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OVERPRESSURE CHAMBER FOR TESTING IN HIGH AIR PURITY CONDITIONS

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Key words: volatile organic compounds, VOC emissions, emission measurement, chamber method, measurement chamber.

Abstract: Volatile organic compounds (VOC) released into the air contribute to adverse climate changes, discomfort, and harmful effects on the human body. Principal sources of VOC emissions are industrial processes and, in the case of rooms for human residence, building materials and indoor furnishings. VOC emissions are monitored and restricted in line with legislation prevailing in the European Union and most countries of the world. The chamber method is used to measure noxious substance emissions from building materials and indoor furnishings.

The paper analyses the impact of regulatory requirements concerning emission test parameters and apparatus on technical solutions as part of air preparation assemblies and emission measurement chambers. The design of a 0.225 m³ steel chamber is presented, which was developed by our Institute based on our experience in the design and construction of similar apparatus. Results of verification testing are described.

Nadciśnieniowa komora do badań w warunkach wysokiej czystości atmosfery

Słowa kluczowe: lotne związki organiczne, emisja VOC, pomiar emisji, metoda komorowa, komora pomiarowa.

Streszczenie: Lotne związki organiczne (VOC) uwalniane do atmosfery są przyczyną niekorzystnych zmian klimatycznych oraz dyskomfortu i szkodliwego oddziaływania na organizm ludzki. Źródłem emisji VOC są głównie procesy przemysłowe, a w przypadku pomieszczeń przeznaczonych do przebywania ludzi materiały budowlane i elementy wyposażenia wnętrz. Zgodnie z prawem Unii Europejskiej i większości krajów świata emisja VOC jest monitorowana i ograniczana. Do oceny emisji substancji szkodliwych z materiałów budowlanych i elementów wyposażenia budynków stosowana jest metoda komorowa.

W artykule przedstawiono analizę wpływu wymagań normatywnych dotyczących parametrów testów emisyjnych i aparatury do ich realizacji na rozwiązania techniczne zespołów przygotowania atmosfery i komór badawczych do pomiaru emisji. Przedstawiono rozwiązanie konstrukcyjne komory stalowej o pojemności 0,225 m³ opracowane przez autorów artykułu na podstawie dotychczasowych doświadczeń w projektowaniu i budowie aparatury o podobnym zastosowaniu. Zamieszczono fotografie wykonanego zestawu pomiarowego i opisano wyniki badań weryfikacyjnych.

Introduction

Volatile organic compounds (VOC) are a group of organic compounds that easily become vapour or gas, exhibit high vapour or gas pressures, a low solubility in water, and boiling temperatures in the range of 50–250°C [1].

Volatile organic compounds are side products in a number of industrial processes and sources of

environmental pollution. Secondary pollution with substances arising from chemical reactions involving VOC in the environment is of particular importance [2–4]. Harmful VOC emissions are primarily associated with furniture, buildings, and plastic industry products. Lacquers, impregnates, paints, bituminous masses, binders, glues, and plastics in floors, painting and lacquer coats of furniture and rooms, floor coverings, water insulating barriers, etc. are some sources of the pollution. Therefore, a number of countries globally

(including Poland) have in place standards limiting the environment emissions by setting maximum acceptable VOC content in products.

The effects of VOC released from building materials, furniture, and other products on air quality in rooms for permanent human habitation are equally important. The quality of indoor air has a substantial influence on health and the mood of humans spending approximately 80% of their time indoors. Volatile organic compound emissions from building materials located indoors are a parameter determining the quality of air in rooms [5–7]. In the European Union, acceptable VOC concentrations in the indoor air are governed by applicable legislation [8–10], with test methods stipulated by standards.

The evaluation of products with regard to indoor air emissions of noxious substances consists in the testing of furnishing materials or finished products in testing chambers [11, 12]. Chambers of 0.05 m³ to 50 m³ featuring stable temperatures and relative humidity are utilised, commonly at room temperature (21–23°C)

and average humidity (45–55% RH). Figure 1 shows a schematic full measurement chamber installation, while Figure 2 contains photos of sample chambers of varying volumes.

