A General Correlation of the Flammability
of Natural and Synthetic Polymers*

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Synopsis

The concept that flammability is fundamentally related to the potential thermal energy available per unit of volume of material emerged from attempts to correlate the effect of composition variables on the flammability of neoprene vulcanizates as measured by the oxygen index (O.I.) test. The origins of this test clearly show that it is a highly specific measure of flammability—the tendency of a composition to continue to burn once ignited—and that it is thermodynamically related to the heat of combustion of materials. This relationship is developed to a linear correlation which includes a wide variety of synthetic and natural materials and permits reasonable prediction of O.I. values from elemental analysis. Polymeric materials containing carbon and oxygen in atom ratios of less than 6 to 1 are more flammable than predicted. The effect of atmospheric temperature on O.I. can be predicted in relation to the O.I. value at normal temperature. This effect is shown to be independent of the composition of the material being tested. These two correlations permit the construction of a simple general map of flammability against which experimental data can be compared and judgments made with respect to the significant variables involved. There appears to be a significant relation between O.I. data, as viewed from these correlations, and the data of other flammability tests.

BURNING CHARACTERISTICS OF
POLYCHLOROPRENE COMPOSITIONS

Polychloroprene has long been known as an elastomer with superior flame-resistant characteristics. Measurements of its flammability have been discussed before.[1-3] A recent study[4] examined the effects of composition on the oxygen index (O.I.) (the minimum fraction of oxygen in the test atmosphere which will just support a flame) for these materials.[5,6] Consideration of the effect of different fillers, plasticizers, and waxes on the flammability of slab vulcanizates which ranged from O.I. values as low as 0.28 to values as high as 0.573 required a fundamental correlation before a sound understanding could be achieved.

The compositions involved in this study are shown in Table I where the major variables concern the use of calcium carbonate, clay, carbon black, hydrated aluminum oxide, and antimony oxide to produce a variety of effects.

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TABLE I

Oxygen Index Values for Various Neoprene Vulcanizates in Relation to Compound Variables

Compound reference (1947-)	Fillers[b]	Polymer HCl, equiv./100 of cpd.	Equiv. acid Acceptor present	Net HCl equiv.	HCl found[a]	CO_2 $+H_2O$, moles	Combustible, %	ΔH Comb., kcal/g	O.I.	1/O.I.
3.	50 clay, 40 black, 19 oil + wax	0.505	0.14	0.36	(0.24)	—	55.	5.1	0.359	2.79
6.	gum vulcanizate	1.00	0.28	0.72	—	—	56.	5.4	0.410	2.49
7.	35 fine $CaCO_3$	0.77	0.70	0.07	—	0.35	42.7	4.1	0.290	3.45
8.	55 fine $CaCO_3$	0.67	0.85	-0.18	0	0.42	37.6	3.6	0.298	3.36
9.	75 fine $CaCO_3$	0.60	0.98	-0.38	(0)	0.50	33.6	3.2	0.305	3.28
10.	55 coarse $CaCO_3$	0.67	0.85	-0.18	(0)	0.42	37.6	3.6	0.327	3.06
11.	55 fine $CaCO_3$ + 78 clay + 10 TCP	0.44	0.56	-0.12	(0)	0.22	27.4	2.6	0.285	3.51
12.	55 fine $CaCO_3$ + 73 $Al_2O_3 \cdot 3H_2O$ + 10 TCP	0.45	0.85	-0.40	0	0.82	28.0	2.7	0.415	2.41
13.	55 fine $CaCO_3$ + 54 black + 10 TCP	0.485	0.85	-0.36	(0)	0.24	53.0	4.7	0.299	3.34
14.	20 clay, 302 $Al_2O_3 \cdot 3H_2O$, 15 Sb_2O_3	0.62	0.32	0.30	—	0.33	37.2	3.5	0.505	1.96
15.	104 clay, 5 black, 15 Sb_2O_3 + 10 TCP	0.46	0.25	0.21	—	—	30.4	2.9	0.515	1.94
16.	104 clay, 5 black, 15 Sb_2O_3 + 8 Sundex	0.46	0.25	0.21	0.124	—	31.0	3.0	0.536	1.86
17.	5 black, 97 $Al_2O_3 \cdot 3H_2O$, 15 Sb_2O_3 + 10 TCP	0.47	0.26	0.21	—	0.82	31.4	3.0	0.573	1.73
18.	52 clay, 41 black, 15 Sb_2O_3 + 10 TCP	0.49	0.27	0.22	(0.12)	—	48.3	4.3	0.458	2.18
19.	52 clay, 41 black, 15 Sb_2O_3 + 8 Sundex	0.50	0.27	0.23	—	—	49.0	4.4	0.446	2.24
20.	52 clay, 41 black, 8 Sundex	0.53	0.15	0.38	0.24	—	50.2	4.7	0.461	2.17
Neoprene foam	30 $Al_2O_3 \cdot 3H_2O$, 5 Sb_2O_3, 10 organic	0.71	0.18	0.53	—	0.37	47.6	4.7	0.357	2.80

