Journal of the Serbian Chemical Society
JSCS@tmf.bg.ac.rs • www.shd.org.rs/JSCS
J. Serb. Chem. Soc. *84* (1) 55–68 (2019)
JSCS–5164
UDC 691.2/.5+504.054–039.7+547.562:519.245
Original scientific paper

Validation and uncertainty estimation of an analytical method for the determination of phenolic compounds in concrete

BRANISLAVA G. SAVIĆ[1]*, IVANA J. MIHAJLOVIĆ[2], SLOBODAN M. MILUTINOVIĆ[1], MINA M. SEOVIĆ[1], ŽELJKA M. NIKOLIĆ[1], MILOŠ S. TOŠIĆ[1] and TANJA P. BRDARIĆ[1]

[1]*Vinča Institute of Nuclear Sciences, University of Belgrade, Department of Physical Chemistry, Mike Petrovića Alasa 12–14, 11351 Vinča, Belgrade, Serbia and* [2]*University of Novi Sad, Faculty of Technical Sciences, Department of Environmental Engineering, Trg Dositeja Obradovića 6, 21000 Novi Sad, Serbia*

(Received 18 May, Revised 3 December, accepted 7 December 2018)

Abstract: Organic contaminants from building materials negatively affect the health of people. This study presents an analytical method for the simultaneous identification and quantification of 9 phenolic compounds, *i.e.*, phenol, 2-chlorophenol, 2,4-dimethylphenol, 2,4-dichlorophenol, 2,6-dichlorophenol, 4-chloro-3-methylphenol, 2,4,6-trichlorophenol, 2,3,4,6-tetrahlorophenol and pentachlorophenol, in concrete by a gas chromatographic method with mass spectrometric detection (GC–MS). By comparing the MS spectra of the test compounds with MS spectra of analytical standards, reliable identification was achieved. The method could be applied in a given range (from 0.01 to 7.5 mg kg^{-1}) with appropriate parameters of precision, accuracy, repeatability and linearity. The developed method could be used for quality control testing of phenols in concrete during the construction of new buildings, old residences and construction waste. The measurement uncertainty of the phenolic compounds in concrete was evaluated using two approaches, *i.e.*, GUM recommendations and a Monte Carlo method. Disagreement of those methods was observed. The Monte Carlo method could be used in the evaluation of combined measurement uncertainty for the determination of phenolic compounds in concrete.

Keywords: building material; GUM; Monte Carlo.

INTRODUCTION

Phenol and phenol derivates represent a very important group of pollutants that are ubiquitous in the environment. Due to the exceptional toxicity of most phenolic compounds with bioaccumulation effect in animals and plants, and carcinogenicity to human health, the US Environmental Protection Agency - USEPA has classified phenolic compounds as a group of hazardous pollutants.[1] Similarly,

---

*Corresponding author E-mail: branislava@vin.bg.ac.rs

https://doi.org/10.2298/JSC180518106S

the European Union Directives 80/778/EC[2] and 93/72/EEC Annex I classified phenols as Xn, R20/21/22 (harmful by inhalation, contact with skin, and if swallowed) and N, R51/53 (toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment).[3] Phenolic compounds are present in the air, water and soil as a result of natural and anthropogenic activities (plastic, pharmaceutical, petrochemical, and explosive industry and agricultural activities).[4,5] In addition, phenolic derivates are found in petroleum products, such as coal tar and creosote, and could be released by wood combustion and auto exhaust gases.[6]

Building materials such as insulation, floor and wall coverings, wood preservatives (*i.e.*, biocides), disinfectants and cigarette smoke are potential sources of phenol in residential area indoor air.[7] Although pollution of indoor air with phenols originates from building materials used for construction building, there is no EU legislation relating to the quality of building materials to date. To the best of our knowledge there is a proposal for lowest concentrations of interest for dangerous substances emitted from building materials into the indoor air by EU-LCI Working Group.[8] Concrete as a building material is composed of water, aggregate (rock, crushed stone, sand or gravel), a binder or paste such as cement and may contain additives.[9] As such, it presents a complex matrix for the analysis of the potential presence of organic pollutants. In previous research, a new ultra performance liquid chromatography-photodiode array (UPLC-PDA) method for the analysis of polycyclic aromatic hydrocarbons in concrete was validated.[10] Many analytical approaches have been used for trace-level analysis of phenols in water, wastewater and soil using instrumental techniques such as gas chromatography-electron-capture detection (GC-ECD),[11] liquid chromatography with ultraviolet detection[12] and high-performance liquid chromatography (HPLC).[13–15]

Although the use of the GC–MS technique for analysis of phenolic compounds in soil and water has already been reported,[16–19] to the best of our knowledge, a method for the determination of phenolic compounds in concrete as construction material has not hitherto been developed.