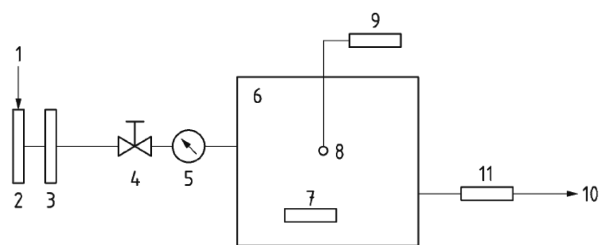


Fig. 1. Schematic chamber installation for testing VOC emissions from building materials [8]: 1 – air inlet, 2 – air filter, 3 – air conditioning system, 4 – air flow regulator, 5 – air flow meter, 6 – test chamber, 7 – fan including air speed meter, 8 – temperature and humidity sensor, 9 – temperature and humidity monitoring system, 10 – air outlet, 11 – sampling set

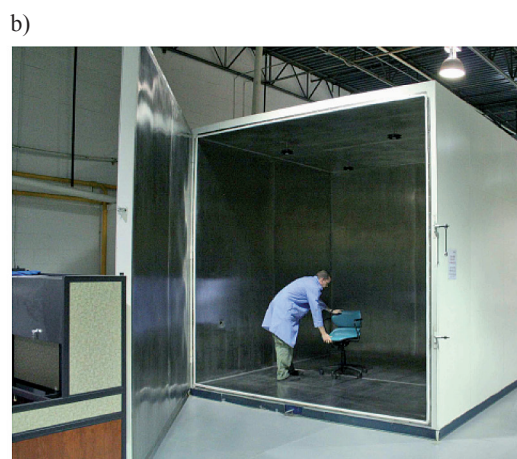


Fig. 2. VOC emission testing chamber: a) 0.053 m³, b) approx. 50 m³ [https://www.mastest.com/voc-testing]

For the purpose of assessing concentrations of particular chemicals, the air from a measurement chamber is driven to samplers, where the compounds are retained and concentrated. At the end of the chamber testing, VOCs are recovered from sorbents by means of thermal desorption or solvent extraction, for instance, and analysed using liquid or gas chromatography combined with mass spectrometry [13–15].

LUKASIEWICZ Research Network – Institute for Sustainable Technologies in Radom has for a dozen years produced sets for testing of VOC emissions from samples of building materials and wood-based products that include 0.1–1.0 m³ chambers [17].

The authors present the process of designing and improving the production of measurement sets that conform to standard requirements of PN-EN ISO 16000-9 [8] and allow for non-regulatory testing according to individual schedules of experimentation.

1. Regulatory requirements and resultant technical conditions of a measurement chamber

PN-EN ISO 16000-9:2009 defines physical parameters and air purity in a measurement chamber where emissions from the surface of a tested material samples take place. The standard requirements determine the design and production and operation technologies of the apparatus for the measurement procedures described there.

Air purity is defined as concentration of the total volatile organic compounds (TVOC) in the chamber background, which should be below $20 \mu\text{g}/\text{m}^3$, while the concentration of an individual target VOC should be lower than $2 \mu\text{g}/\text{m}^3$. The emission test chamber and sampling system elements in contact with emitted VOC must be made of polished stainless steel or glass. Other materials may be part of mixing or sealing elements, but they should be low-emission and low-adsorption, with no impact on the chamber's background concentration to be confirmed with testing.

This requirement considerably limits the range of design materials and technologies that can be used to build air preparation assemblies and the chambers proper. Teflon and fluorine rubber (Viton) are recommended, other than the materials listed in the standard. Plastics, glues, rubber, and sealing and polishing pastes are contraindicated design and auxiliary materials. This causes the following difficulties with construction of the apparatus:

- Metal tank and chamber parts must be welded from the inside (difficult access) in order to prevent gaps where hard to remove impurities may accumulate.
- Chemical etching of walls and discolorations is impossible, as hard to remove substances may deposit on metallic surfaces.
- Welding deformations interfere with effective and easy to maintain sealing of the chamber lid with a high-rigidity Viton seal.
- The structure cannot be sealed additionally with sealing pastes and elastomer seals, which are universally used in machine design.
- Easy access to all chamber internal surfaces must be provided for cleaning (assembled structures).
- Glass chambers are of limited applicability due to the properties of glues used to join glass.

Air hermetic sealing should protect a chamber against uncontrolled exchange of air with the environment. A chamber can be considered sealed if air loss is below 0.5% of its volume a minute at an overpressure of 1 000 Pa.

This involves application of effective chamber wall sealing, including its loading door, the axle of a fan triggering air flow around a sample, process penetrations, and measurement apparatus. Sliding seals of the fan axle and often dismantled wall sealing must be

made of Viton. This is a relatively hard sealing material, which requires high precision of the parts being sealed, and a high, evenly distributed pressure forces. A sliding friction couple in 'dry' operation is required for the fan axle. Stationary sensor seals can be made of Teflon or using standard Viton seals.

Air temperature at the time of testing should be $23 \pm 1^\circ\text{C}$. This requires the application of low-power heating elements in the test chamber or the air-conditioning of the entire laboratory room. An air temperature measurement and control system is required inside the chamber. The heating elements are best placed outside the chamber. Any heaters inside may interfere with test results as particular VOC components are introduced or degrade thermally.

