

# Non-polarisable dry electrode based on NASICON ceramic

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**Abstract**—A NASICON-type ceramic (high sodium ion conductor) is proposed to record bioelectric signals. The electrode does not need gel before its application. The principle of the measurements is based on a sodium ion exchange between the skin and the material. Electrical measurements performed in saline solutions show that the electrode is slightly polarisable. The skin-electrode impedance was investigated. The impedance decreases as a function of the time of application. The resistive component is the major source of the impedance change. This can be explained by the perspiration process which occurs immediately with time after the application of the NASICON-based electrode on the skin. The skin condition is also an important parameter. NaCl saline solution or abrasion causes the resistance to decrease markedly.

**Keywords**—Bioelectrode, Impedance, NASICON, Skin

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## 1 Introduction

IT HAS been demonstrated that the NASICON (Na super ionic conductor) type of ceramic could be used to measure bioelectric signals on the skin, and there was no need to gel the electrode before its application (GONDRA *et al.*, 1992). Owing to the mechanical properties of the NASICON (robustness and shock resistance), the electrode was expected to be of great endurance, easy to clean and reusable for numerous measurements.

This paper is concerned with the electrical properties of this new type of electrode. The electrical performance in saline solutions (electrode–electrolyte interface) are reported and analysed with respect to some of the fundamental parameters of electrodes reviewed by McAdams (MCADAMS, 1992).

The electrode–skin interface impedance is a relevant parameter to assess the ability of an electrode to work properly. Impedance measurements of the NASICON-based electrode–skin interface are reported as a function of the time of application and the skin condition.

## 2 Principle of bioelectric signal recording with NASICON

NASICON, with the general formula  $\text{Na}_3\text{Zr}_2\text{Si}_2\text{PO}_{12}$ , is a ceramic material which is a good conductor through  $\text{Na}^+$  ions at room temperature (conductivity equal to  $10^{-3} \text{ Scm}^{-1}$ )

(HONG, 1976; COLOMBAN, 1986). The recording of bioelectric signals on the skin with this material is based on the existence of an equilibrium between the NASICON and the skin. When NASICON is brought into contact with the skin, a  $\text{Na}^+$  ion exchange between the two phases occurs:



The equilibrium condition can be written by the equality of the electrochemical potential of the  $\text{Na}^+$  ions in the two phases:

$$\bar{\mu}_{\text{Na}^+, \text{skin}} = \bar{\mu}_{\text{Na}^+, \text{NASICON}} \quad (2)$$

By separating  $\bar{\mu}_{\text{Na}^+}$  into a chemical and electrical contribution, eqn. 2 becomes

$$\begin{aligned} \mu_{\text{Na}^+, \text{skin}}^0 + RT \ln C_{\text{Na}^+, \text{skin}} + F\phi_{\text{skin}} \\ = \mu_{\text{Na}^+, \text{NAS}}^0 + RT \ln C_{\text{Na}^+, \text{NAS}} + F\phi_{\text{NAS}} \end{aligned} \quad (3)$$

Here  $C_{\text{Na}^+, \text{skin}}$  and  $C_{\text{Na}^+, \text{NAS}}$  are the  $\text{Na}^+$  ion concentrations in the skin and the NASICON phase, respectively.  $\phi_{\text{skin}}$  and  $\phi_{\text{NAS}}$  are the inner electric potentials of the skin and the NASICON material, respectively. As has been discussed by Fabry *et al.*, (1993), if the  $\text{Na}^+$  ion concentrations in the skin and in the membrane can be regarded as constant, eqn. 3 becomes

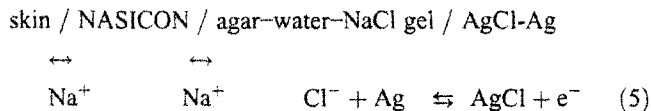
$$\phi_{\text{NAS}} = \phi_{\text{skin}} + \phi^0 \quad (4)$$

where  $\phi^0$  is a constant. The inner electric potential of the NASICON is therefore representative of the skin potential. Any change of the skin potential will be transduced as a change in NASICON potential. To ensure the transduction of this signal to the metallic wire, an electrochemical reversible internal contact is necessary (FABRY *et al.*, 1988a, b). In the present work it consists of an agar–water gel that contains sodium chloride.