In this work, a new analytical method for the determination of phenolic compounds from concrete samples is presented. The main validation parameters of developed method, such as linearity, limit of detection and quantification, repeatability and trueness, were determined. This method could be useful for monitoring phenolic compounds during the construction of new buildings, and related buildings, known as sick-building syndrome considering that the inhalation of phenols may be dangerous for human health, causing systemic damage to the nervous system.[20]

The second aim of the present study was a critical assessment of two approaches for the estimation of combined measurement uncertainty. Both the Guide to Expression of Uncertainty in Measurement (GUM) recommendations[21] and the Monte Carlo method[22] were used for estimating the uncertainty associated with the analysis of phenolic compounds in concrete. Each of these methods has its adventages in different areas: chemistry,[23,24] pharmacy,[25] geodesy,[26] spectrometry,[27] biomedical science,[28] *etc.*

EXPERIMENTAL

Reagents and materials

All chemical reagents were of analytical grade or better. Methanol (99 %, analytical grade) was purchased from Macron Fine Chemicals (USA). Potassium carbonate (pro analysis) was purchased from Lachner Industrial Company. Cyclohexane (HPLC grade) was purchased from Fisher Chemicals. The following chemicals: hydrochloric acid, acetic anhydride, sodium hydroxide, sodium sulphate anhydrous were purchased from Sigma–Aldrich, Merck Reagenzien, Hemos and Centrohem, respectively. A standard stock solution of phenolic compounds, *i.e.*, Phenol-Mix 15 (phenol, 2-chlorophenol, 2,4-dimethylphenol, 2,4--dichlorophenol, 2,6-dichlorophenol, 4-chloro-3-methylphenol, 2,4,6-trichlorophenol, 2,3,4,6--tetrahlorophenol, pentachlorophenol), in methanol, 2000 mg L^{-1}, was purchased from Dr. Ehrenstorfer GmbH. A working solution of 50 mg L^{-1} was prepared by diluting the basic solution of Phenol-Mix 15 with methanol.

Sampling

Sampling of concrete walls was performed in the dimensions 10 cm×10 cm using a drill, with a diamond plate to the depth of 2 cm. The concrete was then collected in a hermetically sealed container and transported to the laboratory. Further, the sample was homogenized to a particle size <1 μm using a hydraulic press without heating.

Extraction

Powdered concrete (10 g) was mixed with 75 mL of methanol and the pH was adjusted to <3 by adding concentrated hydrochloric acid. Extraction was performed by sonification for 10 min and then 30 min on a mechanical shaker (200–300 rpm). After the particles had settled, the supernatant was filtered. An aliquot of 10 mL was transferred to a 100-mL separation funnel and 50 mL of aqueous potassium carbonate solution (0.1 M) was added. Then, 2 mL of sodium hydroxide (0.5 M) and 1 mL of acetic anhydride were added to the extract. The extract was gently rotated for 2 min in the separating funnel during the release of carbon dioxide and left to stand 10 min with occasional shaking. Thereafter, 10 mL of cyclohexane was added, the mixture intensely shaken and the two phases were separated. The cyclohexane phase (upper phase) was transferred to a 20-mL headspace bottle in which 2 g of anhydrous sodium sulfate had previously been added. The extract was stored in a refrigerator at 4 °C and analyzed within 48 h; 1 mL of extract was taken for GC–MS analysis. Blank and spiked samples were prepared in the same way as a real sample.

Identification of compounds from the standard solution was performed by comparing the characteristic mass spectrum of the compound from the library with the characteristic MS spectrum of the individual phenol in the standard solution.

Instrument and analytical conditions

Chromatographic analyses were performed on the gas chromatograph with mass detector, Agilent Technologies 7890B GC System, Agilent Technologies 5977MSD. A fused silica GC capillary column Agilent J&W (HP-5ms 30 m×0.25 mm *i.d.*, film thicknesses 0.25 μm) was used. The gas chromatographic conditions were a programmed temperature of the capillary column from 60 °C (0.2 min hold) at 30 °C min^{-1} to 150 °C (0 min hold), 8 °C min^{-1} to 179 °C

(0 min hold), 20 °C min^{-1} to 240 °C (2 min hold), 50 °C min^{-1} to 320 °C (hold 5 min constant). The temperature of the splitless injector was 250 °C. The injection volume was 1 µL. Helium, grade 5.0 was used as the carrier gas at constant flow rate of 1.9 mL min^{-1}. The transfer line temperature was 310 °C. The ionization occurred with a kinetic energy of the impacting electrons of 70 eV. Mass spectra and reconstructed chromatograms (total ion current [*TIC*]) were obtained by automatic scanning in the mass range m/z 45–300. The GC–MS data were processed with Agilent GC–MS MassHunter software and the NIST 14 mass spectral library.

Uncertainty estimation

In order to achieve a standardized way of expressing measurement results, in 1993 seven international organizations prepared the GUM.[21] GUM embraces many aspects of uncertainty evaluation. Therefore, it was widely used, respected and recognized by many as the basic document for uncertainty estimation. However, some deficiencies and limitations were noticed in this document. In order to revise and promote the use of GUM and the International Vocabulary of Basic and General Terms in Metrology (VIM), and prepare supplemental guides for GUM,[21,22,29-32] the Joint Committee for Guides in Metrology (JCGM) was formed. The basic document in the JCGM 100 series[21] is based on the law of propagation of uncertainty (LPU).[33] LPU can be used under the following conditions: only one output quantity appears in the model; the model is explicit, *i.e.,* it can be written as $c = f(X_1,\ldots, X_N)$ (although the function c does not need to be analytic); and, the model is well approximated by its linear expansion around the best estimates of the input quantities.

