FERMILAB-TM-2146 July 2001

Fermilab

April 16, 2001

Flammability Tests on D0 Run II Muon PDT Gas and P-10 Gas

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We have done a series of measurements with mixtures of Argon, CF4 and CH4 to demonstrate that the mixture chosen for RunII (84%Argon, 8%CH4, 8%CF4) is not flammable. The tests were conducted in the Meson Detector Building in a test cell similar in construction to a cell of a Muon PDT. In order to establish the viability of our test set-up, we first repeated the demonstration that P-10 gas (90% Argon, 10% CH4) is in fact flammable, contrary to the classification by the U.S. DOT.
U.S.DOT regulation 173.115 defines "flammable gas" as:

(1) Is ignitable (at 14.7 psi) when in a mixture of 13% or less with air: or
(2) Has a flammability range (at 14.7 psi) with air of at least 12% regardless of the Lower Explosive Limit (LEL).

P-10 has a LEL of about 40% and a flammability range of about 10%, so P-10 is not flammable according to the U.S. DOT definition. The point here is that the DOT classifications are to serve the DOT's function to ensure transportation safety, and are not necessarily appropriate for other situations.

The first configuration of our test cell, however, apparently failed to ignite P-10. With the guidance of Bill Nuttall of CERN, we modified our test cell to make it more like the standard flammability testing setups, with a large viewing window and a spark gap in the middle of the cell. In this second configuration P-10 was easily and reliably ignitable. After becoming more familiar with the visible indicators of combustion of P-10 (water vapor cloud formation, pressure changes and gas venting) we re-tested with the initial configuration, and found that the mixture actually had been burning, and that we had just missed all the indications. The data from CERN showed that P-10 burns rather slowly, with about a one second rise time for the pressure to reach the maximum of four atmospheres overpressure. In our tests we saw no signs of any flame, but only a water vapor cloud. (Some preliminary tests with the same cell using Argon-Ethane and air had a much more impressive burn, with rapid venting and a red flash clearly visible.)

Our test cell (Figure 1) is a box 1.5"x4"x36" inside dimension. The entire top plate is spring loaded and lifts with about 0.5 psi. This is a large enough vent (4"x36" to prevent any large pressure build up in the cell. There are several clear plastic viewing windows that allow the region of the spark gaps to be observed. The spark for ignition comes from a Droege High Voltage Supply with a parallel capacitor on the output. When the breakdown occurs, the capacitor is discharged into the ionized gas between the electrodes. Most of the energy, however, is dissipated through resistive heating of the wiring. We are able to estimate how much of the spark's energy is deposited in the gas with a tiny gas calorimeter (Figure 2). The calorimeter is made from an air filled plastic syringe into which we have inserted two copper rods to serve as a spark gap. To make the measurement, we hold the nose of the syringe under water and turn on the spark. The air gets heated by the spark and some of it bubbles out through the nose of the syringe. When the gas cools, some of the water is sucked back into the syringe. By measuring the amount of water in the syringe we can calculate a lower bound on the energy deposited by the spark into the air. With our largest capacitor (0.1 microF.) and our highest voltage (7.5kV), we estimate that 100 milliJoules is deposited in the gas by the spark. This is a small fraction of the stored energy of the capacitor (2.8 J). We use a series of capacitors to measure the minimum energy required to ignite the gas under study. We also use a gas chromatograph to complement our visual observations. We can measure the initial mix and the combustion products and determine the amount of fuel consumed.

The usual test procedure is to connect an open 500 cc. sample bottle to the gas outlet port of the test cell, and purge the cell gas through the sample bottle for several volume changes. The cell volume is 3.5 liters and we purge with a flow rate of about two liters per minute. The sample bottle is then valved off and removed, and another sample bottle (under vacuum) is attached to the test cell. Yet another bottle (under vacuum) is attached to the opposite end of the test cell so that we can sample both ends after

ignition. The inlet gas port is then valved off and the High Voltage source is turned up until there is a spark. We note the voltage of the supply when the spark occurs. Immediately after ignition, valves to the two sample bottles are opened to draw gas from the test cell. The valves are then closed again and the bottles removed to bring them to our gas chromatograph for analysis. When P-10 is ignited we can see a vapor cloud in the viewing window for a fraction of a second, and we can hear the spring loaded lid to the test cell lift and vent some gas. The lid re-seals very quickly and the pressure in the test cell goes sub-atmospheric as the gas cools and the water vapor condenses. Even if there were no venting, the pressure would go sub-atmospheric just from the condensation of the water vapor that is made during combustion.

