DEVELOPMENT OF A MEPS BASED CLEAN UP FOR THE ANALYSIS OF DIOXINS AND STOCKHOLM CONVENTION POPs

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Introduction

Materials and Methods

Table 1. Detailed information of samples in follow-up MEPS study

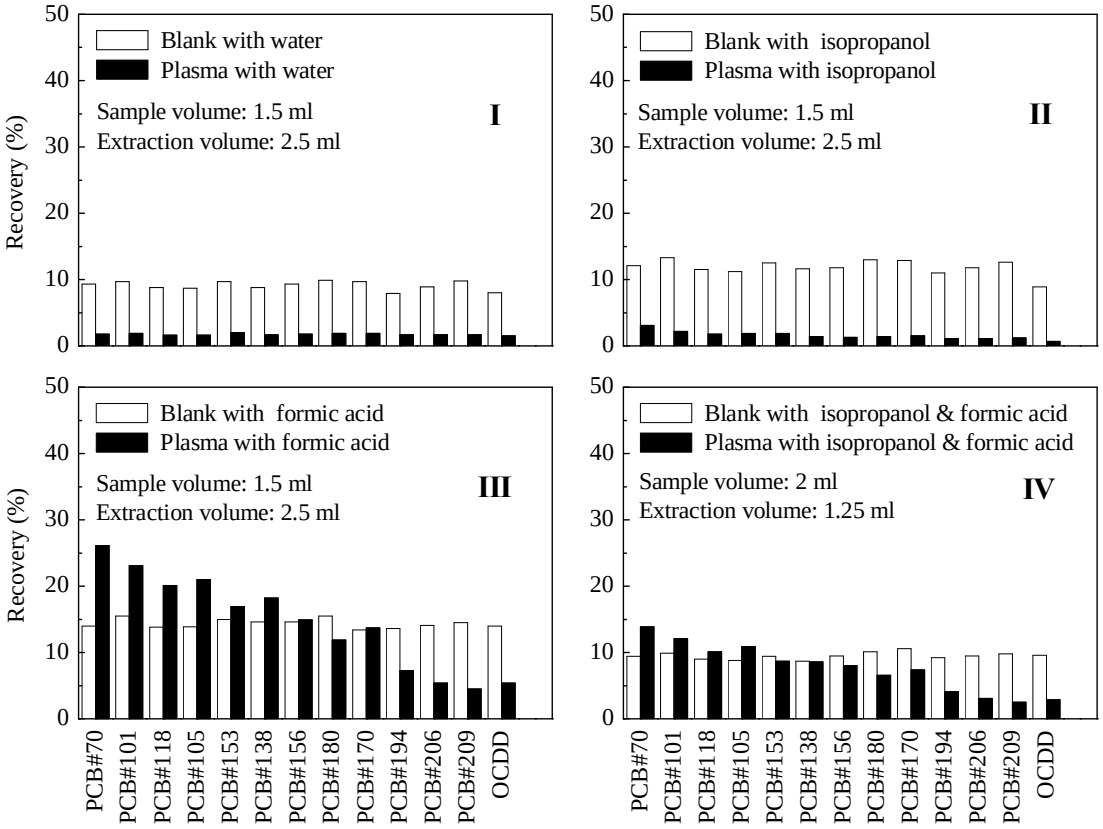
ID	Type	Makeup	Pretreatment	Extraction	Cleanup	Elution
1	Blank	100 εl formic acid	100 εl water	10×250 εl	2×250 εl water	hexane
2	Plasma	100 εl formic acid	100 εl water	10×250 εl	2×250 εl water	hexane
3	Blank	100 εl formic acid	100 εl water	10×250 εl	2×250 εl 5% methanol	hexane
4	Plasma	100 εl formic acid	100 εl water	10×250 εl	2×250 εl 5% methanol	hexane
5	Blank	100 εl formic acid	100 εl water	10×250 εl	2×250 εl water	hexane: DCM
6	Plasma	100 εl formic acid	100 εl water	10×250 εl	2×250 εl water	hexane: DCM
7	Blank	100 εl formic acid	100 εl water	10×250 εl	2×50 εl 5% methanol	hexane: DCM
8	Plasma	100 εl formic acid	100 εl water	10×250 εl	2×250 εl 5% methanol	hexane: DCM
9	Blank	100 εl formic acid	100 εl water	10×250 εl	2×250 εl water	Toluene
10	Plasma	100 εl formic acid	100 εl water	10×250 εl	2×250 εl water	Toluene
11	Blank	100 εl formic acid	100 εl water	10×250 εl	2×250 εl 5% methanol	Toluene
12	Plasma	100 εl formic acid	100 εl water	10×250 εl	2×250 εl 5% methanol	Toluene

Results and discussion

The recoveries of internal standards in blank and plasma samples of various makeup types were shown in Table S1 and Fig. 1. For blank samples, the recoveries of internal standards were quite even (around 10%) irrespective of preparation methods. On the contrary, the plasma samples were quite sensitive to the type of preparation. For Type I and II preparations, the average recovery of internal standards for plasma samples was below 5%, while it reached nearly 15% by using formic acid (Type III). Besides, for both Type III and IV, the internal standards of PCBs for plasma samples decreased with increasing level of bromination.

