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High performance conductivity monitoring instrument with pulsating sensor

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We present an unconventional but high performance pulsating conductivity monitoring instrument constructed by using a new class of sensor, viz., pulsating sensor developed in house. The design consists of a special type of logic gate oscillator (LGO) powered by 5 V direct current (dc). The conductivity cell constitutes a part of LGO. In this conductivity meter the primary signal generated from the sensor is directly in digital domain which is easily transmitted either to a personal computer or to a stand-alone embedded system. Hence it reduces the hardware components making the instrumentation highly simplified. The output of the instrument is pulse frequency which is directly related to the conductivity of solution used for measurement. The relation between pulse frequency and conductivity is determined following a multipoint calibration technique developed in our laboratory using a series of KCl standards. The performance of the instrument is demonstrated with a few examples which reveal the diverse application of this new approach of measurement technique. © 2010 American Institute of Physics. [doi:10.1063/1.3449552]

I. INTRODUCTION

Conventional conductivity monitoring instruments designed for monitoring ionic conductivity of solutions work on the principle of alternating current bridge circuit.^{1–3} The use of alternating current with sufficiently high frequency avoids electrolysis and polarization. In this type of instrument the primary attention has been focused to measure conductivity of ionic solutions at different measurement ranges starting from less than $0.1 \mu\text{S cm}^{-1}$ to several mS cm^{-1} . Accordingly many high precision instruments with different types of conductivity probes are readily available and are being used at present. Moreover conductivity instruments with four electrode system are recently introduced to avoid polarization effect and the fouling of electrodes during measurement of highly conducting solution.⁴ A low cost battery operated conductivity meter using operational amplifiers has been reported for class room demonstration.⁵ da Rocha *et al.*⁶ reported a low cost high performance conductivity meter composed of a triangular wave generator, a current-to-voltage converter, and a precision half-wave rectifier implemented with only one integrated circuit TL084. This instrument was designed for conductivity measurement of solution in the range less than $0.1 \mu\text{S cm}^{-1}$ to 20 mS cm^{-1} . Munoz *et al.*⁷ presented an analog electronic conditioning circuit which converted electrical conductivity of solution into voltage. The authors have focused their attention to avoid polarization in order to give reliable results in the entire range.

Besides mere conductivity measurement it is possible to

deploy the conductance based measurement techniques in many physicochemical applications in a simpler way if the conductivity monitoring technique and the methodologies are suitably designed for rapid monitoring of shift in conductance with high precision and sensitivity. With this objective we have developed appropriate conductivity sensing and measurement systems which operate entirely in digital domain with time dependent monitoring facilities in single or multichannel modes with or without using personal computer (PC). This inexpensive but high performance conductivity monitoring systems have emerged progressively for basic and applied measurements in many physical and analytical applications.

II. SENSING METHODOLOGY

In our laboratory a new class of digital sensors has been developed and deployed in many physicochemical applications.^{8–17} Unlike conventional sensors these sensors generate the first electronic response in digital domain, avoiding intermediate signal processing stages of preamplification, postamplification, analog to digital conversion, etc. Sensors are constructed to respond very sensitively to shifts in one of the four properties namely: (i) resistivity (or conductivity), (ii) dielectric permeability, (iii) inductance, and (iv) electromotive force of the physical or physicochemical system being probed. These four different classes of sensors, individually or in combination, offer an extensive scope for monitoring diverse parameters. Any parameter that causes either of the above property to change, directly or indirectly, becomes measurable. In each of these four types of sensors the signal output is digital pulse frequency which carries information of the physical parameter being sensed. Hence these sensors are called pulsating sensors. The relation be-

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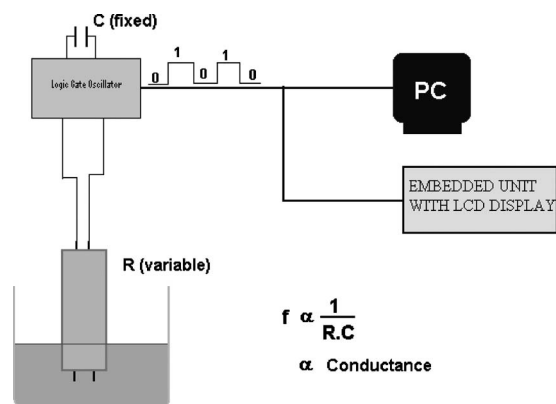


FIG. 1. Schematic representation of a miniaturized LGO (conductivity cell constitutes a part of LGO).

tween frequency and the sensed parameter is determined by appropriate calibration. Toward conductance based measurements the following approach is being followed.

