

LEAK FLOW RATE MEASUREMENT OF MOBILE AIR CONDITIONING COMPONENTS WORKING WITH CO₂

D. CLODIC, Y. YU

Ecole des Mines de Paris, Center for Energy and Processes
60, boulevard Saint Michel – 75272 Paris Cedex 06, France

Phone: +33 1 40 51 92 49, Fax: +33 1 46 34 24 91, e-mail: denis.clodic@ensmp.fr

ABSTRACT

The Center for Energy and Processes-Paris (CEP) started to develop test benches for the measurements of refrigerant leak flow rates several years ago. For the last three years the laboratory has been measuring CO₂ (R-744) leak flow rates from various components: fittings, hoses, valves, and compressors. The measurement apparatuses are based either on infrared spectrophotometry or infrared photoacoustic spectroscopy.

The paper presents the variation of emissions as a function of both pressure and temperature. From those measurements emission laws can be elaborated.

1. THE ACCUMULATION METHOD

The Center for Energy and Processes (CEP) has more than 10 years of experience on the development of method of test and apparatuses for the measurements of leak flow rates in realistic conditions for different refrigerants: R-134a and now CO₂ (R-744) [Clodic D. and F. Fayolle, 2001, Clodic D. and M. Ben Yahia, 2001, Torbey N. and D. Clodic, 2002].

As indicated in Figure 1 the test method for the measurement of leak flow rates can be divided into two families, either by setting the tested component under pressure or by setting the tested component under vacuum. For simulations of realistic conditions of mobile air conditioning system operation, the component or the system has to be set under pressure and preferably at different temperatures.

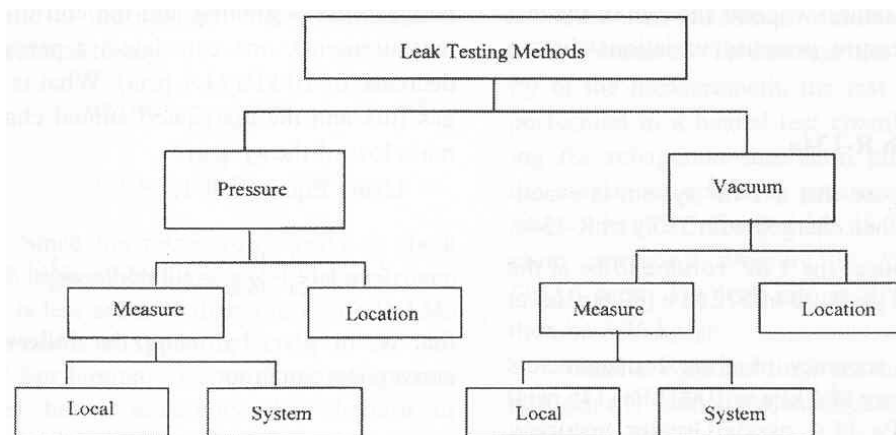
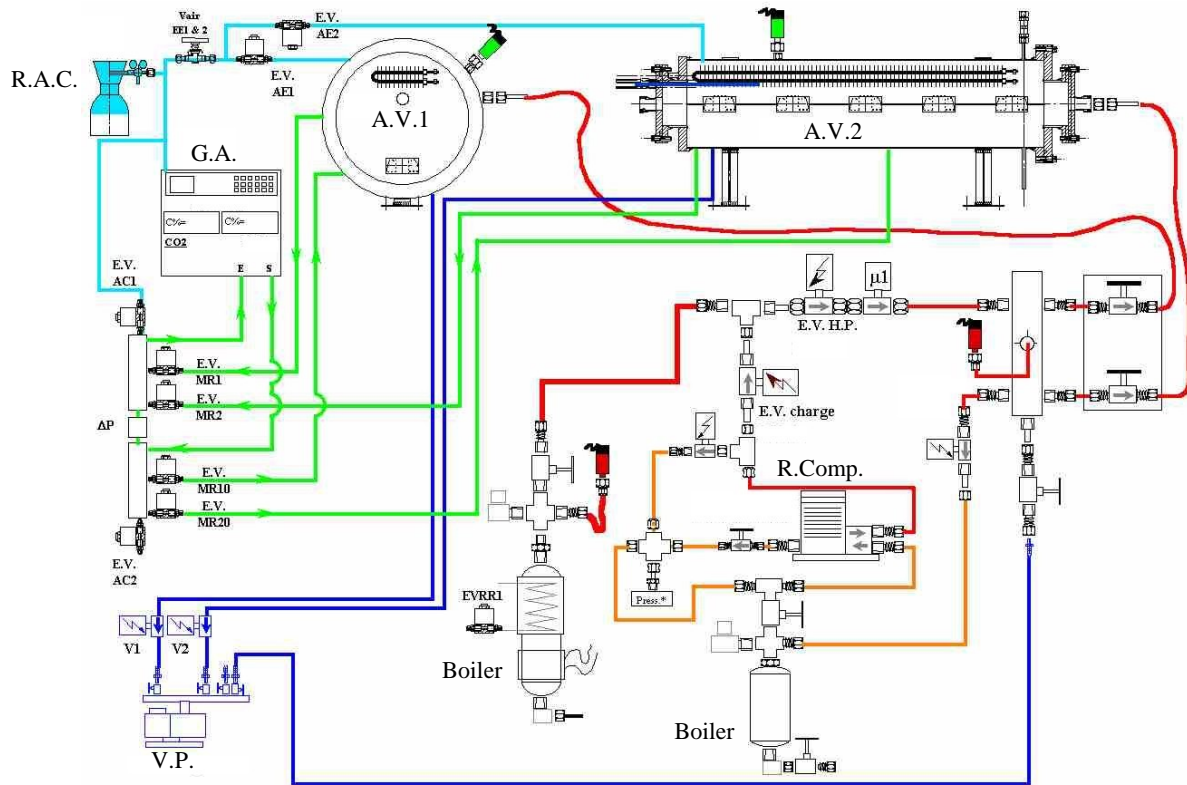


