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RESISTANCE OF BARRIER MATERIALS AGAINST TOXIC COMPOUNDS PERMEATION AND ITS EVALUATION IN ACCORDANCE WITH NEW EUROPEAN NORMS

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***Abstract**: Study of barrier material resistance against permeation of toxic compounds belongs to a category of chemical warfare agents and toxic industrial chemicals are permanently insufficiently solved problems. This work deals with changes in approaches to testing barrier materials against toxic compounds permeation in static and dynamic conditions. An aim of this information is to describe specifications of cancelled norms of ČSN EN ISO 6529 (2002) a ČSN EN 374-3 (2004) with newly established norms ČSN EN 16523-1 a 16523-2 (2015). Authors focused on specification and comments of changes in such parts of norms concerning their scope, terms and definitions. A selected problem was solved with the help of analyse of newly established norms in a form of comments. Conditions for specification of understanding of mentioned problems in a context of established or occasionally prepared methods of testing resistance barrier materials against toxic compounds permeation have been formed with elaborated research.*

Keywords: Protective clothing, barrier material, toxic compound, permeation, norm

1. Introduction

The restricted amount of deployable forces composed of highly specialized units of the Czech Armed Forces Chemical Corps can be limiting within the fulfilment of operational tasks connected with problems concerning the liquidation of consequences of either the employment of Weapons of Mass Destruction or the leakage of Toxic Industrial Materials. The quality of used individual protective equipment whose testing should be a challenge for responsible institution can significantly affect the quality of operational tasks fulfilment. The purpose of chemical protective clothing and equipment is to shield or isolate individuals from the chemical, physical, and biological hazards that may be encountered during hazardous materials operations within military operations [1].

The protective properties are the most important parameters for selection of well-suited materials for construction of individual protective equipment. These properties determine resistance ability of given individual protective equipment against the highly toxic agents and industrial harmful substances. Procedures according to CSN EN ISO 6529 are developed within the Nuclear, Biological and Chemical Defence Institute of the University of Defence for this purpose. These procedures respect regulations of the

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American norm F739 [2] from a category of ASTM (American Society for Testing and Materials) that solve mentioned problem on an army level [3,4].

2. Norm ČSN EN ISO 6529

Norm of ČSN EN ISO 6529 (83 2732) Protective clothing - Protection against chemicals - Determination of resistance of protective clothing materials to permeation by liquids and gases was cancelled to the date of the 1st of May 2016 with the Bulletin of the Czech Office for Standards, Metrology And Testing number 7, 2016 [5]. No subsequent norm that would replace this norm is mentioned in this bulletin. Although updated version from 2013 [6] exists it is possible to legitimately assume that it has not been established within the Czech Republic environment. This norm superseded the norm ČSN EN 369 (83 2732) from February 1995 [7]. With comparison of the version of this norm from 2013 has been found up that it works with the same terms (definitions) and, moreover, authors legitimately assume that some its provisions have been handed over from ČSN EN 16523-1 a 16523-2 norms. It is thus a question which norm is currently usable and applicable as a compensation of cancelled ČSN EN ISO 6529 (2002) in the Czech Republic environment. Professional public agreed that for compensation should be used just ČSN EN 16523-1 a 16523-2.

This norm in its introduction determines that workers involved in the production, use, transportation, and emergency response with liquid and gaseous chemicals (and also soldiers designated to chemical consequence management disasters – authors´ note) can be exposed to numerous compounds capable of causing harm upon contact with the human body. The deleterious effects of these chemicals can range from acute trauma such as skin irritation and burn to chronic degenerative disease, such as cancer. Since engineering controls may not eliminate all possible exposures, attention is often placed on reducing the potential for direct skin contact through the use of protective clothing that resists permeation, penetration and degradation.

Furthermore, it is introduced that these test methods are normally used to evaluate the barrier effectiveness of materials used for protective clothing and specimens from finished items of protective clothing against permeation of either liquid or gaseous chemicals. Under the term of "Finished items of protective clothing" must be included gloves, arm shields, aprons, suits, hoods, boots, etc. The phrase "specimens from finished items" encompasses seamed and other discontinuous regions as well as the usual continuous regions of protective clothing items. Options are provided for conducting this testing under both conditions of continuous or intermittent contact with the chemicals.

