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Review Paper

OVERVIEW OF COMMON METHODS FOR FIRE TESTING

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Abstract. *Fire testing is critical for assessing the behavior of building materials under fire conditions, providing indispensable insights into important variables such as flammability, heat release rate, and smoke production. This paper provides an overview of common fire testing methods, including the procedures for test selection, sample preparation, data collection, and analysis. These fire testing methods are outlined in standards issued by world-respected standardization bodies such as ISO, EN, ASTM, and UL. These standards guarantee and ensure the consistency and reliability of prescribed methods. Moreover, this paper presents a detailed description of a custom-made installation hosted at the Fire Protection Laboratory at the Faculty of Occupational Safety, University of Niš, used for evaluating the flammability parameters of a broad spectrum of materials. This hyphenated installation, comprising a mass loss calorimeter and an FTIR gas analyzer, can be used for comprehensive and simultaneous real-time measurement of flammability parameters and fire effluent analysis.*

Key words: *fire testing, ignitability, flame spread, fire effluents, heat release rate, mass loss calorimeter, gas analyzer.*

1. INTRODUCTION

Fire testing is crucial for ensuring building and occupant safety by assessing how building materials respond to fire, considering factors like flammability, smoke production, and structural integrity. Understanding common fire testing methods helps in selecting materials that mitigate fire hazards and enhance life safety. Typically, fire testing procedures

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involve the following steps: Test selection, Sample preparation, Testing procedure, Data collection and Analysis and classification and reporting.

Selecting the correct fire test is essential to guarantee the safety and effectiveness of building materials. Before performing any specific tests, it's critical to understand the fire properties being evaluated. The most important properties are: flammability, fire resistance, smoke production, and heat release. Several factors influence which fire test is most appropriate for a particular need. However, two are the most important: prospective material use and building code requirements. The intended use of the material plays a significant role. Fire resistance testing is crucial for structural elements, while flammability is the primary concern for building products. Local building codes typically include specific fire testing requirements suited to various building materials and their intended uses.

ISO, ASTM, and EN standards provide guidelines for gathering material samples that accurately represent the bulk material. This process may include sampling from multiple locations within a batch or using specific extraction tools. Certain materials may require conditioning before testing to achieve the desired moisture content, which ensures consistent results since moisture has a significant influence on fire behavior. Conditioning is frequently performed using ovens or desiccators. Tests frequently require that samples are cut or shaped to precise dimensions based on the test method. This ensures proper placement within the testing apparatus and uniform heat exposure throughout the test. Sample preparation for composite materials with multiple layers may include separating the layers or testing them individually, depending on the test requirements. For materials with coatings or finishes, tests may require preparing samples with and without the treatment to assess its effect on fire performance. Depending on the material, specific safety precautions such as wearing appropriate personal protective equipment (PPE) or providing adequate ventilation for dust control may be required during sample preparation. A thorough sample preparation is vital to achieve reliable fire test results. Poorly prepared samples can produce inaccurate data, jeopardizing the overall validity of the test. Standardized sample preparation procedures ensure consistency and repeatability, allowing for meaningful comparisons of different materials.

To obtain consistent and repeatable results, specialized equipment and controlled laboratory environments are used. Usually, in order to guarantee adherence to relevant testing requirements, these facilities follow particular accreditations. The tested sample is inserted into the testing apparatus in line with the chosen methodology, which frequently requires the use of special fixtures or configurations to replicate real-world situations. The sample may need to be positioned horizontally for a floor application or vertically to replicate a wall in some tests. A standardized ignition source, such as an electrical radiant panel or pilot flame, is introduced to initiate combustion, depending on the type of test. The requirements specified in the test standard regulate the choice and positioning of the ignition source.

The data acquisition system is used to systematically document key data throughout the test procedure. Usually, the following parameters are documented: Temperature (in almost all cases K- type thermocouples are used), Time to ignition (the time it takes for the sample to achieve a specific ignition criterion, such as flaming combustion), Heat flux (the rate of heat transfer through a surface per unit area i.e. the amount of heat energy that flows through a specific area over a given period of time), Heat release rate (defines the rate of heat transfer through a surface per unit area), Smoke density (the concentration of smoke particles generated during combustion), Gas/Vapor emission (structure of fire effluents - gases released during combustion, which can be important for assessing toxicity hazards) and Fire spread (rate of flames spread across the sample's surface).

The material is classified or given a fire rating in accordance with applicable standards based on the test results. These classifications or ratings show how well a material performs in terms of smoke production, flame spread, fire effluent toxicity, and fire resistance.

Because fire testing involves inherent risks, strict adherence to safety precautions is required. Thorough safety protocols are essential during the testing process to protect people and the surrounding environment. This includes adhering to hazard management procedures, using efficient ventilation systems, fire suppression equipment, and flame-resistant clothing. Additionally, testing facilities need to maintain strict quality control procedures to ensure the accuracy and consistency of their test results. This entails following established testing procedures, regularly calibrating equipment, and participating in proficiency testing programs.

