Dielectric properties of refrigerant fluids HFO-1336mzz-E, HFC-245fa and HFE-7100

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ABSTRACT

This paper presents the comparison of the dielectric properties of three different refrigerant fluids, HFE-7100, HFC-245fa, and HFO-1336mzz(E), the latter having a global warming potential two orders of magnitude lower than the other two. Refrigerant fluids are used, among others, in generator circuit breaker applications where good thermal and chemical properties (including low global warming potential) must be coupled with excellent dielectric withstand and low electric conductivity. The paper describes in detail the applied measurement techniques and the results obtained, as well as comparisons to previous measurements and literature. The comparison includes dielectric breakdown in liquid and vapor phase, DC conductivity, real permittivity and dissipation factor.

Index Terms — dielectric liquids, conductivity measurement, permittivity measurement, cooling, dielectric breakdown, dielectric losses, circuit breakers, generators

1 INTRODUCTION

THE dielectric properties of refrigerant fluids are extremely important in high and medium voltage applications such as generator circuit breakers (GCB), which require thermally effective cooling systems and at the same time need to be able to withstand the high voltages (several tenths of kVs) present in the system.

A very important property of such fluids is also the global warming potential (GWP). Historically, high GWP refrigerant fluids have been used, giving priority to performance related technical properties with respect to other aspects. Nowadays, the main targets in the development of new systems must not include only sheer performance. Low environmental impact has increased priority, and new fluids become of interest.

In this paper we compare three fluids:

1. Hydrofluorether HFE-7100, see Figure 1a;

- 2. 1,1,1,3,3-Pentafluoropropane (in the following: HFC-245fa or also R245fa, according to [1]), see Figure 1b;
- 3. (E)-1,1,1,4,4,4-Hexafluoro-2-butene in the following HFO-1336mzz(E), see Figure 1c;



Figure 1. Fluids evaluated in the present paper: HFE-7100 ($C_5H_3F_9O$) (a), HFC-245fa ($C_3H_3F_5$) (b), and HFO-1336mzz(E) (2- $C_4H_2F_6$ -E) (c).

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Figure 1 shows the molecules of HFE-7100, HFC-245fa and HFO-1336mzz(E).

Table 1 reports some of the most relevant properties of the three substances, including GWP, toxicity and safety class. For all three fluids toxicity and safety are acceptable: HFO-1336mzz-E has a higher toxicity which does not represent a problem in most applications, HFC-245fa has a lower safety class. Nevertheless, HFO-1336mzz-E has a global warming potential of two orders of magnitude lower than the other two.

Table 1. General p	property comparison: I	HFE-7100, HFO	C-245fa and HFO-
	1336mzz(1	E).	

Name (acc. ASHRAE 15)	HFE-7100	HFC-245fa	HFO- 1336mzz(E)
Chemical Formula	$C_5H_3F_9O$	$C_3H_3F_5$	$2\text{-}C_4H_2F_6$
Molecular mass (g/mol)	250	134	164.1
CAS number	163702-07-6	460-73-1	407-60-3
Boiling point @ 1 bar (°C)	61 [2]	15.3 [3] [4][5][6]	7.4 [7]
Melting point (°C)	135	107	76
Critical temperature (°C)	195.3	154	137.7
GWP (100 year horizon)	421[8], 297 [9]	858 [8] [3], 1030 [1]	9 [7]
Ozone depletion potential (ODP)	0	0	0
Acute Inhalation Toxicity (LC50, 4-hr Rat, ppm)	> 100000 [2]	> 200000 [4]	> 17000 [10]
Flammability	Possibly [2]	No [4][11]	No [12]
Safety class (acc. ASHRAE 15)	A1	B1 [1]	A1 [1][3][12] [7]

In this paper these three fluids are further compared in terms of breakdown in liquid and vapor phase, DC conductivity, real permittivity and dissipation factor. To the author's knowledge, the measurement of the dielectric breakdown of HFO-1336mzz-E has never been published, neither in liquid nor in vapor phase, as well as its direct comparison to another fluid adopting the same measurement technique.

