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Method validation for the determination of Chloramphenicol, a banned veterinary antibiotic in shrimps, by using liquid chromatography-tandem mass spectroscopy

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Chloramphenicol is one of the most widely used antibiotics for both food and domestic animals. It is a broad spectrum antibiotic which interferes with protein synthesis of many Gram-negative and Gram-positive bacteria and has toxic effects on human. Maximum Residue Level (MRL) of Chloramphenicol in shrimp is 0.5 μg kg^{-1}. In Europe, Chloramphenicol is currently banned from use on animals used as food sources and animal feed products. Although unauthorized, Chloramphenicol was used in Shrimp culture in Asia, where shrimp are cultured primarily for the European market. The main objective of the present study was to develop a simple and rapid method to analyse Chloramphenicol residues in shrimps using Liquid Chromatography-tandem mass spectroscopy (LC-MS/MS). Shrimp samples were homogenised and directly extracted with acetonitrile. HPLC grade standard Chloramphenicol (98%) was used for all experiments. The chromatographic separation was carried out on a C-18 column under isocratic elution conditions. The mass analyser was operated in the negative polarity of multiple reaction monitoring (MRM) mode. The mass spectrum of the precursor ion was found at m/z =320.9 and product ions were obtained at m/z=256.9 and 152.0. The linearity of the plot was obtained between 0.05 μg L^{-1} – 100 μg L^{-1} and the r2 value was greater than 0.99. The obtained average recoveries against seven replicates at 2, 45 and 90 μg kg^{-1} levels were 85, 110 and 115%, respectively. Accuracy of the proposed method was within the range of (-2.74) – (+2.74). The LOQ and LOD values were 0.076 μg kg^{-1} and 0.055 μg kg^{-1} respectively. The validation parameters demonstrated good linearity, accuracy, precision, and recovery. The LOD and LOQ of Chloramphenicol in shrimps showed greater sensitivity with respect to required MRL. Moreover, the proposed method provides a simple sample preparation in a shorter time. Hence, this study has proven to be efficient and suitable for monitoring Chloramphenicol in shrimps.

Key words: Chloramphenicol, Shrimp, LC-MS/MS, MRM, Accuracy

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Removal of toluene in an intramural environment using *Sansevieria trifasciata*

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Volatile Organic Compounds (VOCs) are a group of chemicals that can be emitted as gasses into indoor air at room temperature. Benzene, toluene, ethylbenzene, and xylene are the most common hazardous VOCs that persist in indoor air. *Sansevieria trifasciata* is a common and hardy ornamental plant that survives indoors for long periods. An experimental model was used to study its removal capabilities of indoor air VOCs under ambient conditions using toluene as the pollutant. Three independent experiments were conducted using two airtight glass chambers (1 m^3), one as a test chamber and one as the control. The test chamber consisted of

a healthy plant (3 replicates, 1191.41 ± 45.99 cm^2). An overhead light box with five 18 W fluorescent tubes was used to provide constant light. A thermohygrometer was placed in the chamber to measure the temperature and humidity in the chamber. The chambers were completely sealed to minimize leakages. Toluene (220 µL) was introduced to the sealed chamber through the injecting port. After 2 hours of equilibration, air samples (10.0 mL) were collected using a gas-tight syringe and manually desorbed into carbon disulfide (2 mL). Samples were analyzed using Gas Chromatography-Mass Spectrometry (GC-MS). The concentration of toluene in the chamber was determined for three consecutive days within 24-hour time intervals. Toluene reduction was observed compared to the control chamber. Three independent experiments revealed that the plant's toluene removal was 20.13 ± 3.20, 21.52 ± 0.90, 37.67 ± 5.20 $\mu g.m^{-3}cm^{-2}$ respectively on the 1st, 2nd, 3rd day. Its toluene removal efficiency was 0.80 ± 0.06 $\mu g.\ m^{-3}.\ h^{-1}.\ cm^{-2}$ under ambient conditions. The results indicate that *S. trifasciata* is a good botanical purifier of toluene in indoor air. Indoor plants not only beautify indoor environments but also remove VOCs from air.

Keywords: Toluene, indoor air quality, *S. trifasciata*.

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Development and validation of new method for the simultaneous determination of Clotrimazole and Beclomethasone Dipropionate by HPLC in topical cream formulation

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New topical cream formulations for dermatological treatment against broad spectrum antimycotic agent and various inflammatory conditions contain a combination of Clotrimazole (CT) and Beclomethasone Dipropionate (BD). The National Medicines Regulatory Authority and National Medicines Quality Assurance Laboratory require validated analytical methods in order to obtain reliable test results. This work is focused on developing a simple, precise, and rapid HPLC method for simultaneous determination of CT and BD in drug products. The method development and optimization were carried out using a C18, (Pursuit 5, ODS) 250 mm x 4.6 mm, 5 µm column with acetonitrile–0.1% tri-ethylamine (75:25, v/v) as the mobile phase at 40 ± 2 °C (Column temperature). The flow rate of 1.0 ml/min was used for good separation at the detection wavelength of 239 nm. The validation of the proposed method was carried out for specificity, linearity, accuracy, precision, limit of detection, and robustness test as per the ICH guidelines. Under the above conditions, the retention times of CT and BD were 4.415 min and 5.482 min respectively. The method was found to be linear with a correlation coefficient of 0.9997 for CT and 0.9996 for BD. Chromatograms of standard and sample solutions of CT and BD were compared. Sample chromatograms indicated peak purity confirming the specificity of the method. Precision study showed that the percentage relative standard deviation was within acceptable limits, and the mean recovery was found to be within the range of 99.40 - 99.60 % for CT and 99.44 - 99.81 % for BD in cream base. On investigating the robustness of the assay method, no significant change in chromatographic parameters (difference in mean is less than 2%) was observed. The results of the statistical analysis demonstrated that the values of the validation parameters are acceptable, and therefore, this method can be used for the routine analysis and quantitative determination of CT and BD in cream formulations.

Keywords: Clotrimazole, Beclomethasone Dipropionate, Tri-ethylamine, correlation coefficient, antimycotic