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BEFORE THE

ENVIRONMENTAL PROTECTION AGENCY

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AD HOC MEETING

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COMMON CONCERNS ON ANALYTICAL METHODS

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Waterside Mall
West Tower, Room 908
401 M Street, S.W.
Washington, D.C.

January 5, 1981

The above entitled meeting convened, pursuant to notice, at 1:48 p.m., Mr. Courtney Riordan, presiding official.

APPEARANCES:

On behalf of the Environmental Protection Agency:

COURTNEY RIORDAN, OMSQA

RICHARD SCHECHTER, Office of General Counsel

ROBERT MEDZ, OMSQA/WWMMRD

JAMES LICHTENBERG, EMSL-CI

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ROBERT L. BOOTH, EMSL-CI

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On behalf of the Chemical Manufacturers' Association:

RON KAGEL, Dow Chemical Co.

WILLIAM G. KROCHTA, PPG Industries, Inc.

JAMES E. NORRIS, CIBA-GEIGY

DAVID R. NARRS, SOHIO

DONALD J. DAHN, Monsanto

JUDY SHAW, API

JAMES S. SMITH, Allied Chemical

JANET MATEY, CMA

P R O C E E D I N G S

MR. RIORDAN: On behalf of EPA, I would like to call this meeting to order.

My name is Courtney Riordan. I am Deputy Assistant Administrator for Monitoring Systems and Quality Assurance in the Office of Research and Development at EPA.

The purpose of this meeting is for members of the research arm of EPA to meet with representatives from industry and academia to discuss the technical merit of the proposed 304(h) methods which were initially promulgated as proposed in December of 1979 and fully commented on during the last year and are now in the process of review for final promulgation.

I would like to turn the meeting over briefly to Richard Schechter from the Office of General Counsel who will have a few comments regarding some documentation that has been made available prior to the meeting. I would like to then open the meeting up to Ron Kagel to make some introductory comments as well.

MR. SCHECHTER: I am Richard Schechter. I just have a few procedural comments, one of which is the comment period on this proposed rulemaking was closed and now we are for practical purposes reopening it for a short period of time to have this meeting and then

afterwards to see if there are any public comments to this meeting.

The purpose of the meeting is discussion of the technical aspects of the 304(h) proposed rulemaking. We would like to limit the discussion to those issues.

Staff of the Agency did make available a document which indicates what our rulemaking may be in the future, but it is totally unofficial in nature. The only official document concerning the rulemaking is the proposed rulemaking which was promulgated months ago.

The document with indicated test methods which was released by staff within the last week or so can only be considered an informal and draft document and not reflect any official Agency position.

Thank you.

MR. RIORDAN: Ron?

MR. KAGEL: I am Ron Kagel with Dow Chemical.

Just to bring everybody up to speed on this meeting, let me go into a little background on how this occurred.

Apparently Bob Finnegan of Finnegan Instruments had some discussions with Dick Dowd regarding the GCMS methodology versus the GC methodology. Following those discussions, Dick Dowd talked to me to get our input.

It appeared to me at that time that higher

management in R&D/EPA was unaware of industry's concerns and problems with the Section 304(h) methodology. Consequently after that, Courtney and I had discussions. I outlined our concerns and objections and offered to arrange this meeting and try to air some of these matters also involving CMA, API and we hope the UWAG people will join us shortly.

I think our concerns fall into three primary categories and I would like to run through these quickly; the GC methodology we feel is not appropriate for compliance monitoring of a complex facility. We have voiced these objections as early as 1974. We did so again in our comments last year.

The second point is there appears to be some confusion right now on what constitutes a 304(h) method.

At the Norfolk conference in January of 1980, Dean Neptune, Effluent Guidelines Division, declared that the methods being used to propose effluent guidelines were equivalent to the 304(h) methods.

We have seen no evidence this is true. There are methods other than the proposed 304(h) methods that were and still are being used to generate the effluent guidelines. There appears to be some rif between Effluent Guidelines, the R&D Branch and Enforcement. I wrote a letter to Bob Schaffer last year suggesting

that perhaps the three of us could sit down, R&D, Enforcement and Effluent Guidelines, and resolve some of these problems together with CMA and other industrial representatives. Perhaps we can get into that today; perhaps that is beyond the scope of this meeting. I think it is something that ought to be considered in the future because I do not believe we need more than one set of 304(h) methods floating around.

CMA has has consistently maintained that 304(h) methodology should be validated and that quality assurance/quality control procedures should be followed.

We submitted a validation protocol to the Effluent Guidelines Division and the R&D Branch in December of 1975. We also submitted definitions for detection limits, limits of determination, et cetera, to R&D in 1976 and 1977. We have been reinforcing these positions over the intervening years.

