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New Type of Capacitive Detector for Determination of Ions

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A new type of capacitive detector for the determination of ions was designed and fabricated. It consists of two thin layers of gold (thickness 24 nm) sputtered on a glass membrane (20 mm × 20 mm × 0.2 mm) as a dielectric. The upper metal layer of the capacitor had a circular gap (28 mm^2) in the center as the sensing area. The capacitor was coupled with an inductor in parallel configuration to form a resonance circuit, whose frequency was determined. Solutions containing ions applied to the sensing area caused an increase in the capacitance owing to an enhanced charge density on the sensing area. The upper limit of detection for ions was around 10^{-3} mol dm^{-3} of monovalent cations and anions. Higher concentrations did not produce higher capacitance. The lower detection limits were around 5×10^{-5} mol dm^{-3}. Studies were focused on the influence of the metal film thickness, size of the sensing area, response time, dynamic range, sensitivity, detection limit and long-term stability of the detector. The response was stable and reproducible for over 5 months. The signal responses were compared to conductivity measurements under flow injection conditions with standard solutions and mineral water samples. The results show a good correlation between both methods of measurement.

Key Words: Ion capacitive sensor, Capacitive detector, Transducer, Capacitive sensor, Ion-sensitive capacitor, Ion detector.

INTRODUCTION

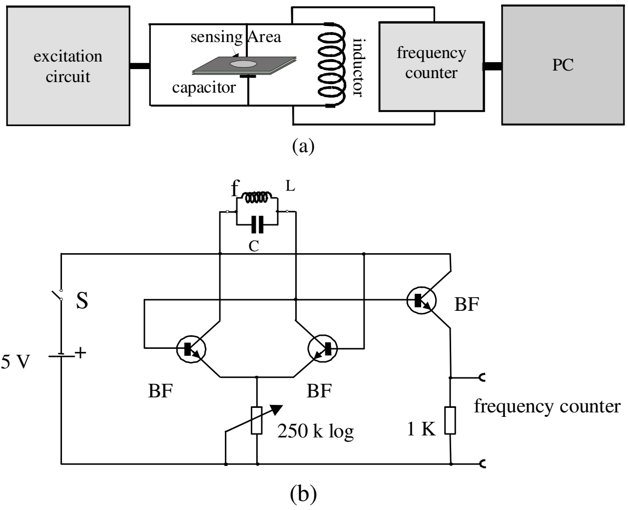
Chemical sensors are devices to yield information about the chemical composition of a system usually connecting chemical and electrical domains by transduction of the chemical information into an electric signal. In many applications the response of the sensors should be selective for the analyte. Moreover, the devices should be fast, inexpensive and have a lifetime in the order of months or years[1-4]. Ion-detecting sensors and transducers are increasingly becoming popular in clinical, biological, pharmaceutical, food, industrial and environmental analysis due to their high simplicity, fast response, feasibility, sensitivity and easy instrumentation[5,6]. They are usually based on potentiometric (ion-selective electrodes, ISEs) or conductometric techniques (detectors for flow systems). Capacitive sensors are nowadays widespread, either as mechanical sensors for monitoring accelerating and decelerating forces or as chemical sensors primarily for the determination of humidity in air[7,8]. Both types rely on a change of the permittivity of the dielectric of a capacitor, either by a moveable suspended inert mass or by adsorption or desorption of water. Indirectly chemical capacitors play an important role with ion-selective field-effect transistors, ISFETs, where the ion-sensing membrane constitutes in fact a plate of a capacitor[8]. Similarly, ion-sensitive capacitors, ISCAPs, can be based on the field effect of ion-sensitive layers in an electrolyte-insulator-semiconductor (EIS) structure[9-12]. Closely related and in fact of the same category as capacitive devices are impedance sensors because the frequency-dependent resistivity of a capacitor is measured[13].

The capacitance, *i.e.*, the charge storing capability of a plate capacitor, is dependent on the permittivity of the dielectric separating the plates, of the plate area and the plate distance. Whereas most of the conventional capacitive sensors are based on a change of the permittivity. To our best of knowledge, no report on transducer for a chemical sensor working on the change of the plate area. The work presented here deals with fundamental investigations studying the possibilities of designing ion capacitive sensors with practically constant permittivity and physical dimensions, but in fact changing the plate area by contact with ions. The focus was set on basic investigations of the operation mode and analytical parameters. The new type of ion capacitive detector seems very promising in a way that it could represent rather universal ionic detectors, similar to conductivity detectors that are simple, reliable and inexpensive for the quantification of anions and cations down almost to the micro molar level, with two enormous advantages. There are no Faradaic reactions occurring on the sensor surface and there is no need for a reference electrode as with other electrochemical sensors[14]. Miniaturization of similar designs would favour applications with chip-sized analytical systems.