Moreover, also the other dielectric properties are not available in literature for HFO-1336mzz-E, and limited data is provided by the manufacturers.

The paper is structured as follows. Section 2 shows the results and the measurement technique applied to evaluate the dielectric breakdown in vapor and liquid phase. Section 3 reports the comparison of the DC conductivity, the real permittivity and dissipation factor of the three fluids. Finally, in Section 4 we draw some conclusions.

2 DIELECTRIC BREAKDOWN IN VAPOR AND LIQUID PHASE

The breakdown (BD) field in both vapor and liquid phase is tested using a vessel with Rogowski shaped aluminum electrodes. Their surface is sandblasted to a technically relevant roughness value (on average, $R_z=30 \mu m$). A sketch of



Figure 2. CAD sketch of the vessel used for BD tests (a), Rogowski electrodes in new conditions (b).

the vessel is shown in Figure 2. The electrodes have 10 mm spacing for vapor phase measurements and 5 mm spacing for liquid phase measurements.

For vapor phase measurements, the vessel is vacuumed to 1 mbar pressure, then filled with the required amount of substance in order to reach the desired pressure.

For the tests in liquid phase, the substance is poured from its pressurized bottle into the sealed vessel (previously under vacuum) which is filled with the required mass, constantly measured by scales, to reach the proper filling level, above the electrodes. The boiling point is increased, and the substance is kept in liquid state by maintaining the sealed vessel pressure sufficiently high. One electrode is fixed to ground, to the other one a step DC voltage generated by a Van de Graaff (VdG) generator is applied. The voltage step has a rise time of approximately 200 ns to peak.

From application of this voltage shape it is possible to have information on both DC and Lightning Impulse (LI) probability distributions. These are derived by assuming that each breakdown is a "DC" breakdown, whereas all BDs that happened after a certain threshold (time to BD > 10 ms) are to be considered as holds for a virtual "LI" application. Therefore, the values provided in the following as "LI" should not be mistaken by values obtained by an IEC standard compliant LI waveform application – they are nevertheless still representative and typically compatible with standard LI measurements.

The dielectric strength of a substances is evaluated with statistical methods [13], based on a high number (200-300) of voltage applications, according to three possible different methodologies: up and down method (U&D), random method and voltage prediction.

In the U&D method the applied voltage is increased until first BD, then it is decreased by a fixed step, until a hold is reached, then increased again. The procedure goes on iteratively until the maximum number of shots is reached. This procedure identifies very accurately the 50% BD probability.

The random method takes the user defined voltage range and steps, then an algorithm selects a random sequence, in order to cover all the range with the selected total number of shots.

The voltage prediction method exploits a custom algorithm



Figure 3. DC and LI vapor phase 50% BD probability (based on 200 shots, voltage prediction). The uncertainty interval corresponds to 1σ . All data shown in the figure are for positive polarity applied to the top electrode. The measurements performed with negative polarity, not shown here, do not differ substantially and are within the uncertainty range. The HFC-245fa breakdown values are obtained using the Paschen curve provided in [14] and [15] and with a 1 cm gap.

that calculates the next voltage application which will provide maximum information on the BD probability distribution.

Each test here reported is performed in one specific polarity (either positive or negative, i.e. no polarity reversal is done during one test) and consists of 200 shots, unless differently specified (300 shots for two of the liquid HFC-245fa tests). These 300 shots test provided the same results as the 200 shots tests, for HFC-245fa, confirming that a statistic on 200 shots is sufficient.

The waiting time between shot to shot has been set to 1 minute for all gaseous phase measurements and 5 minutes for all liquid measurements except the first experiment on liquid where it was set to 1 minute. A difference between 1 and 5 minutes waiting time in liquid measurements could not be seen, nevertheless the higher waiting time was chosen for all the other experiments. This guarantees that in case bubbles are formed they are given enough time to dissolve again, thus avoiding preconditioning of the next voltage application.

