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Measurement of electrical resistivity of hydrofluorocarbons – A review

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ABSTRACT

The expansion of applications involving hydrofluorocarbons has generated a demand for devices capable of measuring the electrical volume resistivity of such liquids across diverse operating conditions. The narrow operating range of commercial offerings, particularly in regard to pressure, compels researchers to develop custom cells for the desired test conditions. A review of resistivity cell designs developed over the past three decades is presented. Academic studies in the past have focussed on the development of cells for the purpose of testing the resistivity of refrigerants in liquid phase under high pressures. The fundamental principles underlying resistivity measurement are discussed while emphasis is placed on practical aspects of cell design. The review addresses facets including contemporary standards, limitations and constructional details of academic and commercial cells. It should serve as a guide for future researchers attempting to develop custom resistivity cells for dielectric liquids.

1. Introduction

The electrical volume resistivity of dielectric liquids is an important parameter that offers information on the insulating ability, purity and behaviour of a liquid in the presence of an electric field. Resolving the resistivity of dielectric liquids has therefore become an important requisite for multiple industrial applications. Dielectric liquids serve to suppress arcing and corona discharge as well as functioning as coolants and electrical insulators. Determining the resistivity permits one to identify and test the performance of such liquids when used in high voltage applications including transformers, capacitors, cables and high voltage switchgear [1]. Likewise, the widespread use of hermetic and semi-hermetic compressors in refrigeration has created a demand to verify the dielectric properties of the refrigerants used in these systems and to test potential alternatives where high resistivities are required [2]. More recently, identifying the resistivity of dielectric liquids has piqued the interest of the pharmaceutical industry where dielectric hydrofluorocarbons (HFCs) are widely used as propellants in metered dose inhalers. The resistivity of these propellants impacts the charge characteristics of the active pharmaceutical ingredients delivered to patients thereby affecting drug deposition in the lungs. Determining the resistivity of these HFCs is therefore critical to understand their suitability and effectiveness as inhaler propellants [3]. Besides, these applications constitute only a handful of examples where dielectric liquids are currently used. As such, there is a demand for performing resistivity tests across a wide range of operating conditions. Unfortunately, commercial resistivity cells, as of present, only accommodate the needs of the power industry due to market forces where they're designed to test the specific chemical profiles of the liquids used in this industry. These include a small number of oils comprising mineral, ester and silicone. This forces academics to develop their own cells when investigating the resistivity of alternative liquids, especially when said liquids require operating pressures outside that of atmospheric conditions. Herein a comprehensive review of studies and factors concerning resistivity cell design is presented.

2. Theory of electrical bulk resistivity

The volume resistivity of a dielectric liquid is the quotient of the applied electrical field strength and the current density [4]. It's an intrinsic property that measures a liquid's ability to resist the flow of electric current and depends on the test conditions when performing the measurement. A material with a high resistivity denotes the lack of free electrons, ions and ion forming particles as well as indicating the presence of a low concentration of electrically conductive contaminants. It can be used to analyse the deviation of a material from the desired dielectric characteristics [4]. It's represented by the symbol ρ with an SI unit of Ωm. Resistivity is equal to the reciprocal of the electrical conductivity σ, with the latter having an SI system unit of Siemens per metre

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Nomenclature

ρ	Electrical resistivity (Ωm)
σ	Electrical conductivity (S/m)
k	Ionic mobility (m s^{-1}(V m^{-1})$^{-1}$)
q	Volume charge density (C m^{-3})
I	Current (A)
V	Voltage (V)
R	Resistance (Ω)
J	Current density (A/m^2)
E	Electric field strength (V/m)
L	Distance between electrodes (m)
A	Cross-sectional area of material under test (m^2)
U	Velocity of ions (m/s)
D	Molecular diffusion coefficient (m^2/s)
n	Number density (m^3)
e	Elementary charge (C)
k_B	Boltzmann constant (J/K)
T	Absolute temperature (K)
a	Stokes radius of an ion (m)
η	Dynamic viscosity (Pa s)
v	Concentration (m^{-3})
K_R	Recombination constant
K_D	Dissociation constant
ε	Permittivity of the material (F/m)
λ	Debye length (m)
u	Fluid velocity (m/s)
t	Time (s)
ρ_f	Fluid mass density (kg/m^3)
p	Pressure (Pa)
g	Acceleration due to gravity (m/s^2)
σm	Moleon conductivity (S/m)
r	Particle radius (m)
δ	Dielectric loss angle
Tan δ	Dissipation factor
ω	Angular frequency (rad/s)
A_T	Constant related to the nature of ions in a liquid
E_{ac}	Activation energy (eV)
n_0	Unperturbed concentration of particles
$i_i(t)$	Capacitor charging current component (A)
$i_p(t)$	Polarisation current component (A)
$i_{ss}(t)$	Space charge current component (A)
$i_c(t)$	Conduction current component (A)
t_a	Time duration of DC voltage step (s)
t_r	Time duration of resorption current (s)
$i_d(t)$	Discharging current component (A)
$i_{dp}(t)$	Depolarisation current component (A)
$i'_{ss}(t)$	Space charge current component at the discharge state (A)
r_1	Radius of inner electrode of a concentric cell (m)
r_2	Inside radius of the outer electrode of a concentric cell (m)

(S/m). The volume resistivity may be expressed as:

$$\rho = \sum_i \frac{1}{k_i q_i} \tag{1}$$

where the medium possesses charge carriers of species i with mobility k_i and volume charge density of q_i. The resistivity of a dielectric liquid is considered to be representative only when the liquid is at thermodynamic equilibrium where the measuring conditions don't significantly affect the mobility and density of the charge carriers [5]. When this condition is satisfied, the dielectric acts as an RC parallel circuit where the conduction current is equal to I = V/R under an applied voltage V. The current density J can be expressed as:

$$J = \frac{E}{\rho} \tag{2}$$

when ohm's law is obeyed by applying a low enough voltage so that the electric field E doesn't disrupt the thermodynamic equilibrium. Thus, by measuring the current flowing through a cell one can derive the value for the resistivity of the liquid. In a case where the cross section is uniform and the electric field and current density is constant, the resistivity for a material between parallel plates can be calculated from the resistance using the equation:

$$R = \rho \frac{L}{A} \tag{3}$$

which rearranges to:

$$\rho = \frac{V}{I}\left(\frac{A}{L}\right) \tag{4}$$

where.

R = Resistance L = Distance between electrodes V = Voltage.

ρ = Resistivity A = Cross-sectional area of material under test I = Current.

The term $\left(\frac{L}{A}\right)$ is referred to as the cell constant with SI unit m^{-1}. It's a geometrical factor used to convert the measured resistance to a resistivity value. Although, the true value of the cell constant can only be determined experimentally via calibration with liquids of known resistivities. This is because (for liquid dielectrics) in the absence of fouling, the geometrical surface area of the electrodes is always lower than that of the electrochemical area, where electron transfer takes place [6].

