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TITLE: REPORT ON ANALYSIS OF PCBs IN HUMAN SERUM

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ABSTRACT: An interim report on the analysis of PCB's in human serum is presented. The problems encountered with the Mound samples and the steps taken to resolve these problems are discussed.

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INTERIN REPORT ON ANALYSIS OF PCB's IN HUMAN SERUM

INTRODUCTION

In September 1981 a report was given to B. Ward and G. Butler on the progress of Monsanto's PCB in human blood analysis program conducted at the Ultratrace Facility of Dayton Laboratory. At that time it was decided to proceed with testing of the method on real samples. It was arranged to acquire blood samples from 10 donors. Samples were received from volunteers at the Mound Facility - MRC in mid-November and the analysis commenced.

RESULTS AND DISCUSSION

The initial analyses using the procedure outlined in St. Louis were completed by the end of November. For quality control purposes, spikes were conducted using a pooled human serum which is commercially available that was then fortified with PCB's. The results of these analyses are shown in Table 1. The average levels found compare reasonably with previously reported human serum levels of 17 ppb (See Journal of American Medical Association 245, 2505 (1981)). The low recoveries were thus attributed to a poor spiking procedure and an alternate method was attempted. This involved spiking the serum using a solution of PCBs in acetone. Four of the Mound samples were reprocessed using this technique.

Initially it appeared that this spiking technique was sucessful but closer evaluation of the data indicated that no more than one half of the PCB's were observed. Thus a series of experiments were run to test the transfer efficiency of PCBs in serum. These indicated that total recovery of all PCB's was on the order of 50%. The results also indicated that any PCB losses due to transfer of serum to the extraction solvent was less than 10%. With the high extraction efficiencies of PCB's into hexane we thus suspected that the losses could be a problem related to the 10% $AgNO_3$-silica gel column.

A complete elution study was then conducted on silica prepared within 3 weeks of the study. A standard PCB mixture was placed on the head of the column and 50 mL of hexane was passed through the column collecting 5 mL fractions. Capillary GC-EC indicated incomplete elution in the first 30 mL of hexane which was the quantity of hexane that had been collected in the Mound studies. This volume was based on the early column elution studies that were analyzed by packed column GC. The more definitive capillary

TABLE 1 MOUND DONOR RESULTS IN ppb

Donor	Sample	Replicate	Average	Monitrol Spike Recovery
1	11.4	a	11.4	b
2	7.9	5.4	6.7	44%
3	4.5	7.2	5.9	32%
4	36.9	12.4	24.7	b
5	9.0	9.4	9.2	14%
6	16.0	15.6	15.8	45%
7	14.8	14.5	14.7	4%
8	10.0	5.8	7.9	6%
9	20.4	17.3	18.8	5%
10[c]	2.7	3.9	3.3	44%

[a]Sample contaminated

[b]No recovery calculated as Average concentration is greater than Spiked sample total PCB concentration

[c]Used experimental vacutainer, original donor 10 sample was lost due to breakage

analysis showed a clear fractionation pattern of PCBs on the liquid adsorption chromatography column. It showed that the higher chlorinated biphenyls are eluted off the column first and subsequently elution follows through the sequentially lower chlorinated species. This work was repeated with a freshly prepared batch of $AgNO_3$-silica gel. The new batch was prepared in a very strict manner controlling conditions to more accurately follow the published procedures instead of the modified procedures that had been used earlier in the method development. The results of this study showed that all PCB's were eluted off the column by 25 mL of hexane with recoveries of 85%. This study also showed that the fractionation of PCB's followed the same pattern as the previous study, but total elution occurred in a smaller total volume more in line with what had been observed in the initial elution studies. Samples of each of these batches of silica gel are being kept to conduct a storage study.

With this information the chromatograms from the Mound donor samples were re-examined. In particular we examined the acetone spiked samples. They show that the low recoveries are accounted for in the front half of the chromatograms which show the absence of the lower chlorinated PCB's. Unfortunately no sample of the $AgNO_3$-silica gel used in that study remains to perform an elution study on that batch. We can conclude that the low recoveries are due to incomplete column elution of PCBs with 30 mL of eluant. In order to prevent this problem from reoccurring, we have now instituted some additional quality assurance steps.

The first QA procedure we are instigating is to perform a complete elution study of each new batch of $AgNO_3$-silica gel. We will also now carry through with each work-up procedure a simulated spike that will be applied directly to the head of the column and column recovery will be calculated. Currently we are conducting a time study to discover the elution characteristic changes of the silica with time. This will enable us to ensure a greater degree of column reproducibility.

The PCB in human serum method is now undergoing further testing using blood from three donors here at the Dayton Lab. This will give us a better idea of the status of the method and whether or not it is ready to resume field testing.