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Original Research

COMPACT VOC EMISSION TEST CHAMBER

KOMPAKTOWA KOMORA DO BADAŃ EMISJI LOTNYCH ZWIĄZKÓW ORGANICZNYCH

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Abstract

The publication presents the test method and construction of a compact VOC emission test chamber. Volatile organic compounds (VOCs) are mainly occur in paint and construction materials containing chemical resins, bitumen, binders, adhesives, etc., as well as residues of unreacted monomers. VOC emission testing is carried out on product samples in environmental chambers in accordance with PN-EN ISO 16000-9. The developed chamber, together with a proprietary control and test parameter acquisition system, allows testing at a stabilized temperature and humidity of air washing over the sample at a specified rate. Qualitative identification and quantitative testing of compounds present in the air inside chambers is carried out using a chromatograph, and the test results are compared with regulations on permissible chemical pollutants in the air of buildings and in atmospheric air. The developed chamber with horizontal sample loading is another version of VOC chambers, developed in response to market demands, offered and implemented in research laboratories by the Łukasiewicz Institute for Sustainable Technologies in Radom.

Keywords: test methodology, VOC, health protection, building materials, control

Streszczenie

Publikacja przedstawia metodę badań oraz budowę kompaktowej komory do badań emisji lotnych związków organicznych. Lotne związki organiczne (VOC) występują głównie w wyrobach malarskich oraz budowlanych zawierających żywice chemoutwardzalne, bitumy, lepiszcza, kleje itp., a także pozostałości nieprzereagowanych monomerów. Badanie emisji VOC odbywa się na próbkach wyrobów w klimatyzowanych komorach zgodnie z normą PN-EN ISO 16000-9. Opracowana komora wraz z autorskim systemem sterowania i akwizycji parametrów badań pozwala na prowadzenie badań w stabilizowanej temperaturze i wilgotności powietrza omywającego próbkę z określoną prędkością. Identyfikację jakościową i badanie ilościowe związków występujących w powietrzu komór przeprowadza się za pomocą chromatografu, a wyniki badań porównuje z przepisami dotyczącymi dopuszczalnych zanieczyszczeń chemicznych w powietrzu budynków i w powietrzu atmosferycznym. Opracowana komora z poziomym załadunkiem próbek jest kolejną wersją komór VOC, powstałą w odpowiedzi na oczekiwania rynku, oferowanych i wdrażanych w laboratoriach badawczych przez Ł-ITEE w Radomiu.

Słowa kluczowe: metodyka badań, VOC, ochrona zdrowia, materiały budowlane, sterowanie

1. Introduction

Volatile organic compounds (VOCs) are mainly found in painting products (Afshari et al., 2003). They can also be found in numerous other building products made of plastics, containing chemically cured resins, bitumen (Boczkaj et al., 2014), binders, adhesives, etc. (Liu et al., 2012), as well as residues of unreacted monomers. VOC emission testing is carried out on product samples in environmental chambers in accordance with PN-EN ISO 16000-9, "Indoor air.

Part 9: Determination of the emission of volatile organic compounds from building products and furnishing. Emission test chamber method" (PN-EN ISO 16000-9). Thermostatic chambers at the appropriate temperature and humidity, and ventilated at a certain speed are used for testing (Koziol et al., 2019; Wei Wenjuan et al., 2012; Zhibini et al., 2003). Qualitative identification and quantitative testing of compounds present in the air inside chambers is carried out using a chromatograph (Demeestere et al., 2008; Niesłochowski, 2013; Ribes et al., 2007), and the test results are compared with regulations on permissible chemical pollutants in the air of buildings and in atmospheric air (Regulation of the Minister of Environment, 2002; Ordinance of the Ministry of Health and Social Welfare, 1996). VOC emission tests have become standard tests of hygienic properties of certain building products and materials, as well as common products (Oppl, 2014; Liu et al., 2012).

The rationale for carrying out the project is the need for an innovative solution (Invernizzi et al., 2021) that relies on the horizontal layout of the emission test chamber, which facilitates the placement of test objects inside it, especially those of higher mass. Another innovation is the ability to carry out tests at temperatures higher than those required by PN-EN ISO 16000-9, which makes it possible to accelerate tests and reproduce special conditions of use, such as intensively heated thermal roof insulation.

