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Assessment of smoke toxicity of complex products using the steady state tube furnace (ISO/TS 19700)

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ABSTRACT

The ISO/TS 19700 steady state tube furnace is a bench-scale method for assessing smoke toxicity of materials. It allows the ventilation conditions to be controlled during the test, allowing the toxicity of each fire stage to be assessed. Currently, the scope of ISO/TS 19700 limits its application to testing of "homogeneous thermoplastics". This work investigates extensions to its use to more complex commercial products (plasterboard, energy cable, wood-based boards and flexible polyurethane foam). In order to achieve results representative of specific fire stages, certain extensions to the protocol were made. These include adjusting feed rate of the sample; adjusting the mass load; increasing the ventilation of preliminary runs; and provision for reporting data for flaming fire stages for essentially non-combustible samples. The products were tested in smouldering; and well-ventilated, small under-ventilated and large (post-flashover) under-ventilated flaming conditions, as defined in ISO 19706. With these adjustments it was found that the steady state tube furnace was able to assess the toxic product yields of more complex products, of unknown composition.

1. Introduction

In the 1960s, UK fire deaths increased, due to the shift from traditional materials to synthetic polymers, which are more flammable and produce more toxic smoke when burning. In response to this, in the 1980s the government implemented regulations limiting flammability of domestic products [1,2,3]. While those regulations coincided with a reduction in the overall number of fire deaths, the main causes of death and injury shifted from burns to inhalation of smoke and toxic gases. Smoke toxicity remains unregulated in furniture and construction products, but is regulated in mass transport, as it is accepted that victims may not be able to escape from those settings. The same argument can be applied to high-rise buildings, or buildings with elderly or mobility-impaired occupants. This paper investigates the assessment of smoke toxicity for four commonly-used heterogeneous products of unknown composition. With a robust means of quantification, smoke toxicity assessment could be applied to such products in the same way as flammability, for example through the Euroclass system.

Fires may grow through certain stages: smouldering, ignition, well-ventilated flaming, fully-developed under-ventilated flaming, flashover and finally decay. Research predicting the carbon monoxide evolution from flames of simple hydrocarbons, reviewed by Pitts [4], has shown the importance of the equivalence ratio, ϕ, for predicting the CO yield from the oxygen depletion in flaming conditions. For many hydrocarbon polymers, CO yield increases rapidly with increase in ϕ, almost independent of polymer [5]. In addition, a close correlation between CO formation and HCN formation has been established in studies of full-scale fires [6,7], as the formation of both species appear to be favourable under the same poorly ventilated fire conditions.

ISO 19706:2011 defines a number of stages by their oxygen (O_2) concentrations, temperatures, carbon dioxide (CO_2) and carbon monoxide (CO) concentrations, and other parameters [8]. Out of those, the four most significant stages in smoke toxicity are shown in Table 1.

The equivalence ratio ϕ is defined by Eq. (1) for flaming combustion. It is a dimensionless value expressing availability of O_2 for complete combustion of the material.

$$\phi = \frac{actual fuel/air\ ratio}{stoichiometric\ fuel/air\ ratio} \tag{1}$$

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Table 1
Key fire stages for smoke toxicity assessment, defined in ISO 19706 [8].

Fire stage		ϕ	$[CO]/[CO_2]$
Non-flaming:	1b Oxidative pyrolysis, including smouldering	-	0.1 – 1
Flaming:	2 Well-ventilated	$\ll 1$	<0.05
	3a Under-ventilated	$\gg 1$	0.2 – 0.4
	3b Large under-ventilated/post-flashover	$\gg 1$	0.1 – 0.4

In the presence of sufficient fuel, an enclosure fire will grow until it is ventilation controlled, becoming under-ventilated. If there is sufficient fuel and the enclosure is large enough, the fire continues to flashover.

In flammability testing, the typical worst-case scenario is well-ventilated flaming, where the oxygen concentration is greatest. In smoke toxicity assessment, the toxic product yields typically increase by a factor of 10 to 50 following the transition from well-ventilated to under-ventilated flaming [9] as $\phi > 1$. In order to provide data suitable for hazard assessments, it is preferable to isolate specific fire stages, to be able to predict the impact of each stage on the smoke toxicity.

During smouldering, the highest toxic product yields are often recorded, because the absence of a flame results in a high concentration of products of incomplete combustion. As the rate of mass loss during non-flaming combustion is usually very low, this stage does not usually present a significant hazard. Once ignition occurs, the mass loss rate increases dramatically, while the main products are CO_2 and water. When the fire becomes under-ventilated, the yields of toxic products such as CO and hydrogen cyanide (HCN) increase [9].

The factors affecting fire smoke toxicity [10–12], have been investigated in terms of the yields of toxic gases and other combustion products (smoke, particulates) [13,14] as a function of fire condition for a wide range of polymeric materials. For smoke toxicity, the most important fire stages are the under-ventilated (stages 3a and 3b) because they yield the largest quantities of toxic effluents.

Smoke toxicity is driven by four factors, listed roughly in order of importance:

1. **The flammability of the material.** The more it burns the more smoke and toxic gases will be produced.
2. **The presence of a flame.** Non-flaming fires produce small quantities of highly toxic smoke but flaming fires produce much larger volumes of usually less toxic smoke.
3. **Ventilation condition.** As the availability of O_2 decreases, so the smoke will contain more products of incomplete combustion. These include CO, hydrogen cyanide (HCN), organics and particulate matter. Most fire deaths result from inhalation of smoke from under-ventilated fires [2].
4. **The material composition.** If the material contains any hetero-elements, such as nitrogen or halogens, other toxic gases will be produced. When nitrogen is present in the fuel, HCN is often produced, which is around 20 times more toxic than CO. Addition of halogens or organophosphates leads to production of irritant gases, but it also affects the chemistry in flames. Halogens and organophosphates are gas-phase inhibitors, which hinder the flame reactions, increasing the yields of all products of incomplete combustion including CO, HCN, organics and smoke.

