

DISCUSSION ON THE PROCESSING OF NUCLEAR EMULSIONS

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Gottstein : In his talk Prof. Teucher showed us several ways in which one can process nuclear emulsions and obtain good results. He admitted that his survey was rather subjective, and we have here sitting by my side four experts on processing who all treat their emulsions in a different manner and who all get their satisfactory results. Now we have seen this morning that this might not be so very surprising. There are some few hundred parameters in the whole processing process which one can vary and nobody really knows what happens if one varies these parameters. What usually happens is that in a particular laboratory people try some method which they think might work and if it works then they stick to it, and they are very careful not to change anything because one never knows what happens when one changes

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Gottstein  
(cont.)

: the procedure. On the other hand the technique is rather lengthy and tedious and sometimes one suspects that there are stages which one can modify in a manner that makes the whole thing shorter and easier. For instance Dr. Dahl-Jensen has processed emulsions completely cutting out the soaking time, introducing the emulsion immediately in the cold developer, and he obtained very good emulsions. He also did away with the stop-bath and it worked. Now of course, if one has a good stack exposed at the CERN-machine, one does not dare to deviate from the well-tested route because one might risk to lose the stack. Therefore very few people have really invested much time into investigating the procedure of processing itself. Therefore, I think, it is very useful if we now ask the experts who are here, why do they process as they do and why don't they do it in a different manner. Particularly I should be very grateful if they would tell us something about their failures because that is something from which one can learn most. Now I will hand over this discussion to the experts and let us start with the rolling-on process. May I ask Dr. Vanderhaeghe what he thinks about the rolling-on procedure which Prof. Teucher described this morning?

Vanderhaeghe : I would like to repeat first what Prof. Teucher said this morning that if there are a few points of disagreement between practices of different laboratories, at least on this side of the Atlantic all routines have the same common root so that their differences are generally not very important. Now, about rolling-on, I think that the over-all procedure described this morning by Prof. Teucher is very near to what we are used to doing at CERN and there is only one point of disagreement, which is quite important.

- Vanderhaeghe (cont.) : It is the way in which you prepare the gelatine. We think that it is much better to heat the gelatine up to about 40° to 50° (it does not matter so much), and then cool it down as quickly as possible. I understand very well the argument of Prof. Teucher that it might be better to cool it down slowly in order to have a very uniform solution but, on the other hand, it is known that keeping gelatine at 50° for too long a time breaks the gelatine chains which weakens its sticking properties. One remark about the time of immersion in the gelatine: if your pellicles are a bit too hard, you have to keep them for more than ten seconds in the gelatine in order to have them soft enough to put them easily on the glass.
- Gottstein : I noted that there is one more difference, at least between the CERN-procedure and what Prof. Teucher described, namely that you use a sponge and really clean the emulsion and the glass rather carefully, don't you?
- Teucher : Whereas Vanderhaeghe just immerses the emulsion, is that true?
- Vanderhaeghe : It is true that we wipe off very gently the gelatine left on the emulsion and we clean the glass.
- Teucher : We only clean the glass, we never touch the emulsion. Let me mention one other point which seems to me rather important during the rolling. Sometimes people want to really see what they are doing and they use the yellow safe-lights in a way which is extremely dangerous. I think it has been proved by now that part of the black deposit at the top of the emulsion is due to yellow safe-light. Do not forget that the Ilford

Teucher  
(cont.)

: safe-lights should be used for indirect lighting and with not more than 15 watt behind one filter. So I would really like to recommend some caution when using the safe-lights; you cannot remove the deposits at the bottom of the emulsion.

Dahl-Jensen

: First, a few words of warning. In the early stages of processing it was believed that if you want to get a very good glue, you should boil your gelatine solution for some time to make it feel really sticky. It is true that it is a better glue, but it is not gelatine and later on the pellicles tend to strip off the glass again. Therefore I can confirm what Vanderhaeghe said, that you should be very careful in melting your gelatine. He quoted the figure 40°C; we actually use only 38°C. Another thing is that the wetting agent you put into your sticking solution could be dangerous. You cannot use every wetting agent you find, but you should choose one of those meant for photography because some sulpho-compounds can act as a sensitizer or desensitizer for the emulsion, and you can therefore get a very undesirable blackening of the surface. Another thing we have forgotten to mention is that the glass-plates prepared by Ilford for sticking onto are rather sensitive. It is recommended to keep them in the Ilford package in a cold place to prevent bacteria from eating the gelatine, because if you form islands on these glass-plates where there is no gelatine, your pellicles will not stick and you will automatically have some bubbles created. Our method of sticking down is somewhat different from what Teucher described. We use the method of painting, i.e. we have a thick rubber mat on which we place the glass which has been cleaned in a dry state using a brush; we clean the pellicle, too, with a dry brush, then we paint the gelatine onto the glass plate. We stick the emulsion first along one line