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The electrochemical equilibria that allow the transmission of the electric signal are



If the exchange of  $\text{Na}^+$  ions between the skin and the NASICON membrane is sufficiently rapid, the electrode will require no gel on the skin and it will be slightly polarisable.

### 3 Experimental

#### 3.1 Electrode assembling

The synthesis of NASICON ceramics has been described in detail elsewhere (CANEIRO *et al.*, 1991). In the present work, the membranes were sintered at  $1000^\circ\text{C}$ . The conductivity of the samples, measured by impedance spectroscopy, was equal to  $2.5 \times 10^{-4} \text{ S cm}^{-1}$  at room temperature. The membranes were between 2 and 3 mm thick and between 7 and 10 mm in diameter. Before they were used, the membranes were polished with successive waterproof silicon carbide papers and diamond paste down to  $1 \mu\text{m}$ .

The electrode was assembled as shown in Fig. 1. The NASICON pellet was sealed in an inert support. The internal contact was a 3% agar water gel containing  $1 \text{ mol l}^{-1}$  of NaCl. It was prepared in the usual way. The silver wire was chloridised by electrolysis. This internal reference system was isolated from the ambient atmosphere by a silicon cement.

#### 3.2 Impedance measurements

The impedance data were obtained using a Solartron 1250 frequency response analyser coupled to a 1186 electrochemical interface.\* A three-electrode configuration mode was used. The experimental set-up is depicted elsewhere (GONDRAN *et al.*, 1992). Impedance measurements were performed in the 65 kHz–1 Hz frequency range. They were carried out at room temperature.

In saline solutions (1 M NaCl), the counter electrode was Ag–AgCl wire and the reference electrode a NASICON-based electrode. Impedance measurements were performed in potentiostatic mode. The DC voltage was equal to zero. The amplitude of the applied sinusoidal voltage was 20 mV RMS.

Impedance measurements on the skin were performed on the forearm. The tested electrodes were strapped on the ventral

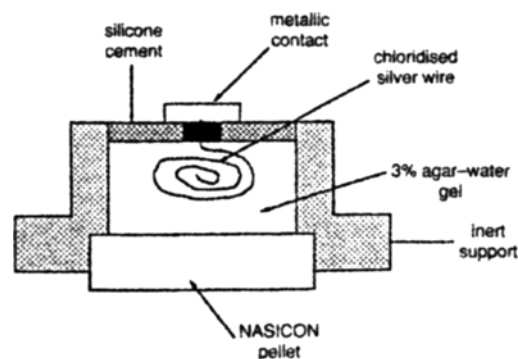


Fig. 1 Electrode construction

\* Schlumberger

side of the forearm with adhesive rubber. Two pregelled Ag–AgCl ECG electrodes were used as reference and counter electrodes, respectively. They were attached to the forearm. The impedance was measured in galvanostatic mode (DC current equal to zero). The amplitude of the applied sinusoidal current was  $1 \mu\text{A}$  RMS. The ambient relative humidity was not controlled during the measurements.

### 4 Electrical properties of the electrode in NaCl solutions

The DC voltage, bias current tolerance and AC impedance of the NASICON-based electrodes were evaluated in a saline solution (1 M NaCl).

#### 4.1 DC voltage

The DC voltage of a pair of NASICON-based electrodes was measured with a high input impedance millivoltmeter, after a 1 min stabilisation period, for 14 pairs of electrodes. It never exceeds 5 mV. The DC offset stability was tested over 15 h. For the 14 pairs of electrodes the EMF drift never exceeds 3.5 mV. The average drift rate was  $30 \mu\text{V s}^{-1}$ .