EXPERIMENTAL

All reagents used were analytical grade (Fluka and Merck). All solutions were prepared with deionized water.

Fabrication of the capacitor: On glass lamellas (cover slides for microscopy, 20 mm × 20 mm × 0.2 mm) a self-adhesive paper of circular shape (diameter 4-10 mm, typically 6 mm) was attached in centre on one side. Gold was sputtered on both sides with a sputtering device (BAL-TEC, SCD 005 Sputter Coater). The thickness of the sputtered metal layers was controlled by the sputtering time and current (usually 180 s at 30 mA corresponding to a final thickness of 24 nm). The raw device was polished with fine sand paper on the four edges to remove the metal layer connecting both sides of the glass membrane. Copper braid wires (around 1 cm of length) were attached to each side of the capacitor by using a silver conductive glue. The other ends of the copper wires were soldered to an inductor (470 μH) to form an LC oscillating circuit. The whole device except the sensing area and a concentric contact zone around it (typically 2 mm) was sealed with a two-component epoxy glue (UHU plus, binder and hardener) to prevent solution-resistive effects (Fig. 1).



(a)

(b)

Fig. 1. Basic setup of the measurement (a) and excitation circuit (b), after[15] for the LC

The general scheme of the experimental set-up employed for measurements is shown in Fig. 1. It consists of the following parts: the oscillator consisting of the capacitor and an inductor (470 μH), an excitation circuit and a frequency counter (MXC-1600 universal counter, VoltCraft). The latter was connected to a personal computer *via* its RS 232 interface for recording the data.

The excitation circuit shown in Fig. 1a is able to stimulate the LC oscillators up to 100 MHz resonance frequency (modified after[15]). The excitation circuit was placed into a metallic housing. The whole arrangement with the detector was put into a metallic cage to eliminate parasitic signals from the environment.

Data recording and evaluation of data was done by a personal computer using a laboratory-written software in visual basic. In standard configuration the software read one data point per second from the frequency counter.

Measurement: After connecting the detector with the measuring system and switching on the excitation circuit the resonance frequency of the dry LC-oscillator was measured. Samples were applied by injection of 100-250 μL (typically 150 μL) of the test solution onto the sensing area.

Frequency spectra were recorded with a spectrum analyzer (Velleman PC-LAB 2000, Taiwan) controlled by the corresponding software on a personal computer.

RESULTS AND DISCUSSION

Operating principle of the ion capacitor: A capacitor is a device where two conductive layers are separated by an insulator (dielectric)[7]. Capacitors as chemical sensors usually rely on a change of the characteristics of the permittivity of the dielectric layer.

The concept of the capacitive ion detector presented here is different. The fundamental layout of two metallic layers (sputtered layers of gold) is separated by the dielectric (glass lamella) as in a conventional plate capacitor setup. On one side the layer has a zone without metallic film, representing the sensing area, which means that the area of the plate which facilitates charge accumulation on both sides, is the total area of the sensor minus this sensing area, because within the latter no charge compensation of eventually accumulated (or depleted) electrons in the back plane is possible (Fig. 4).

If a solution containing ions is applied, the ions may cover the sensing area and thus increasing the conductive area may act as counter-charges facilitating additional polarization of the backplane. According to the capacitance of a capacitor with parallel plates (eqn. 1) the capacitor's area virtually increases in case that the solution has contact with the surrounding metal layer because the dielectric permittivity practically remains constant.

As the amount of charges available in the solution is limited the change of the capacitance is a function of the concentration within certain limits; lowest capacitance is obtained with solutions with no or very small amounts of ions and highest when the sensing area is completely covered with ions.

$$C = \varepsilon\varepsilon_0 \frac{A}{d} \text{ with } A = f(c_i) \qquad (1)$$

C = capacitance, ε = dielectric permittivity, ε_0 = dielectric constant, A = area of the (smaller) plate, d distance between the two plates.

The fact that the permittivity stays constant was proved by the fact that no change of capacitance was observed in case that the solution did not have any physical contact with the gold layer surrounding the sensing area. In case that solvent molecule would penetrate into the dielectric and thus alter its permittivity, C should change independent on the contact.

