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Experimental and Theoretical Analysis of Refrigerant Charge Extraction Methods

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ABSTRACT

According to the European Commission, heating, cooling and domestic hot water (DHW) production implies 79% of the total energy consumed in households. Moreover, as 84% of this energy is still generated from fossil fuels, it is required taking measures in order to improve the current situation. One way to increase the decarbonization in households is the use of heat pumps powered by renewable energy sources. Additionally, the energy captured by heat pumps is considered renewable when a certain efficiency value is achieved (2009/28/CE directive).

Nowadays, refrigerants used by heat pumps are mainly hydrofluorocarbons (HFCs) which have a high global warming potential (GWP) and have a clear deadline defined at F-Gas EU Regulation 517/2014. For this reason, natural refrigerants will play an important role within heating, cooling and DHW production at the household sector. However, most of the natural refrigerants with acceptable thermodynamic properties are at least slightly flammable or toxic, and its use has an associated risk due to this condition. That is the cause that the amount of refrigerant charge must comply with the maximum refrigerant charge limited by regulations, and the prediction of the nominal refrigerant charge amount inside a heat pump has become an important matter within the design process of a heat pump.

There already exist prediction models of refrigerant charge but, under certain conditions, they do not fit the reality. The current prediction methods which use simulation models may be improved by knowing the actual distribution of the refrigerant inside the different components of a real heat pump.

In order to empirically determine the refrigerant charge amount inside each part of the heat pump, several methods can be used, but the most employed one is the "remove and weight" technique. This technique consists in extracting the refrigerant charge of each element of the heat pump in a sample cylinder in order to weight it, after isolating each component from another. A similar but less precise approach of this method is employed when recovering refrigerant in maintenance or decommissioning tasks.

In this contribution, a theoretical and experimental study of different charge extraction methods is presented. In the study, every method has been analyzed according to different factors: percentage of refrigerant extracted, security measures employed, the time needed until stability is reached and cost of the technique.

The results of this study will help in the selection of best extraction method according to the precision of the results needed, the available resources and the training of the staff which in the end will be performing the experiments.

1. INTRODUCTION

Within the lifetime of a heat pump or a laboratory prototype which uses refrigerant as fluid, there can be several times when it is needed to extract the refrigerant located inside. One of the possible reasons is to avoid venting the refrigerant to the atmosphere after its use, due to the environmental impact of this action; and there is also a common use of the extraction, which is to know the charge distribution in the components of a heat pump.

Several studies have been done in order to know the refrigerant distribution of the heat pump, as mentioned in the works of Corberán *et al.*(2008), Cremaschi (2004), Li *et al.*(2015) and Peuker, (2016). In this matter, the different techniques used can be divided into two big groups, On-Line measurement Method (OLM) or Quick Closing Valves Method (QCVM), both mentioned in the work of Peuker (2016). The first group, OLM, consist in measuring the refrigerant inside each component while the system is working, i.e., without stopping the heat pump. Contrarily, the QCVM consist in closing suddenly valves placed in the refrigerant circuit to isolate each part from each other when the desired steady conditions are reached. There also exists a hybrid method between the OLM and the QCVM, which is the Quasi On-Line measurement Method (QOLM) developed by Ding *et al.* (2009).

If QCVM is used, after the isolation of each component, the amount of refrigerant inside them must be measured. There are mainly two ways refrigerant measurement inside a part of the refrigerant circuit, using an expansion tank, and the called Remove and Weight Technique (RWT). The expansion tank technique is widely explained in the work of Björk (2005). Besides, the RWT consist in connecting a sample cylinder to each part where the refrigerant is trapped and induce a pressure difference between both parts, making the refrigerant flow to the sample cylinder. The common way to induce this pressure difference is either by a temperature difference or mechanically. However, in the mechanic extraction there is always a remanent refrigerant which cannot be extracted and remains trapped inside the refrigerant recovery machine. In order to stablish the temperature difference, a sample cylinder is usually chilled by being immersed in a liquid nitrogen or ice bath, but the choice of this chilling method used is not normally done after having studied which is the best chilling medium for the final application. There is also other method mentioned in the work of Lee *et al.*(2020) which is part of the work of the project ASHRAE RP-1785 where the whole components are separated physically and weighted with the refrigerant and lubricant still inside.

