Fluorocarbon composition and leak rates monitoring in the RICH Fluids System

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Abstract

Due to detector aging, leaks have occurred between the two fluorocarbon systems (Liquid radiator and Gas radiator) which control the circulation of the Cherenkov emitting media for the RICH detectors of DELPHI. A *Vapour Pressure Cell* was installed in June 1998 to monitor the composition of the fluorocarbons. This provides a means to ensure that the data quality and the detector security would not be endangered by the mixing of the fluids. By using process parameters from the separation plant, this also gives an estimation of the leak rates between the two fluids systems and of their evolution. We report here on results and running experience from three years of data taking.

1 Introduction

The DELPHI experiment [1] is the only one of the four multipurpose experiments operating at the LEP collider to incorporate Ring Imaging CHerenkov (RICH) counters to perform particle identification. The Barrel RICH detector [2] is used for identification of charged tracks in the momentum range of 0.7-25 GeV/c and it covers polar angles between 40° and 140°. The detector uses two different fluorocarbons as Cherenkov emitting media. C_5F_{12} is used in the *gas radiator* and C_6F_{14} is used in the *liquid radiator*. Media in two different phases are used to provide a larger active range. It has been discovered that these fluids interact with the composite materials inside the detector, inducing shrinks and swells which give rise to leaks between the two initially separated fluids systems. A mixing of the Cherenkov media influences the data quality since it causes variations of the refractive indexes. It also endangers the detector security, since a mixing above certain threshold values could lead to condensation in the gas radiator and boiling in the Liquid radiator. This scenario lead to the installation of a distillation plant [3] in the Barrel RICH fluids systems in June 1996. The quality of the fluorocarbons has been stabilised but an additional measurement device was needed in order to secure data quality and prevent detector harm. There was also a need to monitor the evolution of the leak rates between the radiators. To achieve this, a *Vapour Pressure Cell* was installed in June 1998; the device takes samples alternatively from the liquid radiator and the gas radiator, and measures the composition of these binary mixtures. The measurement is based on a method to monitor the composition of fluorocarbons developed earlier by the RICH group at DELPHI [4], [5]. The design and commissioning of the measuring device are described in more detail in [6].

2 The RICH Detectors

2.1 The Fluids System

For the performance of the detector the fluids quality needs to be maintained, with respect to UV-transparency from 160 to 220 nm and low contamination by oxygen and water. To achieve this, there are separate fluids systems for the gas and the liquid radiators. These systems clean the fluids and monitor their quality. For the gas radiator system, the fluorocarbon is stored in liquid form in a tank and then evaporated and injected in the radiator. The physical properties of the fluorocarbons used in the Barrel RICH detector are shown in table 1.

	Boiling point	Liquid density	Gas density (1 bar)	n at 7 eV
C_5F_{12}	28 °C	1630 kg/m^3	12.5 kg/m^3	1.00178 (Gas)
C_6F_{14}	56 °C	1682 kg/m^3		1.2827 (Liquid)

Table 1: Physical properties of the Barrel RICH Cherenkov media

With the installation of the distillation plant the two fluids systems became interconnected as shown in Figure 1. The return lines from both liquid and gas radiators lead to the *Mixing Tank*, where the two fluids are collected together. Then the mixture is
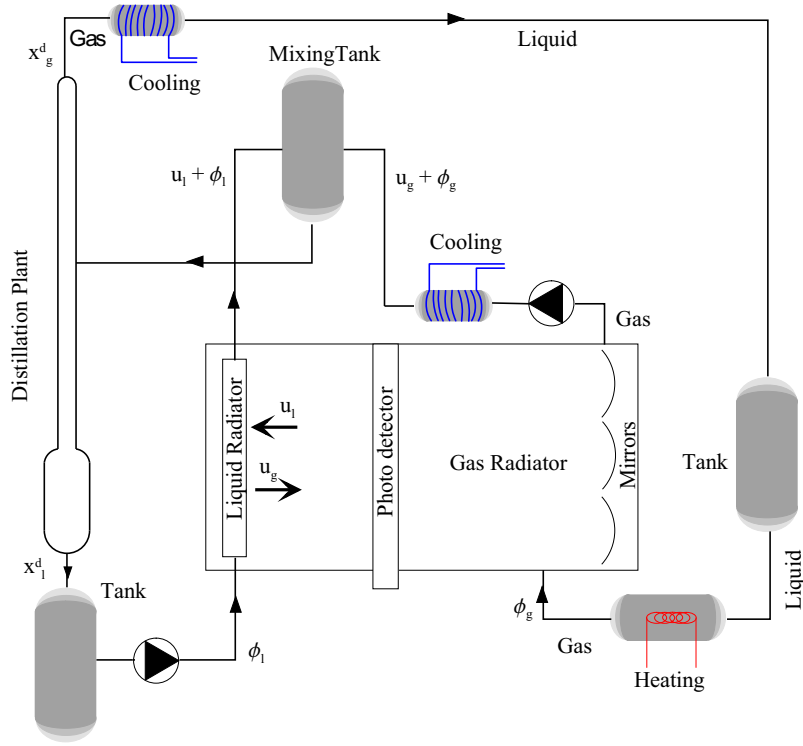
Figure 1: The Barrel RICH fluids systems with the distillation plant.