Relative air humidity should be $50 \pm 3\%$, which requires an air humidification/ drying assembly featuring humidity control. Such an assembly should be part of a clean air preparation system and be made of glass or stainless steel cleaners connected with Teflon tubes. Air flow control valves should be of stainless steel.

Rate of air flow near the surface of a tested sample should be in the range 0.1 to 0.3 m/s. This requires a fan driven by an electric motor with a rotational speed control system to be placed inside the chamber. The rate of air flow depends on sample shape and placement in the chamber, among other things, which requires its measurement and regulation. The fan motor should be outside the chamber due to its materials (plastics, lacquers, bearing lubricants). The metal fan propeller should be mounted on a shaft that is rotationally packed in the chamber wall.

2. Problem formulation

The first emission testing chambers produced by the Institute for Sustainable Technologies were welded from stainless steel sheets or glued glass (Fig. 3). Precision and the pleasing appearance of the steel chambers were priorities. Weld joints were ground and polished and thermal discolorations etched with dedicated preparations. Regrettably, the cleaning of the chambers failed to prevent emissions from polishing pastes or other shining agents with electric and chemical action. The glass chambers were joined with glues used to make glass aquaria, construction structures, and in medicine. These technologies were satisfactory for formaldehyde emission testing, where background cleanliness requirements are less stringent.

In the case of high-precision testing, impurity emissions from internal chamber surfaces caused unacceptable background impurities. This is illustrated in a chromatogram (Fig. 4) where the purity of the air supplied to the steel chamber and the air containing VOCs emitted by its internal surfaces and equipment are compared.

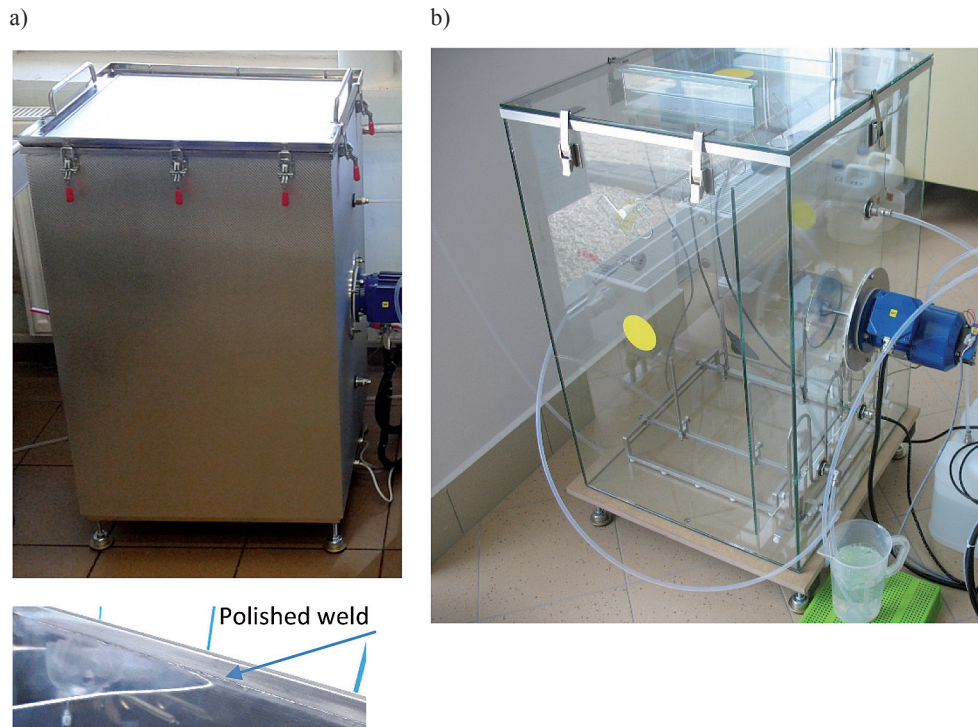


Fig. 3. Chambers featuring ‘enhanced air background inside the testing chamber’: a) steel chamber including polished welds, b) glued glass chamber