The results of the task are used in the design and manufacturing activities of the Institute's Prototyping Center, which for a number of years has been the developer and supplier of VOC emission test equipment kits to scientific (Fig. 1a) and industrial (Fig. 1b) laboratories in Poland (Koziol et al., 2019).

a) b)

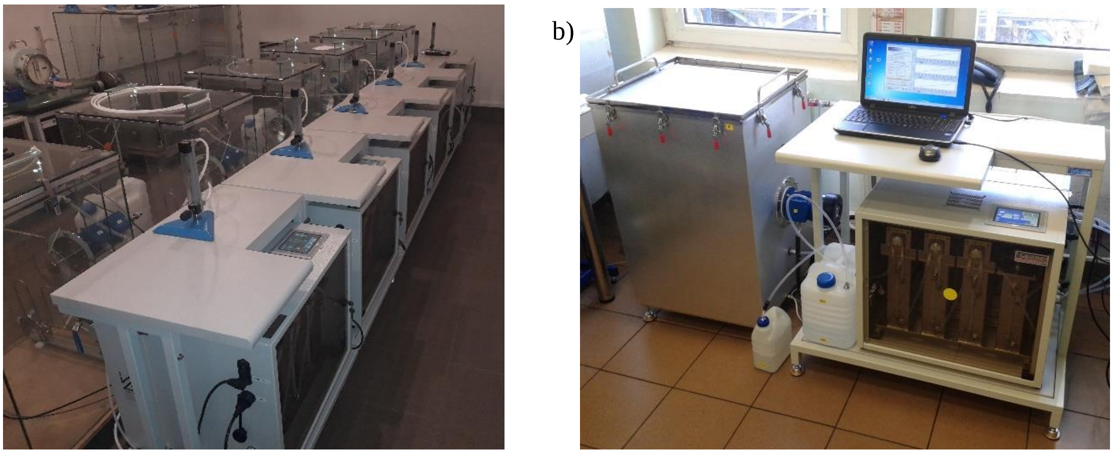

Fig. 1. Examples of VOV chambers implemented in: a) research centers, b) industry

2. The concept of the test chamber

The functional diagram of the VOC emission test stand (Fig. 2) was developed based on the recommendations in Annex C of PN-EN ISO 16000-9. In terms of functionality, three main modules can be distinguished: the emission test chamber, the air preparation module and the control system.

Emission test chambers and sampling system components in contact with emitted VOCs should be made of polished stainless steel or glass. The design of the chamber should provide:

- adequate tightness and strength to allow operation under positive pressure to avoid the influence of the laboratory atmosphere on the measurement result,
- the ability to maintain temperature and humidity at the desired level,
- flow velocity of homogenized air over the surface of the test sample.

The purpose of the air preparation module is to supply the test space of the chamber with the appropriate amount of air of the desired purity and humidity.

The role of the control system is to maintain the set operating parameters of the test stand and to acquire and archive measurement data.

