2, 0}^I + a_2 (c_{\text{NiCl}_2, 0}^I)^2 \quad (15)$$

Using a simplex optimizing method (the algorithm by Nelder and Mead), the following values of a_i ($i=0, 1, 2$) were found: $a_0 = -6.36 \times 10^{-10} \text{ m s}^{-1}$; $a_1 = 2.29 \times 10^{-8} \text{ m}^4 \text{ s}^{-1} \text{ kmol}^{-1}$; $a_2 = 3.44 \times 10^{-9} \text{ m}^7 \text{ s}^{-1} \text{ kmol}^{-2}$. In Fig. 2, the values of P_{Cl^-} calculated from polynomial (15) are drawn by the solid line.

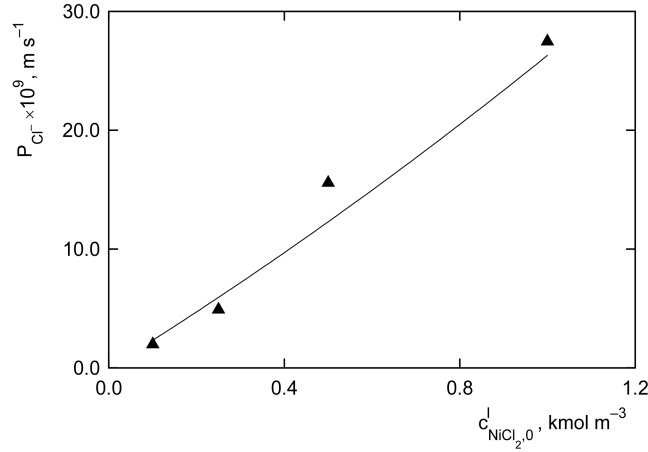


Fig. 2 Dependence of permeability coefficient for Cl^- ions upon initial nickel chloride concentration in compartment I - $\text{NiCl}_2/\text{membrane}/\text{H}_2\text{O}$ system

4.2. $\text{HCl}+\text{NiCl}_2/\text{membrane}/\text{H}_2\text{O}$ system

In an $\text{HCl}+\text{NiCl}_2$ aqueous solution the following species exist (Kotrly and Šucha 1985): H^+ , Cl^- , Ni^{2+} and NiCl^+ . Their actual concentrations and the derivative $d[\text{Cl}^-]^k/dc_{\text{Cl}^-}^k$ ($k=\text{I, II}$) can be found by solving the set of Eqs. (9), (11), to which Eq. (16) (new electroneutrality condition) and Eq. (17) (balance of hydrogen ion) are added

$$[\text{H}^+] + 2[\text{Ni}^{2+}] + [\text{NiCl}^+] - [\text{Cl}^-] = 0 \quad (16)$$

$$[\text{H}^+] - c_{\text{HCl}} = 0 \quad (17)$$

(Note: In Eq. (17) the total concentration of Cl^- ions is equal to $c_{\text{HCl}} + 2c_{\text{NiCl}_2}$.)

The differentiation of Eqs. (9), (11), (16) and (17) gives

$$\beta_1[\text{Ni}^{2+}] \frac{d[\text{Cl}^-]}{dc_{\text{Cl}^-}} + \beta_1[\text{Cl}^-] \frac{d[\text{Ni}^{2+}]}{dc_{\text{Cl}^-}} - \frac{d[\text{NiCl}^+]}{dc_{\text{Cl}^-}} = 0 \quad (18)$$

$$\frac{d[\text{NiCl}^+]}{dc_{\text{Cl}^-}} + \frac{d[\text{Cl}^-]}{dc_{\text{Cl}^-}} = 1 \quad (19)$$

$$\frac{d[\text{H}^+]}{dc_{\text{Cl}^-}} + 2 \frac{d[\text{Ni}^{2+}]}{dc_{\text{Cl}^-}} + \frac{d[\text{NiCl}^+]}{dc_{\text{Cl}^-}} - \frac{d[\text{Cl}^-]}{dc_{\text{Cl}^-}} = 0 \quad (20)$$

$$\frac{d[\text{H}^+]}{dc_{\text{Cl}^-}} = \frac{dc_{\text{HCl}}}{dc_{\text{Cl}^-}} \quad (21)$$

By solving the set of Eqs. (18)-(21), one can obtain

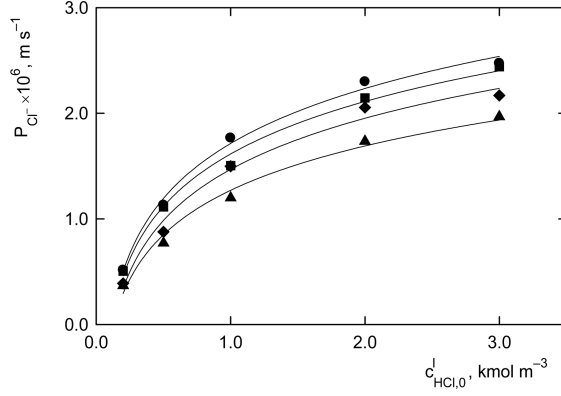


Fig. 3 Dependence of permeability coefficient for Cl^- ions upon initial hydrochloric acid concentration in compartment I - $\text{HCl}+\text{NiCl}_2/\text{membrane}/\text{H}_2\text{O}$ system: $\bullet - c_{\text{NiCl}_2,0} = 0.1 \text{ kmol m}^{-3}$, $\blacksquare - c_{\text{NiCl}_2,0} = 0.25 \text{ kmol m}^{-3}$, $\blacklozenge - c_{\text{NiCl}_2,0} = 0.5 \text{ kmol m}^{-3}$, $\blacktriangle - c_{\text{NiCl}_2,0} = 1.0 \text{ kmol m}^{-3}$