- Dahl-Jensen (cont.) : and then by hand we press it down into contact with the glass. Using a plastic foil (of 50 - 100  $\mu$  thickness) placed on top of the emulsion, it is then gently rolled from the middle and outwards, as Teucher described. Using this method you will not get a wet air-surface of the pellicle when it is being rolled on the glass, and so you will reduce the danger of surface fading due to wetting, whilst you will still have fading at the bottom.
- Gottstein : Do you do this painting of gelatine before you put the treated glass into water?
- Dahl-Jensen : We paint the dry prepared glass-plate from Ilford; thus we do not have to wet it and put it under the water.
- Heckman : At Berkeley we do it just about the opposite way, and perhaps it started a couple of years ago when we decided that any immersion of emulsion in water is generally bad for the eradication of your surfaces. So I found perhaps we could make a dry mount, and by dry I mean that the only liquid which would enter into the mounting would be pure ethyl-alcohol. So the idea was to wet the emulsion in alcohol. The length of time for which the emulsion stays in the alcohol is not critical, and we would then mount it on the dry glass. This has been partially successful. There was some sticking, but not good enough. So we then decided we would add water to our alcohol solution, and now we have settled down to an alcohol solution between 65 and 75% in which we immerse our emulsions. This is just to actually wet the surfaces, and if you pull out your emulsion and look at it and shake it, you can see it dry in about 30 seconds. The glass-plate, however, is what we immerse in a gelatine solution. I do not know details of our gelatine solution, but I think it is very similar to the one that Teucher mentioned. Our solution

Heckman  
(cont.)

: in which we immerse the glass is formular, diluted by a factor of 2 by water; so it just has the strength of the solution. Our technique of mounting is the following. First we inspect the glass to make sure it is free from dirt; we also wash it with a solution of a detergent (Persolol, or something of this order), and we can inspect each pellicle. We do not wash very much the emulsion per se, but it is somewhat cleansed when we immerse it in the alcohol solution. So we have a vessel of alcohol, which is 65 or 75%, into which we can immerse our emulsion; hence we have an assembly line. One person throws an emulsion in and leaves the emulsion there for 3-5 minutes, and it does not make any difference. So if you are in the process of rolling-on and you break a glass or something drops, or something goes wrong and you have emulsion in this liquid, you do not have to panic. Then we have another vessel which is full of our 50% gelatine solution, and we immerse our glass in that. Then we have beside these a foam-rubber pad. Now we are ready to mount. We take a piece which we call parafilm-- it is similar to your polyethylene, though it is a little bit elastic, but it is a waterproof membrane. We put that over the foam-rubber. We take the pellicle out and lay it on the foam-rubber pad, and then we put one of the leading edges of the emulsion (rolling the emulsion out) on the pad and we put a bead of gelatine. You may call this bead of gelatine a 100% strength gelatine solution. As soon as it hits the alcohol on the emulsion it coagulates. Now we take the plate; we are looking through the plate all the time, and you can see how it is going on. Then you put the plate at an angle on the leading edge and it forms a wave-- you can see it contact. Now I demonstrate: your emulsion is here, and then you can see by putting your weight on

Heckman (cont.) : like this (it is rolled on) the wave going on, and then if anything is trapped you can see it, then you can back up and turn it; and then the last thing you do is to put your finger underneath and press the emulsion a little bit so that all the excess gelatine is just oozed right out the edge. Then we put a weight on for 30 seconds, take it off and roll it gently to remove the excess gelatine from underneath the pellicle and the surface is dry as soon as you turn it over and strip off the parafilm which you put on the foam-rubber in the first place. We start processing actually two hours after we have done this. But it is normally delayed. So, in a sense, I think that our techniques are similar. We use the same materials, but I think that we have just reversed what we soak and what we do not soak.

Vanderhaeghe : I am a little surprised about this 50% gelatine solution. Is it 50% by weight?