After obtaining the standard uncertainty using LPU, the GUM approach uses the Welch–Satterthwaite formula to obtain the effective degrees of freedom, necessary to calculate the expanded uncertainty. Since the analytical evaluation of the effective degrees of freedom is still an unsolved problem, the GUM is not always appropriate. Moreover, GUM assumes the validity of the central limit theorem, *i.e.*, it assumes that the probability density function (PDF) of the output is approximately normal and can be represented by a Student *t*-distribution. In some cases, this resulting distribution may have an asymmetric behavior or tend to a normal distribution, which implies less validity of GUM.[34]

One way to overcome these constraints is to use methods that contain more information for estimation of measurement uncertainty than GUM. This work presents a methodology that uses the Monte Carlo method for propagation of distributions, which is described in JCGM 101.[22] The difference between the two approaches is illustrated by Fig. 1a and b, which present propagation of uncertainties and propagation of distributions, respectively. In the case of distributions propagation, complete information of the input quantities is available, while in the case of propagation of uncertainties, the input contains only expectation and standard deviation of the input quantities (only the first and the second central moments).

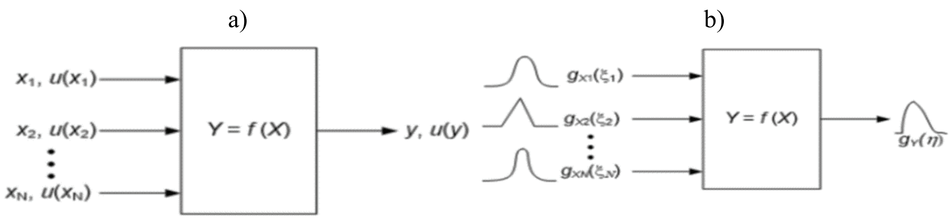


Fig. 1 Monte Carlo method for propagation of distributions, a) propagation of uncertainties; b) propagation of distributions.

The Monte Carlo approach for the evaluation of uncertainty is a reliable tool when the GUM framework is not adequate. Therefore, besides analysis of the measurement uncertainty by the GUM method, the Monte Carlo method used for an estimation of measurement uncertainty in the C++ programming language is implemented.

A procedure for application of Monte Carlo method was performed in the following way: the number of histories M in the MC simulation was selected; N input values and an estimate of their distribution functions were defined; M vectors were generated, by sampling from the PDFs assigned to the input quantities X_i, $i = 1,\ldots,N$; for each vector, the corresponding model Y was created; the obtained values of the models were sorted into increasing order; the output value was estimated and the uncertainty was measured; for the chosen coverage probability p, an appropriate coverage interval was formed (in case of asymmetric PDF, it is the shortest 100 p percentage coverage interval).

RESULTS AND DISCUSSION

Selectivity

Selectivity describes the ability of an analytical method to differentiate various substances in the sample and is applicable to a method in which two or more components are separated and quantified in a complex matrix.[35] The selectivity of the presented method was tested by recording the chromatogram of a spiked sample (7.5 mg kg^{-1}) and blank sample under the previously established chromatographic conditions (see Fig. 2). The resolution successive peak, as a quantification measure of selectivity, was calculated by Eq. (1):

$$R_{2,1} = \frac{2\left(t_{\mathrm{R}_2} - t_{\mathrm{R}_1}\right)}{w_2 + w_1} \tag{1}$$

where t_{R_1} is the retention time, in seconds, of the first peak, t_{R_2} is the retention time, in seconds, of the second peak, w_1 is the peak width on the time axis, in s, of first peak and w_2 is the peak width on the time axis, in seconds, of the second peak.

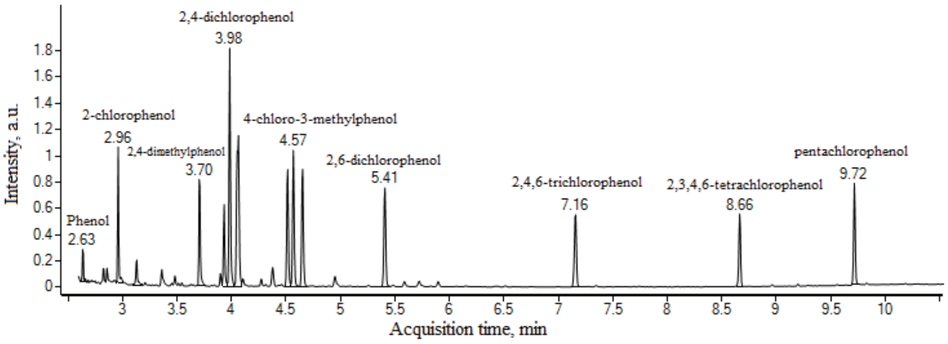


Fig. 2. Chromatogram of a spiked concrete sample (7.5 mg kg^{-1} of each phenol compound).

Resolution for all selected peaks was in the range from 0.5–1.4. The indicated values satisfy the criteria defined in the Eurochem guide,[36] implying that a good selectivity is obtained when using the proposed method for most phenolic compounds.