The conductivity sensor consisting of a pair of inert electrodes placed in the sensing medium is a part of a miniaturized resistance-capacitance type logic gate oscillator (LGO) and it constitutes the resistance of LGO. A fixed capacitor is used in the circuit. When the LGO is powered by 5 V dc it oscillates between two logic states: “0” and “1.” Thus, the signal generated by LGO is a train of rectangular pulses of 5 V amplitude from which the pulse frequency (f) is determined. These digital pulses can be interfaced with any digital input/output card for further data processing without any auxiliary amplification or signal processing. The relation between frequency and conductance in this timing oscillator circuit is given by the following equation:

$$f \propto 1/(RC), \quad (1)$$

where f =frequency, R =resistance, and C =capacitance.

For conductance based sensors C is kept constant and R is variable. Since $R=1/K$, where K is the conductance, Eq. (1) can be presented as

$$f \propto K. \quad (2)$$

Since, $K=\kappa/x$ where κ is the conductivity and x is the cell constant, Eq. (2) can be presented as

$$f \propto \kappa. \quad (3)$$

For a narrow range of application the frequency and conductivity show excellent linear relation. However for a wide range of application it has been observed that the linearity deviates. Hence in order to avoid error in measurement we have adopted a multipoint calibration technique to bring out a relation between frequency and conductivity of solution as described in Sec. V. The principle of conductivity measurement using a miniaturized LGO is illustrated in Fig. 1.

III. INSTRUMENTATION

For measurements, the probe specific oscillator is either connected to a PC via a power supply cum signal routing unit or connected directly to a stand-alone embedded processor with built-in power supply, pulse counting, and computation facilities or can be connected to a handheld frequency

meter. The instrumentation has also been simplified for multichannel measurements where signals from different probes are handled simultaneously.

The electronics for signal routing and data acquisition is designed to perform the following operations: (i) counts the input pulses from all the input channels simultaneously for a fixed duration, (ii) determines the frequencies, (iii) converts the frequency to appropriate parameter being sensed from the pulsating sensor using preloaded coefficients of polynomial equations, (iv) displays both frequency and corresponding parameter in graphical interface, and (v) stores the data in a nonvolatile memory with time stamp.

The pulses received from the sensor can be treated in two different ways. The train of pulses can be sent directly to the PC through its parallel port along with a reference frequency. Since the output is transistor-transistor logic pulses, no other pulse conditioning circuits are required. The indigenously developed software processes the train of pulses and calculates the frequency. It further converts frequency to conductivity using a predetermined mathematical relation between frequency and conductivity.

In case of stand-alone system the pulses are sent to a 16 bit counter interfaced with embedded controller, which counts the pulses for a fixed duration of time. The frequency is being calculated by registering the number of pulses and counting duration. The microcontroller calculates the frequency and converts it into the measured parameter using a polynomial equation. Both frequency and conductivity are displayed either in liquid crystal display or in light emitting diode and stored in a nonvolatile memory with time stamp. The configuration of stand-alone system is done by using PC via serial port interface. Block diagrams of conductivity monitoring unit with complete instrumentation and embedded microcontroller unit are shown in Figs. 2(a) and 2(b), respectively.

