Chapter 2
Experimental Techniques for Conductivity Measurement And a brief Review of Earlier Literature
CHAPTER 2

EXPERIMENTAL TECHNIQUES FOR CONDUCTIVITY MEASUREMENT AND A BRIEF REVIEW OF EARLIER LITERATURE

2.1 INTRODUCTION

Objective of this chapter is to deal with the theoretical aspect of the equivalent circuit of electrical conductivity cell and various methods are employed for the measurement of the electrical conductivity of solutions and also special attention is given to DC contact technique of conductance measurement, which is the technique adopted in the present study. A brief literature review on the technique and instrumentation is included at the end of the chapter followed by the description of the experimental technique developed in the present study.

2.2 ELECTROLYTIC CONDUCTIVITY CELL – THEORETICAL ASPECTS

A Cell consisting of two platinum electrodes dipped in an ionic solution can be represented by an equivalent circuit having combination of $R_{w1}$ and $R_{w2}$ are the resistances of the connecting wires which are usually negligible, $C_{dl1}$ and $C_{dl2}$ are the double layer capacitances of the two electrodes, $C_p$ represents the interelectrode capacitance in parallel with the cell and $R_{sol}$ is the resistance of the solution between the electrodes. $R_{f1}$ and $R_{f2}$ represent the Faradic
resistances at two electrodes (i.e. the electrical equivalent of any possible electrode reactions) are represented by $R_{f1}$ and $R_{f2}$ to such a circuit.

![Fig 2.1 Equivalent circuit of conductance cell](image)

- Faradaic resistances at electrodes ($R_{f1, f2}$)
- Connecting wire resistances ($R_{w1, w2}$)
- Solution resistance ($R_{sol}$)
- Double layer capacitances ($C_{dl1, dl2}$)
- Interelectrode capacitance ($C_p$)

A small dc voltage applied to such a circuit, a brief transient occurs and no electrochemical process takes place. If the applied voltage is increased, the current will flow through the resistive components and $R_{sol}$. On the other hand, if an ac voltage is applied, alternating current will flow through the $C_{dl}$'s, $R_{sol}$ and $C_p$. This prevents building of voltages across $R_{f1}$ and $R_{f2}$ which would lead to Faradic current flow. This situation demands for a small value of $C_p$ and large $C_{dl}$'s, so that the effect of solution resistance ($R_{sol}$) can be studied by itself.

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Practically, the double layer capacitance is increased by coating the platinum electrodes with spongy platinum black, which also greatly enhances the effective surface area. \( C_p \) is significant only in case of high-resistance solution, when large electrodes close together must be selected in order to keep resistance within the large of the measuring instrument.

2.2.1 Types of Conductivity Cells:

(a). 2-POLE CELL

In a traditional 2-pole cell, an alternating current is applied between the 2 poles and the resulting voltage is measured. The aim is to measure the solution resistance (\( R_{sol} \)) only. However the resistance (\( R_{el} \)) caused by polarisation of the electrodes and the field effect interferes with the measurement, and both \( R_{sol} \) and \( R_{el} \) are measured.

(b). 3-POLE CELL

The 3-pole cell is not as popular now as it has been replaced by the 4-pole one. The advantage of this design was that the third pole which was linked to pole 1 allowed the field lines to be guided and confined in an optimal manner, limiting dispersion in the measurement and minimizing influences on the measurement such as beaker volume and position of the cell in the beaker (field effect). It guarantees a better reproducibility when determining the cell constant and therefore more reproducible results.

(c). 4-POLE CELL

In a 4-pole cell, a current is applied to the outer rings (1 and 4) in such a way that a constant potential difference is maintained between the inner rings (2 and
3). As this voltage measurement takes place with a negligible current, these two electrodes are not polarized (R2 = R3 = 0). The conductivity will be directly operational to the applied current. The geometry of 4-pole cells with an outer tube minimizes the beaker field effect, due to the measurement volume being well defined within the tube. The position of the conductivity cell in the measuring vessel or the sample volume therefore has no influence on the measurement.

