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# CONTROLLED ATMOSPHERE BENCH-SCALE CALORIMETRY REVISITED

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## ABSTRACT

The standard Cone Calorimeter has been designed with an “open configuration”, allowing for testing of specimens through use of freely driven room air for combustion. For testing specimens in oxygen depleted atmospheres or in fuel rich combustion a modified apparatus working under controlled atmosphere can be used. To our knowledge there is very few publications describing the use of such modified cone calorimeters and providing data regarding the effect of ventilation on the fire properties. In the open literature it was reported end of the 1990’s that substantial burning can occurred outside the test chamber when such a device is used, the amount of oxygen available to combustion exceeding the amount that was fed to the combustion chamber. In such a case (leading to post-burning outside the test chamber), it has been proposed to correct the experimental data by replacing the oxygen mass feeding rate by the actual rate of oxygen consumption. This paper analyses the proposed correction, and suggests that this might not be fully satisfactory by comparison to data obtained in another bench scale calorimeter with controlled atmosphere. The capacity of a Cone Calorimeter fitted with a controlled atmosphere device will be more in depth investigated in a new research project starting in 2007.

## INTRODUCTION

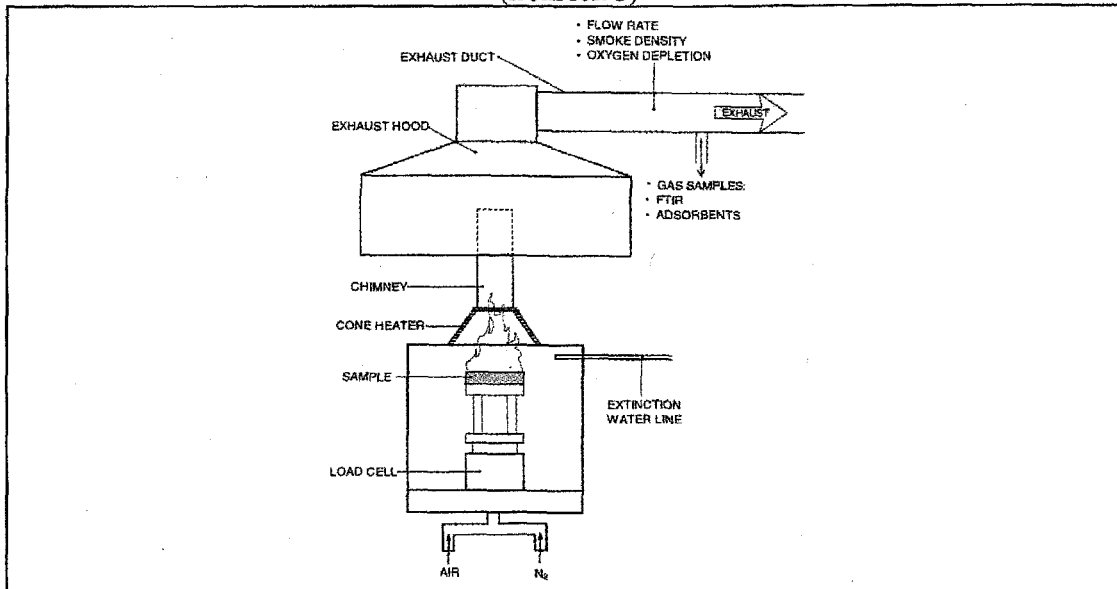
The Cone Calorimeter is commonly used for the measurement of main fire properties of products and materials such as the heat release rate, the effective heat of combustion, the mass loss rate, the time to ignition<sup>1,2</sup>. The standard Cone Calorimeter has been designed with an “open configuration”, allowing for testing of specimens through use of freely driven room air for combustion. For testing specimens in oxygen depleted atmospheres (air vitiation effect) or in fuel rich combustion (ventilation effect) a modified apparatus working under controlled atmosphere can be used. To our knowledge there is very few publications describing the use of such modified cone calorimeters and providing data regarding the effect of ventilation on the fire properties.

On the one hand, a cone calorimeter for controlled-atmosphere studies in vitiated atmospheres has been constructed at NIST<sup>3</sup> in the beginning of the 1990’s. The results obtained in this controlled-atmosphere unit regarding the effect of the air vitiation on the heat release rate and carbon monoxide and smoke produced by flames were published by Mulholland et al<sup>4</sup>. It is important to notice that the controlled-atmosphere NIST unit was used in a closed configuration. The combustion air was supplied at the bottom of the Pyrex enclosure and the combustion products were removed from the top of the enclosure. There was no dilution of the combustion gases before the instrumentation section in the exhaust duct. While some of the changes between the standard unit and the controlled-atmosphere unit were minor, the number of changes is large and affects nearly all of the systems on the apparatus (the cost for constructing the controlled-atmospheres unit was estimated to be more than double that for the standard unit)<sup>3</sup>.