IV. CONDUCTIVITY PROBES

There is a lot of flexibility in probe design depending upon specific application. In some cases the probe specific miniaturized oscillator is directly fixed into the probe head using epoxy resin and in some cases it is placed separately near the probe. The noise free signal from LGO can be easily transmitted to a long distance, about 200 m without any loss of signal. Such advantage with simple instrumentation could be realized due to evolution of such pulsating sensor. The schematic representation of some of the conductivity probes made for conductivity monitoring in static, dynamic, or flowing systems is given in Fig. 3. They are mostly made using Teflon rods of various dimensions depending upon the nature of application. A pair of inert platinum electrodes is used as the sensing material. Probes as shown in Figs. 3(b) and 3(a) are of vertical types specially designed for general laboratory use and for industrial applications, respectively. In each case a pair of Pt electrodes is fixed into the bottom part and connected to the associated electronics of LGO housed in the upper portion of the probe. All electronic components housed in the probe are embedded using epoxy resin. Probe as shown in Fig. 3(a) is pipe mountable type which is used as

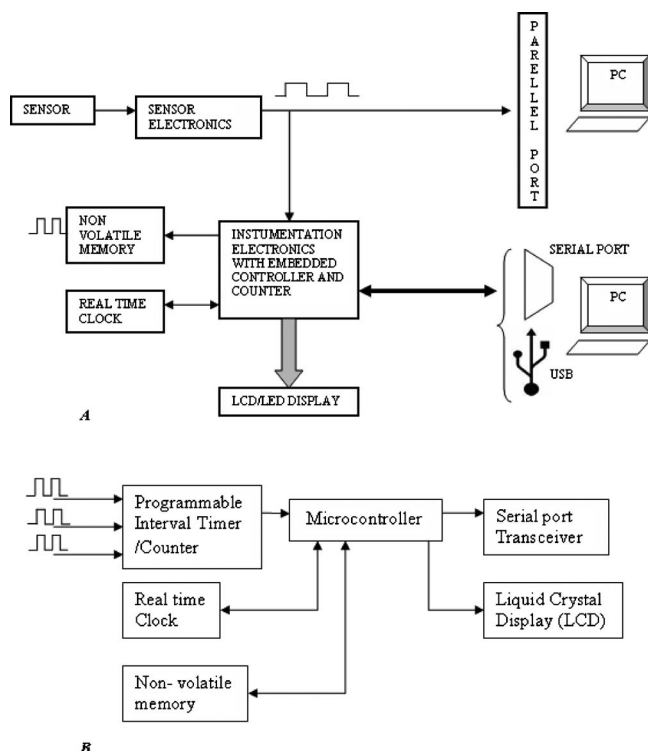


FIG. 2. Block diagrams of instrumentation for (a) conductivity monitoring instrument and (b) embedded microcontroller.

a flow cell in a pipe line. Stainless steel flange provided in the probe helps to fix it onto the pipe line. Flow type conductivity probe as shown in Fig. 3(c) is used in sampling line of steam generator unit of Fast Breeder Test Reactor,

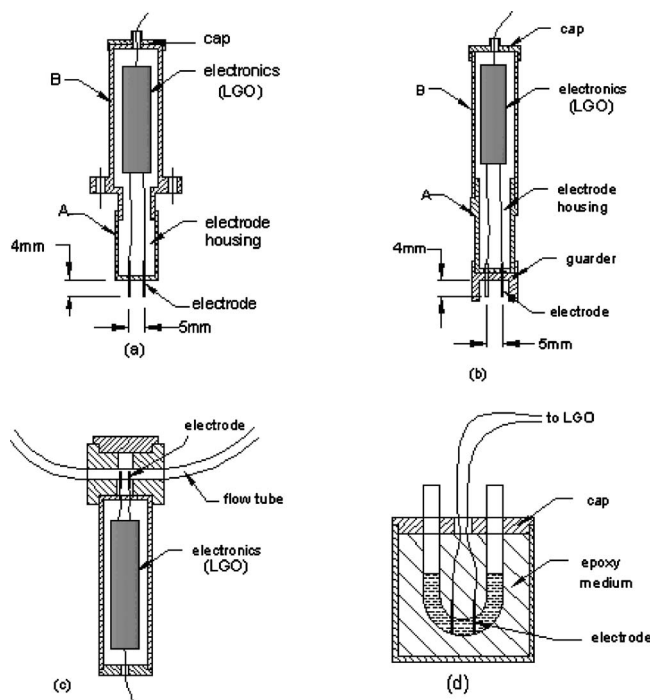


FIG. 3. Schematic of different types of conductivity probes. (a) Pipe line mountable probe for industrial application. (b) Vertical type conductivity probe for general laboratory use. (c) Flow type conductivity probe for online conductivity measurement of high purity water. (d) A miniconductivity cell for conductivity measurement in a small volume of sample (0.5 ml).

