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A NOVEL VALIDATED METHOD FOR EXTRACTION AND ESTIMATION OF DIMETHYL FUMARATE FROM VARIOUS TEXTILE MATRICES ON GC/MS.

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ABSTRACT:

A novel and rapid analytical method was developed for the determination of extractable dimethyl fumarate (DMFu) in various materials, including leather, polyurethane (PU) leather, textiles, rubber, and silica gel pellets, using gas chromatography-mass spectrometry (GC/MS). DMFu was extracted using ethyl acetate and analyzed on a DB-624 capillary column with helium gas (99.9% purity) as the mobile phase. One target ion and two qualifier ions were selected for the identification and quantification of DMFu. Key pre-treatment parameters, such as extraction method, solvent, and time, were optimized to achieve optimal performance. Under the optimized conditions, the method demonstrated a recovery rate of 91% to 98% for the standard. The calibration curve exhibited excellent linearity with a regression value of 0.9995 and a working range of 0.01–0.50 mg L^{-1}. The relative standard deviation (RSD) was ≤10% for test specimens and ≤5% for the pure standard. The method's limit of detection (LOD) and limit of quantification (LOQ) were 5×10^{-4} mg L^{-1} and 5×10^{-3} mg L^{-1}, respectively. The developed method was successfully applied to real samples, revealing DMFu concentrations ranging from 1.34 mg L^{-1} to 5.12 mg L^{-1}. This approach provides a reliable and efficient means for the accurate determination of DMFu in diverse materials.

KEYWORDS: Dimethyl fumarate (DMFu), Solvent extraction, Textile and Leather samples, GC/MS Analysis.

INTRODUCTION:

Dimethyl fumarate (DMFu) is a widely used anti-mold agent employed to protect consumer products such as leather goods, polyurethane (PU) leather, and textiles during storage and transportation, particularly in humid climates. It is commonly applied to prevent fungal growth, ensuring the integrity of products like shoes, purses, and other leather or textile-based items. DMFu is often incorporated into desiccant sachets or pads placed inside product packaging to control humidity during transit (1-4). However, over time, DMFu can evaporate and impregnate the materials, leading to potential deterioration of the products. In some cases, it is directly sprayed onto the surface of the items. Despite its effectiveness as a preservative, DMFu has raised significant health and safety concerns, particularly in the European market, due to its adverse

effects on human health (5-8).

DMFu has been linked to severe allergic reactions, skin irritation, and respiratory issues upon exposure. When DMFu evaporates inside packaging, it can condense on the surface of products, leading to painful dermatitis even at low concentrations. Symptoms of DMFu exposure include itching, redness, burns, and temporary breathing difficulties. These health concerns first gained attention in Europe following reports of adverse reactions linked to household consumer products imported from Asian countries (9-12). Numerous cases were documented, particularly involving contaminated leather footwear, which prompted regulatory action. In response to these health risks, the European Commission issued Directive 2009/251/EC, which banned the import of products containing DMFu and set a strict limit of 0.1 mg/kg for its presence in consumer goods. This was further reinforced by Commission Regulation 412/2012, which permanently prohibited the use of DMFu in products imported into Europe (13-16).

The toxicity of DMFu has been extensively studied using animal models, which have provided insights into its potential health hazards. These studies have highlighted the need for stringent monitoring and control of DMFu levels in consumer products (17-20). Analytical methods for detecting and quantifying DMFu typically involve solvent extraction followed by analysis using gas chromatography (GC) coupled with electron impact ionization mass spectrometry (EI/MS) or high-performance liquid chromatography (HPLC/UV). These techniques have been employed to assess DMFu contamination in various materials, including leather, textiles, and rubber (20-23). In this study, a novel and efficient method was developed for the extraction and quantification of DMFu from real test specimens, including silica gel pellets, rubber, leather, PU leather, and textiles. Ultrasound-assisted solvent extraction was employed to enhance the efficiency of DMFu recovery, followed by analysis using gas chromatography-mass spectrometry (GC/MS). The method was optimized for key parameters such as extraction solvent, time, and technique to ensure accurate and reliable results. The developed method was validated for its sensitivity, precision, and accuracy, with a focus on meeting regulatory requirements for DMFu detection in consumer products.