$$CH4 + 2O2 \rightarrow 2H2O + CO2$$

Since the burning of P-10 mixed with air is not nearly so dramatic as burning Argon-Ethane with air, we were a bit surprised to find that the burning the P-10 mix was a complete burn that consumed almost all the Methane in the test cell. Figure 3 shows the gas composition before ignition. The peaks for N2, Argon and CH4 are prominent in the figure. The Oxygen peak is the shoulder on the left side of the Argon peak. (The positions of all the peaks have been established by running each gas separately through the chromatograph.) Figure 4 is a sample taken after ignition. The Oxygen shoulder is almost completely gone and the Methane peak has been reduced to shadow of its former self. Figures 5 and 6 show the same "before" and "after" samples, but with a longer x-axis to pick out the components that take longer to get through the chromatograph column. In the "after" plot, a peak for CO2 and a peak for H2O have appeared. The ignition energy threshold for P-10 in our setup was about one milliJoule. We used a 0.001 microFarad capacitor in parallel with the High Voltage Supply. A smaller capacitor would not initiate the burn. We also used a 0.1 microFarad capacitor to ignite P-10 mixed with air, but there was very little difference in the burn. We varied the mix of air and P-10 and measured the flammability range to be from 44% air to 54% air when measured by floating ball flow meters with no correction for gas density.

The mix of 80/10/10 Argon/CH4/CF4 barely burned at all. The test cell lid did not lift and there was only a wimpy vapor cloud near the spark. However, we could demonstrate that some combustion was occurring by opening a ¼" copper tube vent and draping a flexible plastic flag over the vent. When the mix was ignited, the flag flapped as some gas vented through the copper tube. We also took samples after the spark to examine with the chromatograph. Figure 7 shows the gas components after ignition, with a tiny amount of CO2 and H2O visible. Figures 8 shows the same data re-plotted with an expanded vertical scale to make the CO2 and H2O more prominent. Both of these plots are for a sample taken near the spark. Figure 9 shows the sample taken at the far end of the cell, away from the spark. Here there are much smaller percentages of H2O and CO2. We deduce from this that the combustion does not propagate far from the ignition source for this gas mixture, at least in the horizontal orientation. For most purposes, this partial burning would not fit the criteria for "flammable", inasmuch as the combustion stops before all the fuel is consumed. The capacitor used to ignite the 80/10/10 mixture was 0.028 microFarads, 28 times larger than the capacitor used to ignite the P-10 mixture. This corresponds to about 28 milliJoules ignition energy. The range of flammability was from 47% air to 51% air, using the same flow meters described above.

Our tests with 84/8/8 showed no signs of even partial burning. There were no puffs of gas coming out of the vent tube, even for the largest capacitors. There were no signs of vapor clouds in the windows. We then mixed the 80/10/10 with 84/8/8 to make 82/9/9. This was then mixed with air and put into our test cell. There were no hints of combustion with this mix either.

Our final tests were with the apparatus in a vertical position. The 80/10/10 mixed with air burned a bit more emphatically in this orientation. We could hear the gas escaping through the ¼" vent after ignition, as well as see the vent flag flutter. We took samples from both ends of the cell after ignition and looked at them with the chromatograph. The bottom end (near the spark) showed about the same amount of CO2 as we had found in the near end of the test cell, in the horizontal configuration. The top end (far end) also showed about the same amount of CO2, whereas in the horizontal orientation there was barely a trace. It appears that the combustion does propagate vertically but still only consumes a small amount of the methane available.

The main conclusion from this study is that we are glad that we never filled our chambers with P-10, since the ignition energy is roughly the same as the energy stored in the high voltage de-coupling

capacitors on each wire. In a system such as ours, with thousands of cells, sparking in some cells is not an unusual occurrence. It is also not too unusual to have considerable air inside a chamber during filling operations. The second conclusion is that adding CF4 to the mix greatly increases the energy required to ignite the mixture and renders the combustion self extinguishing, at least in our cell configuration. Finally, reducing the CH4 component to 8% with 8% CF4 and 84% Argon gives a non-flammable mix even with a monster spark source.

Figure 1.

