MEASUREMENT OF BATTERY SEPARATOR RESISTANCES IN LOW IMPEDANCE CONDUCTIVITY
BY A. C. BRIDGE TECHNIQUES

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Measurement of the resistance of battery separator membranes is frequently accomplished by taking the difference between the resistances of an electrolyte filled conductivity cell with and without the separator inserted between the electrodes. For low resistance separators this may involve measurements of impedances of a few tenths of an ohm. It is shown via equivalent circuit analysis and experimental data on a low impedance cell filled with 45% aqueous KOH solution that ac bridge measurements of separator resistances can be seriously in error if proper account is not taken of electrode impedances.
A new separator material for alkaline batteries is presently under development. One of the criteria that membranes made from this material must meet is a low electrical resistance. Since resistance measurements are used during development to accept or reject a material, it is important that the technique used yield accurate values.

This investigation was undertaken to examine the reliability of measuring very low separator resistances ($< 0.01 \Omega$) in low impedance conductivity cells.

J. R. Dixon
By direction
TABLE OF CONTENTS

INTRODUCTION .......................................................... 3
EXPERIMENTAL ............................................................ 7
RESULTS AND DISCUSSION .............................................. 8
CONCLUSIONS ............................................................. 10

ILLUSTRATIONS

Figure Title Page

1. A.C. Wheatstone bridge for measuring equivalent parallel resistance
   \( R_p \) and capacitance \( C_p \) of a conductivity cell. 11
2. Approximate equivalent circuits of a conductivity cell containing
   (a) electrolyte solution; (b) electrolyte solution plus separator. 11
3. Conductivity cell for measurement of membrane resistances. 12
4. Equivalent parallel and series resistances and capacitances versus
   frequency of a conductivity cell filled with 45% KOH solution at
   25.0°C using unplatinized Pt electrodes. Insert is an enlarged
   plot of the data at high frequency. 13
5. Equivalent parallel and series resistances and capacitances versus
   frequency of a conductivity cell filled with 45% KOH solution at
   25.0°C using platinized Pt electrodes. 14
6. Equivalent circuit of conductivity cell containing electrolyte. 15

TABLES

Table Title Page

1. Calculated values of apparent electrolyte plus lead resistance
   \( R_p \) and apparent separator resistance \( R_{sep,app} \) as a function
   of double layer capacitance, \( C_{dl} \), and frequency, \( f \), for the circuits
   of Figs. 2a and 2b. Assumed value of the other components
   were \( R_L = 0.1 \, \Omega \), \( R_{sol} = 0.1 \, \Omega \) and \( R_{sep} = 0.01 \, \Omega \). 6

APPENDIX 1. Equivalent Circuit Analysis of Fig. 6. 16
INTRODUCTION

In order to increase the electrochemical efficiency, batteries are designed to have a low internal resistance. The major contributors to the internal resistance of a battery are the electrolyte and the separator. The separator is usually a membrane permeable to the electrolyte ions and is used to prevent direct mixing of the anolyte and catholyte. Efforts to improve battery performance are often centered on the development of a stable separator which contributes relatively little to the internal resistance, ideally, a small fraction of the electrolyte resistance.

Assessment of the contribution of a separator material to the battery internal resistance is typically made first by measuring the resistance of the electrolyte solution, $R_{sol}$, between two plane parallel electrodes in a conductivity cell. After this the separator is inserted between the two electrodes in the electrolyte filled cell and the new resistance, $(R_{sol} + R_{sep})$, measured. The contribution of separator to the internal resistance, $R_{sep}$, is then the difference between the two resistance measurements. Plainly an accurate measurement of $R_{sep}$ requires that $R_{sep}$ not be negligible compared to $R_{sol}$, i.e., $R_{sep}$ must contribute an accurately measurable increment to $R_{sol}$.

A case in point for the present paper is a separator for silver-zinc batteries using 30-45% KOH solution as electrolyte in which a good separator material may contribute as little as 15.5 $\Omega/m^2$ (0.01 $\Omega/in^2$) to the internal resistance. An accurate measurement of $R_{sep}$ thus requires that $R_{sol}$ be of the order of 155 $\Omega/m^2$ (0.1 $\Omega/in^2$) or smaller.

