cryogenic grinding, for hair analysis. The hair is milled to fine powder using the Cryogenic Mill. The fine powder is directly extracted with acidic methanol. The residue evaporated under N_2 stream was derivatized with pentafluoroacetic anhydride and injected into GC/MS with SIM mode. The standard calibration curves for methamphetamine and amphetamine were obtained from the hair powder blanks spiked with methamphetmaine and amphetamine standards. The recoveries of amphetamine and methamphetamine from the spiked hair powder blanks were over 96 %. The correlation coefficients of the standard calibration curves for amphetamine and methamphetamine were over 0.997. Ten hair samples which were already analysed with hair cutting preparations were analyzed with cryogenic grinding preparations and the results were compared with each other. Amphetamine and methamphetamine extraction were much improved by cryogenic grinding. These results showed that extraction of amphetamine and methamphetamine in hair was dependent on the hair sample preparations. Furthermore, the time consumed for sample preparation decreased when the hair sample preparation was done by cryogenic grinding. These all results suggested that this cryogenic grinding could be utilized as one of the useful sample preparations for hair analysis.

[PA4-2] [04/18/2002 (Thr) 14:00 - 17:00 / Hall E]

Gas chromatographic/Mass spectrometric Determination of 2-Chlorobenzylidene malononitrile (CS gas) metabolites, 2-Chlorohippuric acid and 2-Chloromercapturic acid, in Postmortem Specimen, Liver

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There were several kinds of lacrimators, commonly called tear gases or riot control agents, used for incapacitating or dispersing of rioters in the contaminated environment or chemical warfare. The representative and popular lacrimatory agents used are 2-chlorobenzylidine malononitrile (CS gas) and chloroacetophenone (CN gas). The major urinary metabolites of CS in rats were reported to be 2-chlorohippuric acid and 2-chloromercapturic acid. Our main goals are to develop GC/MS analysis methodology of these two metabolites using new derivatizing reagent, trimethylsilyldiazomethane, in postmortem specimen, liver. The liver samples was taken from the postmortems of which the cause of was due to the intoxication of CS. The samples were homogenated and the metabolites under acidic condition were extracted with Isolute C_{18} column. The residues were derivatized with trimethylsilyldiazomethane ($TMSCHN_2$) to methylate the hydroxy grops. These solutions were injected into the GC/MS. To quantitate the concentration of 2-chlorohippuric acid and 2-chloromercapturic acid in samples 168 m/z and 125 m/z were selected, respectively. The concentrations of 2-chlorohippuric acid and 2-chloromercapturic acid in different postmortem specimen were calculated from the standard calibration curve and blank blood and the results were shown in Table 1.
The concentrations of 2-chlorohippuric acid and 2-chloromercapturic acid in the control blood from hospital were 46.3 ng/mL and 7.2 ng/mL, respectively. However, the concentrations of 2-chlorohippuric acid and 2-chloromercapturic acid in postmortem specimen, in m073, m074, and m077 were over 130 ng/mL and over 628 ng/mL, respectively. In case of 2-chloromercapturic acid, the concentrations in postmortem specimen were about 100 times higher than that in normal blood. This results suggested that the dead persons would be intoxicated with C.S. Generally almost all the papers pertaining to 2-chlorohippuric acid and 2-chloromercapturic acid analysis with GC or GC/MS reported that first the hydroxyl groups of 2-chlorohippuric acid and 2-chloromercapturic acid were derivatized with diazomethane. U. langenbeck, et al reported that hippuric acid was most efficiently derivatized with diazomethane among bis (trimethylsilyl)acetamide, bis(trimethylsilyl)trifluoroacetamide, and trimethylphenylammonium hydroxide derivatizing reagents. However, this diazomethane is highly toxic, in situ prepared and should be treated with care and safe. Newly applied methylating reagent, trimethylsilyldiazomethane, was relatively not toxic and very quantitavely reacted with the hydroxyl group of carboxylic acid at room temperature but with same reactivity as diazomethane. Our results suggested that this trimethylsilyldiazomethane was very usfull substituent for diazomethane for methylation of 2-chlorohippuric acid and 2-chloromercapturic acid.

[PA4-3] [04/18/2002 (Thr) 14:00 - 17:00 / Hall E]

Down-regulation of Cytochrome P450 1A1 expression by o,p'-DDT

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