Flammability Test Cell

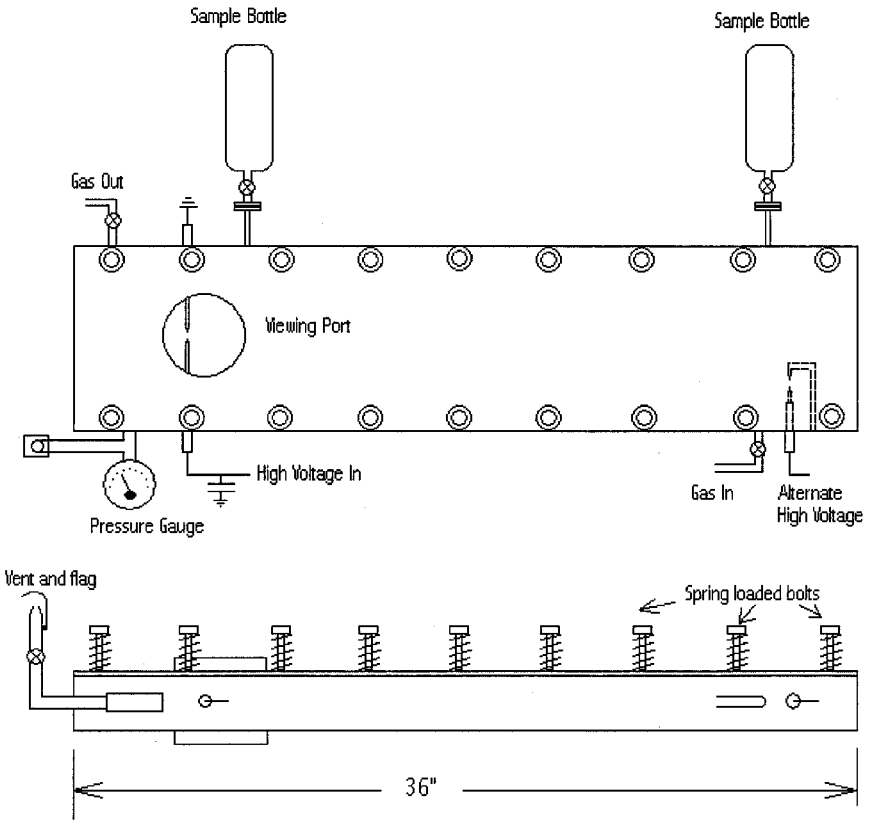
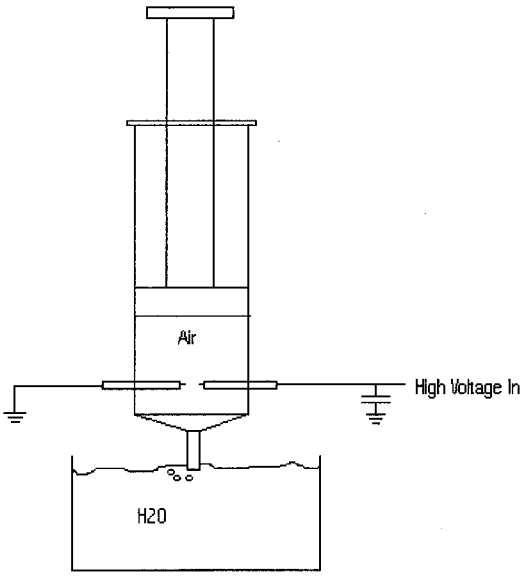


