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OXYGEN INDEX - AN APPROXIMATE VALUE FOR THE EVALUATION OF COMBUSTION CHARACTERISTICS

I. Zartmann, D. Reinwardt and A. Franke

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OXYGEN INDEX - AN APPROXIMATE VALUE FOR THE EVALUATION OF COMBUSTION CHARACTERISTICS

By: Chem.-Ing. Isolde Zartmann, Dipl.-Ing. Dieter Reinwardt and Dr.-Ing. Albrecht Franke

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The oxygen index has gained international recognition for the determination of combustion characteristics of plastic material.

The amounts of oxygen and nitrogen were more accurately determined for existing test equipment in order to specify the oxygen index as precisely and as reproducible as possible.

The study outlines parameters such as the size of the ignition flame, ignition of test pieces, test piece sizes and test temperature. The minimum oxygen index was determined by the dimension and duration of the fire. The results are sufficiently accurate for factory operating conditions and are also reproducible.

1. Introduction

It is necessary to know the combustion behavior of plastic materials in order to process them correctly and without danger. This requires the determination of flammability criteria such as ignition characteristics, flame propagation, heat release and smoke formation which will give a relatively good idea of the material's combustion characteristics. The oxygen index procedure serves to determine the ignition resistance or relative flammability of an organic material.

The oxygen index procedure has gained wide recognition for the plastics producing industry as a routine test to check the uniformity of properties during production and also for researchers in developing flame-retardant materials.

The procedure is usable for different shapes and structures of materials. Numerical results ranging from easily flammable to practically non-flammable materials offer an efficient and simple check method suitable for routine tests.

The oxygen index is a laboratory value and not directly applicable to actual situations. It does not permit a final and conclusive judgement of combustion behavior but is regarded as a characteristic value.

2. Test procedure

The procedure is based on the candle test as described by Fenimore and Martin (1) in 1966 which since then has been developed further and perfected. It is generally used in the plastics industry.

The principle is based on the determination of a minimum concentration of oxygen in a flow of oxygen/nitrogen mixture in which an upright test piece is just capable of maintaining combustion from the top to the bottom.

The test piece is fixed perpendicularly in a glass cylinder. A predetermined mixture of oxygen/nitrogen flows upwards through the cylinder and surrounds the test piece, the upper end of which is ignited by a propane flame. If combustion is maintained then the oxygen is gradually reduced until the flame goes out at a predetermined section length or after a predetermined time span. The parts per volume of oxygen found at this condition is converted as per

$$n(\%) = 100\frac{O_2}{O_2 + N_2}$$

to the LOJ value expressing it as a volume percent statement.

Efforts to standardize this procedure in the GDR are outlined in Standard Proposal TGL 25 253/01, 1975 (2) and in the RGW Standard Proposal RS-4168, 1973 (3).

This procedure was standardized in 1970 in the US standard ASTM D 2863 and revised in 1976 (4).

3. Test equipment

Devices for this test procedure are readily available from several US and British companies (5). Some DDR facilities have acquired such devices. Several laboratories also use devices built by themselves which maintain measuring principles and dimensions as per the standard. There are differences when measuring flow quantities of oxygen and nitrogen.

Most of the homemade devices as well as the one of the Michigan & Company measure both gases via calibrated rotameters which are then mixed and conducted to the test cylinder. The measuring scale and setting marks thus give the adjustment and reading accuracy of the oxygen portion which ranges between 0.2 and 0.5 % by volume. The device by the Stanton Redcroft Company measures the O_2 portion after admixture via an oxygen analysator which offers an adjustment and reading accuracy of 0.1% by volume.

The measuring device available at our place was designed and made by the VEB Leuna works "Walter Ulbricht" and can determine the LOJ values in real life situations. Flow quantities are controlled via a rotameter with an adjustment and reading accuracy of 0.5% by volume.

Following test trials of the device technical changes were made to increase the measuring accuracy:

Replacement of the somewhat inaccurate needle valve with a precision valve as per Prof. Rossignol in order to eliminate fluctuations in the oxygen and nitrogen flow.

Addition of an auxiliary air controller in the nitrogen line downstream of the reducing valve (measuring range 0...0.5 MPa, control accuracy 0.01 MPa).

Installation of a light source behind the rotameter for better reading of indicators.

Improving the test piece clamping device by inserting a metal cylinder of 100 mm height.

A backfill of 5 mm diameter glass beads produced a mixing capability as required by the Standard or the Proposed Standard.

Furthermore, by testing a large number of plastic materials (foils, foam, compact material) we were able to create comparative values and criteria between TGL standard 25 253/01 and ASTM D 2863-76 which, when properly applied, will produce good and reproducible results.