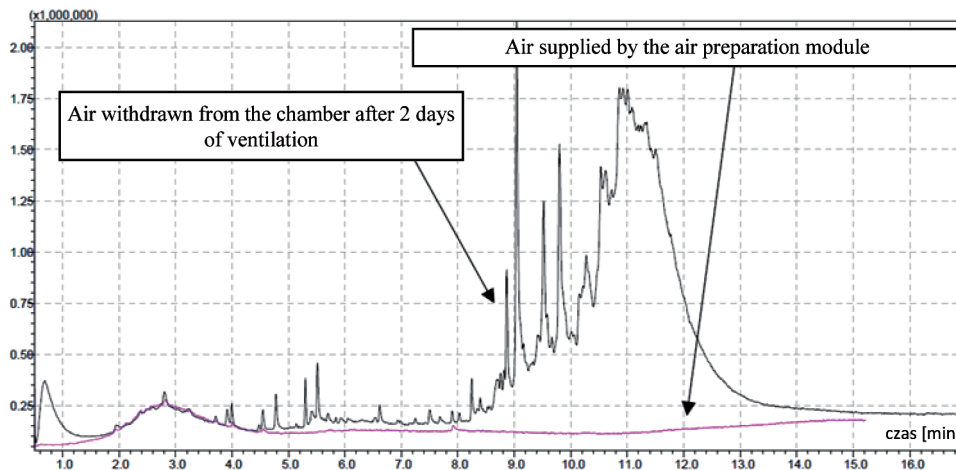


Fig. 4. Chromatogram generated for VOCs in the air supplied by the air preparation module and withdrawn from inside a steel emission chamber

The glass chambers display similar characteristics: the background is polluted with substances released from the glue joint. This is shown in the chromatogram (Fig. 5) of chemicals characteristic, inter alia, for glues contained in the air withdrawn from inside the chamber.

Wall rigidity is another problem with the steel chambers. A sheet fixed along its perimeter, over-rigid on welding, and thermally deformed, tends to bifurcate under the force of pressure. In effect, the chamber volume changes, depending on a temporary curvature (convexity or concavity) of a steel wall (Fig. 6).

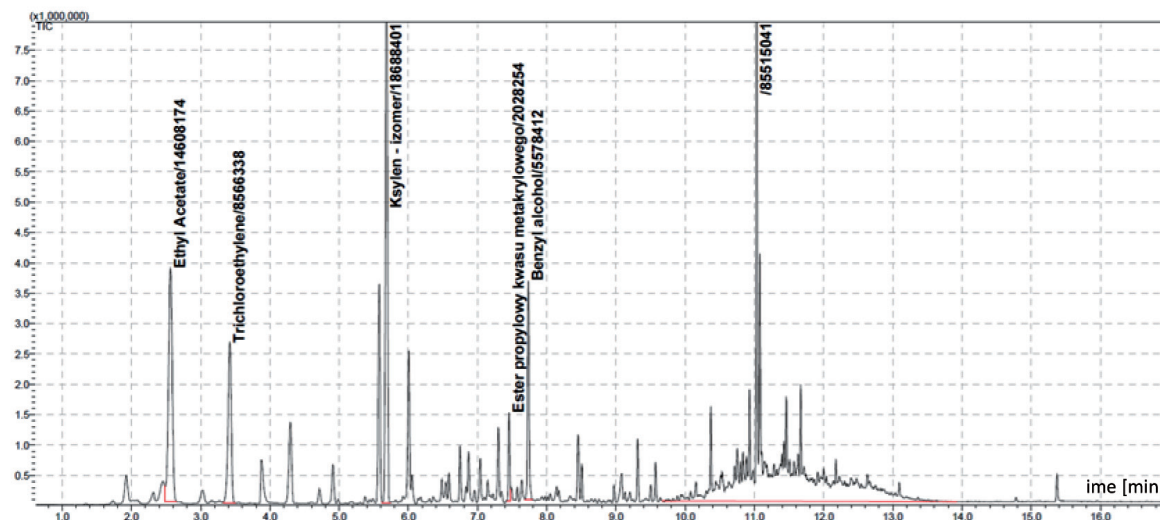


Fig. 5. Chromatogram generated for VOCs in the air withdrawn from inside a glass emission chamber

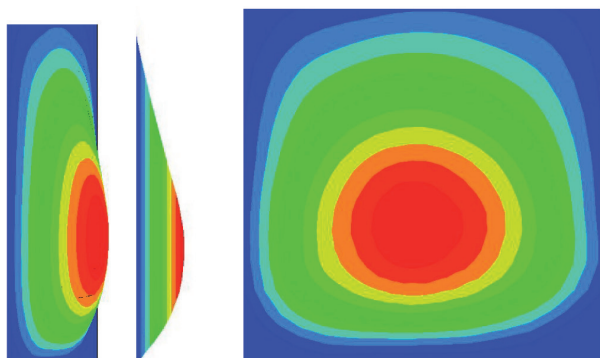


Fig. 6. 3D model of bifurcation of a testing chamber's side wall

3. Design of a testing chamber in conditions of high air purity

Based on the technical requirements and experience with the design and production of emission testing apparatus, a new, modernised 0.225 m³ chamber for the testing of volatile organic compound emissions was designed.