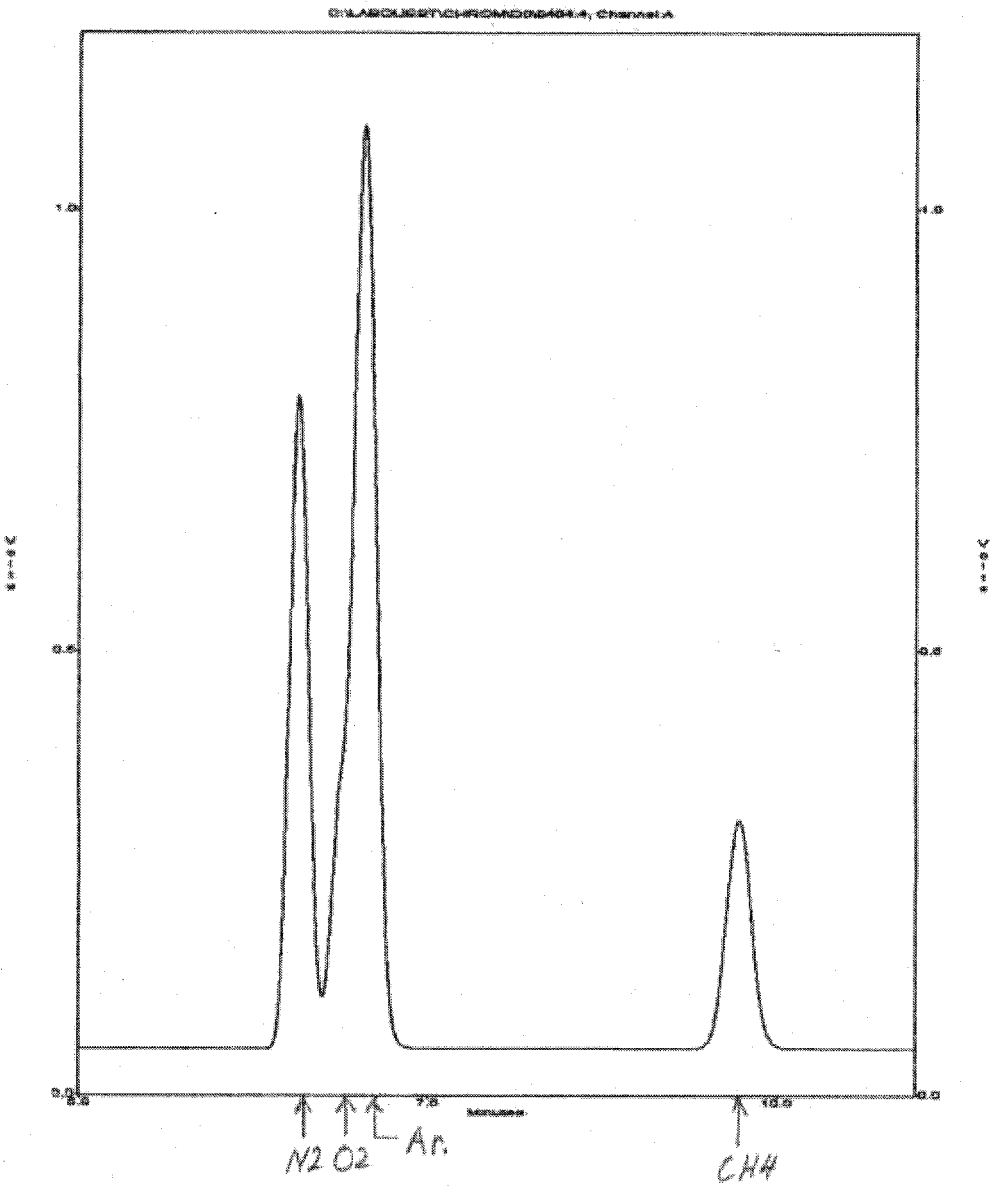
Figure 2

Spark Calorimeter



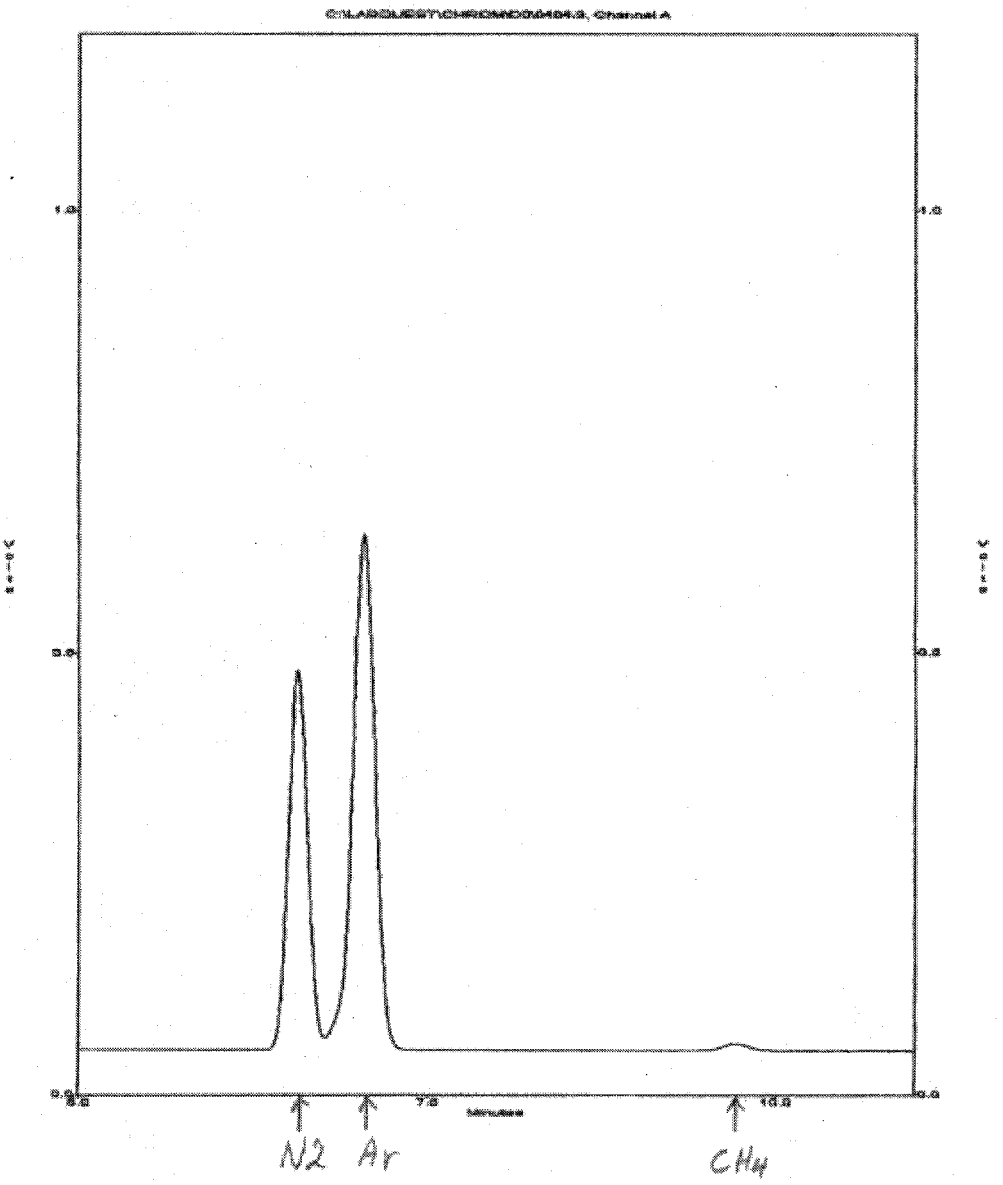
P-10 "BEFORE"

FIGURE 3.