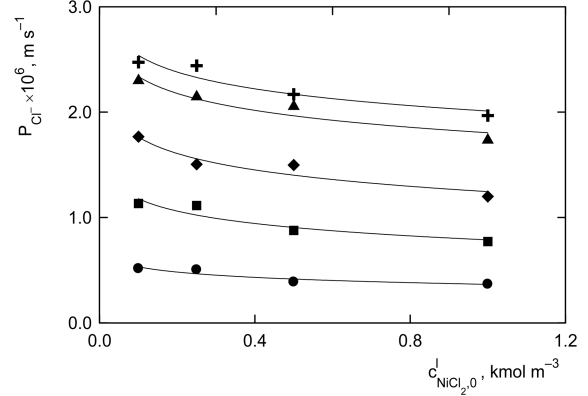


Fig. 4 Dependence of permeability coefficient for Cl^- ions upon initial nickel chloride concentration in compartment I - $\text{HCl}+\text{NiCl}_2/\text{membrane}/\text{H}_2\text{O}$ system: $\bullet - c_{\text{HCl},0} = 0.2 \text{ kmol m}^{-3}$, $\blacksquare - c_{\text{HCl},0} = 0.5 \text{ kmol m}^{-3}$, $\blacklozenge - c_{\text{HCl},0} = 1.0 \text{ kmol m}^{-3}$, $\blacktriangle - c_{\text{HCl},0} = 2.0 \text{ kmol m}^{-3}$, $\blackplus - c_{\text{HCl},0} = 3.0 \text{ kmol m}^{-3}$

$$\frac{d[\text{Cl}^-]}{dc_{\text{Cl}^-}} = \frac{2 + \beta_1[\text{Cl}^-] \left(1 + \frac{dc_{\text{HCl}}}{dc_{\text{Cl}^-}}\right)}{2 \{1 + \beta_1([\text{Cl}^-] + [\text{Ni}^{2+}])\}} \quad (22)$$

where $\frac{dc_{\text{HCl}}}{dc_{\text{Cl}^-}} = 1$.

Similarly as in the previous case, Eq. (7) was numerically integrated and this procedure was combined with an optimizing procedure searching for a minimum of the objective function, where instead of the salt concentration the total concentration of Cl^- ions in compartment II was used.

The dependences of the permeability coefficient of the Neosepta-AFN membrane upon the initial acid and salt concentrations in compartment I are presented in Figs. 3 and 4. Fig. 3 concerns the dependence $P_{\text{Cl}^-} = f(c_{\text{HCl},0})$ (here, the parameter of the individual curves is the initial salt concentration), while Fig. 4 concerns the dependence $P_{\text{Cl}^-} = f(c_{\text{NiCl}_2,0})$ (here, the parameter of the individual curves is $c_{\text{HCl},0}$). All the dependences presented reveal that the permeability coefficient for Cl^- ions, P_{Cl^-} , is affected by both the initial acid and initial salt concentration in compartment I. The permeability coefficient decreases with an increasing salt concentration but it increases with an increasing acid concentration. Moreover, the permeability coefficient is of the same order of magnitude as that for hydrochloric acid (Palatý and Žáková 2007).

5. Conclusion

Dialysis of aqueous solution of $\text{HCl}+\text{NiCl}_2$ was investigated in a two-compartment cell. In all the experiments, the anion-exchange membrane Neosepta-AFN was used. In order quantify the dialysis process, the permeability coefficient of the membrane for Cl^- ions was used. This quantity was

determined from the time dependences of the total concentration of Cl^- ions in compartment initially filled with water as a stripping agent. For this purpose, the basic differential equation describing the dependence of the total concentration of Cl^- ions upon time was numerically integrated and this step was combined with the optimizing procedure. The results showed that the permeability coefficient for Cl^- ions is a satisfactory characteristic of the dialysis process.

Acknowledgements

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Nomenclature

A	membrane area, m^2
a_0	coefficient in Eq. (15), m s^{-1}
a_1	coefficient in Eq. (15), $\text{m}^4 \text{s}^{-1} \text{kmol}^{-1}$
a_2	coefficient in Eq. (15), $\text{m}^7 \text{s}^{-1} \text{kmol}^{-2}$
c	molar (total) concentration, kmol m^{-3}
F	objective function
f	general function
J	molar flux, $\text{kmol m}^{-2} \text{s}^{-1}$
n	amount, kmol
n	number of experimental points
P	permeability coefficient, m s^{-1}
V	volume, m^3
β_1	complexity constant
τ	time, s
$[]$	actual concentration, kmol m^{-3} , in Eq. (9) dimensionless

Superscripts

I	referred to compartment I
II	referred to compartment II

Subscripts

$calc$	calculated
Cl^-	referred to Cl^- ions
exp	experimental
HCl	referred to hydrochloric acid
if	interfacial
M	referred to membrane
NiCl_2	referred to nickel chloride
0	initial