On the other hand, a cone calorimeter fitted with a controlled-atmosphere apparatus was constructed at the laboratory of VTT Building Technology/Fire Technology to investigate the effect of ventilation conditions on the burning characteristics and fire effluents of chemicals<sup>5</sup>. Exhaust duct system, gas measurements and smoke measurements were the same as in the standard Cone Calorimeter method.

Under the cone heater an enclosure to produce the reduced ventilation conditions was constructed with a short chimney on top of the cone heater in order to prevent air flowing into the chamber from above (Fig. 1)<sup>6</sup>. The mixture of air and nitrogen was supplied at the bottom of the chamber with both flow rates which could be adjusted between 0.5 and 4 l/s. The maximum flow through the chamber being 8 l/s, the fire effluents were diluted with ambient air in the exhaust hood (before the measurement section). It was reported that substantial burning can occur outside the test chamber when such a device is used, the amount of oxygen available to combustion exceeding the amount that was fed to the combustion chamber. In such a case (leading to post-burning outside the test chamber), it has been proposed to correct the experimental data by replacing the oxygen mass feeding rate by the actual rate of oxygen consumption. This paper analyses the proposed correction using data obtained in the Fire Propagation Apparatus.

Figure 1. Schematic view of the ventilation controlled cone calorimeter constructed at VTT, Finland (from ref. 5)