Additionally it was found that apolar liquids not possessing a dipole moment did not cause any change of the capacitance of the dry detector, also in accordance with the model depicted above. Water possessing a dipole moment (and also containing small amount of ions) does provoke a capacitance change of the device.

Thus, with the capacitor presented here the use of a reference electrode, as it is common with other ion-sensitive capacitive devices to create a potential reference point in the solution, such as with ISCAPs and ISFETs, is obsolete because no membrane potentials are involved in its working principle. This simplifies the experimental setup significantly. On the other hand there is hardly any selectivity towards specific ions to be expected in the basic configuration of the detector except the size and the mobility of the ions.

Capacitor design: As sketched in the previous paragraph the design relies on the classical metal-insulator-metal sandwich structure (MIM) of a plate capacitor, which simplifies the construction and production of the capacitor significantly. A glass lamella, which is a usual cover slide for microscopic studies with a thickness of around 0.2 mm and a size of 20 mm × 20 mm, is sputtered on both sides with a thin gold metal film (usual thickness 24 nm). Before sputtering, on one side a mask of self adhesion paper with the shape of a circle (usual diameter 4-10 mm) is attached and removed after the deposition of the metal. Thus, a hole ("sensing area") is created in the metal film (Fig. 2c). The whole device except the sensing area and an ambient brim of the metal layer is sealed with an epoxy glue to prevent resistive effects between sensing and back layer when submerging the device in solutions.

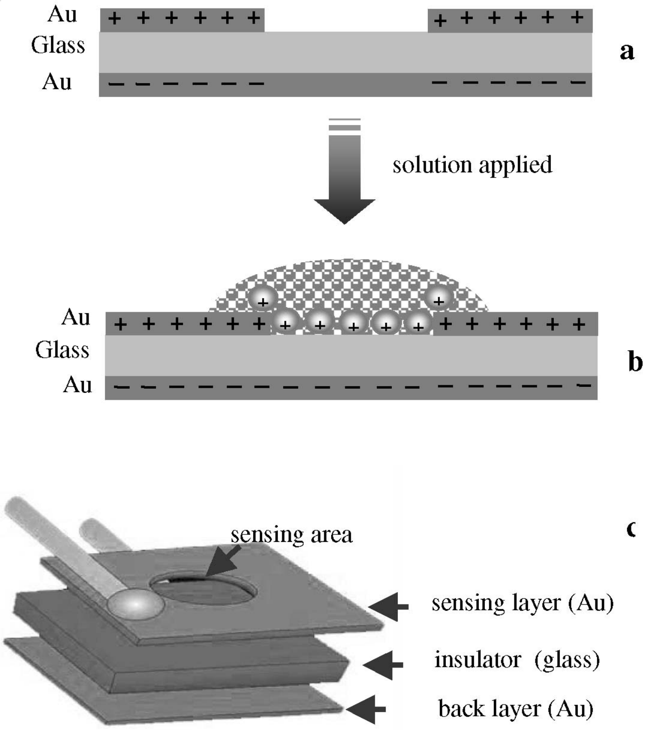


Fig. 2. Plate polarizations in the absence (a) and the presence (b) of a solution containing ions, and exploded view (c) of the capacitor

One of the simplest ways to determine the capacitance is to combine the capacitor with an inductor in parallel configuration generating an oscillating circuit where the resonance frequency ν is dependent on the capacitance C and the inductivity L (eqn. 2). As the inductance is constant (470 µH) the capacitance is related to the ion concentration applied to the sensing area.

$$\nu = \frac{1}{2\pi\sqrt{LC}} \tag{2}$$

Characterization of the detectors: The capacitor was characterized by changing design and experimental parameters which could influence its performance with respect to analytical applications. The dielectric medium (glass lamella) remained the same for all studies. The influence of type and thickness of the metallic layer, sensing and contact area were investigated in details. Moreover, the ion capacitor was evaluated with various ions in terms of linear range, sensitivity, detection limit, reproducibility, response time and long term use.