injected and separated in the distillation plant and finally transferred back to the gas (liquid) radiator system. The components for cleaning and monitoring the fluids are not shown in this schematic picture. The leaks between the two systems are considered to be constant over periods of the order of one day. This gives the possibility to make use of a simple mathematical model for the process of mixing and demixing. Each radiator system is considered as a constant volume V_i fed by the input flow ϕ_i with the purity x_i^d from the distillation plant and by a flow u_i due to leaks between the system. Furthermore, the volume is drained by the flow $u_i + \phi_i$ to the mixing tank. The symbols are defined in Figure 1, with i representing g (l) for the gas (liquid) radiator system. Under the assumption of perfect mixing the volume fraction is given by a first order differential equation. The model used is described in more detail in [6]. Since the parameters of the system are expected to be approximately stable in time, the *steady-state* solution shown in Equation 1 is considered.

$$x_i^{ss} = \lim_{t \to \infty} x_i = (\phi \cdot x_i^d)/(\phi_i + u_i) \tag{1}$$

The characteristic parameters of the systems at the time (1996) of the installation of the Distillation Plant are given in table 2. The purities of the fluids from the distillation plant were measured at the commissioning [3]. The steady state solutions given by these values are shown in Figure 2.

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	Purity x_i^d	Flow ϕ_i
Gas Radiator	0.981	276 Litres/day
Liquid Radiator	0.971	960 Litres/day

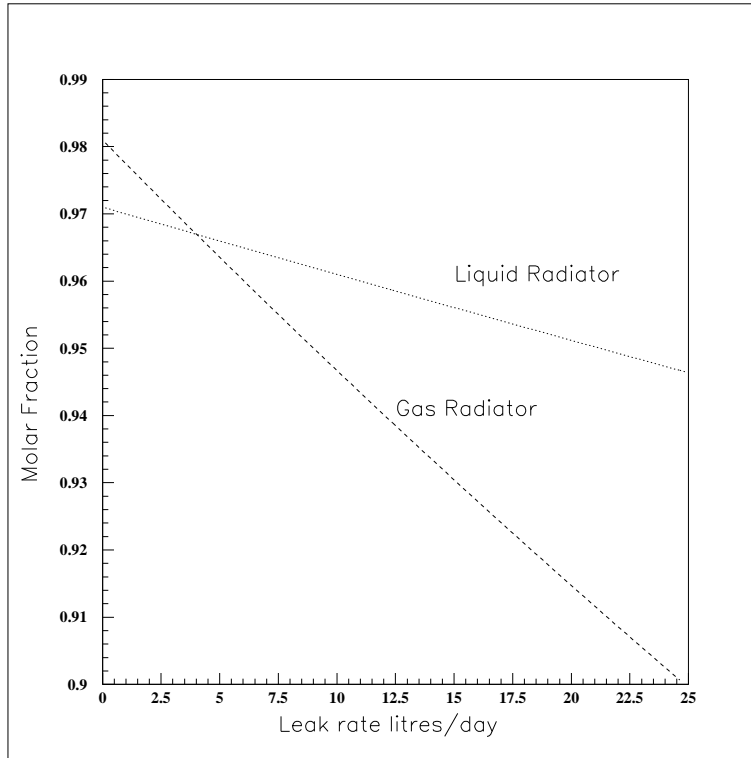Table 2: Parameters of the fluid systems in 1996

Figure 2: Purity of the fluorocarbons as a function of leak rates in the range between 0 and 25 litres per day.