The usual method of carrying out resistance measurements of this type is by means of a conventional ac Wheatstone bridge at audio frequencies. The cell impedance is balanced against a parallel resistance-capacitance combination ($R_p$, $C_p$) as shown in Fig. 1. The balancing resistance $R_p$ is usually identified

with the cell resistance, $R_{\text{sol}}$ or $(R_{\text{sep}} + R_{\text{sol}})$. As has been pointed out numerous times in the literature\textsuperscript{2}, the equivalent ac circuit of the cell is not a parallel resistance-capacitance combination, i.e., it does not correspond to the balancing circuit configuration, $R_p$ in parallel with $C_p$. This in turn can lead to large errors in the measurement of $R_{\text{sol}}$ and $(R_{\text{sep}} + R_{\text{sol}})$, as shown below.

The simplest approximations\textsuperscript{2} to the true equivalent circuits of a low impedance conductivity cell containing electrolyte and electrolyte plus separator are shown respectively in Figs. 2a and 2b. $R_L$ is the resistance of the leads between the bridge and conductivity cell, and $C_{\text{dl}}$ represents the double layer capacitances at the electrode surfaces. Hence the true first approximation of the equivalent circuit of the cell has the resistive elements ($R_L$, $R_{\text{sol}}$, and $R_{\text{sep}}$) in series with a capacitance, $C_{\text{dl}}$.

For Fig. 2a, the complex impedance is

$$Z(a) = (R_L + R_{\text{sol}}) + \frac{1}{i\omega C_{\text{dl}}}$$  \hspace{1cm} (1)

where $\omega = 2\pi f$ is the angular frequency. If the circuit of Fig. 2a were measured on the bridge of Fig. 1, the equivalent parallel resistance would be

$$R_p(a) = \frac{\frac{1}{\text{Re}(1/Z(a))}}{R_L + R_{\text{sol}}}$$  \hspace{1cm} (2)

where $\text{Re}$ designates the real part of the complex number. Similarly the complex impedance of the circuit of Fig. 2b is

$$Z(b) = (R_L + R_{\text{sol}} + R_{\text{sep}}) + \frac{1}{i\omega C_{\text{dl}}}$$  \hspace{1cm} (3)

and the equivalent parallel resistance is

$$R_p(b) = \frac{\frac{1}{\text{Re}(1/Z(b))}}{R_L + R_{\text{sol}} + R_{\text{sep}}}$$  \hspace{1cm} (4)

In the usual procedure for determination of the separator resistance using a

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bridge like that of Fig. 1, the apparent separator resistance would be taken to be

\[ R_{\text{sep,app}} = R_p(b) - R_p(a) = R_{\text{sep}} \left[ 1 - \frac{1}{(R_L + R_{\text{sol}} + R_{\text{sep}})(R_L + R_{\text{sol}})(1/\omega C_{\text{dl}})^2} \right] \]  

(5)

In Table 1, we show the results of calculations via Eqs. (2), (4), and (5) of the apparent electrolyte plus lead resistance \( R_p(a) \) and the apparent separator resistance \( R_{\text{sep,app}} \) as a function of \( C_{\text{dl}} \) and frequency in the audio range. We have used values of \( R_L \), \( R_{\text{sol}} \), and \( R_{\text{sep}} \) typical for low resistance separator membranes in a highly conducting electrolyte such as 45% aqueous KOH solution. Clearly \( R_p(a) \) approaches the actual value, 0.2 Ω, of \( (R_L + R_{\text{sol}}) \) and \( R_{\text{sep,app}} \) approaches the actual value, 0.01 Ω, of \( R_{\text{sep}} \) only in the limits of high frequency and/or high double layer capacitance.