[a] Based on IRATE analysis, equiv./100 of cpd. values in parentheses assumed by analogy to compounds 8, 12, 16, or 20.

[b] Additionally, stock contained 4 phr MgO, 5 ZnO, 2 Antiox, 1 NA-22, and carbon black and process oils or plasticizer. Numbers are parts per hundred of neoprene.
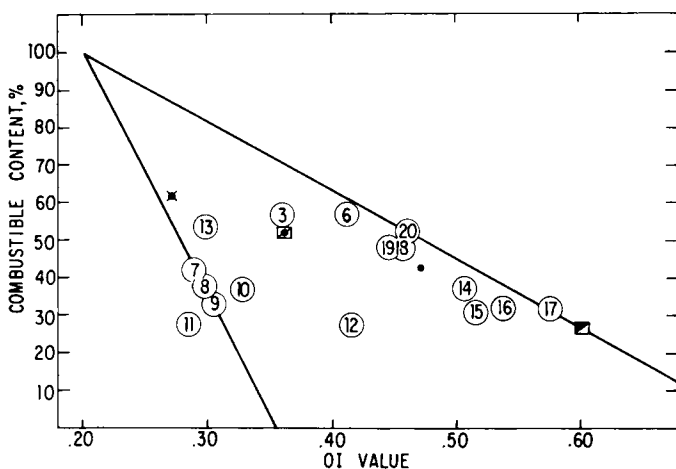
Fig. 1. Relation of combustible content to oxygen index of halogen-containing polymers: circled numbers = neoprene compositions, Table II, (●) rigid poly(vinyl chloride); (◉) phosphate ester-plasticized poly(vinyl chloride); (✗) dioctyl phthalate-plasticized poly(vinyl chloride); (◪) poly(vinylidene chloride); (□) neoprene foam cushion.

It occurred to us that the flammability of these compositions should be in some way related to the concentration of combustible material (in general, the total concentration of carbon + hydrogen) in the composition. Consideration was also given to the amount of HCl which could be evolved from the vulcanizate, which varied depending on the amount of acid acceptor present, and to the generation of carbon dioxide and water from appropriate fillers.

The correlation of per cent combustibles for these neoprene compositions (Table I) versus the O.I. values is shown in Figure 1. It is apparent that a wedge-shaped area is defined by the data points. The lower or left-hand boundary represents those neoprene compositions containing an excess of small particle size calcium carbonate over that required to absorb all the HCl potentially available from the polymer. The upper or right-hand boundary is defined by those compositions which were demonstrated to release HCl copiously. Most practical neoprene compositions will be within the indicated range as, for example, a practical neoprene wire covering, compound 3, and a commercial neoprene foam.

The fact that other halogenated polymers such as rigid poly(vinyl chloride) and poly(vinylidene chloride) also were close to the upper line was the first indication that a broad correlation might be available.

CORRELATION OF OXYGEN INDEX VALUES WITH SPECIFIC HEAT OF COMBUSTION

Further consideration of the concept of the per cent combustibles convinced us that flammability must be significantly related to the potential heat energy available per unit volume or per square centimeter of solid surface. Calculation of the heat of combustion by standard techniques