Kalpakkam, India in order to monitor conductivity of high purity water round the clock. It is clearly seen in the figure that the electrodes are fixed onto a small Teflon tube and LGO is mounted over the probe head. This flow type cell is connected to the sampling line. In some specific study it is required to measure conductivity of a very small volume of sample solution. For such application we have designed a cell using which about 0.5 ml sample can be measured comfortably.

V. CALIBRATION OF CONDUCTIVITY SENSOR

Usually the conventional conductivity meters are calibrated using single point calibration technique, i.e., by using cell constant approach.¹⁸ It is well known that this method uses only one standard KCl solution for calibration. In the present work we have introduced a multipoint calibration technique to establish a relation between pulse frequency and conductivity. A series of standard KCl solutions whose conductivities cover the entire application range is selected. Frequency of each standard is measured using the conductivity meter. In order to evaluate the conductivity of KCl solutions used in above application, the following technique is adopted. The equivalent conductance values of a series of KCl solutions available in literature are used.¹⁹ A relation between equivalent conductance versus square root of $[KCl]$ following the Debye-Huckel principle¹⁹ is determined ($[KCl]$ represents the concentration of KCl expressed in gram equivalent l^{-1}). Using this relation, the equivalent conductance of each standard KCl solution taken for calibration is evaluated. From the relation between equivalent conductance and specific conductance as shown in Eq. (4), the specific conductance of each KCl standard is determined:

$$\kappa = (\Lambda[KCl])/1000, \quad (4)$$

where κ =specific conductance ($S\text{ cm}^{-1}$) and Λ =equivalent conductance ($S\text{ cm}^2\text{ equivalent}^{-1}$)

A calibration plot using frequency and specific conductance is generated. Using least squares fitting, a polynomial relation between frequency and specific conductance is evaluated which is used in the system software to give direct display of conductivity value during measurement. A calibration plot using a typical conductivity cell is shown in Fig. 4.

VI. PERFORMANCE OF CONDUCTIVITY MONITORING INSTRUMENT

We present a few examples, both in analytical as well as in physical applications, which have been successfully carried out in simpler way using this digital conductometry.

A. Conductivity measurement in dilute KCl solutions

Before describing the specific examples we demonstrate the response of our PC based conductivity monitoring device toward small change in conductivity of aqueous solution with stepwise addition of dilute KCl solution. To 10 ml Millipore distilled water (DW) in a laboratory made conductivity cell 0.01M KCl was added in 10 μl steps. The solution was stirred continuously for homogeneous mixing. The

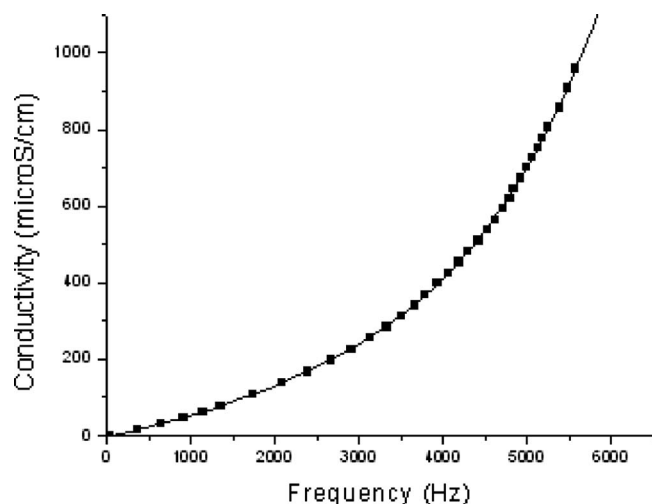


FIG. 4. Pulse frequency to conductivity relation for a typical laboratory made conductivity cell, range: from 1 to 1000 $\mu\text{S cm}^{-1}$ [relation between frequency and conductivity is shown by the following equation: $\kappa = 1.6937 + 0.03009f + 2.6536e - 5f^2 - 7.1589e - 9f^3 + 1.24962e - 12f^4$, where κ : conductivity ($\mu\text{S cm}^{-1}$) and f : frequency (Hz)].