Linearity

Linearity was tested by linear regression analysis using a calibration standard of acetylated phenolic compounds at five concentration levels (0.05–1.0 mg L^{-1}). For each concentration level, three consecutive measurements were performed. The chromatogram corresponding to the lowest level of the calibration curve is shown in Fig. 2.

Based on the results, parameters of the regression equations (the slope, intercept and correlation coefficient) were determined, Table I.

Examination of the regression line showed that the method is linear with a regression coefficient, $R > 0.99$ for all phenolic compounds in the examined range.

The limits of detection (*LOD*) and quantification (*LOQ*) were determined statistically using regression analysis functions obtained from linearity according to the equations:

$$LOD = 3.3\left(\frac{SD}{b}\right) \tag{2}$$

$$LOQ = 10\left(\frac{SD}{b}\right) \tag{3}$$

where b is the slope of the calibration curve and SD is the residual standard deviation. The obtained *LOD* and *LOQ* values are presented in Table I.

Trueness

To test the trueness of the method, a recovery test was applied. Spiked samples of concrete were prepared for three concentration levels in the range of the calibration curve. The determined concentrations were compared with the spiked amounts. The ratio between the obtained and expected concentrations was calculated and expressed as a percentage, which represents the recovery. The mean value, standard deviation and relative standard deviation (*RSD*) were calculated. For GC–MS analysis, the obtained recoveries ranged from 79 to 91 %, except for 2,4-dimethylphenol (see Table I). According to Eurachem,[36] these mean values of the recoveries were within the acceptable range.

Repeatability

The repeatability of a method is one way of expressing precision within the laboratory, under the same operating conditions over a short period of time, with the same analyst on the same equipment. Repeatability represents agreement

TABLE I. Summary of the method validation data

Compound	R_t min	Acetylated compounds				R	LOD mg kg^{-1}	LOQ mg kg$_{–}^{1}$	Repeatability RSD / %	Precision RSD / %	Recovery %
		1st mass	Relative intensity, %	2nd mass	Relative intensity, %						
Phenol	2.96±0.05	94	100	66	26	0.995	0.01	0.01	0.04	0.08	79
2-Chlorophenol	3.70±0.05	128	100	130	34	0.999	0.01	0.01	0.04	0.08	87
2,4-Dimethylphenol	3.98±0.05	122	100	107	85	0.997	0.03	0.08	0.03	0.06	34
2,4-Dichlorophenol	4.65±0.05	162	100	164	66	0.998	0.03	0.08	0.05	0.07	87
2,6-Dichlorophenol	4.51±0.05	162	100	164	67	0.999	0.01	0.01	0.05	0.10	91
4-Chloro-3-methylphenol	4.56±0.05	107	100	144	60	0.998	0.01	0.01	0.04	0.07	84
2,4,6-Trichlorophenol	5.40±0.05	196	100	164	66	0.997	0.01	0.01	0.04	0.06	88
2,3,4,6-Tetrachlorophenol	7.15±0.05	230	78	232	100	0.992	0.03	0.08	0.03	0.09	89
Pentachlorophenol	8.66±0.05	264	66	266	100	0.987	0.03	0.10	0.05	0.05	85

between the independent test results and it is expressed in the form of the standard deviation or the relative standard deviation (*RSD*). The *RSD* or the coefficient of variation (*CV*), as it is alternatively termed, can be useful in order to eliminate the influence of the concentration. For the determination of the repeatability of the method, six spiked samples with 3.75 mg kg^{-1} of phenols solution were prepared and the GC–MS analysis was performed.

The mean values of concentration and *RSD* were calculated. The obtained results are given in Table I. The *RSD* values ranged from 0.03 to 0.05 %. Based on Eurachem,[36] the calculated *RSD* values were within the acceptable range.

Intermediate precision

The purpose of this test was to check the trueness of the analytical method and to prove that the impact of small variations in the method parameters (different analysts and times of operation) on the trueness was negligible. For this validation characteristic, samples spiked with 3.75 mg kg^{-1} of phenols solution were prepared and injected in triplet during five days. The obtained results were compared with the results of the previous day. The *SD* and the *RSD* were calculated, Table I.

Determination of measurement uncertainty

The content of phenol in a concrete sample was determined according to Eq. (4):

$$c = \frac{y-a}{b}\frac{V}{m}\frac{100}{R} \tag{4}$$

where y is the peak area of a phenol compound, a and b are the intercept and the slope of straight-line fit to a data set, respectively obtained by the application of the method of least squares, V is the volume of solution, m is the mass of sample and R is the recovery.

In accordance with the GUM,[21] all potential sources of uncertainty in the measurement were considered. In addition, the contribution to the uncertainty of the measurements was estimated in a way that uses all available information about the measurement procedure (considering that during propagation of uncertainty and propagation of distribution, no further information than that already known was transmitted). In order to identify all relevant uncertainty sources, a cause–effect diagram was drafted (Fig. 3). All necessary information for uncertainty calculations for both methods are summarized in Table II presenting the data for phenol and in Tables S-I, S-III, S-V, S-VII, S-IX, S-XI, S-XIII and S-XV of the Supplementary material to this paper for the eight other compounds.