#### 4.2 Current tolerance

The response of the NASICON-based electrode potential when an anodic current of 200 nA passes through the electrode is shown in Fig. 2. As expected, the potential (measured against a Ag–AgCl reference electrode) slightly deviates from its equilibrium value. After 1 h the drift rate is low (less than  $2 \mu\text{Vs}^{-1}$ ). This test was performed anodically and cathodically on 10 electrodes for a time period of 1 h. For all the electrodes, the anodic overvoltage was less than 14 mV (average value 6 mV) and the cathode overvoltage was less than 21 mV (average value 7 mV).

#### 4.3 AC impedance

Fig. 3 shows the Nyquist plot of the NASICON-based electrode impedance measured after a short stabilisation period in 1 M NaCl. The diagram shows an 'almost straight line.' Its high-frequency intercept with the real axis, denoted  $R_{ms}$ , corresponds to the NASICON pellet and solution resistances. This NASICON pellet resistance depends on the geometric factor of the sample. Typically, for a membrane 10 mm in diameter and 1 mm thick, the resistance  $R_{ms}$  is equal to 500  $\Omega$ . The NASICON resistivity is equal to 400  $\Omega\text{cm}$  at room temperature. The low-frequency part of the diagram can be ascribed to the Ag–AgCl/agar–water gel interface. The impedance  $Z^*(10 \text{ Hz})$  defined as

$$Z^*(10 \text{ Hz}) = Z(10 \text{ Hz}) - R_{ms} \quad (6)$$

was used to evaluate the internal reference system. For 15

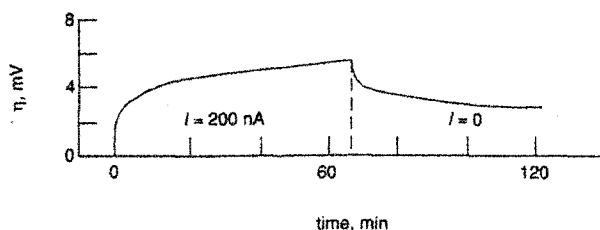


Fig. 2 NASICON-based electrode overpotential versus time in 1 M NaCl solution for anodic current of 200 nA

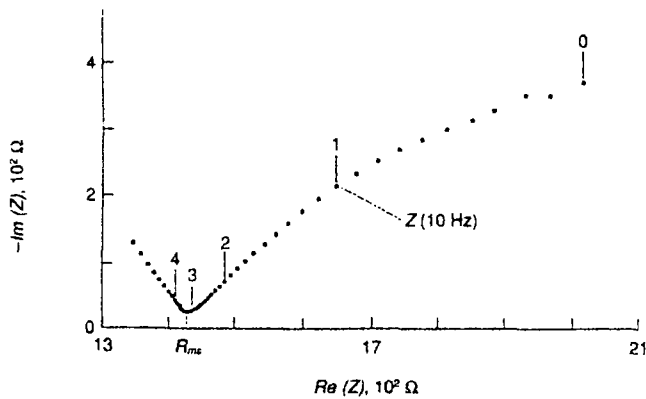


Fig. 3 Nyquist plot of NASICON-based electrode impedance in 1 M NaCl solution; parameter is frequency logarithm

electrodes  $Z^*(10 \text{ Hz})$  was found never to exceed  $500 \Omega$  (average value  $380 \Omega$ ).

The electrode AC impedance of the electrodes was measured over a period of several months. Between each measurement the electrodes were stored without any conditioning (storage in air, without any water supply). The impedance diagram obtained after 103 days is shown in Fig. 4. An additional high-frequency semicircle appears as a function of the time. It can be related to the  $\text{Na}^+$  ion transfer across the NASICON/agar-water gel interface (SIEBERT *et al.*, 1990). As expected, the resistance  $R_{ms}$  does not vary with the time.