Influence of the type of sputtered metal: Two metals (gold and platinum) were studied with aqueous solutions of KCl, KNO_3, KI, NaCl and $CaCl_2$ (0.001 mol/L); the metal films of the capacitors were prepared with a thickness of 24 nm and a sensing area of 28 mm^2. The results are shown in Fig. 3. The responses of gold are proportionally higher than those of platinum. Obviously the charge-storing capacity of gold is higher than of platinum, probably due to its significantly higher electric conductivity (2.35×10^{-6} Ω cm *versus* 10.6×10^{-6} Ω cm specific resistivity). Nevertheless, both metals yield similar responses. Calcium chloride does not differ from salts consisting of monovalent ions only, because the concentration (0.001 mol/L) is practically in the saturation region of the detector. With lower concentrations differences may be noticed in the corresponding calibration curves due to higher equivalence concentrations.

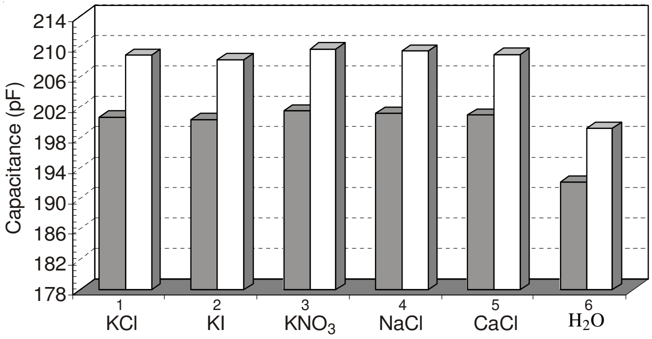


Fig. 3. Capacitance of detectors prepared from different metals (shaded bars Pt, white bars Au); concentration of salt solutions 0.001 mol/L; thickness of sputtered metal layer 24 nm; glass thickness of glass lamella 0.2 mm; total size 20 mm × 20 mm; sensing area 28 mm^2; inductance 470 µH; applied volume 200 µL; batch operation

Qualitatively there was no significant difference between the two sputtered metals. For subsequent studies capacitors with gold layers were used. The thickness and the homogeneity of the metal layer are of importance for the performance of the detector; the parameters were studies by means of scanning electron microscopy (SEM).

The thicknesses of the sputtered layers were varied from 6-31 nm. The results indicated that a thickness of 12.50 nm or more yields constant, reproducible capacitance. SEM studies showed that with this thickness complete coverage of the glass substrate occurs whereas more or less interconnected islands of metal deposits appear with thicknesses below. Therefore the thickness of the sputtered layers should be at least 12.50 nm. For the studies presented here the thickness of the metal layers was 24 nm which guaranteed sufficient homogeneity of the layers for producing reproducible detectors.

Influence of the size of the sensing area: Different diameters of the circular sensing area were studied comparing the capacities of the dry detector with the solution (Fig. 4). As expected two trends can be observed. First, the larger the sensing area is, the smaller is the basic capacitance with a constant total area. This is, of course, completely coherent with the model description presented above, because the metallized zone decreases with increasing sensing area and thus the charge-storing capacity decreases. On the other hand increasing sizes of the sensing zone produce more significant changes in capacitance after application of water or salt solutions because the increase of conductive area becomes more significant as well. From the data "relative metal film equivalents" (rmfe) were evaluated (Table-1). This parameter should somehow estimate the capacitance change which is produced by an equivalent coverage of the sensing area with a metallic film. The relative metal film equivalent was estimated by referring the capacitance of the detector with analyte (water or salt solution) to the capacitance of the dry device. The total capacitance without sensing area was estimated as 210 pF and was experimentally proven by completely covering the area with silver conductive glue. As can be seen in Table-1 the virtual metal-equivalent coverage decreases with increasing sensing area. This seems reasonable because there should be a significant influence of the brim of the metal film towards the sensing area due to the different shapes and sizes of the sensing plane and the back plane as well as resistive effects within the covering layer.

The dynamic range of the detectors was found to be independent on the size of the sensing area spanning in average a range of 5.0×10^{-5}-1.0×10^{-3} mol/L with a detection limit of 3.0×10^{-5} mol/L.