2.3 EXPERIMENTAL TECHNIQUES

Electrolytic conductivity can be measured as the ability of a solution to carry an electric current. When a voltage in applied between two electrodes immersed in the solution, an electric current flows due to the migration of ions under the influence of the electric field. As in case of metallic conductors, solutions of electrolytes also obey the Ohm’s Law, with deviations arising only in cases of high voltages & high frequency currents being passed through the solution.

There are various techniques by means of which conductance of a solution may be determined. These techniques are broadly classified as -

• DC contact measurement.
• Null comparison measurement.
• Capacitive contact measurement.

A brief explanation of the individual techniques is as follows -
2.3.1 DC Contact measurement

One of the important aspects in the measurement of conductance is establishing an effective contact between substance ordinary metallic conductors. If this is achieved, measurement becomes very simple. The ideal contact between the test substance and measuring device should have perfect conductance and introduces no additional voltages into the circuit such as the thermal or electrochemical potentials. A deviation from this occurs in case of dissimilar conductor composition. Metallic contacts for most of the devices, substances and measurements are sufficiently idle and the simple relationship of Ohm’s Law can be used as the basic of measurement. An Ohmmeter is a device that provides the resistance of conductor in Ohms \( \Omega \), when connected across the ends of the conductor. A simple circuit of this kind is shown in figure 2.2

After initial adjustment, i.e. short-circuiting the probe and adjusting the potentiometer to lead full scale \( (I_1) \), then the total resistance of the circuit is given by

\[
R = R_m + R_f + R_a \quad \text{----------------------------------------------- (2.1)}
\]

![Fig 2.2 Ohmmeter](image.png)
When the test probes are connected across the unknown resistance ‘$R_u$’,
the current value decreases to $I_2$, which is depends on the value of ‘$R_u$’.

Therefore,

$$I_2 = I_1 \frac{R}{R + R_u}, \quad R_u = [I_1/I_2 - 1] R \quad \text{--------- (2.2)}$$

If suppose, a battery of 1.5v and 1 mA meter is used, the total circuit
resistance will be 1.5 KΩ, for full-scale deflection.

$$R_u = [I_1/I_2 - 1] 1.5 \times 10^3 \quad \text{--------- (2.3)}$$

If mid-scale reflection $I_2$ is made $I_1/2$. The unknown resistance is
1.5KΩ. Then the relationship between the unknown resistance and current
measurement is linear only for a certain range.

This drawback can be overcome by using linear- resistance-to-voltage
converters. From Ohm’s law, $V=IR$, it is observed that the voltage across a
resistor is proportional to the resistance for a constant current value. For
relation $I=VG$, $G$ is the conductance, which is proportional to the current
flowing through the resistor for a constant voltage. Therefore, the first case
requires a constant current source and second one requires a Current
measurement with negligible voltage. The above requirements can be met by
an operational amplifier.

As shown in fig 2.3, $R_i$ is connected to the voltage source $V$ and a
current $I=V/R_i$ is generated as a result using operational amplifier. This current
is proportional to the conductance of $R_i$ because the potential of the summing
point is maintaining zero potential, the input current $I$, produces an output voltage.

$$V_o = I R_f$$
$$V_o = -\frac{V}{R}, R_f = -\frac{V}{G}R_f \quad \text{(2.4)}$$

Thus the circuit produces an output voltage directly proportional to the conductance of $R$, within the output voltage and current capabilities of the operational Amplifier.

Fig 2.3 Current follower circuit for resistance measurement
2.3.2 Null comparison measurement

In this technique, the effect of an unknown resistance must be compared with the effect of a variable standard resistance under nearly identical conditions as possible. This can be realized by keeping the unknown and standard resistances can be kept in two arms of a wheat stone bridge, and varying the standard resistance until the difference between the voltages or current between the two circuits is zero.