3. Nature of charge carriers and mobility

It is believed that in dielectric liquids including those of high purity, low concentrations of impurities are present in the range of parts per billion [7]. The charge carriers in these liquids, at relatively low electric field strengths, are ions that result from the spontaneous dissociation of these impurities as opposed to electrons due to the life time of electrons being very short (lower than 10^{-4} s) [5]. The velocity of ions U in a liquid at rest with a constant temperature can be expressed by:

$$U = kE - D\frac{\nabla n}{n} \tag{5}$$

with ionic mobility k, molecular diffusion coefficient D and ion number density n. The first term represents the contribution due to ionic drift while the second accounts for molecular diffusion. Molecular diffusion may be neglected at ambient temperature when the applied electric field is above the order of 0.025 V/m [8]. The diffusion coefficient equates to:

$$D = \frac{k \cdot k_B T}{e} \tag{6}$$

where e is the elementary charge, k_B is the Boltzmann constant and T is the absolute temperature. Ions draw neutral molecules toward themselves according to solvation and are more extensively solvated with smaller ionic radii. Ionic mobility is proportional to the net charge of the particle and inversely proportional to the Stokes radius a of the ion (effective radius of solvated ion). When a solvated ion moves with a constant drift velocity after being accelerated by an electric field, it is subject to equal and opposite forces of dynamic viscosity η and coulomb force [9]. The ionic mobility can therefore be given by:

$$k = \frac{e}{6\pi\eta a} \tag{7}$$

The simplest model of conductivity at low electric field strengths is based on the assumption that impurities are in the form of ion pairs AB with concentration ν dissolved in a dielectric liquid which dissociate into ions A^+ and B^-. The global equilibrium reaction between the monovalent ions A^+ and B^- and the neutral AB molecules may then be expressed by:

$$AB \underset{K_R}{\overset{K_D \leftrightarrow}{\qquad}} A^+ + B^- \tag{8}$$

K_R in Equation (8) represents the recombination constant and K_D the dissociation constant. The number of ions generated is equal to those that recombine at thermodynamic equilibrium causing K_{D^ν} to equal $K_{R^{n_+ n_-}}$ with $n\pm$ denoting the ionic densities [9]. Thus, $n_+ = n_- = \sqrt{K_{D^\nu}/K_R}$ and conductivity may be expressed as:

$$\sigma = (k_+ + k_-)e\sqrt{\frac{K_{D^\nu}}{K_R}} \tag{9}$$

K_D increases when the applied electric field strength increases while K_R is independent of the electric field and can be approximated with relative confidence by Langevin's expression for ionised gases:

$$K_R = (k_+ + k_-)\frac{e}{\varepsilon} \tag{10}$$

where ε is the permittivity. The steady-state current-voltage characteristics for a given liquid resulting from field enhanced disassociation can then be drawn by making a number of assumptions. These include the lack of injection, diffusion and fluid motion (including electrohydrodynamic convection), instantaneous discharge of ions at the electrodes and an ohmic regime being observed where the field is uniform across the interelectrode gap [9]. The initial current density is then given by:

$$J_0 = \sigma E = (k_+ + k_-)e\sqrt{\frac{K_{D^\nu}}{K_R}} \cdot \left(\frac{V}{L}\right)` \tag{11}$$

3.1. *Interfacial phenomena at the electrodes*

It is commonly assumed that an ideal behaviour of ion neutralisation at the electrodes is observed which is generally not the case. They may experience a delayed discharge and develop a unipolar charged layer that is released upon field reversal. Moreover, the immersion of a solid (electrodes) in a liquid causes a spontaneous unipolar layer of ions near the solid to develop where the thickness of the noncharge neutrality region is given by the Debye length λ:

$$\lambda = \sqrt{\frac{(k_+ + k_-)\varepsilon kT}{e\sigma}} \tag{12}$$

which depends on the surface properties of the solid and the liquid. The measured resistivity of a liquid is lowered if the two layers converge as a result of a high λ relative to the electrode gap [9]. At the electrode boundaries, the non-charge neutrality region in the vicinity of the electrodes is known as the electrical double layer. This consists of two parallel layers of charge surrounding the electrodes. It includes the first layer, the surface charge, where ions adsorb onto the electrodes as a result of electrostatic interactions and a second diffuse layer of ions experiencing a coulomb force toward the surface charge. The extent of net charge and penetration depth into the liquid volume of the double layer are determined by ion migration, convection and diffusion. The accumulation of charge at the electrodes causes enhanced local electric fields to be developed at the interface [1]. The local electric field at the electrodes may also be amplified in practise due to the presence of microasperities at the electrode surface where highly polished surfaces such as steel and aluminium contain microasperities per cm^2 of the order 10^8 and an average radius of curvature below 2 μm. This field enhancement is important in studies relating to electrical breakdown as breakdown initiates where the field is largest [8].

3.2. *Electrohydrodynamic convection*

Electrohydrodynamic (EHD) effects on the resistivity may also need to be considered. EHD convection can develop in the dielectric due to coulomb forces on the net space charge and is usually observed in dielectric liquids with high concentrations of impurities ($\rho \leq 10^{14}$ Ωm) [1,8]. EHD may be generated due to charge injection, charge depletion and motion of unipolar charged layers which are explained in more detail in Section 6.3. It causes turbulent fluid flow to occur leading to convection currents in addition to conduction currents. Diffusive currents can be considered negligible, therefore in an ohmic regime; the current density may be expressed by:

$$J = \sigma E + qu \tag{13}$$

with charge density q and fluid velocity u. The first term in equation (13) represents conduction current while the second denotes convection current. The governing EHD equations for an ohmic model of resistivity with incompressible, viscous and homogenous dielectric liquids are the following:

$$\nabla \cdot \boldsymbol{u} = 0 \tag{14}$$

$$\rho_f\left(\frac{\partial \boldsymbol{u}}{\partial t} + \boldsymbol{u}\mathrm{n}\nabla\boldsymbol{u}\right) = -\nabla \mathrm{p} + \eta\nabla^2\boldsymbol{u} + q\boldsymbol{E} - \left(\frac{E^2}{2}\right)\nabla\varepsilon + \rho_f\boldsymbol{g} \tag{15}$$

$$\nabla \mathrm{n}(\varepsilon\mathbf{E}) = \mathrm{q}, \;\; \nabla \times \boldsymbol{E} = 0 \tag{16}$$

$$\frac{\partial q}{\partial t} + \nabla\cdot(\sigma\boldsymbol{E} + q\boldsymbol{u}) = 0 \tag{17}$$

where t is the time, ρ_f is the fluid mass density, p is the pressure and $\boldsymbol{g}$ is the acceleration due to gravity [8]. The contribution of EHD to the conductivity of a dielectric liquid is usually considered negligible below an applied field of the order 10^5 V/m [10]. This means that EHD may affect the results attained when applying electric field strengths recommended by international standards, since recommended field strength are of the order 10^5–10^6 V/m (see Table 1), depending on the liquid properties and electrode material. Contemporary standards relating to liquid resistivity measurement have not yet addressed this issue.

3.3. *Conduction due to small particles*

In dielectric liquids, charge transport may also emanate from small particles known as moleons leading to a contribution to the current. The main source of moleons in resistivity tests is attributed to the introduction of fresh electrodes into a cell where their removal is considered difficult even with vigorous flushing. The equation for moleon conductivity is given by:

$$\sigma_m = 2\pi^5\varepsilon^2 r^3 n_0 / 27\eta \tag{18}$$

where r is the particle radius and n_0 the unperturbed concentration of particles. This relation may not always correlate with experimental data, particularly for cases with EHD characteristics [8].

4. AC and DC resistivity

The current measured in a resistivity cell, and for that case any plane

Table 1
Standards relating to the measurement of dielectric liquid resistivity [15].