The relatively low recovery of internal standards obtained in this study might be attributed to following reasons: 1) the ratio of extraction volume to sample volume is too sample (less than 2); 2) the solvent for cleanup and elution (water and hexane, respectively)

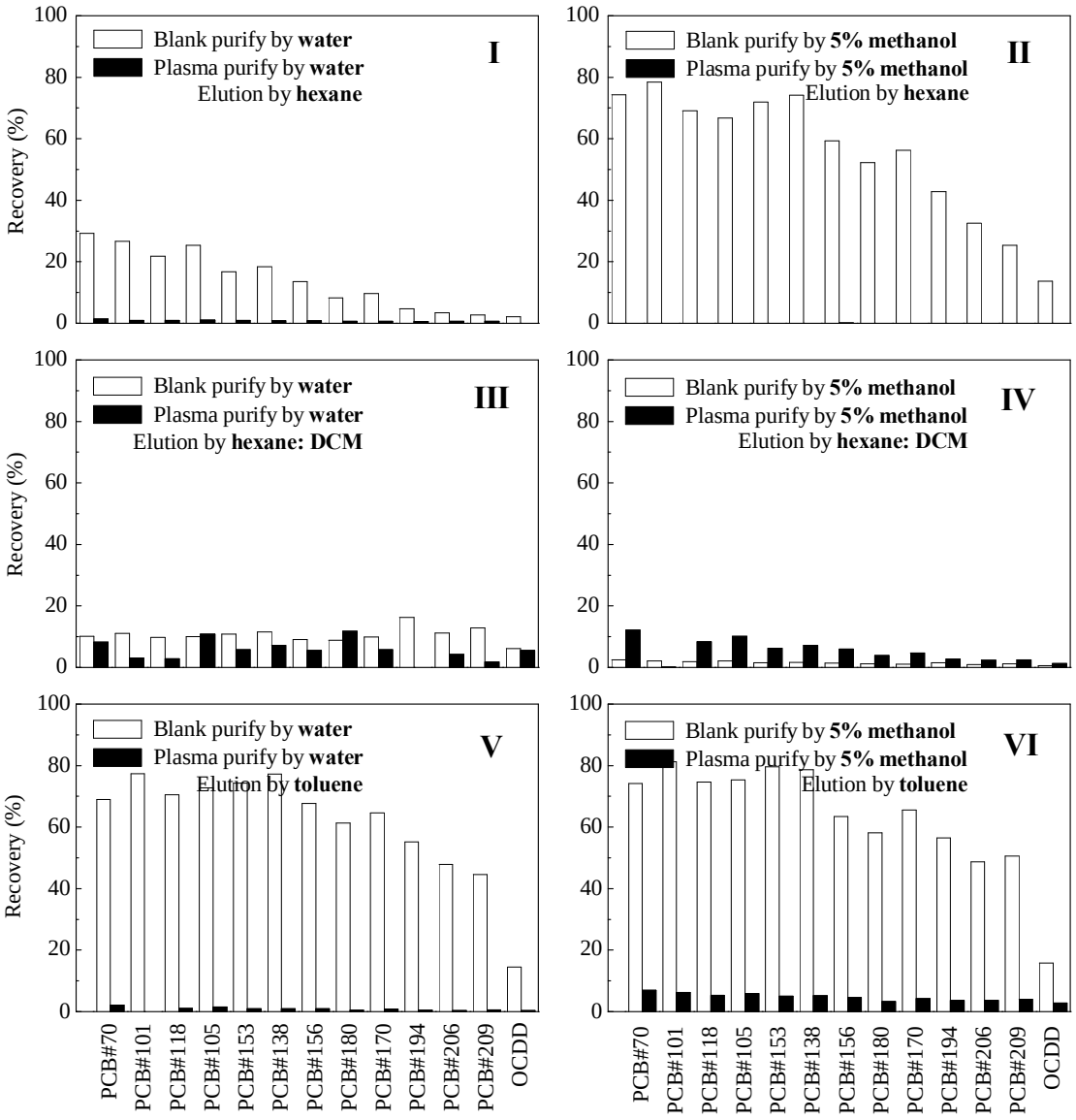
might not be optimal. In order to get better recovery of internal standards for both blank and plasma samples, a follow-up study was performed by increasing the extraction /sample volume ratio and using various cleanup/elution solvents.



The recoveries of internal standards in blank and plasma samples of various cleanup and elution types were listed in Table S2 and were shown in Fig. 1.

For blank samples, quite good results (mean recovery > 50%) were obtained for treatment II, V and VI. Compared with makeup Type III (Fig. 1) in preliminary study, the average recovery of internal standards by treatment II increased 280%. This could be attributed to the increased extraction /sample volume ratio (8.3 vs. 1.7) and the polarity of the cleanup solvent (5% methanol vs. water). Among the 6 treatments, sample purified by 5% methanol and eluted by toluene exhibited best result, with an average recovery of 63.3%.

However, for plasma samples, the recovery of internal standards did not improved much in comparison with the preliminary study. This might be due to the interference of matrix. Besides, the real sample was much more difficult to aspirate compared with blank samples. Nevertheless, the recovery of the internal standards for plasma samples might be increased by adding procedures like silica gel cleanup.



Acknowledgment