TABLE II
Oxygen Index and Specific Heat of Combustion of Polymeric Materials

Material	O.I.	1/O.I.	ΔH_{comb}, kcal/g[d]
Polyoxymethylene	0.15[b]	6.66	4.05
Polyacrylonitrile	0.169	5.93	7.3
Polybutadiene (poly BD)	0.183	5.45	10.78
Polyethylene	0.184[b]	5.45	11.1
Cotton	0.184	5.45	4.0
Polystyrene (PS)[a]	0.192	5.21	9.9
PS + 5% ST-23P (trisdibromylpropyl phosphate)[a]	0.232	4.31	9.7[c]
PS + 10% ST-23P (trisdibromylpropyl phosphate)[a]	0.252	3.97	9.25[c]
PS + 15% ST-23P (trisdibromylpropyl phosphate)[a]	0.288	3.48	8.83[c]
Poly(vinyl alcohol) (PVA)	0.225	4.45	5.98
Poly(ethylene terphthalate) (PET)	0.227	4.40	5.3[c]
Nylon	0.239	4.18	7.5
Wool	0.265	3.17	6.35[c]
Exp. urethane foam (du Pont)	0.25–.0.28	3.57	7.5[e]
Polycarbonate (Lexan)	0.27	3.70	7.4
Aromatic polyamide (APA)	0.286	3.50	6.85
Hypalon	0.27	3.70	6.80[c]
Neoprene (gum)	0.40	2.50	5.8[c]
Neoprene foam (Toyad Co.)	0.36	2.78	4.7
Rigid poly(vinyl chloride) (PVC)	0.47	2.13	4.34
Poly(vinylidene chloride)	0.60[b]	1.66	2.52
Poly(tetrafluoroethylene) (PTFE)	0.95	1.05	1.0

[a] Data of DiPietro & Stepnicka.[9]
[b] Data of Fenimore & Martin.[5]
[c] Calculated based on a composition.
[d] Experimental values reported in literature or determined.
[e] Calculation of P. S. Bauchwitz.

showed there was indeed an almost proportional relationship between the per cent combustibles and the specific heat of combustion of the neoprene vulcanizates of Table I.   The plot of these data shown in Figure 2 is substantially identical to the plot of Figure 1.

The question of where other synthetic or natural polymers might fall on a similar O.I. versus heat of combustion plot led to the data summarized in Table II where the oxygen index values are taken from published literature. Initial attempts to plot these data demonstrated a strong correlation but led to curves which had only empirical meaning.

Consideration of the basic concepts involved led to the conclusion that there was probably a hyperbolic relationship between the heat of combustion and the O.I.   The need for a reciprocal plot became obvious, and the correlation shown in Figure 3 resulted.   It is apparent that 14 synthetic or natural unfilled polymers, covering a wide range of flammability and chemical compositions, fall sufficiently close to a straight line to warrant the conclusion that the specific heat of combustion of solid materials and their oxygen index are fundamentally related.
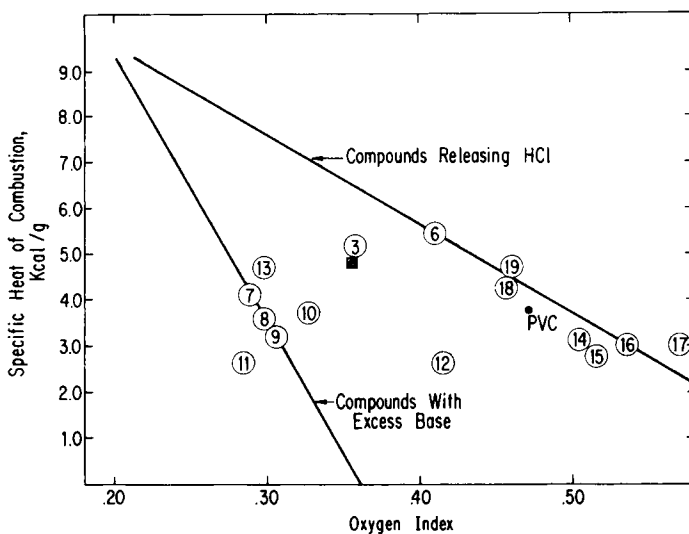
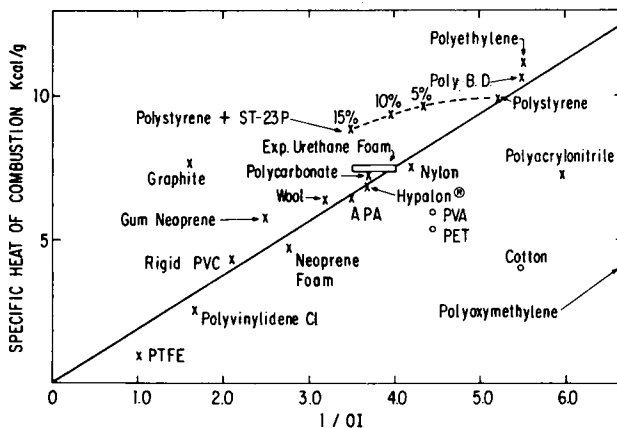Fig. 2. Specific heat of combustion of neoprene stocks vs. oxygen index.