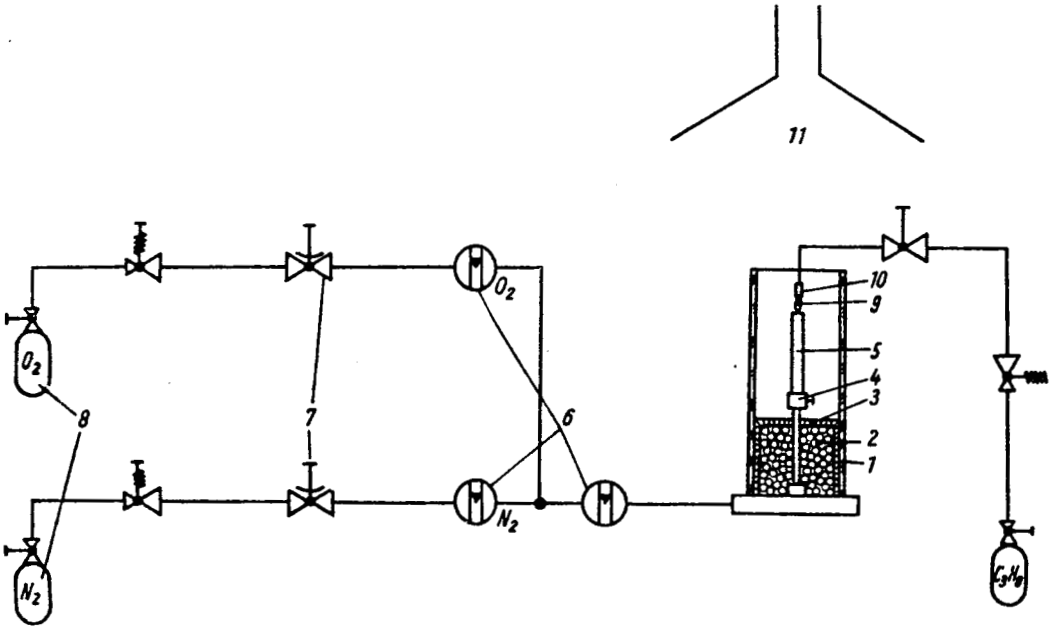

Fig. 1 Schematic arrangement of the test facility "Oxygen Index"

4. Test facility

The test facility was installed as per the flow schematic shown in Fig. 1.

The test device proper consists of a test container as well as the regulator and measuring section.

The test container consists of a glass cylinder (1) with a minimum diameter of 55 mm and a minimum height of 400 mm. Glass or metal beads (2) of 3 to 5 mm diameter are poured from a height of 80 to

100 mm to increase the mixing capability. A strainer (3) is placed above the beads to catch burning cinders. The test piece holder (4) guarantees a perpendicular stationing of foils and rods. The two gas flows are regulated by needle valves (7). Nitrogen and oxygen of at least 98% purity is taken from gas bottles (8).

The test piece is ignited by a propane flame (9) of about 10 mm length which comes out of a nozzle (10) of 2.5 mm diameter. The test facility is placed under an extractor which is switched on during testing.

5. Critical parameters and special aspects during testing

Evaluation based on the aforementioned Standard and Proposed Standard shows a number of differences with respect to ignition of the test piece, its size and measured results.

5.1 Ignition of the test piece

A comparision of the various ignition parameters showed that they have a definite influence on the test results and must, therefore, be kept as constant as possible. The tests were carried out with a propane flame (technical propane). The test flame thermically heats the test piece up to the point of ignition. The longer the flame contact the higher the thermical influence on the test piece which means that the result will show a low LOJ value. It should, therefore, be established, prior to testing a new material, at what point in time the surface of the material starts to burn after contact with the flame. This time span must then be applied uniformly to that particular material.

Ignition times fluctuate for each type of material and are usually shorter for foam and foils than for compact material. The ignition period should be minimized and kept constant since LOJ values are also very much temperature-dependent. The given temperature of 23°C + 2K prior to testing the material, as well as the gases and gas container must not be varied. Since ignition and burning heats

the glass cylinder, it is important to let the unit cool down to room temperature before beginning a new test. Working with several cylinders avoids unnecessary waiting time between the tests.

5.2 Test piece size

Five to ten test pieces will be required to test a particular material. According to the proposed TGL 25 253/01 and RS 4168-73 only solid plastic substances can be tested.

Test piece dimensions (mm):

Width	10 ± 0.5
Thickness	4 ± 0.5
Length	80 to 120

ASTM D 2863-76 permits also the testing of foam and sheet material (foil) which can be clamped into a frame.

Test piece dimensions (mm):

	Solid	Foam	Foil
Width	6.5 ± 0.5	12.5 ± 0.5	52 ± 0.5
Thickness	3.0 ± 0.5	12.5 ± 0.5	as available
Length	70 to 150	125 to 150	140 ± 5

The dimensions for foam and foil listed in ASTM D 2863-76 have proved to be very satisfactory. Solids, however, should follow proposed Standard 25 253/01 since such dimensions are also applicable for other material tests.