2. Classes of Fire Tests

Fire tests have been developed to address nearly every aspect of fire behavior. Leading organizations such as the Society of Fire Protection Engineers (SFPE) [1], the National Fire Protection Association (NFPA) [2], ASTM International (formerly the American Society for Testing and Materials) [3], the International Organization for Standardization (ISO) [4], European Norms (EN) [5], and Underwriters Laboratories (UL) [6] are responsible for developing fire-related tests and standards in the USA, EU, and internationally. Their mandate covers two key categories for fire testing: fire resistance and reaction to fire.

Fire resistance tests are designed to check whether building construction assemblies prevent fires from spreading from the point of origin through the assembly and into adjacent compartments. These kinds of tests are usually performed on building structural elements and fire-resistant barriers (walls, floors, columns, doors, and windows). Also, these tests assess the duration for which a structural element of a building can endure exposure to a standardized fire without collapsing or impairing its intended functionality.

Reaction to fire tests evaluates how materials behave when exposed to a fire source, focusing on their immediate response. Unlike fire resistance tests, they do not measure the duration a material can endure fire exposure. Understanding how materials react to fire allows building designers, architects, and fire safety professionals to make informed material selection decisions and implement fire risk reduction strategies within buildings.

3. Fire Resistance Test Standards

Fire resistance test standards ensure that structures meet the fire protection and separation standards outlined in building codes. These kinds of standards are crucial for ensuring safe construction by evaluating the performance of building elements under fire conditions. In these tests, the "fire resistance rating" indicates the duration the assembly can withstand standard exposure before reaching a "critical endpoint". For example, in the case of walls and partitions, the "critical endpoint" is reached when a specific temperature rise (the value of which depends on the standard) above the initial temperature of the test specimen on the unexposed side is attained. The construction element undergoes exposure to a progressively intense fire, following a standard time-temperature curve. The standard time-temperature curve is a key component of fire resistance testing. It essentially depicts a controlled temperature increase over time, simulating a specific fire scenario within a testing furnace. It's noteworthy that the fire resistance rating pertains to the entire tested assembly rather than its individual components. Tests are performed in Fire Resistance Test Furnaces.

Internationally, ISO 834 Fire-resistance tests — Elements of building construction comprise a series of standards covering various aspects of fire resistance testing. This battery of standards, consisting of 14 individual standards, focuses on both loadbearing and non-loadbearing vertical and horizontal separating elements, as well as beams, columns, ceiling elements, structural steel elements, beams, and bars.

Fire resistance tests for load-bearing structural elements also assess the specimen's ability to carry loads under test conditions. In the USA for fire resistance testing following standards apply: ASTM E119 Standard Test Methods for Fire Tests of Building Construction and Materials [7]. Requirements for ISO, ASTM, and NFPA standards are very similar, with minor differences arising primarily in the standard time-temperature curves.

Few studies have explored the precision of fire resistance testing due to its complex and costly nature [8]. This creates a discrepancy between code requirements and actual test results, where even slight differences are expected. Variability in test results can stem from the precision of the fire test, execution variability in the laboratory, and differences in the test specimen. This also implies that manufacturers may use "selective testing" by running multiple tests, possibly in different laboratories, until they achieve the desired result, thereby avoiding the need for additional testing. The reproducibility and consistency of test results is an ongoing topic of discussion and debate.

The standard time-temperature curves often don't capture the real-world complexities of fires with varying fuels, ventilation, and growth patterns. Their simplified assumptions and one-size-fits-all approach might miss crucial details in specific building configurations or fire safety concerns. Additionally, the lack of customization hinders them from reflecting unique situations or fire protection objectives. Ariyanayagam [9] highlights the limitations of standard time-temperature curves in fire resistance testing. He pointed out that the ISO 834 curve was developed in the early 1900s when wood was the primary fuel source, and emphasized that current standard time-temperature curves do not accurately represent modern building materials, which include thermoplastic materials, synthetic foams, and fabrics, among others. Therefore, standard curves should be used cautiously and complemented by further analysis for a more complete understanding of fire safety performance.

4. Reaction to Fire Test Standards

Most of the ongoing fire testing primarily focuses on examining reaction-to-fire characteristics. These tests typically evaluate properties such as ignition capacity, ease of extinction, flame propagation, smoke and release of toxic gases and heat release rate [10].

4.1. Ignitibility

Ignitability refers to a material's tendency to ignite and sustain combustion [10]. Key properties that characterize the ignition are minimum ignition temperature, critical/minimal heat flux required to initiate the ignition and time to ignition under the specified incident heat flux. The ignitibility of materials has been extensively studied by Babrauskas, who condensed a significant body of knowledge on the subject in the seminal Ignition Handbook [11].

While ignition temperature is crucial, only a few standards specifically address the procedures for determining it. The standards mainly refer to plastic materials. These are the following standards ASTM D1929-23 Standard Test Method for Determining Ignition Temperature of Plastics [12] and ISO 871:2022 Plastics - Determination of ignition temperature using a hot-air furnace [13]. Although these standards provide valuable information regarding the ignition temperatures of plastics, their applicability to other materials may be limited [14]. They are primarily designed for plastics and might not work well for other materials. The controlled lab environment may not perfectly reflect real fires, and the single ignition source used in the test might not represent all fire scenarios. Additionally, the test only provides one data point (ignition temperature) and doesn't consider factors like airflow, which can significantly impact ignition behavior in real-world fires. Therefore, while these standards are very useful, they should be used alongside other tests and considerations to provide a more complete overview of a material's fire safety characteristics.