Fig. 3. Correlation of specific heat of combustion with reciprocal of oxygen index (see Table II for abbreviations).

In a more rigorous treatment, the correlating parameter should not be the specific heat of combustion but the ratio of the specific heat of combustion to the specific heat of vaporization of the combustible. This ratio is very essential to the transfer number B, and it is well established that the burning rate is proportional to ln $(1 + B)$ or, for small B, proportional to B alone.

The polymers falling well below the line are polyacrylonitrile and the four highly oxygenated polymers poly(vinyl alcohol); poly(ethylene terephthalate), cotton, and polyoxymethylene. Oxygenated polymers having a carbon-to-oxygen ratio of 6 or higher fit the correlation. The other oxygenated polymers are displaced from the line in the same order as their

carbon-to-oxygen ratio decreases.   Polyacrylonitrile is known to have an exothermic thermal decomposition mechanism.   For most polymers, addition of fillers, as already shown for neoprene, alters the O.I. values in irregular ways.

ORIGINS OF THE OXYGEN INDEX TEST

It has long been recognized that combustion requires fuel, oxygen, and heat.   In considerations used in many flammability tests, the nature and composition of the fuel has been given the greatest amount of study.   The application of flame retardants to synthetic polymers and fabrics is an example of emphasis on the fuel component of the fire triangle.   The effects of the variation in oxygen concentration or the effects of added heat on the flammability phenomenon have been considered in relation to power generation, but they have been given relatively little attention in the flammability testing of plastics and fabrics until recently.

Flaming combustion requires the presence of a fuel–air mixture whose composition lies within the explosive limits for that particular system.   A flame, therefore, becomes a standing explosion wave the velocity of which is sharply influenced by the ratio of fuel to oxygen in the mixture.   This is true for both premixed (as in a Bunsen burner) and for diffusion flames where the combustible mixture is created by normal diffusion and turbulence of the fuel vapor and the oxygen-containing gas.   In studies of diffusion flames, it has been shown by Simmons and Wolfhard[7] that the flame temperatures of diffusion and premixed flames are substantially equivalent.

It is interesting to note that the oxygen index test was first used[7] to study the effects of oxygen concentration on the flammability of gaseous and volatile fuels.   An extensive study by Coward and Jones[8] for fuels ranging from hydrogen, hydrocarbons, alcohols, and many common solvents had shown that as air is diluted with nitrogen, the explosion limits for the fuel are narrowed and that at the limiting dilution the fuel-to-oxygen ratio in the system is very nearly stoichiometric (Fig. 4).   Simmons and Wolfhard recognized this fact and showed that these fuels are characterized by a
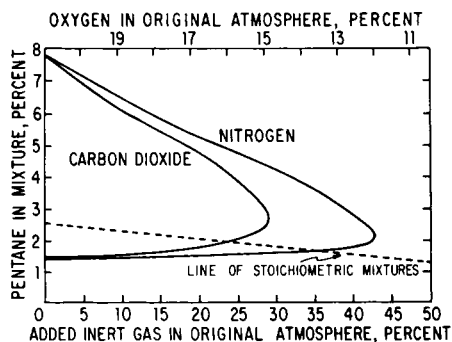

Fig. 4. Limits of flammability of pentane in mixtures of air and nitrogen, and air and carbon dioxide.   Data of Coward and Jones.[8]

TABLE III
Oxygen Index and Limiting Temperature of Volatile Fuels[a]

	O.I.	1/O.I.	ΔH_{comb}, kcal/mole	ΔH_{comb}, kcal/g	Sp. heat, cal g/°C	Limiting temp., °C
Hydrogen	0.054	18.5	68.4	34.2	3.39	811
CO	0.076	13.2	67.9	2.42	0.25	1177
C_2H_2	0.085	11.8	312	12.0	0.383	1271
CH_3OH	0.111	9.0	170.9	5.35	0.390	1285
C_2H_4	0.105	9.52	332	11.8	0.359	1335
C_2H_5OH	0.126	7.95	328	7.1	0.406	1405
C_3H_8	0.127	7.87	526	12.0	—	1922
C_6H_6	0.133	7.54	787	10.0	0.32	1540
$(CH_2)_6$	0.134	7.46	939	11.2	0.413	1495
CH_4	0.139	7.20	210.8	13.2	0.528	1518
C (graphite)[b]	0.635	1.58	94.3	7.7		—