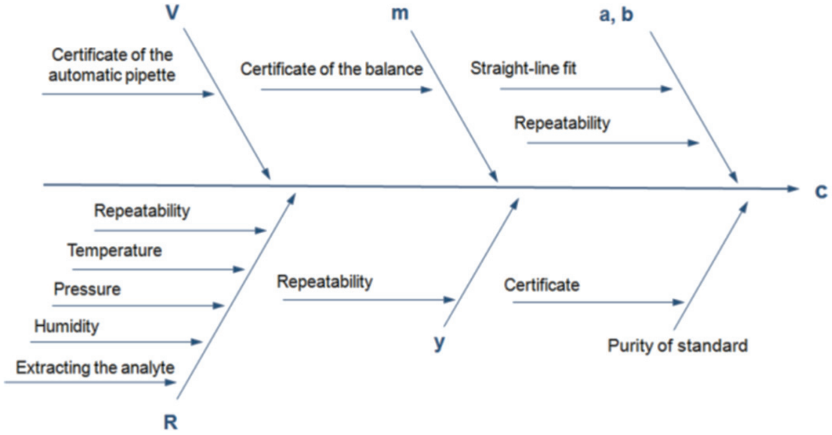


Fig. 3 Cause–effect diagram.

TABLE II. Uncertainty sources and associated distributions with their respective parameters for the estimation of uncertainty for the compound phenol; *SD* – standard deviation; *DF* – degrees of freedom

Uncertainty source	Distribution	Parameters of a distribution
Volume (*V*)	Normal	Mean: 75 mL; *SD*: 2.55 mL
Mass (*m*)	Normal	Mean: 10 g; *SD*: 0.22 g
Recovery (*R*)	Student's *t* location-scale	Mean: 78.80 %; *SD*: 6.81 %; *DF*: 3
The area of peak (*y*)	Student's *t* location-scale	Mean: 194071; *SD*: 9500; *DF*: 3
Intercept (*a*)	Student's *t* location-scale	Mean: 20055; *SD*: 2912; *DF*: 3
Slope (*b*)	Student's *t* location-scale	Mean: 464675 l mg^{-1}; *SD*: 5657 l mg^{-1}; *DF*: 3
Purity of standard	Uniform	Min: –0.00617 mg kg^{-1}; Max: 0.00617 mg kg^{-1}

The last parameter to be defined is the number of histories *M* in the Monte Carlo calculations. According to the "Evaluation of measurement data – Supplement 1" the "Guide to the expression of uncertainty in measurement" – Propagation of distribution using a Monte Carlo method",[22] this number can be chosen as *a priori* in the following way:

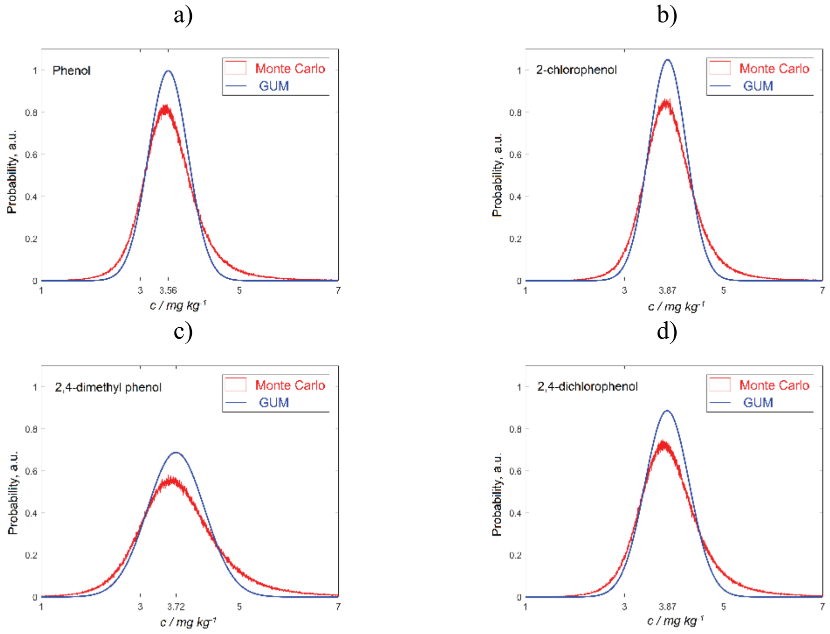
$$M > 10^6 (100-p)^{-1} \tag{5}$$

where *p* is a selected coverage probability. Therefore, when the coverage probability is 95 % (as in the present case), *M* should be at least higher than 2×10^5. Since a larger number of histories *M* causes better convergence of the result, *M* was chosen to be 5×10^6. The results were obtained by processing the set of available information using the GUM uncertainty approach. Corresponding statistical parameters were obtained by Monte Carlo simulation for the compound phenol and the results are presented in Table III. The results for the eight other compounds are presented in Tables S-II, S-IV, S-VI, S-VIII, S-X, S-XII, S-XIV and S-XVI of the Supplementary material. The following parameters were obtained in the Monte Carlo simulation: low endpoint for 95 % and high endpoint for 95 %. They represent the low and high limit of the shortest-length 95 % confidence interval, respectively. It could be seen that for all compounds, the Monte Carlo approach predicts a larger spread of data around a central value. For example, from Table III, it could be noticed that one half of the shortest interval is 1.29 mg kg$^{–1}$ compared to the expanded uncertainty of 0.95 mg kg$^{–1}$.