Figure 7 shows a 3D model of a complete chamber. The external sheath of the chamber body (Fig. 8b) made of thin steel sheets provides a pleasing appearance in harmony with laboratory equipment. The fan motor is placed inside a ventilated guard that provides for its cooling.

The sealed inner jacket of the chamber (Fig. 8a) is a welded structure including reinforcing ribs welded from the outside to individual points of the coating walls. The figure also shows the welded supports of transport handles, a connection flange for the fan driver, and a duct for an electric installation between electric heaters between the bottom of the chamber and the fan guard.

The outer jacket (Fig. 8b) matches the dimensions of the inner one by touching the rib surfaces and is pressed against them with clamps of the lid (9), mounted from the outside (Fig. 9), along with handles (11), the guard (8), and terminal masking shields (7).

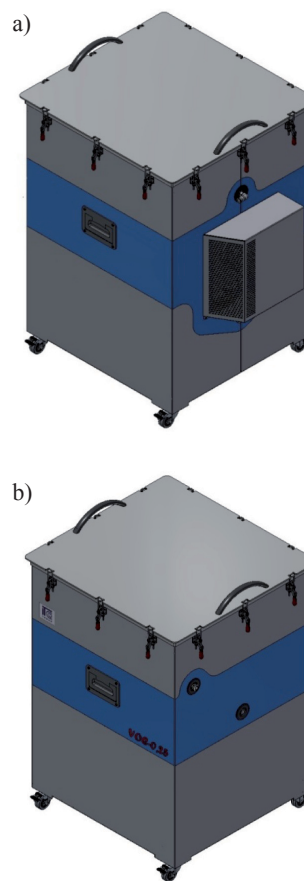


Fig. 7. 3D model of a complete 0.225 m³ chamber; a) view from the fan motor, b) view from the process terminals

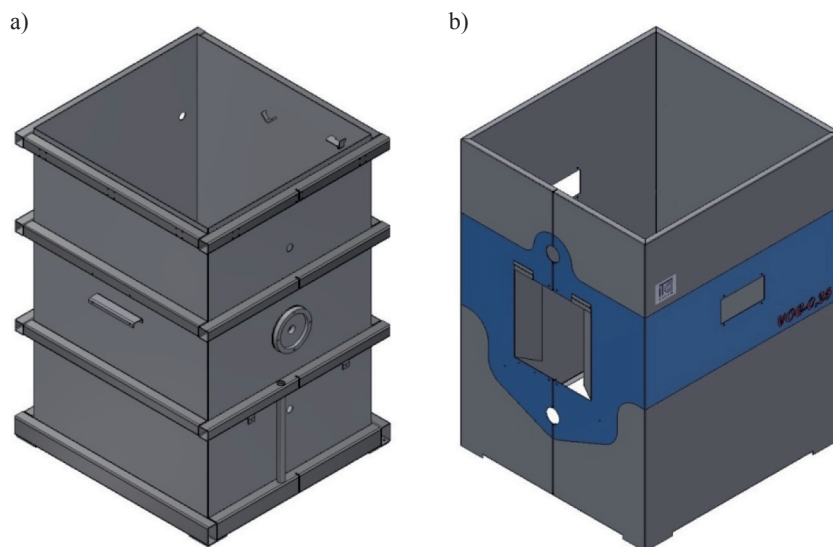


Fig. 8. 3D model of a complete 0.225 m³ chamber: a) the inner jacket, b) the outer jacket

Detailed design solutions of the modernised chamber are presented in a 3D model and a partial cross-section (Fig. 9).

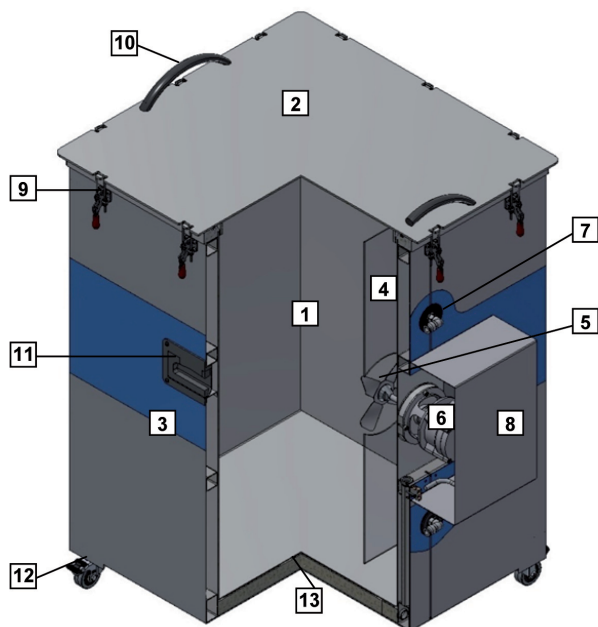


Fig. 9. 3D model of a complete 0.225 m³ chamber: 1 – inner jacket, 2 – lid, 3 – outer jacket, 4 – dismantled partition, 5 – fan airscrew, 6 – fan motor, 7 – process/measurement terminal, 8 – fan motor guard, 9 – lid clamp, 10 – lid grip, 11 – chamber transport handle, 12 – transport wheels, 13 – thermal insulation of heating elements

