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TRACE EVIDENCE ANALYSIS OF ENDOSULFAN BY GC-MS: ATTEMPTED HOMICIDAL POISONING

Forensic Science

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ABSTRACT

We report here a case of 70-years-old woman who drunk water kept in earthen pot; which was intentionally contaminated by Endosulfan and ethyl alcohol. Due to consumption of this water victim felt some uneasiness. Chemical analysis of the water sample by Gas chromatography Mass Spectrometry (GC-MS) revealed organochlorine insecticide Endosulfan. In addition to this water was also found to be contaminated with Ethyl alcohol.

KEYWORDS

Homicidal poisoning, Endosulfan, Organochlorine insecticide, GC-MS.

Introduction:
Endosulfan (M. F. $C_9H_6Cl_6O_3S$; 6,7,8,9,10,10-hexachloro-1,5,5a, 6,9,9a-hexahydro-6,9-methano- 2,4,3-benzodioxathiepine-3-oxide) is an organochlorine insecticide and acaricide; introduced in 1954 by Farbwerke Hoechst AG under the name of Thiodan. It is a mixture of two stereoisomer (α-endosulfan and ß-endosulfan) in the ratio 7:3. It belongs to cyclodienes group and widely used in agriculture since 1960 [1]. The global ban on the manufacture and use of endosulfan was recommended by the Stockholm Convention held in April 2011 because of its adverse effects on human health and the environment. Exposure to Endosulfan can occur through inhalation, ingestion, eye or skin contact; all routes of exposure can be hazardous. Endosulfan causes overstimulation of central nervous system (CNS) and respiratory effects in humans. Accidental and suicidal poisoning with Endosulfan is reported in literature [2-5]. J. L. Blanco-Coronado et al. describes the report of six patients with acute Endosulfan intoxication [2]. Also a case series of Endosulfan poisoning was described by Cebicci H. and his group in detail [6].

Case History:
A case was registered by the married woman of 70 years under IPC 328 against her brother-in-law for allegedly mixing of something in drinking water kept in earthen pot, to cause some intentional health hazard to her and her husband. Fortunately, Victim felt some uneasiness, when consumed water of that earthen pot. So, she approached the nearest police station to register a complaint. Police authority seized water of earthen pot and sent to forensic laboratory for chemical analysis.

No other biological or non-biological evidence except water of earthen pot was seized by Police officer from scene of crime.

Experimental:

Gas Chromatography Mass Spectrometry method:
Endosulfan was determined in ether extract of earthen pot water by Gas chromatography mass spectrometry (GC-MS) operated in EI mode with following parameter. Figure 1 shows chromatogram for Endosulfan.

Column: HP-5MS, 5% Phenyl Methyl Silox. 325 °C: 30 m x 250 µm x 0.25 µm.

Oven program: 70 °C for 5 min. then 10 °C/min to 280 °C for 30 min.
Run time: 56 min.

Injector Temperature: 220 °C

Transfer line Temperature: 280 °C

Mass Range: 40-550 amu.

Carrier Gas (He) Flow Rate: 1 mL/min.

Mode: Splitless

Results and Discussion:
Initially, water sample was tested for volatile poisons. Ethyl alcohol and petroleum hydrocarbons were found to be positive.

In routine forensic toxicological analysis, insecticides (poisons) are generally analyzed by thin-layer chromatography (TLC). For TLC analysis water sample was extracted in diethyl ether. Hexane-acetone (8:2, by volume) mixture was used as solvent system for ether extract residues. The samples were spotted on TLC plates with fine capillary tubes along with pure insecticides as a standard. The plates were dried, and the chromatogram was developed in a presaturated tank containing the solvent system as mentioned above. After developing the plates, the extra solvent was evaporated (dried) in fume hood. The plates were then sprayed with various chromogenic sprays; but few of them showed positive response like o-toludine in acetone, Sodium hydroxide-Nickel amine, Zinc chloride-Diphenyl amine in acetone etc. which is not conclusive. So, sample (ether extract) was subjected for GC-MS analysis. The GC-MS analysis revealed organochlorine insecticide Endosulfan, both isomers. **(Figure 1 and 2)**

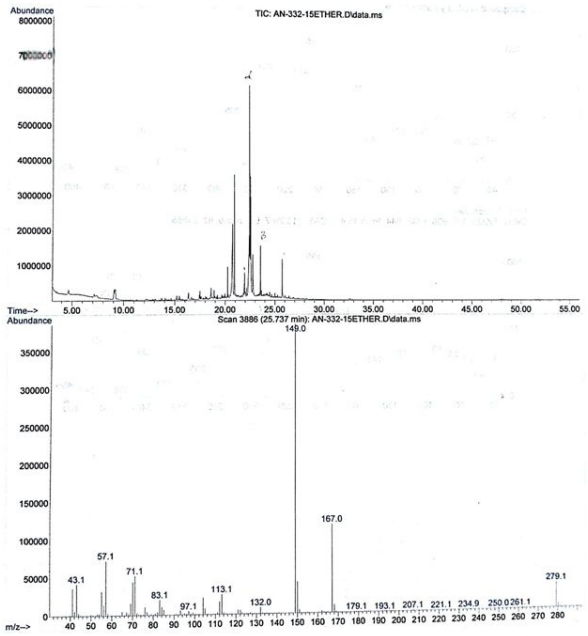

Figure 1: Gas chromatograph of Endosulfan.

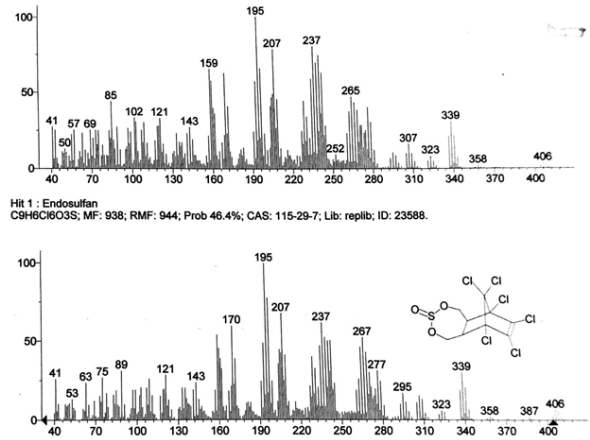

Figure 2: Mass spectrum of Endosulfan

Conclusion:

Endosulfan use is prohibited in India since 2011. Cases of poisoning due to endosulfan showed us that insecticide which is banned also may be the cause of intoxication. The work presented in the paper indicates that the trace amount of endosulfan was extracted from water sample of earthen pot and analyzed by Gas Chromatography-Mass Spectroscopy (GC-MS). The water sample was also found to be ethyl alcohol and petroleum hydrocarbons positive. In the present case, Brother-in-law of victim was found to be accused of mixing ethyl alcohol and organochloro insecticide endosulfan (Thiodan) in the drinking water to cause some intentional health hazard to victim and her family. Fortunately, the life of victim and her family members were saved.

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