### 5 AC impedance of the NASICON-based electrode-skin interface

The NASICON-based electrode was applied on dry skin prealably washed with acetone. A typical impedance locus is shown in Fig. 5. In the 10 kHz–1 Hz frequency range, the locus of the electrode-skin interface has the form of a depressed semicircle and satisfies the classical Cole-Cole law (COLE and COLE, 1941):

$$Z = \frac{Z_0 - Z_\infty}{1 + (\tau_m j\omega)^\alpha} \quad (7)$$

where  $Z_\infty$  and  $Z_0$  denote, respectively, the high-frequency and static values of  $Z$ , and  $\tau$  is a relaxation time. Compared with the classical debye expression, this equation represents the effect of a distribution of relaxation times. The parameter  $\alpha$  measures the breadth of this distribution. Such a diagram is classically observed (YAMAMOTO and YAMAMOTO, 1976, 1977;

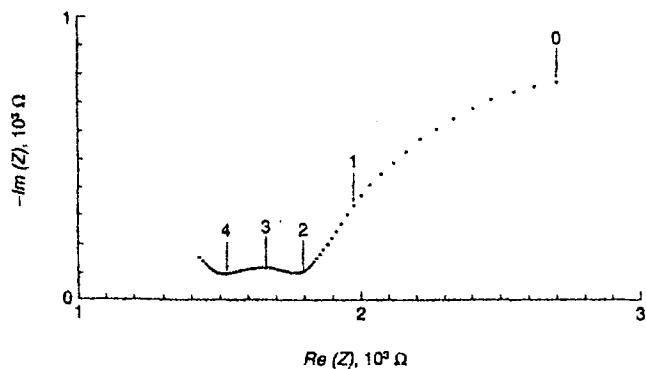


Fig. 4 Nyquist plot of impedance of NASICON-based electrode in 1 M NaCl solution 103 days after electrode has been constructed; parameter is frequency logarithm

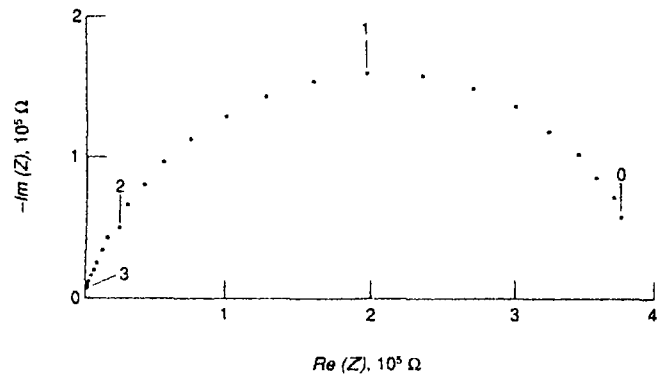


Fig. 5 Nyquist plot of impedance of skin-NASICON-based electrode interface 10 min after electrode application (NASICON diameter = 1.07 cm); parameter on the semi-circle is frequency logarithm

YAMAMOTO *et al.*, 1986; GRIMNES, 1983; JOSSINET *et al.*, 1991).

In the present work the skin-electrode interface will be treated as an inhomogeneous medium, represented by a static resistance in parallel with a static capacitance spacially distributed around a mean value. The general characteristics of the skin electrode impedance will be described by the three following parameters: the resistance  $R$  corresponding to the intercept of the semicircle with the real axis:

$$R = Z_0 - Z_\infty \quad (8)$$

the capacitive effect  $C$  calculated from

$$RC\omega_m = 1 \quad (9)$$

where  $\omega_m$  is the frequency at the top of the semi-circle; and the phase angle depression  $\phi$  which is related to the parameter  $\alpha$  by

$$\phi = (1 - \alpha) \frac{\pi}{2} \quad (10)$$

$R$  and  $C$  give the mean value around with the static resistance and capacitance are distributed. The values of these parameters typically lie within the range 50–500 k $\Omega$  for  $R$ , 20–80 nF for  $C$ , and 15–20° for  $\phi$ .

The electrode-skin interface impedance includes the electrode impedance, the electrode-skin interface impedance due to  $\text{Na}^+$  exchange at the interface, and the skin impedance. As seen in Fig. 3, the electrode impedance is far less than that observed in Fig. 5. The impedance of the  $\text{Na}^+$  ion transfer across the NASICON-saline solution interface has been studied elsewhere (SIEBERT *et al.*, 1990). The resistance was of the order of  $100 \Omega \text{ cm}^2$  for 0.1 M NaCl saline solution and the capacitive effect of the order of  $2 \mu\text{Fcm}^{-2}$ . The large resistive and low capacitive values deduced from the present work indicate that the skin-electrode interface impedance is predominantly the skin impedance. This has already been proposed to explain the electrode-skin behaviour (CLAR *et al.*, 1975; YAMAMOTO and YAMAMOTO, 1976; GRIMNES, 1983; KOHLI *et al.*, 1985; JOSSINET *et al.*, 1991).