Standard	IEC 61620:1998 [16]	IEC 60247:2004 [4]	ASTM D1169-19 [17]
Title	Insulating liquids – Determination of the dielectric dissipation factor by measurement of the conductance and capacitance – Test method	Insulating liquids – Measurement of relative permittivity, dielectric dissipation factor (tan δ) and d.c. resistivity	Standard Test Method for Specific Resistance (Resistivity) of Electrical Insulating Liquids
Quantities	Dielectric dissipation factor (resistivity measurements simply a means of dissipation factor determination)	Relative permittivity, dielectric dissipation factor (tan δ), DC resistivity	DC resistivity
Definition	Resistivity is related to an initial current density during a short period of time.	DC resistivity is related to "steady-state current density".	DC resistivity is related to a current density "at a given instant of time".
Method	Current measurement, trapezoidal voltage	Current measurement, DC voltage	Current measurement, DC voltage
Field strength	≤100 kV/m	≤250 kV/m	0.2 to 1.2 MV/m
Electrification time	0.4–5 s (trapezoidal square wave, f = 0.1–1 Hz, rise time 1–100 ms)	1 min	1 min direct polarity/5 min short circuit/1 min reversed polarity

capacitor, when applying a DC voltage step of duration t_a can be expressed as a time variable component $i_1(t)$ equal to the sum of four disparate conduction processes:

$$i_1(t) = i_i(t) + i_p(t) + i_{ss}(t) + i_c(t) \tag{19}$$

where $i_i(t)$ is the capacitor charging current component, $i_p(t)$ is the polarisation current, $i_{ss}(t)$ is the space charge current component and $i_c(t)$ is the conduction current component (see Fig. 1).

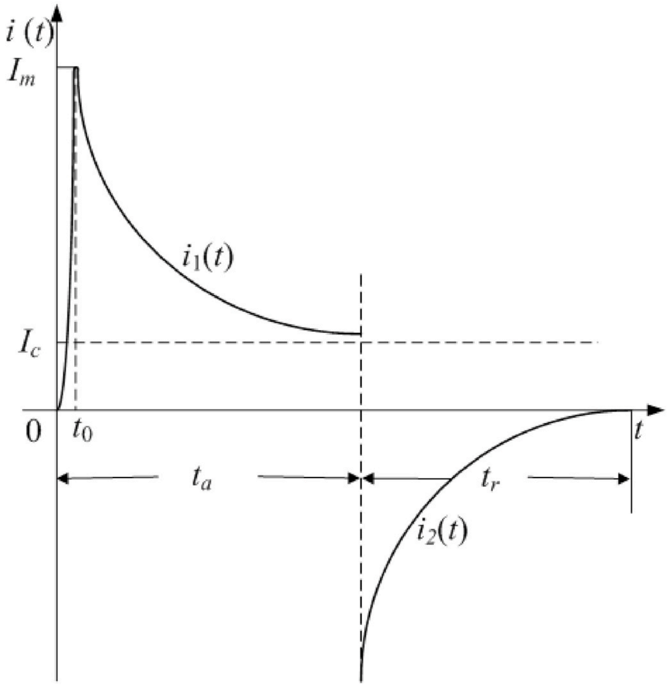

Fig. 1. General form of time variation of current through a capacitor with (t_a) and without (t_r) an applied voltage [11].

The charging current $i_i(t) = \varepsilon A \partial E/\partial t$ in a capacitor under DC is equivalent to the displacement current and is reduced to zero when the potential difference across the capacitor reaches the source voltage, immediately after t_0. The current measured prior to t_0 is therefore usually disregarded [11].

The polarisation component represents the current caused by transient charge-carrier drift processes due to ions being attracted to or repelled from electrodes based on their polarity.

Space charge is a concept where charge is treated as a continuum of charges distributed within a volume inside the dielectric as opposed to point like charges. The space charge current component for a capacitor may be a result of charge injection from the electrodes, electrical double layers or dielectric ionisation depending on the electric field strength [12]. This current reduces to zero after a limited time as the charge carriers are neutralised at the electrodes or become trapped in a localised state within the dielectric [11].

The conduction current is a voltage independent component that is characterised by the inherent resistivity of the dielectric medium. It is dependent upon the chemical nature of the dielectric - the type and number of charged species, the chemical reactions they undergo and their velocity [8,11].

The resistivity of the dielectric at any given time t can be found by subtracting the resorption current (current measured after switching off the voltage) from the absorption current (current measured after voltage is applied) when applying a DC voltage. The resorption current is a transient component described by the sum of three other components:

$$i_2(t) = i_d(t) + i_{dp}(t) + i'_{ss}(t) \tag{20}$$

where $i_d(t)$ is the discharging current component, $i_{dp}(t)$ is the depolarisation current component and $i'_{ss}(t)$ is the space charge current component at the discharge state. The polarisation and space charge components of the DC resistivity are time varying and reduce to zero after a long enough measurement time leaving the steady-state component corresponding to the conduction current. On the other hand, the AC resistivity is only composed of two components, a steady state conduction component that is independent of frequency and a frequency dependent component [11,13].

AC voltage sources are often used to determine the conductivity of liquids with high conductivities. This is because the use of AC limits the presence of "polarisation" effects due to ion drift by causing the ions to oscillate with the applied frequency about their positions, preventing them from accumulating at the electrodes. Lower frequencies are typically used with higher resistivities as polarisation effects are less significant [6]. However, the use of AC, when measuring the resistivity of dielectric samples, is limited by the presence of large capacitive currents relative to the resistive current, preventing the latter from being measured accurately [14].

IEC 60247 and ASTM D1169 standards advise readers to measure the resistivity at the 'steady state' as opposed to the initial 'true resistivity'. The true resistivity of the fluid can only be found immediately after applying the voltage with low voltages, which can be accomplished with an AC voltage source as stipulated by IEC 61620. Polarisation processes (induced electric dipole moments) take place as soon as the voltage is applied and within seconds, an initial resistivity can be measured according to IEC 61620 as conduction currents become dominant. Conversely, IEC 60247 and ASTM D1169 propose the use of relatively high voltages for a protracted time [4]. The steady state in materials is typically arrived at within 60 s where the resistivity is determined according to IEC 60247 and ASTM D1169 (see Table 1), while it may take longer for materials with high resistivities [15]. Thus, the type of voltage source is an important consideration when deciding on the type of resistivity intending to be measured.

5. Standards for resistivity measurement of dielectric liquids

There exists different international standards for measuring the resistivity of insulating liquids including IEC 61620, IEC 60247 and ASTM D 1169. The fundamental differences between these standards relating to resistivity measurement are summarised in Table 1.

IEC 61620 is intended to offer guidance on the determination of the dielectric dissipation factor and was not designed to standardise resistivity measurement. However, an approach for measuring the initial resistivity is outlined as a means of measuring the dissipation factor based on equation (16):

$$Tan\ \delta = \frac{1}{\varepsilon\omega\rho} \tag{21}$$

where δ is the dielectric loss angle whose tangent is the dissipation factor and ω is the angular frequency [16]. The resistivity value is sampled at the flat current level of every half period using a trapezoidal square wave voltage (frequency = 0.1 Hz–1 Hz, rise time = 1 ms–100 ms). The use of low electric field strengths, below 100 kV/m, is proposed in the standard and an electrification time between 0.4 and 5 s. The resistivity is defined as being related to an initial current density within a short time of energisation. Nonetheless, the steady-state resistivity is different from the initial resistivity measured using the method proposed in this standard and it's impossible to establish a relationship between these two quantities [15].

IEC 60247 relates DC resistivity to a steady-state current density and the outlined method for measuring the resistivity involves measuring the current through the insulating liquid after applying a DC voltage. The maximum field strength proposed in this standard is 250 kV/m with a suggested conventional arbitrary time of electrification of 60 s [4].