The CMA positions that go back to 1975 were the basis for the guidelines on environmental measurements that were published by the ACS in December of 1980. We are concerned that EPA R&D seems to be choosing a path that differs from that taken by the recognized scientific community on these matters.

We feel the 20 lab round robin is a very positive step. Unfortunately we do not have the data

and the benefit of the results of those round robins and we will not have those until long after the methods are promulgated. I think this is contrary to good scientific practice. It also raises and I think unnecessarily so the basis for legal challenges in the future.

We in CMA and I know this is true of API and UWAG, have labored to resolve some of the issues with EPA at the technical level. We are still committed to resolve them at the technical level rather than at the legal level.

I think today, if I understood Dick Dowd correctly, we have the ear of EPA R&D management. I think we should take advantage of that situation.

MR. RIORDAN: When you say GCA is not appropriate for compliance monitoring, could you elaborate on the basis of that statement so we can have an understanding of some of the technical difficulties?

MR. KAGEL: In a complex manufacturing facility, and I will restrict my remarks to a large facility, the nature of the waste in that facility changes, if not day by day, hour by hour. This is primarily because in addition to continuous operations there are batch operations and things come and things go rather quickly.

GC methodology is very sensitive. It is not specific. Interferences from both priority pollutants and non-priority pollutants can obscure portions of the chromatogram, can interfere and lead to erroneous results.

I believe Shell has done some extensive studies between GC mass spec and GC comparing the methods within their own labs, within the contractor lab. Those data were submitted with the CMA comments. I believe they were also submitted with the API comments. They were submitted independently by Shell to the Agency.

They very clearly show there are problems with the GC methods in terms of interferences and erroneously high results.

MR. RIORDAN: What were they using for confirmation, were they using GCMS?

MR. KAGEL: Yes. I think it is true that most of the large companies will use GC mass spec for doing this monitoring rather than GC. In a small location where there are a limited number of products being made, it very well may be the appropriate thing to use GC, provided the GC methods are appropriately GC mass spec based, that is at the beginning, all the peaks in the chromatogram are identified by GC mass spec and if there are any interferences or things that go wrong during the course of a run, then that is backed and reconfirmed by

GC mass spec.

That is probably the biggest problem we see.

MR. RIORDAN: The empirical basis for the question of the GC is the Shell data base that was submitted during the comment period?

MR. KAGEL: It is hard data.

MR. RIORDAN: That is the set of data we could look to, to find inconsistencies with the GC methodology?

MR. KAGEL: That is correct.

MR. MARRS: My name is Dave Marrs with SOHIO. We submitted some data on the LC method which showed not being able to differentiate PNAs overlapping unresolved both priority pollutant and non-priority pollutant isomers. The same is true with the phenols.

MR. RIORDAN: Did you submit that data?

MR. MARRS: Yes, through API.

MR. RIORDAN: Is it my understanding you support the GCMS method as the method to be used for compliance?

MR. KAGEL: As circumstances dictate, yes. We would prefer to use GCMS.

MR. MARRS: I think our objection is certainly GC versus GCMS but none of the methodology so far has been validated to the point that it would stand up

through a concensus method writing association, none of the methods as proposed on December 3rd.

MR. KAGEL: I can cite a couple of numbers out of the Shell data. In the raw waste, GC mass spec versus GC, the Shell GC mass spec ran 2100 mg/l. Radian, an independent contractor, round 1400 mg/l. Radian doing the same analysis by GC found 13,000 mg/l. EPA doing the analysis through MRI by GC found 12,000 and 13,900. There is quite a difference there, a factor of six.

That is one of the things we have a problem with. GC may falsely read high. If it is a compliance situation, you may be in a compliance situation when you really are not because of the methodology.

If the methodology were appropriately validated with precision and accuracy, I think we could eliminate some of these problems.

MR. RIORDAN: Those numbers were based on analysis of a single sample? Was that a split sample?

MR. KAGEL: I will have to read up on that.

MR. RIORDAN: Yes?

MR. NORRIS: Jim Norris with Ciba-Geigy. I think there is one point that needs to be clarified, in the event there is any confusion. The CMA position is as an absolute analytical technique, we think GC mass spec is probably less subject to the sorts of

interference as positive and negative that we see engaged with GC. There should be no confusion that our position with regard to 624 and 625, which were the methods published in the Federal Register on December 3, 1979, we find some deficiencies in those methods. We are not offering them as the standards of the world at this juncture.

If I remember correctly, the Shell work was done by Shell's own GC mass spec technologies and not necessarily calling upon 624 and 625.

MR. RIORDAN: In your comments you also provide recommendations on what represents validation in your mind for a method to be accepted. Is that correct?