It is important to note that the minimum ignition temperature is measured as an additional parameter during heat and smoke release tests. Some of the most commonly used standards for determining heat and smoke release are: ISO 5660 - Reaction-to-fire tests — Heat release, smoke production and mass loss rate — Part 1: Heat release rate (cone calorimeter method) and smoke production rate (dynamic measurement) [15]; ASTM E2058 - Standard Test Methods for Measurement of Material Flammability Using a Fire Propagation Apparatus (FPA) [16]; ASTM E1354 - Standard Test Method for Heat and Visible Smoke Release Rates for Materials and Products Using an Oxygen Consumption Calorimeter [17]; ASTM E1321 - Standard Test Method for Determining Material Ignition and Flame Spread Properties [18] and ISO 5657 - Fire tests — Reaction to fire — Ignitability of building products subjected to direct impingement of flame [19]. These standards are reviewed in the heat release rate section.

4.2. Ease of extinction

ASTM D2863 Standard Test Method for Measuring the Minimum Oxygen Concentration to Support Candle-Like Combustion of Plastics (Oxygen Index) [20] and ISO 4589-2 Plastics Determination of burning behaviour by oxygen index Part 2: Ambient-temperature test are two similar standards used for measuring materials ease of extinction [21]. They describe a method for determining the combustion behavior of plastics by measuring the oxygen index at ambient temperature. The oxygen index indicates the lowest concentration of oxygen in an oxygen-nitrogen mixture that supports material combustion. This test provides critical information about the flammability of plastics in normal environmental conditions. The method requires just a small sample of material to be tested and has excellent reproducibility and repeatability. Because of this, these standards stand out as cost-effective and highly accurate quality control tests for combustible materials.

The limitations of the quoted testing methods include their focus on ambient temperature testing, which may not fully represent real-world fire conditions with elevated temperatures. They primarily assess burning behavior using the oxygen index method, limiting their scope to plastics and potentially excluding other materials. Environmental factors such as humidity and airflow are not always considered, and test result interpretation may vary due to factors such as sample preparation and testing conditions. Despite providing valuable insights, these standards/methods of testing have limitations that should be acknowledged when assessing fire safety.

4.3. Flame spread

Understanding flame spread is crucial for predicting fire growth and flashover. Flame spread tests are widely used to gauge material performance in fire situations. The angle formed between the exposed side and the horizontal reference plane can be used to categorize flame spread tests. [22]. This angle largely influences the rate at which gaseous combustion products heat the surface of the tested material [23].

The ASTM E84 Standard Test Method for Surface Burning Characteristics of Building Materials [24], commonly known as the Steiner Tunnel Test, is widely used in the USA for assessing the surface flammability of building materials. The Steiner test is widely used in North America to determine if building materials meet the requirements set forth by building codes. The method of preparing samples for this test is defined by the accompanying ASTM standards.

As a result of testing according to ASTM E84, the dimensionless flame spread index (FSI) is obtained. The FSI is calculated based on the distance and time it takes for flames to spread along the surface of a specimen compared to a reference material under controlled conditions. A lower FSI indicates slower flame spread and better fire resistance of the material. The problem with this test is that FSI is a relative quantity and as such cannot be used in any way in fire modeling.

In North America, a number of standards and tests have been developed for testing the flame spread of pipes and cables based on ASTM E84 such as NFPA 262 Standard Method of Test for Flame Travel and Smoke of Wires and Cables for Use in Air-Handling Spaces [24]. There is no ISO standard corresponding to the Steiner Tunnel Surface Flame spread test.

In addition to the Steiner test, the Lateral Ignition and Flame Spread Test (LIFT) ASTM E1321 Standard Test Method for Determining Material Ignition and Flame Spread Properties [18] and ISO 5658 Reaction to fire tests Spread of flame Part 2: Lateral spread on building and transport products in vertical configuration [26], along with ASTM E162 Standard Test Method for Surface Flammability of Materials Using a Radiant Heat Energy Source [27], are utilized to assess flame spread. The LIFT test was developed to improve the ASTM E162 standard by providing engineering variables derived from the test, which can be utilized in fire safety calculations and predicting full-scale flame spread performance [28]. LIFT is used to determine the value of critical heat flux, surface temperature required for flame spread and thermal inertia of tested material. Additionally, a flame spread parameter is obtained as an output, which can be used to compare fire responses of different materials.

While ISO 5658 provides a number of useful variables for fire safety calculations, according to ISO, it would be insufficient to rely solely on this standard for describing or evaluating the fire hazard of materials, products, or assemblies in actual fire situations.

4.4. Smoke and toxic gas release

Smoke from fires poses several significant problems. Two are the most important: reduced visibility and toxicity.