The sealed joining of the chamber jacket and the lid is shown in Figure 10. The easy lid mounting and sealing are made possible by the flat lid plate and a seal

in a groove in the massive steel flange of the chamber's inner jacket. The clamps (7) (Fig. 10) with an adjustable length of their shackle provide for precise control of the distribution of the seal pressure forces all around the lid circumference.

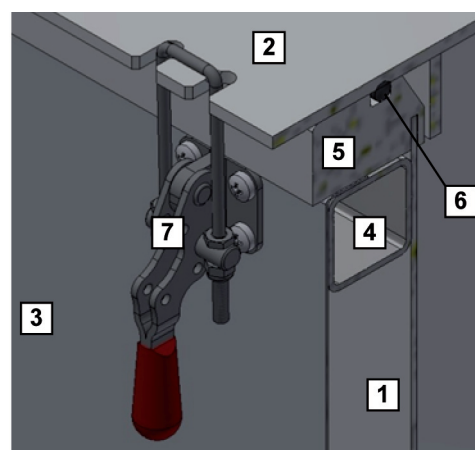


Fig. 10. 3D model of a chamber – the lid sealing: 1 – inner jacket, 2 – lid, 3 – outer jacket, 4 – inner jacket reinforcing rib, 5 – massive flange of the inner jacket, 6 – Viton “o-ring”, 7 – lid clamp

4. VOC emission measurement apparatus including a modernised chamber

The photographs (Fig. 11) show a full prototype apparatus for air sampling in the chamber for testing VOC emissions. Samples of test materials are placed on an openwork support frame inside a 0.225 m³ steel chamber. The internal air movement caused by a fan triggers the flow of required speed near a sample surface. The air is supplied to the chamber by a compressor

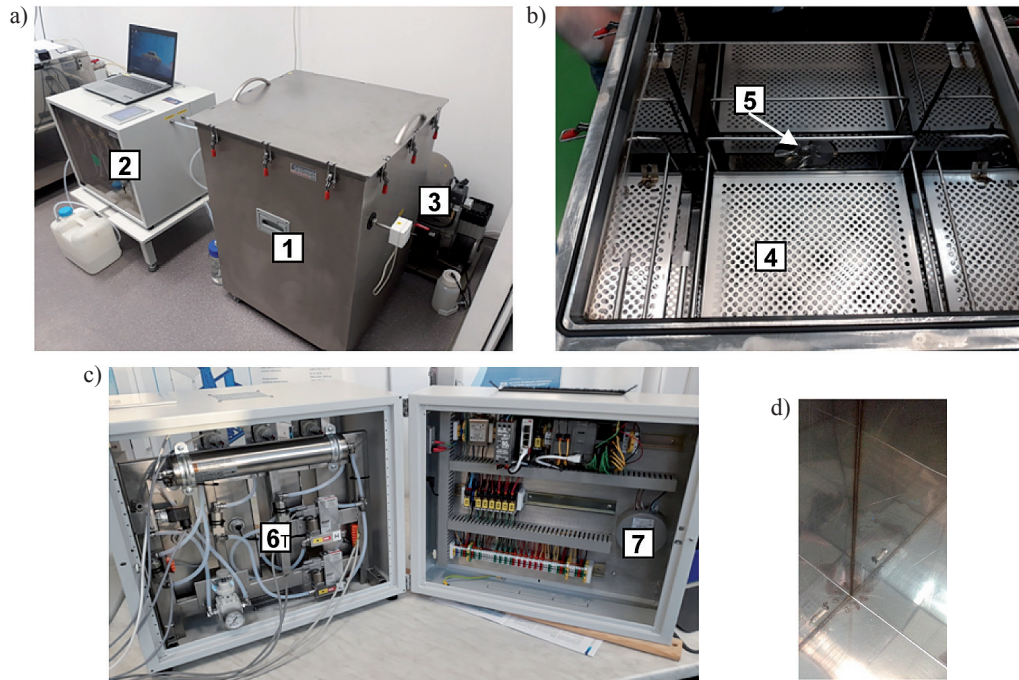


Fig. 11. VOC emission test stand including a 0,225 m³ chamber: a) complete testing set, b) chamber interior, c) design of the air preparation assembly, d) a weld inside the chamber – free from polishing treatment. 1 – emission chamber, 2 – air preparation assembly, 3 – compressor, 4 – support frame for samples, 5 – fan, 6 – filters, air and control apparatus, 7 – electronic control systems

pumping it through an air preparation assembly, where it is cleaned by active carbon filters. Air humidity is controlled as well. The apparatus responsible for supplying a specific stream of clean air of a required temperature and humidity is controlled by programmed electronic systems. The entire test that produces a sample containing absorbed VOC is controlled by a computer with dedicated operator software.