The findings of this research contribute to the growing body of knowledge on DMFu analysis and provide a robust analytical framework for monitoring its presence in consumer goods. By addressing the challenges associated with DMFu detection, this study supports efforts to ensure product safety and compliance with international regulations, ultimately protecting consumers from the harmful effects of this hazardous substance.

EXPERIMENTAL:

INSTRUMENTATION:

An analytical balance (Model: ML 204/01, Mettler Toledo) was used to weigh the test specimens. The chromatographic analysis was performed using a GC-2010plus system (Shimadzu) equipped with an auto sampler (AOC-20s), an auto injector (AOC-20i), and a mass selective detector (QP2010ultra-plus). The system was operated using GC/MS Solution software (version 2.70, Shimadzu Corporation), supported by the NIST 147 and 127 mass spectral libraries for compound identification. Separation of DMFu was achieved using a DB-624 capillary column (320 µm × 30 m × 1.8 µm) with a maximum temperature capacity of 350 °C. Electron impact (EI) mass spectrometry was employed to confirm the presence and structural identity of DMFu. For quantification and peak identification, the system was operated in full scan mode (m/z = 40 to 150) with a 1.2 kV electron multiplier and 70 eV EI ionization. The target ion was set at m/z 113, while m/z 85, 59, and 114 were selected as qualifier ions for accurate identification and quantification of DMFu.

CHROMATOGRAPHIC CONDITIONS:

Auto-tuning of the mass-selective detector was conducted using perfluorotributylamine (PFTBA), with key ions monitored at m/z 69, 219, and 502. The chromatographic system was operated in split-less injection mode, with a purge flow rate of 2.0 mL/min following the injection of test specimens. High-purity helium (99.99%) was used as the carrier gas at a constant flow rate of 1.0 mL/min. The injection volume for test specimens was set at 1.0 µL. Temperature settings were optimized as follows: the injector temperature was maintained at 250 °C, the ion source temperature at 200 °C, and the interface temperature at 260 °C to ensure efficient vaporization, ionization, and transfer of analytes.

TEMPERATURE PROGRAM:

The initial column temperature was set at 50 °C and held for 1.0 minute after injection. Subsequently, the temperature was increased at a rate of 70 °C per minute, rising from 50 °C to 250 °C, and then held at 250 °C for 3.0 minutes. The total runtime for the analysis was 6.68 minutes, ensuring efficient separation and detection of the target analyte within a short timeframe.

CHEMICALS AND SOLVENTS:

High-performance liquid chromatography (HPLC) grade chemicals and organic solvents were procured from local suppliers. Acetone and ethyl acetate were obtained from Lab Line Suppliers, while the working standard, dimethyl fumarate (DMFu), was purchased from Chem Service, a local supplier based in Lahore, Pakistan. All chemicals and solvents were of high purity and used without further purification to ensure the accuracy and reliability of the analytical results.

REAGENT PREPARATION:

A working standard solution of dimethyl fumarate (DMFu) at a concentration of 1000 mg/L was prepared by accurately weighing 50.0 ± 0.5 mg of DMFu and dissolving it in ethyl acetate within a 50 mL volumetric flask. From this working standard, an intermediate standard solution of 10 mg/L was prepared. This intermediate standard was then serially diluted to obtain a series of calibration standards with concentrations of 0.01, 0.025, 0.05, 0.10, 0.25, and 0.50 mg/L. These calibration standards were used to establish the calibration curve for the quantitative analysis of DMFu in the test specimens.

TEST SPECIMEN COLLECTION:

Silica gel pellets and various commercial textile products, including finished garments, printed and non-printed fabrics, leather, polyurethane (PU) leather, and rubber articles, were collected from retail stores and textile processing industries in Lahore, Pakistan, during the period from February to May 2019. These samples were selected to represent a diverse range of consumer goods commonly available in the market, ensuring a comprehensive analysis of dimethyl fumarate (DMFu) contamination across different materials.