MR. KAGEL: That is correct. Essentially it is the same -- this is the article published in December of last year, analytical chemistry, guidelines from the ACS.

What we proposed was a validation scheme very simply 7-7-7, following the N-N-N type scheme or seven is recommended, to determine recovery, precision and accuracy and so forth.

MR. SCHECHTER: Do we have a copy?

MR. KAGEL: That was just published in 1980. I submitted informal copies to the Effluent Guidelines Division. I think I submitted an informal copy to R&D

Cincinnati while it was still in draft.

MR. SCHECHTER: Why don't we submit it so it will be part of the formal record.

MR. KAGEL: All right. We have proposed in the past dealing with validation inputs to that article. Dow input strongly. CMA did input also.

MR. MARRS: In the December 3rd Federal Register, EPA had an appendix on quality assurance on 624 and 625. One of the things we tried to get across in our comments through API was we believed that method validation is distinct from quality assurance, that method validation is the responsibility of the Agency, not the responsibility of the user.

The way that was structured on December 3rd is they had each individual user validating the GCMS methodology. There was no criteria as to what was an acceptable validation.

MR. RIORDAN: What is the distinction between quality assurance and validation?

MR. MARRS: In our thinking, very often in a concensus method, for example, which ASTM would publish, there would be with that method performance criteria. For example, there had been established through interlaboratory testing that the analyst could meet and say I have done this right or I have to do it again

because something is wrong. In the method as published by the Agency, that has not been provided. The analyst is to develop his own confidence levels; not only does that duplicate effort with everybody doing it, it costs about $50,000 per lab to do it. Not only is it expensive but a bad laboratory would have a wider set of control limits. They would not have any reason to know they were doing things poorly and ought to clean their act up a little bit.

MR. KAGEL: If you follow the ACS article on methods validation, the way they have it lined out, this is something you do once on a method for a matrix. A method which is valid for my waste may not be valid for your waste. It may be matrix specific because of interferences. It is something that need be done only once. It is not an ongoing thing that is done every day or every week.

The protocol is laid out. The steps are followed. As long as the protocol is not changed, then the validation should be verified.

MR. RIORDAN: Does the article say it would have to be done for every matrix or is "matrix" defined as an industrial effluent from one plant? Would it be good enough that a matrix represents effluents from the industry?

MR. KAGEL: I do not think you can be that broad with the whole industry. I think it has to be matrix specific.

Part of the problem is in the chemical industry you will find that most of the effluents have high salt contents. These vary from plant to plant. This has a dramatic effect on the recovery in a particular effluent. There are other things there. I think it is well known that priority pollutants constitute probably less than one percent of a total effluent, there are other things there that will interfere.

It is matrix specific.

MR. RIORDAN: Would validation in your terms have to be done on each plant effluent? Would validation be possible?

MR. KAGEL: We differ on this.

MR. MARRS: Because the effluents are more uniform.

MR. KAGEL: Ours are not. I can guarantee you that the effluent in Midland, Michigan is not the same as the effluent of Dow/Texas and so forth. It is dependent on the product mix of the plant.

MR. LASETER: You indicated earlier that will change based on the operations and variations within the plant itself.

MR. KAGEL: That is correct.

MR. LASETER: How would you identify some standard matrix, if you increased the content, I do not know what would happen.

MR. KAGEL: I do not know if you can identify some standard matrix. That is why I think what we are really talking about is a program to identify when that happens, when you have the change. I know it is kind of sticky. It is a tough one to wrestle with.

MR. DAHN: Don Dahn, Monsanto.

I think what we are talking about is a hierachy of method validation as part of the QA/QC program. When you start off and you are developing a new set of methods for use under 304(h), that is something you expect to have broad utility on. You should check under a fairly wide variety of circumstances whether these methods are rugged. If they are found to be rugged under a variety of circumstances, then they are good methods to be published.

There ought to be another set of QA/QC procedures that I validate that method in my own laboratory that shows I can use that method competently as an individual laboratory on some standard matrices and further, I have to have some experiments that tell me that set of methods is working under my own matrix.

Under my day to day operations, I have to have some QA/QC procedures that show something has not gone off, that I have a reasonable expectation that my matrix is being constant.

All of these things have to be balanced against the cost. If you did too many method validation procedures all along the way, you would soon be spending more on method validation than on the experimentation.

You have to have a hierachy within the 304(h) methods before they are published, they ought to have a pretty rigorous method validation. When you get down to what an individual laboratory is doing using that method day to day, it would be much less on any particular sample and therefore less costly.

MR. KAGEL: I think this varies depending on the size of the location. We have been using a set of methods for some time now and they are validated for our waste. Even though we have batch operations up and down, the changes are so slight that it does not really interfere with the methods. When it does, we know it does and we go back to the GC mass spec and find out what is going on.