Most bench-scale methods used for generation of toxic effluents are based on flammability tests, and can only reproduce a single well-ventilated fire stage or combustion condition. Those bench-scale methods in closed chambers, derived from the smoke density chamber (SDC) ISO 5659:2017 [15] where the sample is exposed to a fixed heat flux, do not have steady state combustion conditions, as the oxygen concentration decreases during the test, while static tube furnace tests, such as the NF X 70-100, the sample temperature, and hence the rate of pyrolysis, increases during the test. The controlled atmosphere cone calorimeter [16] (CACC) ISO/TS 5660-5:2020, and the Fire Propagation Apparatus (FPA) [17] (ASTM E 2058 and ISO 12136:2011) have approximately constant combustion conditions and the facility to control the ventilation condition.

The steady state tube furnace (SSTF) also has constant combustion conditions. It has been developed from a British Standard (BS 7990) [18] into the first international standard (ISO/TS 19700) [19] for bench-scale measurement of fire toxicity [20], specifically as a tool for fire safety engineering. It was found that ISO/TS 19700 currently offers the best means among the three test methods evaluated for conducting tests at pre-determined and controlled equivalence ratios, superior to the CACC or SDC [21].

The repeatability and reproducibility of the SSTF have been quantified [22,23], relating its yields to other national and international standards (the smoke density chamber (SDC), ISO 5659 modified for toxicity measurements as ISO draft DIS 21489, the French railway test NF X 70-100, the FPA and DIN 53436) [24] and finally to the limited comparable data available from large scale tests [25,26]. This work showed that under controlled conditions, robust quantification of fire toxicity was achievable.

The steady state tube furnace (SSTF) facilitates generation of fire effluents at various fire stages by forcing the sample to burn under pre-set ventilation conditions. Each of the fire stages described in ISO 19706 can be replicated individually using the equivalence ratio ϕ and CO/CO_2 ratio to specify the ventilation condition,. To test at specific equivalence ratio, the SSTF requires controlled air supplies and controlled feed rate of the fuel, in order to burn the sample in predetermined ventilation conditions.

The apparatus, shown in Fig. 1, consists of an 800 mm single-zone tube furnace with a 47 mm internal diameter quartz tube passing through it, from the sample inlet to the mixing chamber. This set up is compliant with the ISO/TS 19700:2016 standard. The sample and the primary air are introduced from the right-hand side.

The sample is driven in the furnace at a fixed rate, typically at 1 g min^{-1}. As it enters the furnace, the heat flux increases, until the sample reaches the critical heat flux for ignition, when it ignites and continues to burn. The flaming eventually stabilises, resulting in steady burning, called the steady state. The effluent flows through the tube leading to a mixing chamber where it is diluted with secondary air. The effluent sample collected during this steady state is representative of the selected fire condition.

The SSTF has been reported for the assessment of smoke toxicity of a wide variety of polymeric materials under different fire conditions. Published reports include investigations of hemp, flax and ramie fibres [27], polyurethane foams [28,29], acrylonitrile-butadiene-styrene (ABS) copolymers [30], and the effect of adding montmorillonite clay to ABS [31]. The method has been applied to wider groups of materials, including carpets [32], household components [33], and building materials such as wood and insulation [21,34–36]. The SSTF has also been used to assess industrial products typically stored in warehouses [37], and recently, the smoke toxicity of pesticides [38, 39] under different fire conditions. A model for predicting the toxicity of enclosures, by combining yields of toxicants from SSTF with flammability parameters, for use in fire hazard assessments, has been proposed [40]. A comparison of four methods for assessment of smoke toxicity, including the SSTF has been reported [41].

In addition, the SSTF has been used to generate particulate emissions as a function of fire stage from a PVC carpet and a wood-based board [42], and the smoke particle size distributions and morphology for wood and polypropylene [43].

This study investigates the suitability of the steady state tube furnace to assess the smoke toxicity of inhomogeneous and non-thermoplastic products. In addition, the standard was written to assess the smoke toxicity of combustible products, making it difficult to compare the contribution to smoke toxicity of products which do, and do not,

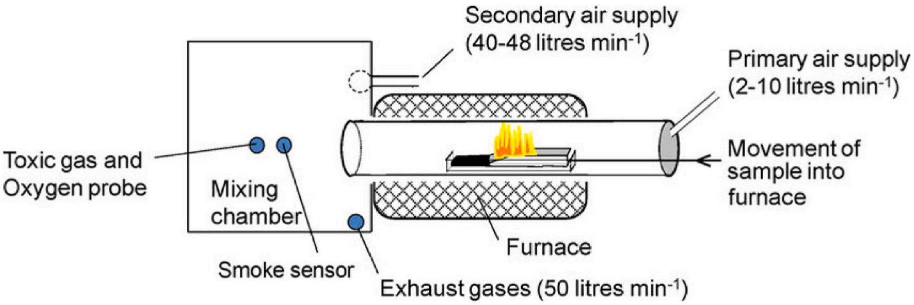

Fig. 1. Schematic of the steady state tube furnace, ISO/TS 19700.

undergo flaming combustion. Certain construction products, such as cables, are constructed with a fire protecting sheath surrounding a combustible interior. When such products are linearly homogeneous, they can be tested whole, giving a more realistic assessment of their actual fire behaviour.

2. Materials

Four samples were selected to represent the different types of complexity found in commercial products. Each was of unknown formulation, often containing more than one component. They were: plasterboard (or gypsum board), representing a non-combustible product with combustible wrapping; oriented strand board (OSB), an engineered timber product, chosen for its macroscopic composition of wood shavings and glue, and its tendency to char; a low-smoke, zero-halogen energy cable, representing a layered construction with a combustible core and a heavily fire retarded outer sheath; flexible polyurethane foam (PUF), a product with high flammability, low density (also showing very high smoke toxicity, and widely used in furniture products).

The materials were obtained from commercial manufacturers. Plasterboard was obtained from Gyproc®, Saint Gobain. The cable was supplied by Eland Cables. The OSB was obtained from Norbord, and the PUF was supplied by Carpenter ApS (which was made for the mainland European market, without the addition of flame retardants).

Of the four samples, only the cable consisted of multiple combustible components. Around 60% of the cable mass was copper conductor, which was included in the test, but remained unchanged and was disregarded from the toxic product yield calculation. The remaining 40 % of mass was the “plastic” cable components, of which the sheathing (believed to consist of ethylene-vinyl acetate and aluminium hydroxide fire retardant in approximately 1:2 ratio) made up 75.3%; and the inner insulation (consisting of cross-linked polyethylene) comprised the remaining 24.7%.

