Q. Now, will you please describe in more or less historic order the methods used for pesticide residue analyses, oh, say since 1942 when DDT was generally used? Now, in doing that please do not give us the details of each procedure but name the various methods and the improvements and generally what have they been?

A. Well, the first analyses that we were aware of from a standpoint of literature was the Schechter-Haller Colorimetric method for DDT. We didn't start using it right away, only because we had no specific request for that particular procedure. Our first exposure was the fly-bioassay which came about due to a request in an entirely different area.

Q. Roughly will you describe the fly-bioassay technique without too much detail?

A. Well, essentially consists of an extraction of—as we conducted at that time—crops and a dean-up of the extract, followed by taking down the extract to a residue to which flies were exposed and after a period of exposure the remaining alive flies were counted. Standards were conducted similarly and results compared to the standards so conducted at the same time.

Q. Now, is this a very accurate scientific procedure for identifying and measuring the residues in death of flies.

A. It was anything but. Actually we had problems because people would come and ask us to analyze for DDT by this procedure. So, therefore, you'd use DDT as a standard and calculate your results to DDT, but also somebody else might request that you analyze for what they said would be dieldrin or heptachlor or anything else present and then we would *set* up similar standards and, of course, then we would calculate our results to whatever the person so required. Unfortunately of course we learned as we went along too that there were products - with the fly-bioassay and there was also

problems of—if the cleanup was not adequate for the particular samples used, the waxes or fats which might ensue in the residue would cause the flies' wings to become moribund and they could not get up and leave the flask and so we'd count them as dead.

Q. And they were really alive?

A. **I** would have to assume that this occurred, yes.

Q. In any event it's not very accurate?

A. No, no, it was not very accurate.

Q. It's still extensively used though is it not, in certain areas?

A. Yes, it is used in a great number of areas in the analysis of canned food where they use it as a very rapid screening method for doing a lot of samples easily.

Q. All right. Now let's get a little more information on the next method which you have already identified as the Schechter-Haller colorimetric method. Can you describe that procedure?

A. Well, again it consisted of extraction of a crop as we initially used it. And cleanup by various *means. Again* taking the residue down—the extract down to a residue, treating it with nitric and sulphuric acids, causing it to be nitrated. The nitrated product could be thereafter reacted with sodium methylate and a blue color would then ensue if DDT were present and speaking of DDT, this color then could be read in a spectrophotometer or suitable colorimeter.

Q. What was the sensitivity of the Schacter-Haller method, that is, how many parts per whatever it would be could you measure with this method?

Q. —of DDT, I'm talking about.

A. Of DDT, the ordinary level was in the neighborhood of a tenth of a part per million. It could be extended somewhat lower than that by taking larger samples but

this usually lead to other problems so that the ordinary sensitivity was around a tenth of a part per million.

Q. All right. When was that first generally used by chemists in this country?

A. Well, the initial papers on this by both Mr. Schechter and **Dr. Haller** appeared **in 1943,**1944, and 1945, so I would assume **that** chemists had started using it back in those days.

Q. Now, was that technique superseded by more accurate techniques?

A. **I** wouldn't necessarily say that it was more accurate techniques but it was superseded by other techniques which could **analyze for** more pesticides. . . . Multiple detection analyses.

Q. And this was a great advantage was it, over the old methods?

A. Well, it *was* an advantage **if one** was looking for more than **one** pesticide or **if** one was looking at an unknown —an unknown being something that could contain any pesticide if any were present and so it was a great advantage to the chemist to be able to use a procedure which would analyze for more than **one** thing.

Q. All right. Will you identify those methods, please, Mr. Coon?

A. Well, the initial one that we were exposed to of course was **the** paper chromatographic procedure.

Q. Will you explain that briefly, please?

A. Well, this consisted again of a cleanup, an extraction cleanup and after passing through a column a portion of the cleaned-up extract was placed on a sheet of filter paper which in **most** instances was cut to an eight-by-eight *square.* After the extract had dried off, the paper was placed in a chromatographic tank which contained mixed solvents. The solvents passed up the paper by capillary action and of course passed through the, spot

containing the suspected residues. The residues would move within the solvent and would separate so that they would not all show up at one spot After the chromatogram had developed to the point of where the solvents had reached a predesignated spot near the top of the paper the paper was removed from the tank, dried and then sprayed with a chromogenic reagent consisting of silver nitrate and two phenoxyethanol. This was in the early 1960's. Dr. Mill's paper as an official procedure for paper chromatography appeared in 1960 or 1961.

Q.. Is this Dr. T. A. Mills of the Food and Drug Administration?

A. Yes, that is.

Q. Now, what is thin layer chromatography, is that a variant of the paper chromatography?

A. It's just a variation of the paper chromatogram, substituting for paper a glass plate which has been coated with an absorbent.

Q. Now, what was the next major development in measurement of residues as far as procedures are concerned?

A. Well, the next one that we used for this multiple detection was gas chromatography.

Q. Before we get to the gas chromatography, which we may be referring to as GC, will you tell us the sensitivity of the paper and the thin layer methods which you have described?

A. Well, they were in the same relative range of the colorimetric procedure for DDT. With care one could detect a tenth of a part per million of crops; as it concerned milk and butter products, the best that one could do was in the range of a part per million.

Q. All right, now, the present method is gas—

A. Chromatography.

EXAMINER VAN SUSTEREN: We are going to call it GC from now on.

MR. YANNACONE: Is it GC or GLC?

WITNESS: Well, I would say that it could be either. The common way we describe it is GLC.

Q. All right, GLC. Will you describe that method, please?

A. Well, I would have to say that the extraction cleanup and subsequent passage through a column was essentially the same, that we had used in the past except that it was extended to the extent that we used much more refined chemicals, solvents, care in cleaning; in other words, the procedure itself demanded much more from the chemist than some of the preceding procedures.

Q. Now, before you describe it further, will you tell us the sensitivity of this method as contrasted to the prior method you have described?

A. Well, this method could easily detect in products containing low fat, low waxes, something in the neighborhood of one part per million. But it was ordinarily—is ordinarily used for residues of .005 to one part per million.

A. I said parts per million. I shouldn't go back and forth. It'd be better if I use one term. Speaking of gas chromatography, it might be better if we used a part per billion.

Q. Now, when you use that scale then, describe the sensitivity of this instrument and these procedures?

A. Yes, it is in the range of a part per billion, depending upon *the* material which you are working, to something in the neighborhood of a part per million, depending on sample size and the samples themselves.

Q. Can you describe how this instrument works and just the mechanics of working it?

A. Yes, it essentially consists of a source of carrier gas, a port for sample entry, a column attached to the port; a detector at the end of the column for measuring the compounds as they peel off the column, an amplifier to enhance the signal from the detector, and a recorder to put it on record.

Q. What sort of a recorder is used? How does it actually come out visually, the record of this analysis?

A. Well, it has a paper as we use it, a ten-inch paper on which **a** pen records the chemicals as they emerge as a peak.

MR. YANNACONE: Excuse me, Mr. Stafford, could we stop a minute. Is this a standard X-Y recorder?

WITNESS: Yes, pretty much so.