

Q. Now just tell us what in the regular course of your business when you see this chromatogram going through, Chromatogram 8, what do you do, what do you tell your client, how do you tell your client? You call him up? You write him a letter? What do you do?

A. We do it either way, and sometimes both ways.

Q. And—

A. Call him first and then tell them that we think we see something that is—could cause problems in the analysis. And if they asked for confirmation in writing, of course we would send it to them.

Q. And what would you send them in writing?

A. We would send them in writing the values obtained as DDT, DDD, and DDE from these two charts compared to standards back here in the previous one, two, three, four charts.

Q. And you would send them with each of these figures separate, DDE, DDD, and DDT, right?

A. Yes, we would send them that, and on the report we would certainly mark something to the effect that the DDT and DDD in our estimation could be in error.

Q. All right, this is what I'm trying to get on the record. Do you have a standard qualification that you put on your analytical report where this occasion arises in the \$25 exam?

A. We do not have a standard qualification, no.

Q. You have gotten thousands of requests for DDT residue analyses, right?

A. Right, yes.

Q. And where you or Mr. Hughes sees this suspicious hump in one of these chromatograms, you mean to say that you don't have a standard warning or qualification or reservation clause that you put in your analytical report?

A. No, there's not been that number of samples to have warranted it since we have determined that actually compounds such as PCB's are present.

Q. And this is since the fall of '67?

A. That's right.

Q. Roughly how many analyses have you done on DDT since then, roughly? For DDT and its metabolites by request since the fall of 1967 when you discovered the PCBs?

EXAMINER VAN SUSTEREN: He personally?

MR. YANNACONE: No, his department.

EXAMINER VAN SUSTEREN: All right.

WITNESS: Seven to eight thousand.

Q. And you haven't had enough PCB interference in those to warrant preparing a standard disclaimer clause or reservation clause, have you?

A. Not in those particular samples.

Q. Now of those, do you recall what percentage would be environmental; the same, roughly 60 percent?

A. Roughly 60 percent.

Q. So in those 4,000 environmental samples you haven't found enough PCB interference to warrant a standard disclaimer or reservation to your client?

A. That is correct.

Q. All right. Fine. Now, let's assume that you do find this suspicious peak, and you were going to write a letter to your client, this client, what would you tell them in the letter in the regular course of your business?

Q. What do you tell him in the regular course of your business?

A. We suggest that the sample—portion of the sample, depending on time, if we still have the original extract and can work with it at that time, of actually doing the initial extract, we would recommend that the extract be hydrolyzed, and subsequently analyzed for whatever may show up on the gas chromatograph.

If the time lapse is such that there would be a question in doing the extract, we would start with a whole new sample, if we would have it or they could submit it; and in this case we would prepare a new extract, reinject it into the chromatograph to make sure that we see the same pattern that we saw before.

HYDROLYZE THE SAMPLE!

Q. Stop a minute. The same pattern up to the end of the—in this ; case, chromatogram No. 8, the \$25 chromatogram?

A. That is correct. And then we would hydrolyze a portion of this: extract, reinject, and give them a best estimate that we could from that of what we then see.

Q. Now the hydrolysis—Which I think you said was 11 alkaline hydrolysis?

A. Yes. This is right directly out of the FDA residue manual for hydrolyzing extracts containing DDT and DDD.

Q. Yesterday I think you told us that you hydrolyzed with sodium, hydroxide. Doesn't the FDA manual specify potassium hydroxide?

A. I—to this moment I would not know for sure. I would guess that it certainly could.

Q. You're sure you do it with sodium hydroxide, though?

A. No, sir, I'm not sure that I do it with sodium hydroxide. We do it according to the FDA manual.

Q. Now when did you start doing this particular process, this alkaline hydrolysis process?

A. I would say we started sometime in the spring of 1968.

Q. In other words, it's your testimony, sworn testimony that were doing this hydrolysis on clients' samples prior to January of 1967?

A. To the best of my knowledge, we started on our own; we certainly were working with clients' samples; but we were working on our own without pay for this work to—only to learn for ourselves what we were—thought we had been seeing. To the best of my knowledge, we were using an alkaline hydrolysis at that time.

Q. What time?

A. It was—we started with the two procedures which were in the literature at that time. One was that you could add alkali and change the DDT and DDD to compounds which would not then appear at the same retention times as DDT and DDD.

Q. Among them, DDE?

A. This would be one of the compounds which would be formed. The other procedure at that time was the nitration step, which I mentioned yesterday. We actually started with it first, because our understanding was that it should remove from the chromatogram all three, DDT, DDD, and DDE.

Q. Well, Mr. Coon, doesn't it make a difference in that nitration procedure whether you use fuming nitric acid at 100 degrees, or whether you use just plain nitric acid at zero degrees?

A. It would make a great deal of difference.

Q. Which one did you use?

A. We used probably those two and everything in between.

Q. Now with respect to the alkaline hydrolysis and saponification technique, do you know Robert Risebrough?

A. Yes, I do.

Q. You have met Dr. Risebrough, haven't you?

A. Yes, I have.

Q. You have discussed analytical chemical procedures, gas chromatographic analysis for pesticide residues and polychlorinated biphenyl residues with him, haven't you?

A. I have.

Q. And you have discussed them with Dr. Wurster, haven't you?

A. Yes, I have.

Q. And you have discussed with Dr. Hickey, haven't you?

A. Yes.

Q. You have discussed them with Dr. Lucille Stickel, haven't you?

A. Yes, I have.

Q. And you discussed them with all four of them during January of 1969, didn't you?

A. That would be the approximate date.

Q. Didn't you at that time make the statement that you had not yet begun to saponify your samples as a check for polychlorinated biphenyls?