Figure 1 -Testing methods for leak tightness control, (ASTM, 2004).

In order to reach realistic conditions of measurements including presence of oil, and possible interaction between the refrigerant (CO₂) and elastomer material, the working fluid is preferably used as a tracer gas.

The accumulation method consists in installing the tested component inside a hood and measuring the raise of concentration of the working fluid (here CO₂) in the hood. The accumulation method presents the possibility of setting the accuracy of the leak flow rate measurement according to the measurement duration. The longer the measurement, the higher the accuracy providing that the leak flow rate is in steady and that all testing conditions are constant. Based on those principles, test benches for components and systems have been developed according to the lay out presented in Figure 2.



R.A.C.: Reconstituted air cylinder, R. Comp.: Recovery compressor, V.P.: Vacuum pump, A.V.: Accumulation volume

Figure 2. CEP test bench lay out.

Figure 2 shows two accumulation volume (AV1 and AV2). Fans are installed in each of them in order to reach homogeneous concentration rapidly. An electrical resistance allows to control the temperature inside the volume. Each accumulation volume is connected to:

- a vacuum pump (VP) to evacuate the accumulation volume down to 1 Pa abs.
- a cylinder of reconstituted air (N₂ + O₂) in order to fill up the accumulation volume with only those components (CO₂ and H₂O concentrations are nil); the internal pressure inside the accumulation volume is usually set just above the current atmospheric pressure;
- a gas analyzer (G.A.) with a continuous circulation between the accumulation volume and the analyzer.

The component installed inside the accumulation volume is connected to a CO₂ boiler allowing to control the pressure inside the component under test.

At the beginning of the test the CO₂ concentration in the accumulation volume is equal to zero, and so the current atmospheric concentration has no influence on the measurement. Moreover, the water vapor concentration is also nil, which avoids any compensation for the measurement by infrared technologies. Heating or cooling the boiler leads to control the CO₂ pressure inside the tested component and the heating system inside the accumulation volume allows fixing the temperature independently of the pressure. The continuous monitoring of the concentration allows fixing the accuracy and the measurement duration.

2. CONCENTRATION MEASUREMENT BY INFRARED SPECTROSCOPY

Currently two technologies are available for the measurement of CO₂: infrared spectrophotometry (Figure 3) and infrared photo-acoustic spectroscopy (Figure 4). Both methods are based on the absorption of infrared energy on characteristic wave lengths. Both apparatuses have an infrared source.