ASTM D 1169 [17] relates DC resistivity to a current density "at a given instant of time". The standard proposes for current to be measured after a DC voltage is applied at a field strength between 200 kV/m and 1.2 MV/m with an electrification time of 60 s. It also suggests to average two measurements with both polarities to compensate for effects relating to polarity, however, research has shown for this to not be enough of a conditioning where a number of subsequent polarity reversals is required [15].

6. Factors that influence the resistivity of dielectric liquids

6.1. Moisture

The conductivity of pure water is 6×10^{-6} S/m at 25 °C. It dissociates to H+ and OH- which gives rise to the stated conductivity value. It contributes free charge carriers to the sample therefore serving to reduce the measured bulk electrical resistivity [18]. This is exhibited in Fig. 2 where the DC conductivity of insulating liquids (hydrocarbons) increases with the amount of moisture. A water content of 20 ppm in new mineral oil can lead to an order of magnitude increase in conductivity [19,20].

The presence of water is an important factor that affects the electrical strength of insulating liquids where even "0.01% water in transformer oil reduces its electrical strength to 20% of the dry oil value" [21]. The severity of the impact of moisture on the dielectric properties of a medium depends on the solubility of water in the liquid and the relative saturation. Dissolved water has a lower impact on the resistivity than emulsified or free water. The solubility of water in a liquid is defined as the total amount of water capable of being dissolved at a particular temperature. The dependence of solubility on the temperature follows an exponential relationship [22].

6.2. Temperature

Resistivity has an inverse dependence on the temperature that is generally exponential [4]. This makes it important to ensure that measurements are made under precise temperature conditions. The dependence of resistivity on the temperature is largely due to the temperature dependence of the liquid viscosity. Increasing the temperature increases the motion of the charge carriers and lowers the viscosity of the liquid causing an increase in the mobility of the carriers. It also increases the number of charge carriers due to the dissociation coefficient increasing. This leads to a higher conductivity in the sample. The product of the mobility of ions in highly insulating liquids and viscosity is approximately constant as the temperature is varied according to an empirical relation known as Walden's rule:

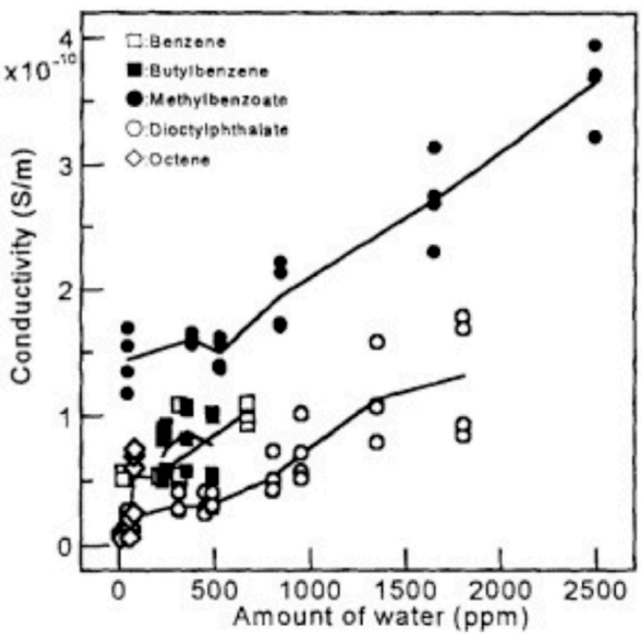

Fig. 2. Graph displaying the effect of water content on the conductivity of hydrocarbon liquids [20].

$$k\cdot\eta \sim 10^{-11}\ \left(m^2 / Vs\right) \tag{22}$$

It denotes the fact that mobility depends on the temperature to the same extent as the viscosity. The viscosity is therefore inversely proportional to the mobility which allows an expression to be derived relating the temperature to the conductivity for most insulating liquids:

$$\sigma(T) \approx A_T e^{(-E_{ac}/k_B T)} \tag{23}$$

where T is the absolute temperature in Kelvin, A_T is a constant related to the nature of the ions in the liquid and E_{ac} (eV) is the activation energy [19].

6.3. Electric field

The conductivity of insulating liquids when measured with increasing electric field leads to an established trend that may be visualised graphically in Fig. 3. The graph can be separated into three regions (numbered in the figure as 1, 2 and 3) including an initial ohmic region with low field strength where the current density is proportional to the electric field. This is followed by a region with an intermediate field strength where current density appears to be saturated. Finally, a region with large field strengths is observed where the current density increases rapidly with electric field before resulting in electrical breakdown.

The field strength dependence of the resistivity can be explained by ion drift and generation processes. Ions can be generated in the bulk via disassociation and at the electrodes via injection. In the initial low field

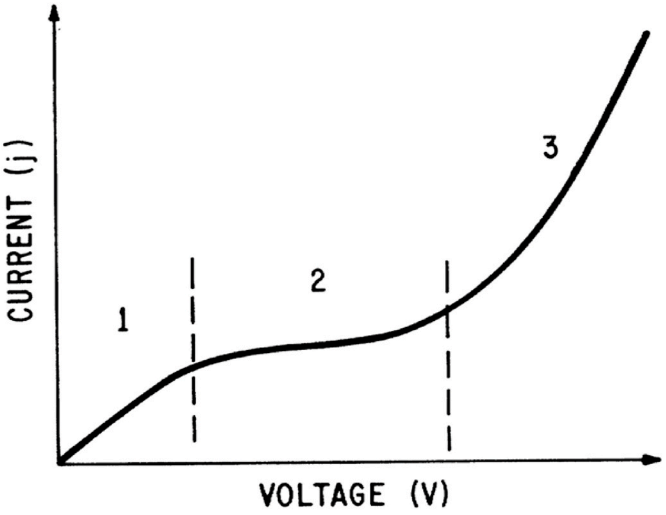

Fig. 3. Shape of the current-voltage graph for a dielectric liquid [23].

region, ions migrate to the counter electrode due to the force from the electric field where they accumulate or are partly neutralised. Free ions are generated by the electric field and can be replaced by newly generated ions in the dielectric leading to a constant resistivity value. At higher field strengths, the ion drift velocity increases. The intermediate field region involves free ion density decreasing as they are displaced at a faster rate than they are generated. Thus, the current density enters a saturated state where the dissociation and recombination rate stay constant and the resistivity increases with increasing field strength. During the high-field region, the ionic disassociating rate increases to an extent where the rate becomes independent of the applied voltage leading to more charge carriers and decreasing resistivity with field strength [15,19]. In this region above the saturation voltage, charge is also injected from the electrodes contributing to the increase in charge carriers and conductivity. Charge injection upsets the thermodynamic equilibrium so that the resistivity varies with position and time [1]. One such method of injection is Schottky emission, which involves thermionic emission of electrons from a metal electrode into the conduction band of the liquid, enhanced by reducing the work function with an electric field and correcting for the image force. Another method involves a process called Fowler-Nordheim field emission where electrons migrate to the liquid from the surface of a metal electrode via quantum mechanical tunnelling under high electric fields (above $\sim 10^8$ V/m) [8]. Injection is dependent upon the impurities present in the tested medium, the nature of the liquid (including polarity) and the electrode material [9]. It usually becomes dominant only at applied electric fields above 100 kV/m [24]. The current density in the steady state will also become space charge limited if the injected charge density is high [9].