2.2 The consequences of mixing

For the detector hardware security, a mixing above certain ratios would be extremely dangerous.

Mixing changes the vapour pressure of the fluids which could lead to boiling in the liquid radiator or condensation in the gas radiator. A phase transition of this kind would seriously disturb the flows in the fluids system and interfere with the pressure regulation. Phase transitions occur when the mixing ratios exceed 20% (50%) in the gas (liquid) radiator.

Mixing of the two radiating media leads to a change in their refractive indexes. The alignment of the detector at LEP II is based mainly on data taken at the Z^0 resonance peak in the beginning and in the end of the year. The band in Figure 3 shows the uncertainty of the refractive index estimated from the mean Cherenkov angle from data taken at the Z^0 peak. An attempt to estimate the refractive index using high energy data is illustrated by the points in Figure 3. The points represent the deviations from the mean value of the refractive index estimated using high energy data collected during a ten-day
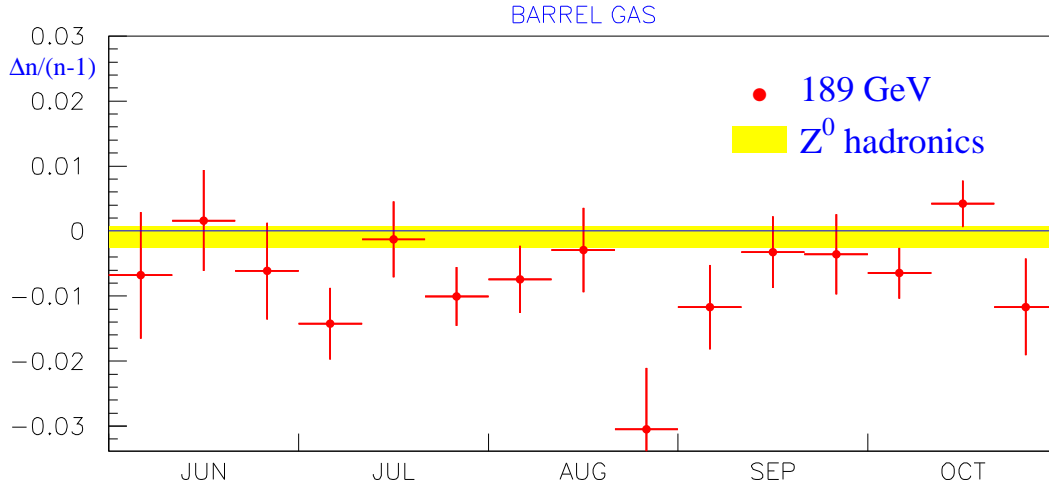
Figure 3: An attempt to monitor the refractive index of the Barrel RICH Gas Radiator during 1998 high energy run.

period. The electron-positron cross section at high energies is too low to give enough statistics to follow the time evolutions of parameters such as the refractive index. The statistical uncertainty of this method is illustrated by the variations between the points in the plot. Clearly, an alternative monitoring of the fluids quality is needed.

The emission angle (θ_C) of the Cherenkov photons from a particle passing through a medium is given by the formula

$$\cos \theta_C = \frac{1}{n\beta} \qquad (2)$$

where n is the refractive index of the medium and β is the velocity of the particle: variations of the refractive index influence the data quality. If these variations lead to a shift in the Cherenkov angle larger than 1 (2.5) mrad for photons produced inside the gas (liquid) radiator, that is, comparable with the resolution of the measurement of the Cherenkov angle for a track [7], the measured distributions of charged particles would be altered.