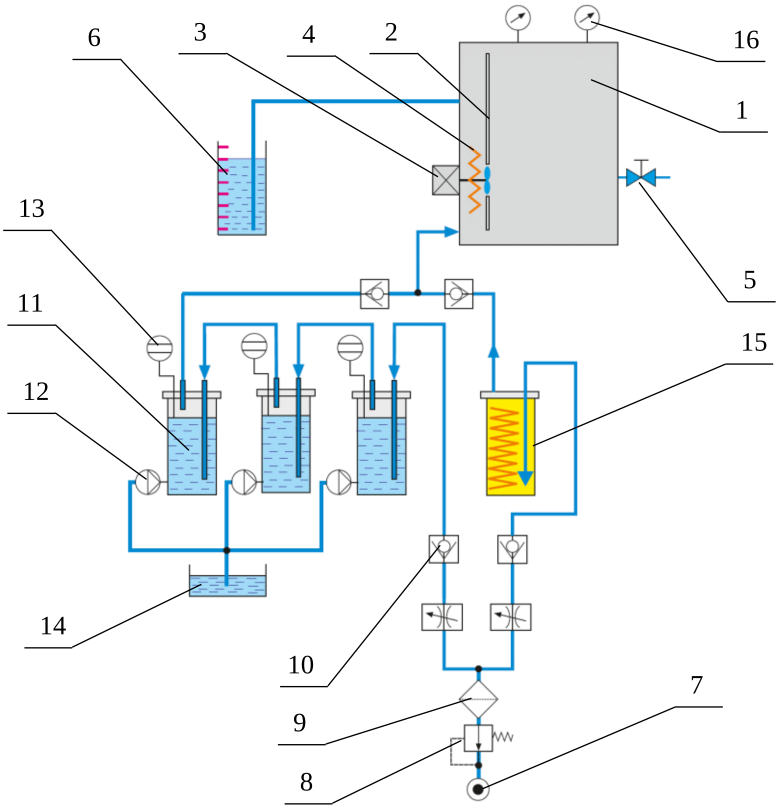

Fig. 2. Functional diagram of the test stand for testing VOC emissions: 1 - emission chamber, 2 - partition, 3 - fan, 4 - heater, 5 - sample intake valve, 6 - hydrostatic tank, 7 - compressor, 8 - pressure regulator, 9 - filter, 10 - check valve, 11 - washing bottle, 12 - pump, 13 - level gauge, 14 - buffer tank, 15 - dehumidifier, 16 - temperature and humidity sensor

Table 1. Basic parameters of the designed test chamber

Parameter	Value
Capacity	225 dm^3 ± 0.0045 dm^3
Maximum operating pressure	500 Pa
Air temperature in the chamber	max +65°C
Relative humidity of the supplied air	max RH 50% ±5% RH
Air velocity at the sample surface	0.1 ÷ 0.3 m/s

3. Research device design

Based on the assumptions, virtual 3D CAD models of the complete device (Fig. 3) and all the elements that make up its structure were developed. The model was developed in Autodesk Inventor Professional.

Test chamber 1 is made of stainless steel and is the shape of a cuboid with a capacity of 225 liters (Fig. 4). It is equipped with a front door 2 with bolt locks 3. The required tightness of the test space is provided by a type 4 O-ring made of low-emission fluorocarbon-based rubber.

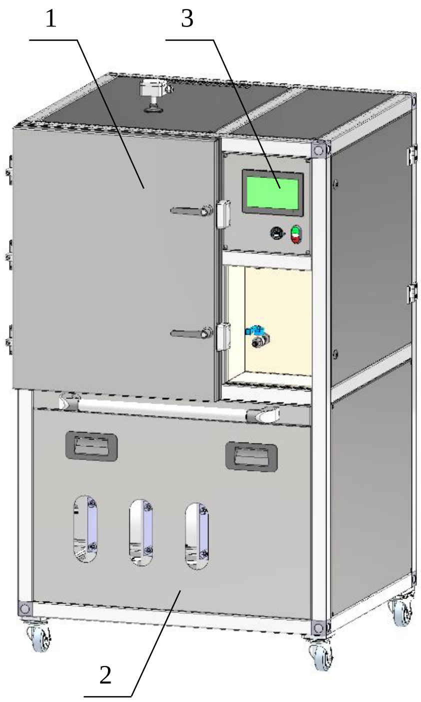

Fig. 3. CAD model of the VOC emissions testing device: 1 - test chamber, 2 - air preparation module, 3 - control system