Heckman : You might say it is a solution containing  $7\frac{1}{2}$  g per 100 cc of water, instead of the 15.

Dahl-Jensen : We were asked to tell about failures in the processing. We had, around Christmas time, three weeks of processing with 12 batches of emulsion. The last three batches consisted of 80 pellicles belonging to the E19 experiment together with five or six pellicles belonging to a spurious scattering exposure, and 40 NIKFI emulsions belonging to Bombay. The first batch to be rolled-on were the 80 pellicles, then the five or six pellicles belonging to another emulsion batch from Ilford which were mounted on other glasses than the 80 pellicles, and finally the 40 NIKFI emulsions. In the fix-bath later on, we found the 80 pellicles did not show any bubbles, but that the five

Dahl-Jensen (cont.) : or six spurious scattering plates showed around 100 bubbles per plate. Of course, we looked very carefully at the glass-plates we had left from the sticking down and tried to see if we could not find any failures in them. But the gelatine coating from Ilford is very thin and it was very difficult to detect if there were any islands in it. The only difference we can suspect was that these five or six glass-plates were not perfect, because all the premises in this processing were the same for three different kinds of emulsions, and only these five or six plates showed a very large number of bubbles.

Teucher : We have not said anything so far about the rolling-on of NIKFI emulsions, and so I will take the liberty of quoting one paper by the Berlin Group. They have done quite some work on it, and one thing which is very much different from the preparation of the Ilford emulsions for the mounting is that with NIKFI emulsions it is not absolutely clear just from looking at them which side should be mounted to the glass. In Ilford it is very clear; you have one shiny side which should be mounted, and this you have to use. For the NIKFI emulsions, the side you want to glue to the glass has first to be cleaned. You have to rub away something between 3 or 4  $\mu$ , using an alcohol-water solution of about 25 or 30%. If you do not do this, you will have a black deposit at the bottom surface which cannot be removed later, and you cannot look through this under the microscope. The other procedure is not completely different, although the solution used is somewhat different: but if you want to look into it, I think all this has been published in "Nuclear Instruments" in great detail.



- Gottstein : I should be interested to know if anyone can tell us anything more about the correlation between numbers of bubbles appearing and the procedure applied. Sometimes it is, as you know, a nuisance that one has a lot of bubbles. Dahl-Jensen has just told us of an example, but maybe some of the other experts know a way of avoiding bubbles. Can anything be said about this?
- Vanderhaeghe : Certainly cleanness is very important. I can say that for the last two years we have avoided trouble with bubbling just by taking care, putting on gloves, and not touching either the back of the emulsion or the glass. But unfortunately this is not the only cause of blistering. Recently we had exactly the same kind of trouble as Dahl-Jensen; we had no time to investigate it carefully, but most probably it came from faults in the coating on the glass.
- Gottstein : Teucher told us this morning not to use gloves, but maybe it depends a little bit on the hands one has.
- Vanderhaeghe : No, I agree perfectly that inside the gelatine solution you do not need gloves; but for all other operations such as marking, gridding, and so on, i.e. every dry operation, it is much better to wear gloves.
- Bull : Is there a minimum time that should elapse between rolling-on and the beginning of the processing? Such a suggestion was made at Bristol some time ago.
- Teucher : We always did the mounting in the afternoon, and usually we waited more than 24 hours. But once we had to do it the next day, and also sometimes we had to wait, let us say five or six days, until we could really start processing. In my experience I found no correlation whatsoever between bubbles and this time.

Vanderhaeghe : I agree with Teucher. Nevertheless, a long time ago when this was suggested by Prowse in Bristol that it was better to wait for a certain time after mounting, we did some tests in Brussels and we agreed with this conclusion. But nowadays, with our present procedure, it is no longer necessary, and usually we do the mounting in the afternoon and process next morning without any more trouble. Sometimes we started processing immediately after the rolling-on, and it was still all right. This is, of course, better, because you reduce the fading.

Heckman : Our standard technique is also waiting until the next morning after rolling-on in one afternoon, so typically eight to ten hours. When we roll-on, in principle our emulsion is dry; at least the surface is dry and the interface between the glass and the emulsion is wet with gelatine. After we roll on and the surface dries, we notice that there are islands on the emulsion which swell relatively to anything else, and they start swelling just as if the rolling-on process did not squeeze all the gelatine up. There were pockets of moisture still there, which may lead one to suspect that adhesion at that point is not as good as at some place else, and we felt we should wait until the moisture has been able to withdraw from that into the emulsion itself and somewhat dries. So there are areas which appeared to us to be probably not as well stuck as they could be, owing to islands of moisture which are not squeezed up during the rolling process. So I would hesitate to say that we ever tried to correlate areas such as these with any bubbles that we observed. In general, it happens on every plate without exception that there are four or five areas which have bubbles with swelling, preferential swelling, and our bubble count has varied from a very few (less than 1%) to the complete catastrophe.