Types of ions and calibration curves: The chemical nature as well as the ion size and ionic mobility seem to be of some, but not very significant importance for the capacitance change of the device. A variety of different electrolytes were studied. The results are summarized in Fig. 5. Water yields a capacitance of the detector higher than air. The increased value is due to the dipole character and a small amount of mobile hydronium and hydroxyl ions of the solvent. Tests with non-polar organic solvent (*e.g.*, hexane) revealed that the resulting capacitance is practically identical to the capacitance of the dry detector. All solutions containing strong electrolytes (practically completely dissociated in ions without salt hydrolysis) show comparable result as expected. $CaCl_2$, containing 3 ions and having an equivalent concentration of two times the molar concentration of monovalent ions, does not exceed the capacitance of binary compounds significantly and is comparable to potassium nitrate because at a concentration of 0.001 mol/L saturation is practically obtained already. Due to the nature of the measurement, *i.e.*, an electric field alternating with the resonance frequency, it may be assumed that besides the size the mobility of ions exerts some influence as well, because during the quick polarity change the ions must reorient correspondingly. Thus, slight differences of the capacitance produced by different salts of the same concentration can be explained. Of course, weak electrolytes such as acetic acid provoke smaller effects because of incomplete dissociation.

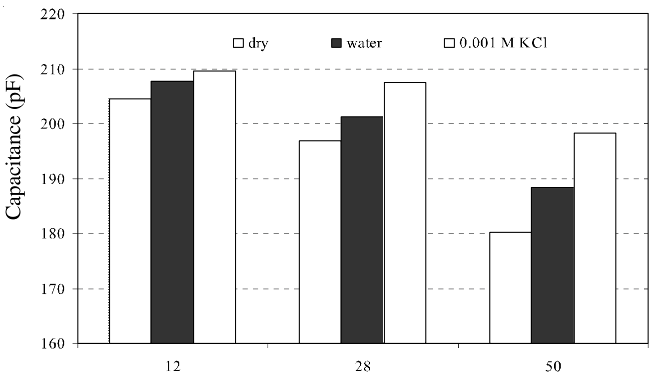


Fig. 4. Dependence of the capacitance on the size of the sensing area; gold layer thickness 24 nm; glass thickness 0.2 mm, total size 20 mm × 20 mm total size; inductance 470 µH; applied volume 200 µL; batch operation

TABLE-1
RELATIVE METAL FILM EQUIVALENTS FOR THE SENSOR IN DEPENDENCE ON THE SENSING AREA; PRACTICAL PARAMETERS AS IN FIG. 8

Sensing area (mm^2)	Water (%)	KCl 0.001 (mol L^{-1}) (%)
12	57.0	93.5
28	32.8	84.3
50	21.6	50.0

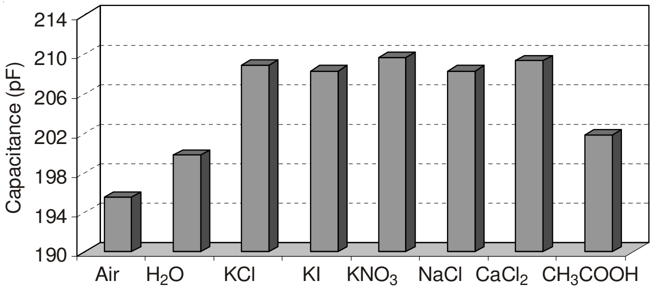


Fig. 5. Capacitance of the detector after application of aqueous solutions of various compound (0.001 mol/L); sensing area of 28 mm^2; gold layer thickness of 24 nm; 200 µm glass thickness with 20 mm × 20 mm total size; inductance 470 µH; 200 µL applied volume; batch operation

Fig. 6 shows the capacitance of the ion detector in dependence on the concentration. Sodium and potassium chloride behave practically identical, whereas the calibration curve is shifted towards lower concentrations with calcium chloride. The observed shift of the curves corresponds roughly to 0.3 due to double the amount of charges per formula unit as compared to alkali halides. Quasi-linear dynamic ranges can be determined from the calibration plot as 7×10^{-5}-1×10^{-3}, 1×10^{-4}-1.25×10^{-3} and 1×10^{-4}-1.25×10^{-3} mol/L, respectively, with linear regression equations as $y = -8.20x + 241.90$, $y = -10.20x + 246$ and $y = -10.35x + 245.75$ (y in pF, x as negative