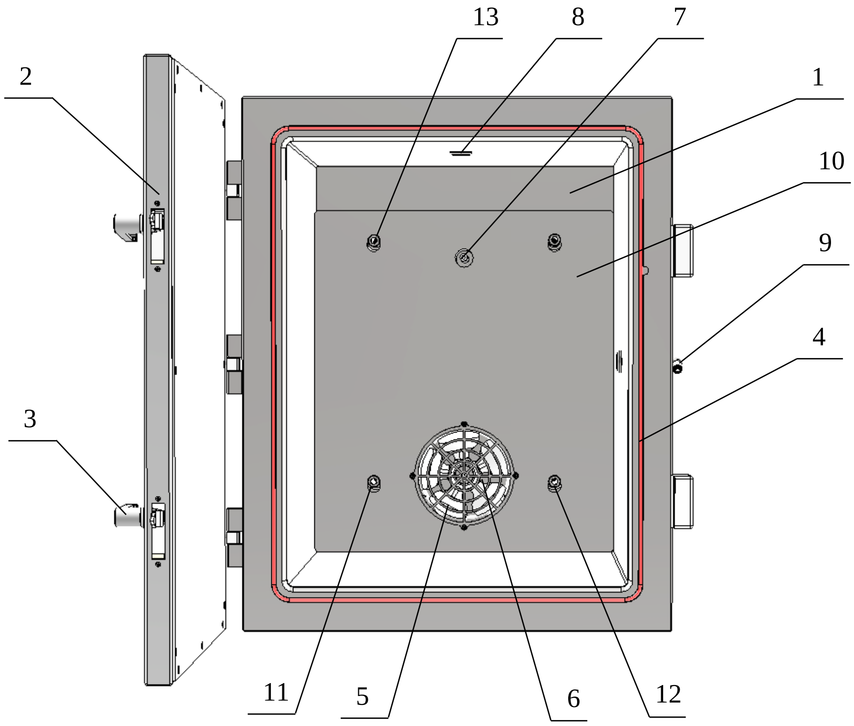

Fig. 4. CAD model of the test chamber: 1 - emission chamber, 2 - front door, 3 - bolt lock, 4 - seal, 5 - heating module, 6 - fan, 7 - temperature and humidity sensor, 8 - air velocity sensor grommet, 9 - air intake port, 10 - partition, 11 - dry air inlet, 12 - humid air inlet, 13 - air outlet

The proper circulation of air in the chamber, its homogenization and the flow of air at the appropriate speed around the samples is provided by a fan (6) with a drive located outside the chamber together with a partition (10). Heating module (5) is responsible for maintaining the set temperature during testing or warming up at the set temperature, such as 60°C. A temperature and humidity sensor (7) is located in the rear wall of the chamber. At the top of the chamber there is a grommet (8) that allows the insertion of an air velocity sensor washing over the tested sample. On the right side of the chamber, there is a port (9) connected to a ball valve for drawing the air to be analyzed. Symmetrically, on both sides of the fan are placed air inlets for dry (11) and humid (12) air. The flow of air through the chamber is provided by two fittings (13) located at the top of the chamber and connected to a water tank that provides positive pressure in the test chamber.

The air preparation module is located at the bottom of the test device (Fig. 5). The supporting structure is a rack (1) attached to two ball guides (2) that allow the module to be pulled out from inside the unit for service purposes. An oil-free compressor (3) with a minimum output of 500 l/h is located at the rear of the rack. Compressed air flows through pressure regulator (4) and carbon filter (5). Pre-cleaned air is directed to two flow regulators (6). The first regulates the value of the air flow through three washing bottles connected in series, filled with demineralized water. The second regulates the value of the air flow through the dehumidifier (7) filled with silica gel. The ratio of the values of the humid and dry air streams is responsible for the humidity of the air supplied to the emission chamber, while the sum of the streams determines the air flow rate through the chamber.

Each washing bottle, which cleans and humidifies air, is equipped with an optical level gauge (8) and a water level sensor (13). The water level in the washing bottles is topped up automatically with peristaltic pumps (9) drawing water from a buffer tank (12). The dehumidifier is equipped with a sight glass that allows to visually assess the moisture level in the silica gel and decide whether it needs to be dried.