It is specified, that these test methods provide various options for reporting test results in terms of breakthrough time, permeation rate and cumulative permeation to allow a comparison of protective clothing material permeation resistance. These parameters are key measures of the effectiveness of a clothing material to act as a barrier to the test chemical. Such information is used in the comparison of clothing materials during the process of selecting clothing for protection from hazardous chemicals. Long breakthrough detection times and normalized breakthrough detection times as well as low permeation rates are characteristic of the best barriers.

It is recommended, that resistance to penetration by liquid chemicals should be determined by using ISO 6530 Protective clothing - Protection against liquid chemicals - Test method for resistance of materials to penetration by liquids while resistance to penetration by liquid chemicals under pressure should be determined by using ISO 13994 Clothing for protection against liquid chemicals - Determination of the resistance of

protective clothing materials to penetration by liquids under pressure.

Regarding personnel requirements, it is in the introduction that it has been assumed in the drafting of this International Standard that the execution of its provisions is entrusted to appropriately qualified and experienced people for whose guidance it has been prepared and that appropriate precautions will be taken to avoid injury to health and contamination of the environment.

Based of literature background research performed within 2013 to 2017 at present, no quantitative information exists about acceptable levels of dermal contact. Therefore, the data obtained using this test method cannot be used to infer safe exposure levels.

This International Standard described laboratory test methods that enabled a determination of the resistance of materials used in protective clothing to permeation by liquid or gaseous chemicals under the conditions of either continuous or intermittent contact. Methods were divided as follow:

- method A was applicable to the testing of liquid chemicals, either volatile or soluble in water, expected to be in continuous contact with the protective clothing material;
- method B was applicable to the testing of gaseous chemicals expected to be in continuous contact with the protective clothing material;
- method C was applicable to the testing of liquid chemicals, either volatile or soluble in water, expected to be in intermittent contact with the protective clothing material.

It is very important to stress, that these test methods were only suitable for the testing of air-impermeable protective clothing materials. They assess the permeation resistance of the protective clothing material under laboratory conditions in terms of breakthrough time, permeation rate, and cumulative permeation. These test methods also enabled observations to be made of the effects of the test liquid on the protective clothing material under test. These test methods addresses only the performance of materials or certain material constructions (e.g. seams) used in protective clothing and did not address the design, overall construction and components, or interfaces of garments or other factors which may affect the overall protection offered by the protective clothing.

Moreover, it is necessary to emphasize that these tests did not necessarily simulate conditions to which clothing materials are likely to be exposed in practice. The use of test data should therefore be restricted to broad comparative assessment of such material according to their permeation-resistance characteristics.

3. ČSN EN 16523-1 a 16523-2 (2015)

Norms ČSN EN 16523-1 Determination of material resistance to permeation by chemicals - Part 1: Permeation by liquid chemical under conditions of continuous contact and 16523-2 Determination of protective material resistance to permeation by chemicals - Part 2: permeation by gaseous chemical under conditions of continuous contact are introduces with an attribute: “Part or the whole norm is in English”. Based on the text: “The ČSN EN 374-3 (83 2310) from May 2004 is replaced with this norm” it is possible to assume that a connection between the norm of ČSN EN ISO 6529 is not prime but only secondary. Both mentioned norms are listed among European norms approved for direct application as ČSN in Bulletin [8].

3.1 ČSN EN 16523-1

In the introduction of the norm there is mentioned that the test method described in the first part of ČSN EN 16523 (thus ČSN EN 16523-1) is intended to be used to evaluate the barrier effectiveness of materials used for protective clothing, gloves and footwear materials against permeation by liquid chemicals.

Furthermore, it is specified, that this

method does not assess the chemical degradation or penetration of the material. Resistance to penetration by liquid chemicals can be determined by using for example ISO 6530 Protective clothing - Protection against liquid chemicals - Test method for resistance of materials to penetration by liquids while resistance to penetration by liquid chemicals under pressure can be determined by using for example ISO 13994 Clothing for protection against liquid chemicals - Determination of the resistance of protective clothing materials to penetration by liquids under pressure. Resistance to chemical degradation can be determined by EN 374-4 Protective gloves against chemicals and micro-organisms. Determination of resistance to degradation by chemicals for gloves and EN 13832-1:2006 Footwear protecting against chemicals - Part 1: Terminology and test methods, in the part 4.2 for footwear.