real time shift in conductivity due to stepwise addition of KCl was captured online which is shown in Fig. 5. Results of conductivity measurements in each step of addition are shown in Table I. It shows very fast response (less than 20 ms) with excellent precision (relative standard deviation $< 0.5\%$) and high sensitivity. It is quite obvious that conductivity shift less than 50 nS cm^{-1} is easily detectable.

In order to check the performance of our instrument we measured the conductivity of a series of sample solutions with wide range of conductivities and compared the results with those obtained from a commercially available conductivity meter. Results obtained from both the instruments agreed well (refer to Table II).

B. Response toward concentration of boron at $\mu\text{g l}^{-1}$ levels

In order to demonstrate the minor shift in conductivity with stepwise change in concentration of boron at $\mu\text{g l}^{-1}$ levels in a specially treated aqueous solution the following experiment was conducted. To 50 ml of DW in a conductivity cell, 1 g of mannitol was added. The solution was stirred to mix uniformly. The background conductivity of mannitol

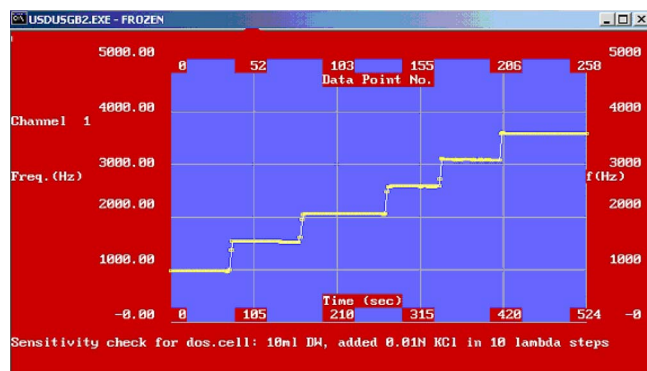


FIG. 5. (Color online) A typical online plot to show small change in conductivity during stepwise addition of KCl to DW.

TABLE I. Response toward small shift in conductivity with stepwise addition of dilute KCl to DW (0.01 M KCl added to 5 ml DW in a conductivity cell in 5 μl steps, sensitivity: 368 Hz per $\mu\text{S cm}^{-1}$, and conductivity shift is less than 50 nS cm^{-1} is detectable).

	Conductivity ($\mu\text{S cm}^{-1}$)	Digital pulse frequency (Hz)
DW	1.2	976 ± 3.1
Step 1	2.6	1534 ± 2.3
Step 2	4.0	2063 ± 1.5
Step 3	5.4	2587 ± 1.9
Step 4	6.9	3093 ± 1.9
Step 5	8.3	3594 ± 2.4

solution was measured. To this standard boric acid solution containing 1 mg l^{-1} B was added in 0.5 ml steps. Small shift in conductivity due to addition of boric acid to mannitol solution was recorded online (refer to Fig. 6). The results are shown in Table III. Boric acid reacts with mannitol to form boron mannitol complex which is also a weak acid; but it has better conducting power than boric acid. It is quite interesting to note that a change in concentration of boron by 56 ppb results to a change in conductivity by 0.17 $\mu\text{S cm}^{-1}$ only. Such a small shift in conductivity due to change in concentration of boron at ppb level could be easily measured with excellent precision using the present technique. This interesting finding inspired us to develop a suitable analytical technique for rapid assay of boron in mixed oxide fuel of Fast Breeder Reactor (FBR).