TEST SPECIMEN PREPARATION:

For rubber, polyurethane (PU) fabric, leather, and other textile test specimens, the samples were cut into small pieces measuring approximately 5 mm × 5 mm. In the case of silica gel specimens,the samples were finely ground into a powder to ensure homogeneity. Three specimens, labeled A, B, and C, were selected as matrix blanks. These blank specimens were spiked with pure dimethyl fumarate (DMFu) standard and analyzed using various solvents,

extraction times, and methods to optimize the extraction process. The results indicated that the ultrasonic extraction method using ethyl acetate as the solvent for 40 minutes provided the best recovery efficiency. Consequently, ethyl acetate was chosen as the extraction solvent, 40 minutes as the extraction time, and the ultrasonic method as the preferred extraction technique. Detailed results of this optimization process are summarized in Figure 1 and Tables S1, S2, and S3 in the supplementary data.

TEST SPECIMEN EXTRACTION:

An accurately weighed 1.00 g test specimen was placed into a culture tube, and 10 mL of ethyl acetate was added to the tube, which was then tightly capped. The mixture was sonicated at 70 °C for 40 minutes to ensure efficient extraction of dimethyl fumarate (DMFu). After sonication, the mixture was filtered using glass wool and a 0.45 µm syringe filter to prepare the sample for GC/MS analysis. The 40-minute extraction process was found to be sufficient for the complete extraction of DMFu, eliminating the need for additional extraction steps. This optimized procedure ensured high recovery and reliable quantification of DMFu in the test specimens.

INSTRUMENTAL ANALYSIS BY GC/MS:

Before conducting any batch of test specimens, the following GC/MS instrument parameters were ensured. The instrument was conditioned according to the specified instrumental conditions (Section I) and temperature program (Section II). A calibration curve for dimethyl fumarate (DMFu) was established using the peak area versus analyte concentration, demonstrating a linear regression coefficient (r^2) of > 0.9995. To ensure the absence of contamination, method blanks and specimen blanks were analyzed. A 0.1 mg/L standard solution was used to assess instrument sensitivity. For recovery estimation, 0.5 mg/L solutions of laboratory quality control, specimen spike, and calibration standard check were injected into the instrument. The recovery of these solutions fell within the acceptable range of 85–115% (results were summarized in Table S4 in the supplementary data).

Test samples were then injected into the instrument for analysis. The presence or absence of the target analyte was confirmed by comparing the mass spectrum and retention time (RT) with those of the standard. The retention time of the test specimen components was required to be within ± 0.01 RT units of the standard component's relative RTs. If the quantitation wavelength response ratio exceeded the range of the prepared calibration curve, the test specimen extract was diluted to bring it within the required range. For specimens that fell outside the calibration range, retesting was performed by either increasing the final volume of ethyl acetate or reducing the test specimen weight to ensure accurate quantification.

RESULTS AND DISCUSSION:

ANALYTICAL METHOD VALIDATION:

Working range and linearity: A six-point calibration curve was established, with concentrations of 0.01, 0.025, 0.05, 0.1, 0.25, and 0.5 mg/L. The curve demonstrated excellent linearity, with regression coefficients (r^2) ranging from 0.9995 to 0.9997. The working range of the calibration curve spanned from 0.01 mg/L to 0.5 mg/L. Each concentration level was prepared and analyzed in triplicate to ensure precision and accuracy. Detailed results of the calibration curve are summarized in Figure S1 in the supplementary data.

Specificity: To evaluate the potential impact of matrix or reagent interference on the

chromatographic technique, matrix blanks, reagent blanks, and pure standards were analyzed. The method blank and matrix blank showed no detectable signals, confirming the absence of contamination from reagents or the matrix. The results of sensitivity checks, laboratory quality control, specimen spikes, and calibration standard checks demonstrated recoveries within the acceptable range of 100 ± 15%. These findings are summarized in Table S4 in the supplementary data, highlighting the reliability and accuracy of the analytical method.

Accuracy: A test specimen and a blank solution were spiked with pure standard at concentrations of 0.1 mg/L, 0.3 mg/L, and 0.5 mg/L. Each concentration level was prepared in replicates and analyzed three times to ensure precision and accuracy. The recovery rates for these individually prepared replicates ranged from 91.0% to 99.4%, demonstrating the method's effectiveness in accurately quantifying dimethyl fumarate (DMFu) across different concentration levels. Detailed results of these recovery studies are summarized in Table S5 in the supplementary data.