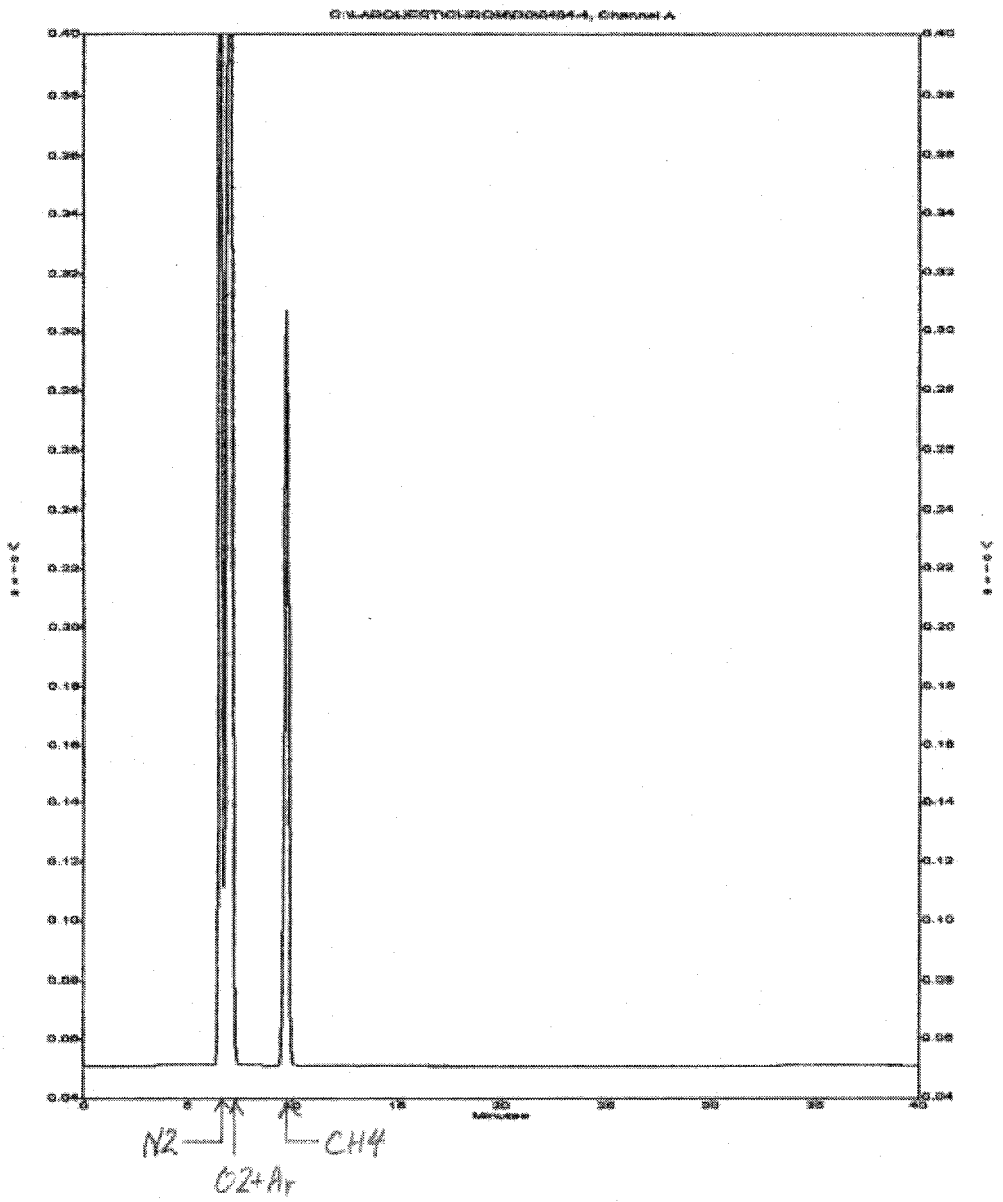
P-10 "AFTER"

FIGURE 4.