7. Resistivity cell types and requirements

High range resistivity cells may be categorised into two basic groups according to their geometric designs including a parallel plate arrangement and a concentric cylinder arrangement. A cylinder cell is considered more favourable in most cases and has been adopted by commercial entities as a result. Some academic studies, however, have utilised the parallel arrangement citing the desire to investigate the effect of a variable inter-electrode distance or due to the simplicity of the design. The main appeal behind the cylinder arrangement is the lower cell constant offered when measuring a similar volume of fluid to a parallel cell. This is a result of the higher surface area of the electrodes that are exposed to the liquid in a cylindrical cell. A lower cell constant allows one to increase the measuring range of the cell, reduce the volume of liquid required for measurement and reduce the size of the cell. The equations for the cell constant in each case offer a mathematical rationale for the existence of lower cell constants inherent in cylinder cells where:

$$\frac{\ln\left(\frac{r_2}{r_1}\right)}{2\pi L} \tag{24}$$

is the equation for the cylinder case and

$$\frac{L}{A} \tag{25}$$

for a parallel cell where L is the distance between the electrodes, A the cross-sectional area of the measuring electrode (in parallel cells) while r_2 is the inside radius of the outer electrode and r_1 is the radius of the inner electrode (in a cylinder cell). The natural logarithm in equation (24) possesses a relationship where the range tends to zero exponentially as the ratio $\left(\frac{r_2}{r_1}\right)$ tends to 1. Thus, for a set difference between r_2 and r_1, we may exponentially reduce the size of the cell constant in cylinder cells by linearly increasing the diameters of the concentric cylinders. Increasing the length of the cylinders and area of the measuring electrode in a parallel cell also reduces the cell constant linearly in both cases, as can be seen in Equations (24) and (25). The dimensions result in volume and materials increasing proportional to the length for a cylinder and proportional to the square of the plate dimension. An exponential increase in size is thus required for parallel cells to reduce the cell constant in the same way a linear increase in cylinder length reduces it. This is not ideal in the parallel case due to possible size and volume constraints.

The design of the cell must also meet certain requirements to perform satisfactorily as outlined in the aforementioned international standards. It should allow the component parts to be cleaned easily and thoroughly. It should permit the cell to be used at the required constant temperature and offer a way to measure and control the temperature of the liquid. The materials used in constructing the cell should be capable of withstanding the temperatures and pressures intended to be applied. In addition, the electrodes' surface in contact with the liquid should have a smooth finish to ensure easy cleaning. The measuring electrode should also be guarded to facilitate a high order of measurement accuracy by limiting any erroneous impact of fringe fields [4].

The fringe field effect is when part of the measuring field falls outside the geometric space of the measuring volume and can lead to a mismeasurement of the fluid resistivity as a result. The use of a guard electrode serves to absorb the current flowing through the fringe field, ensuring a homogenous field while confining the current to a defined area and limiting interference with the field lines. An inhomogenous electric field causes the resistivity to be a function of spatial coordinates rather than being represantative of the bulk medium [25]. Therefore, it is important to ensure a homogenous field within the measuring volume to obtain a representative value for the resistivity. This is another reason why it is recommended that the surface of the electrodes be smooth to limit electric field amplification caused by rough surfaces.

Likewise, the insulating materials incorporated in the cell should not absorb or be affected negatively by the test liquids or solvents used for cleaning. The resistivity of these insulating materials must also be high, especially the material used between the measuring electrode and the guard. Examples of insulating materials that are considered satisfactory for such a purpose include borosilicate glass, steatite, boron nitride, TFE-fluorocarbon and quartz [17]. Additionally, the electrode material must be corrosion resistant and introduce no errors under the conditions of the test. Stainless steel is often recommended for this purpose [4].

8. Previous studies – Resistivity cell design

Resistivity cells developed by academics have largely been for the purpose of testing the resistivity of liquid refrigerants. These refrigerants have to be pressurised under large pressures to maintain a liquid phase at room temperatures due to their low boiling points. As a result, the cells they develop mainly focus on delivering a similar performance to that of commercial cells while maintaining a large pressure within the cell. Although, the need for large pressures within the cell places considerable emphasis on ensuring effective evacuation of the system prior to tests to prevent water vapour from condensing into moisture. The measurement process therefore becomes more complicated than that used in commercial cells as nitrogen purging and vacuum pumping are typically employed. Unfortunately, the aforementioned standards do not offer procedural information on the measurement of resistivity for dielectric liquids under large pressures. Thus, the lack of a standardised method for limiting the presence of moisture when conducting such resistivity tests may be a source of variation for results attained by related studies. Table 2 lists a selected number of studies into the resistivity of liquid refrigerants including the type tested, the results attained and details about the cells used in the studies. The following sections summarise the selected studies including those that utilised parallel plate cells initially followed by studies involving concentric cylinder cells.

8.1. *Meurer S. et al. (2001)*

The cell used by Meurer et al. [2] was a parallel plate cell displayed in Fig. 4 with a measurement electrode surrounded by a guard and a 2 mm interelectrode gap. Epoxy was used to separate the measuring and guard electrodes. They tested the AC resistivity of different refrigerants and used a low 50 mV voltage with a frequency of 1 kHz after experiencing the dissociation-field effect at higher voltages with some of the refrigerants. The refrigerants tested in the study include R404A, R407C, R410A, R507 and R134a. The measurements were conducted between temperatures of 23 °C and 26 °C in liquid phase at saturated pressures and the cell was flushed with nitrogen and evacuated prior to each measurement. The total error of each resistivity measurement was assumed to be a maximum of 15% due to the tolerances of the structural parts. The cell possesses a relatively large cell constant of 1.02 m^{-1} compared to Feja S. (2012) with 0.113 m^{-1} which may be a significant factor behind why the resistivity measured for R134a is two orders of magnitude lower than that measured by Feja S. (2012). The large cell constant may be attributed to the use of a parallel plate cell compared to a concentric cylinder cell where the latter is capable of attaining lower cell constants when measuring the resistivity of similar volumes of dielectric liquid.

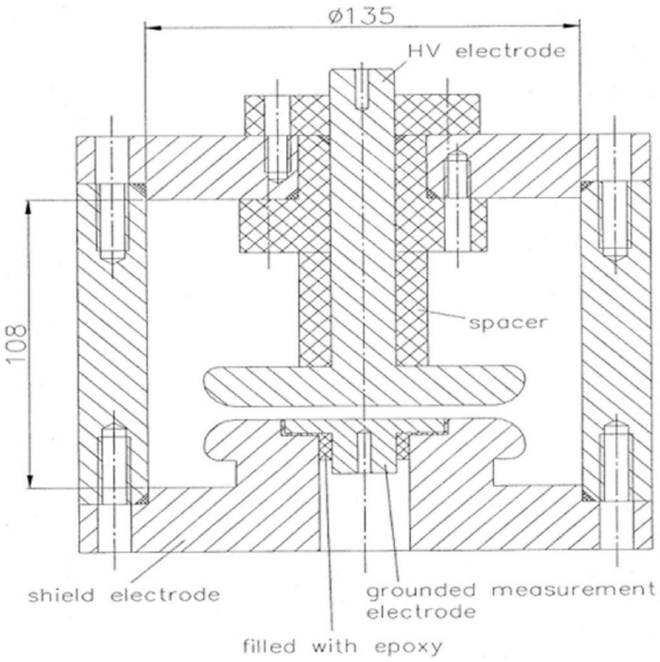

Fig. 4. Parallel plate cell design utilised by Meurer et al. (2001) [2].