Such an arrangement is shown in fig 2.4

![Null comparison measurement wheat stone bridge](image)

**Fig 2.4 Null comparison measurement wheat stone bridge**

When the bridge is balanced (the well-known condition is \( P/Q = R/S \)), we have

\[
\frac{R_c}{R_v} = \frac{R_a}{R_b}
\]

i.e. \( R_c = R_v \left( \frac{R_a}{R_b} \right) \) \( \text{------------------- (2.5)} \)

Therefore, the unknown resistance \( R_c \) can be determined by knowing the values of standard resistance values \( R_a \) and \( R_b \), and the variable resistance \( R_v \).
An assumption made in the DC contact and null comparison measurement discussed above is that the probe leads and contact to the measured conductor are assumed to be ideal. An ideal and contact has zero resistance and has no voltage generated by thermal, electrochemical or any other effect. Generally, the above discussion applies for high resistance measurements. For low resistance measurement, the four contact methods shown in fig 2.5 (a) are suitable. But the demerit with this technique is that it cannot work with electrolytic solutions. For example, it is not possible to measure the resistance of a copper Sulphate solution using an ordinary ohmmeter. The reason for this is that the contact between the metallic electrode and electrolytic solution is far from ideal. This problem is overcomed by using one of the capacitance contact techniques described in the following sections.

Fig 2.5 (b) depicts the type of cell arrangement used for the measurement of electrolytic conductance. In this technique, the conductance of the part of solution that is in the capillary tubing between the two vials of the H cell, the reference type of electrodes are commonly used for the voltmeter probes.
Fig 2.5 (a) Low Resistance Measurement Four Contact Method

Fig 2.5(b) Four Contact Measurement of Electrolytic Conductivity

Fig. 2.5(b) Four Contact Measurement of Electrolytic Conductivity
2.3.3 Capacitive contact measurement of conductance

The basis of this technique is avoiding the effect of potential differences arising at the electrode solution interface by taking advantage of the capacitative behaviour of the double layer at the electrode surface to make AC conduction with the solution. At relatively low frequencies, a conductance cell may be represented as the double layer capacitance $C_d$ in series with the solution resistance $R_s$ as shown in fig 2.6(a) if the excitation voltage is sinusoidal of voltage $V_s$ as in Fig. 2.6(b), then the instantaneous current $I$ is given by,

$$i = I \sin wt$$  \hspace{1cm} (2.6)

$I$ = the maximum current

$w$ = angular frequency (rad/s)

$t$ = time (s)

From Ohm’s law, the instantaneous voltage $V_{Rs}$ across $R_s$ can be written as

$$V_{Rs} = I R_s \sin wt$$  \hspace{1cm} (2.7)

Similarly the voltage across the capacitance may be obtained as

$$V_c = I X_c \sin (wt-\pi/2)$$  \hspace{1cm} (2.8)

Where $X_c$ is the capacitive reactance

$X_c = 1/\omega C_d$  It is the measure of opposition to the flow of charge in a capacitor and hence it is also expressed in ohms.
Fig. 2.6 (a) Equivalent circuit of conductance Cell at low frequencies
(b) Current voltage Relationships
(c) Frequency dependence of impedance and phase angle
The relation between $X_c$ and frequency is inverse in nature i.e. $X_c$ decreases as frequency increases and vice-versa. Typical values for the double layer capacitive reactance for aqueous solutions are 10-100 $\mu$F/cm². Thus, the capacitive reactance for a 1cm² electrode with a 10-$\mu$F capacitance at an applied frequency of 1.6 KHz is 10 $\Omega$.

Another important parameter is the impedance denoted as 'Z', which is the total opposition to the current in an AC circuit, for the network under consideration.

$$Z = \sqrt{X_C^2 + R_s^2} = \sqrt{(1/wcd)^2 + R_s^2} \quad (2.9)$$

From expressions 2.8 and 2.9, it is clear that as the signal frequency is increase i, $X_c$ decreases, $z$ approaches $R_s$ and consequently $G$ approaches $1/Z$. The potential across $C_d$ also decreases gradually and the phase angle $\phi$ between $V_s$ and $i$ approaches zero, as shown in fig. 2.6(c).

