Statistics
Special Study 72-5
Job No. 8505016

# ROUND ROBIN STUDY - PCB IN WATER

## INTRODUCTION

In order to establish the accuracy and precision in the method used for estimating PCB content in the environment, a round robin testing experiment was set up. Samples were prepared by spiking water with PCB's at the level of about 500 ppb. Individual preparations included blanks (no PCB). Aroclor 1242 only, Aroclor 1254 only, Aroclor 1260 only, all 50-50 two component mixtures, and a 1/3, 1/3, 1/3 preparation containing all three Aroclors. Six samples of each mixture were prepared, and three of each sent to the W. G. Krummrich Plant Lab, and three tested by Applied Sciences (cf Table I). Tables II and III show the results obtained by the laboratories.

Each result, expressed in ppb of the individual components was converted to % of the total PCB added. Analysis of variance was run on all the data taken together (Table IV) and on each lab independently. Two obviously defective samples were deleted from the Krummrich data.

## CONCLUSIONS

1. There was a significant difference between the laboratories in per cent of total PCB found. Significance - 99.0%.

2. There was a significant difference between the laboratories in variety of results. Significance - 99.9%.

3. The 95% confidence limits for individual results were as follows:

| | |
|---|---|
| Either Lab | 87.5 ± 55.2% |
| WGK | 81.3 ± 46.4% |
| Applied Sciences | 93.7 ± 19.2% |

4. The repeatability for the individual labs, based on repeat tests of samples having identical prepared compositions is as follows:

| | |
|---|---|
| WGK | ±42.0% |
| Applied Sciences | ±11.6% |

5. In general, Applied Sciences comes closer to properly identifying the individual Aroclors, with one exception. Applied Sciences found Aroclor 1254 in a mixture of Aroclor 1242 and Aroclor 1260. See below:

| Mixture | Present | Found WGK | Found A/S |
|---|---|---|---|
| 1 | 1242 | 1242,(1254) | 1242 |
| 2 | 1242,1254 | 1242,1254,(1260) | 1242,1254 |
| 3 | 1242,1254,1260 | 1242,( ),1260 | 1242,1254,1260 |
| 4 | 1254 | 1254,(1260) | 1254 |
| 5 | 1242,1260 | 1242,1260 | 1242,(1254),1260 |
| 6 | 1260 | (1248),1260 | 1260 |
| 7 | 1254,1260 | (1248),1254,1260 | 1254,1260 |

Numbers in parentheses indicate misidentifications.

DSW 346559

Apparently the technique is not yet sufficiently developed to permit accurate identification of mixtures of Aroclors.

6. A previous study, based on fewer data points gave the following confidence limits (95%):

Total PCB 113.8 ± 58.7% (3 laboratories participating)

Confidence limits of means for individual labs:

| | |
|---|---|
| Applied Sciences | 99.3 ± 48.8% |
| WGK | 133.6 ± 48.8% |
| Anniston | 118.0 ± 48.8% |

Note that the previous results tended to show higher recovery than the present set.

DISCUSSION

Prior to the analysis of variance, two test results from W. G. Krummrich were discarded. These were samples B26 and B29 on Table II. It was felt that there might have been an error in the preparation of these samples.

The Applied Sciences laboratory had significantly more precise results on samples having the same composition than on overall % recovery over all samples. This lower variability did not show up on the Krummrich tests (WGK showed the same variability within compositions as between compositions). It is possible that the improved precision for Applied Sciences was due to similarities in technique (dilution, etc.) on samples having identical compositions.

Details of the statistical analyses on this and the previous study are available.

db

Monsanto Industrial Chemicals Co.
Applied Sciences
St. Louis, Missouri

3/72 - J. D. Hinchen, R. E. Keller



STLCOPCB4084582

TABLE 2

# Round Robin Water Samples - Spike Level

| Sample | Aroclor | Level (ppb) |
|---|---|---|
| A-1<br>B-23<br>C-45 | None | Blank |
| A-2, 9, 16<br>B-24, 31, 38<br>C-46, 53, 60 | 1242 | 502 |
| A-3, 10, 17<br>B-25, 32, 39<br>C-47, 54, 61 | 1242<br>1254 | 251<br>250 |
| A-4, 11, 18<br>B-26, 33, 40<br>C-48, 55, 62 | 1242<br>1254<br>1260 | 251<br>250<br>250 |
| A-5, 12, 19<br>B-27, 34, 41<br>C-49, 56, 63 | 1254 | 501 |
| A-6, 13, 20<br>B-28, 35, 42<br>C-50, 57, 64 | 1242<br>1260 | 251<br>250 |
| A-7, 14, 21<br>B-29, 36, 43<br>C-51, 58, 65 | 1260 | 501 |
| A-8, 15, 22<br>B-30, 37, 44<br>C-52, 59, 66 | 1254<br>1260 | 250<br>250 |

A - Applied Sciences
B - WGK
C - Amiston.