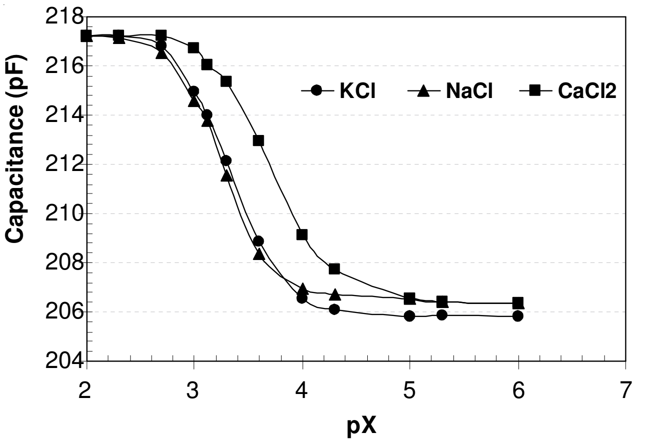


Fig. 6. Dependence of the capacitance on the type and concentration of electrolytes; sensing area 28 mm^2; gold layer 24 nm; glass thickness 0.,2 mm; total size 20 mm × 20 mm; inductance 470 µH; applied volume 200 µL; batch operation

decadic logarithm of the concentration) and correlation coefficients of 0.9986, 0.9988 and 0.9987, respectively. The sensitivities of the device for $CaCl_2$, KCl and NaCl are 8.20, 10.20 and 10.35 pF per concentration decade, respectively.

Table-2 summarizes the upper and lower detection limits, UDL and LDL, as determined from the data by intersection of the tangents of the quasi-linear parts of the plots of the calibration curves. In fact the actual dynamic range of the detectors is rather small, spanning one order of magnitude only. On the other hand, such devices might be ideal as "digital" sensors, indicating just the presence (> 10^{-3} mol/L) or absence (< 10^{-4} mol/L) of ions.

TALBE-2
UPPER AND LOWER DETECTION LIMITS FOR SALTS DETERMINED WITH THE ION CAPACITIVE SENSOR; SENSING AREA 28 mm^2; METAL LAYER 24 nm; GLASS THICKNESS 200 µm WITH 20 × 20 TOTAL SIZE; INDUCTANCE 470 µH; APPLIED VOLUME 200 µL; BATCH OPERATION

Salts	Upper detection limit		Lower detection limit	
	pX	mol/L	Px	mol/L
NaCl	2.6	1.6×10^{-3}	3.8	1.25×10^{-4}
KCl	2.6	1.6×10^{-3}	3.8	1.25×10^{-4}
$CaCl_2$	2.9	1.3×10^{-3}	4.4	4.00×10^{-5}

Response time: The response time of the ion capacitor is influenced by the process of formation of an equilibrium distribution of the cations and anions at the glass-solution interface where they can easily and quickly change positions to follow the polarity changes of the electric field. The result shows that the signal detected upon application of the solution (2.5×10^{-4} mol/L) to the detector is fully established in about 5 s where 90 % of the signal is obtained after 3 s. Thus, the response is reasonably fast. Very similar response times are found with higher and lower concentrations.

From point of view of ionic movements one would expect an even faster response; probably wetting of the sensor surface and of the metallic layer plays some role.

Long-term stability: Capacitors were stored in ambient air at room temperature over 5 months and tested each month with standard solutions. The devices showed excellent repeatability over 5 months with variations of the blank signal (air) of less than 0.4 % and of KCl solution (10^{-3} mol/L) of less than 0.2 %. Higher deviations after half of a year are probably due to the formation of gold oxide layers on the surface.

Reproducibility and repeatability: To determine the repeatability of the signal, a capacitor was repeatedly measured with air, water, air and solution; it was found to be usually better than 0.5 % RSD. In order to check the reproducibility three different detectors were prepared; the RSD was around 3 % which is acceptable.

Comparison of capacity and conductivity detection: In order to estimate the performance of the ion capacitor, it was used in a flow through cell with flow injection analysis. The detector response was compared to a conventional conductivity detector, which was inserted between the sample injector and the detector with the capacitive detector (Fig. 7). There was a clear relation between conductivity and capacitance (Fig. 8). A rather linear proportionality could be found between the capacitance and the logarithm of conductance (Fig. 8). As it is obvious from the previous investigations the upper limit of detection restricts applications of the ion capacitive detector to concentrations higher than 10^{-3} mol/L.

Samples: The ion capacitor and the conductivity detector were used to analyze water samples with flow injection analysis (Table-3 and Fig. 9). As can be seen, there is even a good quasi-linear relation for the samples between conductivity and capacitance, particularly if the measured values are not too close to the upper or lower limit of the capacitive detector.