It is clear from above discussion that it is desirable to measure $R_s$ at high frequencies, in order to make $X_c$ negligible compared to $R_s$. But certain other problems arise at high frequencies. The main idea is to decrease $X_c$ An usual method of realizing this is to increase the surface area and hence the capacitance by platinizing the electrodes which is the process of electrodepositing a layer of platinum black on to the platinum electrodes, usually from a solution of chloroplatinic acid.

When the frequency of the applied signal $i$, above several KHz, even then the impedance of the conductance cell tries to deviate from $R_s$. Since the
solution medium is itself a dielectric situated between two parallel charged surfaces, the equivalent circuit of such a situation is shown in fig 2.7(a). Here the signal is applied to a capacitor $C_p$ in parallel with solution resistance, $R_s$. The magnitude of the capacitance is given by the expression.

$$C_p = 10^9 \frac{D}{4 \pi c^2} \left(\frac{1}{a}\right) \quad (2.10)$$

Where $D$, $C$ and $1/a$ represent dielectric constant of the medium, velocity of light and the cell constant respectively. The dielectric constant of a dilute aqueous solution is approximately 80, and if the cell constant is assumed to be $1 \text{ cm}^m$, then the $C_p$ value will be about 10pf. If the applied signal frequency is 1.6 KHz, this results in a reactance $X_c=10M\Omega$. The same analysis holds good for a series circuit with the exception being that the voltages across $R_s$ and $C_p$ different but the currents $i_c$ and $i_R$ in $C_p$ and $R$ being same.

The instantaneous current are given by

$$i_c = \frac{V_s}{X_c} \quad \text{and} \quad i_R = \frac{V_s}{R}$$

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The current flowing through the resistor in phase with the applied voltage, while the current in the capacitor leads the applied voltage by 90° as shown in fig. 2.7(b). The net current is given by

\[ i = \frac{V_s}{\sqrt{X_c^2 + R^2}} \]

--- 2.11

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Therefore, the cell impedance is expressed as

\[ Z = \frac{V_s}{i} \sqrt{\frac{1}{X_c^2} + \frac{1}{R^2}} \tag{2.12} \]

If \( X_c \approx 10 \text{ M\(\Omega\).} \) Then current path through \( C_p \) becomes appreciable only when solution resistance approaches 100 K\(\Omega\). This kind of situation can be overcome by choosing the appropriate cell constant and concentration of electrolyte in the solution of interest. Other factors also try to add up to the parallel capacitance like, contact capacitance at the junction of the cell leads and the measurement system, lead capacitance and the capacitance due to the dielectric properties of the thermostating medium. This problem can be solved by using an oil bath rather than water bath for thermo stating and choosing a proper cell design.

Fig. 2.8 (a) shows the simplified representation of the combination of high and low frequency models of the conductance. The response of the system to an ac excitation is shown in fig 2.8(b).
Fig. 2.8 (b) Frequency dependence of impedance

From above the graph it is evident that the solution resistance can be measured effectively in the plateau or the narrow mid frequency range. For most of the solutions if the plateau is located, the solution resistance is directly computed by measuring the cell impedance. The following section describes the measurement of cell impedance.

2.3.4. Impedance measurements

In order to measure the electrolytic conductance, the double layer capacitance Conductance Cd and the capacitance across the solution resistance 'Cₙ' is to be compensated one method to realize this in the impedance measurement techniques. the block diagram of which is illustrated in Fig. 2.9 (a).
A sinusoidal signal is applied across the conductance cell. The resulting signal is amplified & detected in such way so as to produce an output signal proportional to the resistive component of the cell impedance. Fig 2.9 (b) depicts the circuit for measuring impedance of the cell based on the above principle. Diodes D1 and D2 perform the function of rectification (demodulation) of the resulting signal of the ac excitation of the cell (Fig. 2.9(c). the switches S₁ and S₂ are used to calibrate the output and selection of amplification factor respectively. The output of the circuit is directly proportional to the conductance of the cell (i.e. 1/Z₀).

(a) Block Diagram

(b) Circuit Diagram
Fig 2.9 Impedance measurement by phase selective demodulation

(C) Wave forms
(d) Circuit diagram of unknown cell

This type of instrument can be constructed very easily in the laboratory for a students use. Other area’s application of the circuits includes conductmetric titrations, reaction kinetic studies of slow reaction, liquid chromatography.