As significant we consider that this method provides tests results in terms of breakthrough time. This parameter is a key measure of the effectiveness of a material to act as a barrier to the challenge chemical. Such information is used in the comparison of the performances of personal protective equipment materials during the process of selecting personal protective equipment for protection from hazardous chemicals. Long breakthrough times are characteristic of high permeation resistance. Breakthrough time does not provide a correlation between protection and the toxicity of the chemicals tested, only cumulative permeation can provide this information.

It has been assumed in the drafting of the first part of EN 16523 that the execution of its provisions will be entrusted to appropriately qualified and experienced people with a sound understanding of analytical chemistry. Appropriate precautions should be taken when carrying out this type of testing in order to avoid injury to health and contamination of the environment.

In the part of “Scope” it is mentioned that this European Standard specifies a test method for the determination of the resistance of protective clothing, gloves and footwear materials to permeation by potential hazardous liquid chemicals under the condition of continuous contact. This test method is applicable to the assessment of protection against liquid chemicals that can be collected only by liquid or gaseous collecting media. This test method is not adapted for the assessment of chemical mixtures, except for aqueous solutions.

3.2 ČSN EN 16523-2

This European Standard is used in conjunction with EN 16523-1. A future part of EN 16523 will explain the use of the series of standards EN 16523.This standard includes only the specific aspects linked with the testing with gaseous challenge chemicals.

This European Standard specifies a test method for the determination of the resistance of protective clothing, gloves and footwear materials to permeation by potentially hazardous gaseous chemicals under the condition of continuous contact. This test method is applicable to the assessment of protection against gaseous chemicals that can be collected only by liquid or gaseous collecting media. This test method is not adapted for the assessment of gaseous chemical mixtures. This test method describes the modifications to EN 16523-1 necessary to test against gaseous chemicals that can be collected by liquid or gaseous collecting media.

4. Comparison of norms ČSN EN ISO 6529 a ČSN EN 16523

4.1 Introduction and scope

Based on above mentioned facts it is possible to claim that norms from the ČSN EN 16523 category are usable for testing resistivity of barrier materials against the permeation of toxic compounds. A scope of their usage is, however, little bit narrower mainly from the view of a number of parameters that are for evaluation utilized.

Norms of ČSN EN 16523-1 and 16523-2 establish the only one parameter which is the breakthrough time while ČSN EN 6529 operates with terms as the breakthrough detection time, permeation ration and cumulative permeation. For support of this statement it is possible to introduce that norms ČSN EN 16523-1 a 16523-2 use this information to comparison of the performances of personal protective equipment materials during the process of selecting personal protective equipment for protection from hazardous chemicals. In case of ČSN EN ISO 6529 we can meet the statement that this information is used for comparison of materials during the process of selecting clothing for protection from hazardous chemicals. Long breakthrough detection times and normalized breakthrough detection times as well as low permeation rates are characteristic of the best barriers. This statement can be also interpreted in the way that new norms are more focused on determination of barrier material resistance against permeation of chemical compounds in static conditions and in contrary the cancelled one on dynamic conditions. In fact, however, typical terms for static and dynamic conditions are used in both norms. In light of above mentioned comparison it can be deduced that an information base for evaluation of achieved data will be lower in case of new norms.

As relative formal can be assumed the statement that norm ČSN EN ISO 6529 enabled testing samples of barrier materials used for protective clothing and simultaneously even samples from finished parts of protective clothing designated for protection against permeation of liquid and gaseous chemicals. In the norm of ČSN EN 16523-1 is mentioned that the test method is designated to be used to evaluate the barrier effectiveness of materials used for protective clothing, gloves and footwear materials against permeation by liquid chemicals. It is thus the question whether this statement enables testing already established barrier materials of protective clothing.

The difference can be also found in the part concerning requirements on personnel. Whereas in the norm ČSN EN ISO 6529 is determined an obligation of performance of the measurement by properly qualified and experienced persons so in the norm ČSN EN 16523-1 is the same obligation determined to qualified and experienced persons that thoroughly understand analytical chemistry. The rate of thoroughness is not, however, determinate neither demanded education not the length of practice.