Precision: A specimen solution containing the target level of the analyte was prepared, and 10 replicates were generated using the final method protocol. These replicates were analyzed over six consecutive days to evaluate the method's precision. The relative standard deviation (RSD) for intra-day analysis was 1.6%, while the RSD for inter-day analysis was 2.5%. These results demonstrate the method's high precision and reproducibility, both within a single day and across multiple days.

LOD and LOQ: Spike solutions and blank solutions with progressively decreasing concentrations of the analyte were prepared to determine the limit of detection (LOD) and limit of quantification (LOQ) for the proposed analytical method. These solutions were analyzed using the developed method, and the LOD and LOQ were determined based on the lowest concentration of the analyte that could be accurately detected and quantified, respectively. The limit of detection was found to be 5×10^{-4} mg/L, while the limit of quantification was 5×10^{-3} mg/L. These values demonstrate the method's high sensitivity and ability to detect and quantify dimethyl fumarate (DMFu) at very low concentrations.

Selectivity: The selectivity of the developed GC/MS method was evaluated by preparing mixtures of the analyte (DMFu) with textile test specimens. The recovery of DMFu in the presence of potential interferences was found to be within the range of 100 ± 15%, confirming the method's ability to accurately quantify DMFu even in complex matrices. These results, demonstrating the method's robustness and selectivity, are summarized in Table S5 in the supplementary data.

Stability: The stability of the analyte (DMFu) in the presence of other components in the solution was evaluated by observing the percentage deviation of results obtained over a three-day period. The deviation of the analyte concentration was found to be less than 3% during this period, indicating excellent stability of DMFu in the solution. This result confirms that the analyte remains stable and reliable for analysis over a short-term storage duration.

Robustness: To assess the robustness of the developed method, chromatographic parameters were intentionally altered. The column oven temperature was varied from the original range of 50–250 °C to 45–245 °C and 55–255 °C, while the carrier gas flow rate was adjusted from 2.0 mL/min to 1.95 mL/min and 2.05 mL/min. The results demonstrated that these changes had no significant impact on the analyte (DMFu), as the deviation of the analyte recovery remained within the acceptable range of 85–115%. These findings, summarized in Table 2, confirm the method's robustness and reliability under slightly modified experimental conditions.

TEXTILE SAMPLES SCREENED FOR DMFU CONTENTS:

The analysis of dimethyl fumarate (DMFu) in test specimens revealed a range of concentrations,

with the lowest detected value being 1.34 mg/L and the highest value being 5.12 mg/L. Among the analyzed samples, sample D contained 2.23 mg/L of DMFu, sample E contained 5.09 mg/L, sample F contained 2.11 mg/L, sample G contained 1.99 mg/L, sample H contained 1.34 mg/L, sample I contained 4.65 mg/L, and sample J had the highest concentration at 5.12 mg/L. Over a four-month period, a total of 65 test specimens were analyzed, of which 7 specimens showed detectable levels of DMFu. These results are summarized in Table 1.

The chromatographic analysis demonstrated excellent peak resolution for DMFu, with the analyte eluting at a retention time of 6.37 minutes (results shown in Figure 2). The mass spectrum of DMFu identified the parent ion mass fragment at m/z 113, with daughter ions at m/z 59 and m/z 85, confirming the presence and structure of DMFu (mass spectrum as shown in Figure 3). The DMFu content leached from the test specimens ranged from 1.34 mg/L to 5.12 mg/L, as detailed in Table 1.

Table 1. Results of detectable test specimens analyzed on GC/MS

Test specimens	Test specimen description	DMFu mg/L	Test specimens	Test specimen description	DMFu mg/L
A	PU Leather	BDL	F	Blue denim pant	2.11
B	Rubber	BDL	G	Black denim pant	1.96
C	Silica gel	BDL	H	Brown Leather	1.34
D	Rubber	2.23	I	Yellow fabric	4.65
E	PU Leather	5.09	J	Silica gel	5.12

Textile samples A, B, and C were utilized as blank matrices to optimize extraction parameters, including extraction time, method, and solvent for DMFu analysis. The results of these optimization studies are summarized in Figure 1 and Tables S1, S2, and S3 in the supplementary data. These findings highlight the method's effectiveness in accurately detecting and quantifying DMFu in various test specimens.

Table 2. Results of robustness study of DMFu for GC/MS instrument.