C. Trace level analysis and chemical speciation

Rapid conductivity monitoring technique using pulsating sensor is deployed to develop an innovative rapid titration facility.¹¹ Such a facility was used for rapid assay of boron down to sub-ppm levels present as boric acid in aqueous solution. Though the absolute conductivities are sensitive to impurity ions, the conductometric titration technique can be easily followed for assay of boric acid in presence of impurities. The dilute solution of boric acid containing HCl as impurity was mixed with mannitol and titrated against standard NaOH. Figure 7 shows a high resolution conductometric titration plot for assay of boron at sub-ppm levels in aqueous solution containing HCl and trace CO_2 as impurities. From titration plot distinct regions corresponding to each chemical specimen present in the sample solution could be easily identified. The sudden decrease in conductivity in the first region is due to replacement of highly conducting H^+ ion (equivalent conductance: $350.1 \text{ S cm}^2 \text{ equivalent}^{-1}$) by less conducting Na^+ ion (equivalent conductance: $50.1 \text{ S cm}^2 \text{ equivalent}^{-1}$).²⁰ The undissociated boric acid-mannitol complex (represented as HX) reacts with NaOH as per the following reaction:



Due to progressive addition of NaOH during titration the nonconducting HX is replaced by conducting Na^+ and X^- ions. Hence it shows a positive slope in the second region.

TABLE II. Comparison of results. (For each sample, five measurements were taken to evaluate precision in measurement.)

Description of sample	Conductivity ($\mu\text{S cm}^{-1}$) using pulsating conductivity meter	Conductivity ($\mu\text{S cm}^{-1}$) using conductivity meter. Model: Hach Sension 378
KCl solution 1	2.45 ± 0.02	2.45 ± 0.08
KCl solution 2	3.56 ± 0.03	4.10 ± 0.11
KCl solution 3	7.89 ± 0.03	7.50 ± 0.13
KCl solution 4	9.67 ± 0.03	10.21 ± 0.43
KCl solution 5	879.78 ± 2.22	855.45 ± 6.3
KCl solution 6	1201.72 ± 3.34	1243 ± 6.78
KCl solution 7	956.06 ± 3.40	959.55 ± 5.55
DW 1	1.23 ± 0.01	1.19 ± 0.06
DW 2	1.13 ± 0.00	1.18 ± 0.05
DW 3	1.1 ± 0.01	0.94 ± 0.04
Deminearlised water 1	1.20 ± 0.00	1.20 ± 0.05
Deminearlised water 1	1.18 ± 0.02	1.25 ± 0.04
Sea water 1	$53,078 \pm 50$	$54,447 \pm 258$
Sea water 2	$56,087 \pm 54$	$55,678 \pm 317$
Sea water 3	$58,956 \pm 51$	$59,234 \pm 222$
Tap water 1	320.56 ± 2.21	323.23 ± 5.11
Lake water 1	338.89 ± 2.29	338.12 ± 3.33
Lake water 2	376.56 ± 3.12	382.00 ± 3.39

Slope change in the third region with increase in conductivity is due to presence of residual CO_2 in the sample. Further steady increase in conductivity with slope change in the fourth region is due to addition of excess NaOH . Hence all reacting species present in the sample are quantitatively determined using this simple titration approach. This titration technique will be suitable for rapid assay of boric acid in heavy water moderator of pressurized heavy water reactor. The technique was also demonstrated in the quantitative determination of chemical species in sodium aerosols produced in sodium fire.⁸ We also reported assay of carbon in uranium powder/pellets and solutions of uranyl nitrate using this technique.⁹

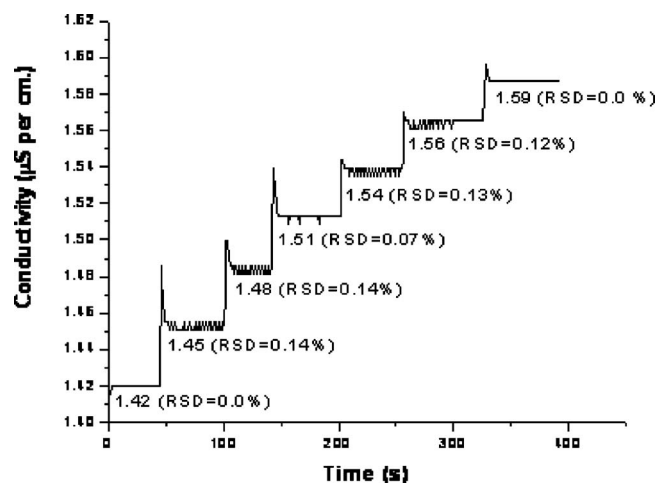


FIG. 6. Response toward online shift in conductivity during addition of boric acid at trace level to 2% (w/v) mannitol solution.