[a] Original data by Simons and Wolfhard.[7]
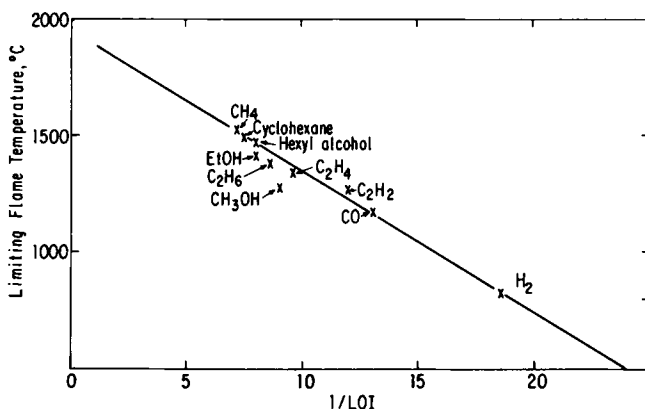[b] Data of Fenimore and Martin.[5]

Fig. 5. Limiting flame temperature of fuels vs. reciprocal of oxygen index. Data from Simons and Wolfhard.[7]

limiting flame temperature which could be calculated from the oxygen index and the stoichiometric quantity of oxygen required for complete combustion. The limiting flame temperature is calculated from the molar heat of combustion and the heat capacity of the nitrogen and oxygen at the limit. This limiting temperature represents the lowest flame temperature required to maintain flammable combustion. Their O.I. and limiting temperature data are summarized in Table III. A plot of their limiting flame temperature versus the reciprocal of the oxygen index is shown in Figure 5. This is in reality a plot of potential heat energy per unit volume available in a gaseous fuel versus a function of the oxygen index. Because we are dealing with gases the molar heat of combustion is a measure of the thermal energy available per unit volume. As the density of a fuel increases from gas to liquid to solid, it becomes necessary to use the specific heat of com-
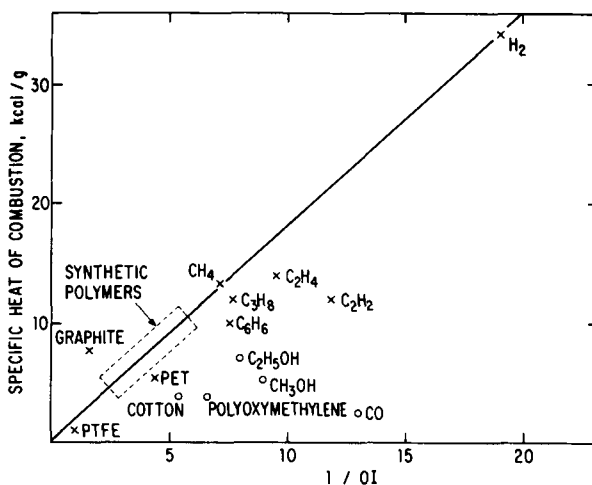
Fig. 6. Correlation of oxygen index and specific heat of combustion for gases, liquids, and polymers.

bustion as the measure of available energy, rather than the molar heat to achieve a similar correlation.

Curiosity led us to plot the oxygen index values of the volatile fuels, using the specific heat of combustion versus 1/O.I. plot of Figure 3. This plot (Fig. 6) places methane and hydrogen on the same straight line as the polymers of Figure 3. Note also that the oxygenated fuels are off the curve in the same manner as poly(ethylene terephthalate) or cotton.

Simmons and Wolfhard were very careful to note that the oxygen index measures the limiting conditions for the combustion of a stoichiometric mixture of fuel and oxygen and that this value represents the limit of conditions necessary to maintain a flame. For solid materials, O.I. values must similarly measure the limiting conditions for flaming combustion of a near stoichiometric mixture of pyrolysis vapors and oxygen diluted with nitrogen. O.I. values are therefore fundamental measures of flammability (i.e., the tendency of a material to continue to burn, once ignited), and they are not related to the ignition process. (The author's definition of flammability is somewhat broad. By a strict definition of flammability one should mean the rate at which a flame propagates across a flammable surface. The O.I. test, then, is related to the burning rate of a given combustible.)