It is well established that practically all the impedance of the skin results from the stratum corneum. The impedance diagram shape can therefore be assigned to the dielectric response of the stratum corneum. Fig. 6 gives a schematic picture of the stratum corneum structure. It consists of an insulating lipid cellular matrix containing pores. The stratum corneum thickness is typically in the range 10–40  $\mu\text{m}$  with an average of 20  $\mu\text{m}$ . There is evidence (CULLANDER and GUY, 1991; SCOTT and WHITE, 1992) that the charge transport occurs mainly through the channels. From this model, the static resistance and capacitance of the stratum corneum result from

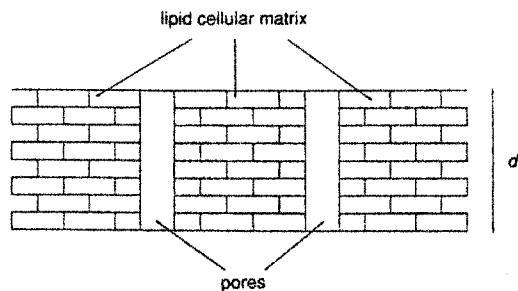


Fig. 6 *Stratum corneum according to Cullander and Guy (CULLANDER and GUY, 1992); d is average thickness of stratum corneum*

the parallel combination of the static resistance and capacitance of the lipid matrix and the pores:

$$\frac{1}{R_{stratum\ corneum}} = \frac{1}{R_{lipid\ matrix}} + \frac{1}{R_{pore}} \quad (11)$$

$$C_{stratum\ corneum} = C_{lipid\ matrix} + C_{pore} \quad (12)$$

Fig. 7 shows an example of long-term variation (8 h time period) of the impedance parameters  $R$ ,  $C$  and  $\phi$  against time after application of a dry NASICON-based electrode on dry skin. For comparison, the results obtained with a Ag-AgCl pregelled electrode ( $gSA^*$ ) fixed on the forearm near the NASICON-based electrode are indicated. As it was shown (YAMAMOTO and YAMAMOTO, 1986) that the skin admittance is approximately proportional to the electrode surface area, the resistance and capacitance reported in the figure have been normalized to the geometric surface area of the electrode. For the Ag-AgCl electrode, the surface was calculated with the diameter of the gel.

It can be seen from this Figure that the resistance decreases as a function of the time. An exponential law is obeyed. The time constant depends on the patient's skin (sweat duct filling). The drier the skin, the higher the time constant. Whatever the characteristics of the patient, the time necessary to reach a steady-state value is lower with NASICON-based electrodes than with pregelled Ag-AgCl electrodes. The capacitance remains relatively constant (a slight increase a few minutes after the electrode was applied). The phase angle depression is observed to increase. The increase is lower with NASICON than with pregelled Ag-AgCl electrodes. The same results have been reported (JOSSINET and MCADAMS 1991) for wet gel and hydrogel electrodes. It can be concluded that the resistance is the major source of the observed impedance changes.

The variation of the impedance parameters can be explained by the perspiration process which has been reported (YAMAMOTO and YAMAMOTO, 1977) to occur immediately after dry electrodes are applied to the skin. Perspiration has two main effects, as described below.

First, ionic shunt: the electrolyte penetrates into the pores of the stratum corneum, which become more conductive. This ionic shunt decreases the resistance of the stratum corneum since eqn. 11 becomes

$$R_{stratum\ corneum} \approx R_{pore} \quad (13)$$

Little effect should be observed in the skin capacitance, which is mainly related to the dielectric nature of the lipid matrix:

$$C_{stratum\ corneum} \approx C_{lipid\ matrix} \quad (14)$$

Secondly, there is the geometric effect: a dry electrode is only partially in contact with the skin. With the accumulation of perspiration beneath its surface, the electrode becomes wet.