In order to better understand the products being tested, analysis of carbon, hydrogen and nitrogen was undertaken in duplicate on the materials, using the Flash 2000 Organic Elemental Analyser, Thermo Scientific, USA. The results are shown in Table 2. The nitrogen content of the plasterboard is unexpectedly high (the individual values were 6.82 and 7.75%).

Table 2
Elemental analysis.

Material	C %	H %	N %
Plasterboard - plaster	1.9	1.7	7.3
Plasterboard - outer	40.2	5.2	2.4
Cable - outer sheath	35.1	6.8	2.3
Cable - inner insulation	85.5	14.6	2.8
OSB	48.3	6.1	8.7
PUF	59.6	8.0	7.4

2.1. *Sample preparation*

Test specimens of each product were prepared for testing in the steady state tube furnace, as shown in Fig. 2. The lengths of the samples were varied in order to provide enough material to reliably obtain steady state burning for a minimum of 5 min, but also to create a uniformly burnt residue from which to quantify the mass loss. The plasterboard sheet was cut into 20 mm wide strips, with overall size 20 x 12 × 700 mm. This prepared sample retained both the paper wrappings on each side of the product, replicating its real-life application. The cable was cut into 600 mm long segments and tested without further preparation. The OSB was cut into 4 x 11 × 600 mm lengths. This was done to lower the combustible load of the sample. The PUF was cut into 25 x 50 × 700 mm strips, which were then bound with a fine steel wire to create a sample with higher density.

3. Methodology

The fire stages most relevant to smoke toxicity, investigated using the steady state tube furnace, are shown in Table 3. The standard specifies that for the test to represent a particular fire condition, the equivalence ratio and flaming requirement must be met. If flaming does not occur for stages 2, 3a or 3b, the furnace temperature can be raised in 25 °C steps, or lowered if flaming does occur for the oxidative pyrolysis (smouldering) stage. It is important to note that the specified furnace temperature is the measured temperature in the middle of the furnace. Most samples ignite at a lower temperature, before reaching the middle of the furnace.

The fire effluent composition is quantified during steady state burning, which should last for a minimum of 5 min. The standard specifies requirements for the stability of O_2 and CO_2 concentrations (expressed as volume fractions in ISO/TS 19700:2016) during the steady state.

The O_2 and CO_2 volume fractions must have a long-term trend which varies by less than $\pm 0.02\ \text{min}^{-1}$ (i.e. 2 % per minute) and short-term fluctuation, expressed as the standard deviation of the volume fraction divided by the average value of the volume fraction, over the 5 min period which is less than 0.20. Fig. 3 shows a well-ventilated cable test, alongside the range of the maximum standard deviation the concentrations are allowed to reach (shaded) and still be within the short-term fluctuation requirements.

In addition to measuring the O_2, CO_2 and CO, other components necessary for smoke toxicity assessment were quantified, such as acid gases and HCN. The ISO/TS 19700:2016 standard states “The concentrations of all effluent components (gases and respirable aerosols) that contribute significantly to the computation of the toxic hazard shall be measured during the steady-state test period. The methods of analysis shall conform to the specifications in ISO 19701 and ISO 19702”.

The gases measured were hydrogen chloride (HCl), hydrogen bromide (HBr), hydrogen fluoride (HF), nitrogen oxides (NO_x), sulphur dioxide (SO_2) and HCN. ISO 19701:2013 [44] specifies capturing of the gases in bubbler solutions, followed by analytical procedures. (ISO

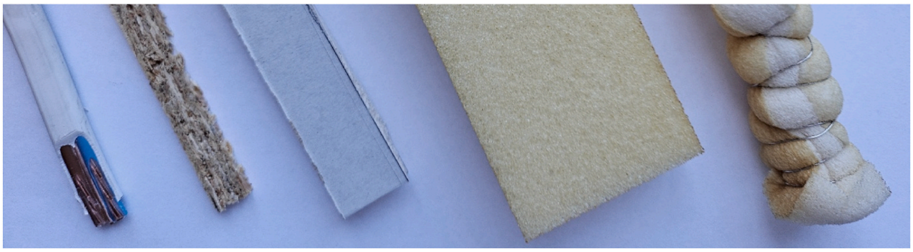

Fig. 2. Samples (from left: cable, OSB, plasterboard, PUF before binding, PUF after binding).

Table 3
Test conditions specified in ISO/TS 19700 to replicate each fire stage.

Fire stage	Furnace temperature/°C	Equivalence ratio ϕ	Flaming
1a Oxidative pyrolysis	350	-	No
2 Well-ventilated	650	0.5 – 0.75	Yes
3a Small under-ventilated	650	2.0 ± 0.2	Yes
3b Post-flashover (or large under-ventilated)	825	2.0 ± 0.2	Yes

19702:2024 specifies methods for gas phase Fourier transformation infrared analysis (FTIR) which was not used in this study, because of the problems of calibration and interference from water vapour.) Adjustments were made to HCN analysis protocol, based on recent literature [45].

For products of unknown composition, the equivalence ratio can be calculated from the oxygen requirement of a preliminary well-ventilated test. This test is carried out at 900 °C, in excess air. From the total O_2 requirement, the stoichiometric O_2 to fuel ratio (Ψ_0) can be determined. This allows tests at other primary air flows to have known equivalence ratio.

The samples tested are complex commercial products, so some parameters had to be adjusted to fit their dimensions and properties. ISO/TS 19700 recommends a sample boat speed of 40 mm min^{-1} (section 5.6.1) and a combustible loading of approximately 25 mg min^{-1} (section 8.2) giving a feed rate of combustible material of 1 g min^{-1}. The primary air flow is then adjusted to give the specified equivalence ratio. Table 4 specifies the sample feed rate of the whole product, primary air used, and the resulting equivalence ratio that resulted in steady burning of each product. The whole product includes the gypsum in the plaster board, and the copper conductors in the cable.

3.1. *Verification test, and example of a non-compliant test*

Fig. 4 shows an example of an SSTF verification run, using a uniform strip of polymethylmethacrylate (PMMA) in well-ventilated conditions, showing the variation in concentrations of O_2, CO_2 and CO over time. The graph shows ignition and flame stabilisation (300-400s), after which CO and CO_2 concentrations stabilise (450-1100s) with very little fluctuation. For the test to be valid, this phase needs to meet the trend and fluctuation criteria for at least 5 min.