TABLE III. Results obtained using the GUM and Monte Carlo uncertainty approach for uncertainty estimation for the compound phenol

Parameter (GUM)	Value, mg kg^{-1}	Parameter (MC)	Value, mg kg^{-1}
Mean	3.56	Median	3.56
Combined standard uncertainty	0.40	Low endpoint for 95 %	2.39
Expanded uncertainty for 95%	0.95	High endpoint for 95 %	4.96

Deviation of the PDF of the output estimated by Monte Carlo simulation from the distribution assigned to the measured and through GUM analysis is shown in Fig. 4. It could be seen that these two ways of expressing the measurement results are in disagreement, *i.e.*, the resulting PDFs from Monte Carlo simulations are skewed to the right and their coverage intervals are larger than the coverage intervals obtained by the GUM approach. Since these disagreements come from the assumptions and approximation included in the GUM Uncertainty Framework, the Monte Carlo method described in JCGM 101:2008 is proposed for the analysis of this kind of uncertainty.



a)
b)
c)
d)

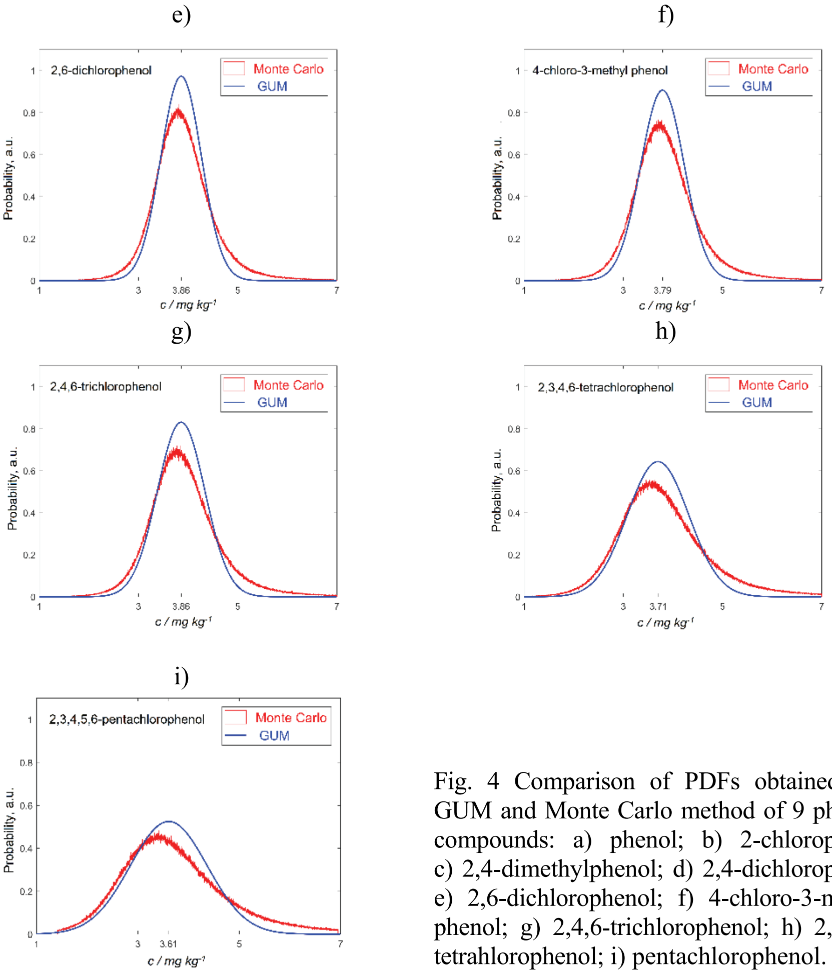


Fig. 4 Comparison of PDFs obtained with GUM and Monte Carlo method of 9 phenolic compounds: a) phenol; b) 2-chlorophenol; c) 2,4-dimethylphenol; d) 2,4-dichlorophenol; e) 2,6-dichlorophenol; f) 4-chloro-3-methylphenol; g) 2,4,6-trichlorophenol; h) 2,3,4,6--tetrahlorophenol; i) pentachlorophenol.

CONCLUSIONS

The gas chromatographic method with mass spectrometric detection was used for the simultaneous identification and quantification of nine phenolic compounds in concrete. The analytical technique was developed in the presented study.

Considering satisfactory values for precision, accuracy, repeatability and linearity, it could be concluded that it is possible to use this novel analytical method for determination of phenol and halophenols in concrete in new and old residential buildings, in order to prevent possible exposure of humans. It was shown that there is a mismatch between the estimation of combined uncertainty of measurement using GUM recommendations and the Monte Carlo simulation method. Monte Carlo simulation was proposed as a more reliable method for the estimation of measurement uncertainty in the analysis of phenolic compounds in concrete.