At present it is not known to what degree these limiting conditions have been satisfied in actual measurements of separator resistances. Indeed, it is similarly unknown to what degree the simple circuits of Fig. 2 are a sufficiently accurate representation of the cell for obtaining an accurate determination of \( R_{\text{sep}} \) by measuring the equivalent series resistance of the cell. We do note, however, from Table 1 that a correct determination of \( (R_L + R_{\text{sol}}) \) guarantees a correct determination of \( R_{\text{sep}} \). Consequently, in the present study we have carried out a series of resistance and capacitance measurements at various frequencies in order to determine what errors, if any, may arise in these determinations. The measurements were made on a cell used in measurements of separator resistances and filled with 45% KOH.
TABLE 1  Calculated values of apparent electrolyte plus lead resistance $R_p^{(a)}$ and apparent separator resistance $R_{sep,app}$ as a function of double layer capacitance $C_{dl}$ and frequency $f$ for the circuits of Figs. 2a and 2b. Assumed values of the other components were

$R_L = 0.1 \, \Omega$

$R_{sol} = 0.1 \, \Omega$

$R_{sep} = 0.01 \, \Omega$

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<th>$C_{dl}$ (\mu F)</th>
<th>$R_p^{(a)}$ (\Omega)</th>
<th>$R_{sep,app}$ (\Omega)</th>
<th>$R_p^{(a)}$ (\Omega)</th>
<th>$R_{sep,app}$ (\Omega)</th>
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<th>$R_{sep,app}$ (\Omega)</th>
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<td>-6.03 x $10^3$</td>
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<td>12.9</td>
<td>-0.593</td>
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<td>$10^3$</td>
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<td>$10^6$</td>
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EXPERIMENTAL SECTION

The conductivity cell used in these experiments was obtained from RAI Research Corporation and is shown in Fig. 3. It consists of two heavy gauge square Pt electrodes, 2.54 cm on a side mounted plane parallel and 0.25 cm apart in a lucite holder. The cell can be disassembled and the electrodes removed. The cell also contains a provision for inserting a separator film between the two electrodes. This feature was not used in the present study.

In order to assess the effect of electrode double layer capacitance, measurements were carried out for two electrode conditions: bright, polished platinum electrodes (unplatinized) and electrodes coated electrolytically with a heavy deposit of platinum black (platinized). The fine granular Pt black deposit causes a large increase in the electrode surface area, leading to an increase in double layer capacitance.

The assembled cell was filled with Fisher Scientific 45 wt % aqueous KOH solution and allowed to stand overnight, refilled with fresh solution and the equivalent parallel resistance and capacitance, $R_p$ and $C_p$, measured at 25.0 $\pm$ 0.1°C over the frequency range 50 to 5000 Hz. Resistance measurements (accuracy 1%) were made using a General Radio 1650B impedance bridge. An external capacitance balancing decade box adjustable in steps of $10^{-4}$ µF was connected via shielded cables in parallel with the balancing resistance slide wire in the bridge. Current (ac) was supplied by a Hewlett Packard 200 CD Oscillator; frequency was measured with an Anadex CF 500R counter.

A similar set of measurements of $R_p$ and $C_p$ were made at 25°C with the conductivity cell with platinized electrodes filled with 0.01 M KCl aqueous solution in order to obtain the calibration constant for the cell. To obtain the resistance of the leads to the cell, $R_L$, the cell was filled with mercury to short circuit the two electrodes and the resistance measured; this gave a value of $R_L = 0.173 \ \Omega$.

A capillary conductivity cell with platinized platinum electrodes whose cell constant (22.3 cm$^{-1}$) had been determined previously by calibration with 0.01 M KCl solution was used to measure the conductivity of the 45% KOH solution. The equivalent parallel resistance of this cell filled with the KOH solution was 47.9 $\Omega$ at 25.0°C and 1 kHz.
RESULTS AND DISCUSSION

The resistance, \( R_p \), of the cell filled with 0.01 M KCl solution was 27.5 ± 0.1 \( \Omega \) and independent of frequency over the entire frequency range. This indicates (cf. Eq. (2)) that contributions to \( R_p \) from frequency dependent electrode impedance terms such as \( (1/\omega C_d) \) are negligible compared to the contribution from the \( (R_L + R_{sol}) \) term, so that \( (R_L + R_{sol}) \) may be taken as the measured \( R_p \) value of 27.5 \( \Omega \). Subtracting the measured value of \( R_L \) from this gives \( R_{sol} = 27.3 \Omega \). The cell calibration constant, \( (L/A) \), may then be calculated from

\[
K_{sol} = \frac{(L/A)}{R_{sol}}
\]

where \( K_{sol} \) is the conductivity of the electrolyte solution. Using \( K_{sol} = 0.0014127 \mho^{-1} \text{cm}^{-1} \) for 0.01 M KCl solution at 25°C, the cell constant \( (L/A) \) is 0.0386 cm\(^{-1}\). This is in good agreement with the less precise \( (L/A) \) value of 0.039 cm\(^{-1}\) calculated from the electrode area \( (A = (2.54 \text{cm})^2) \) and electrode separation \( (L = 0.25 \text{cm}) \).