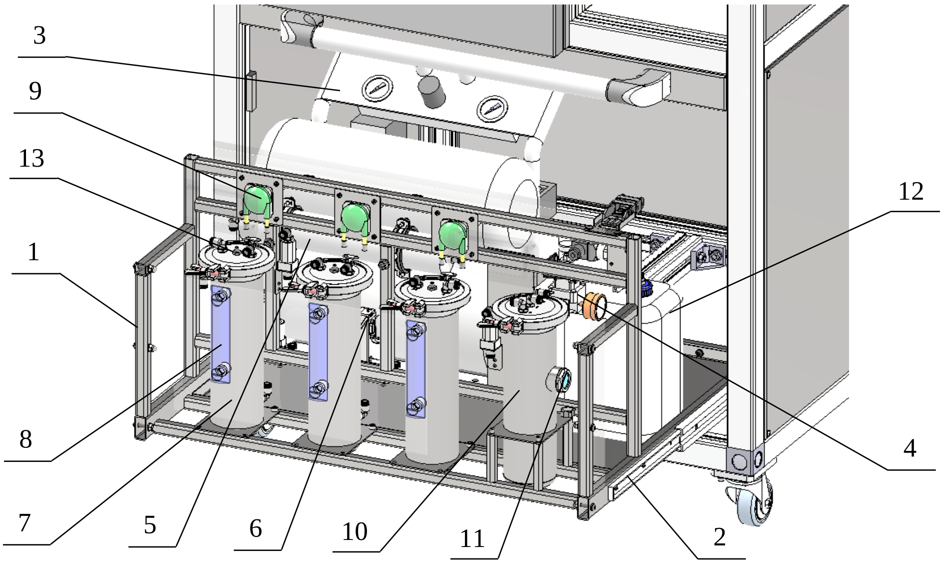

Fig. 5. Air preparation module: 1 - rack, 2 - ball guides, 3 - compressor, 4 - pressure regulator, 5 - carbon filter, 6 - air flow regulators, 7 - washing bottle, 8 - level gauge, 9 - peristaltic pump, 10 - dehumidifier, 11 - sight glass, 12 - buffer tank, 13 - level sensor

Modern equipment, and especially research equipment, is characterized by the integration of mechanical systems with advanced control systems. The designed control system (Fig. 6) provides the following functions:

- controlling the temperature of the air in the chamber,
- controlling the humidity of the air in the chamber,
- adjusting the amount of airflow through the chamber,
- adjusting the air velocity over the sample
- automatic replenishment of demineralized water level.

Regardless of the parameter settings, the control system will enable:

- writing a data record to the device memory every ½ h or 1 h,
- erasing device memory,
- browsing stored data records using the control panel,
- adjusting time and date displayed on the device (the set date and time are used to save the data record),
- adjusting the contrast of the control panel, operation in Polish and English.

The developed user interface will include graphical visualizations of the test processes carried out by the device. The use of mnemonic icons for the control panel interface (Fig. 7) is envisioned to facilitate menu navigation and simplify operation.

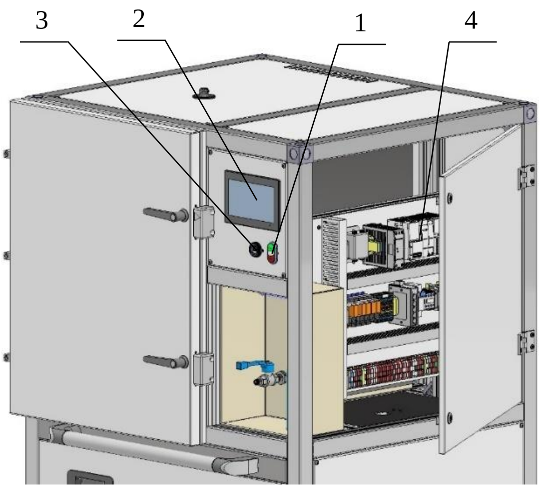

Fig. 6. VOC emission test chamber control system components: 1 - START-STOP button, 2 - control panel, 3 - RJ communication connection, 4 - PLC controller