The hold and breakdown distributions are then fitted to a logistic distribution and the 50 % probability and σ are provided.

2.1 VAPOR PHASE DIELECTRIC BREAKDOWN

Figure 3 shows the 50% probability BD electric fields (and σ) of HFO-1336mzz-E at different pressures obtained using a voltage prediction algorithm with 200 shots. Two different purity levels of the substance are compared¹. The differences are within uncertainties, as well as the differences between polarities (only positive polarity measurements are shown in the figure). The uncertainties (σ from the BD probability fitting algorithm) are plotted with a coverage factor of 1.

Such measurements in gas phase are relevant also for GCB heat pipe applications since both phases are typically present

¹ Gas chromatography (before tests) provided a 99 % purity for the "high purity" sample and approximately 80 % purity for the "low purity" sample.

in such heat pipes.

The measurements show a rather good dielectric strength of HFO-1336mzz-E. To the author's knowledge such measurements have never been shown before and are still not available for HFE-7100 and HFC-245fa with the same technique, but a comparison to SF_6 (using the same vessel and methodology) is shown in Figure 3. The same figure also shows HFC-245fa breakdown values obtained using the Paschen curve provided in [14] and [15] and with a 1 cm gap, as the measurements performed in the vessel of Figure 2.

2.2 LIQUID PHASE DIELECTRIC BREAKDOWN

Figure 4 shows the comparison of liquid of HFO-1336mzz(E) and HFC-245fa 50% BD fields. HFO-1336mzz(E) was first tested with U&D method (1 minute and 5 minutes waiting time showed almost no difference). Then it was possible to test it with the voltage prediction algorithm (waiting time was kept equal to 5 minute).

All measurements are compatible and have overlapping uncertainty intervals.

Due to the more conductive behavior of HFC-245fa, it was not possible to test it with the standard voltage prediction method. As soon as the step DC is applied to the vessel, the voltage drops fast enough to be interpreted as a breakdown. Therefore, algorithms such as voltage prediction or U&D, which base the selection of the next voltage(s) to be applied on the result of the last experiment(s) do not converge for this liquid.

Thus, the BD experiments for HFC-245fa were done with a random voltage application technique, in a range between 60 and 180 kV, with 200 and 300 shots.

Both liquids show very good dielectric strength compared to the one of HFE-7100 reported in [16]. Even if obtained with a different technique, the AC values in [16] at room temperature may be put in comparison to the DC values reported in this paper.

Paper [16] reports several measurement, including ageing effect, and an AC 50% BD probability of 97.4 kV/cm at 295 K



Figure 4. DC (a) and LI (b) liquid phase 50% BD probability and σ (based on 200 shots, unless specified). The uncertainty interval corresponds to 1 σ .

in new conditions, which slightly increases as more BDs happen in the fluid, to reach maximum value of 100-110 kV/cm. These values are compatible with what reported on $3M^{TM}$ data sheet: 28 kV (RMS) with a 0.1 inch electrode gap, i.e. 110.2 kV/cm.

In conclusion, in the liquid phase, the dielectric strength of HFO-1336mzz(E) is higher than both HFC-245fa and HFE-7100, the latter having the worse performance, approximately half the HFO-1336mzz(E) one.

3 OTHER DIELECTRIC PROPERTIES

In addition to general properties such as the ones in Table 1 and to the breakdown properties described in the previous section², other dielectric properties have a direct impact on cooling performance of a cooling medium.

These are:

- conductivity
- complex permittivity, $\varepsilon = \varepsilon' j\varepsilon''$
- Dissipation factor, tan δ.

These properties determine how much power is lost in such a medium when it is subject to a DC or an AC electric field. Therefore, they are used to quantify the possible temperature raise due to presence of high E field, and to dimension the maximum power specifications of a GCB.