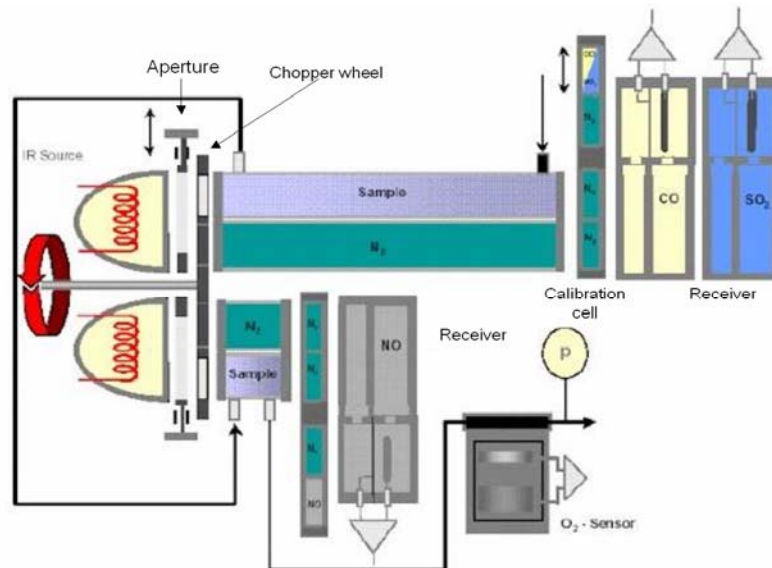


Figure 3 - Infrared spectrophotometer (by courtesy of ABB).

The URAS 14 photometer compares the energy going through the volume filled with a non absorbent gas (nitrogen) to the cell where the sample of the gas (N₂ + O₂ + CO₂) coming from the accumulation volume. The difference of the signal between the two measurement cells is analyzed by a sensor (diaphragm condensator). The infrared energy absorbed by the sample is proportional to the CO₂ concentration in the sample cell. The sensitivity of the apparatus is of 0.5 ppm.

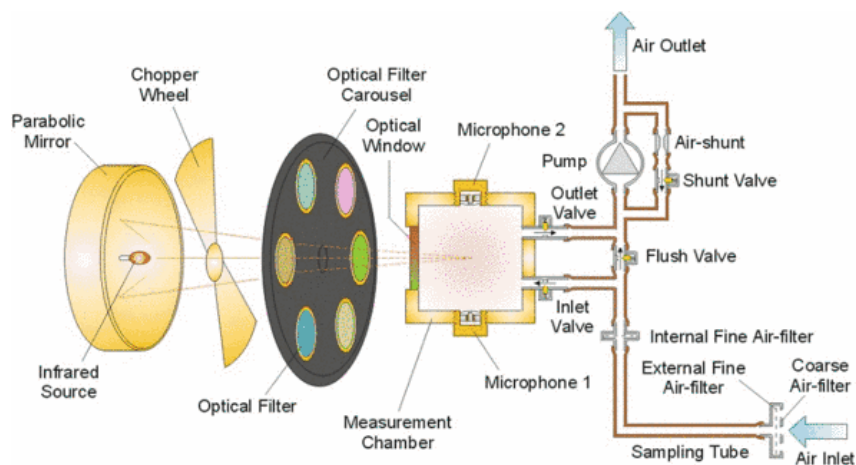


Figure 4 - Infrared photo-acoustics spectroscopy (by courtesy of INNOVA).

The infrared photo-acoustic spectroscopy is based on the conversion of the infrared energy in acoustic energy detected by a microphone. In the Innova 1314 concept an optical filter narrows the infrared wave lengths according to the infrared spectrum of the substance to be detected. The sample coming from the concentration volume is heated by the infrared energy leading to a raise of temperature and pressure in the measurement chamber (see Figure 4). The pressure generates an acoustic wave detected by the microphone. The sensitivity of this apparatus is of 15 ppb.

Using those apparatuses in the test bench shown in Figure 2 leads to a continuous measurement of the raise of the CO₂ concentration in the accumulation volume for various pressures and temperatures. Doing so it is possible to establish behavior laws for leak flow rates of components and systems (Clodic D. and L. Palandre, 2005).

3. CALIBRATION OF THE TEST BENCH

The uncertainties are calculated based on the relationship between the measured value and the CO₂ leak flow rate, which are established by eq. (1) and (2).

$$\dot{N} = \frac{V_{\text{acc}}}{V_{\text{mol}}} \frac{\partial C_{R-744}}{\partial t} \quad (1)$$

$$\dot{m} = \dot{N} \cdot M \quad (2)$$

Eq. (1) allows to establish the molar flow rate \dot{N} using the variation of the CO₂ concentration along time multiplied by the ratio of the accumulation volume to the molar volume. Then this molar leak flow rate is converted into the mass leak flow rate using eq. (2).