The norm ČSN EN ISO 6529 also specified an availability of usage of the method on the subject of air-impermeable protective clothing materials. They assess the permeation resistance of the protective clothing material under laboratory conditions. The norm of ČSN EN 16523-1 mentions no such as restriction. In practice it can mean its usage even for measurement of permeable barrier materials in different scope of temperature. Absence of this restriction can be a benefit to superior specification of barrier materials usability in extreme conditions in that their users can operate.

Very important regulation was introduced in the ČSN EN ISO 6529 norm that these tests did not necessarily simulate conditions to which clothing materials are likely to be exposed in practice. The use of test data should therefore be restricted to broad comparative assessment of such material according to their permeation-resistance characteristics. This regulation miss in the ČSN EN 16523-1 norm and thus it is possible to lead a discussion concerning the problem how to do a particular test and mainly to interpret achieved results.

4.2Terms and definitions

It is possible to discover some differences and thus changes even in terminology those bounds to subjected problems. Changes in definitions, terms and their meaning are summarized in table 1. …”

Table 1: Comparison of terms and definitions between norms ČSN EN ISO 6529 and ČSN EN 16523

CSN EN ISO 6529 („old norm“)	**CSN EN 16523 („new norm“)**	**Note – difference**
Analytical technique	Analytical technique	Note 1.
Breakthrough detection time	-	
Closed-loop	Closed loop	Tiny different in English grammar. Note 2.
Collection medium	Collecting medium	Tiny different in English grammar. Note 3.
Contact time	-	
Cumulative permeation mass	-	
Cycle time	-	
Degradation	-	
-	Limit of quantification	Note 4.
-	Liquid challenge chemical Gaseous challenge chemical	The second term is mentioned as the only one in the norm ČSN EN 16523-2. Note 5.
Minimum detectable mass permeated	-	
Minimum detectable permeation rate	Minimum detectable permeation rate	Note 6.
Normalization permeation mass	-	
Normalization permeation rate	Normalized permeation rate	Tiny different in English grammar. Note 7.
Normalized breakthrough detection time (open-loop)	-	
Normalized breakthrough detection time (closed-loop)	Normalized breakthrough time	The same meaning as the term: Normalized breakthrough detection time (closed-loop) Note 8.
Open-loop	Open loop	Tiny different in English grammar. Note 9.
Penetration	-	
Permeation	Permeation	Note 10.
Permeation mass		
-	Response time	Note 11.
Permeation rate	Permeation rate	Note 12.
Protective clothing material	-	
Purge time	-	
Steady-state permeation rate	Steady-state permeation rate	
Test chemical	-	Synonym to terms liquid challenge chemical(ČSN EN 16523-1) and gaseous challenge chemical(ČSN EN 16523-2)

Notes:

1: In an old norm there is mentioned: “Procedure whereby the concentration of a chemical in a collection medium is quantitatively determined” while in a new one: “Method of identifying and quantifying the amount of permeated chemical in the collection medium.”

There is evident that the new norm also focuses on identification of the chemical which in term of the test chemical is mostly known so this regulation slightly loses its sense. It is, however, necessary to add that within permeation of chemical compounds mixtures it can be interesting to deal with that problem. In the old norm there is mentioned “gas and liquid chromatography” and vice versa in the new one “mass spectrometry”. Other analytical techniques are the same. Methods gas and liquid chromatography are introduced in a supplement of the definition and it is mentioned: “Although liquid and/or gas chromatography are separation techniques rather than detection methods they can be used in conjunction with suitable detectors to quantify the amount of permeated chemical in the collection medium.”

2: While the old norm defines this term that: “Refers to a testing mode in which the collection medium is fixed.” so the new one defines it: “System in which is collecting medium is re-circulated or stirred through the sampling compartments of the test cell.” Further, the note differs. While in the old norm there is: “The collection medium volume may change slightly from sampling without replacement of the sampled collection medium.” so in the new one is written: “Closed loop systems are not commonly used with gaseous collection media.”