The refractive index of a binary mixture is given by

$$n = x_1^v \cdot n_1 + x_2^v \cdot n_2 \qquad (3)$$

where x_i^v is the volume fraction of the ith component. Thus a variation in the mixing ratio of the two components of Δx^v propagates as a shift of the saturated ($\beta = 1$) Cherenkov angle as

$$\Delta\theta_C = \frac{(n_2 - n_1) \cdot \Delta x^v}{n \cdot \sqrt{n^2 - 1}} \qquad (4)$$

where the n in the denominator represents the refractive index of the fluid of the mixture. This expression sets the upper limits of the acceptable deviations of the mixing ratios
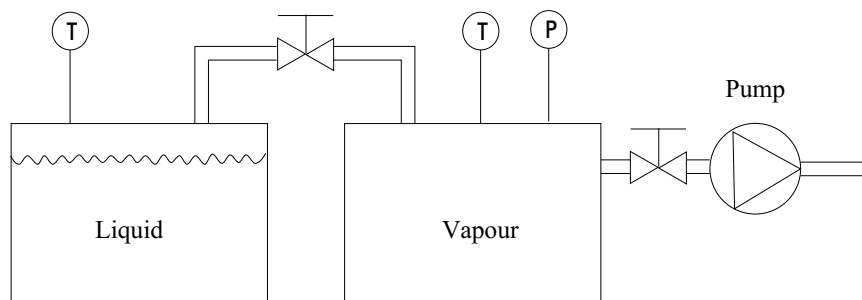
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Figure 4: Schematic layout of the *vapour pressure cell*.

from the values at the time of the Z^0 calibration run. Variations $\Delta x^v < 0.06$ (0.27) in the gas (liquid) radiator would not influence the data quality. Expressed in molar fractions, these limits are $\Delta x^m < 0.07$ (0.25) in the gas (liquid) radiator.

3 The Vapour Pressure Cell

3.1 Principle of Operation

A liquid enclosed in an evacuated volume evaporates. This evaporation builds up a pressure and after some time equilibrium is reached between the liquid and the gas phase. The equilibrium pressure is characteristic for each fluid and it depends only on the temperature. This pressure is called the *vapour pressure* and can be parametrised with good approximation over a large temperature range by the formula:

$$P_V(T) = P_0 \cdot e^{-W/T} \tag{5}$$

where T is the absolute temperature and P_0 and W are parameters characteristic of the fluid. For a mixture of two liquids A and B enclosed in an evacuated volume, the vapour pressure is described by *Raoult's law*

$$P_V(T) = P_V^A(T) \cdot x^A + P_V^B(T) \cdot x^B. \tag{6}$$

Here x^A and x^B denote the molar fractions of the liquid phases, and $x^A = 1 - x^B$. From the simultaneous measurement of the temperature and the vapour pressure of a binary mixture, the composition of the liquid phase is deduced.

The schematic layout of the Vapour Pressure Cell (VPC) consists of two cells (Figure 4): one cell containing the liquid and another that becomes filled with the vapour. These two cells are connected by a valve and a pump is connected to the vapour cell. A sample from either the gas or the liquid radiator is collected from the fluids systems after it has left the radiators and before it enters the distillation plant for separation. The flow form the selected system is diverted through the liquid cell before entering the mixing tank of the distillation plant. The cell is flushed for a certain time to assure the collection of a representative sample. Then the cell is left at rest for another time period to let the system reach thermal equilibrium.

To measure the vapour pressure, the vapour cell is first evacuated. Then the connection valve between the two cells is opened to allow the pressure to build up. After a certain
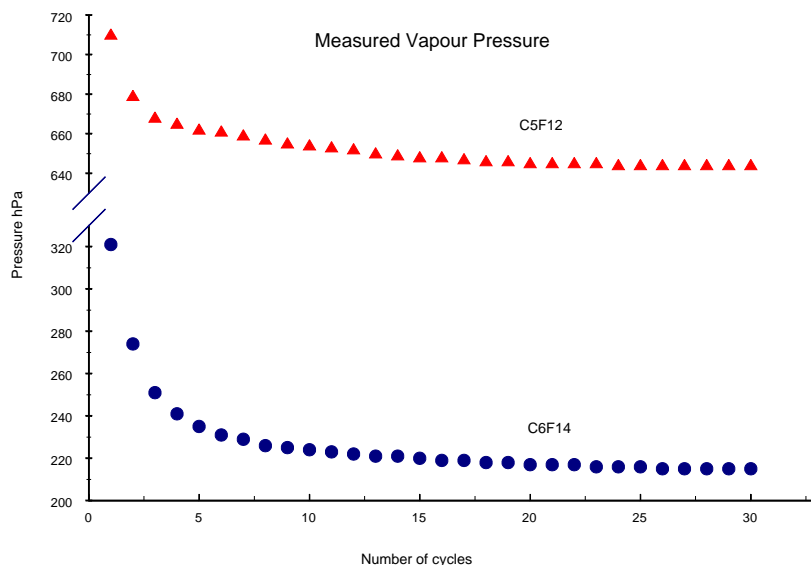
Figure 5: Measured vapour pressures for C_5F_{12} and C_6F_{14} during the degassing process.

time, the pressure is measured, together with the temperature of the liquid and the vapour. The molar fractions of the dual mixture are calculated from these measurements and from values from the literature of the vapour pressures.