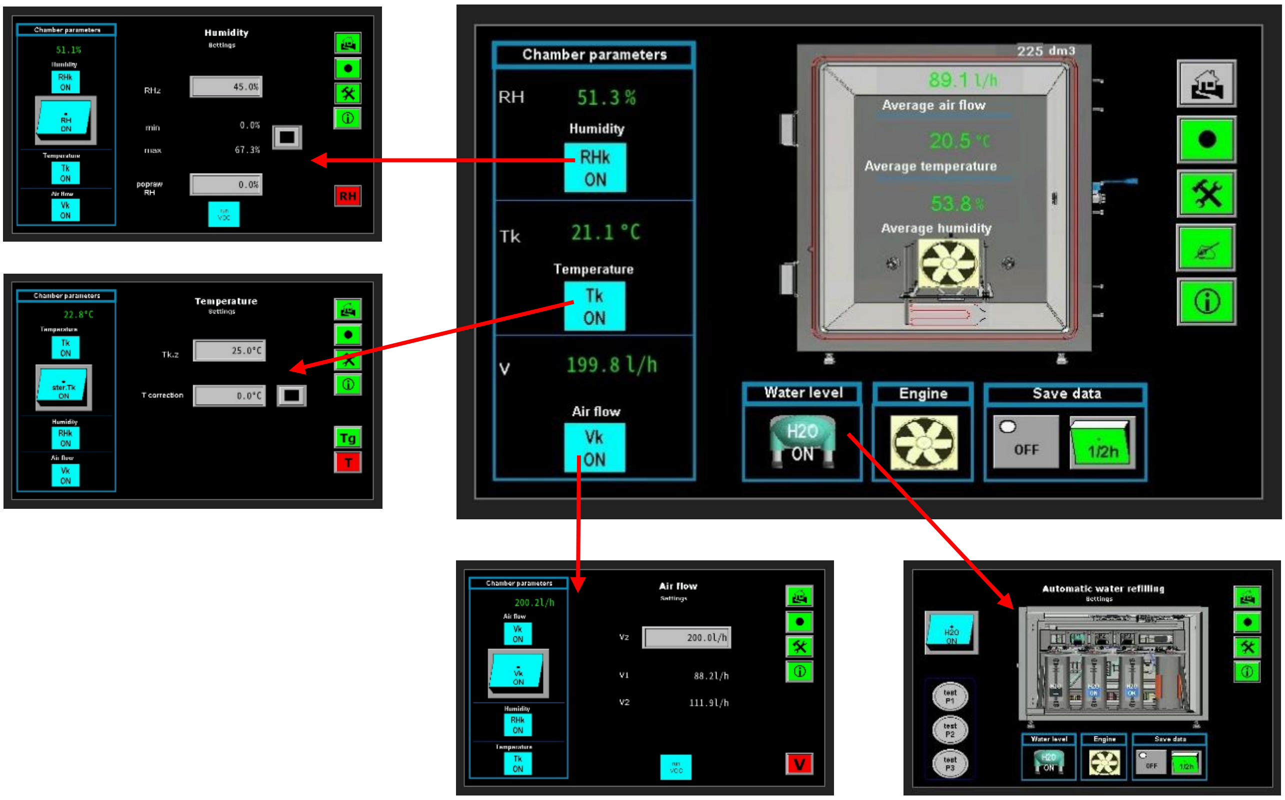

Fig. 7. Control panel screen design

4. Prototype verification

On the basis of the assumptions made and the model and technical documentation developed, a prototype VOC emission test stand with a horizontal emission chamber was built (Fig. 8).

The sampling port for air contaminated by the VOC emitted by the tested product (Fig. 9), is located in a recess in which a tank of water is placed to establish the positive air pressure in the chamber during testing.

In the process of verifying the prototype, test and operational studies were conducted under operating conditions consistent with the real ones.

The following inspection procedures were implemented to check the compliance of the device's operating parameters with normative requirements: checking cleanliness, tightness, airflow, temperature and humidity.

Fig. 8. Prototype of the test stand: 1 - emission chamber, 2 - control panel, 3 - air sampling port and water tank, 4 - pull-out shelf, 5 - air preparation module

a) b)

