Laboratory Demonstration

COMBUSTION PROJECT: EXPLOSIVE LIMITS

S. SANDLER
University of Toronto
Toronto, Ontario, Canada

FOR SEVERAL YEARS an apparatus built according to a description by G. W. Jones [1] has been used by freshman engineering students taking the combustion option in the Department of Chemical Engineering at the University of Toronto, to study the explosive limits of a typical hydrocarbon, as well as a number of basic physical chemistry principles associated with the measurement. It was felt that a modification of the apparatus to make it suitable for demonstration purposes using a "hands-on" approach was desirable. The following criteria for such a design were then established:

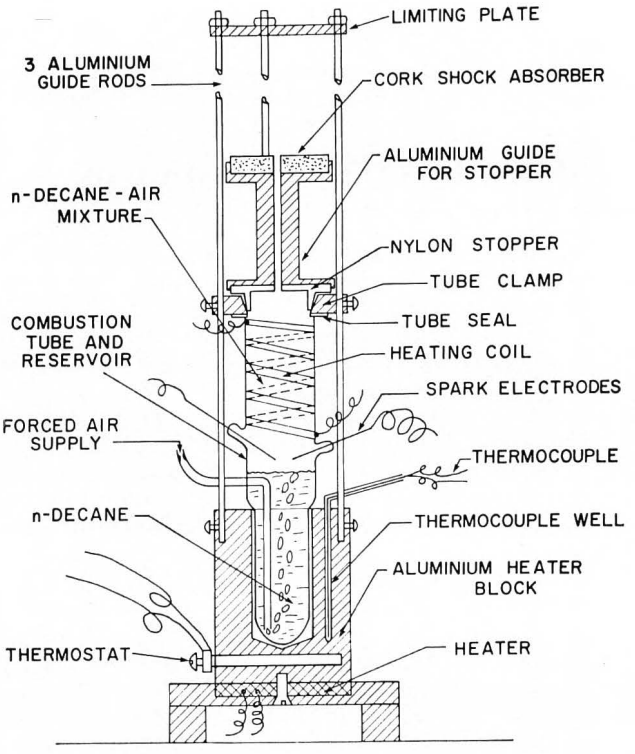

FIGURE 1. Explosive Limits Apparatus.

- **It should be capable of illustrating certain principles (e.g. existence of explosive limits, the vapour-pressure temperature relationship, relative rates of flame propagation, approach to equilibrium).**
- **It should be simple to operate, requiring only the pushing of buttons, and observation of the effects.**
- **It should be safe when operated by entirely unskilled people, including children at grade school level.**
- **It should yield reproducible results without requiring the intervention of an operator.**
- **It should be essentially self-explanatory.**
- **The effects should be sufficiently impressive to attract students to the apparatus and to stimulate them to raise further questions and perhaps try further experiments in this field.**
- **The unit should be essentially portable and yet heavy enough so that it could not readily be removed from any location in which it was set up.**

The design to be described here apparently satisfies these criteria. It is hoped that the description will serve to point out the problems and the solutions associated with the re-design of a relatively simple laboratory-scale apparatus to conform to the rather more stringent criteria of a demonstration unit.

DEMONSTRATION UNIT

A DIAGRAM OF SOME of the features of the design of the demonstration unit as finally adopted is shown in Figure 1. The vapour generator and combustion tube combination is made of Pyrex glass and consists of a lower reservoir for the fluid under test, 75 mm long and 25 mm in diameter, which sits in the heated block at the test temperature, and an upper separately heated combustion chamber, 150 mm long and 30 mm in diameter. A glass tube entering the reservoir near the top extends to a position close to the bottom of the reservoir and serves to lead air pumped by a fish-tank aerator at the rear of the unit through a tube partially packed with molecular sieve 5A and restricted at the end, through the test fluid and into the combustion chamber.