PMMA is used as a verification test to confirm the SSTF instrument is correctly set-up and calibrated. It also serves as an example of what the test results are meant to look like. Achieving the same results with non-homogeneous complex products can be less straightforward. Fig. 5 is an example of an unsuccessful PUF run, which despite following the same procedure as the PMMA test has failed to meet the steady state: the short-term fluctuations between 450 and 900 s of the O_2 and CO_2 are bigger than the standard allows. This was due to the sample flaming inconsistently, with flickering and reignition, as the flame moved out of the furnace against the direction of sample and primary air flow. In subsequent tests, the conditions were adjusted to prevent this.

The following sections describe the adjustments necessary to successfully test each product, showing both well-ventilated and under-ventilated conditions.

Table 4
Conditions of SSTF tests.

Material	Fire stage	Sample feed rate /g min^{-1}	Primary air/L min^{-1}	Equivalence ratio
Plaster board	Smouldering	4.2	2	-
	W-V^{NF}	4.2	10	-
Cable	Smouldering	4.5	2	-
	W-V	4.5	16.8	0.76
	Small U-V	4.5	4.2	2.02
	Large U-V	4.5	4.2	2.09
OSB	Smouldering	0.75	2	-
	W-V	0.75	6	0.50
	Small U-V	0.75	1.25	1.97
	Large U-V	0.75	1.25	2.10
PUF	Smouldering	1	2	-
	W-V	2.5	20	0.50
	Small U-V	2.5	5	1.94
	Large U-V	2.5	5	1.99

^{NF} Plasterboard did not flame during the well-ventilated testing, therefore it does not have an equivalence ratio.

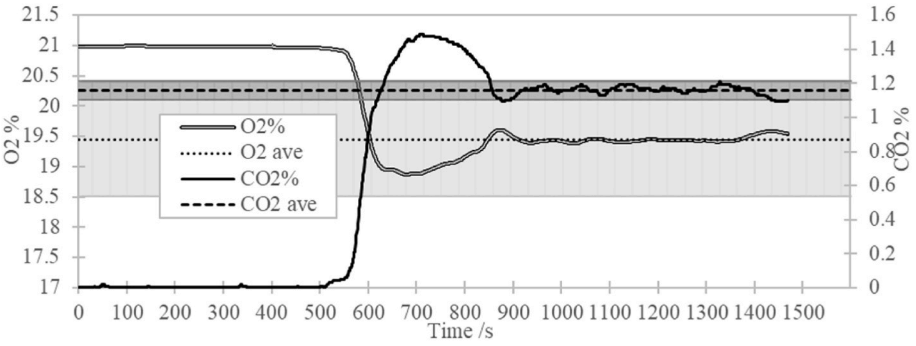

Fig. 3. CO_2 and O_2 concentrations from a SSTF test showing short-term fluctuation requirement for steady state (shaded).

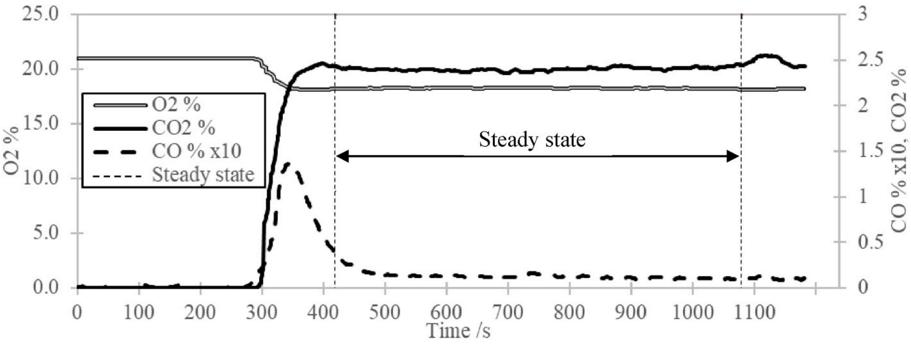

Fig. 4. PMMA Variation of O_2, CO and CO_2 concentrations during burn in well-ventilated conditions.

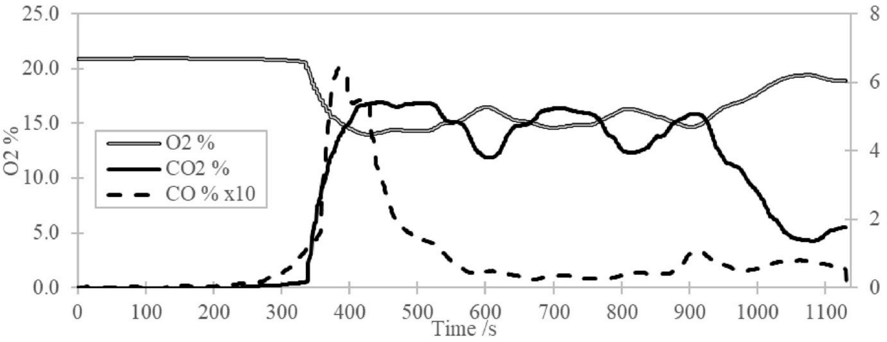

Fig. 5. PUF - Variation of O_2, CO and CO_2 concentrations during an unsuccessful burn.

3.2. Plasterboard

The first sample is the plasterboard, non-combustible gypsum coated with a thick layer of paper. Flaming did not occur, even in the furnace at 900 °C in well-ventilated conditions. Therefore, data is only reported for the smouldering stage and the non-flaming test at 900 °C. The smouldering test is shown below, in Fig. 6. This shows CO_2 and CO concentrations which are so low as to be indistinguishable from background concentrations (seen in the first 200 s of the test). The data cannot be considered to represent a steady state.

The 900 °C well-ventilated (non-flaming) test is shown in Fig. 7. This shows measurable concentrations of CO_2 and CO, which meet the steady state requirements, despite the long-term upward trend in CO_2 concentration.

The current version of ISO/TS 19700:2016 has no specific guidance for assessing the smoke toxicity of non-combustible materials under conditions corresponding to each fire stage. However, there are many applications of construction products where it would be useful to have comparative data for combustible and non-combustible products.