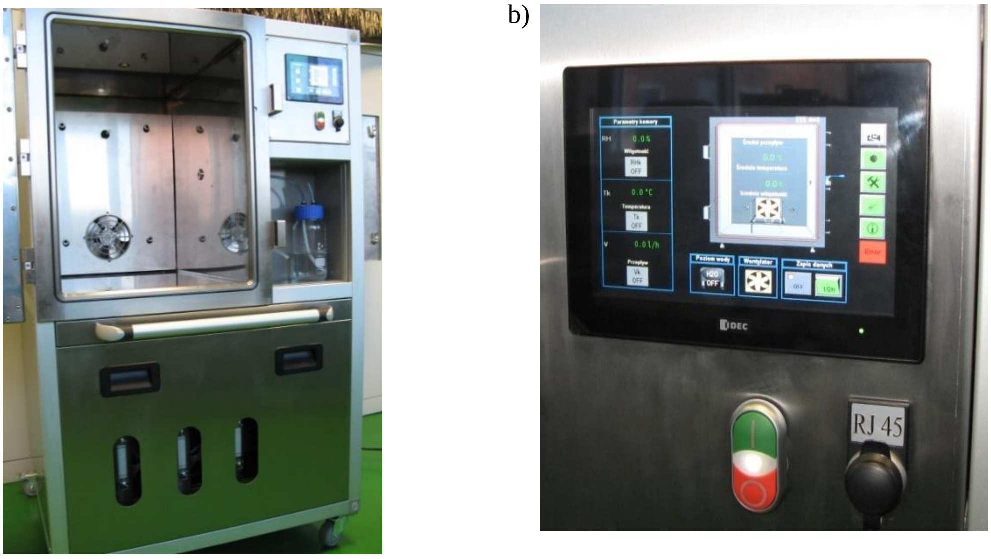

Fig. 9. Prototype of the test stand: a) view with the emission chamber open, b) control panel

4.1. Checking cleanliness of the air in the emission chamber

In accordance with the standard PN-EN ISO 16000-9: Part 9, Section 8.2, the quality of the supply air should meet the following criteria:

- the concentration of total volatile organic compounds (TVOCs) in the chamber should be less than 20 μg/m3,
- The concentration of a single targeted VOC in the chamber should be less than 2 μg/m^3.

Air samples for testing were taken in accordance with ISO 16000-6:2021 (3 dm^3, flow rate of 6 dm^3/h). For adsorption of organic compounds, steel sorption tubes filled with 200 mg of Perkin Elmer Tenax TA were used and conditioned at 300°C before each analysis.

Determination of the concentration of VOCs was performed in accordance with PN-EN ISO 16017-1:2006. A Perkin Elmer Clarus 680 gas chromatograph equipped with a Clarus 680C MS mass detector and TurboMatrix 650 ATD thermal desorber was used for the analysis. Certified reference material – VOC-Mix 12 from Restek was used for quantitative analysis. Concentrations of other organic compounds were obtained using the equivalent method for toluene. The list of identified compounds is shown in Table 2.

The chromatogram obtained by analyzing the air sample from the test chamber and by analyzing the standard solution is shown in Fig. 10.

The resulting sum VOC concentration does not exceed the concentration of the maximum allowable concentration of volatile organic compounds according to ISO 16000-6 “Determination of volatile organic compounds in indoor and test chamber air by active sampling on Tenax TA sorbent, thermal desorption and gas chromatography using MS or MS-FID”.

Table 2. List of identified compounds

Identified compound	Concentration on tube [μg]	Actual concentration [μg/m^3]
1,2-dichlorobenzene	0.004589	1.481
Cis-3,3,5-trimethylcyclohexanol	0.000210	0.068
Benzyl alcohol	0.000358	0.115
Butoxyethoxy ethanol	0.000126	0.041
3,3,5-trimethylcyclohexyl methacrylate	0.000112	0.036
	TOTAL VOC [μg/m^3]	**1.741**

a)
b)