The loss tangent is defined as the ratio of the lossy and the loss-less reactions to the electric field E:

$$\tan \delta = \frac{\omega \varepsilon'' + \sigma}{\omega \varepsilon'} = \tan \delta_P + \tan \delta_C \tag{1}$$

As it can be seen in equation (1), tan δ is the sum of two components, a loss factor by polarization tan δ_P and a loss factor by conduction tan δ_C . Nevertheless, in practice, energy dissipation due polarization may be indistinguishable from the loss due to, for example, space charge effects in insulators.

In the following we summarize the presently available information on permittivity, conductivity and tan δ of HFO-1336mzz(E), HFE-7100 and HFC-245fa indicating when results are provided for the first time. These three quantities depend, in first approximation, on frequency, applied field and temperature and not always they can be measured independently. For conductivity, it is possible to measure the DC one, as a rough indication of the type of losses in the medium.

3.1 DC CONDUCTIVITY

The DC conductivity can be deduced by applying a DC voltage to a capacitive test cell containing the fluid and measuring the resulting current, I(t), by definition for an infinite time:

$$\sigma = \lim_{t \to \infty} \frac{I(t)\varepsilon_0}{VC_0}$$
(2)

² Other properties such as enthalpy, density, viscosity, boiling curve, critical temperature etc. have also an important role in the cooling behavior of a refrigerant but their analysis is not within the scope of the present paper.



Figure 5. Liquid HFO-1336mzz(E) DC conductivity measurements (a) and the used capacitive vessel, 1 kV/mm applied field (b).

where C_0 is the capacitance of the empty test cell.

It should be noted that I(t) is a polarization current (executing complete polarization/de-polarization cycles may speed up the measurements [17], but this technique was not applied).

Equation (2) is valid under the following assumptions:

- Inside the vessel, space charge and material with different polarization can be neglected.
- The field-configuration is homogeneous (or quasihomogeneous).

At present time HFO-1336mzz(E) conductivity measurements have never been reported, as well as their direct comparison to those of other fluids. Figure 5a shows measurements executed using the test cell reported in Figure



Figure 6. Liquid HFC-245fa DC conductivity measured in the vessel shown in Figure 2.

5b for temperature between 253 and 363 K.

Figure 5a shows that larger temperatures result in lower viscosity (hence higher ion mobility) and larger currents, having also a faster decay.

The same figure also shows measurements of liquid HFO-1336mzz(E) DC conductivity (cross symbols) executed by using the same capacitive vessel used for breakdown experiments shown in Figure 2. These measurements were performed using a Megger instrument or a FUG HV power supply and a Keithley multimeter, with similar results.

The measurements obtained with the latter technique are one order of magnitude lower and may be considered as a lower limit, since the vessel and the measurement set-up were not specifically designed for the purpose.

When the same vessel and technique are applied to liquid HFC-245fa DC conductivity measurements the results are several orders of magnitude higher, as shown in Figure 6, but compatible to values reported in other sources, [18] and [15], even if only AC values are available.

In [18], the resistivity is measured at 60 Hz, with voltage application of 10 V on a 3-8 mm gap, between 298.15 K and 323.15 K. The conductivity values at ambient temperature range between $4 \cdot 10^{-8}$ S/m and $5 \cdot 10^{-8}$ S/m. [15] reports a value of AC conductivity measured at 1 kHz and at ambient temperature of $1.4685 \cdot 10^{-9}$ S/m.

Interestingly, the HFC-245fa DC conductivity is not only 3 orders of magnitude higher than the HFO-1336mzz(E) measured in the same vessel and with the same technique, but also shows a completely different behavior, with an extremely low decay of the conductivity. This slow and progressive reduction in the current may be induced by field reduction due to progressive charge accumulation, the DC conductivity of a polarizable liquid being sensitive to the applied field. We note that for DC HV applications this phenomenon should not be overlooked, and a less ionizable liquid such as HFO-1336mzz(E) would be in general a better choice.

The high conductivity of HFC-245fa is also affecting the dielectric breakdown experiments, as reported in Section 2.2. It causes the fast voltage drop, which can be interpreted in certain cases as a breakdown.