8.2. *Dschung F. And Kindersberger J. (2016)*

The test cell used by Dschung and Kindersberger [26] was a parallel plate cell consisting of modified stainless steel flanges mounted to both ends of an aluminium pipe with an inner diameter of 100 mm and gaskets installed between the pipe and flanges. The cell is shown in Fig. 5. Coaxial bushings were used to connect the electrodes from outside the cell where the top electrode was connected to the voltage source. The bushings were able to withstand a pressure of up to 6 MPa and temperatures of up to 100 °C. The frame was made of PEEK (Polyether ether ketone) and the electrodes of stainless steel. The measuring electrode had a diameter of 24.2 mm and was glued to a PEEK support structure with PEEK material separating the measuring and guard electrodes by 1 mm. The distance between the measuring electrode and anode was set at 2 mm. The guard ring was connected to the volume limiter which was in galvanic contact with the grounded lower flange. The volume limiter served to reduce the required volume of liquid.

They investigated the DC resistivity of the samples across a range of temperatures. An average field strength of 250 kV/m was applied for 60 min with the current in the last 10 min being used to calculate the resistivity after a steady state had been fulfilled. They found no clear trend with respect to temperature between the 20 °C and 90 °C they tested. During their investigation they found R134a showing indications of decomposing when stressed with 250 kV/m after several hours and recommended the long-term stability of the HFC to be proven prior to application. The HFCs were also investigated at commercial grade purity with >99.5% for R134a and >99.0% for R227ea. The procedure used by Dschung and Kindersberger involved scavenging air in the cell before inserting the liquid refrigerant and performing a short test with 500 V to

Table 2
Summary of key parameters for selected studies into the resistivities of refrigerants.

Study	Fellows B. et al. (1991)	Bryan J. & Seyed-Yagoobi J. (1996)	Meurer C. et al. (2001)	Feja S. (2012)	Dschung F. & Kindersberger J. (2016)
Type of power source	AC & DC	DC	AC	DC	DC
Design	Concentric cylinder cell	Concentric cylinder cell	Parallel plate cell	Concentric cylinder cell	Parallel plate cell
Refrigerant	(1) R134a, (2) R152a	R404A[a]	R134a	(1) R134a, (2) R152a, (3) R744	(1) R134a, (2) R227ea
Resistivity (liquid phase, saturation pressures, ≈25 °C[b])	(1-DC) 6.6 × 10^8 Ωm, (1-AC) 1.8 × 10^6 Ωm (2-DC) 2.2 × 10^7 Ωm, (2-AC) 5 × 10^5 Ωm	3.1 × 10^7 Ωm	1.089 × 10^6 Ωm	(1) 10^8 Ωm, (2) 10^7 Ωm, (3) >10^{14} Ωm	(1) 6 × 10^6 Ωm, (2) 1.3 × 10^8 Ωm
Cell constant	Not known	0.1901 m^{-1}	1.02 m^{-1}	0.113 m^{-1}	4.35 m^{-1}

[a] R404A was measured at a maximum temperature of 10 °C.

[b] The temperatures used in these studies varied between 20 °C and 26 °C.

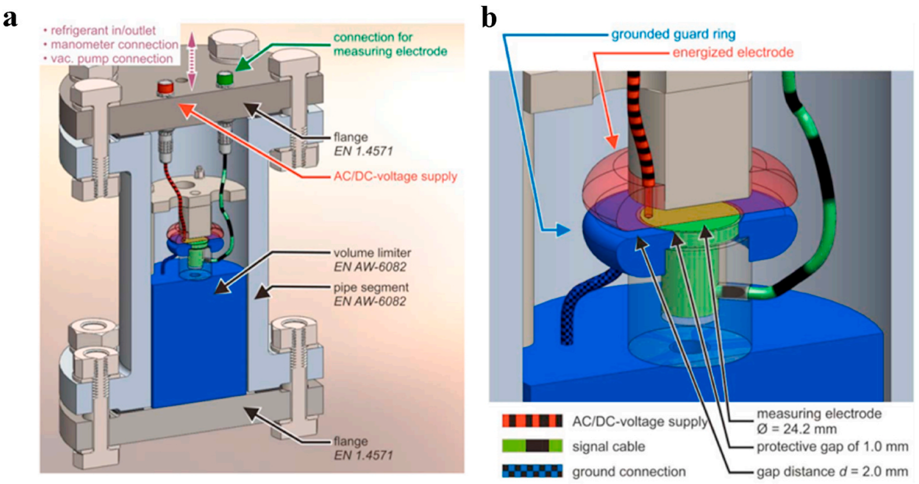

Fig. 5. Illustration of (a) complete parallel cell utilised by Dschung and Kindersberger (2016) to measure the resistivity of HFCs and (b) a close up of the cell geometry [26].

ensure a correct filling level. The measured values allow one to easily discern whether the refrigerant is in its gaseous or liquid state as they differ by orders of magnitude. A manometer was connected to the top flange as shown in Fig. 5 and was used to monitor the pressure in the cell to ensure a correct amount of refrigerant was in liquid phase. The pressure was used to determine the temperature of the liquid using a temperature pressure correlation. 500 V with a positive polarity was applied for 60 min with current recorded every 2 s and the current in the last 10 min was used to calculate the resistivity. The polarity was then reversed to negative 500 V while repeating the same process again. This was then all repeated with different temperature values and repeated four times for each refrigerant. The results for the resistivities of the refrigerants as a function of temperature can be seen in Figs. 6 and 7.

Dschung and Kindersberger [26] use a similar parallel plate design to that of Meurer et al. [2] while exploring the resistivity of the same refrigerant R134A, with a larger cell constant of 4.35 m^{-1} compared to 1.02 m^{-1} in the latter. Dschung and Kindersberger [26] also apply a different voltage source, using DC while Meurer et al. [2] make use of AC and achieve a similar value for the resistivity of R134a with the same order of magnitude of 10^6 Ωm. The effects of this difference in cell constant size and the difference in voltage source applied therefore appears muted in this case. Furthermore, the cell developed by Dschung and Kindersberger [26] uses PEEK material to separate the measuring and guard electrodes by 1 mm compared to Meurer et al. [2] who use a 2 mm interelectrode gap with epoxy as the separating material. The difference in the interelectrode gap in this case is relatively small and should not contribute to any significant difference in the measured electrical resistivity. However, it should be noted that as the interelectrode gap increases, the current measured by the measuring electrode increases proportionally (to an extent depending on the size of the fringe field) due to the fringe field effect leading to a lower resistivity measurement. Both epoxy and PEEK are insulating materials with high enough resistivities to be considered satisfactory.

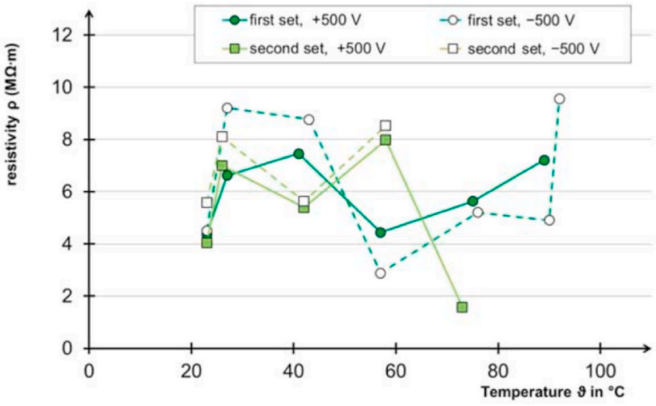

Fig. 6. R134a resistivity results as a function of temperature [26].