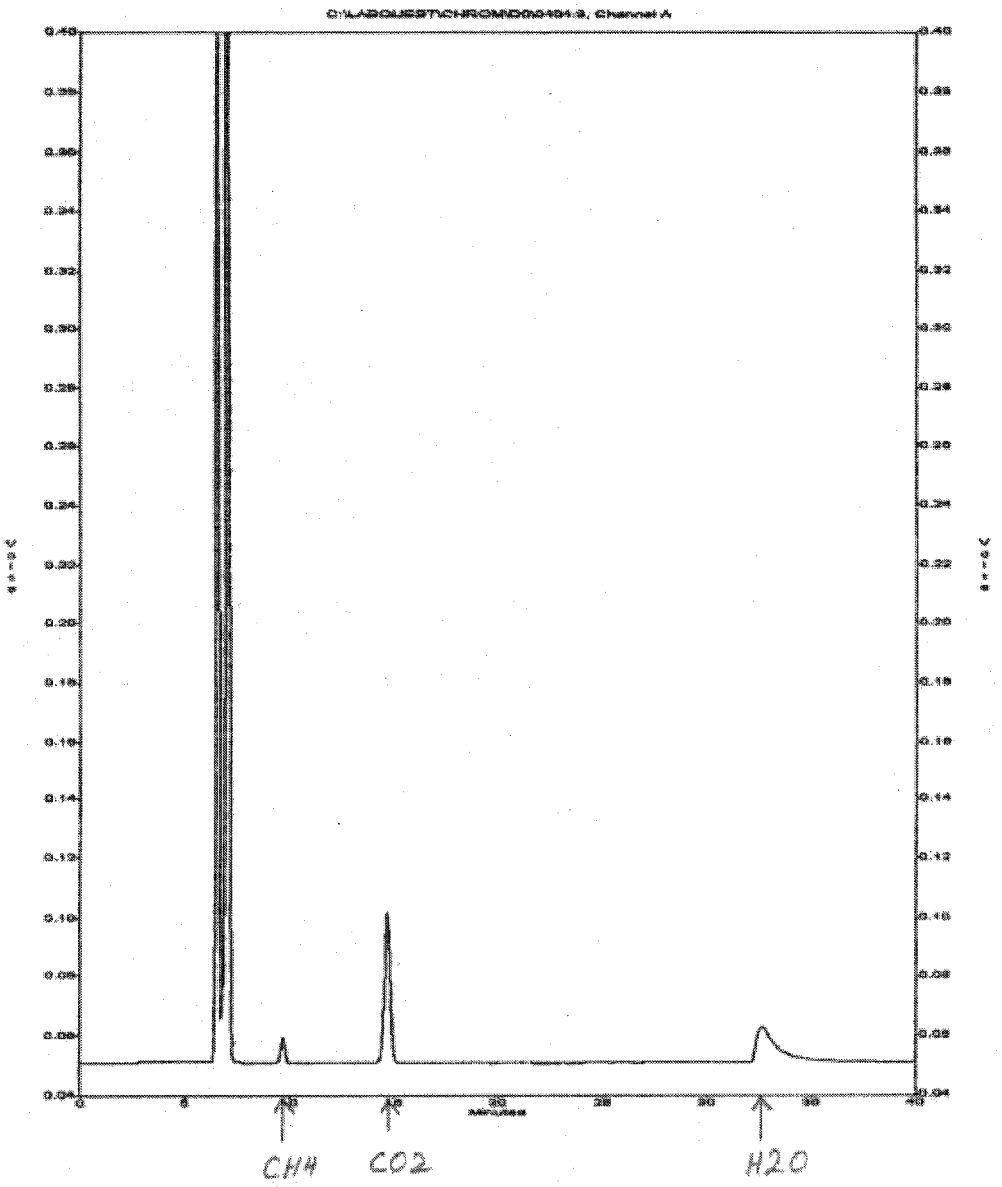
P-10 "BEFORE"

FIGURE 5.



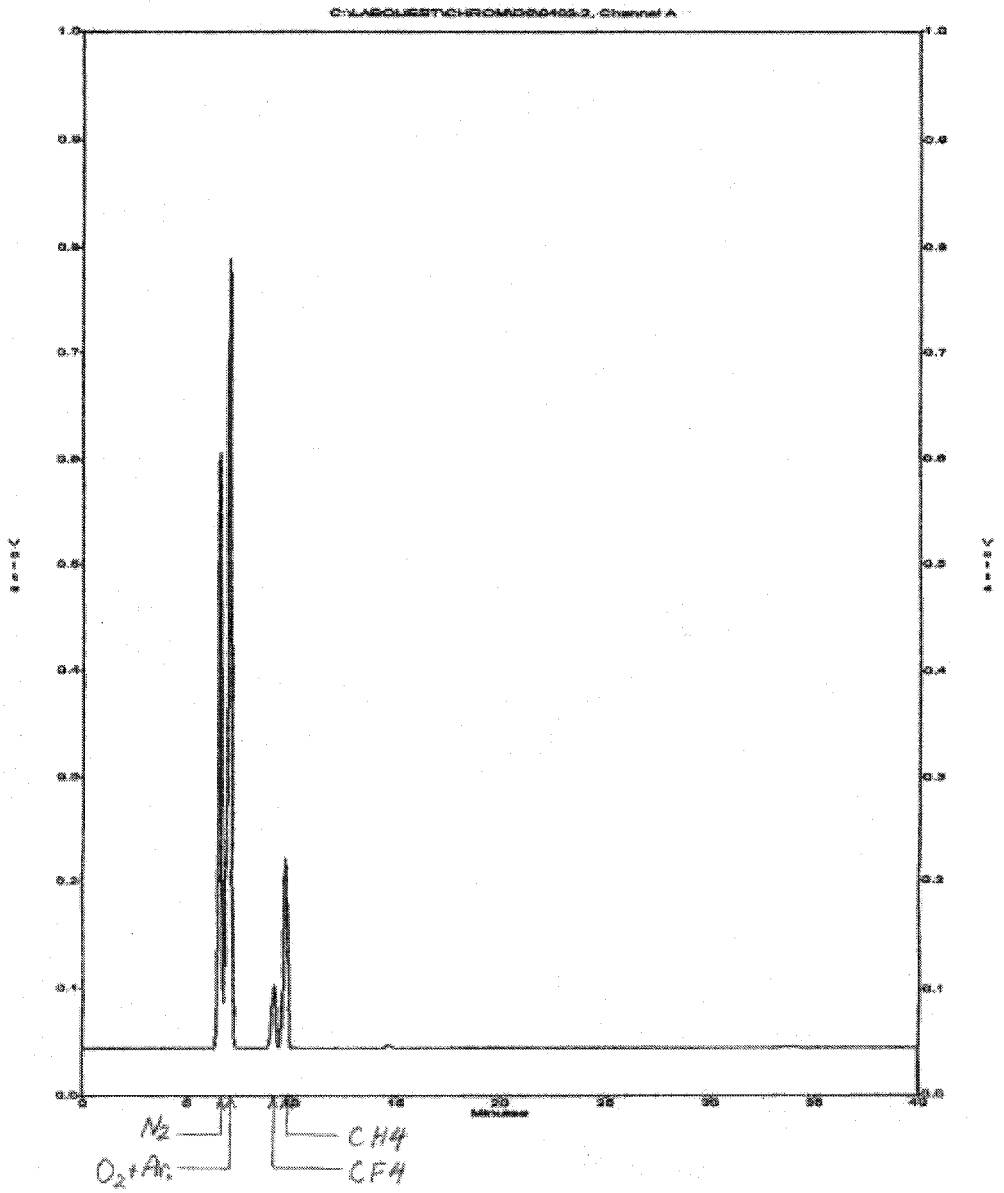
P-10 "AFTER"