Since O.I. is related to stoichiometric compositions and since in a practical system explosive limits are generally entered by approach from the low side, it is a necessary conclusion that ignition of a material may occur at a lower oxygen concentration than indicated by the O.I.

EFFECT OF TEMPERATURE ON OXYGEN INDEX

Recent efforts to consider the effect of increased ambient temperatures on flammability has shown, as expected, that flammability increases with
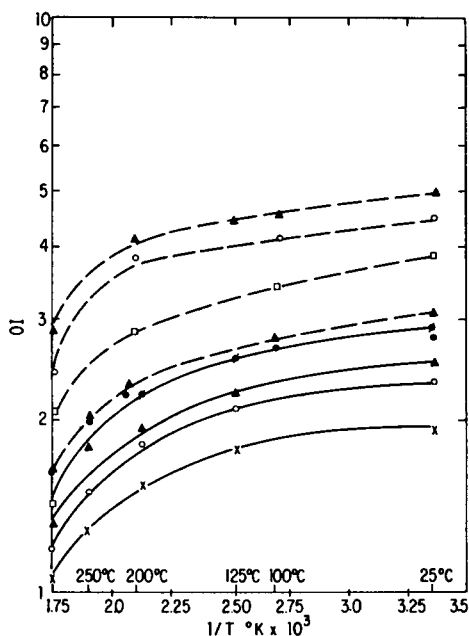
Fig. 7. Effect of atmospheric temperature on oxygen index: (——) neoprene compositions; (— —) polystyrene and flame-retarded polystyrene compositions. Data of DiPietro and Stepniczka.[9]

temperature. The effect of temperatures on the neoprene compositions of Table I was studied by preheating the nitrogen–oxygen mixtures in the O.I. apparatus. The resultant data are tabulated in Table IV. Attempts to correlate these data using a variety of parameters, including an Arrhenius plot, failed to provide any indication of logical relationship. However, the similarity in slopes of the Arrhenius plot between a variety of neoprene compositions and data of De Pietro and Stepniczka (Fig. 7) led to tabulation of the data (Table V) in terms of the per cent of the room-temperature O.I. retained at some higher temperature.

The data shown in Table V is remarkable in that it considers 28 different polymeric compositions having room-temperature O.I. values ranging from 0.10 to 0.50 and including polymers containing chlorine, ester groups, nitrile groups, and a variety of flame retardants, yet the per cent change in O.I. is a constant for the temperatures indicated. We can only conclude that increasing the temperature of the atmosphere produces effects which are totally independent of the composition under test.

We have attempted to correlate these average values with the temperatures in a variety of ways. The only satisfactory correlation we have achieved is that shown in Figure 8, where the values of the constant at 100°, 200°, and 300°C are linearly proportional to the 3/2 power of the absolute temperature. This relationship was suggested because the temperature coefficient of diffusional processes is frequently correlated in this

TABLE IV
Effect of Temperature of Atmosphere on O.I. of Neoprene Compositions

Compound no.	Description of additives	O.I.[a]			
		25°C	100°C	200°C	300°C
3	clay, black, oil	.325	.284	.227	.157
6	gum vulc.	.264	.233	.192	.164
7	fine $CaCO_3$	.276	.250	.191	.153
8	fine $CaCO_3$	.269	.239	.187	.149
9	fine $CaCO_3$	.264	.233	.186	.146
10	coarse $CaCO_3$	.280	.269	.220	.164
11	fine $CaCO_3$ + clay	.307	.276	.227	.183
12	fine $CaCO_3$ + $Al_2O \cdot 3H_2O$	.387	.342	.284	.197
13	fine $CaCO_3$ + black	.276	.250	.190	.160
14	clay, $Al_2O_3 \cdot 3H_2O$–Sb_2O_3	.449	.414	.387	.244
15	clay, Sb_2O_3	.414	.387	.287	.200
16	clay, Sb_2O_3	.414	.387	.290	.205
17	$Al_2O_3 \cdot 3H_2O$, Sb_2O_3	.499	.460	.414	.287
18	clay, black, Sb_2O_3	.357	.309	.280	.233
19	clay, black, Sb_2O_3	.449	.414	.387	.209
20	Clay, black	.325	.284	.239	.189