Chamber sealing tests confirmed the problems with generating overpressure had been fully resolved. The rigid, massive chamber flange made of a thick stainless plate facilitated the welding technology as well. The ribs reinforcing the chamber's inner jacket helped to stabilise its geometry, facilitating assembly and operation of the internal equipment.

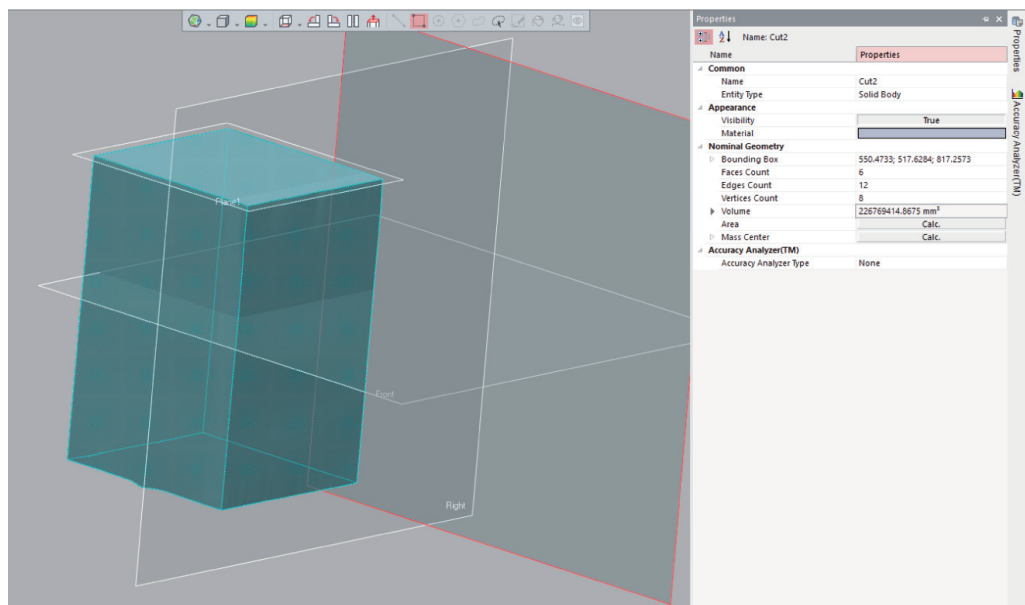


Fig. 12. Results of 3D scanning of 0.225 m³ chamber interior

Precision of the chamber's inner jacket was verified with a laser 3D scanner. The results are presented as a screen shot from the monitoring programme (Fig. 12). The flatness of the chamber walls and its volume were checked. The measurement results affirm the required dimension tolerances are preserved. The measured chamber volume is 0.2267 m³, thus, it is within the regulatory tolerance of $\pm 1\%$.

With a view to research and development work on new generations of structural coatings and plastics, a capacity for test programming in the chamber beyond the standard regulations is provided for. To this end, the chamber design and the control system of the apparatus allow for testing in the following variability ranges of the environment parameters:

- Temperature: the ambient temperature to 60°C
- Relative humidity: 10–90%

- Air pressure: 1000–40000 Pa
- Air flow intensity: 50–500 l/h

The environment parameters can be controlled with an accuracy of $\pm 3\%$ of the range.

5. Results of air cleanness testing in the modernised emission chamber

VOC concentration in the chamber's background was tested. The samples were aspiration withdrawn under constant pressure and mass flow using TD sorption tubes (Tenax TA). The concentrations assayed by means of gas chromatography coupled with mass spectrometry had the values listed in Table 1. Volatile organic compounds in the chamber background reached a total concentration below its statutory limit value, i.e. 20 $\mu\text{g}/\text{m}^3$ (Table 1).