For what concerns HFE-7100, values ranging between $1-4\cdot10^{-8}$ S/m are reported in [16] at ambient temperature and under 250 V/mm field, therefore in similar range as HFC-245fa.

In conclusion, the DC conductivity of liquid HFO-1336mzz(E) is orders of magnitude lower than both HFC-245fa and HFE-7100, therefore a suitable candidate for HV applications.

3.2 REAL PERMITTIVITY

We report here for the first time the real permittivity and the dissipation factor of HFO-1336mzz(E) and HFC-245fa, obtained with AC spectroscopy using a 1260A



Figure 7. Real permittivity of liquid HFO-1336mzz(E) (a) and HFC-245fa (b) obtained with dielectric spectroscopy similarly to what done in [16] and verified using the test vessel of Figure 2 and AC voltage application. The dielectric spectroscopy measurements have been obtained using reference liquid Novec 649 and equation (3) with and ε'_n =1.8 (dashed lines, lower uncertainty bound) or 1.9 (continuous lines, upper uncertainty bound). Literature values are also reported when available.

impedance/gain-phase analyzer and a Solartron 1296 dielectric interface, similarly to what done in [16].

Further verification tests were also performed with the setup shown Figure 2, by applying high voltage (up to 20 kV) AC signal at frequency f from a voltage transformer and measuring the current with a Keithley 2002 Multimeter. With this technique it was possible to measure the real part of the permittivity for fluids HFO-1336mzz(E) and HFC-245fa and compare it to the dielectric spectroscopy results, as shown in Figure 7. This figure also reports literature values [14, 15, 18, 19] for HFC-245fa permittivity. Literature values and verification measurements of liquid HFC-245fa are all compatible with the full frequency sweep in the range 1-100 kHz, which is here shown for the first time.

Calculation of the real permittivity from real capacitance measurements in AC is done using the following formula:

$$\frac{\varepsilon_x'-1}{\varepsilon_n'-1} = \frac{C_x - C_a}{C_n - C_a},\tag{3}$$

where C_x is the capacitance of the vessel filled with the substance with unknown real permittivity ε'_x , C_a is the vessel

capacitance in air, and C_n is the measured capacitance with vessel filled with a reference substance with known permittivity ε'_n . For the dielectric spectroscopy measurements of Figure 7 the reference substance is Novec 649, whose real permittivity is known with some uncertainty and ranges at room temperature between 1.8 and 1.9, as reported in [16] and [20].

For what concerns HFE-7100, measurements obtained with the same dielectric spectroscopy technique are reported in [16].

The comparison between the three liquids shows the following:

- At ambient temperature HFO-1336mzz(E) has the typical behavior of an insulating material with dilute carrier density and well-defined dielectric constant. At higher temperatures, the real permittivity of HFO-1336mzz(E) increases at lower frequencies (<100 Hz), most probably due to higher charge mobility caused by higher temperature. Real permittivity range is 2-4 at 50 Hz for a temperature between 253 and 363 K.
- HFC-245fa is capacitive when the frequency is higher than 1 kHz. When the frequency is lower than 1 kHz the behavior is resistive, and the relative permittivity increases dramatically. In other words, in this low frequency range, the capacitive behavior assumption fails. We also note that the higher this frequency turning point is, the more conductive is the liquid. At 50 Hz and ambient temperature the real permittivity has a wide scatter, 40-500, depending on liquid sample and its purity.

The behavior at lower frequencies is very important for a wide range of applications – the measurements previously available in literature (Figure 7b) could not show this, since performed at higher frequencies.

- HFE-7100 has a resistive behavior at low frequency, the turning point is lower than HFC-245fa, though (50-100 Hz instead of 1 kHz of HFC-245fa). Increasing temperature increases the turning point, as also observed for the other liquids. At 50 Hz the real permittivity is 8-80 in a temperature range between 253 and 293 K.
- For HFO-1336mzz(E) and HFC-245fa the real permittivity decreases with increasing applied field this behavior is more marked in the HFC-245fa fluid.