The technique of phase selective demodulation is used for frequency less than 10KHz. An advantage of this technique is that it separates the resistive (real) and capacitive (quadrature) components of the cell impedance. This is achieved by a process of cross correlation. The instrument use to accomplish this task and the waveforms are shown in Fig. 2.10. Waveforms (a) and (c) shows that the magnitude of the correlation integral is proportional to the resistive component of the cell impedance at constant frequency. A simple graphical multiplication of waveform (ii) and $V_s$ in (i) gives the correlation...
waveform (iii). That will cancel upon integration and unshaded region represents \( \int R V_s \, dt \). The cell in the circuit becomes impedance to the current follower. When a sinusoidal signal (\( V_s \)) is applied across. The cell, the output of the operational amplifier is proportional to and in phase with the current \( I \), as shown in waveform (i). Since \( I \) is in phase with \( V_R \), the bipolar square waveform (ii) is obtained. This is realized by a comparator so that the output alternates between the positive and negative limits of the op-Amp.
Fig. 2.10 Conductance measurement by phase selective
Demodulation Circuit schematic and waveforms.

The output of the comparator is fed to the four-quadrant multiplier, which performs the multiplication of the bipolar square wave with the input signal (sinusoidal) $V_s$. The output of the multiplier is then passed through an active low pass filter designed using op-Amp and the output dc voltage is
proportional to R. By using the phase selective demodulation technique an accuracy and resolution better than 1 part of $10^4$ over a frequency range of 0.005 Hz to 10KHz was obtained Fig. 2.11 shows an ac wheatstone bridge, which has been the traditional instrument of choice for accurate conductance measurement which are relatively free of capacitance effects. The balance condition for the AC Bridge is same as that for the DC Bridge except, when reactive elements are present, then the impedance vectors must be substituted for resistances in the arms of the bridge.

At the balance condition, the points B and D on the AC Bridge must be equal in magnitude as well as in phase. The balance point can be detected by an oscilloscope.

Assuming that the value of parallel capacitance of the cell is negligible, the bridge balances when $R_s = R_{cell}$ and $C_s = C_{cell}$. By balancing the bridge carefully, one can obtain accuracies of the order of 0.01%.

It is understood from above discussion, that it is the parallel plate capacitance and double layer capacitance, which poses problems with regard to the measurement of solution conductance.

Large current flow through the solution leads to the Faraday process, which charges the double layer capacitance. Also these capacitances are involved in measuring signal frequencies.
2.4 A BRIEF REVIEW OF EARLIER LITERATURE ON EXPERIMENTAL TECHNIQUES

A Bipolar current method for determining solution resistance was described by Daum and Nelson [6]. It involved successive application of opposite polarity to a conductance cell. The resulting voltage was integrated to determine the area of the voltage time curve. They have further shown that this area is proportional to the solution resistance, and is independent of the value of parallel capacitance $C_p$ over a wide range of values. The paper also details the instrumentation for rapid accumulation of data points. The instruments utility was demonstrated by the studies on KCl solution and acetyl chloride ethanolysis reaction.
A wide band, precision, dc coupled lock-in detector and gated integrator for electrochemical measurements was developed by Bentz et al [7]. The design was simple and was capable of measuring ac cell admittance from 0.005 Hz to 10KHz over an impedance range of $10^2 \Omega$ to $10^{14} \Omega$ with 1% absolute accuracy. The instrument was calibrated by studying the reaction rate of ethanol-acetyl chloride esterification by monitoring conductivity.

Caserta et al [8] developed a bipolar pulse conductivity system for use as a detector in chemical rate determinations. This instrument possesses a wide dynamic range ($10^{-1}$ to $10^{-8} \Omega$) and the signal-to-noise ratios of up to $6 \times 10^3$ for single data acquisition and $6 \times 10^5$ for ensemble averages of 2000 acquisition were obtained. The accuracy of the system was reported to be in the range of 1% to 0.005% without calibration and 0.02% with calibration. Discrete conductance measurements were done in 30 μs when operated at its maximum data rate. Numerical correction factor for temperature changes, which occur in the cell, were also illustrated in this paper.