Similarly the resistance \( R_p \) of the 45% KOH solution in the capillary conductivity cell is sufficiently large that it may be used directly to calculate the conductivity of this solution. This gives 0.465 \( \mho^{-1} \text{cm}^{-1} \) for \( K_{sol} \) of the 45% KOH solution at 25°C.

In Figs. 4 and 5 the bridge measurements of \( R_p \) and \( C_p \) respectively are plotted versus frequency for the 45% KOH solution in the conductivity cell with unplatinized and platinized electrodes. Also plotted are the equivalent series resistance \( R_s \) and capacitance \( C_s \) calculated from the complex admittance

\[
Y = \frac{1}{R_p} + i\omega C_p
\]

via the equations

\[
R_s = \text{Re}(1/Y) = \frac{R_p}{1 + (\omega R_p C_p)^2}
\]

\[ C_s = -\frac{1}{\omega \text{Im}(1/Y)} = C_p \left[ 1 + \frac{1}{(\omega R_p C_p)^2} \right] \]  

(9)

where \( \text{Re} \) and \( \text{Im} \) designate the real and imaginary parts respectively.

If the circuit of Fig. 2a were an accurate representation of the cell equivalent circuit, \( R_s \) and \( C_s \) would be frequency independent and equal respectively to \((R_L + R_{\text{sol}}) \) and \( C_{\text{dl}} \). \( R_s \) and \( C_s \) are not frequency independent, but are less so than \( R_p \) and \( C_p \), so that the equivalent circuit of Fig. 2a is clearly a better approximation to the cell circuit than a parallel \( R_p \), \( C_p \) combination.

A more realistic representation \(^2\), \(^4\), \(^5\) of the equivalent cell circuit is shown in Fig. 6. \( R_{W}(\omega) + C_{W}(\omega) \) are the resistive and capacitive components of the so-called Warburg impedance, which arises from concentration polarization due to electrode reactions, and \( R_F \) is the so-called Faradaic impedance associated with the electrode reaction itself. In Fig. 6 we have neglected capacitance between the conductivity cell leads and capacitance due to the dielectric constant of the electrolyte, since these contribute a negligible amount to the audio frequency impedance of the low impedance cell under consideration.\(^2\)

Equivalent circuit analysis of Fig. 6 (Appendix 1) gives for the equivalent series resistance:

\[
R_s = R_L + R_{\text{sol}} + \frac{\left[ R_F + R_{W}(\omega) \right]^2 + \left[ \frac{1}{\omega C_{W}(\omega)} \right]^2 \left[ R_F + R_{W}(\omega) \right]}{\left[ R_F + R_{W}(\omega) \right]^2 + \left\{ \omega C_{\text{dl}} \left[ \left( R_F + R_{W}(\omega) \right)^2 + \left( \frac{1}{\omega C_{W}(\omega)} \right)^2 \right] + \left( \frac{1}{\omega C_{W}(\omega)} \right)^2 \right\}^2}.
\]

(10)

Both \( R_{W}(\omega) \) and \( \left( \frac{1}{\omega C_{W}(\omega)} \right) \) are frequency dependent with an \( \omega^{-1/2} \) dependence,\(^4\) so that the high frequency limit of \( R_s \) is

\[
\lim_{\omega \to \infty} R_s = R_L + R_{\text{sol}}
\]

(11)

Equivalent circuit analysis of Fig. 6 shows that \( R_p \) approaches an identical high frequency limit, but at a slower rate (compare Eq. (2)).

The data of Figs. 4 and 5 exhibit this predicted behavior, that is
(a) both $R_S$ and $R_p$ converge to the same limiting values at high
frequencies, $R_S$ more quickly than $R_p$;
(b) the resistance values for both platinized and unplatinized
electrodes converge to the same high frequency limit.