Smoke clouds can severely limit visibility, making it difficult to escape a burning building. ASTM E662 Standard Test Method for Specific Optical Density of Smoke Generated by Solid Materials [29] and ISO 5659 Plastics - Smoke generation Part 2: Determination of optical density by a single-chamber test [30] standards are used for testing the potency of certain materials to emit smoke. In ASTM E662 a material sample measuring 7.5 x 7.5 mm is placed vertically and exposed to a heat flux of 25 kW/m^2 inside the specially designed chamber. The amount of smoke emitted by materials during combustion is measured indirectly by measuring the light transmitted.

The test has faced significant criticism due to its static nature, as it does not yield reliable values that accurately reflect real fire conditions. Despite these limitations, the test continues to be widely utilized for evaluating both existing materials and the development of new ones. ISO 5659 provides significant improvements over ASTM E662 by allowing testing up to a heat flux of 50 kW/m^2, horizontal sample orientation and real-time mass loss measurement. However, the most reliable data is obtained in full-scale tests, which involve testing the material on a full scale.

Although surprising at first glance, statistical data show that smoke toxicity is not the most significant fire hazard. It has been found that there is a strong correlation between fire fatalities and the amount of CO released during a fire [31,31,32]. Since CO is always produced during a fire due to incomplete combustion, the amount of CO generated will depend on the size of the fire. Therefore, objects and materials that release a smaller amount of heat per unit of time by burning carry a smaller fire hazard, regardless of the toxicity of the compounds released by burning them.

Through the working group ISO/TC92/SC3 - "Fire threat to people and environment", ISO has defined a number of standards related to fire chemistry [33]. Among these, ISO 19702 [33] provides guidance for sampling and analyzing toxic gases and vapors in fire effluents using Fourier Transform Infrared (FTIR) spectroscopy. Additionally, ISO/TS 21397 [35] addresses FTIR analysis of fire effluents in cone calorimeter tests, while ISO 13344 [36] focuses on estimating the lethal toxic potency of fire effluents. For calculating pollutant emissions, ISO 19703 [37] is recommended including calculating species yields, equivalence ratios, and combustion efficiency in experimental fires.

4.5. Heat release rate

Heat release rate (HRR) stands out as the most crucial parameter in assessing the size of a fire and its associated hazard [32]. HRR is significantly more important in assessing fire hazard than factors such as ignitability, fire spread, and toxic effluent emissions.

The first instrument for measuring HRR was developed in the late 1960s at Ohio State University (OSU) in the USA [38]. This instrument is now standardized through the ASTM E906 Standard Test Method for Heat and Visible Smoke Release Rates for Materials and Products Using a Thermopile Method [39]. It became the first method for determining HRR to be regulated after the Federal Aviation Administration (FAA) established a clear correlation between HRR size and the time needed to rescue passengers from a burning aircraft under real-scale conditions [40]. Around the same time, Tewerson from FM Global began developing a fire propagation apparatus, a device designed for measuring HRR and fire release. Unlike OSU devices that rely on temperature measurement, this apparatus calculates HRR by measuring the concentration of CO2 in combustion products, providing a more reliable method.

Today, the most widely used instrument for determining heat release rate is the bench-scale cone calorimeter, designed by Babrauskas from NIST in the early 1980s. This device exposes a sample measuring 10 x 10 cm to a uniform incident heat flux of up to 100 kW/m^2 using a truncated cone heater. Combustion products are conveyed through a duct where oxygen content, and optionally the concentration of CO, CO2, and H2O, are precisely measured in real time. The HRR is calculated based on the oxygen depletion principle. Additionally, the device is utilized to determine the flammability characteristics of materials, mass loss over time, and smoke release. The device gained popularity after establishing a correlation between the results obtained on the cone calorimeter and full-scale tests [41]. It's considered one of the most widely used devices in fire research and fire hazard assessment.

Although small-scale testing is practical, it may not provide insight into the flammability characteristics of whole products, especially if they are highly heterogeneous or contain multiple layers. For such cases, medium and large-scale tests based on the oxygen depletion principle are utilized.

The Intermediate Scale Calorimeter (ICAL) is a device used in North America for medium-scale fire tests. The test method is standardized through ASTM E1623 Standard Test Method for Determination of Fire and Thermal Parameters of Materials, Products, and Systems Using an Intermediate Scale Calorimeter (ICAL) [42]. A flat sample measuring 1 x 1m is exposed to a radiant heater source (gas-fired radiant panel) with a range of heat fluxes up to 50 kW/m^2. This test has very good correlations with full-scale tests, but it is still not included in the regulatory framework.

On the other hand, in the EU, the Single Burning Item (SBI) test EN 13823 Reaction to fire tests for building products - Building products excluding floorings exposed to the thermal attack by a single burning item [43] is a regulatory intermediate-scale test that is used to regulate building products. In the SBI test, two samples measuring 1.5 x 1.5 m and 1.5 x 0.5 m are exposed to a 30 kW radiation source (propane gas burner). During the test, heat release rate, smoke release, flame spread, ignitability and flaming droplets are determined. FIGRA (fire growth rate) and SMOGRA (smoke growth rate) are two condensed variables that are used for regulatory and classification purposes.