D. Toward development of online radiation dosimetry

The conductometric technique has been utilized toward development of a low cost online dosimeters based on *in situ* measurement of a radiation induced reaction product. The feasibility studies were carried out with saturated solution of chloroform in water. Its conductivity increased with time due to progressive buildup of highly conducting HCl on exposure to gamma irradiation. It was followed online with an appropriately designed test devices. About 5 ml of saturated chloroform in water was taken in a small conductivity cell. The cell was kept inside a Co-60 chamber. It was connected to a miniaturized LGO kept outside the radiation chamber. The signal received from LGO was transmitted to a stand-alone system and in some cases to a PC placed about 100 m away through a signal routing cum power supply unit. Figure 8 shows an online plot of increase in conductivity due to accumulation of conducting HCl on exposure to radiation. Results are highly encouraging and low cost online dosimeters are well in sight.

TABLE III. Shift in conductivity as a function of boric acid concentration at ppb level.

Concentration of boron ($\mu\text{g l}^{-1}$)	Conductivity ($\mu\text{S cm}^{-1}$)	Shift in conductivity from blank solution ($\mu\text{S cm}^{-1}$)
0.0	1.42 ± 0.0	0.0
9.9	1.45 ± 0.002	0.03
19.6	1.48 ± 0.002	0.06
29.12	1.51 ± 0.001	0.09
38.46	1.54 ± 0.002	0.12
47.61	1.56 ± 0.002	0.14
56.6	1.59 ± 0.0	0.17

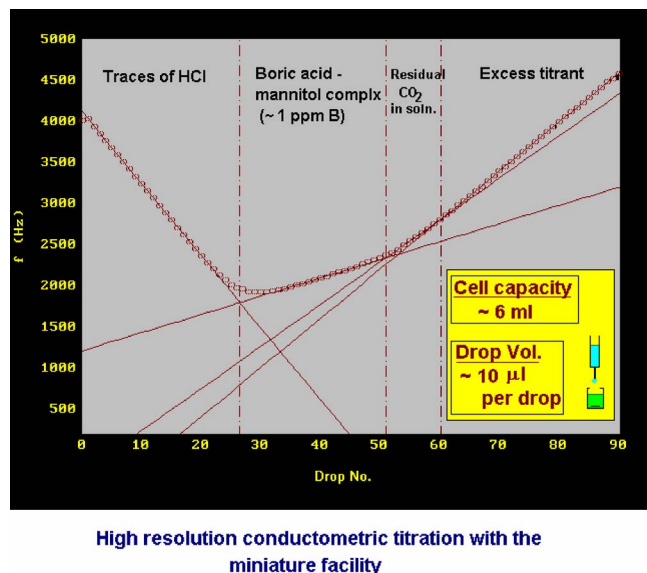


FIG. 7. (Color online) High resolution conductometric titration plot for assay of boron in aqueous solution.

E. Characterization of liquid surface oscillations

A PC based plug-and-use type stand-alone real-time system with very little auxiliary hardware was developed and deployed for online characterization of free surface oscillations of the water pool in $\frac{1}{4}$ -scale model that simulates hydrodynamic and flow induced vibrations in liquid sodium pool of Prototype Fast Breeder Reactor, Kalpakkam, India. The probe was made to respond to rapid changes in conductance as the water level fluctuated within a pair of electrodes immersed partially in an agitated water pool. The pulse frequency at a given moment carried information with respect to the prevailing water level within the electrodes at that instant. A simple procedure was used for generating a calibration equation for relating the pulse frequency to immersion depth.