In a smaller operation which is all batch, that can be a real problem.

The initial method is GC mass spec based.

MR. RIORDAN: Do any of the industries represented here use GC as a routine monitoring device, either for process control or effluent monitoring?

MR. DAHN: Certainly.

MR. RIORDAN: Have you had experience in terms of data that shows this is less sensitive than some other methods or goes out of control more often? Is this because the extremes are fairly constant in terms of chemical composition that you are able to use them?

MR. KAGEL: Sensitivity is not a problem. It is specificity.

MR. DAHN: I think we could produce a wide variety of examples where GC as a method worked very effectively at measuring an effluent for a particular compound in a particular wastestream. I think we could also dig up examples where problems occur with using GC which would be less likely to occur with GC mass spec.

The point I would like to get across is you really have to make sure you have the experiments that tell you whether or not a method is valid before you can use it for compliance monitoring.

MR. RIORDAN: You are saying basically that EPA should do that?

MR. DAHN: It seems to me that EPA's responsibility is to validate the method and show it is

rugged and it works under a wide variety of circumstances. It is the individual laboratory's responsibility to show they can use that method competently and it is also their responsibility to have a record of some quality control and quality assurance data that shows the method has been applied correctly throughout the history of the use of that method.

MR. RIORDAN: In your comments did you recommend what might be a reasonable validation scheme for a 304(h) type broad utility method? Is that in the CMA article?

MR. KAGEL: When did we submit that? December, 1975 was the first time I sent it in and I sent it in again in January, February and March.

I was trying to get a response from someone in EPA and they never did get that. I was forced to write a rather harsh letter a year later about where the response was and we got a response about a year after that.

They were submitted some time ago. I do not know whether we made reference to that in our comments specifically. We did make reference to the letters we had sent off to Dr. Ballinger on detection limits and limits on determination.

MR. DAHN: In the CMA comments, there are

comments on the 20 lab round robin tests as being a beneficial part of such a validation scheme.

MR. LASETER: Are we saying low resolution, is th[illegible] primarily what we are speaking of or are you taking [illegible]igh resolution with selective detectors to begin to minimize some of the interferences using GC as a tool or using GC [illegible] the generic sense. I am having a problem following.

Do the people who are addressing this from industry primarily use low resolution type detection systems?

MR. NORRIS: I have seen good work done with such systems, as long as the matrix remains fairly constant and you have done your homework beforehand to know what a particular RF represents. I have seen the whole shooting match go out the window when a new product would come on line and then the peak would be apparently enhanced and you would get an exceedingly high reading and people could not accept this as a reality. You would go back and do your high resolution and say, this other process had some components in the effluent that dilute at the same time as the peak that we were previously looking at, at the RF.

You really cannot make a blanket statement. We have had experience with both.

MR. KAGEL: I think we are talking primarily about packed column. Some of the methods have specific detectors, others are just general FID. I think we would have to get into specifics.

MR. MARRS: I think that point is more general; regardless of the method, the homework has to be done before it hits the streets.

MR. KROCHTA: I think we need to be more specific because we are using a combination of which you would consider high resolution and slow resolution columns. For example, using less capital area columns which are considered high resolution and some applications, for example, PNAs, the phenols, versus maybe low resolution, on the volatiles.

We are using mass spec in addition to the high resolution tolerance.

I might add we are talking about validation. I do not think this is unique with CMA or API. There was just a recent article in "Environmental Science and Technology" and this article referred to the fact that they were asking EPA for validation of 304(h) methods. It was about a month ago.

MR. RIORDAN: Basically what you are saying is you do have some data based upon the Shell experience which shows -- I guess that was low

resolution gas -- provides results which are inconsistent with that of GCMS results. I guess the GCMS is considered to be in that instant the more accurate measurement. Is that right?

MR. KAGEL: These were the 600 series methods that were applied for tolulene.

MR. RIORDAN: That is basically the data to look at in terms of inconsistency at this point.

MR. KAGEL: That is the best data available at this point in time.

MR. RIORDAN: In terms of method validation, you have a general position which says that EPA should not promulgate a 304(h) method for general application for identifying and measuring pollutants and effluent unless it has gone through a fairly vigorous validation procedure. You define "validation" as being the application of the method to several different waste streams under interlaboratory colaborative conditions.

MR. SCHECHTER: Basically a reflection of the same position that was in the comments.

MR. KAGEL: Yes.

MR. DAHN: My feeling is you should be able to give some direction to the user of that method as to whether that method is applicable to a given circumstance by giving him a set of experiments he should

do before using the method. It may turn out a specific method is not applicable but if it is, then he can use it. That was the point Dave made earlier with the fact that there was not any performance criteria included in the 624 and 625 section.