An automatic measurement of the conductivity of an electrolyte solution by FFT (Fast Fourier Transform) electrochemical impedance spectroscopy was developed by Diard et al [9]. An new method was devised for synthesizing the equivalent circuit of the conductivity cell. A Hewlett Packard 3562 a dynamic signal analyzer was used to measure the electrolyte solution resistance. With this method, it was possible to envisage automatic process control by conductivity measurement.
Boiko and Boiko [10] have developed a new cell to measure the electrical conductivity of the solutions. The cell design made the unlimited dilution of solution in cell possible. A Siphon is used to maintain a constant solution level.

To eliminate various problems encountered with other methods, Johnson and Enke [11] have developed a new method. An added advantage of this method is its efficiency in measuring very rapid changes in conductivity. Application and limitations of several ac bridge techniques and models were analysed. The analysis is done with respect to polarization, the series ($C_a$) and parallel ($C_p$) cell capacitances and frequency required. By using a phase-angle voltmeter as a null detector in the conductance bridge the ideal frequency was shown to be proportional to $1/\sqrt{C_{dl} \cdot C_p}$.

**Review of Earlier Work**

Conductivity is a widely used parameter for characterizing the electrical properties of solids and liquids. Many researchers attempted to measure conductivity employing different types of techniques. As the dc conductivity measurements have many drawbacks like lack of sensitivity, accuracy and polarization effects at the electrodes, most of the researchers used different conductivity techniques as a function of temperature and frequency.

A detailed review of various experimental techniques used for the measurement of conductivity was covered in literature. Hence, instead of
taking a detailed review an attempt is made here to mention some of the missing links.

Conductance measurements were performed by Szaro [12] with the help of case sensitive detectors. He observed that self-balancing bridges are appropriate for conductance measurements if the loss tangent is low and phase sensitive detection is used to perform conductance measurements over a range of frequency.

Staudt [13] and Schon [14] developed an automatic system for frequency independent impedance measurements based on computer controlled network analysis. A microprocessor developed by Boukamp [15] to overcome the difficulty of measuring impedance in a specific range of frequency. He measured the impedance ranging from 0.1 mΩ to 1.2999 Ω by switching the internal reference impedance. HP 9825 computer controls this impedance analyzer.

The AC conductivity of solid electrolyte by Bruce and West [16] in terms of equivalent circuits consisting of resistors and capacitors. The ac conductivity of solid electrolyte is analysed by Bruce and West in terms of equivalent circuits consisting of resistors and capacitors. They found that ac response does not correspond to that predicted by simple ideal circuits; for instance semicircles in the complex plane. Empirical functions based on Cole-Cole Cole-Davison [17] expressions have been used to fit the experimental
results. They conducted two terminal ac measurements over the frequency range $10^{-3} - 10^7$ using a combination of bridge and automated phase sensitive detection technique as detailed below.

<table>
<thead>
<tr>
<th>Frequency Range</th>
<th>Details Instrumentation employed</th>
</tr>
</thead>
<tbody>
<tr>
<td>100Hz – 70KHz</td>
<td>Wayne Kerr B224 bridge Brook deal 9472 signal generator. Total accuracy better than 3% was observed.</td>
</tr>
<tr>
<td>70KHz – 7MHz</td>
<td>B602 Rf bridge SR268L combined source detector unit.</td>
</tr>
<tr>
<td>$10^{-3} - 10^4$</td>
<td>Schlumbega Soltrron frequency response analyser model 1170</td>
</tr>
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</table>

A three-electrode geometry for the measurement of conductivity developed Aswani Sharma and Bhatia [18] with a GRU (USA) capacitance bridge assembly model 1620A. They measure capacitance and conductance in the frequency range $100Hz - 10KHz$ and temperature range 180K-475 DC conductivity was measure using a Keithly electrometer model 160D. They found that at higher temperature $\sigma(\omega)$ exhibits a much faster increase with temperature and frequency dependent conductivity increases approximately linearly with frequency in the range $10Hz$-$10^8Hz$.