However, the most reliable method to evaluate the fire performance of materials and products is through full-scale testing. ASTM and ISO organizations have developed several full-scale tests, with the room-corner test being the most widely used. ASTM E2257 Standard Test Method For Room Fire Test Of Wall And Ceiling Materials And Assemblies [44] test serves as an alternative to Steiner testing, offering various engineering quantities useful for fire calculations and material/product comparisons.

5. DESCRIPTION OF CUSTOM-MADE INSTALLATION FOR EVALUATION OF FLAMMABILITY PARAMETERS

Unlike the oxygen bomb calorimeter, which undergoes combustion with complete oxidation, small bench-scale devices such as the fire propagation apparatus, cone calorimeter, and mass loss calorimeter analyze flammability parameters as they manifest in real fires, under specific heat flux conditions and constant ventilation.

Despite the simplicity of measuring the flammability parameters of materials in fire conditions with a mass loss calorimeter, a limitation of this instrument is the lack of analysis of evolved gases and soot smoke particles. Using the mass loss calorimeter with an appropriate hood enables users to conduct thermal exposure studies under nearly identical conditions to those employed in the cone calorimeter. This setup enables the visual observation of specimen reactions and the measurement of flammability parameters. The fire model incorporated in the mass loss calorimeter complies with the specifications of the ISO 5660 Cone Calorimeter, which ensures that equivalent results are achieved compared to the cone calorimeter for various materials [45].

The primary objective of coupling the mass loss calorimeter and FTIR gas analyzer is to conduct more detailed analyses of the toxic gases emitted during specific stages of thermal decomposition and flame combustion of the samples tested in the cone calorimeter.

The parameters measured with the mass loss calorimeter are: heat release rate (HHR), time to ignition (tig), and mass loss rate (MLR), Total Heat release (THR), and Effective Heat of Combustion (EHC) of materials. While measuring the flammability parameters of materials in fire conditions with mass loss calorimeter is straightforward, a limitation of this device is its inability to analyze evolved gases.

In this regard, at the Faculty of Occupational Safety in Niš, University of Niš, Serbia, within the Fire Protection Laboratory (https://www.znrfak.ni.ac.rs/fpl/), a custom-made hood was developed for simultaneous determination of the flammability characteristics of materials of various structures and types, and fire effluents resulting from the combustion process in real-time (see Fig 1). To perform this kind of experiment, the laboratory is equipped with the following equipment:

- Bench scale device, mass loss calorimeter (Fire Testing Technology (FTT), UK)
- FTIR gas analyzer, GASMET DX4000 (Gasmet, Finland)

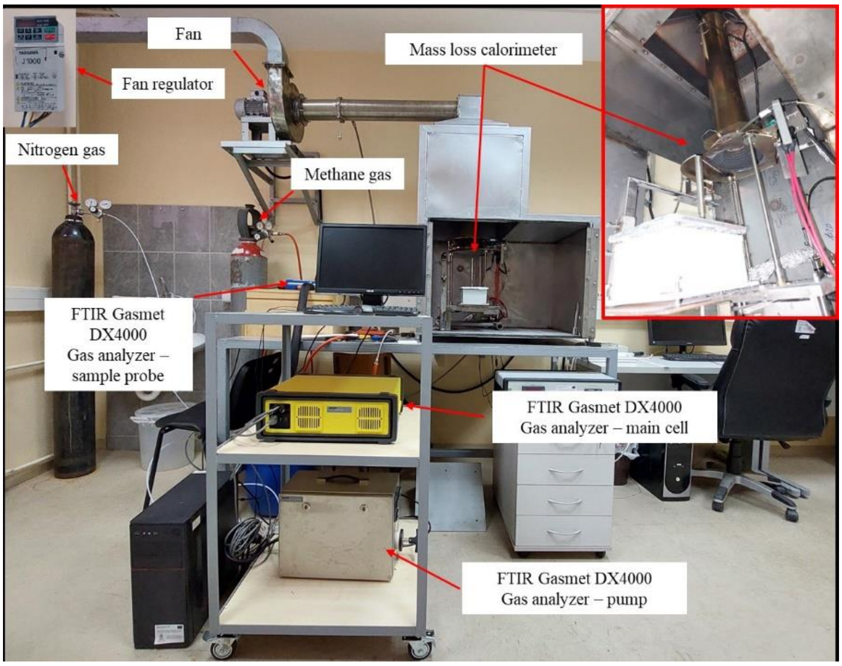

Fig. 1 Experimental installation with the adapted mass loss calorimeter [46]

5.1. Mass loss calorimeter

Heat release rate, as the single most important variable in determining the hazard from a fire, can be calculated by measuring the depletion of oxygen in the fire atmosphere. Devices for measuring oxygen concentration, particularly those of the paramagnetic type, have now reached a level of precision capable of detecting the subtle variations in oxygen levels necessary for determining heat release rates. Given that heat release apparatuses based on oxygen consumption principles tend to be quite expensive, an alternative solution may lie in devices utilizing the thermopile technique, such as those found in mass loss calorimeters.

The mass loss calorimeter features a thermopile consisting of four thermocouples positioned at the top of a chimney, directly connected to the conical heater. This chimney collects all the smoke released during the fire test emitted during the fire test, while the thermopile assesses the heat release rate.