3: Terms are in detail defined in other parts of both norms. In their expression a supplementary comment practically does not differ. Nonetheless, a single term is in the old norm defined as: “Liquid or gas that does not affect the measured permeation and in which the test chemical is freely soluble or adsorb to a saturated concentration greater than 0.5 % by mass or by volume.” In the new norm there is little bit simpler definition: It is mentioned: “Liquid or gas on the "inner" clean side of the test sample in which any permeated chemical is collected.“

4: It deals with the new term. It is defined as: “Minimum quantity of a substance which can be measured.” In the note there is further mentioned: “It is the value where the uncertainty of measurement is equal to 50 % of the determined value.”

5: It deals with the new and old term, thus a synonym to the term of test chemical. It is defined as: “Liquid chemical that is used to challenge the protective clothing, gloves and footwear material specimen.” In the part of norm ČSN EN 16523-2 is defined the term that completes its meaning concerning gaseous chemical compound.

6: The term is defined in both norms in the same way. The difference is, however, in the texts of notes. In the old norm it is specified with one note as amended: “This value is not necessarily the intrinsic limit of detection for the analytical instrument.” In the now norm there are three notes therewith the first one is the same. The two others are introduces subsequently: “Minimum detectable permeation rate is usually based upon 3 times the average background noise.” and “useful information can be found on the following websites:http://www.measurementuncertainty.org/guide/app_f.thml and http://iupac.org/publications/pac/1997/pdf/6902x0297.pdf“.

7: Terms do not differ only grammatically but also significantly. In the old norm it is defined as: “Permeation rate used for determining the normalized breakthrough detection time in an open-loop permeation test.” and accompanied by comments as amended by:“This method provides two choices of normalized permeation rates: 0.1 $\mu g.cm^{-2}.min^{-1}$ nebo 1 $\mu g.cm^{-2}.min^{-1}$.“

New norm is slightly shorter and introduces only: “Arbitrary fixed figure of 1 μg.cm^{-2}.min^{-1}“. Furthermore, it is accompanied with two notes, thus: “This optimized figure is a compromise between test method parameters (mixing, flow rate, detection limits, etc.) and the specific analysis possibilities at the present time, to ensure a better repeatability and reproducibility of the test.” and “In some standards the normalized permeation rate is fixed in lower concentration (for example 0,1 μg.cm^{-2}.min^{-1}). However, the parameters of the others standards, flow rate, mixing are often less severe than the present test method. The tests are only design to evaluate protective clothing, gloves and footwear.” Despite the fact that finally both data concerning normalized permeation rate are established so the ability of detection in lower values of normalized permeation rate does not have to be the result of flow rate, mixing but mainly sensitivity of the detection system.

8. Definition of introduced term in the new norm exactly corresponds with the definition of normalized breakthrough detection time in relation to systems working on the open loop base which is the part of the old norm. The term that would be an equivalent for systems working on the open loop base is not established in the new norm.

9: While the old norm defines this term that: “Testing mode in which fresh collection medium flows continuously through the collection chamber of the test cell and is not reused or recycled.” so the new one defines it as: “System in which the collection medium passes through the sampling compartment of the test cell without recirculation.” Further, the note there is in the new norm: “Closed loop systems are not commonly used with gaseous collection media.”

10: Definitions are the same in both norms. Within the description of the 1st phase of the permeation process is newly used the term “absorption” instead of “sorption”. Authors assume that the certain rate of generality would be kept whereas the resistance against permeation of chemical compounds is possible to also study in the scope of permeable (porous) barrier materials when primarily mechanisms of adsorption occur and not only in the scope of isolative (non-porous, impermeable) ones.

11: The new term deals with. It is defined as: “The time between the actual arrival of the challenge chemical on the collection side of the specimen and the time when analytical instrumentation responds to it.” This term can have its invaluable meaning just within methods working in dynamic conditions and using analytical techniques for self evaluation of resistivity against permeation of the chemical compound.

13: Definitions are quite the same each other. In the old norm there is introduced that permeation rate is: “Quantity of test chemical that passes through the protective clothing material for a given exposed surface are per unit time.” In new norm these is introduced that permeation rate is: “Mass of challenge chemical permeating unit area of the protective clothing, gloves and footwear per unit time.” Even here stressing of “exposed” seems to be more suitable considering the fact that permeation is a physical process that runs practically anytime and under any conditions and through all types of materials.