Test specimen	Spike level (mg/L)[a]	Flow rate, 2.0 mL/min	Flow rate, 2.05 mL/min	Flow rate, 1.95 mL/min	Column oven temperature 100-250 ˚C	Column oven temperature 95-245 ˚C	Column oven temperature 105-255 ˚C
A	0.1	0.088	0.079	0.078	0.096	0.081	0.085
B	0.2	0.201	0.206	0.205	0.203	0.205	0.207
C	0.5	0.503	0.508	0.506	0.495	0.492	0.494

[a]=Three replicate measurements

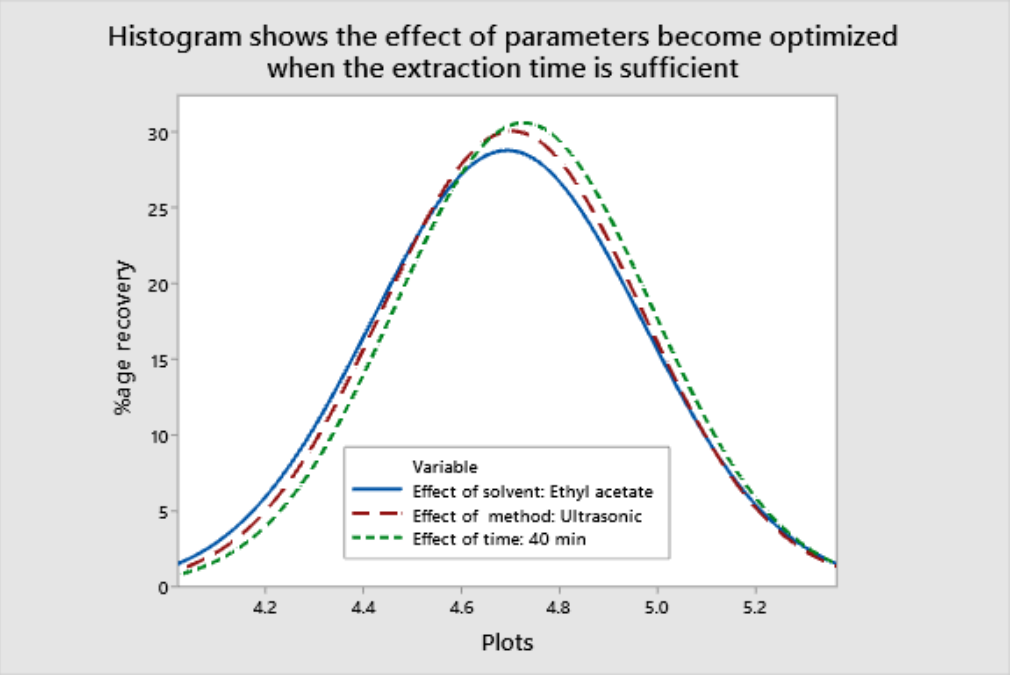

Figure 1. Shows extraction efficiencies of DMFU contents (average results in mg/L) for textile samples spiked with known concentrations of analyte for extraction, method, time and solvent.