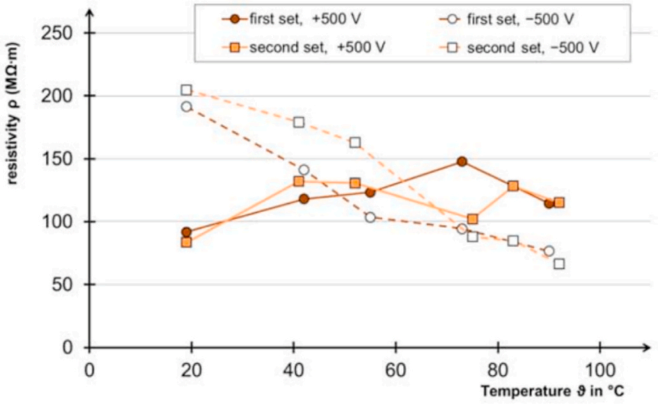

Fig. 7. R227ea resistivity results as a function of temperature [26].

Similar to Meurer et al. [2], the cell by Dschung and Kindersberger [26] measures a resistivity value for R134a which is two orders of magnitude lower than that measured by Feja S. (2012). The factors causing this may similarly be attributed to the use of a parallel plate cell with a higher cell constant.

8.3. Bryan J. And seyed-yagoobi J. (1996)

Bryan and Yagoobi [27] used a cylinder cell shown in Fig. 8 consisting of two cylindrical electrodes enclosed by a housing where they are all electrically insulated from each other and made of 304L stainless steel. The housing serves as a guard electrode and pressure vessel with either nylon 6/6 or Teflon being used as insulators to separate the electrodes and housing depending on the fluid and operating conditions used. Two electrical connections to the cell exist at the base of the device with coaxial cables being used. Three resistive temperature devices, two servicing ports and a pressure port are mounted at the top of the housing. The seals used are fluid compatible O-rings and the cell can be completely disassembled for cleaning. The cell possesses a relatively low cell constant value of 0.1901 m^{-1} owing to the concentric cylinder

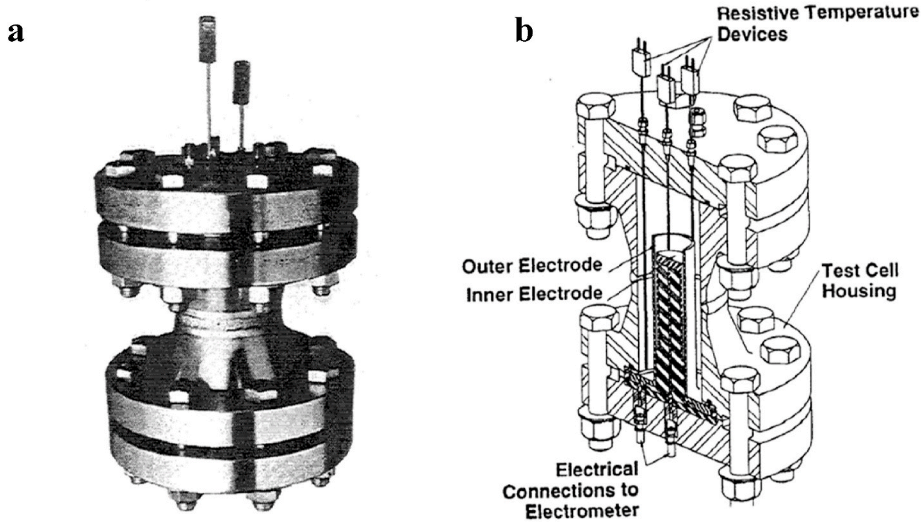

Fig. 8. (a) Image of assembled and (b) cross sectional drawing of resistivity cell [27].

geometry. The cross sectional drawing of the cell provided by the author in Fig. 8 offers little in the way of clarity and is thus difficult to interpret.

They investigated the DC resistivity of an HFC refrigerant called R404A with a 2.55 mm electrode separation and field strength of 39.2 kV/m. The resistivity was tested over a temperature range between 248 K and 283 K with a corresponding saturation pressure range of 0.26 MPa–0.83 MPa. An electrification time of 36 s was used after it was determined as the time needed for current to settle with a less than 1% change occurring. The cell was pressure tested to 10.3 MPa and between temperatures of 210 K and 350 K. A typical test involved cleaning with acetone and evacuating to a 50 μm vacuum before purging with nitrogen gas to remove moisture. The cell was cooled with liquid nitrogen to 250 K before inserting the refrigerant. It was further cooled before beginning measurement of resistivity as the cell warmed to ambient temperature at 10° below the lowest temperature of the temperature range. The cell was then heated to 10° above the highest temperature of the range before starting measurement of resistivity as the cell cooled to ambient temperature. Once a test was completed the fluid was deserviced and the cell was disassembled for cleaning. The time required to obtain the resistivity data for the refrigerant across the temperature range was two days due to the slow change in temperature. The results of the investigation are displayed in Fig. 9.

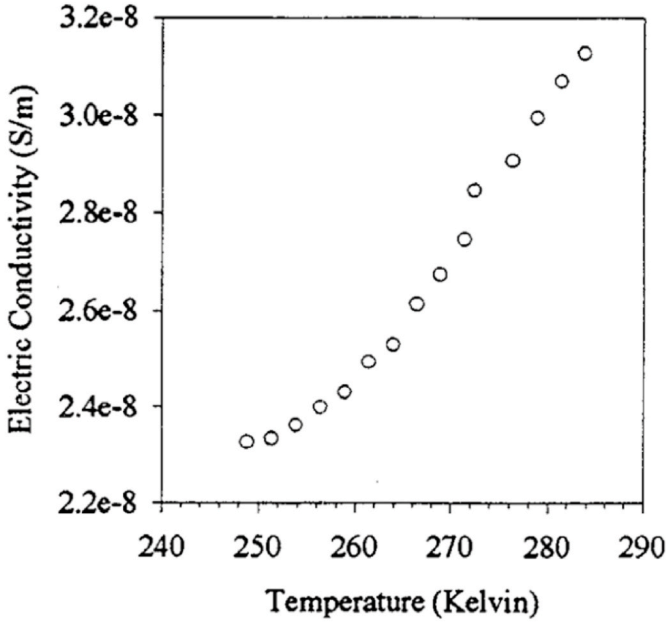

Fig. 9. Electrical conductivity of R404A as a function of temperature [27].

8.4. Feja S. (2012)

Feja [28] tested the DC resistivity using a field strength of 250 kV/m. He did not use a guard ring and instead calibrated the test cell with a reference liquid in advance. The test cell was calibrated after each cleaning process with n-Heptane at room temperature. He made no mention of flushing the cell with nitrogen to limit moisture content. The cell has a large proven measuring range that is evidenced by the experimentally determined resistivity of liquid carbon dioxide (R744) at greater than 10^{14} Ωm. This aligns well with the fact that cell also possesses the lowest cell constant with a value of 0.113 m^{-1} out of the studies discussed. A thermostat bath was used to vary the temperature and keep it constant when performing a test. A Pt100 sensor connected to the inner electrode was used to monitor the temperature. The resulting resistivity values for the refrigerants tested across a temperature range are plotted in Fig. 10.