The air will pick up a proportion of the vapour

Professor Sandler received his B.A.Sc. and M.A.Sc. from the University of Toronto. His principal research interest has been in kinetics and mechanism of the oxidation, decomposition, ignition and detonation of fuel vapours and gases as well as of the associated instrumental methods of chemical analysis. After ten years with Defense Research Board of Canada as a Principal Scientific Research Officer on combustion research, he joined the staff of the Department of Chemical Engineering, University of Toronto. Besides his teaching and research work, Professor Sandler is an active consultant on combustion and analytical matters with Chemical Engineering Research Consultants Ltd. He has also been very active in the Chemical Institute of Canada, as chairman of the Toronto Section, as Councillor "A" of the national body, as a tour speaker and as organizer and chairman of the first three Toronto Symposia on Gas Chromatography. He is presently a Fellow of the Chemical Institute of Canada.

of the test fluid which will depend on its vapour pressure at the test temperature and the degree to which equilibration of the air with the fluid is allowed to occur. With the relatively low air flow rate used and after passage of air for at least 30 seconds, a steady state condition is set up which is not far from the equilibrium state. Hence, a vapour pressure-temperature curve for the test fluid (in this case, n-decane) with associated vapour-air composition scales as in Figure 2 can be used to estimate the mixture strength corresponding to operation at any test temperature, as read on a pyrometer. An iron-constantan thermocouple imbedded in the heater block provides the impulse for this reading. The heater for the block is a 300 watt element in its base controlled to within $\pm 0.5C^{\circ}$ by a thermostat within the block.

An auxiliary heater coil is necessary to prevent condensation of the vapour in the combustion chamber by maintaining it at or slightly above the test temperature. This less critical temperature is maintained constantly by providing an appropriate voltage from a small transformer to the auxiliary heater of each unit.

After creating the desired mixture of fuel vapour and air, a spark is passed across a 1/4-inch gap between two platinum electrodes located in the lower part of the combustion chamber. The liquid level in the reservoir is usually about 1/2 inch below the electrodes. However, the actual position of the liquid with respect to the electrodes is not critical and the unit may be operated for several days before make-up liquid is required.

Flame propagation is signalled in a number of ways. When the lights are turned off, it is possible to see a flame, initially generated at the electrodes, actually propagated through the mixture if it has a composition somewhere within the explosive limits. In addition, the pressure generated by the

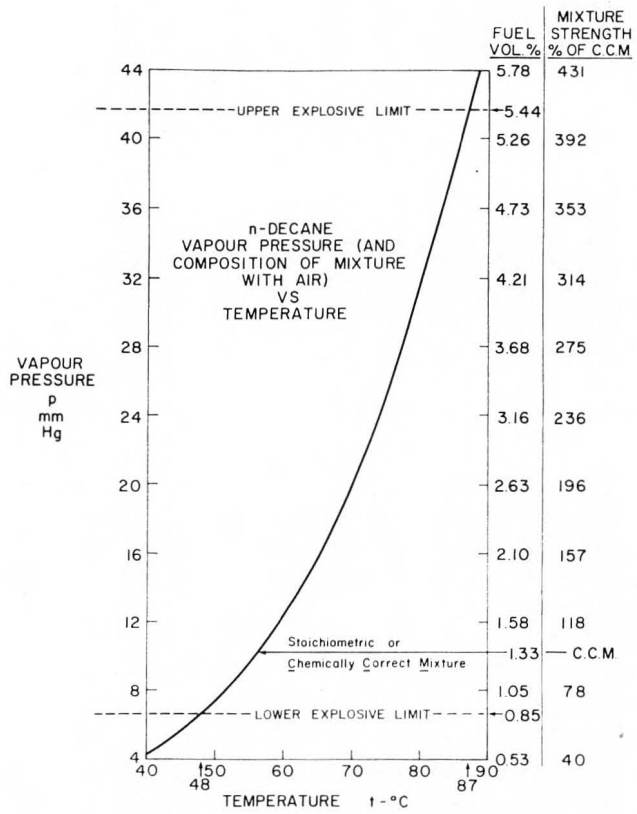

FIGURE 2. Vapor Pressure Plot.

expanding gaseous products of the combustion may be sufficient to lift the Nylon or Teflon stopper off the combustion tube and raise the aluminum guide holding this stopper to some level above its rest position. If the explosion is sufficiently vigorous, that is, if the flame velocity is sufficiently great, this guide will travel all the way up to strike the upper limiting plate. A cork shock absorber is incorporated into the upper end of this guide for the purpose. Subsequently, the guide

returns to its rest position and, *if properly designed,* seats itself snugly into the mouth of the combustion tube in preparation for the next test. For proper reproducibility of the results, it has been found necessary to machine the stopper out of either Nylon or Teflon, to adjust the weight of the guide to allow a vigorous return and seating of the stopper after an explosion without jamming and to machine the stopper so that it will, at one and the same time, seat solidly but along a