The liquid sample is saturated with dissolved gases, mainly nitrogen. While the vapour pressure is formed, part of this gas evaporates and contributes to the measured pressure. This gives a considerable error in molar fractions. To extract the dissolved gases from the liquid, the vapour cell is pumped and refilled with vapour several times. After about twenty-five cycles, the measured pressure reaches a plateau and this is interpreted as a complete extraction of the dissolved gases (Figure 5). Due to the higher vapour pressure for the most volatile component, more C_5F_{12} than C_6F_{14} is extracted in each cycle. This changes the composition of the binary mixture and enriches the liquid in the less volatile component. The final measurement will therefore be done on a liquid with a different mixing ratio than the original. This error is however small, and it can be corrected for in the analysis of the data.

3.2   Integration in the Fluids System

The two cells of the VPC are made of two concentric cylinders with a lid and a bottom. The inner chamber is used as vapour cell and the outer chamber as liquid cell. This design was chosen to assure a good thermal contact between the two cells. The cell is situated close to where return lines from the gas and liquid Radiator system enters the mixing tank. Thus, it is possible to divert the flow of one of the return lines through the VPC before it enters the mixing tank. The cell is flushed with liquid for two hours to make sure that the collected sample is representative for the mixture in the vessel. Then the VPC is left at rest for one hour to smoothen out temperature gradients, before the cyclic measurement process is performed.

The measurement cycle is controlled by a Programmable Logical Controller (PLC). It
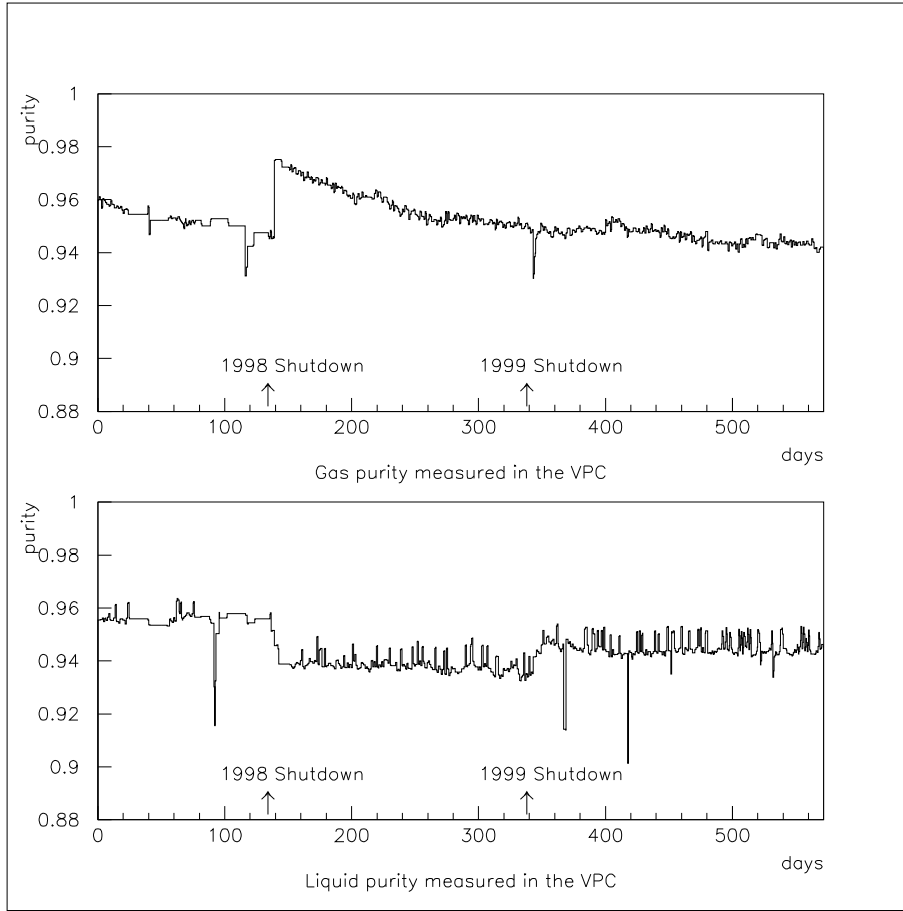
Figure 6: Purity of the Cherenkov media as measured in the Vapour Pressure Cell.