3.3. Cable

The preliminary cable tests failed to meet the steady state requirements due to unstable flaming, where the sample would repeatedly stop flaming then reignite. This was believed to result from insufficient fuel for sustained ignition. The high non-combustible content of the cable (the copper wire) and the high mineral filler content of the fire retarded sheath led to an actual combustible fraction (e.g. organic polymer) being close to 8.8% of the total mass (including copper) or 22% of the combustible mass (of polymeric compounds). To overcome this the feed rate was increased to raise the combustible mass to 4.5 g min^{-1}, by increasing the drive speed of the sample boat, resulting in the steady state requirements being met.

Fig. 8 shows the SSTF test of a length of cable in the well-ventilated scenario, reaching steady state between 900 and 1400 s. Fig. 9 shows the SSTF test of cable in the small under-ventilated scenario. In this case the test meets the steady state criteria, although there is greater fluctuation in the CO concentration.

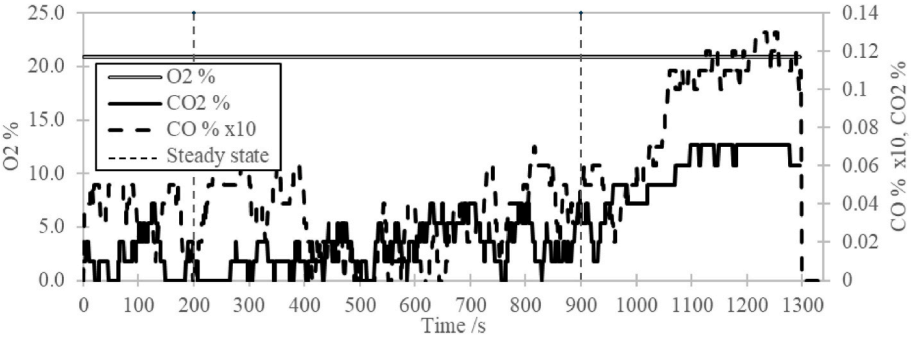

Fig. 6. Plasterboard - Variation of O_2, CO and CO_2 concentrations during burn in smouldering conditions (the digital noise reflects the very low CO and CO_2 concentrations, close to the sensor detection limits).

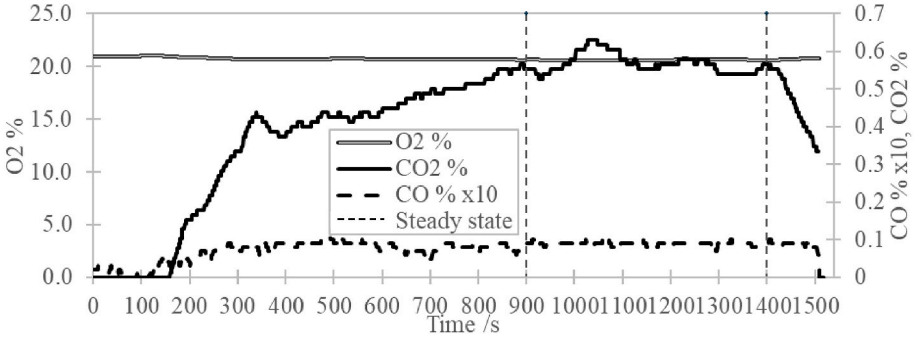

Fig. 7. Plasterboard - Variation of O_2, CO and CO_2 concentrations during burn at 900 °C in well-ventilated conditions, non-flaming.

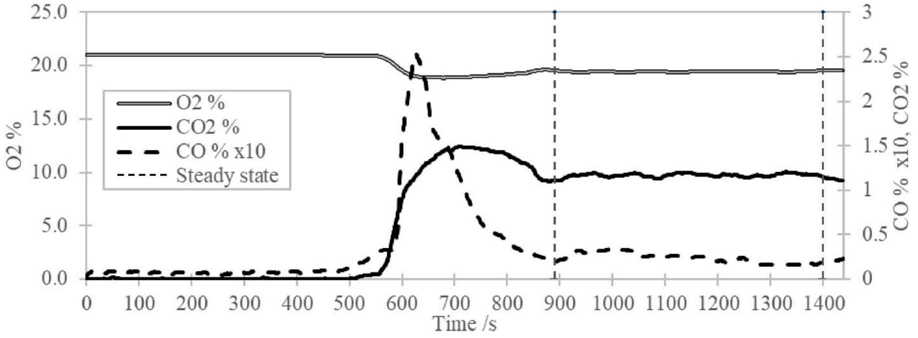

Fig. 8. Cable - Variation of O_2, CO and CO_2 concentrations during burn in well-ventilated conditions.

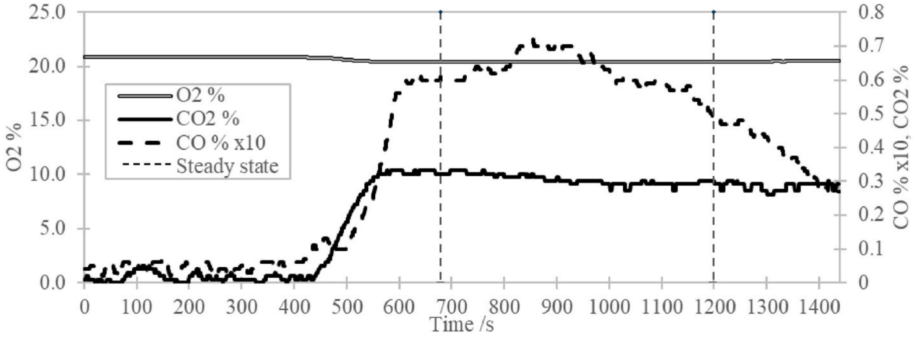

Fig. 9. Cable - Variation of O_2, CO and CO_2 concentrations during burn in small under-ventilated conditions.