[a] The samples used in these tests were thinner than recommended by ASTM D-2863. This accounts for the differences in 25°C O.I. values listed here vs. Table I.
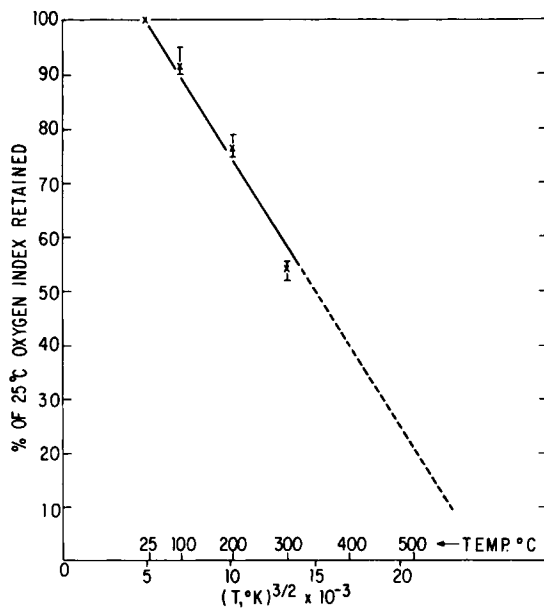

Fig. 8. Effect of temperature on oxygen index.

TABLE V
Per Cent of 25°C O.I. Retained at Higher Temperatures

Polymer samples	Average % (± standard deviation)		
	100°C	200°C	300°C
16 Neoprene	90.2 ± 2.8	74.5 ± 5.9	55.1 ± 5.0
4 Polystyrene[a]	93.1 ± 1.7	78.6 ± 1.6	52 ± 2.0
4 ABS[a]	95.3 ± 3.7	79.1 ± 3.2	53.0 ± 3.4
4 Polyester–styrene[a]	92.5 ± 2.2	80.6 ± 2.0	55.3 ± 4.3

[a] Original data of DePietro and Stepniczka.[9]

TABLE VI
Calculation of Temperature at Which a Given 25°C Oxygen Index
Will be Reduced to 0.21

O.I. measured at 25°C	Retention of O.I., %[a]	$(T)^{1/2} \times 10^{-3}$, °K[b]	Temp. (T)	
			°K	°C
0.21	100	5.15	298	25
0.25	89	7.2	373	100
0.30	70	10.8	489	216
0.35	60	13.0	553	280
0.40	52.5	14.5	595	322
0.45	46.7	15.5	622	349
0.50	42.0	16.5	648	375
0.55	38.2	17.2	666	393

[a] 0.21/Measured O.I. × 100.
[b] Temperature function from Fig. 8 for indicated % retention of O.I.

manner.   It certainly indicates that diffusional processes are more important than the chemical activation of pyrolysis which would only plot logarithmically.

This temperature effect is also reflected in the long-recognized fact that explosive limits of a fuel–air mixture broaden as the temperature goes up. For most volatile fuels there is a straight-line relation between the temperature and the flammability limit.

If extrapolation of the plot in Figure 8 to higher temperatures is justified, it becomes apparent that there will be few carbon containing materials capable of resisting ignition and burning at temperatures between 400° and 500°C.   It also quantifies the observations that it is increasingly difficult to maintain flame retardancy as temperatures increase up to the maximum use temperature of organic polymers.

The knowledge of this temperature effect now permits application of a temperature scale to Figure 3, as shown in Figure 9.   This map indicates that those materials having oxygen index values of 0.28 at room temperature will have the O.I. value of normal air at a temperature of 200°C and that those materials having a room temperature O.I. oxygen index value of 0.38 will have an atmospheric O.I. value at 300°C.
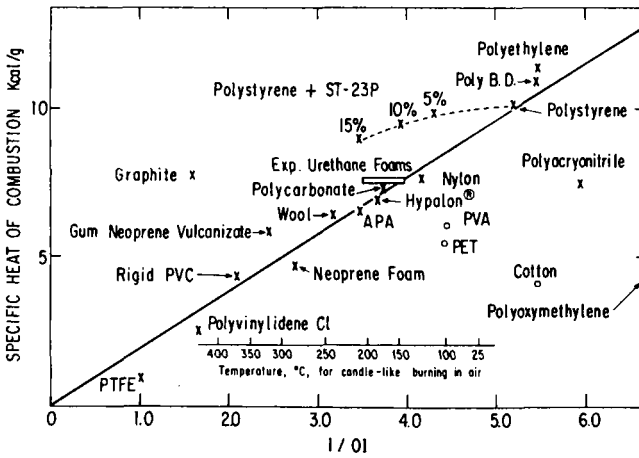
Fig. 9. General map of oxygen index of materials and temperature effect scale (see Table II for abbreviations).
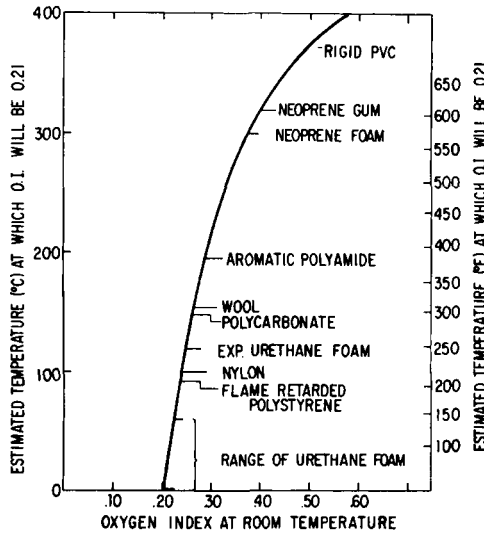