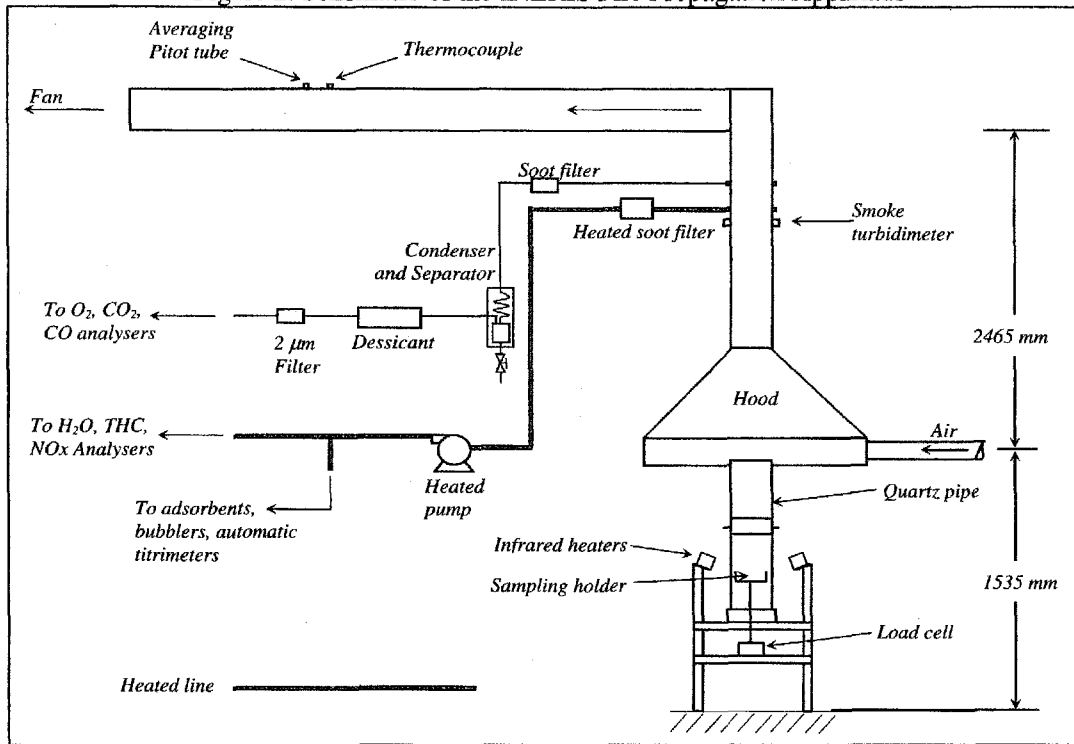


## LEARNINGS FROM THE USE OF FIRE PROPAGATION APPARATUS

Figure 2 shows a schematic view of the calorimeter implemented in a purpose-built section of INERIS reaction to fire laboratory. The lower part of the calorimeter is the combustion chamber which comprises essentially the sample holder system and associated weighing cell, the ignition device and infra red heaters. In addition, the combustion or propagation tests may take place in a controlled volume physically delimited by a quartz tube made of two cylindrical superimposed parts. A range of three mass flow meters allow the operator to set the desired mass flow rate of air to the required value (up to 300 NL.min<sup>-1</sup>) and to modify and adjust the composition of the inlet flow (e.g. mixtures of air, CO<sub>2</sub>, N<sub>2</sub>, O<sub>2</sub>) for research purposes. In particular, the inlet air flow may be enriched or depleted in oxygen. The infra-red heaters system is designed in a way it can supply an even heat flux to the specimen surface at whatever desired value in the range of 0 to 60 kw.m<sup>-2</sup>. The upper part of the apparatus is the exhaust system collecting all combustion product releases and dilution air and comprising main instrumentation section (see figure 1).

The combustion chamber allows the user to adjust the fire ventilation of the test in terms of fuel enrichment of the combustion process. This is done in practice quantitatively through the measurement of the parameter called the equivalence ratio  $\Phi$  (actual fuel-to air ratio normalized by the stoichiometric value of same ratio), a parameter commonly adopted by fire scientists<sup>7</sup>. In any experiment, the equivalence ratio may be targeted at any desired level to feature well ventilated or under-ventilated fire conditions, in its simplest mode by varying the air inlet flow rate.

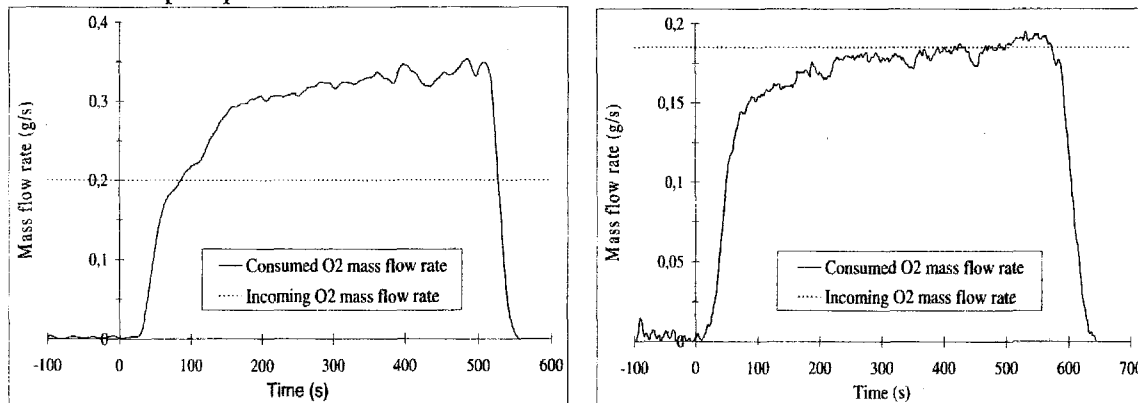
Figure 2. Schematic of the INERIS Fire Propagation Apparatus



Similarly to the cone calorimeter it has been shown that some conditions require a careful control of oxygen flows to determine the true equivalence ratio when very low air flow rate is adjusted (under-ventilated fire conditions)<sup>8</sup>. In such a case, the O<sub>2</sub> consumed as taken from heat release data could be more important than air available by normal air distribution system in the bottom part of the FPA.

Figure 3(a) illustrates such a situation in the case of heptane, a highly flammable hydrocarbon where a control of the actual oxygen consumption by the fire process reveals that an extra supply of air other than normal inlet flow occurred in that experiment.