is performed once a day for each of the two systems. Pressures and temperatures are read by the PLC and further on transmitted via the RICH fluids network to DELPHI's VMS cluster. The data is analysed and presented by software running on the Alpha stations.

4   Measurements

4.1   Results

The fluids composition at the outlet of the radiators has been measured with the method described in Chapter 3 during three years of data taking. The molar fraction of C_5F_{12} in the gas radiator and the fraction of C_6F_{14} in the liquid radiator are shown as a function of time in Figure 6. The composition of the fluids is influenced by the performance of the distillation plant and by the leak rates between the two radiators as discussed in Chapter 1. The relation between the input purities to the radiators and the leak rates is given by Equation 1. To estimate the leak rates between the radiators it is necessary to monitor the output purities from the distillation plant. Two important components of the distillation plant are the boiler, where the liquid phase is extracted and the condenser, where gas phase is extracted. The purities of the extracted fluids are calculated from the temperature and absolute pressure of the condenser and the boiler, which act according to

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the principle of a vapour pressure cell as described in Chapter 3. The N_2 dissolved in the fluids is estimated to contribute with a partial pressure of 4 kPa in the condenser and 1.5 kPa in the boiler. The results of these measurements are shown in Figure 7 for the three years of running. The steps between consecutive years wich can be seen in both Figures 6 and 7 are due to the different flows in the liquid radiator system. The C_5F_{12} flow is driven by the pressure inside the gas radiator which is automatically set and mantained, while the flow inside the liquid radiator is set via hand valves at the beginning of every run period, and it can change substantially from year to year. The values of the flow inside the liquid radiator are shown in table 3.

Year	Liquid Radiator flow (l/day)
1998	745
1999	936
2000	768

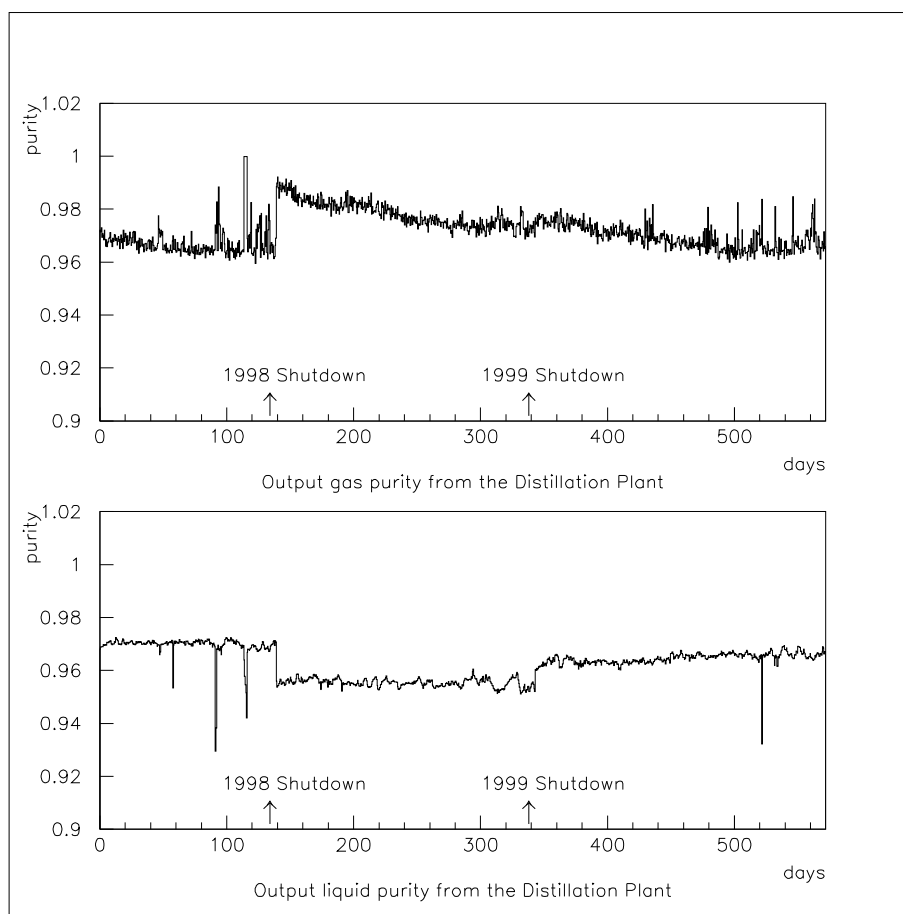Table 3: Flow through the liquid radiator during the years