3.4. Oriented strand board OSB

In the preliminary tests, the OSB samples did not reach the steady state requirements as the large mass of OSB char continued to smoulder slowly, increasing the CO and CO_2 concentrations as the test went on. To overcome this problem, the feed rate of the sample was reduced to allow fuller combustion during the flaming stage. This modification is compliant with the standard, but not specifically described. By using a thinner sample (4 x 10 × 650 mm) and lowering the feed rate to 0.75 g min^{-1}, the CO and CO_2 concentrations stabilise, giving steady state

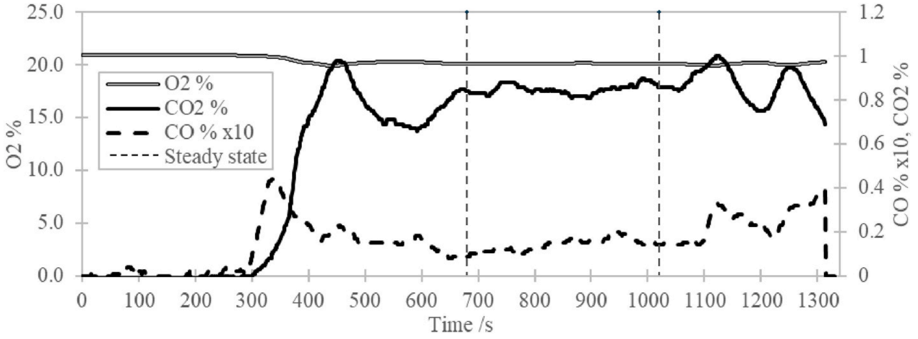

Fig. 10. OSB - Variation of O_2, CO and CO_2 concentrations during burn in well-ventilated conditions.

burning. To ensure that the feed rate adjustment did not affect the stoichiometric O_2 requirements, a preliminary stoichiometric test at 900 °C was run at the new feed rate, to ensure consistency of the results.

Fig. 10 shows a well-ventilated SSTF run of OSB. The CO and CO_2 concentrations are reasonably stable from 500 to 1200 s, but even more so from 650 to 1050 s. In this instance the steady state yields were taken from 670 to 1020 s.

Fig. 11 shows the small under-ventilated stage of OSB test, which again meets the steady state requirements, although there is more fluctuation in the CO concentration, particularly after 900 s.

3.5. Polyurethane foam

For a flexible foam sample to provide adequate mass load (according to the standard 20 g over a 20 min run), the volume of the sample would be too big to allow adequate air flow through the furnace tube, which is 44 mm internal diameter. This problem was solved by binding the foam with a fine steel wire, to create a sample with higher density. This method does not affect the burning behaviour of the sample because flexible polyurethane foam (PUF) decomposes upon heating, mostly into its component monomers, isocyanate and polyol, converting the fuel to liquid which burns as a pool fire.

For easily ignitable samples such as PUF, some adjustments may be necessary to prevent the flame moving against the direction of sample movement, and sometimes even outside the furnace towards the air inlet. In this case, the boat speed was increased to give a sample feed rate of 2.5 g min^{-1}. While this shortens the duration of the test, it prevent the flame from spreading away from the heated zone and was sufficient to meet the O_2 and CO_2 criteria for a 5 min period of steady state burning.

Fig. 12 shows the SSTF test of PUF in the well-ventilated stage. While the test was shorter than with other products, a steady state of 5 min was reached. However, probably due to the short duration of the test, the CO concentration shows significant fluctuation. Fig. 13 shows the small under-ventilated test of PUF. This shows a much more consistent CO concentration during the steady state.

The well-ventilated test shows oscillating flaming, where the flaming periodically increases and decreases, usually more typical of under-ventilated flaming. After ignition, in the presence of sufficient fuel, the flame spreads rapidly, until the oxygen is so depleted it recedes, which may be repeated, giving rise to a puffing phenomena. Fig. 14 shows another well-ventilated test for PUF, using the same settings as for Fig. 12, also demonstrating the same effect and reaching similar volume fractions and yields as the previous well-ventilated PUF test.

3.6. Steady state calculations

As mentioned before, reaching the steady state during the SSTF testing is essential to ensure the data is representative of the fire stage in question. To confirm the steady state has been reached, the standard defines limits to short-term fluctuations and long-term trends that the test must meet to be compliant. In Table 5, the short-term fluctuations and long-term trends are calculated for each SSTF test and compared against their requirements. Each of the reported tests have met both of the steady state trend requirements.

4. Results

Each material was tested at all four fire stages (1b, 2, 3a, 3b), except for plasterboard which did not ignite. The concentrations of O_2, CO_2 and CO were recorded, and the yields are reported in Table 6. The change in O_2 is expressed as oxygen depletion, showing the oxygen consumed by the combustion processes, per g of combustible sample.

Additionally, the concentration of HCN was determined from the bubbler solution after the test run. The limit of detection for HCN is 0.3 ppm in the bubbler solution, which corresponds to 4 ppm in smoke under the sampling conditions used here. All samples, except for PUF, were below the detection limit, as indicated in Table 6.

Initially, each sample was tested following the method in the ISO/TS 19700 without any adjustments. In many cases steady state burning did not occur: because the flame spread along the sample away from the flaming zone; insufficient fuel made the flame unstable; or the sample did not flame at all. With each product, the method was adjusted to obtain steady flaming and assess the smoke toxicity.

Additional comparisons with the theoretical maximum yields of CO_2 can be made from the tests used to determine the stoichiometric oxygen requirement. As they were well-ventilated and at the maximum temperature, they maximised the yields of CO_2, by providing the most complete combustion. These yields are shown at the bottom of Table 6.

5. Discussion

The steady state tube furnace is designed to force burning under specific ventilation conditions and maintain them during the steady state period, the time during the test where the concentrations of O_2 and CO_2 stabilise. To assess the appropriateness of the ventilation conditions, the CO_2 yields in the well-ventilated scenario are compared to the theoretical yields, obtained from the stoichiometric tests at 900 °C.

Additionally, the yields from each fire stage (smouldering, well-ventilated, small under-ventilated and large under-ventilated), correspond to those reported elsewhere.

To determine whether the test results correspond to the fire stages, the CO/CO_2 ratio was compared to the generic values presented in ISO 19706:2011, shown in Table 7. With each stage, except for the cable in small under-ventilated flaming conditions, the CO/CO_2 ratio corresponds to standard's generic values.