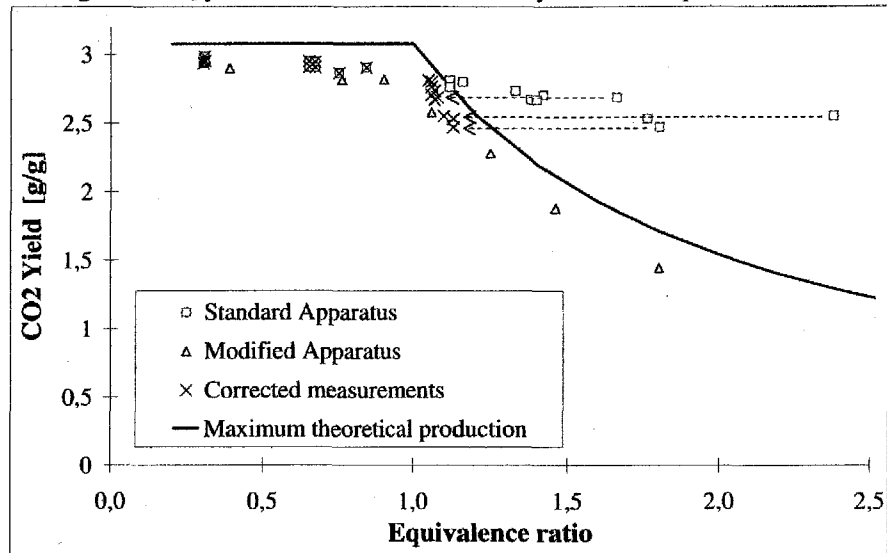
Figure 3. Time evolution of the incoming and consumed oxygen mass flow rates for heptane burn tests in under-ventilated fire conditions. (a) Standard apparatus, (b) Modified apparatus with a section restrictor at top of quartz tube



The same analysis can be done for the CO<sub>2</sub> yield (Fig. 4). Indeed the carbon dioxide produced by the combustion revealed to be more important than the maximum theoretical yield due to the unexpected air entrance in the quartz tube. This unexpected additional air supply revealed to take place at the top of the quartz tube, in the form of air stream entering in opposite direction of main upward gas flow,

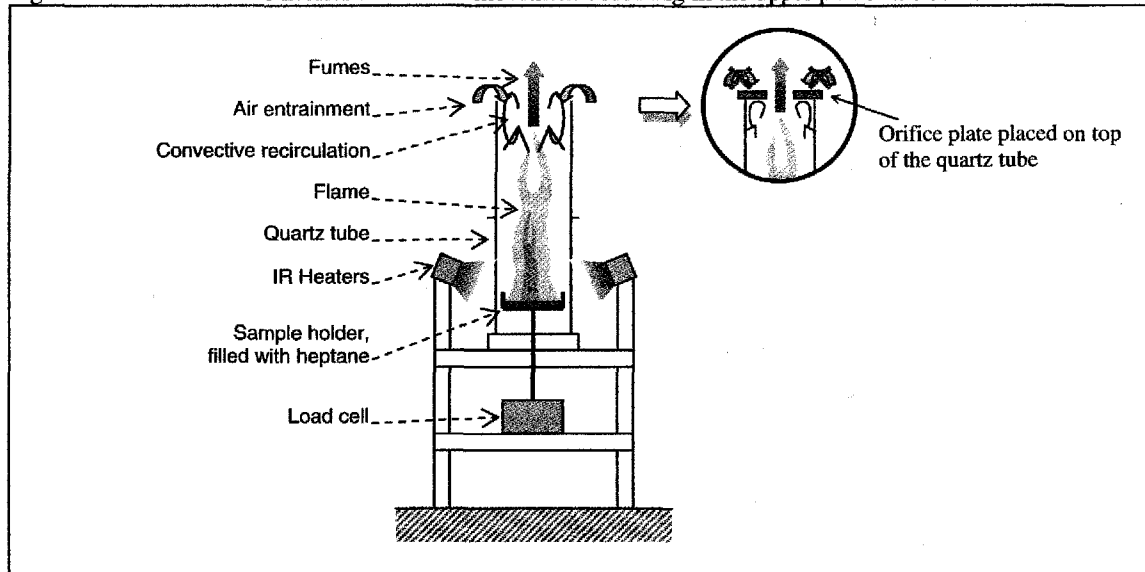
close to the inner walls of the quartz tube (convective movement induced by cold wall effect, see figure 5).

Figure 4. CO<sub>2</sub> yields and maximum theoretical yield versus equivalence ratio.



In order to reduce this phenomenon a technical solution<sup>8</sup> (Fig. 5) consists in reducing the tube outlet diameter, thanks to a disk-shaped refractory piece placed on its top (the diameter was reduced by a factor of ~3). This procedure proves to be a good solution as shown by comparison of the graphs plotted in figure 2 for the oxygen consumption (see also figure 4 for the carbon dioxide yield). The comparison clearly shows that using such a component to prevent any counter flow of air from the top part of the quartz tube is efficient.