Dahl-Jensen : I can mention that our best processings have been those where we have started the processing immediately after sticking down. By best, I mean no, or extremely few, bubbles and a high sensitivity. One may expect that the sensitivity decreases with time after the rolling-on. If you then process immediately after the sticking down, you should expect a higher processed sensitivity, and there are hints that this is really so.

Teucher : I would like to comment on Heckman's experience. We had the same experience of this local swelling of the emulsion when we had to process the big stack in Chicago. This, I think, usually occurs about half-an-hour after the mounting. Suddenly you can see really such a swelling of sometimes  $\frac{1}{2}$  cm<sup>2</sup>, sometimes even more than that; but one should not be too anxious because in our case it always disappeared about the next morning. It gradually disappeared and we could not find at this point that bubbles occurred later. So if you ever will notice it, just forget it.

Finney : After sticking down the emulsion and before the processing starts, are the plates kept under pressure?

Heckman : We mount the glass on to the emulsion; we weight it very lightly just to keep the edges from folding away essentially for maybe one minute, then we roll and then we stack. We stack each pellicle as we do it until we get maybe three or four inches of glass and pellicles altogether. We might put a little weight on it, but there is no direct intent to weight them for any length of time. But I think, in fact, they are under weight for about 20 minutes during the process, but as soon as we get to stack them each glass and pellicle would peel off the parafilm and dry it.

- Gottstein : Is this stacking dangerous, or do you have some poly-ethylene in between it?
- Heckman : From the initial moment of the rolling-on we put the parafilm on the pellicle and we leave it on, so that no gelatine sees the top surface of the emulsion and makes it a very clean product. We leave that parafilm on for about a total of half-an-hour before we peel it off to dry it.
- Bovet : After having heard such complicated processes for sticking the emulsions on glass, I would like to mention that it is possible to stick the emulsion quite easily by immersion in cold water, say at 5°C. Dr. Manfredini in Rome has done so several years ago.
- Gottstein : Perhaps each member of the panel would describe briefly what kind of processing machine they use.
- Heckman : We are the wet-hot stage protagonists, and in Berkeley we are very tight for space. In fact, the emulsion group there has been in operation for about 12 years, and we started up the emulsion work in a dark room which was perhaps 9 by 12 feet in area. When we first started up we were using one by three inch emulsions, and this was plenty of room. Then all of us came to cubic feet of emulsion, and we had to process them still in this 9 by 12 foot room. So we had the problem of how we were going to handle such large volumes of emulsion using a dry-hot stage. So the idea was to try a wet-hot stage. The first results were quite encouraging, and so we continued to do it this way. We would merely cool, presoak, standard presoak, standard developer at 5° for 2½ hours, and then we would withdraw about 50% of the

Heckman  
(cont.)

: developer and mix it with hot water so that the resulting solution would be a 50% strength developer at 22°C. Then we dump the rest of our cold developer from the emulsions and rapidly run in the warm developer; we leave it for an hour or so at 22°C and then use standard stop and fix. In this way we think we alleviated two problems. First, we could do a very good size stack in one small 50 gallon tank. In fact, we have now a tank which is designed to handle up to about 400 sheets, each a foot square. These pellicles are racked up so that the glasses are separated only about  $\frac{1}{8}$  inch from each other, so we have a rack which is a solid layer of emulsions, and by having complete immersion all the time we do not have the problem of contact with the air which would tend to oxydize and eradicate the surface, which was in the early days one of our main concerns. Another advantage of this particular technique is that a great deal of the work has been considerably lessened. In the processing of a large stack the mounting, gridding, density measurements, etc., are a major task and require everybody in the group to take part. This is a one-day effort. After the plates have been rolled-on we put them into the rack, put them into this big tank, and start processing. This is a one-man job only. Our technician runs through the process all by himself, and he has nothing else to do but to make sure that the solutions are mixed, flowing in, flowing out. From the time the pellicles are racked-up initially and dried and placed in the tank they are not touched or moved, except for eliminating air bubbles between the glass, until they are withdrawn from the tank and dried. We might check bubble formation, but on a 200 or 300 pellicle stack it is practically impossible to check every pellicle. So we do nothing about bubbles, we just let them exist and

Heckman (cont.): let them dry out. We do not puncture them, and we find that the emulsion in the middle of the hubble is perfectly usable except for the crater that goes around each one. We have never lost a pellicle because a bubble grew to any considerable size; we had them perhaps up to 1 to 1.5 inch in diameter which is not uncommon.