3.3 DISSIPATION FACTOR

The loss (or dissipation) factor by polarization, tan δ_P ($\varepsilon''/\varepsilon'$) obtained from dielectric spectroscopy measurements for liquid HFO-1336mzz(E) and HFC-245fa is also reported here for the first time (see Figure 8) and compared to the HFE-7100 results reported in [16] and measured with the same technique.

From this comparison we observe:

• HFO-1336mzz(E) has a lower tan δ_P than all other fluids (0.005-0.05 at 50 Hz in a temperature range between 253 and 363 K), therefore also reduced thermal losses.



Figure 8. Dissipation factor of liquid HFO-1336mzz(E) (a) and HFC-245fa (b) obtained with dielectric spectroscopy, as in [16].

- HFC-245fa has a higher dissipation factor (10-100 at 50 Hz on different samples), therefore a worse dielectric behavior with respect to the other two fluids and a larger scatter. The available value for dissipation factor from literature is 3.1187 @ 1 kHz, in [14] and [15], about 1 order of magnitude lower than the range here obtained.
- HFE-7100 has intermediate performance (2-80 at 50 Hz in a temperature range between 253 and 303 K).

4 CONCLUSIONS

In this paper the comparison of the dielectric properties of three different refrigerant fluids, HFO-1336mzz(E), HFC-245fa and HFE-7100 has been shown.

The comparison has included dielectric breakdown in liquid and vapor phase, DC conductivity, real permittivity and dissipation factor, as well as other general properties, e.g. GWP.

The paper has provided for the first time measurements for all dielectric properties of HFO-1336mzz(E), performed at variable temperature. Furthermore, also the HFC-245fa dielectric properties measured with the same approach were here reported for the first time – the available literature results (for specific frequencies or temperatures) were also reported for comparisons.

The most relevant findings from this thorough comparison are:

- HFO-1336mzz-E has a global warming potential of two orders of magnitude lower than the other two.
- In vapor phase, the dielectric strength of HFO-1336mzz(E) is compatible with high voltage application, as well as the one of HFC-245fa, where values are available. Information on HFE-7100 is not available.
- In liquid phase, the dielectric strength of HFO-1336mzz(E) is higher than both HFC-245fa and HFE-7100, the latter having the worse performance, approximately half the HFO-1336mzz(E) one.
- The DC conductivity of liquid HFO-1336mzz(E) is several orders of magnitude lower than both HFC-245fa and HFE-7100, therefore extremely suitable for HV applications. The observed behavior of HFC-245fa in both breakdown tests and DC conductivity tests can be explained with a high polarizability. Less ionizable liquids such as HFO-1336mzz(E) are in general a better choice for HV applications.
- At ambient temperature HFO-1336mzz(E) has the typical behavior of an insulating material with dilute carrier density and well-defined dielectric constant (capacitive behavior) whereas both HFC-245fa and HFE-7100 show a resistive behavior for low frequencies. The turning point of HFC-245fa (around 1 kHz) is also very high compared to the HFE-7100 one (50-100 Hz).
- HFO-1336mzz(E) has a lower tan δ_P than all other fluids (0.005-0.05 at 50 Hz in a temperature range between 253 and 363 K), therefore also reduced thermal losses. HFC-245fa has the highest dissipation factor (10-100 at 50 Hz on different samples), therefore a worse dielectric behavior with respect to the other two fluids and a larger scatter. HFE-7100 has an intermediate performance (2-80 at 50 Hz in a temperature range between 253 and 303 K).
- For all three fluids toxicity and safety are acceptable, HFO-1336mzz-E having the highest toxicity and HFC-245fa having the lowest safety class.

In conclusion, HFO-1336mzz(E) seems to be a very good choice for HV applications, e.g. cooling systems of high voltage switchgear, having at the same time a reduced GWP.

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