MR. MARRS: Especially as you get down at the ppb level, uncertainty becomes a major factor. I think there ought to be requirements -- or we think at API, there ought to be some kind of requirements in the method to require the analyst to report not only his results but the uncertainty associated with that result. Twenty-six and 25 ppb may be the same number.

MR. KAGEL: Or 26 and 35 ppb or 26 and 70 may be the same number.

In the letter we submitted some time ago, defining detection limits, we spelled that out very carefully in terms of signal to noise, to make an instrument independent. At that time we said a qualitative result should be 2.5 times signal to noise.

The ACS has adopted a slightly different position since then, that was their original position. They now say it should be three times signal to noise. That is a qualitative number. Yes, it is there, no, it is not there. That is a detection limit.

A quantitative number should be ten times

signal to noise. That is still consistent with the ACS position. Any number between 2.5 and 10 should not be taken as a quantitative number, number that has meaning, but should be taken as an indication it is there and in order to get meaning for that number, it needs to be repeated, to be verified.

That is part of the problem working down by detection limit. We see numbers coming out. We saw this in effluent guidelines' screening phase where the methods were said to have a detection limit of 10 ppb and yet we saw results coming in that two ppb, one-tenth of a ppb, and I do not know what those numbers mean.

MR. RIORDAN: You are saying the method should have a realistic detection limit associated with it?

MR. KAGEL: The method should have a detection limit, yes, a realistic detection limit associated with it. I think what Dave was saying is there should be realistic precision and accuracy associated with the result, such that result will achieve the goals.

MR. MARRS: I think it is a real problem for non-analysts to appreciate what uncertainty is. Engineers do not and lawyers sure do not.

MR. RIORDAN: Can you really say this data is good with 90 percent comfidence?

MR. MARRS: There are two things; it really

depends on how the data is used.

MR. KAGEL: We have run into problems where data out of a data base of 20 points, eight of those points were rejected and a standard of deviation was calculated, it was called a two sigma 95 percent confidence level. That sparked quite a debate at an open meeting.

MR. RIORDAN: In your discussions with Dick, are these basically the points that were covered? Did you cover anything in addition?

MR. KAGEL: I think those were the basic points I covered in our very short discussion. I think there are other points that people may want to bring out.

I wanted to bring up one other point and that was recovery. You go through the exercise now with method 624 and 625 for determining the recovery. Then recovery is not used. The number is reported and the recovery is reported along with the number. I guess I am a little uncertain as to how one makes a decision as to whether that is a good number of not.

Even though it work not to our benefit to have recovery correct numbers, I think there is some argument that recovery correct numbers are probably better than just raw numbers.

If the number is reported with the recovery

and if I get 38 with a recovery of 45 percent, or 38 with a recovery of 85 percent, I do not remember any guidance telling me what is a good number or bad number.

I think that really promotes in the long run bad science. People start reporting numbers with bad recoveries instead of working things over to get good recoveries.

We would like to see all recoveries above 85 percent. That is not always practical.

Some allowance ought to be made for this recovery. Right now it is kind of use of the yardstick. I believe the ACS guidelines do call for recovery correction. This is part of the validation procedure.

MR. LASETER: Even the recovery will change as you start to approach the low end all the way through regardless of the ultimate.

MR. KAGEL: The recovery has to be run at different levels, if that is what you mean. You will trace out a heteroscadastic curve on your recovery and follow the error curve almost perfectly. At very low concentrations, your recovery starts going bananas both ways, plus and minus.

Some of the data we have seen in a preliminary round robin would indicate recoveries jump all the way from 20 percent up to 200 percent.

MR. HARRIS: What level?

MR. KAGEL: I believe that was 100 ppb.

MS. SHAW: Judy Shaw with API. We are doing a project right now with the EPA guidelines people at two oil refineries. We are using the stable label method. We are putting in about 20 stable label compounds. What we are going to do with the data is looking at the stable label spikes, it gives you some indication of the accuracy of 624 and 625. We are just beginning to work with that data.

One of the things is the recovery from one sample to another is different for a particular compound. It is also different between compounds. You might have in one sample 60 percent recovery of benzene and 20 percent of tolulene and in the next sample it might flip flop and be the opposite.

If you look at average recoveries, they are great but the range is terrible.

We are running the extractibles at 100 ppb and the volatiles at 20. That data could be very useful. We only did it up to four weeks. The guidelines people did it for two months, sampling every other day effluent and influent. We can also look at whether or not recovery is a lot different between the effluent which is a pretty messy sample and the influent for

recoveries which is pretty clean.