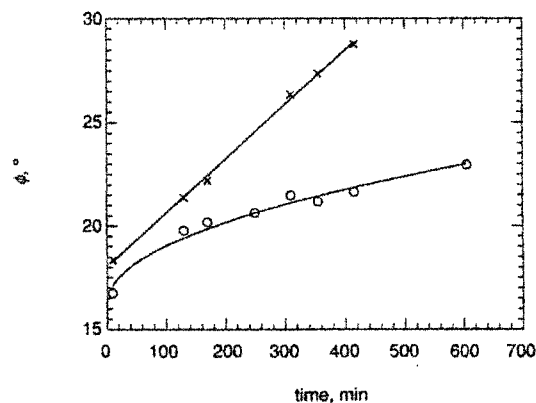
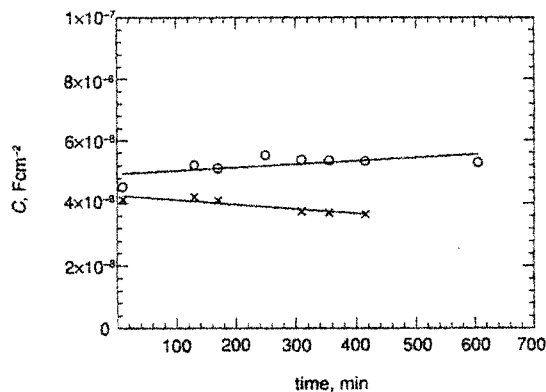
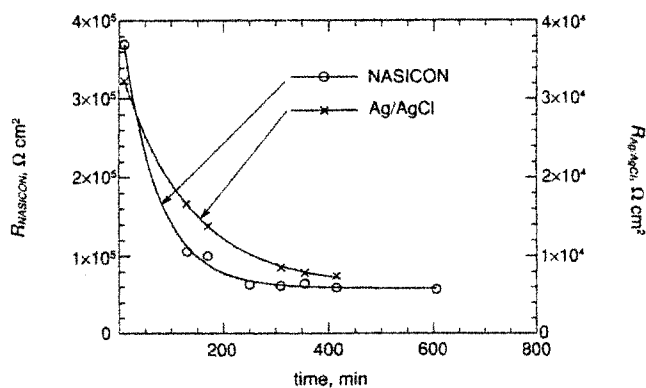


Fig. 7 Long-term variation of  $R$ ,  $C$  and  $\phi$  after application of dry NASICON-based electrode on skin; a pregelled Ag-AgCl ( $gSA^*$ ) electrode serves as control ○ NASICON × Ag/AgCl

This electrolyte spread on the skin surface increases the effective electrode surface area. This enhancement of the contact area is depicted in Fig. 8. The low-frequency distribution of the field lines inside the skin for a dry (Fig. 8a) and a wet (Fig. 8b) electrode are compared. As the skin becomes wet, the resistance is expected to decrease and the capacitance to increase.

These two phenomena are responsible for the abrupt change of the resistance and the enhancement of the capacitance observed during the first few minutes.

The slow long-term decrease of the resistance is of the same type as that observed during the hydration of skin tissue. It has been shown (FOLEY *et al.*, 1992) that the impedance of excised human epidermis exposed to water over a 20 h period decreases as a function of hydration time, whereas the capacitive value remains relatively constant. Some similar results were found (BURNETTE and BAGNIEFSKI, 1988) on excised nude mouse skin. The low penetration of the electrolyte