In this contribution, an experimental and theoretical analysis is done in order to know the actual precision of each chilling method to help the selection for each application.

2. METHODOLOGY

The purpose of this work is to empirically determine the precision of each method of refrigerant extraction, in order to have enough information when it is needy to select one method to be used. The study has several parts.

2.1 Extraction process and tests performed

For the extraction process it has been used a Brazed-Plates Heat Exchanger (BPHE) as a representative part of an actual heat pump where the QCVM has been employed. This experimental setup can be seen in Figure 1a. This BPHE is connected to a refrigerant container in order to be filled with a controlled amount of refrigerant and it is also connected to a sample cylinder in order to extract the refrigerant (Figure 1b and c).

a)





c)





Figure 1: Experimental setup. a) BPHE, b) Sample cylinder, c) sample cylinder connected, d) cooling reservoir.

b)

d)

The process of each test has been as follows:

- 1. Make mechanical vacuum in the sample cylinder and BPHE.
- 2. Tare weight of refrigerant bottle (m_1) and sample cylinder (m_2) .
- 3. Add refrigerant charge to the system. It has been approximately 300g of R134a.
- 4. Weight the refrigerant bottle again (m_3) .
- 5. Wait until thermal equilibrium is reached.
- 6. Start cooling the sample cylinder with the valves closed. (Figure 1d)
- 7. Open the valves to make the refrigerant flow from the BPHE to the sample cylinder.
- 8. Close the valves once the pressure of the system gets stable.
- 9. Warm and dry the sample cylinder.
- 10. Weight the sample cylinder (m₄).

The results recorded are the time spent, final pressure in the system, temperatures of the secondary fluid and the BPHE, and the weight at the beginning and end of the sample cylinder and refrigerant tank.

The cooling media used to create a pressure difference between the sample cylinder and the BPHE are listed in the Table 1.

Cooling medium	Reference temperature	Reference pressure (R134a)	Density of R134a @25°C
Liquid nitrogen (LN2)	-196 °C	<0.0001 bar	$\sim 0 \text{ kg/m}^3$
Phase change materials (-18°C) and glycol (PCM-18)	-18 °C	1.44 bar	6.13 kg/m ³
Ice bath (Ice)	0°C	2.92 bar	12.84 kg/m ³

Table 1: Cooling media employed in the tests

For each cooling medium employed, different tests have been performed. There are two main variances between the tests, which are the place of extraction and the final temperature of the BPHE. Regarding the place of extraction, the refrigerant has been extracted from the top of the heat exchanger (vapor extraction) or from the bottom part (liquid extraction); on the other hand, regarding the temperature of the heat exchanger, it has been studied at room temperature or heated by an electric resistance. All the tests performed, and the main variations of each test are listed in the Table 2.

Table 2: List of tests	performed.
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Cooling medium	Repetitions	Type of extraction	Resistance connected or
			not
Liquid nitrogen (LN2)	4	Liquid extraction	No
Liquid nitrogen (LN2)	3	Liquid extraction	Yes
Liquid nitrogen (LN2)	3	Vapor extraction	No
PCM(-18)	7	Liquid extraction	No
PCM(-18)	3	Liquid extraction	Yes
PCM(-18)	3	Vapor extraction	No
Ice	3	Liquid extraction	No

2.2 Analysis of the process

In order to know the precision of each test, the initial refrigerant mass inserted in the system must be compared to the extracted mass plus the theoretical remaining refrigerant charge in the BPHE. To know the remaining refrigerant charge inside the BPHE it is compulsory to know the inner volume of the components. This has been calculated using the isothermal gas method which is mentioned in the work of Jin & Hrnjak (2016).