A. I wouldn't remember.

Q. Now when you apply the technique of alkaline hydrolysis or saponification, what happens to the various compounds in the residue, if you know? Chemically?

A. Well, the two in question that we were anxious to find out something about were DDT and DDD. DDT will be converted to DDE. And DDD is converted to DDE.

Q. In other words, then what you have effectively done is metabolized the original mixture, much the same way an animal would, or human being, right?

A. To some degree, yes.

Q. Because the DDT goes through certain changes and becomes DDE, and the DDD goes to DDE, right?

A. Yes.

Q. Now let's look at Chromatogram 9 and Chromatogram 10. In Chromatogram 9 I think we have a five microliter sample, right?

A. Right.

Q. Now there's now an off-scale peak?

A. One peak is considerably off scale.

Q. Now do you have a sample of the retention times, a standard of the retention times of saponified mixtures of the standard solutions you originally standardized this chart with?

A. Not on this chart.

Q. But you know what the retention times of the standard saponified versions of those standards are, don't you?

A. I know what the relative retention times are, yes.

Q. So now by analysis you can make a very simple broad determination and take your pen and your ruler and see that the big central peak is essentially unchanged in retention time from the standard and that you may conclude from it is DDE, right?

A. Yes, one can conclude that.

Q. And if we do the same thing again and we pick it off the same way, we lay it off the—in chromatogram No. 10 there is another large central peak that extends well beyond mid-scale up to 70 on this particular relative scale, 75, 74, and you can infer from the retention time that that's DDE, right?

A. Yes.

Q. And you did so do it, did you not?

A. We did so do it.

Q. Now the DDE in that chromatogram designated 10, a one microliter sample, is higher than the DDE peak in chromatogram No. 8, where relatively it only goes up to 55 plus a little bit units, and we may conclude that the difference between those two is metabolized DDT, can't we?

A. Yes, we can.

Q. And that's a change of approximately— (Mr. Yannacone does calculating) 25-30 percent, right?

A. 20 percent of what?

Q. The original DDE?

A. Oh. Yes.

Q. Now because you have standardized this and calibrated it, you can compute quantitatively the change in DDE, right?

A. Yes.

Q. You did so do it?

A. We did so do it.

Q. You found that the original one microliter sampler had 185 picograms of DDE and the saponified sample had 275 picograms of DDE, right?

A. Yes.

Q. And in the saponified sample you can't find any DDT peak, can you?

A. Not that can be identified.

Q. Now saponification does what, if anything, to the polychlorinated biphenyls?

A. The polychlorinated biphenyls that we have subjected to alkaline hydrolysis have remained essentially intact, we have not seen any significant change.

Q. In other words then, in chromatogram No. 10—which can be compared, I take it, quantitatively with chromatogram No. 8, is that correct?

A. Yes, it shows the dilution to obtain the DDE peak.

MR. COON: \$50, Mr. Stafford, for the whole show.

MR. STAFFORD: Oh, that's very cheap. I thank you, Mr. Coon.

MR. YANNACONE : All right.

MR. STAFFORD: I know that isn't the bill we will get, but—

MR. YANNACONE : I'm trying.

EXAMINATION BY MR. YANNACONE

Q. Chromatograms 9 and 10, added to Chromatograms 7 and 8, plus the standards, would enable you to report what to your client?

A. They should enable us to report the amounts of DDT and DDD to a reasonable degree of certainty.

Q. Now in chromatogram No. 10, do you want to show us the chlorinated biphenyl peak, if any?

A. In Chromatogram 10, it was never set up to identify it, and we have not so identified it.

Q. Well, did you find any?

A. Well, I would not—I'd hesitate to say on the basis of Chromatogram 10, because we set that particular chromatogram up to determine the content of DDE, and not to look for or try to identify or anything else as far as the PCB's were concerned.

Q. Now assuming your client has paid, I assume it's an extra \$25 for 9 and 10?

A. Approximately, yes.

A. The knowledge that we presently have would have to—we would have to calculate that and report it as DDE.

Q. But you could do it?

A. Yes.

Q. Is there anything in Chromatograms 9 and 10 which would cause you to doubt the reported amount of DDE shown in the sample in Chromatogram 8, computed and calculated in Chromatogram 8?

A. Again, the presence of—the fact that we do identify several peaks of the PCB's, and that if one—if a peak corresponding to the one which we show on our standard chromatogram to be close to DDE, if any portion of that were present, it would interfere with the DDE peak. But direct knowledge on these chromatograms, there is nothing other than the peaks which we do call and identify as 1254.

Q. You could determine with scientific accuracy to a reasonable level of confidence whether or not there was polychlorinated interference with DDE by simply running this solution through another column, couldn't you? And comparing the total set of results?

A. Well, this is one of the things that we are presently doing. We are doing it through several columns to find out exactly whether we do or do not get—I will have to qualify that as to how many of these columns would show that DDE does not show at another—close to another one of the PCB peaks.

Q. Isn't it a fact the identification of DDT and its metabolites by means of gas chromatography and the confirmation of those results and comparison of those results with different kinds of columns is something that has been done and published at length in the literature; isn't that a fact?

A. There has been a great deal of publication in this area.

Q. And you have examined it, haven't you?

A. Yes, sir, I have.

Q. Now, Mr. Coon, if we were to use the method of gas liquid chromatography with electron capture detection simply as a qualitative analytical tool to identify the presence of certain compounds, one of the characteristics identifying a given compound or mixture of compounds would be the wave form profile that you observe, right?

A. Yes.

Q. In other words then, looking at an environmental sample and knowing the relative retention times, as I'm sure you must, you can pretty well estimate what's present qualitatively from the shape and distribution of the wave form, can't you?

A. Yes.

Q. And certain compounds give certain characteristic wave shapes and profiles on given columns, don't they?

A. As a rule this is true.