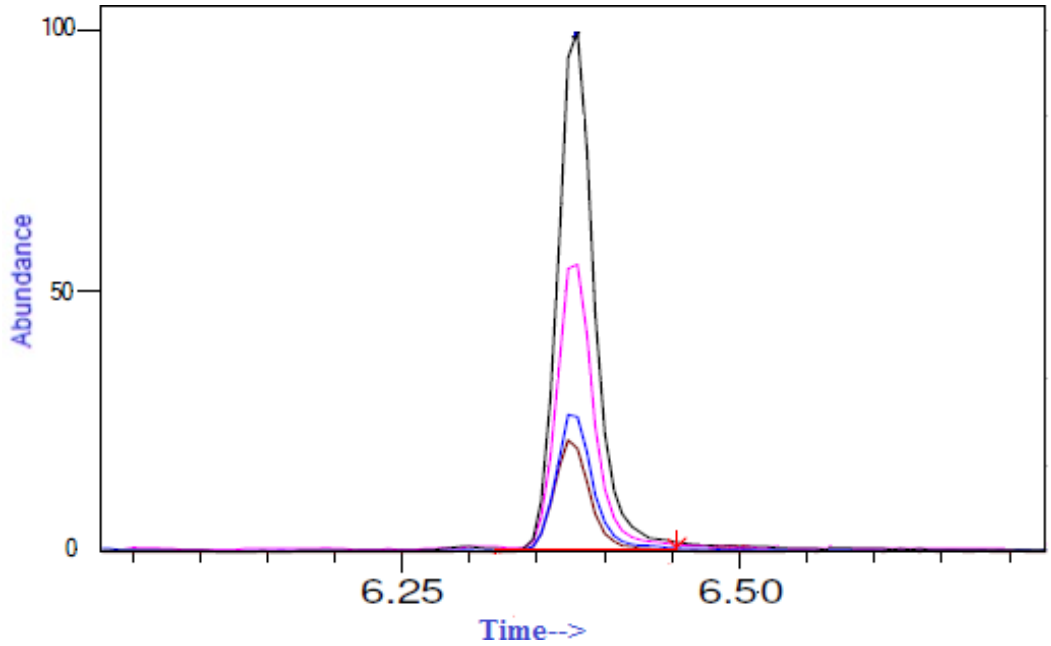

Figure 2. Chromatogram from GC/MS of DMFu, analytical method operated was presented at section I & II.

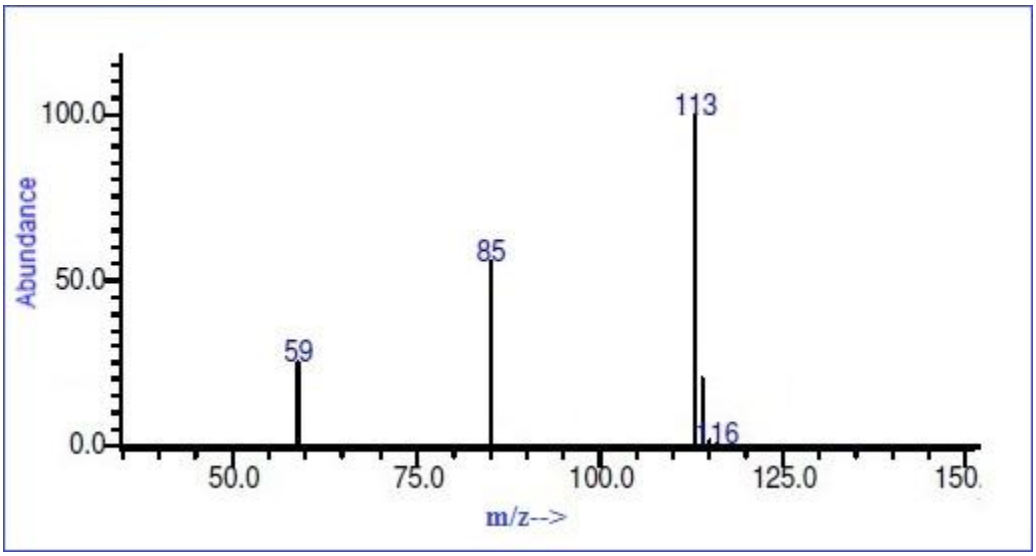

Figure 3. Mass spectrum of DMFU, Parent ion mass fragment was m/z 113, daughter ions were m/z 59 and m/z 85

CONCLUSIONS:

The suitability of the GC/MS technique for the separation and quantitative analysis of dimethyl fumarate (DMFu) in leather, polyurethane (PU) leather, textiles, rubber, and silica gel pellets was successfully demonstrated. The target analyte, DMFu, was analyzed using a DB-624 column with helium as the carrier gas. Under optimized conditions, the method exhibited excellent linearity ($r^2 \geq 0.9995$) within the concentration range of 0.01 to 0.50 mg/L. The recovery rates ranged from 91% to 98%, with a relative standard deviation (RSD) of ≤ 3%. The method's sensitivity was confirmed with a limit of detection (LOD) of 5×10^{-4} mg/L and a limit of quantification (LOQ) of 5×10^{-3} mg/L.

This analytical method is highly effective for ensuring the safety of leather, PU leather, textiles, rubber, and silica gel pellets by accurately quantifying DMFu levels. Additionally, it serves as a valuable tool for biomonitoring human exposure to DMFu, particularly through direct skin contact with contaminated materials. The method's precision, sensitivity, and reliability make it a robust approach for regulatory compliance and consumer safety assessments.

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