Figure 7: Purity of the Cherenkov media after the separation process and before the reinjection into the radiators.

The higher liquid flow in 1999 corresponds to poorer performance of the distillation
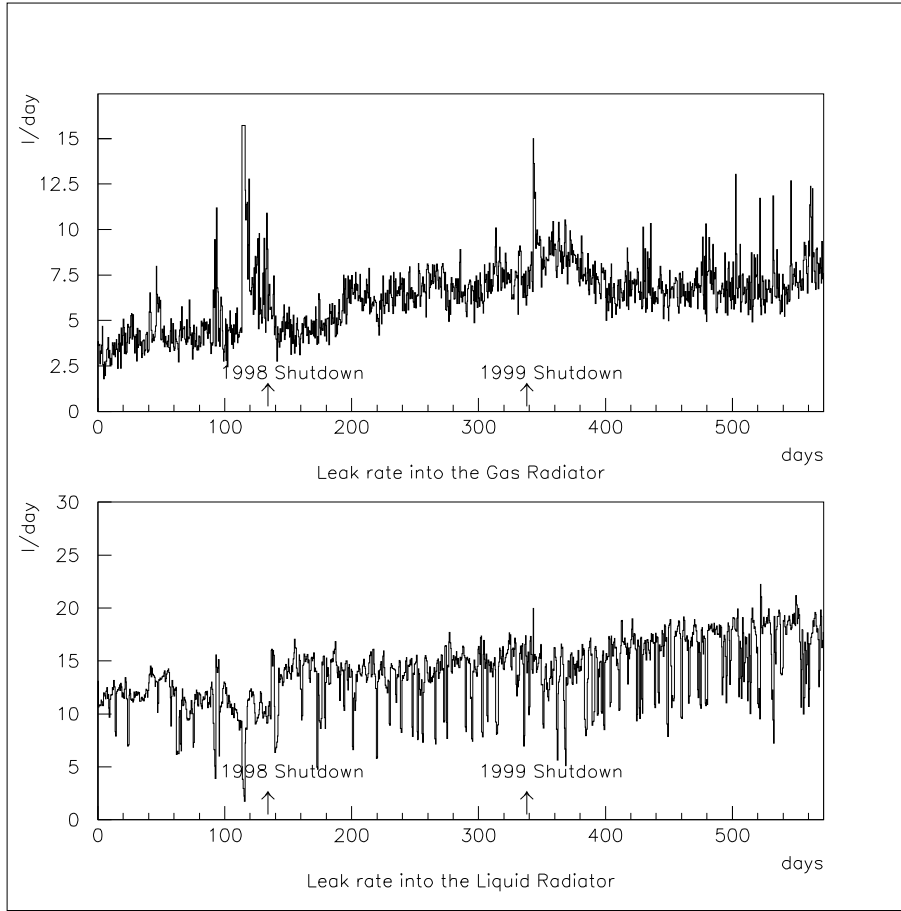
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Figure 8: Estimated leak rates between the two radiators.

plant in evaporating the C_5F_{12} dissolved in the mixing tank, since it had to handle more fluid with respect to the previous year. The leak rates can be estimated from the results presented in Figure 6 and in Figure 7 via Equation 1. The result can be seen in Figure 8. When calculating the leak rate inside the liquid radiator it has been assumed that inside the range of interest the leak rate is proportional to the flow in the radiators, and the results shown are normalised to the year 2000 flow. The assumption seems reasonable from a theoretical point of view, because a bigger amount of C_5F_{12} can be dissolved at the same pressure and temperature in a bigger amount of C_6F_{14}. Also, the obtained curve does not show dramatic steps between consecutive years, so that we have a more realistic estimation of the evolution of the leaks.