The total uncertainty of the leak flow rate is a combination of 4 uncertainty parameters: V_{acc} , T_{amb} , P_{amb} , and $\frac{\partial C}{\partial t}$. T_{amb} and P_{amb} are necessary for the calculation of V_{mol} but the uncertainties are small. The main uncertainties of measurements are related to the precision of the measurement of the accumulation volume V_{acc} (including the sampling circuit), and also the uncertainties of $\frac{\partial C}{\partial t}$.

The calibration process for the assessment of the uncertainties on the accumulation volume V_{acc} includes the following steps.

- Calculation of the accumulation volume
- Detailed calculation of all volumes installed in the accumulation volume.

Those two steps lead to the calculation of the free volume (V_{acc}) in eq. (1). V_{acc} is the volume where the CO₂ concentration is raising along the time.

- Standardization of the infrared analyzer with standardized gas concentrations; those gas concentrations are available with the guarantee of ± 1 ppm (ISO 6142, 2001). Several concentrations are used to verify the offset and the linearity of the infrared spectroscope. This calibration process depends on the apparatus and is typically done every six months.

- Calibration of the calibrated leaks.
Calibrated leaks are available (Figure 5) and have to be first calibrated in a reference accumulation cell, which volume is accurately known. For a fixed temperature inside this accumulation volume, the leak flow rate of the calibrated leak is established.



Figure 5 – Calibrated leaks.

- Verification of the concentration measurement inside the accumulation volume by the calibrated leak. After calibration, the calibrated leak is installed inside the accumulation volume for the verification of the free volume (V_{acc}) as calculated. During the calibration process Eq. (1) is used not to calculate the molar flow rate but to calculate V_{acc} . The least square

method is used for determining $\frac{\partial C}{\partial t}$ by minimizing the sum of squared residuals. The standard error of the regression is written as:

$$s_{\text{regression}} = \sqrt{\frac{\sum_{i=1}^n (C_i - \hat{C}_i)^2}{n-2}} \quad (3)$$

Therefore, the standard error of the slope ($\frac{\partial C}{\partial t}$), which should not be confused with the standard error of the regression ($s_{\text{regression}}$), is calculated as follows:

$$s_{\text{DC}} = \sqrt{\frac{s_{\text{regression}}^2}{\sum_{i=1}^n (t_i - \bar{t})^2}} \quad (4)$$

Eq. (4) shows that a high degree of variation of t_i makes for a small standard deviation s_{DC} .

The study focuses on the number of required samples of concentration measurements. A $s_{\text{regression}}$ value of 0.05 ppm is chosen by experience.

The combined standard uncertainties of the mass flow rate is written:

$$\frac{u(\dot{m})}{\dot{m}} = \sqrt{\left(\frac{u_{T_{\text{amb}}}}{T_{\text{amb}}}\right)^2 + \left(\frac{u_{P_{\text{amb}}}}{P_{\text{amb}}}\right)^2 + \left(\frac{u_{V_{\text{acc}}}}{V_{\text{acc}}}\right)^2 + \left(\frac{u_{\text{DC}}}{\text{DC}}\right)^2} \quad (5)$$

where $\text{DC} = \frac{\partial C}{\partial t}$

According to this analysis, the contribution of the accumulation volume to the overall uncertainty is by far the largest. Figure 6 shows the relative uncertainty of the mass flow rate with regard to the required test duration.

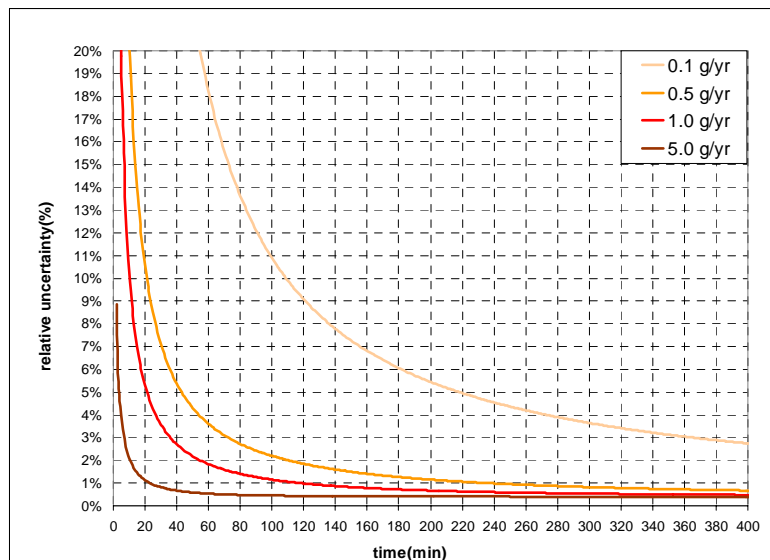


Figure 6 - Relative uncertainty vs. required measuring time.