Table 1. Concentrations of volatile organic compounds in the air withdrawn from the VOC emission testing chamber (the chamber background)

Identified compound	Tube concentration [μg]	Real concentration [$\mu\text{g}/\text{m}^3$]
Acetonitrile	0.0015	0.492
Cyclohexane	0.0014	0.450
Benzene	0.0026	0.858
Toluene	0.0018	0.596
Hexamethylcyclotrisiloxane	0.0019	0.642
Ethylbenzene	0.0021	0.692
p-Xylene	0.0052	1.717
m-Xylene	0.0015	0.496
Styrene	0.0008	0.270
Octamethylcyclotetrasiloxane	0.0012	0.400
Decane	0.0013	0.417
D-Limeone	0.0031	1.042
Benzenealdehyde	0.0066	2.192
Benzyl alcohol	0.0221	7.250
Nananal	0.0013	0.433
Decanal	0.0007	0.217
TOTAL VOC [$\mu\text{g}/\text{m}^3$]		18.262

Figure 13 contains chromatograms illustrating the results of the analysis of the reference material VOC Mix12 and of an air sample withdrawn from the emission chamber.

A certified reference material VOC-Mix 12 by Restek served the purposes of quantitative analysis. Concentrations of the remaining organic compounds were arrived at by means of the equivalent method for toluene.

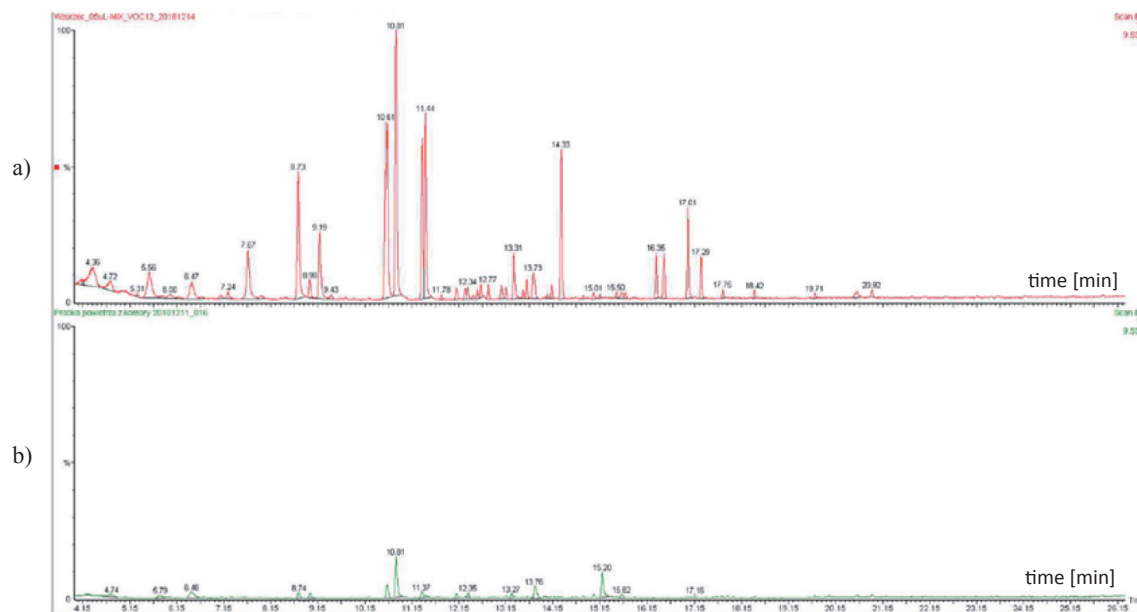


Fig. 13. Chromatogram presenting analysis results for: a) the reference solution VOC-Mix 12 from Restek, b) air sample from the test chamber

Conclusion

Regulatory requirements of conditions and procedures of measuring volatile organic compound emissions from wood-based and construction materials and other products, as well as design and technological solutions applied in the apparatus used for such measurements were analysed following the work described in this paper.

A virtual model of a modernised 0.225 m³ emission measurement chamber, and the design and engineering documentation were developed. A complete prototype of a modified chamber was built and subject to verification testing.

As part of the design modifications by changing the fixing conditions, the rigidity of the chamber walls was enhanced, which prevents bifurcation and uncontrolled changes of test volumes. The change to the production technology implies resignation from weld polishing.

The testing has reaffirmed the effectiveness of the solutions and the fulfilment of all regulatory and user requirements with regard to the apparatus and VOC emission measurement procedures.

The design and technological solutions, as part of the action described above, will be applied to the production of the apparatus, and the design and production of equipment with comparable requirements and purposes, e.g., for the construction of emission chambers with other volumes or chambers for precise physical and chemical analysis.

The solution features a programmable variability range of environment parameters and, as such, can also be applied to non-standard research. VOC emissions may be tested in simulated environment conditions close to real operating conditions that may vary in respect of temperature, pressure, humidity, and air flows.

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