FIGURE 6



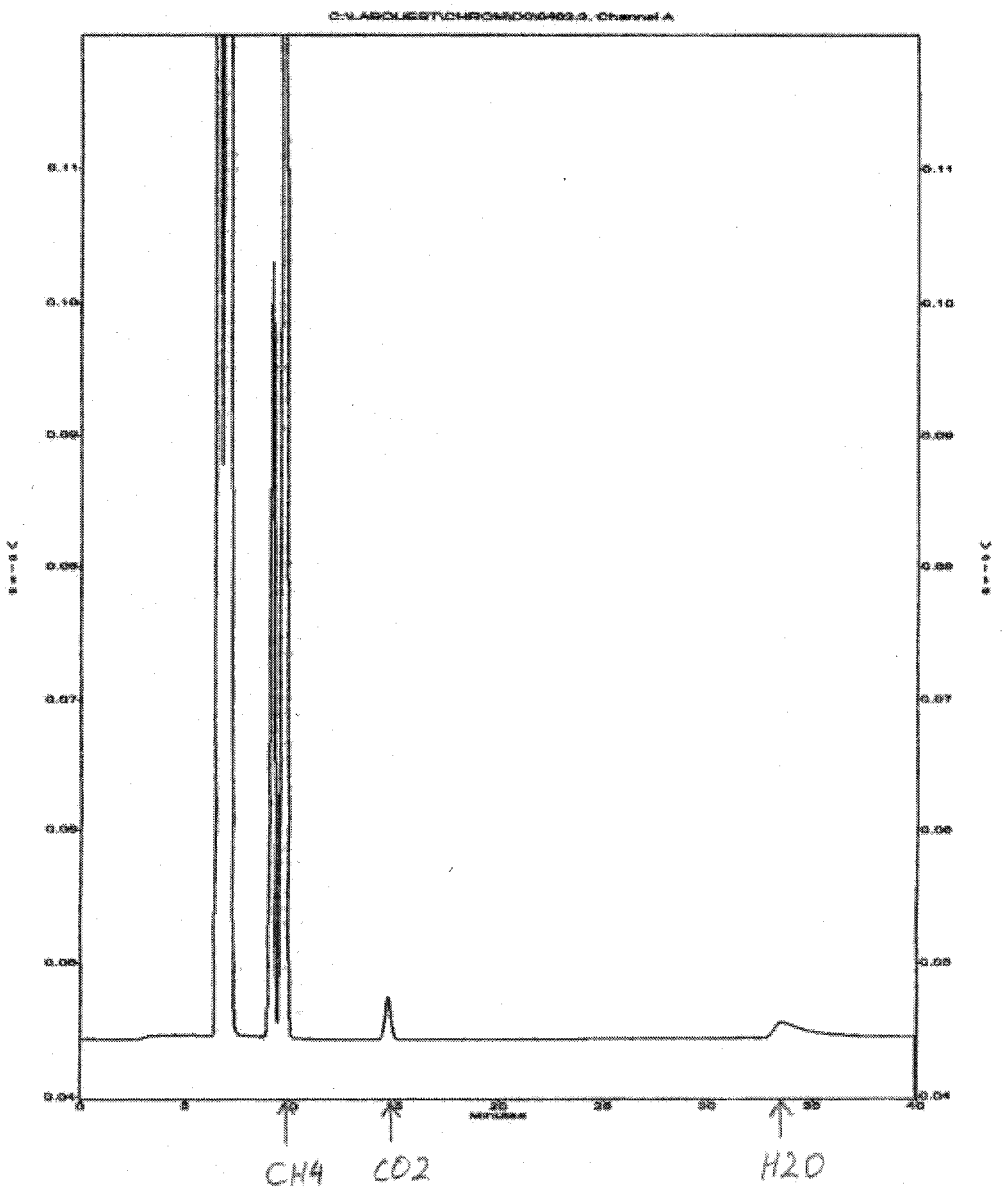
80/10/10 "AFTER"
Near End

FIGURE 7.



80/10/10 "AFTER"