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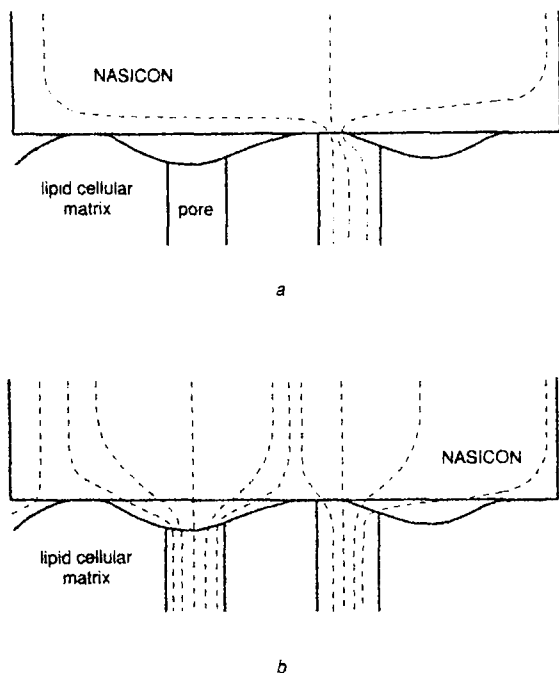


Fig. 8 Effect of electrolyte spreading due to perspiration process on distribution of field lines inside skin; (a) dry contact; (b) wet contact

inside the skin tissues can be responsible for the decrease in resistance observed over longer times.

Table 1 shows the skin-electrode resistance and capacitance for different patients. The impedance was measured 30 min after application of the dry NASICON-based electrode on the dry skin. A pregelled Ag-AgCl electrode fixed on the forearm serves as the reference. The values are of the same order of magnitude as that found for dry electrodes (GRIMNES, 1983). The variations reflect the different sweat duct filling during the measurements.

As for classical Ag-AgCl and metallic electrodes, the skin condition is also an important parameter. Table 2 shows the resistance and capacitance of the NASICON-based electrode-skin contact obtained for different skin preparations. The impedance was measured 5 min after application on the skin. Addition of a sodium chloride solution decreases the resistance and increases the capacitance. As expected, abrasion which removes part of the stratum corneum also causes the resistance to decrease markedly and the capacitance to increase. In agreement with previous work (YAMAMOTO and YAMAMOTO, 1976), the resistance decrease is more important than the capacitance increase. The resistance is divided by 630, whereas the capacitance is multiplied by 17.

Table 1 NASICON-based electrode-skin contact impedance parameters; impedance is measured 30 min after application. Ag-AgCl electrode used as control

subject	electrode	number of tests	R range, $k\Omega cm^2$	C range, $nF cm^{-2}$
1	NASICON	5	83-600	15-51
	Ag-AgCl gSA	1	40	33
2	NASICON	3	152-162	30-126
	Ag-AgCl 3 M	1	85	28
3	NASICON	1	115	36
	Ag-AgCl 3 M	1	80	34
4	NASICON	1	600	11
	Ag-AgCl 3 M	1	97	21
5	NASICON	1	530	20

Table 2 R and C parameters for various conditions of skin preparation; measurements were carried on subject 1. Surface area of NASICON-based electrode was  $0.9 cm^2$

Measurement conditions	R, $k\Omega$	C, nF
dry NASICON/dry skin	760	36
dry NASICON/skin wetted with 1 M NaCl	20	80
dry NASICON/abraded skin	1.2	610
pregelled Ag-AgCl (gSA)/dry skin	19	70

## 6 Conclusion

A new electrode based on a NASICON-type ceramic has been proposed. This electrode does not need gel to measure a bioelectric signal and is slightly polarisable. It could therefore be interesting for application in which an electrode array is used (no occurrence of electrode bridging, owing to the gel) and for EEG recordings.

Electrical measurements performed in 1 M NaCl solution have shown that the EMF drift between two pairs of electrode never exceeds 3.5 mV, the DC polarisation for a pair of electrodes is lower than 35 mV when a current of 200 nA passes through the electrodes for a time period of 1 h and the 10 Hz impedance never exceeds the limit of  $1000 \Omega$  for a NASICON pellet 10 mm in diameter and 1 mm thick.

As for classical Ag-AgCl and metallic electrodes, the skin (stratum corneum) is the dominant part of the skin-electrode interface impedance. The resistive part has been observed to decrease with time after application, whereas the capacitive component is approximately constant. This is due to the perspiration process which occurs when the electrodes are applied on the skin. The skin condition greatly influences the skin-electrode interface impedance. The resistive component is more affected than the capacitive one.

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