We are seeing things from 20, if you put in 100, you get between 20 to 150 or more back. There is no way to predict what you are going to get.

MR. LASETER: Is it reproducible with the same split sample? Are you criticizing the method or the variability in the samples you are using?

MS. SHAW: You have one sample that you are analyzing for everything else.

MR. LASETER: If you reanalyze that same sample, is your scatter because you have taken a second influent sample a few minutes later?

MS. SHAW: No. The scatter is day to day within a sample, if you are getting 20 percent recovery on benzene and 80 percent on tolulene and you are not correcting, you are not getting good numbers.

MR. LASETER: If you run that same identical sample again.

MS. SHAW: We have only done that a little bit so we do not know.

MR. LASETER: You have to satisfy that question on scatter before you work on scatter sample to sample. That is why I am not sure what you are saying.

MR. KAGEL: We have a similar program with the Effluent Guidelines Division. We are studying five

plants, the influent and effluent and looking at data. We are not using stable label. We are using method 624 and 625. We will have several data points per plant where there will be an EPA number, a company number and an independent contractor number, not on every plant but on many of them.

MR. RIORDAN: If there are no more comments, I will take under advisement with the people in ORD and the Agency the comments. I assure you we will give them full consideration when we get around to reviewing the package for final rulemaking.

A transcript of this meeting will be made available to you as well as the public. Can we make it available to them so they can edit the transcript?

MR. SCHECHTER: Anyone can obtain a copy of the transcript from the Reporter.

MR. RIORDAN: Before we go public with the transcript, is it possible to give the participants a chance to edit their comments?

MR. SCHECHTER: I do not believe so. It is not a sensitive transcript that would be used in a judicial proceeding.

What we are going to do is make the transcript available and open the comment period for a 20 day period to allow any interested party to comment on the

transcript of the meeting. In addition we are going to make the package of methods that we are working on, the package made available to this group, available, along with the transcript.

If there are any comments about the transcript of the meeting, including corrections, they can be made during that comment period.

MR. KAGEL: Can we get an update on the Cincinnati folks, as to where the methods are, what has happened to the methods? Dr. Hodz, what is the status of the round robins? What is the status of the definition of "detection limits"?

MR. SCHECHTER: We cannot at this particular meeting discuss anything beyond the proposal.

MR. RIORDAN: We are restricted to discussing what was made public in December and are unable to discuss with any single parties or independent parties what we have been doing in the meantime.

MS. MATEY: You are unable to discuss the changes in the methods since the last time they were proposed, although we have a copy of the draft?

MR. SCHECHTER: I think the situation to be precise is only the Administrator can promulgate a regulation. As a consequence, the package that you have is a package that staff is working on. It does not

represent any official position on the part of the Agency. It may or may not be the methods that we actually promulgate.

We are open to discussing that package with that in mind. The Agency has not taken any official position that it is going to promulgate that group of methods.

With that in mind, we would be willing to discuss it.

MS. MATEY: Dr. Medz is familiar with the changes and what has happened since they were initially proposed. If he or someone else could explain to us what the changes are in this draft --

MR. SCHECHTER: I do not think we can do that. That really goes beyond the scope of this discussion.

MR. RIORDAN: We can receive comments on the draft from you.

MS. MATEY: We just got them.

MR. SCHECHTER: Since there is going to be a 20 day comment period, perhaps we should wait and let you look at the document and then submit any written comments.

I think it is a difficult procedural situation. Ordinarily we would not be in this position but it is a very technical rulemaking. A lot

of it is purely a matter of chemistry. Obviously the Agency wants to get the most input from the regulated community as possible.

On the other hand, the situation precisely is other than the proposed rulemaking, the Agency has not made a proposal. As such, the Agency really cannot comment on what other possible rulemakings we might be considering.

MR. RIORDAN: I think you have given comments and I think they require the attention of the policy makers within the Agency to make a decision.

MR. KAGEL: We raised the question of the 20 lab round robin. Could we go into that now?

MR. RIORDAN: The 20 lab round robin, in terms of the earlier comments or in terms of the results?

MR. SCHECHTER: We have no objection to your discussing it. On the other hand, anything that was discussed in the comments will be considered by the Agency.

MS. SHAW: That was not in the comments because the results were not published.

MR. KAGEL: That would be my major comment, the results were not published. We did open the door to say we ought to have those results before the methods

are promulgated. For what good it did, we reserved the right to reopen the comment period ourselves.

MR. DAHN: You mentioned this is a highly technical issue. I guess the concern I have is we may have a set of methods that work fairly well in professional hands and which professional people can recognize when they are not working and do something to modify it and get the right method.