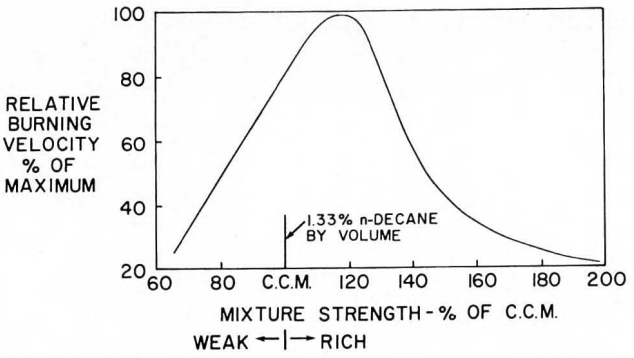

FIGURE 3. Relative Burning Velocities of n-Decane—Air Mixtures.

relatively narrow portion of its circumference. If appropriately set up, such a device exhibits a maximum popping effect during an explosion and permits unattended operation of the unit for a very long time. The noise associated with the propagation of a flame and/or explosion is a third way in which the event can be monitored.

A number of safety features have been incorporated into this design. A relatively high boiling liquid (n-decane boils at 174°C) is generally selected as the test fluid, although the device is amenable to the study of lower boiling materials with suitable modification. With this in view, the normal vapour composition within the confines of the large box containing the units is always well below the lower limit of inflammability of the fuel. However, to doubly ensure this, a fan has been incorporated into the box to expel any vapours to the outside. Observations of the explosion effects are made through a safety glass front and only the push buttons for air and spark are exposed for operation of the units. There is no combination of operating parameters yet found which will result in anything more vigorous happening than has been designed into the apparatus. Operation of an all-glass prototype of this apparatus by students over the past three years under much less carefully controlled conditions has proceeded without the slightest mishap. Even if the stopper does jam into the mouth of the combustion tube, and an explosion is generated, it has been found that blow-back of liquid occurs into the drying agent in the air inlet tube and the explosive force is thereby released without shattering the container.

DISCUSSION

AS WILL BE OBSERVED, three of the combustion tubes and associated apparatus have been incorporated into the demonstration unit to satisfy the educational criterion mentioned above. Table 1, as posted on the unit, gives the generally accepted limits of inflammability for the test fluid, n-decane, in terms of both vapour-air compositions and the corresponding fluid temperatures which would yield such mixtures. Combustion tubes 1 and 3, which are respectively to the left and right of the central apparatus in the unit, are thermostatted at temperatures which will generate respectively a mixture just above the lower explosive limit and a mixture just below the upper explosive limit. The central unit is thermostatted at a temperature corresponding to a mixture near that which would propagate flame at maximum velocity.

The general form of the relationship between mixture composition, within the explosive limits, and flame velocity is shown on the graph in Figure 3, also as posted on the unit. The observations of the explosion intensity in each of the tubes may be compared to this graph, to yield a preliminary explanation of the effects in terms of differences in flame velocities. An in-depth review of the more

TABLE 1

COMBUSTION TEST FLUID NORMAL DECANE

EXPLOSIVE LIMIT	VOL. % IN AIR	CORRESPONDING TEMPERATURE—°C
LOWER	0.85	48
UPPER	5.44	87

rigorous precepts involved in this phenomenon is beyond the scope of this paper. However, an extensive treatment is available in the text by Lewis and von Elbe [2].

It is interesting when demonstrating this unit, to ask students to predict the effect of the grad-

ually increasing test temperature, especially after they have made the observations of the explosion intensity in the first two combustion tubes and before they have observed the effect in the third apparatus. The mildness of the "explosion" in the third tube (at the highest temperature) generally surprises people after they have seen the effects in the other tubes.