The electrode impedances for the platinized electrodes are such that $R_S$ and $R_p$
converge at lower frequencies than for the unplatinized electrodes, so that we
may take the limiting high frequency value of $R_S = 0.261 \Omega$ for the platinized
electrodes and identify it with $(R_L + R_{sol})$. This agrees within experimental
error with the value of $(R_L + R_{sol}) = 0.256 \Omega$ calculated from the independently
measured values of $R_L$, $(L/A)$, and $K_{sol}$ for the 45% of KOH solution:

\[
(R_L + R_{sol}) = R_L + (L/A)/K_{sol}
\]  

CONCLUSIONS

We have shown here that one may obtain correct measurements of $(R_L + R_{sol})$
and hence also obtain correct measurements of $(R_L + R_{sol} + R_{sep})$ and therefore
of $R_{sep}$ by audio frequency ac bridge measurements on low impedance conductivity
cells if some precautions are observed. In particular, since even with
platinized electrodes (cf. Fig. 5) $R_p$ and $R_S$ reach their limiting high frequency
values only at the higher frequency end of the audio range, it is plainly not
sufficient to make a measurement of $R_p$ at a single frequency and assume it equal
to $(R_L + R_{sol})$ or $(R_L + R_{sol} + R_{sep})$. Rather it is recommended that for low
impedance cells $R_p$ and $C_p$ measurements be made as a function of frequency, that
$R_S$ be calculated from $R_p$ and $C_p$ via Eq. 8, and that the limiting high frequency
value of $R_S$ be identified with $(R_L + R_{sol})$ or $(R_L + R_{sol} + R_{sep})$. Heavily
platinized electrodes should be used, since these lead to convergence of $R_S$ to
its limiting value within the observable frequency range (Fig. 5), while unplati-
nized electrodes may require extrapolation of $R_S$ beyond the audio range in
order to obtain the high frequency limit.
FIG. 1  A. C. WHEATSTONE BRIDGE FOR MEASURING EQUIVALENT PARALLEL RESISTANCE $R_p$ AND CAPACITANCE $C_p$ OF CONDUCTIVITY CELL.

FIG. 2 APPROXIMATE EQUIVALENT CIRCUITS OF CONDUCTIVITY CELL CONTAINING (a) ELECTROLYTE SOLUTION AND (b) ELECTROLYTE SOLUTION PLUS SEPARATOR.
FIG. 3 CONDUCTIVITY CELL FOR MEASUREMENT OF MEMBRANE RESISTANCES.
FIG. 4 EQUIVALENT PARALLEL AND SERIES RESISTANCES AND CAPACITANCES VERSUS FREQUENCY OF A CONDUCTIVITY CELL FILLED WITH 45% KOH SOLUTION AT 25.0°C USING UNPLATINIZED Pt ELECTRODES. INSERT IS AN ENLARGED PLOT OF THE DATA AT HIGH FREQUENCY.
FIG. 5 EQUIVALENT PARALLEL AND SERIES RESISTANCES AND CAPACITANCES VERSUS FREQUENCY OF CONDUCTIVITY CELL FILLED WITH 45% KOH SOLUTION AT 25.0°C USING PLATINIZED Pt ELECTRODES.
FIG. 6 EQUIVALENT CIRCUIT OF CONDUCTIVITY CELL CONTAINING ELECTROLYTE.
APPENDIX 1

Equivalent Circuit Analysis of Fig. 6.

\[ Z = R_L + R_{sol} + Z_{el} = R_L + R_{sol} + 1/Y_{el} \]
\[ = R_L + R_{sol} + 1/(Y_1 + Y_2) \]  \hspace{1cm} (I)

where \( Y_1 = i\omega C_{dl} \) \hspace{1cm} (II)

refers to the lower branch of the electrode circuit, and

\[ Y_2 = 1/Z_2 = \frac{1}{R_F + R_W + 1/i\omega C_W} \] \hspace{1cm} (III)

refers to the upper branch of the electrode circuit.

Substituting equations (II) and (III) into equation (I) and taking the real part gives Eq. (10):

\[ R_s = \text{Re} (Z) \]
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