Each material showed an increase in CO/CO_2 ratio with the increase in equivalence ratio. The increase in CO/CO_2 ratio is described in the standard and is demonstrated by previously published work on this apparatus [5,6,9]. The ability to quantify the smoke toxicity under different ventilation conditions is a capability specific to the SSTF apparatus. This work has demonstrated that this capability can be extended to complex products with more problematic burning

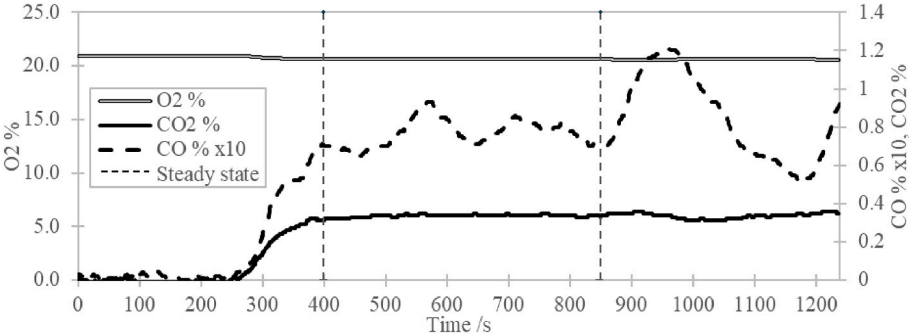

Fig. 11. OSB - Variation of O_2, CO and CO_2 concentrations during burn in small under-ventilated conditions.

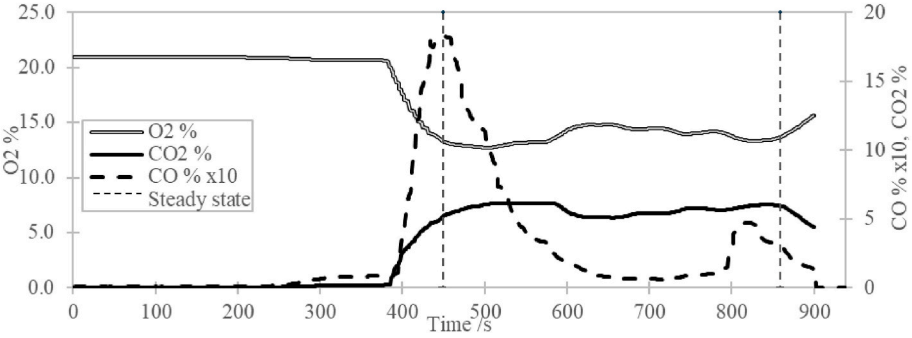

Fig. 12. PUF - Variation of O_2, CO and CO_2 concentrations during burn in well-ventilated conditions.

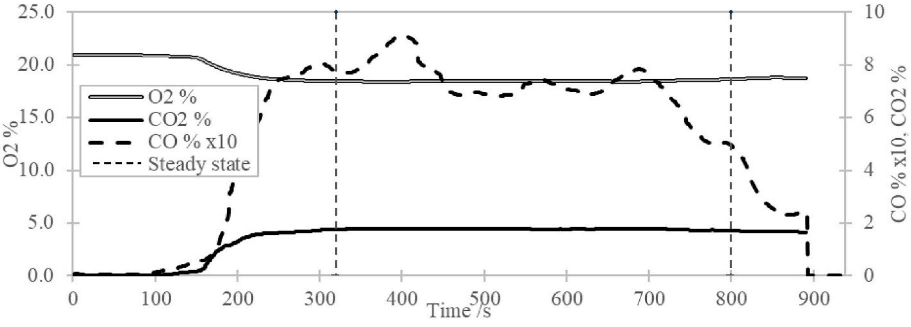

Fig. 13. PUF - O_2, CO and CO_2 concentrations in small under-ventilated conditions.

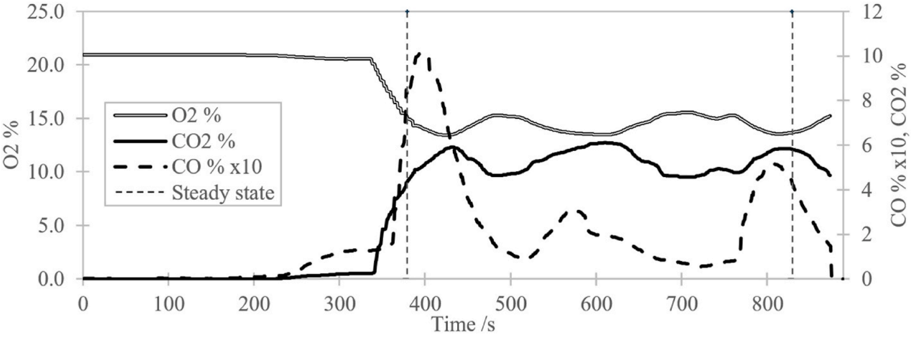

Fig. 14. PUF - Variation of O_2, CO and CO_2 concentrations during burn in well-ventilated conditions.

Table 5
Assessment of compliance with ISO/TS 19700 steady state criteria.

Fire stage	Requirements	Long-term trends		Short-term fluctuations	
		<0.02 min^{-1}		<0.20	
	Sample	$\Delta[O_2]$/min^{-1}	$\Delta[CO_2]$/min^{-1}	$\Delta[O_2]$	$\Delta[CO_2]$
Well-ventilated	Plasterboard	0.00000005	0.000588	0.0007	0.070182
	Cable	0.00000006	0.000003	0.0008	0.014279
	OSB	0.00000010	0.000057	0.0015	0.024956
	PUF	0.00000165	0.000003	0.0372	0.061567
Small under-ventilated	Cable	0.00000007	0.000100	0.0009	0.039828
	OSB	0.00000003	0.000046	0.0004	0.019729
	PUF	0.00000007	0.000115	0.0081	0.062293
Large under-ventilated	Cable	0.00000012	0.000211	0.0020	0.046191
	OSB	0.00000002	0.000077	0.0003	0.018977
	PUF	0.00000054	0.000015	0.0075	0.021274

behaviour.

The sharp increase in smoke toxicity, as exemplified by the CO yield seen in thermoplastics [11], is less evident in these complex products. Typically, the CO yield increases by a factor of between 10 and 50 in the transition from well-ventilated to under-ventilated. In these cases (shown in Table 6), the CO yield increases by a factor between 2 and 6 for the transition to small under-ventilated and between 6 and 8 to large under-ventilated. There are several reasons for this. Simple

Table 6
Yields of the SSTF tests, in g g^{-1} (uncertainty is expressed as ± 1 standard deviation over the steady state period).