Some of the differences and in addition discrepancies can also be traced in the images to clearly explain the terms used.

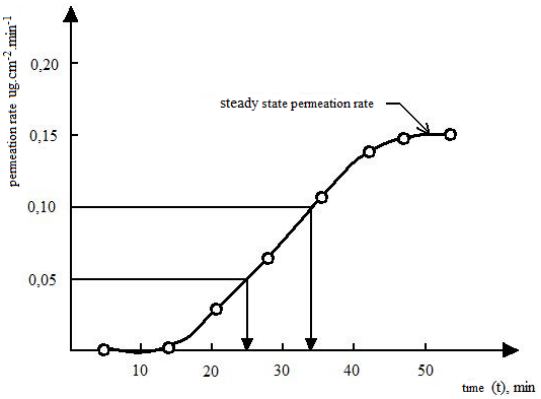
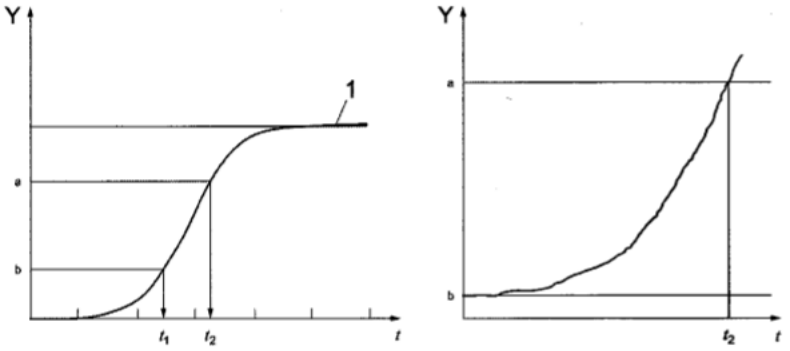


Figure 1: Breakthrough detection time [9]

By the picture 1 that that is illustrated in the old norm is introduced that the breakthrough detection time for a method sensitivity of 0.05 $\mu g.cm^{-2}.min^{-1}$ is 23 min but would be reported at 20 min, which corresponds to the last sampling time preceding the test. The normalized breakthrough detection time at a normalization permeation rate of 0.1 $\mu g.cm^{-2}.min^{-1}$is 33 min, but similarly would be reported at 28 min, which corresponds to the preceding sampling time. The steady-state permeation rate is approximately 0.15 $\mu g.cm^{-2}.min^{-1}$. .



where there is:

Y permeation rate [$\mu g.cm^{-2}.min^{-1}$]
t time
1 steady-state permeation
a normalized permeation rate
b minimum detectable permeation rate
t_2 normalized breakthrough time

Figure 2: Schematic permeation graph showing actual and normalized breakthrough times

It is interesting that already in the name of the picture the term of actual breakthrough time reveals. This term is not defined in the new norm at all. Also the term steady-state permeation is not defined in the new norm. It is possible to assume that the term steady-state permeation rate should be established.

5. ČSN EN 374-3 (2003)

Norm ČSN EN 374-3 Protective gloves against chemicals and micro-organisms - Part 3: Determination of resistance to permeation by chemicals, as it is visible, is focused only on protective aids of the protective gloves type. Norms of ČSN EN 16523-1 and 16523-2 are, however, outspread to determination of resistance against the permeation of chemical compounds not only for gloves bat also for protective clothing and footwear. In the

subject of the norm there is introduced that: "This norm sets designation of resistivity of materials of protective gloves against permeation of potentially dangerous chemicals that are not in a gas form under conditions of a permanent contact."

6. Conclusions

Based on analysis of the cancelled ČSN EN ISO 6529 norm and newly established but non-harmonized standards ČSN EN 16523-1 a ČSN EN 16523-2 can state their general usefulness for the evaluation of barrier properties of construction materials used to manufacture chemical protective clothing. By comparing were, however, identified some fundamental flaws that can have a practical impact on the implementation of results of some previously used and established methods. Absence of terminology relating to the testing of barrier materials with respect to resistance to permeation of chemicals in static conditions should force for reflection and for a broader discussion of this problem, not only at a national but also an international level. Moreover, it is necessary to lead a discussion concerning problems whether the continuity between civilian and army thinking was not disturbed.

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