4. RESULTS OF TESTS AND UNCERTAINTIES

Figure 7 shows two curves: one is the raise of CO₂ concentration inside the accumulation volume (to be read on the right scale), and the other one is the annual leak flow rate (read on the left scale). After 20 minutes, the leak flow rate is in steady state, the measurement is performed every second.

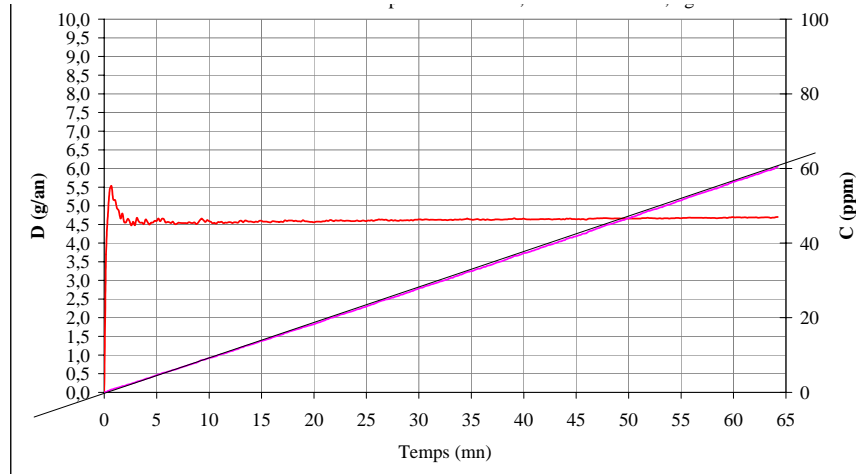


Figure 7 – Concentration variation and leak flow rate.

2,400 elementary measurements of the concentration are used to establish the single value of the annual leak flow rate. Taking into account eq. (5), the leak flow rate is of: 4.6 g/yr \pm 0.04. This example shows the general methodology and the uncertainties. The smaller the leak flow rate, the longer the measurement duration for reaching a given level of uncertainty.

Table 1 shows a typical series of tests carried out for three different pressures and for two temperatures at 50 bars and 100 bars.

Table 1 – Measurement of leak flow rates of a fitting.

Tests	Pressure (bar abs.)	Temperature (°C)	Annual leak flow rate (g/yr)
1	50	50	6.1
2	100	50	10.8
3	100	120	250.0
4	76	30	2.0
5	50	31	1.1

From Table 1 different trends can be derived. For tests 1 and 2, CO₂ is either superheated or in supercritical state but at 76 bars and 30°C, the conditions are just saturated and so liquid has appeared. Due to the significantly higher viscosity of the liquid, the leak flow rate at 76 bars and 30°C is lower than the leak flow rate at 50 bars and 50°C (see Figure 8).

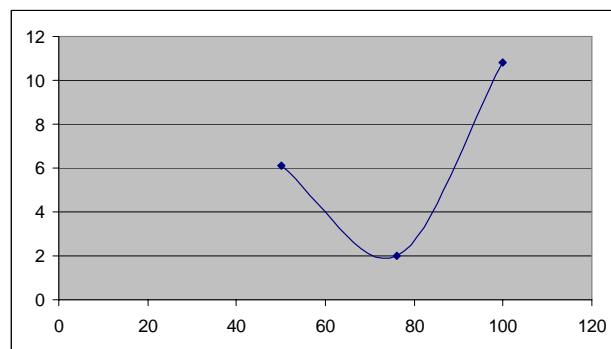


Figure 8 – Variation of leak flow rate according to the

pressure and temperature.

Complementary tests (not shown here) have been done at 76 bars and 40°C and the leak flow rate is significantly higher (around 8 g/yr).

Figures 9 and 10 show the very significant impact of temperature at constant pressure.