Once the volume is known, the theoretical remaining refrigerant mass (m_t) can be calculated by simply multiplying the density, known at the final conditions of pressure and temperature, as shown in equation (1). The density in equation (2) is calculated using the software REFPROP developed by Lemmon *et al* (2013).

$$m_t = \rho \cdot V \tag{1}$$

$$\rho = f(P,T) \tag{2}$$

Besides, the refrigerant extracted (m_6) is equal to the difference between the weight of sample cylinder at the beginning and the end of the test (3).

$$m_6 = m_4 - m_2 \tag{3}$$

The refrigerant inside the system is equal to the difference between the weight of refrigerant tank before and after adding the refrigerant in the system (4).

$$m_5 = m_1 - m_3 \tag{4}$$

And finally, the refrigerant not extracted (m_7) is calculated as the difference between the refrigerant inserted in the system and the refrigerant extracted (5)

$$m_7 = m_5 - m_6 \tag{5}$$

So, the error in the refrigerant mass determination committed in each test(ϵ) is the difference between the refrigerant calculated knowing the finale volume, pressure, and temperature(m_t) and the refrigerant not extracted (m_7), i.e., the refrigerant amount that theoretically should remain in the system at the end of the test minus the refrigerant amount that remains in the system (6):

$$\varepsilon = m_t - m_7 \tag{6}$$

2.3 Uncertainty of the measurements

In the tests, the sensors used, and its uncertainty are listed in the Table 3.

Table 3: Sensors employed

Measurement variable	Sensor	Uncertainty 2σ
Temperature	Type T ThermoCouple class 2	±2°C
Mass	Scale Kern	±0.5g
Pressure	Yokogawa EJA510E	0.008bar

3. RESULTS AND DISCUSSION

3.1 Extraction methods results

In the Table 4 are shown the results of the test campaign mentioned. In this table, can be seen that the refrigerant amount of each test has been approximately 300g of R134a. As well, the most important data are the two last columns and more precisely, the difference of both columns. The lower this difference, the more precise is the method employed.

Test	Initial ref charge, m ₅ (g)	Time (h)	Final T secondary (°C)	Final pressure (bar)	Theoretical pressure (bar)	Amount remaining, <i>m</i> ₇ (g)	Theoretical amount, $m_t(g)$
I NO 1	205	0.20	106	0.006	0	0.6	0
	303	0:30	-190	0.000	~0	0.0	~0
	280	0:30	-196	0.004	~0	0.18	~0
LN2 3	296.9	0:30	-196	0.003	~0	-0.4	~0
LN2 4	306.1	0:30	-196	0.002	~0	-0.8	~0
LN2 5	296.6	0:30	-196	0.003	~0	0.16	~0
LN2 6	290	0:30	-196	0.002	~0	1.04	~0
LN2 7	288	0:30	-196	0.002	~0	0.6	~0
LN2 8	306.2	0:30	-196	0.003	~0	1.7	~0
LN2 9	299.4	0:35	-196	0.002	~0	0.62	~0
LN2 10	285	0:35	-196	0.002	~0	-0.2	~0
PCM(-18) 1	291.4	5:00	-17	1.5	1.51	3.4	4.38
PCM(-18) 2	299.7	1:00	-17.8	1.55	1.46	5	4.43
PCM(-18) 3	292.2	1:00	-17.24	1.486	1.49	3.8	4.32
PCM(-18) 4	303.4	1:00	-16.74	1.566	1.53	3.2	4.33
PCM(-18) 5	300.5	1:00	-16.85	1.495	1.52	4.1	4.34
PCM(-18) 6	292.2	1:00	-18.4	1.426	1.42	3.1	4.07
PCM(-18) 7	292.6	1:00	-16	1.63	1.57	7.3	4.82
PCM(-18) 8	302.8	1:00	-16.8	1.56	1.52	5.6	4.55
PCM(-18) 9	301.6	1:00	-15.2	1.68	1.63	3.8	4.37
PCM(-18) 10	308.9	1:00	-16.2	1.57	1.55	5	4.49
PCM(-18) 11	302.6	1:00	-14.5	1.69	1.68	4.7	4.5
PCM(-18) 12	301.5	0:20	-20.26	1.343	1.31	2.6	3.76
PCM(-18) 13	304.2	1:00	-16.5	1.57	1.54	4.9	4.64
Ice 1	309.8	1:00	-3	2.68	2.62	8.7	7.76
Ice 2	304.2	1:00	-3.8	2.64	2.55	9.3	7.52
Ice3	301.4	1:00	-0.7	2.98	2.85	9.2	8.49

Table 4: Results obtained in the test campaign.