TABLE-3
CONDUCTIVITIES AND CAPACITIES OF WATER SAMPLES; WORKING CONDITIONS: 24 nm GOLD LAYER; 50 mm^2 SENSING AREA; 200 µm THICKNESS GLASS SUBSTRATE WITH 20 mm × 20 mm TOTAL SIZE; 470 µH INDUCTANCE; 1 mL/min FLOW RATE; 100 µL INJECTION VOLUME; FLOW INJECTION ANALYSIS

Samples	Dilution (times)	Conductance (µS)	Capacitance (pF)
Tap water	10	39	201.5
Evian	10	43	201.7
Vittel	10	46	201.8
Sorgente linda	10	48	202.0
Voslauer	10	68	202.5
Juvina	25	80	203.1
Sicheldorfer	50	83	203.2
Johannisbrunnen	50	85	203.3
Romerquelle	10	92	203.4

Conclusion

A new type of capacitive ion detector is presented. The measuring device is made of two thin sputtered layers of gold as electrical charge storage pools separated by a glass membrane as a dielectric. A small area of the upper layer of the capacitor is spared and represents the sensing area. Measurements have been carried out by coupling the capacitor with an inductor coil in parallel configuration to form an oscillating circuit. The ions in solution cause a change in capacitance owing to the enhancing charge density on the sensing area. The sensors are inexpensive, simple to produce and use and stable during storage. Moreover, there is no need of a reference electrode as is the case with ISEs or ISFETs. The dynamic