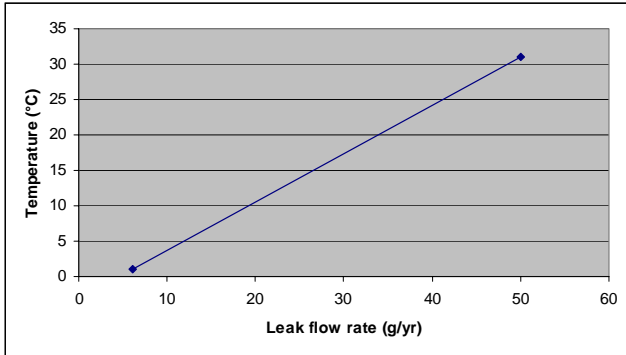


Figure 9 – Leak flow rate at constant pressure (50 bars) and two temperatures.

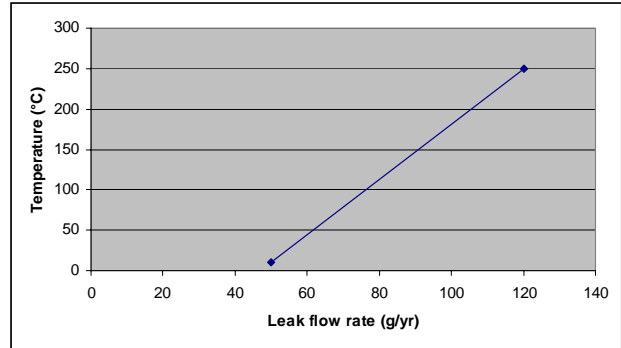


Figure 10 - Leak flow rate at constant pressure (100 bars) and two temperatures.

At 50 bars, the saturating temperature of CO₂ is of 14.3°C so the tests at 31 and 50°C are in superheated conditions. The only variation of 19 K leads to a variation of the leak flow rate from 3.1 to 6.1 g/yr. For 100 bars, when the temperature varies from 50 to 120°C, the leak flow rate varies from 10.8 to 250 g/yr. This last value is non acceptable, but the trend is interesting leading to a necessary systematic study of CO₂ leak flow rates as a function not only of the pressure but also of the temperature, whether in sub-critical conditions or super critical conditions.

5. CONCLUSIONS AND PERSPECTIVES

Leak flow rates measurement have been improved in the last ten years by the development of infrared measurement techniques which allow to measure the leak flow rates in realistic conditions with the working fluid itself used as a tracer gas. The accumulation of the tracer gas in the accumulation volume where the component or the system under test is installed leads to the possibility of using the measurement duration to fix a specified level of uncertainty. Tests performed at the CEP on components designed for the use of CO₂ as a refrigerant in mobile air conditioning systems have shown a significant sensitivity not only to the pressure, which is known for all refrigerants, but also to the temperature at constant pressure. Complementary work is on going in order to elaborate behavior laws for polymer materials as well as emission laws depending on the clearances between fittings and seals.

NOMENCLATURE

•	\dot{N}	the molar mass	(mol/s)	\bar{t}	the mean of all t_i
•	M	the molar mass	(g/mol)	V_{acc}	the volume of the accumulation volume (L)
•	\dot{m}	the mass flow rate	(g/s)	V_{mol}	the molar volume at the defined temperature (g/L)
	n	the number of the samples		$\partial C / \partial t$	the variation of concentration of R-744 in the volume (% v/v per second)
	t_i	the time of the measurement (s)		$\sum_{i=1}^n (C_i - \hat{C}_i)^2$	the sum of squared residuals

REFERENCES

ASTM E 432-71. 1984. Standard recommended guide for the selection of a leak testing method. American Society for Testing and Materials.

Clodic D., L. Palandre. 2005, Measurement of leak flow rates of mobile air conditioning systems, *Proc. SAE 2005, 2005-01-1507*

Clodic D., F. Fayolle. 2001, Test-Bench for Measurement of Leak Flow Rate of MAC Compressors, *Proc. SAE 2001, 01PC-183*.

Clodic D., M. Ben Yahia. 2001, New Test Bench for Measuring Leak Flow Rate of Mobile Air Conditioning Hoses and Fittings. *Proc. Earth Technology Forum. Baltimore, USA*.

ISO 6142. 2001, Gas analysis preparation of gas mixtures for standardization – Gravimetry method.

Torbey N., D. Clodic. 2002, Méthode d'étalonnage sur un étalon primaire des fuites calibrées permettant la vérification des performances des détecteurs de fuites des fluides frigorigènes. Report for ADEME (01 74 084/11243).