These data can be better seen in the Figure 2. As it can be seen, the prediction of each method is accurate enough for standard uses. If it is required the highest precision, liquid nitrogen should be used due to its reduced uncertainty.



Figure 2: Results of refrigerant extraction. a) Ice tests, b) PCM tests and c) LN2 tests.

As shown in Figure 2, there is no main difference in the results of the tests whether the resistance connected or not. However, it can be seen a worse prediction in the test 11, 12 and 13 of PCMs which are the test of vapor extraction, comparing with the other tests performed with PCMs. The effect of this detraction is not seen in the case of liquid nitrogen, where its low temperature level creates a much higher pressure difference, diminishing the effect of extracting from the vapor side of the prototype.

As discussed above, the Table 5 shows the mean results and confidence intervals of the error committed using as chilling medium Ice, PCMs vapor, PCMs liquid, LN2 vapor, LN2 liquid. Since the error is the variable under study, its average and confidence interval are sought to be as nearest to 0 as possible and of lowest range, respectively.

Case	Average	Standard deviation	Confidence interval (20)
	(g)	(g)	(g)
Ice	1.14	0.56	±1.40
PCMs liquid	-0.31	0.67	± 0.48
PCMs vapor	0.52	1.31	±3.25
LN2 liquid	0.20	0.63	±0.58
LN2 vapor	0.71	0.95	±2.37

Table 5: Mean results and interval confidence of the difference of the prediction and the initial mass

As shown in Table 5, results of liquid extraction are the most precise ones both for PCMs and for liquid nitrogen as the average and standard deviations are the nearest to cero. Also, it is important to mention that the results of error obtained for every case are relatively low, being lower as the temperature of the secondary fluid are reduced. With PCMs, the results are good enough for most common applications, as the confidence interval is smaller than the uncertainty of most of the scales used in these processes.

3.2 Uncertainty analysis

To enlighten the error committed in this experimental study, an uncertainty propagation study has been done.

The main variable to be studied is the difference between the refrigerant mass inside the BPHE and the theoretical amount of remaining refrigerant charge i.e., ε in equation (6). As mentioned before, in order to estimate the remaining refrigerant charge, the volume inside the BPHE must be known and this is done by the isothermal gas method. The volume of the BPHE is therefore 0.9±0.11 with a 2 σ confidence interval.

With the known uncertainty of the sensors and the volume, the average mean uncertainty depending on the cooling method used is calculated and shown in the Table 6

Table 6: Average uncertainty of each method employed.

Cooling method	Uncertainty
Liquid Nitrogen	±1 g
PCMs (-18°C)	±1.174 g
Ice	±1.628 g

As it can be seen, the impact of the uncertainty of the theoretical remaining refrigerant increase as the method used allows more refrigerant to stay inside the BPHE after the extraction. In the case of liquid nitrogen, almost all the uncertainty is caused by the uncertainty of the scales, which is $\pm 0.5g$ and there is needed 4 uses of this scale to have the final result. It is also noticeable that in this study, the uncertainty of the sensors used generates higher error than the method employed, consequently, if liquid nitrogen is selected due to its precision, the use of a more precise scale is highly recommended.

4. CONCLUSIONS

To sum up

- Different methods of refrigerant extraction have been studied theoretically and experimentally.
- The precision obtained with all studied methods is better than the ones obtained using the QOLM, and they also are even better compared to QOLM. However, they still have the problem of the time spent during the tests.
- If the required precision is not lower than a gram, using PCMs would be an option as it is no expensive and the error committed and the uncertainty are relatively low. Also, this method does not require additional safety measures.
- For studies where a higher precision is needed, it should be considered to use both liquid nitrogen and a better scale with more precision and less uncertainty. Using only one of these improvements would result in similar levels of total uncertainty.

NOMENCLATURE

Abbreviations:				
DHW	domestic hot water			
OLM	on-line measurement method			
QCVM	quick closing valves method			
QOLM quasi on-line measurement method				
RWT	remove and weight technique	•		
BPHE	brazed plates heat exchanger			
LN2	liquid nitrogen			
PCM	phase change materials			
Parameters:				
m	mass	(g)		
ρ	density	(kg/m^3)		
V	volume	(1)		
Е	precision			

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