DSW 346561

TABLE II

Round Robin Water Samples — WGK

| Sample | Aroclor & Level (ppb) | | | |
|---|---|---|---|---|
| B-23 | 1242 2<br>1254 1 | | | |
| B-24, 31, 38 | 1242 - 298<br>1254 - 64 | 1242 - 222 | 1242 - 273 | 280<br>- 13 |
| B-25, 32, 39 | 1242 - 314<br>1254 - 280 | 1242 - 170<br>1254 - 168 | 1242 - 128<br>1260 - 245 | 435<br>- 13% |
| B-26, 33, 40 | 1242 - 1<br>1254 - 1 | 1242 - 171<br>1260 - 487 | 1242 - 144<br>1260 - 477 | 639<br>- 15% |
| B-27, 34, 41 | 1254 - 448 | 1254 - 232 | 1260 - 462 | 381<br>- 23% |
| B-28, 35, 42 | 1242 - 141<br>1260 - 345 | 1242 - 96<br>1260 - 252 | 1242 - 133<br>1260 - 317 | 428<br>- 12% |
| B-29, 36, 43 | 1260 - 100 | 1260 - 546 | 1248 - 8<br>1260 - 530 | 394<br>- 21% |
| B-30, 37, 44 | 1248 - 120<br>1260 - 96 | 1254 - 285 | 1248 - 50<br>1260 - 513 | 354<br>- 29% |

DSW 346562

Round Robin Water Samples - Applied Sciences

| Sample | Aroclor & Level (ppb) | | | |
|---|---|---|---|---|
| A-1 | 1242 - 15 | | | |
| A-2, 9, 16 | 1242 - 474 | 1242 - 507 | 1242 - 548 | 510<br>+2% |
| A-3, 10, 17 | 1242 - 165<br>1254 - 287 | 1242 - 165<br>1254 - 290 | 1242 - 169<br>1254 - 279 | 452<br>-9% |
| A-4, 11, 18 | 1242 - 160<br>1254 - 253<br>1260 - 129 | 1242 - 172<br>1254 - 281<br>1260 - 216 | 1242 - 227<br>1254 - 272<br>1260 - 215 | 642<br>-11% |
| A-5, 12, 19 | 1254 - 541 | 1254 - 524 | 1254 - 550 | 539<br>+8% |
| A-6, 13, 20 | 1242 - 117<br>1254 - 108<br>1260 - 162 | 1242 - 140<br>1254 - 111<br>1260 - 186 | 1242 - 153<br>1254 - 145<br>1260 - 157 | 436<br>-13% |
| A-7, 14, 21 | 1260 - 436 | 1260 - 430 | 1260 - 455 | 440<br>-12% |
| A-8, 15, 22 | 1254 - 282<br>1260 - 200 | 1254 - 300<br>1260 - 208 | 1254 - 262<br>1260 - 210 | 487<br>-3% |

DSW 346563

TABLE IV

ROUND ROBIN WATER SAMPLES

TOTAL PCB - % OF SPIKE LEVEL

| Composition No. | W. G. Krummrich | Applied Sciences |
|---|---|---|
| 1 | 72.0 | 94.3 |
| | 44.1 | 100.9 |
| | 54.3 | 109.1 |
| 2 | 118.2 | 89.9 |
| | 74.2 | 90.5 |
| | 72.2 | 89.2 |
| 3 | | 72.1 |
| | 87.5 | 89.0 |
| | 82.6 | 95.0 |
| 4 | 89.2 | 107.6 |
| | 46.1 | 104.3 |
| | 91.9 | 109.5 |
| 5 | 96.7 | 83.0 |
| | 69.3 | 87.0 |
| | 89.6 | 90.5 |
| 6 | | 86.8 |
| | 108.7 | 85.6 |
| | 107.1 | 90.5 |
| 7 | 43.2 | 96.4 |
| | 57.0 | 101.6 |
| | 112.6 | 94.4 |

DSW 346564