SUPPLEMENTARY MATERIAL

Additional data are available electronically at the pages of journal website: http://www.shd.org.rs/JSCS/, or from the corresponding author on request.

Acknowledgement. This study was financially supported by Ministry of Education, Science and Technological Development of the Republic of Serbia, Project No. TR 37021.

ИЗВОД

ВАЛИДАЦИЈА И ПРОЦЕНА МЕРНЕ НЕСИГУРНОСТИ АНАЛИТИЧКЕ МЕТОДЕ ЗА ОДРЕЂИВАЊЕ ФЕНОЛНИХ ЈЕДИЊЕЊА У БЕТОНУ

БРАНИСЛАВА Г. САВИЋ[1], ИВАНА Ј. МИХАЈЛОВИЋ[2], СЛОБОДАН М. МИЛУТИНОВИЋ[1], МИНА М. СЕОВИЋ[1], ЖЕЉКА М. НИКОЛИЋ[1], МИЛОШ С. ТОШИЋ[1] и ТАЊА П. БРДАРИЋ[1]

[1]*Институт за нуклеарне науке Винча, Универзитет у Београду, Лабораторија за физичку хемију, Мике Петровића Аласа 12–14, 11351 Винча, Београд и* [2]*Факултет техничких наука, Универзитет у Новом Саду, Департман за заштиту животне средине, Трг Доситеја Обрадовића 6, 21000 Нови Сад*

Органски загађивачи који могу бити присутни у грађевинским материјалима негативно утичу на здравље људи. У раду је представљена аналитичка метода за идентификацију и квантификацију девет фенолних једињења: фенол, 2-хлорфенол, 2,4-диметилфенол, 2,4-дихлорфенол, 2,6-дихлорфенол, 4-хлор-3-метилфенол, 2,4,6-трихлорфенол, 2,3,4,6-тетрахлорфенол, 2,3,4,5,6-пентахлорфенол у бетону, применом гасне хроматографије са масеном спектрометријском детекцијом. Поређењем масених спектара (MS) испитиваних једињења са MS спектрима аналитичких стандарда постигнута је поуздана идентификација. На основу задовољавајућих вредности валидационих параметара (прецизности, тачности, поновљивости и линеарности) може се закључити да је метода применљива у опсегу од 0,01 до 7,5 mg kg^{-1}. Развијена метода би се могла користити при испитивању контроле квалитета бетона на садржај фенола током изградње нових грађевинских објеката, као и за постојеће потенцијално контаминиране објекте. За одређивање мерне несигурности, коришћена су два приступа: препоруке GUM-а и Монте-Карло метод. Уочено је неслагање између наведених метода. Метода нумеричке симулације — Монте Карло се показала као прецизнија метода у процени комбиноване мерне несигурности за анализу садржаја фенолних једињења у бетону.

(Примљено 18. маја, ревидирано 3. децембра, прихваћено 7. децембра 2018)

REFERENCES

1. EPA, US Environmental Protection Agency, https://www.epa.gov/sites/production/files/2015-09/documents/priority-pollutant-list-epa.pdf (Accessed 17 May 2018)
2. 80/778/EEC: *Council Directive of 15 July 1980 relating to the quality of water intended for human consumption* (1980)
3. 93/72/EEC: *Commission Directive of 1 September 1993 adapting to technical progress for the nineteenth time Council Directive 67/548/EEC on the approximation of the laws, regulations and administrative provisions relating to the classification, packaging and labeling of dangerous substances* (1993)
4. J. Michalowicz, W. Duda, *Pol. J. Environ. Stud.* **16** (2007) 347 (https://www.pjoes.com/abstracts/2007/Vol16/No03/02.html)