There are two features visible in the data from 1998, around day 100: a drop in molar fraction for the liquid radiator and one for the gas radiator. The perturbation for the liquid radiator was due to a faulty anti-return valve in the distillation plant. This had the effect of pushing C_5F_{12} into the liquid radiator system. For the gas radiator, the deviation was caused by a faulty three-way-valve preventing the fluid from being distilled for a few days. Consequently, the material was contaminated by C_6F_{14} leaking inside the gas volume.

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4.2　Limitations of the measurement

Errors are propagated from uncertainties in the reading of the pressures and the temperatures. The uncertainty in temperature is assumed to be ± 0.2 °C and in pressure ± 0.3 kPa. This gives an absolute uncertainty of approximately 1.5 %-units in molar fraction. An explicit calculation of these errors is given in [6].

There are two systematic errors whose numerical values are difficult to estimate. The numerically calculated value for the vapour pressure coincides well to the point to which it has been fitted. However, the errors of the intermediate points are unknown. Since the measurements are done within a small temperature range, this error should have the character of an offset. This have been corrected with calibration measurements on liquids with known compositions. The uncertainty in molar fraction should be less than 1 %-unit.

The residual partial pressure from dissolved nitrogen is not fully understood. Measurements with a large number of pump cycles indicate that there is still a contribution to the pressure after the normal 35 pump cycles. This uncertainty should be constant in time and only influences the absolute value of the estimated molar fractions. Therefore, this does not affect the monitoring of time variations in the composition. Assuming a constant residual partial pressure of 1.5 kPa gives reasonable results in agreement with simulations.

In the estimation of the output purities from the distillation plant the main uncertainty is the magnitude of the partial pressure of the dissolved Nitrogen. However, this only affects the calculated absolute values of the leak rates, not their estimated evolution in time. The values chosen for the residual N_2 pressure in the Cherenkov media result in reasonable values for the leak rates. The important parameter to monitor for the hardware integrity of the detector is the time evolution of the leak rates.

5　Conclusions

In June 1998 a vapour pressure cell (VPC) has been implemented in the fluids system of the DELPHI Barrel RICH detector in order to monitor the quality of the Cherenkov media as well as to follow the trend of the leak rates between the liquid and the gas radiators. Three years of running have shown that the VPC measurements are reliable and strongly correlated with the performance of the fluids separation device, the distillation plant. The quality of the fluids is not stable during the years nor from one year to the other, but the variations are well within the tolerable limits as far as data quality is concerned. The leak rates between the radiators, deduced by comparing the fluids compositions in the VPC and in the distillation plant, that is, before and after they have undergone the separation process, have increased in both systems, going from 3 to 7 l/day in the gas radiator and from 12 to 18 l/day in the liquid radiator. Nevertheless, thanks to the good performance over the years of the distillation plant, the contamination levels inside the radiators have always been well below the respective thresholds for hardware danger, namely condensation inside the gas radiator and boiling inside the liquid radiator.

Acknowledgements

References

[1] DELPHI Coll., P. Abreu et al., Performance of the DELPHI Detector, Nucl. Inst. Meth. A378 (1996) 57-100.

[2] E.G. Anazzontsis et al., The Barrel Ring Imaging Cherenkov Detector of DELPHI, Nucl. Inst. Meth. A323 (1992) 351-362.

[3] C. Joram, The Distillation Plant of the DELPHI Barrel RICH Detector, DELPHI 98-53 RICH 94 (1998).

[4] H. Fürstenau et al., Mixing and unmixing of fluorocarbons in the Barrel RICH fluids system, DELPHI 95-46 RICH 69

[5] C. Joram et al., Monitoring of the Composition of the RICH Fluids with Vapour Pressure Cells, DELPHI 98-63 RICH 93 (1998).

[6] L. Eklund, Monitoring the composition of the Cherenkov radiating fluids at DELPHI's RICH detectors. Linköping University, LiTH-IFM-Ex-773. (1998).

[7] W.Adam et al., Alignment and Cherenkov angle resolution of the DELPHI RICH. Detector, Nucl. Inst. Meth. A371 (1996) 228