5.3 Determination of the minimum LOJ value

The LOJ value represents the minimum oxygen concentration in the flow mixture of oxygen and nitrogen which just maintains combustion of the test piece like a candle. In order to find this point one approaches the problem from both sides by searching for the concentration which lies between the dying-down and complete combustion of the piece. The two criteria are used for judgement since from ignition to going out a certain time span will have elapsed and a

certain amount of the test piece will have burned away. According to ASTM D 2863-76 the minimum LOJ value will have been reached when combustion ceases as per one of the following criteria:

	Solid plastic	Foam	Foil
1. Length of burn (mm)	50	75	100
2. Duration of burning (s)	180 ± 3	180 ± 3	-

One of the criteria will be exceeded for the next O_2 concentration.

The next lower O_2 concentration should not produce any of the critical points. The tests should be repeated and must be reproducible.

According to TGL-E 25 253/01 the minimum LOJ value will have been reached when the test piece extinguishes itself after a burning period of 180 ± 3 s. The burnt-off length is measured and recorded as well as other observations such as dripping, charring, etc. Based on a large number of material tests we concluded that the burn length (mm) had to be used most of the time as the determining criteria. This proves that the ASTM D 2863-76 requirements are well founded.

This criteria should be incorporated into the proposed Standard. The combustion time, however, should always be measured as well, since in conjunction with the burnt-off length it offers an indication of flame propagation. Melting, dripping, shrinking, charring, etc. of the test piece should also be recorded in the test report. The portion of the test piece below the reference point should in all cases be undamaged.

The devices used at our laboratory had a regulation and adjustment accuracy of only 0.5% by volume. It may, therefore, be possible that at a preset O_2 concentration only such areas burned which were heated by the flame and then extinguished themselves a short time after flame removal. They might have continued burning by an increase of 0.5% by volume. The minimum LOJ value must then be established by averaging the values.

6. Measuring accuracy and reproducibility

The measuring accuracy depends on the sensitivity of the flow measurement system and capability of precise adjustment of flow quantities of oxygen and nitrogen. The ratio of oxygen in the mixture also plays a role.

Test devices where indication is given by an oxygen analysator have a measuring and reading accuracy based on a scale of 0.1 ...0.2% by volume. Rotameters used by us only offered a setting of 1 mm intervals of flow through levels corresponding to an oxygen index accuracy of 0.5% LOJ value. Repeat tests as well as equipment inspection tests to check for constancy and leakage of the complete system showed a divergence of measuring values as follows:

for compact material and foam up to 0.5% of LOJ value
for flat pieces and foils up to 1.0% of LOJ value.

Reproducibility and measuring results can also be negatively influenced by inhomogeneities, dripping, bending and uneven burning marks of the test piece. If large divergencies are noted then the average values have to be calculated from repeated tests.

Table 1 lists the measuring divergencies of our tests. Tests on four different configurations are shown in columns 1 and 2. This data is compared in columns 3 and 4 with the permitted divergence factors as per ASTM D 2863-76.

A comparison of divergencies from an improved device in our laboratory and devices available at the ASTM facility shows maximum fluctuations of 0.4% of the LOJ value (compare column 1 and 3). Comparing the values of several laboratories (compare column 2 and 4) showed large divergencies from the ASTM data. We may, therefore, conclude that the existing and improved device at our laboratory - despite an inferior reading accuracy - can achieve a similar reproducibility of the measuring value.

Table 1. Comparison of known and calculated measuring divergencies (%)

Test piece	Column 1	Column 2	Column 3	Column 4
	max.div.of meas.results		Data from ASTM D 2863-76	
	in our lab with same device	4 other labs with diff. devices	max. div. in one lab	max. div. at several labs
Solid body LOJ $<21\%$	0.5	0.25 - 0.5	0.1 - 0.3	0.4
Solid body LOJ $>21\%$	0.5	0.5 - 1.5	0.1 - 0.3	0.7 - 1.4
Foam	0.5	0.5 - 1.5	0.1 - 0.3	0.4 - 1.5
Foils and flat pieces	0.5 - 1.0	0.5 - 1.5	≤ 0.6	0.5 - 1.4

Literature

1) Fenimore, C.P., and Martin, F.J., Mod. Plastics, Vol. 43 (1966) Nov. P. 141.

2) Engineering standard: TGL-Proposal 25253/01 - checking of plastic materials, determination of flammability, oxygen index procedure, issue of April 1975.

3) Recommendation for Standards: RS 4168-73 - Plastic materials determination of combustion behavior as per oxygen index of December 1973.

4) Standard: ASTM D 2863-76 - Standard procedure for measuring the minimum oxygen concentration to support the candle-like combustion of plastic material (oxygen index). Translation.

5) Stanton-Redcroft, London/Great Britain, Oxygen Index device, Company prospectus 1978.

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16. Abstract

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