5. W. Anku, M. Mamo, P. Govender, in *Phenolic Compounds - Natural Sources, Importance and Applications*, M. Soto-Hernandez, M. Palma-Tenango, M. del Rosario, Garcia-Mateos, Eds., InTechOpen, London, 2017, p. 419 (http://dx.doi.org/10.5772/66927)
6. J. Wallace, *Phenol*, in *Kirk-Othmer encyclopedia of chemical toxicology*, J. I. Kroschwitz, M. Howe-Grant, Eds., Wiley, New York, 1996, p. 592 (https://www.wiley.com/legacy/products/subject/reference/kirk_contributors.html)
7. A. T. Hodgson, J. D. Wooley, *Assessment of Indoor Concentrations, Indoor Sources and Source Emissions of Selected Volatile Organic Compounds*, Applied Science Division, Berkeley, CA, 1991, p. 90 (https://pubarchive.lbl.gov/islandora/object/ir%3A95348/datastream/PDF/view)
8. Current list of substances with EU-LCI values, n.d, http://www.eu-lci.org/EU-LCI Website/EU-LCI Values.html (Accessed 9 December 2015)
9. S. Kubba, *Handbook of Green Building Design and Construction*, Butterworth-Heinemann, Oxford, 2017, p. 111 (https://www.elsevier.com/books/handbook-of-green-building-design-and-construction/kubba/978-0-12-810433-0)
10. D. D. Acimovic, Z. M. Nikolic, M. S. Tosic, D. S. Milovanovic, V. M. Nikolic, T. P. Brdaric, M. P. Marceta-Kaninski, *J. Hazard. Mater.* **325** (2017) 271 (https://doi.org/10.1016/j.jhazmat.2016.12.008)
11. N. Fattahi, Y. Assadi, M. R. M. Hosseini, *J. Chromatogr, A* **1157** (2007) 23 (http://dx.doi.org/10.1016/j.chroma.2007.04.062)
12. M. C. Alcudia-León, R. Lucena, S. Cárdenas, M. Valcárcel, *J. Chromatogr. A* **1218** (2011) 2176 (http://dx.doi.org/10.1016/j.chroma.2011.02.033)
13. M. L. Barrico, C. Nabais, M. J. Martins, H. Freitas, *Chemosphere* **65** (2006) 482 (http://dx.doi.org/10.1016/j.chemosphere.2006.01.061)
14. N. R. Neng, J. M. F. Nogueira, *Molecules* **19** (2014) 9369 (https://doi.org/10.3390/molecules19079369)
15. L. Zhang, L. Zhang, W. Zhang, Y. Zhang, *Anal. Chim. Acta* **543** (2005) 52 (https://doi.org/10.1016/j.aca.2005.04.025)
16. Á. Kovács, A. Kende, M. Mörtl, G. Volk, T. Rikker, K. Torkos, *J. Chromatogr, A* **1194** (2008) 139 (https://doi.org/10.1016/j.microc.2011.04.007)
17. Á. Kovács, M. Mörtl, A. Kende, *Microchem J.* **99** (2011) 125 (https://doi.org/10.1016/j.microc.2011.04.007)
18. M. L. Davì, F. Gnudi, *Water Res.* **33** (1999) 3213 (https://doi.org/10.1016/S0043-1354(99)00027-5)
19. ISO/TS 17182: *Soil quality —Determination of some selected phenols and chlorophenols — Gas chromatographic method with mass spectrometric detection* (2014)
20. E. Meyer, *Chemistry of Hazardous Materials*, Prentice Hall Building, Englewood Cliffs, NJ, 1989, p. 236
21. JCGM 100: *Evaluation of measurement data – Guide to the expression of uncertainty in measurement* (2008)
22. JCGM 101: *Evaluation of measurement data — Supplement 1 to the "Guide to the expression of uncertainty in measurement" — Propagation of distributions using a Monte Carlo method* (2008)
23. M. Sega, F. Pennecchi, S. Rinaldi, F. Rolle, *Anal. Chim. Acta* **920** (2016) 10 (https://doi.org/10.1016/j.aca.2016.03.032)
24. D. Theodorou, L. Meligotsidou, S. Karavoltsos, A. Burnetas, M. Dassenakis, M. Scoullos, *Talanta* **83** (2011) 1568 (https://doi.org/10.1016/j.talanta.2010.11.059)

25. A. M. Saviano, F. R. Lourenco, *Measurement* **46** (2013) 3924 (https://doi.org/10.1016/j.measurement.2013.08.005)
26. W. Niemeier, D. Tengen, *J. Appl. Geod.* **11** (2017) 67 (https://doi.org/10.1515/jag-2016-0017)
27. O. Sima, M. C. Lepy, *Appl. Radiat. Isot.* **109** (2016) 493 (https://doi.org/10.1016/j.apradiso.2015.11.097)
28. A. Chen, C. Chen, *Measurement* **87** (2016) 27 (https://doi.org/10.1016/j.measurement.2016.03.007)
29. JCGM 102: *Evaluation of measurement data – Supplement 2 to the "Guide to the expression of uncertainty in measurement" – Extension to any number of output quantities* (2011)
30. JCGM 104: *Evaluation of measurement data — An introduction to the "Guide to the expression of uncertainty in measurement" and related documents* (2009)
31. JCGM 106: *Evaluation of measurement data – The role of measurement uncertainty in conformity assessment* (2012)
32. JCGM 200: *International vocabulary of metrology – Basic and general concepts and associated terms* (VIM) (2012)
33. I. Lira, *Evaluating the Measurement Uncertainty – Fundamentals and practical guidance*, Institute of Physics Publishing, London, 2002, p. 45 (http://unina2.on-line.it/sebina/repository/catalogazione/documenti/Lira-Evaluating%20the%20measurement%20uncertainty.%20fundamentals%20and%20practical%20guidance.pdf)
34. V. Chan, *Theory and Applications of Monte Carlo Simulations*, InTech, Rijeka, 2013, p. 27 (http://dx.doi.org/10.5772/45892)
35. M. R. Christopher, W. R. Thomas, *Development and Validation of Analytical Methods*, Pergamon, Oxford, 1996, p 79 (https://www.amazon.com/Development-Validation-Analytical-Pharmaceutical-Biomedical/dp/0080427928)
36. Eurachem - *The Fitness for Purpose of Analytical Methods*, https://www.eurachem.org/images/stories/Guides/pdf/MV_guide_2nd_ed_EN.pdf (Accessed 17 May 2018).