Common characteristics that describe the utility of the mass loss calorimeter include:
- validity and accuracy;
- precision, repeatability, and reproducibility;
- ease of calibration;
- simplified specimen preparation to minimize uncertainties in starting time;
- specimen size can reach up to 100 mm by 100 mm, with variable thickness up to 50 mm;
- ease of data analysis and interpretation;
- simple and affordable enough to be useful for testing laboratories;
- short preparation and testing time;
- ruggedness of apparatus;
- ease of physical installation;
- provided ignition with electric spark (piloted ignition);
- sample mass loss measurement system.

Similar to the cone calorimeter, the mass loss calorimeter employs a conical electric heater to sustain a steady and uniform heat flux of up to 100 kW/m^2, enabling the replication of diverse fire conditions. Furthermore, the heat flux can be easily calibrated before each test.

The parameters measured with mass loss calorimeter are: heat release rate (HHR), time to ignition (tig), mass loss rate (MLR), Total Heat release (THR), and Effective Heat of Combustion (EHC) of materials.

- **Apparatus design**

The mass loss calorimeter instrument consists of the following assemblies:
1. Fire Model which consists of conical radiant heater, heater shutter assembly, spark igniter, 3 thermocouple sockets for cone heater control;
2. Control Unit;
3. Load Cell Assembly;
4. Chimney and Thermopile;
5. Calibration burner and flow meters;
6. Software interface and MLCCalc software.

Figure 2, extracted from the instruction manual for the mass loss calorimeter, illustrates the constituent elements of the device [47].

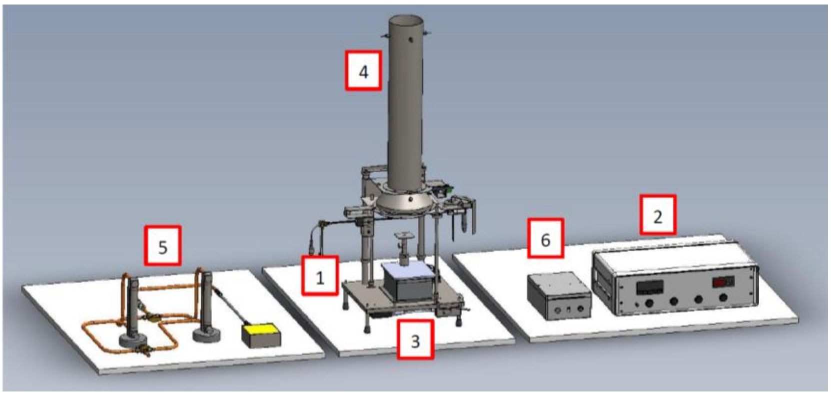

Fig. 2 Mass loss calorimeter components [47]

The basic mass loss calorimeter comprises solely a fire model and a load cell, intended for conducting mass loss and ignitability measurements primarily for screening purposes. However, the device discussed in this paper represents a modified version. To facilitate measurements of the heat release rate, an advanced calculation method was developed by the equipment producer. This method involves integrating an additional thermopile onto the fume stack, that allows for the digital deconvolution of the fume stack's thermal response into radiant and convective energy absorption components. Subsequently, the processed signal yields a composite value that exhibits a linear correlation with the measured HRR of reference materials [45].

- **Conical radiant heater**

The conical radiant heater can attain sufficiently high levels of irradiance, and maintain a uniformly distributed irradiance across the entire surface of the specimen. The room fire burning test reveals that maximum gas temperatures exceed 1000 °C, corresponding to irradiance levels reaching approximately 150 kW/m^2 on the walls and contents. In line with this, similar to the cone calorimeter, the conical heater within the mass loss calorimeter generates controlled heat fluxes ranging from 10 to 100 kW/m^2.

The design of the conical heater was borrowed from the ISO 5657 ignitability apparatus, specifically for its central hole feature [48]. This design effectively prevents the formation of a hot spot at the center of the sample and ensures that flames from the specimen do not splash onto the heater coil. The schematic view of the conical heater is shown in Figure 3.

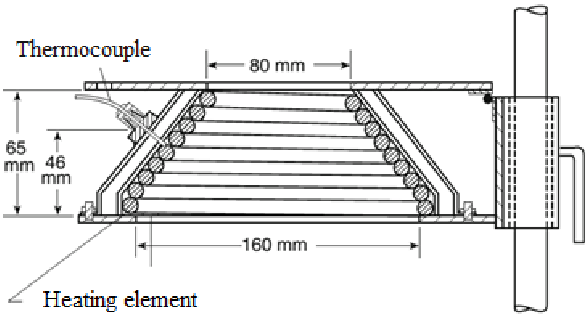

Fig. 3 Cross-section view through the heater [48]

Additionally, the conical heater is equipped with a removable radiation shield, which serves to protect the specimen from irradiance just before the commencement of the test.

- **Load cell**

A standard load cell has a maximum load capacity of 500 grams. The device has an accuracy of 0.1 g and a 90% response time of less than 3 seconds. The load cell measures the real-time change in mass loss of the specimen during the experiment. All data are recorded using the MLCCalc software, and the mass loss data changes are displayed on the control unit.