Near End

FIGURE 8.



80/10/10 "AFTER"
Far End

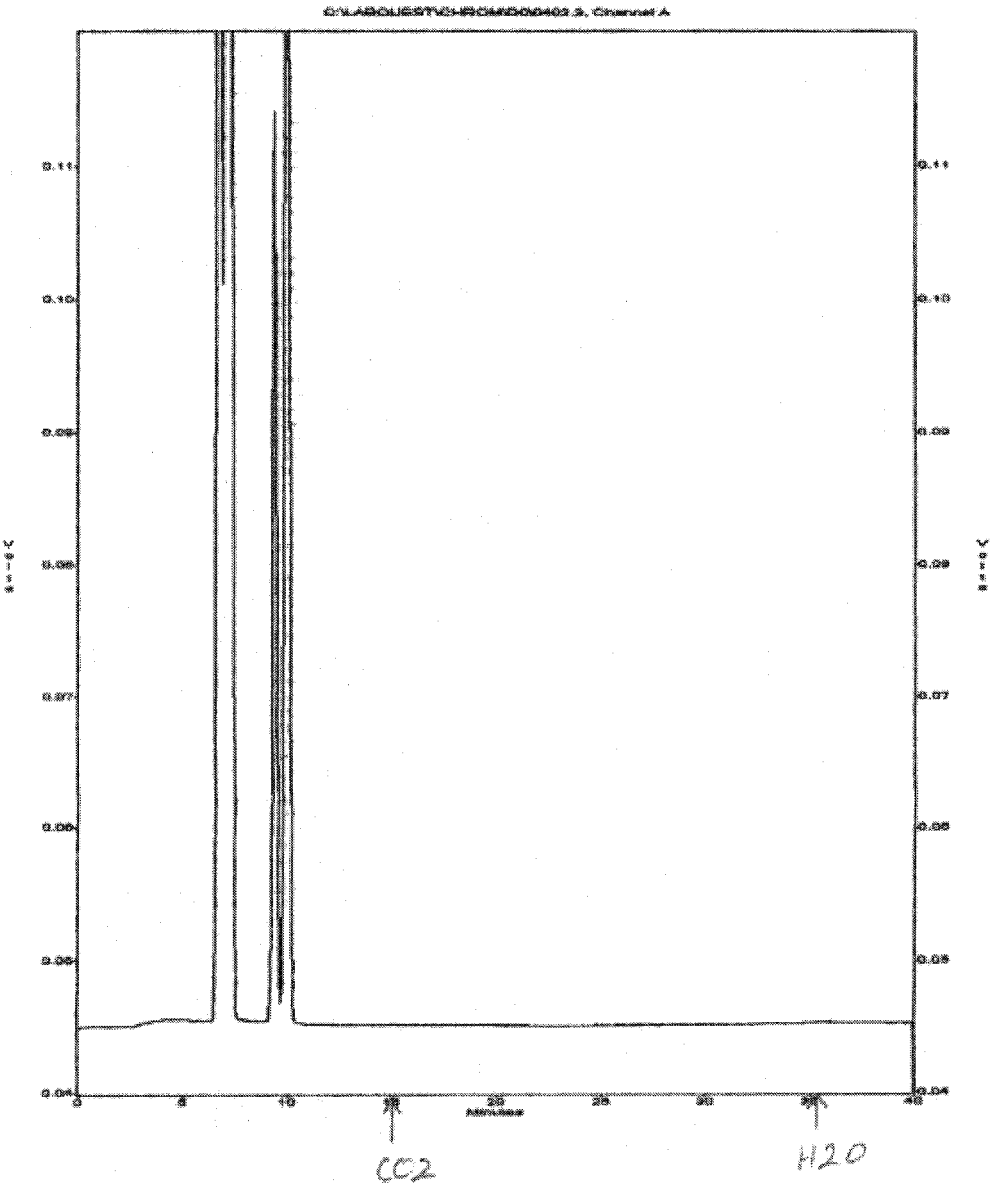



FIGURE 9.