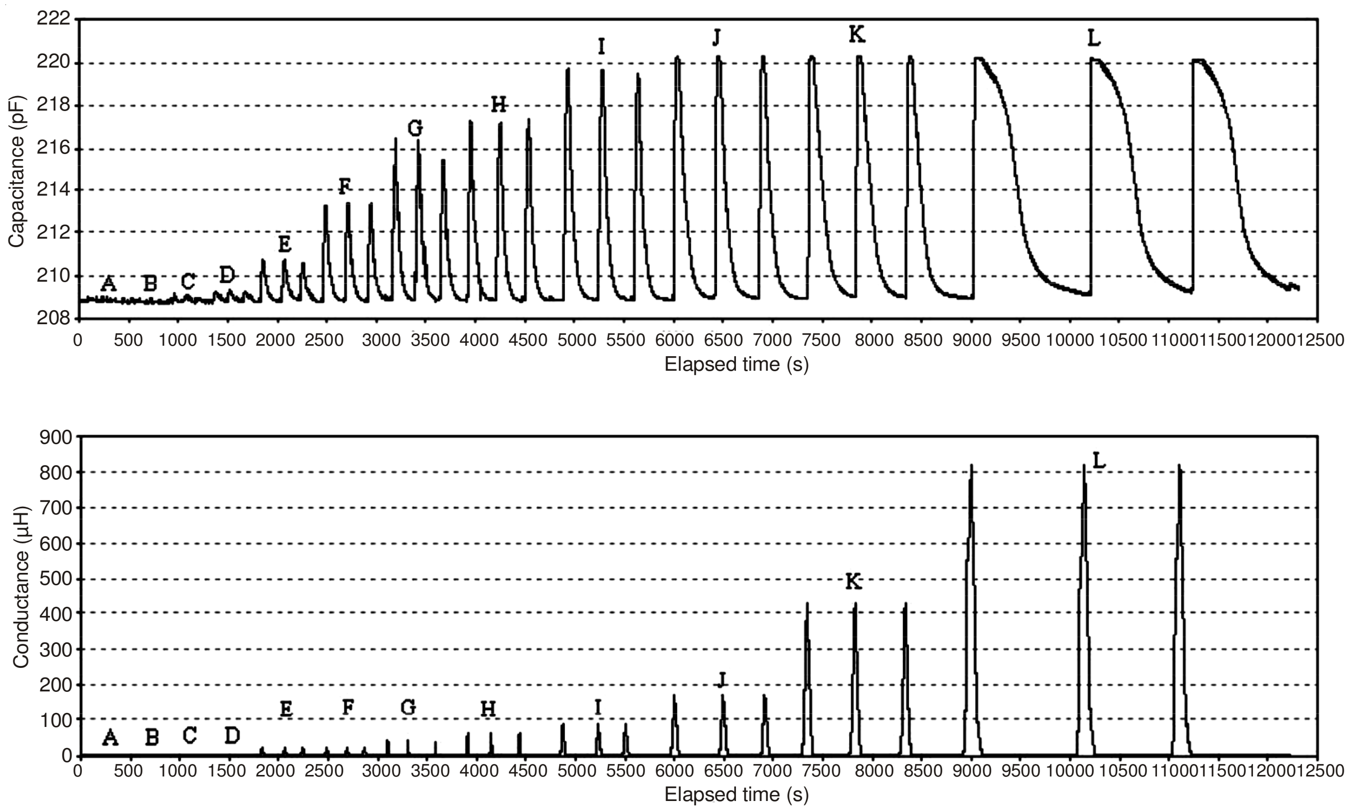


Fig. 7. Comparison of capacitance and conductivity detection with flow injection analysis; (A) 1 × 10^{-6}; (B) 5 × 10^{-6}; (C) 1 × 10^{-5}; (D) 5 × 10^{-5}; (E) 1 × 10^{-4}; (F) 2.5 × 10^{-4}; (G) 5 × 10^{-4}; (H) 7.5 × 10^{-4}; (I) 1 × 10^{-3}; (J) 2 × 10^{-3}; (K) 5 × 10^{-3} and (L) 1 × 10^{-2} mol/L KCl; 24 nm gold layer; 50 mm^2 sensing area; 200 µm thickness of glass substrate with 20 mm × 20 mm total size; 470 µH inductance; 1 mL/min flow rate; 100 µL injection volume; flow injection analysis

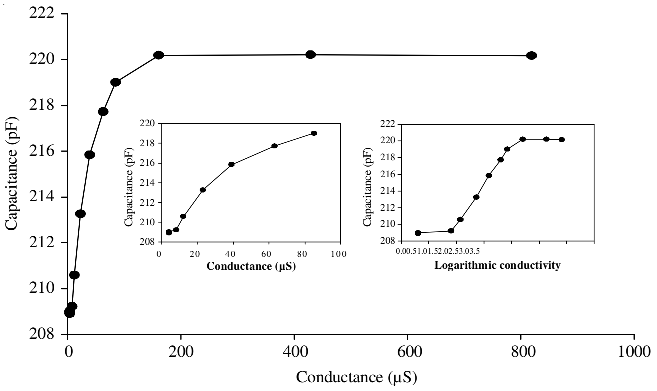


Fig. 8. Comparison of capacitance and conductance of KCl solutions in FIA; 24 nm gold layer; 50 mm^2 sensing area; 200 µm thickness of glass substrate with 20 mm × 20 mm total size; 470 µH inductance; 1 mL/in flow rate; 100 µL injection volume; flow injection analysis

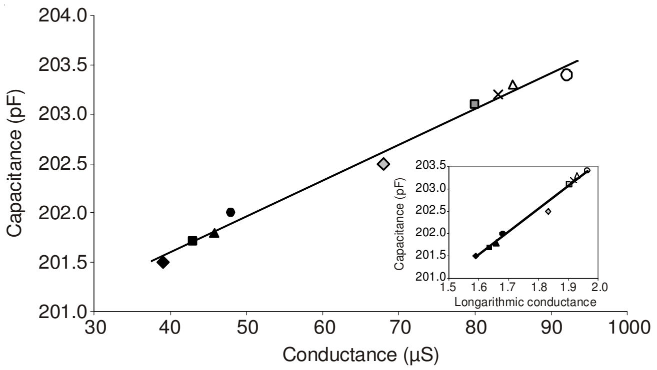


Fig. 9. Capacitance and conductance of water samples (◆ Tap water, ■ Evian, ▲ Vittel, ● Sorgente linda, ◇ Voslauer, ☐ Juvina, × Sicheldorfer, △ Johannisbrunnen and ○ Romerquelle). Working conditions: 24 nm gold layer; 50 mm^2 sensing area; 200 µm thickness of glass substrate with 20 mm × 20 mm total size; 470 µH inductance; 1 mL/min flow rate; 100 µL injection volume; flow injection analysis

range of the capacitor is rather narrow still. But preliminary investigations revealed that coverage with permeable membranes indicated that in fact the signal gets smaller, but the linear range widens. The performance of the ion capacitor corresponds to a conductivity detector. Water samples showed good correlation between the signals of both detectors. Thus, the capacitive detector seems promising for being applied in routine work. The concept of the capacitor seems very interesting in some respects *viz*., it can be easily miniaturized and placed on chips. It may be a simple alternative to conductance measurements. It can be easily applied as a detector in flow systems (comparable to conductivity detection) and it could constitute the transducer for more specific information on the ionic composition of samples by applying membranes with ionophores on the surface of the sensing area. All these topics are currently under investigation.

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