- **Specimen mounting**

Specimens are cut to 100 x 100 mm in size, and with thickness up to 50 mm. With the prepared size and shape, samples are inserted into the sample holder. The sample holder has the shape of a square pan with an opening of external dimensions measuring 106 by 106 mm at the top, and a depth of 25 mm. Equipped with a handle for easy insertion and removal, it also features a mechanism to ensure the central positioning of the specimen under the heater and proper alignment with the load cell. All test specimens must be tested with the retainer frame, which is part of the sample holder (see Fig. 4).

Adjustments for specimen thicknesses less than 50 mm can be made by adding extra refractory pad layers. A spacing of 25 mm needs to be set up between the bottom of the cone and the top of the specimen, to achieve uniform distribution of heat flux across the entire specimen surface. Before positioning the sample holder along with the sample, the sides of the testing specimen need to be protected with aluminum foil, ensuring a single piece fits the bottom and sides and is cut flush with the top.

The following procedure for specimen preparation is:

- Put the retainer frame on a flat surface facing downwards.
- Insert a foil-wrapped specimen into the retainer frame, with the exposed surface facing downwards.
- Put layers of ceramic fiber blanket on top of the sample, until 2 layers extend above the rim of the retainer frame.
- Fit the sample holder into the retainer frame.
- Position the sample holder atop the load cell, ensuring that the specimen holder is centered with respect to the cone heater.

Fig. 4 Sample holder with ceramic fiber insulating material and retainer frame

- **Spark ignitor**

The spark ignitor is placed where it is expected to reach the lower flammable limit as the specimen begins pyrolysis. However, it should not be positioned so close to the specimen surface that minor swelling of the specimen would disrupt the ignition process. In the mass loss calorimeter, the spark ignitors are ideally positioned 13 mm above the center of the specimen. Additionally, users have the option to choose between piloted ignition with a spark ignitor or simultaneous ignition without one.

- **Calibration**

The mass loss calorimeter utilizes three distinct calibrations.

First, calibration of the conical heater is necessary. This calibration aims to establish a correlation between the temperature of the heater and the heat flux it generates. It is conducted using a Schmidt-Boelter water-cooled fluxmeter, specifically calibrated for the heat flux intended for the test.

The temperatures of the conical heaters corresponding to the determined values of the heat flux on the mass loss calorimeter at the Faculty of Occupational Safety in Niš are presented in Table 1. Additionally, a comparative analysis of the temperatures specified in the [49] is provided. The measured temperature corresponding to the flow speed in the duct is 3.4 m/s, while below the cone heater, it is 0.1 ± 0.02 m/s.

Table 1 Heat flux from cone heater and temperature

Irradiance [kW/m^2]	Heater temperature at Faculty of Occupational Safety [°C]	Heater temperature in [49] [°C]
10	416	422
20	557	547
25	609	592
30	649	628
40	720	689
50	778	744
60	835	783
70	883	825

The second calibration is related to thermocouples, which are calibrated using a methane burner. This burner is designed to allow gas to flow out through the sand aggregate, ensuring an even distribution of the flame across the burner's entire surface. Initial calibration of the thermocouples is conducted for heat powers of 0.5 kW, 0.75 kW, 1 kW, 2 kW, 3 kW, 4 kW, and 5 kW. For each of these heat powers, the flow of methane with a purity of at least 99.5% is precisely regulated (refer to Table 2).

Table 2 The Flow of Methane Required for Thermocouple Calibration

Heat power [kW]	The flow of methane [l/min]
0.5	0.83
0.75	1.26
1	1.68
2	3.35
3	5.03
4	6.7
5	8.38

The objective of this calibration is to establish a correlation between the measured temperature on the thermocouples and the current heat output of the methane burner. In Figure 5, an example graph illustrating thermocouple calibration for a heat flux of 50 kW/m^2 can be observed.

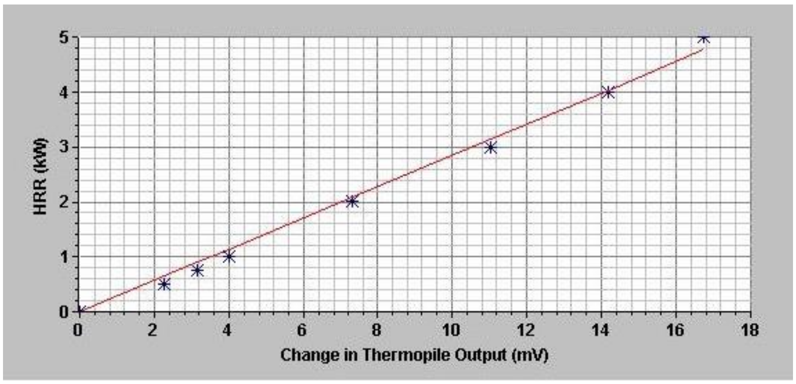

Fig. 5 Example of thermocouple calibration line for a heat flux of 50 kW/m^2

Additionally, alongside this calibration, it is essential to conduct a daily check of the heat release rate at 3 kW before commencing work with the device.