Figure 5. Schematic of the natural convective movement occurring in the upper part of the combustion chamber



## EFFECT OF THE OXYGEN MASS RATE CORRECTION

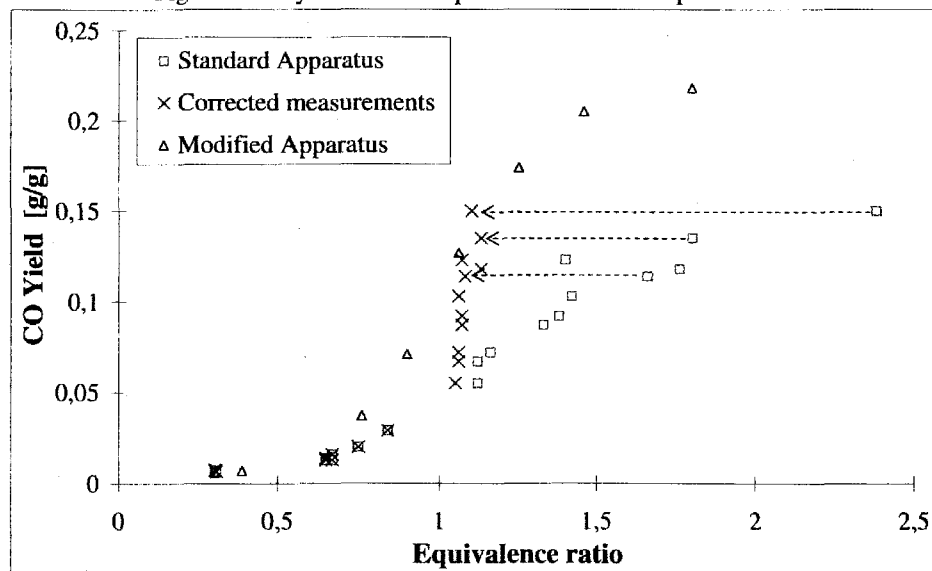
Fuel-rich combustion of heptane has been studied on a Tewarson Calorimeter, in two different configurations:

- Unmodified Tewarson apparatus (standard apparatus), allowing convective recirculation and air entrainment at the top of the quartz tube, and, thus, post-combustion phenomenon.
- Modified Tewarson apparatus, where recirculation is made impossible (using an orifice plate made of mineral material at the top of the quartz pipe) and burning only occurs with available oxygen (controlled mass rate at inlet).

One might wonder whether the correction proposed by Hietaniemi et al applied to the data obtained with the unmodified Tewarson apparatus (by replacing the oxygen mass feeding rate by the actual rate of oxygen consumption) could lead to data which are similar to the ones obtained with the modified Tewarson apparatus. The results are presented in figure 4 for the carbon dioxide yield. By considering only the amount of oxygen that is fed to the combustion chamber ( $\square$ ), the CO<sub>2</sub> production exceeds the maximum theoretical yield in the case of the standard apparatus (post-combustion phenomenon). Hietaniemi et al. correction (X) solves this issue, but does not allow for testing highly fuel-enriched mixes as post-combustion still happens, in opposition with real controlled atmosphere measurements ( $\triangle$ ) (especially for heptane, a very flammable product which leads to high heat release rates).

In a similar way, the data measured for carbon monoxide are presented in figure 6 for the standard Tewarson apparatus and the modified one. The correction proposed by Hietaniemi et al is also presented. When a post-combustion phenomenon is observed it comes that the correction of Hietaniemi et al should be applied in order to obtain more realistic results. However, this correction might not be fully satisfactory by comparison to data obtained in the modified Tewarson Apparatus. The remaining difference comes certainly from the fact that the oxygen mass feeding the combustion chamber is certainly higher than the actual rate of oxygen consumption.

Figure 6. CO yields versus equivalence ratio for heptane fires.



## CONCLUSION

Bench scale fire tests such as the Cone Calorimeter equipped with ventilation controlled atmosphere or the Fire Propagation Apparatus can be used for the establishment of experimental correlations between chemical species yields and the equivalence ratio which can be used as input data in computer models for the prediction of the fumes composition. However, a practical issue may arise

to calculate the actual equivalence ratio in experiments with bench-scale apparatuses like the Cone Calorimeter and other fire calorimeters, since oxygen consumed as derived from heat release data could be more important than the theoretical limit of oxygen supply introduced at the bottom of the test chamber. In fact, secondary combustion air may be brought and participate in the combustion process under some circumstances at ignorance of user from the dilution air stage that occurs before the effluents enter the collecting hood. In such a case, the correction proposed by Hietaniemi et al should be applied in order to obtain more realistic results. However, the proposed correction might be not fully satisfactory since the oxygen mass feeding the combustion chamber is certainly higher than the actual rate of oxygen consumption. The objective of this project will be to improve the confined atmosphere testing equipment for the Cone Calorimeter with proper ventilation control to reproduce the whole range of burning conditions for different materials.

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