Werner Obwald Kelemen [19] devised a high temperature cell for automatic low frequency impedance measurements, which can be used for AC measurements over the frequency range 5Hz to 13MHz. This measuring cell has been used to determine the conductivity upto 600° C. AC resistivity
measurements using network analyzers, HP instruments number (1-1300MHz) and 4192A(5Hz-10MHz) was performed by Behrooz and Zettle [20] for high frequency measurements the sample were mounted in a two probe configuration while low frequency instrument allows four probe measurements. They modeled the sample as a resistor in parallel with reactive component.

By using Hawllet Packard 4191A network analyser, Smith, Baumard and Abelard [21] performed ac impedance measurements. They applied a time dependent voltage $\delta \sin (\omega t)$ across a ceramic sample and the response form the sample is characterised by the complex impedance $Z$ that is a function of $\omega$. The real and imaginary parts $Z$ i.e., $Z^1$ and $Z^{11}$ is determined by using a network analyser in 1MHz – 1GHz range.

Balaya and Sunandana [22] designed an electronic system based on quadrate oscillator, current to voltage converter and phase sensitive detector for measurement of AC conductivity and complex impedance at several frequencies upto 60KHz.

Kulkarni and his co-workers [23] carried out impedance measurements employing impedance analyser model HP4192A over wide range of frequencies 5Hz to 13MHz and temperature 42°C to 650°C. Kulkarni et al [24] and also developed PC-based automated impedance analyser system which can be used in the frequency range 1Hz to 30MHz and temperature range 299K – 573K. This technique is found to be useful for the study of bulk conductivity,
bulk capacitance, grain boundary resistance and grain boundary capacitance at different temperatures. They have also evaluated activation energies. Sanjeev Patil et al reported measurements on superionic materials. The impedance plots for certain range of temperature are found to have low frequency loops.

Subba Rao [25] and his co-workers carried out electrical conductivity measurements on some polymer electrolytes as a function of composition at room temperature employing Keithley Electrometer model 614.

Shareefunndin et al [26] fabricated an electronic system to measure complex admittance parameters using a phase sensitive detector in the frequency range 70-100KHz and temperature ranging 300 to 950K. From the complex admittance plots they evaluated bulk conductance of the sample through which ac conductivity was calculated. They observed that ac conductivity was found to increase in the high frequency region due to reduction in space charge polarization between sample and electrode interface at high frequencies.

Scope and Purpose of the Work

The review of earlier work presented in previous section reveals that bulk of the effort has gone into conductivity measurements by various techniques. All the measuring techniques seem to be quite expensive and complex. It is evident from the review of literature that very little work has been reported on the instrumentation aspects of the developed techniques. It
allows a researcher to borrow the knowledge of already established ac and dc circuit theory and characterize the sample as electronic equivalent circuit. This direct connection between the behavior of sample under test and that of an electronic model circuit consisting of discrete components facilitates physical understanding of the sample. It also provides a wealth of structural and ion-dynamics information about materials. So, a modest attempt is made here to develop an inexpensive, indigenous and a simple electronic instrument for the measurement of conductivity. Using DC contact measurement already presented in Chapter 1.

The complex impedance is in general measured using either bridge methods or oscillometric method, as is evident from the introductory chapter and reviews. Subsequently, quadrature method of detection is widely used for determination of complex impedance. Most of the available circuits are quite complex and involve several IC’s. In this work a modest attempt is made to develop a low cost simple circuit for measurement of complex impedance and conductivity.

1. It is proposed to design a simple laboratory audio generator in the frequency range 20Hz-20KHz.

2. It is proposed to design and develop pc-based system for conductance measurements, with the appropriate temperature controller.
2.5 AIM AND SCOPE OF THE PRESENT STUDY

The advantages and easy availability of the personal computers has increased manifold in the present days. This has led to their introduction in the Curriculum of both under-graduate & Postgraduate courses. Therefore, an attempt is being made to interface the personal computer with the experiment. It involves development of appropriate hardware and software. The software is developed using ‘C’ language.