The automated measurement process, starting from pulse recognition and counting to presentation of various data in real time, was executed entirely by a specially developed user-friendly software with the following features: (i) determination of pulse frequency as a function of time (at intervals less than 20 ms), (ii) online visualization of oscillations along the electrodes, (iii) graphic display of immersion depth as a function of time, (iv) expanded online view of subsets,

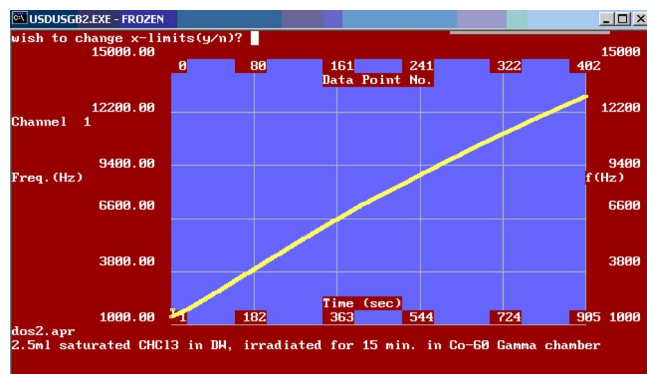


FIG. 8. (Color online) Online plot of increase in conductivity of a typical chemical dosimeter exposed to gamma irradiation.

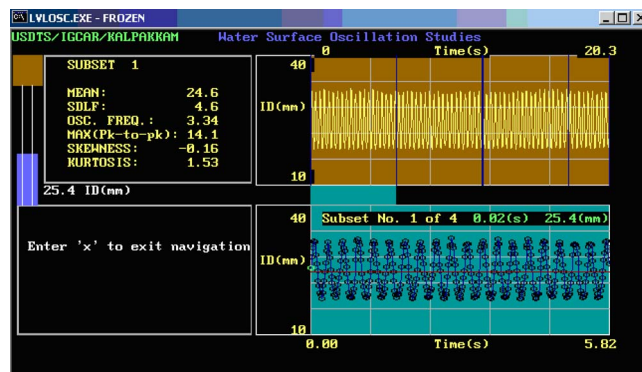


FIG. 9. (Color online) A typical output from a pulsating conductivity monitoring system, characterizing the water surface oscillations simulated by an orbital shaker driven laboratory setup. The bottom window gives the expanded view of the first subset in the run. All parameters for this subset are displayed simultaneously.

and (v) online computation of mean immersion depth (level), standard deviation of level fluctuations, oscillation frequency, peak-to-peak amplitude, skewness and kurtosis, and their display at the end of every subset.

Schematic representation of a shaker driven laboratory set up for generation of rapidly fluctuating water surface and a typical output of the characterization system capturing the oscillations is given in Fig. 9.

VII. DISCUSSION

Some of the examples described above give a clear idea regarding various miscellaneous applications of conductometric technique besides high precision measurement of conductivity of solutions. Rapid response, high precision, high resolution, and high sensitivity in conductivity measurement enabled us (i) to capture small shift in conductivity due to change in boron concentration at ppb levels in an appropriately treated aqueous solution and (ii) to detect small shift in conductivity due to formation of very dilute hydrochloric acid from aqueous solution saturated with chloroform exposed to gamma radiation. Hence it is possible to evolve rapid analytical technique for assay of boron in mixed oxide fuel and to develop a cost effective online radiation dosimeter. Similarly the evolution of a new type of rapid titration facility using pulsating sensors widened a scope for trace level analysis with chemical speciation in much simpler way. Due to very fast response of conductance measurement with online data acquisition we could successfully characterize liquid level oscillation in the context of FBR application. Toward development of a novel technique for void fraction measurement in coolant circuit of pressurized heavy water reactors using conductometric technique work is in progress.¹⁷

VIII. CONCLUSION

Evolution of an unconventional conductivity monitoring instrument as described in this paper widened the scope of conductometry in various applications. With a simple concept it was possible to address many critical applications. Some of the physicochemical applications are briefly described in this paper in order to give awareness to the readers about the

latent potential of conductometry. These examples bring out the enhanced scope for conductometry through pulsating conductivity meter. Direct generation of digital pulses as primary signal which helps in easy signal transmission without any loss, excellent noise immunity, flexibility in probe design, multichannel measurement with online visualization of consequences of induced changes at trace levels, lower detection limits, high sensitivity, high precision, and high speed with rapid response are the key factors behind their high performance.

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