I am concerned when we get to an enforcement situation, the methods have to be rather rigorously followed in order to comply with the conditions of the permit and we may get erroneous results by blindly applying the methods which result in arguments that have enforcement penalties, rather than just technical discussions attached to them.

If at all possible, we would like to have as many of those technical uncertainties ironed out rather than be a result of a dispute between a permitting agency and one of our plants.

MR. RIORDAN: Yes.

MR. KAGEL: We would like to know what the status of the 20 lab round robins was, in terms of promulgation of the methods. Are we going to get a chance to see that data prior to promulgation?

MR. RIORDAN: That is one of the things we are

going to have to consider with that decision.

MR. MARRS: That would be useful from our standpoint to make a good representative hopefully constructive technical comment, if we had a data base to look at. Otherwise we are just shooting from the hip.

MR. SCHECHTER: It would be accurate to say that the Agency's position at the moment is no further information on this proposed rulemaking is going to be released. The Agency could conceivably change its position. At this point, that would be our official position.

MR. KROCHTA: The feeling is not only should that data be available to us but it should be available to EPA for evaluating the methods.

MR. RIORDAN: That is the decision we are going to make, you are basically asking that we do quite a bit more work on validation prior to promulgation of the methods. We have to decide what position to adopt.

MR. KAGEL: I think a good share of the round robins are well along the way now.

MR. MARRS: The draft documents were available last Spring.

MR. RIORDAN: Let's take a brief recess and see if there are any further questions.

(Whereupon, a short recess was held at 2:44 p.m. to 2:52 p.m.)

MR. RIORDAN: Let's reconvene. I wonder if we can summarize and also give everyone the opportunity to ask any additional questions, make any additional statements they feel would be productive to the meeting.

I might start off by trying to summarize the situation as I understand it. My understanding is basically the position the representatives at this meeting have adopted is that EPA should not promulgate the 304(h) methods until we have an adequate validation system, as you have defined it, prior to putting that out for general use in industry, for measuring chemicals and effluents.

We should carry out a validation system which meets criteria. You have provided us with one recommended set of criteria for determining an adequate validation scheme.

MR. MARRS: The Agency has its own also.

MR. RIORDAN: You are talking about the equivalency scheme?

MR. MARRS: Yes.

MR. KAGEL: Are you referring to the good lab practices? What is it, Handbook of Quality Assurance? I think it even proposes the 7-7-7 type validation scheme. That is referred to i the ACS article. There are some guidelines already that exist within the

Agency.

MR. MARRS: Within the scientific community.

MR. KAGEL: I think that adds a lot.

MR. RIORDAN: Would you not agree validation is a flexible term, its design depends upon the use to which you are going to put the data and the time at which you are going to be measuring the data.

MR. KAGEL: Definitely. I think that is pointed out clearly in the ACS article. If you go straight on an N-N-N type validation scheme using Walpole and Myers, you would calculate the number of samples that you would need to properly validate a method, you can come up with numbers like 20-20-20 or 30-30-30 which would be economically infeasible to do.

MR. RIORDAN: The other point I would like to clarify is that you have made the suggestion that GC may not be an appropriate method for measuring constituents and effluents. I think you recognize it could be used, depending upon the wastestream. It could be applicable for use in the GC.

MR. MARRS: My understanding of the 20 lab round robin studies is that data should give us a handle on that.

MR. RIORDAN: As far as the GCMS 624/625, your position is that also needs validation prior to

promulgation, even though it has been used under the conditions that represent adequate validation. Is that correct?

MR. KAGEL: That is correct.

MR. DAHN: I think you can go a little bit beyond that and say you have to do enough experiments to determine what the limitations are, of that methodology and make sure we do not use that for permit purposes in situations where it does not make sense to use it.

MR. RIORDAN: Specifically, you recommend we not promulgate methods until we have the results of the 20 lab collaborative tests available and evaluated, is that another point?

MR. KAGEL: Yes.

MR. RIORDAN: You also made the point that on recovery for the extraction methods, it should be built into the estimate of the concentration.

Another point that was made was in an attempt to build in uncertainty in the methods.

MR. MARRS: Precision and accuracy.

MR. RIORDAN: Precision and accuracy for a method, would it cover your uncertainty?

MR. MARRS: Yes, and they should also be reported.

MR. RIORDAN: As part of the QA?

MR. MARRS: Yes.

MR. RIORDAN: We should report them initially?

MR. MARRS: Anyone who reports a number should.

MR. RIORDAN: Do you do that routinely in your measurements you carry out?

MR. MARRS: We ought to but we do not.

MR. KAGEL: It will vary depending on the application. Sometimes it is done; sometimes it is not. I will tell you the place where it is done most frequently, a result is reported near the detection limit. Then a detection limit is reported.