To measure the mass loss accurately, it is imperative to calibrate the scale before using the device. This calibration is conducted based on the expected mass of the sample, enhancing the precision of the measurement.

5.2. Sampling and measurement of fire effluents

A crucial aspect for evaluating the toxic hazard in fires involves understanding the variety, characteristics, and concentrations of toxic substances and smoke particles emitted in the fire effluent. Due to the frequently harsh conditions prevailing in fires, specialized methods for sampling and analysis are necessary to ensure meaningful results [50].

The GASMET DX-4000 is a gas analyzer specifically engineered for measuring the concentrations of different emission and immission gases. Its operational principle is rooted in utilizing Fourier transform infrared (FTIR) spectroscopy to determine gas concentrations. The DX-4000 analyzer is commonly employed in monitoring stack emissions, catalytic process control, and other scenarios requiring accurate monitoring of multiple gas compounds in hot and humid gases. The heating of the sampling cell ensures a condensation-free sampling process, even at high water vapor concentrations.

The GASMET DX-4000 gas analyzer includes the following components:

- GASMET DX-4000 gas analyzer;
- GASMET - Portable Sampling System;
- portable probe for gas sampling PSP4000-H;
- sampling line.

A sampling probe, designed in accordance with ISO/TS 21397, was used to sample effluents from the extraction system during the combustion process in the mass loss calorimeter [35].

The FTIR Gasmet DX4000 features a library of 16 gases that can be continuously measured: Water vapor (H_2O), Carbon dioxide (CO_2), Carbon monoxide (CO), Nitrous oxide (N_2O), Nitrogen monoxide (NO), Nitrogen dioxide (NO_2), Sulfur dioxide (SO_2), Ammonia (NH_3), Hydrogen chloride (HCl), Hydrogen fluoride (HF), Methane (CH_4), Ethane (C_2H_6), Ethylene (C_2H_4), Propane (C_3H_8), n-Hexane (C_6H_{14}) and Formaldehyde (CHOH).

According to information from the manufacturer, Gasmet's FTIR analyzers do not require any span or re-calibrations after the initial factory calibration. Performing a daily background spectrum measurement using zero gas, such as nitrogen (N_2), is sufficient to maintain measurement accuracy. The gas sampling interval can be configured with different time values, although 3 seconds is the theoretical minimum due to technical limitations of the instrument. Sampled gases were transported to an FTIR gas cell through a heated PTFE sampling line conditioned at 180 °C.

As the mass loss calorimeter and gas analyzer operate independently, it's essential to synchronize the time delay between these instruments. Suitable adjustments can be made before experiments, once all data are collected.

6. Conclusion

This study highlights the importance of rigorous fire testing for building materials to enhance safety and ensure compliance with local codes. By utilizing standardized methods and advanced equipment, reliable data on material flammability and fire effluent characteristics can be obtained.

Numerous studies in the literature focus on determining the flammability characteristics of materials with various structures and purposes using small bench-scale devices, such as Cone Calorimeters and Fire Propagation Apparatus. These instruments are considered state-of-the-art in fire engineering. However, as an alternative to these devices, the less expensive Mass Loss Calorimeter can be considered. This device employs the same fire model as the Cone Calorimeter but includes an additional stainless steel chimney equipped with a thermopile for measuring heat release rate. Hence, it represents a suitable alternative to the Cone Calorimeter for determining the flammability characteristics of materials.

In addition, this paper details a custom-made installation at the Fire Protection Laboratory of the Faculty of Occupational Safety, University of Niš, designed to assess the flammability parameters of various materials. This advanced setup, which includes a Mass Loss Calorimeter and an FTIR gas analyzer, enables thorough and simultaneous real-time measurement of flammability characteristics and fire effluent analysis.

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PREGLED UOBIČAJENIH METODA ZA ISPITIVANJE POŽARA

Ispitivanje materijala na dejstvo požara je ključno za procenu ponašanja građevinskih materijala u uslovima požara. Tokom ispitivanja dobija se neposredan uvid u važne promenljive poput zapaljivosti, brzine oslobađanja toplote i produkcije dima. Ovaj rad daje pregled uobičajenih metoda ispitivanja materijala na dejstvo požara, uključujući procedure za odabir testa, pripremu uzoraka, prikupljanje podataka i analizu. Metode ispitivanja definisane su u standardima koje izdaju globalno respektabilne institucije poput ISO, EN, ASTM i UL. Ovi standardi garantuju i obezbeđuju doslednost i pouzdanost propisanih metoda. Takođe, u ovom radu je dat detaljan opis instalacije u Laboratoriji za zaštitu od požara Fakulteta zaštite na radu Univerziteta u Nišu, koja se koristi za određivanje ključnih požarnih karakteristika za širok dijapazon materijala. Ova instalacija, koja se sastoji od kalorimetra za merenje toplotne snage i FTIR gasnog analizatora, može se koristiti za sveobuhvatno i istovremeno merenje parametara zapaljivosti u realnom vremenu i analizu efluenata požara.

Ključne reči: *ispitivanje požara, zapaljivost, efluenti požara, toplotna snaga, kalorimetar za merenje toplotne snage, gasni analizator.*