A description of the cell has not been offered by the author of the study. At the top of the inner electrode, a white polymeric material can be seen which is thought to be an insulator separating the inner electrode extending toward the bottom and the metal above the polymer, possibly serving as a guard electrode. The surface lining the inside cylinder wall of the metal construction on the right side of Fig. 11 (a) is considered another electrode. The cell possesses a flange at the top with two cylindrical ports extending from the top of the flange.

9. Commercial resistivity cells

The current market leaders in commercial resistivity cell offerings include BAUR GmbH's DTL C and Megger Group Limited's OTD test cell (shown in Fig. 12). The specifications for each of these cells is summarised in Table 3. The OTD and DTL C cells possess similar designs, albeit, a detailed description of the arrangement for the latter cell is not provided by the company. They both measure the resistivity of liquids under atmospheric pressure and do not offer the ability to adjust the applied pressure on the tested liquid. The design of these commercial cells is therefore slightly different from the cells developed by the aforementioned studies accordingly. The OTD has an inner measuring electrode cylinder possessing a hemispherical end being enclosed by a similar larger cylinder acting as the anode. The cell is sealed by a non-

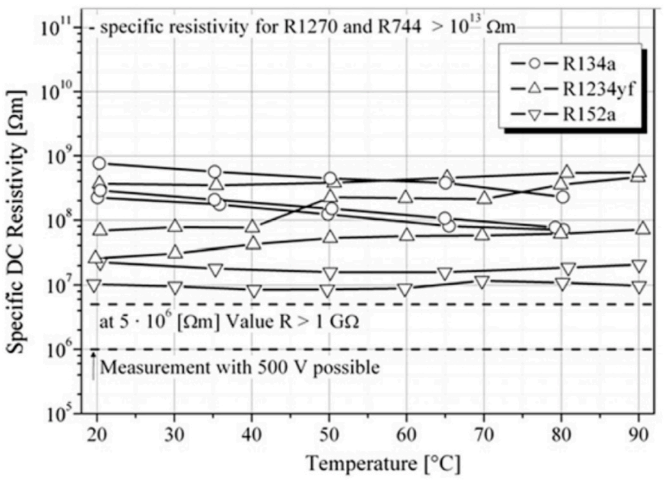

Fig. 10. Resistivity results for R152a, R134a and R1234yf plotted as a function of temperature [28].

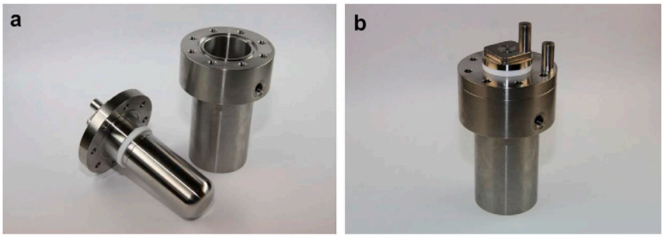

Fig. 11. (a) Inner (left) and outer electrode (right), (b) Assembled test cell [28].

conducting glass covering at the top with an orifice where the liquid enters the cells while the guard and measuring electrode are separated. The guard also serves to distance the orifice from the measuring electrode to prevent electric field distortions around the inlet from extending toward the measuring volume and affecting the electric field homogeneity. The introduction of curvature by the hemispherical ends of the electrodes causes a geometrical enhancement to the electric field. This adversely compromises the electric field homogeneity. However, the degree of this enhancement may be limited by a large radius of curvature. Moreover, a drain valve is situated at the bottom of the cylinder where the fluid exits. The cells also enable easy disassembly for cleaning which helps limit the presence of contaminants [29,30].

10. Recommendations

When designing a high range resistivity cell, it is recommended to utilise a concentric cylinder arrangement to achieve a low cell constant. This is to increase the measuring range of the cell, reduce the volume of liquid required for measurement and reduce the size of the cell. The measuring electrode should also be guarded to achieve a high order of measurement accuracy by limiting any erroneous impact of fringe fields. The surface of the electrodes should be smooth with no sharp edges to prevent charge injection caused by electric field amplification. In terms of procedural recommendations, moisture content in the cell should be limited by cleaning with a solvent such as acetone or n-heptane and evacuating the cell with a vacuum pump before purging with nitrogen gas.

11. Conclusion

An analysis of resistivity cell designs has been presented including a description of considerations that need to be addressed when attempting to develop a resistivity cell for insulating liquids, particularly for HFCs

Table 3
Comparison of specifications for OTD and DTL C commercial resistivity cells.

Test cell	OTD [29]	DTL C [30]
Measurement range	2.5 MΩm to 100 TΩm	2.5 MΩm to 100 TΩm
Temperature range	10 °C–110 °C	11 °C–110 °C
DC voltage range	125 V–500 V	500 V max
Humidity	<60% RH	<100% RH
Accuracy	±1% from calibrated value	±1% from calibrated value

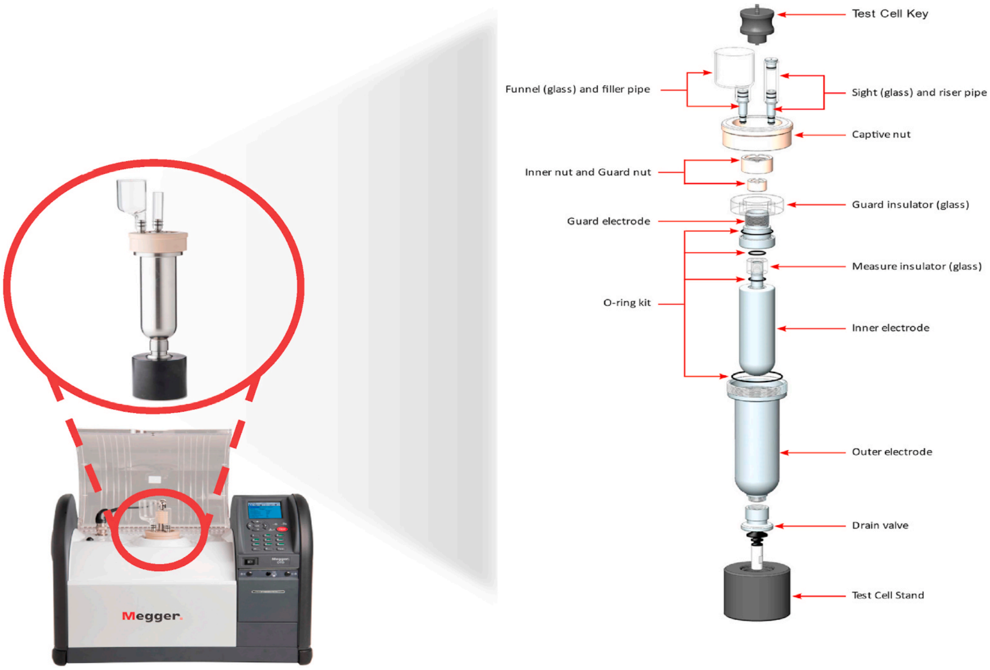

Fig. 12. Arrangement of OTD resistivity cell, courtesy of Megger Ltd [29].

used in pharmaceutical metered dose inhalers and industrial refrigerants. The nature of charge carriers and the associated mobility in dielectric liquids is also detailed. An explanation of the guidelines set out by IEC and ASTM standards relating to resistivity measurement of dielectric liquids is offered including the difference between AC and DC resistivity measurement.

Several research studies into the resistivity of dielectric liquids using individually developed cells have previously been published with the majority of these involved in resolving the resistivity of liquid refrigerants. This article therefore summarises the arrangement of these custom cells and the main findings from the studies. It was suggested that a standardised method be developed for dielectric liquid resistivity tests performed under pressure.

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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