MR. DAHN: I think the reader has a right to expect if he sees a significant figure placed in an analytical result without an uncertainty attached to it, that the uncertainty is some small number. Frequently we have been reporting numbers where that is not true in a lot of cases, where we are a factor of five or ten off.

MR. KAGEL: Normally it is assumed to be plus or minus ten percent. I think that is a normal rule of thumb. Normally, it might not be that much.

A simple thing like significant figures can be very misleading unless you have precision and accuracy associated with the number; 62.5, if it is plus or minus 40 percent, the .5 does happen.

There is another thing I think we have overcome and that is reporting things at the detection limit, the use of less than. It is a very ambiguous term if you do not detect anything and you record it as being not detected, giving the detection limit in parentheses.

There are people that still will report less than the detection limit. That could mean it was there but it was less than the detection limit or it could mean it was not there at all.

I think we need some strides in resolving those difficulties.

MR. HITES: When you are talking about precision, accuracy, sampling errors is always among the highest. How would that get into the procedures?

MR. KAGEL: There is some reference in the ACS paper.

MR. NORRIS: I think Ron you have hit on what is an important issue. We were talking about taking the methods which EPA proposes to promulgate and apply them to a compliance situation and then we have as of now undefined, variations in the analytics, in the sampling technology. These could be added or subtracted.

We are talking about from the industrial perspective, a live or death situation. Somebody could go to jail. Often times the person who is in the field

representing the Agency or the state regulatory body may not be aware of these uncertainties. To him, it is black and white; you are in compliance or you are not.

It is this which goes to the heart of our concern.

MS. SHAW: After it has been promulgated, I think it has to be clear what the variations are, because it is going to be useful for other things and lots of quality decisions and decisions that are going to involve a lot of expenditures. You are going to have states that are going to be doing state water quality standards and state laboratories measuring ambient water quality and deciding whether or not they have to have a state water quality standard.

They are doing waste load allocations. They are going to do all this with methods which right now are reproducible to plus or minus five times.

MR. RIORDAN: I agree, the method should be appropriate to the ends to which it is going to be put.

MS. SHAW: Once they have been promulgated, we do not think the methods are sufficient right now.

MR. RIORDAN: All right. The validation procedure you recommend would be something that we adopt from the literature or from some variation of the 20 lab collaborative tests or some other good practice

but something more than you have seen so far in the promulgation we have made. That is the sum and substance that I have gotten out of the meeting.

MR. MARRS: Certainly ASTM would be another source.

MS. SHAW: There has not been a round robin on 624 and 625. There is really no interlaboratory data.

MR. KAGEL: I was told there was but that was only a rumor.

MS. SHAW: Nothing was mentioned when they were promulgating it.

MR. MARRS: I have a couple of things. A transcript of this meeting is going to sound pretty negative and I think all of us realize we are working with state-of-the-art methods. It is just going to take a lot of work to take a state-of-the-art method and get it to a point where it can be used routinely at another plant. We have to do our homework right.

I guess that is our position.

Speaking for API, we would certainly be willing to participate and help in any way by providing laboratory services or whatever.

MR. KROCHTA: We mentioned this 20 lab round robin study. The five plant study has a lot of built in sampling procedures to give you interlaboratory

precision as well as intralaboratory precision. The data coming from that study should be extremely useful, not only to us but to EPA as well.

I am not sure; we have three plants completed.

MR. KAGEL: Two to go. That should end up this month or next month. We are anticipating a termination date of May, 1981.

MR. KROCHTA: Do you have accuracy as well as precision on data obtained from that?

MR. KAGEL: We have done replicas. We have done spikes. We will have inter- and intralaboratory variability along with it on a limited basis. There will be a lot of data available and we will make those available.

MR. KROCHTA: It should take into consideration the sampling problem as well, in splitting samples.

MR. RIORDAN: Does anyone from EPA have any questions or any points to be clarified?

MR. MEDZ: Most of the things that have been said have been said in the official comments and received. They will be taken into consideration.

MR. RIORDAN: That is the purpose of the meeting, to simply reaffirm the fact that we are taking these into consideration and the decisions made will reflect deliberation on the points made at this meeting.

We appreciate your coming. Thank you.

(Whereupon, the meeting was adjourned at 3:10 p.m.)

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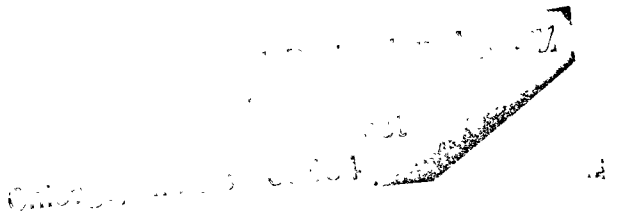
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