It would be desirable to demonstrate, as well, the inability of flame to propagate through the gaseous mixture at compositions just below the lower limit and just above the upper limit. Unfortunately, this would be incompatible with the achievement of the other criteria for this demonstration unit without increasing the number of combustion units to be maintained at different constant temperatures or without increasing the complexity of the operating instructions.

In an arrangement for a laboratory experiment, only one combustion tube and its associated hardware would be required to demonstrate the principles already mentioned as well as several others. Thus, a step-wise increase in temperature and, hence, mixture strength can be achieved by simply altering the thermostat settings appropriately. The whole range of desired mixture strengths could thus be scanned beginning below the lower limit and extending beyond the upper limit. Observations concerning the character of the inflammation and the explosive violence could then be made throughout. In addition, a variety of inflammable liquids could be examined. For example, in our combustion laboratory, the explosive limits for a number of Jet Al fuels have been examined in such an apparatus. It is interesting to note that the lower explosive limit for such fuels is very close to that of n-decane. For present purposes, however, it is preferable to operate with a fuel of which the composition would be invariant over the long period of use.

Another example of a more exotic application of such an apparatus is in the determination of the lower limit of inflammability of a 40% by volume ethanol-water mixture (as in several alcoholic beverages). The degree to which the vapour pressure of alcohol in contact with such a solution exceeds the value calculable on an ideal solution basis, using Raoult's Law, can in fact, be estimated from such a measurement if the vapour pressure-temperature relationship and the lower explosive limit for pure ethanol is known. The extension of the project to achieve this goal is outlined in Table 2. □

TABLE 2

Extension of Project No. 4—Combustion
Lower Limit of Inflammability of an Ethanol-Water Solution

Data: Ethanol (C_2H_5OH – M.W = 46.08)
Density of pure ethanol = 0.789 g/ml at 20°C
Density of 40% v/v ethanol-water solution = 0.952 g/ml at 15.56°C
(% ethanol (W/W) = 33.1%)

$$\log^{10} P^{\circ}_{C2H5OH} = \left(\frac{-0.2185 \times 9673.9}{K}\right) + 8.827392$$

where K = temperature in °K
P°_{C2H5OH} = vapour pressure of C_2H_5OH in mm Hg.
Assume barometric pressure = 760 mm Hg (unless measured)

1. **Determine, experimentally, the temperature at which a 40% v/v solution of ethanol in water reaches the lower limit of inflammability of ethanol vapour in air (determined in tests with pure ethanol to be 4.3 % v/v).**
2. **Assuming ideal solution behaviour and attainment of equilibrium, calculate the concentration of ethanol in air corresponding to the experimentally determined temperature.**
3. **Using the vapour pressure-temperature relationship for ethanol (see data from Handbook of Chemistry and Physics) and again assuming ideal solution behaviour and attainment of equilibrium, calculate the theoretical temperature at which a 40% v/v ethanol-water solution would produce an ethanol vapour-air mixture at the lower limit of inflammability (4.3% v/v).**
4. **Compare the results of 2 and 3 with 1 and explain the discrepancies. Calculate the factor by which the calculated partial pressure in 2 should be multiplied to give the lower limit mixture at the experimentally determined temperature.**
5. **Would you expect an alcoholic beverage containing 40% v/v ethanol as well as certain flavour components and sugar to produce vapour at the lower limit of inflammability at a higher or lower temperature than**
   (a) the theoretical temperature calculated in 3?
   (b) the experimental temperature determined in 1?
   Justify your answers.

REFERENCES

1. G. W. Jones, Inflammation Limits and their Practical Application in Hazardous Industrial Operations. Proceedings of the First and Second Symposia on Combustion—as Reprinted in 1965 by the Combustion Institute, pp. 248-264.
2. B. Lewis and G. von Elbe, Combustion, Flames and Explosions of Gases, 2nd edition, Academic Press, 1961.

ACKNOWLEDGMENT

The demonstration unit described here was constructed in the machine, electrical and glassblowing shops of the Department of Chemical Engineering at the University of Toronto. The author is extremely grateful and thankful to Messrs. John Aslin and Gord Kearns (machine shop), Ken Adams, K. Atia, K. Kim (Electrical shop) and Fred Leslie (glassblowing shop) for their invaluable assistance in bringing this project to fruition.