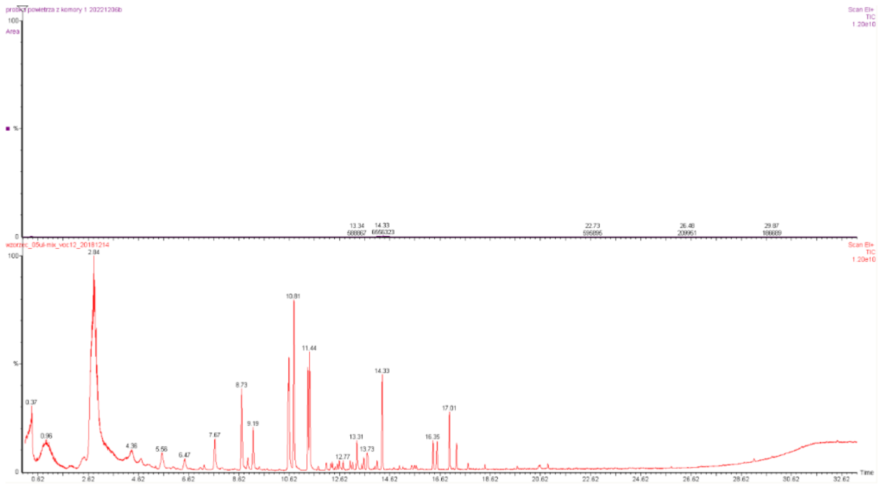

Fig. 10. Chromatogram obtained from the analysis of: a) air sample from the test chamber, b) standard solution

4.2. Tightness and airflow measurement

In accordance with EN ISO 16000-9: Part 9, Section 6.4 emission test chamber is considered air tight if the air loss is less than 5% of the air inlet folw. Two rotameters were used for testing. The first was connected between the output of the air preparation module supplying air to the chamber and its input. The other was connected to an open air sampling valve from the emission test chamber. A summary of the results of the airflow measurements is shown in Table 3.

Table 3. Airflow measurement results.

No.	Flow [dm³/h]			Loss [dm³/h]	Allowable loss [dm³/h]
	Set stream	Inlet stream	Outlet stream		
1	50 ±1	50 ±1	49 ±1	1	2.5
2	100 ±1	98 ±1	95 ±1	3	5
3	150 ±1	148 ±1	143 ±1	5	7.5
4	200 ±1	198 ±1	190 ±1	8	10
5	250 ±1	248 ±1	238 ±1	10	12.5

4.3. Temperature and humidity measurements

In accordance with EN ISO 16000-9: Part 9, Section 8.1 products intended for use in Europe should be tested at 23 °C ± 2 °C and RH 50% ± 5% during emission testing (ISO 554). The results of comparative measurements made with the chamber's instrumentation (thermohygrometer) and a reference gauge are shown in Tables 4 and 5.

Table 4. Temperature measurement results.

No.	Temperature [°C]			Absolute error [°C]	Allowable error [°C]
	set	thermo-hygrometer reading	reference gauge reading		
1	23	23.3 ±0.5	23.3 ±0.2	0.0	±2
2	24	24.0 ±0.5	24.04 ±0.2	0.04	±2
3	25	25.2 ±0.5	25.3 ±0.2	0.1	±2
4	50	52.4 ±0.5	50.5 ±0.2	1.9	±2

Table 5. Relative humidity measurement results.

No.	Humidity [%RH]			Absolute error [%RH]	Allowable error [%RH]
	set	thermo-hygrometer reading	reference gauge reading		
1	20	21.1 ±2	21.6 ±1	0.5	±5
2	40	40.4 ±2	41.7 ±1	1.3	±5
3	45	46.3 ±2	48 ±1	1.7	±5
4	50	50.7 ±2	53.1 ±1	2.4	±5

The results of the tests confirmed the readiness of the completed prototype to carry out standard tests of VOC emissions from samples of building materials, wood-based materials and other products emitting VOCs hazardous to health. Verification of the control system was also carried out towards the realization of the established test procedures. The tests confirmed the correct operation of the measurement and control system.

5. Conclusions

At the Łukasiewicz Institute for Sustainable Technologies in Radom, a prototype compact VOC emission test chamber has been developed and created, and has undergone comprehensive verification tests. The functional tests carried out confirmed the possibility of appropriate adjustment of the physical parameters of the implementation of emission tests while maintaining the air quality of purity required by the normative provisions for testing VOC emissions using the emission test chamber method. The design solution developed in the course of the work in the form of a horizontal VOC emission test chamber was used to fulfill an order for the supply of two test kits (Fig. 11) for a company producing wood-based insulation materials for the construction industry.

Fig. 11. View of implemented VOC emission test chambers

The original design solutions developed at Łukasiewicz - ITEE have been protected by industrial design registration (Rp.xxxxx).

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