Fire stage	Sample	Plasterboard	Cable[a]	OSB	PUF
1b Oxidative pyrolysis/ Smouldering	O_2 depleted	0.004 ± 0.0001	0.005 ± 0.0005	0.344 ± 0.038	0.046 ± 0.005
	CO	0.001 ± 0.0002	0.001 ± 0.0001	0.127 ± 0.010	0.005 ± 0.0006
	CO_2	0.005 ± 0.003	0.012 ± 0.001	0.380 ± 0.027	0.047 ± 0.014
	HCN	N.D.	N.D.	N.D.	N.Q.
2 Well-ventilated flaming	O_2 depleted	0.054 ± 0.008^{NF}	0.561 ± 0.001	0.731 ± 0.026	1.873 ± 0.166
	CO	0.001 ± 0.0002^{NF}	0.007 ± 0.0002	0.011 ± 0.002	0.061 ± 0.055
	CO_2	0.126 ± 0.016^{NF}	0.594 ± 0.002	1.034 ± 0.029	2.028 ± 0.122
	HCN	N.D.	N.D.	N.D.	0.006 ± 0.001
3a Small under-ventilated flaming	O_2 depleted	-	0.205 ± 0.001	0.306 ± 0.004	0.662 ± 0.040
	CO	-	0.012 ± 0.0002	0.065 ± 0.006	0.173 ± 0.020
	CO_2	-	0.166 ± 0.001	0.418 ± 0.010	0.642 ± 0.044
	HCN	-	N.D.	N.D.	0.005 ± 0.0004
3b post flashover flaming	O_2 depleted	-	0.319 ± 0.002	0.290 ± 0.005	0.693 ± 0.037
	CO	-	0.043 ± 0.001	0.082 ± 0.006	0.243 ± 0.050
	CO_2	-	0.265 ± 0.002	0.405 ± 0.008	0.649 ± 0.015
	HCN	-	N.D.	N.D.	0.039 ± 0.004
CO_2 yields – (well-ventilated 900 °C)	MAX CO_2	0.148 ± 0.007^{NF}	1.442 ± 0.038	1.659 ± 0.142	2.026 ± 0.220

^{NF} Plasterboard did not flame during the well-ventilated testing.
N.D. – None detected.
N.Q. – Detected, but below limits of quantification.
[a] For cable, the weight of the wire was removed from the yield calculations.

Table 7
Comparison of CO/CO_2 ratios for each fire stage.

$[CO]/[CO_2]$	Typical values (ISO 19706)	Plasterboard	Cable	OSB	PUF
1b Smouldering	0.1 – 1.0	0.55	0.14	0.53	0.19
2a Well-ventilated	<0.05	0.02	0.02	0.02	0.05
3a Small under-ventilated	0.2 – 0.4	-	0.11	0.25	0.42
3b Large under-ventilated	0.1 – 0.4	-	0.25	0.31	0.60

thermoplastics burn very cleanly in well-ventilated conditions, giving very low CO yields. The highly fire retarded cable failed to burn completely in the small under-ventilated conditions (the oxygen consumption fell from 0.56 to 0.21 g g^{-1} from well-ventilated to small under-ventilated, but increased to 0.32 for the post-flashover condition) preventing fully under-ventilated flaming; the PUF also showed a high CO yield for well-ventilated flaming.

The standard also defines the long-term trend and short-term fluctuations required for a test result to be valid. Each test has met the requirements.

As it has been shown that complex, non-homogeneous products can be satisfactorily tested using the SSTF, with minor modifications to the protocol, the scope of the standard and details could be modified accordingly. For example, the feed rate may need adjusting to match the sample's dimensions or combustible content, while testing products which do not ignite could follow a more specific protocol.

6. Conclusion

The tests have been shown to meet the standard criteria for valid test runs, after adjustments which remain compliant with the existing standard. These adjustments could be incorporated into a revised standard. For example, using the preliminary test for stoichiometric oxygen consumption at 900 °C shows whether the product undergoes flaming combustion. This eliminates the costly and time-consuming process of increasing the furnace temperature in steps of 25 °C for successive runs to establish whether an essentially non-combustible sample will ever undergo flaming combustion. Once this has been established, an additional high temperature, non-flaming fire stage could be added to the testing requirements for such products.

The adjustments to the testing procedure that were used on the samples and shown to facilitate viable steady state burning are as follows: adjusting feed rate to match better the combustible content of the sample (used on cables); increase mass load for samples of low density and/or high flammability (used on polyurethane foam); or lower feed rate and lower mass load to reach steady state on samples which burn at different rates.

The results demonstrate that the steady state tube furnace can be applied to wider range of samples than specified in the scope of the current standard.

However, in order to simplify the smoke toxicity assessment of other materials and products using the state tube furnace, the protocol would benefit from some adjustments. These include: products without enough of combustible load for flaming (such as plasterboard, or any non-combustible board with a combustible paint or other covering); products with significant non-combustible components (such as electrical energy cable, fibre-optic cable, metal-elastomer door seals etc.); products where the fuel changes on burning, such as timber forming a combustible char, supporting rapid initial flaming combustion followed by slower char oxidation (oriented strand board (OSB) and many other timber-based products); products which are difficult to test because of their low density (flexible polyurethane foam (PUF) has been tested but other furniture fillings and foams could also be assessed using the same methodology).

Based on the work reported here, the following modifications could be made to ISO/TS 19700.

1. Change the protocol for fuels of unknown composition or low flammability by starting with the well-ventilated 900 °C test. This will avoid the painstaking process of repeating the test at 25 °C intervals for non-flaming samples, and ensure that appropriate ventilation conditions are used.
2. Explicitly state the need to ensure that the preliminary run at 900 °C is well-ventilated. The current guidance of 1 g min^{-1} combustible fuel feed rate and 10 L min^{-1} primary air provides too little oxygen for polyolefins ($(CH_2)_n$) but too much air flow for heavily-filled products.
3. Explain the need to vary sample dimensions, either by careful cutting (e.g. for wood) or by compression (e.g. for foam).
4. Amplify the need to vary the rates of fuel and air flow into the furnace in order to obtain steady state burning.

5. Modify the scope to widen the use of the steady state tube furnace to materials and products which can be converted to linearly uniform samples for testing.

CRediT authorship contribution statement

Katarina Handlovicova: Writing – review & editing, Writing – original draft, Validation, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Anna A. Stec:** Writing – review & editing, Supervision, Resources, Project administration. **T Richard Hull:** Writing – review & editing, Supervision, Resources, Funding acquisition, Formal analysis, Conceptualization.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Katarina Handlovicova is in receipt of a PhD studentship from the Mineral Wool Manufacturer's Association (MIMA), UK. Richard Hull is the PI for the MIMA sponsored project. The other authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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