

MULTI RESIDUE PESTICIDE ANALYSIS OF α -ENDOSULFAN, β -ENDOSULFAN AND BIFENTHRINE IN BLACK TEA USING GAS CHROMATOGRAPHY-MASS SPECTROMETRY (GC-MS)**W.A.P.P. Nishadini¹, K.R.W. Abeywickrama² and A.C.A. Jayasundera^{1*}**¹*Department of Chemistry, Faculty of Science, University of Peradeniya, Sri Lanka*²*Sri Lanka Tea Board, 574, Galle Road, Colombo 3, Sri Lanka***acaj@pdn.ac.lk*

Pesticide residues in food commodities are currently of concern in the society due to health problems. Blending of imported tea from India and China, which being the most pesticide using countries for tea is considered to be the main reason for pesticide residues in Sri Lankan Tea. Endosulfan (organochlorine) and bifenthrine (pyrethroid) are included as frequently using pesticides in tea and their health risk on human is of great concerned due to intensive utilize. Maximum Residue Level (MRL) for α -endosulfan, β -endosulfan and bifenthrine are 0 mg kg⁻¹, 0 mg kg⁻¹ and 5 mg kg⁻¹ respectively according to Sri Lanka Tea board guidelines.

Residue analysis of α -endosulfan, β -endosulfan and bifenthrine in tea involved extraction of pesticides from tea collected from different elevations (high grown, mid grown and low grown) with hexane, then enrichment and clean up with solid phase extraction and subsequent identification and quantification by GC-MS. Two samples from each elevation were taken. Analysis was carried out with a 2010 Shimadzu gas chromatography coupled to QP 2010 Plus mass spectrometer with an AOC-20s Shimadzu auto sampler. Chromatographic separation was performed on a Rtx 5-SilMS (30 m×0.25 mm I.D., 0.25 μ m f.t) capillary column under single quadrapole ion separation. Helium (purity 99.999%) was used as the carrier gas at a constant pressure of 100.1 k Pa. Injection port temperature was set at 250 °C and 2 μ L was injected through split less injector. Mass spectrometer was operated in Electron Impact Mode (EI) and data acquisition was performed in Selected Ion Monitoring mode (SIM). By the Standard pesticide total chromatogram, retention time of each peak was identified. Retention time of analytes were confirmed by getting characteristic mass spectrum corresponding to each peak.

Detectable amounts of α -endosulfan, β -endosulfan and bifenthrine residues were not observed. Limit of Detection (LoD) can be calculated when signal to noise ratio is 3. LoD values of α -endosulfan, β -endosulfan and bifenthrine were 0.04, 0.04 and 0.01 μ g ml⁻¹ respectively. Method validation was performed by spiking the blank tea samples with 0.7 μ g ml⁻¹ pesticide standards and then by calculating recovery [Recovery percentage = (Detected concentration / Spiked concentration) × 100%]. To calculate the recovery, pesticides were extracted from pesticide spiked tea samples using same analytical procedure. Here recovery percentage has to be between 80%-120% to accept the analytical procedure as a validated procedure.

The study concluded that this method is fast and efficient for pesticide residue analysis and no evidence was recorded for α -endosulfan, β -endosulfan and bifenthrine in Sri Lankan Black Tea from different elevations (low grown, mid grown and high grown). Recovery percentage values for spiked tea samples were between 80 % to 120 %. Therefore this analytical method can be considered as a validated method.