Fig. 10. Temperature for candle-like burning in air.

Figure 8 also permits estimation of the temperature at which any given oxygen index (measured at room temperature) will be reduced to 0.21. In other words, this will be the temperature at which the flammability of a material with a given O.I. will permit candle-like burning in ordinary air. These calculations are presented in Table VI and presented graphically in Figure 10. The position of various polymers having O.I. values greater than 0.21 have been indicated along this curve to provide specific examples of ranges of flammability of well-known materials.

The independence of temperature and composition effects demonstrated by Table IV, we believe, should permit correlation of O.I. values with per-
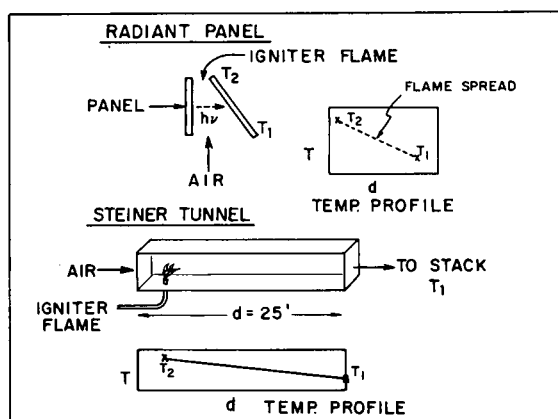
Fig. 11. Relationship of oxygen index to radiant panel and Steiner tunnel test.

formance of materials in any other flammability test where a temperature gradient is imposed on the test sample. Thus, for example, in the "Radiant Panel" test, shown schematically in Figure 11, where radiant energy is imposed on the sample with ordinary air passing across the surface, we believe flame spread will occur to that point on the temperature profile of the sample where the temperature is such to give the sample an O.I. value of 0.21.

Similarly, in the Steiner tunnel test, where the large pilot flame imposes a temperature gradient on the 25-ft-long sample while consuming about 20% of the oxygen, we believe flame spread will occur to that point on the temperature profile where the temperature is equivalent to an oxygen index value of 0.17.

CONCLUSIONS

We believe the following points have been established:

1. The oxygen index (O.I.) test measures the lowest flame temperature at which a stoichiometric mixture of fuel vapor (volatile pyrolysis products) nitrogen and oxygen will continue to burn. It is fundamentally related to the specific heat of combustion of most materials. It is a measure of flammability—the tendency of a material to continue to burn once it has been ignited.

2. The O.I. of many common materials can be reasonably predicted by the equation:

$$\text{O.I.} = 1.9/\text{sp. } \Delta H_{\text{comb}}.$$

3. Since O.I. deals with a substantially stoichiometric flame, it provides a precise definition of flammability. Ignitability, which deals primarily with flame phenomena at the explosive limits, will be broader than the O.I.

4. Polymeric materials containing carbon and oxygen in ratios of less than 6 are more flammable than predicted, and the lower the C/O ratio is, the lower the O.I., regardless of the heat combustion.

5. The effect of atmospheric temperature on O.I. can be generally predicted in relation to the O.I. value at normal temperature. The retained O.I. will be 92%, 78%, and 55% of the 25°C value at 100°, 200°, and 300°C, respectively. The effect of temperature is independent of the composition of the material. This permits estimation of the temperature at which a material will burn in ordinary air.

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