Gottstein: Does Copenhagen also use the wet-hot stage?

Dahl-Jensen: Yes. Our processing plant has been described in the Suppl. Nuovo Cimento 15, 211 (1960) and therefore I shall not go into much detail. But the difference between our equipment and yours is not very large. It consists of separating the processing in two steps. After the development and stop bath we transfer the racks from the stainless steel tanks to a fixing and washing tank where they stay until we start the alcohol drying for which we have a third set of tanks. Our tanks have an area of 50 by 50 cm<sup>2</sup> and are 15 cm deep, in which one can place a rack having 11 slots where 11 plates can be put in. For safety we normally use only ten plates and put a glass plate at the top. We have heating from outside by coils which have been soldered on. We have two of these tanks for the actual development. These are operated in such a way that we start in one of them and, after one hour, we start the other one, and then the procedure is such that our pre-cooler and our thermostats always will be ready for the next operation. When we have completed two batches then we transfer the racks to the fixing equipment. If the silver deposits on the surface of the pellicles are too heavy, we remove them in most cases by gentle rubbing with medical cotton-wool between the stop and fixing stages, but not always. Then these two racks are transferred to the fixing equipment

- Dahl-Jensen (cont.) : and so on. I think these are the only differences between Berkeley's method and ours.
- Vanderhaeghe : I would like to ask a question about the wet-hot stage. Does the Amidol concentration in the solution used during the hot stage depend on the thickness of the emulsion or not? Some people use just the developer without Amidol, some others put half of the Amidol concentration, but I suppose this concentration has something to do with the thickness.
- Dahl-Jensen : Is there any dependence between the thickness of the pellicle and the Amidol concentration in the hot developer? I do not know if there is any variation in the effect of a given Amidol concentration in the hot developer on pellicles of different thickness, because we only have experience with 600  $\mu$  and 400  $\mu$  emulsions. I forgot to say that our second developer (the hot developer) follows a Brussels recipe in which the Amidol has been halved and potassium-bromide doubled, to prevent excessive development on the surface of the pellicle. The reason why we have kept the total concentration of salts more or less equal is that if you have a different concentration then you will have an ion concentration shock giving a sudden change in the swelling. Therefore we are very careful to keep this ion concentration constant.
- Vanderhaeghe : I would like to raise a few points which are slightly different from what Teucher described this morning. First, two remarks about the preparation of the developer. Usually we start by dissolving the sodium-sulphite. It seems that this helps a little to dissolve the boric acid. We put the Amidol in this solution after having cooled it down. Because Amidol oxydizes so easily, it is much

Vanderhaeghe : better to cool down the solution to about the right  
(cont.) temperature, i.e. 5° or 6°C, and then add Amidol which  
still dissolves very easily.

Another point is the temperature of the presoaking.  
Usually we start with the pellicles at room temperature;  
we put them on the slab and we drop the temperature down  
to about 5°C before adding the cold soaking water. Of  
course, we know that this means that the soaking time will  
be longer, but we prefer that just to avoid the exces-  
sive and too sudden swelling. However, there is some  
evidence -- I do not want to go into details now -- that  
anything you do before the hot stage has not much  
influence on distortion and spurious scattering. It  
had been suggested some time ago to do the soaking even  
slower, starting from an alcohol solution and then in-  
troducing water very slowly. It has been tried, I  
think, in Copenhagen and no improvement has been found.

Now a few words about the apparatus. We have two large  
slabs of about 2 sqm, and they are in stainless steel.  
The main reasons for the choice of this material are  
mechanical strength and low cost. I do not think that  
it is really so important to achieve a very good heat con-  
duction, as we all agree that everything has to go very  
slowly, so stainless steel is good enough. It is probably  
more important to have a very flat surface.

I should like to comment about the stop bath. Here,  
then, is a serious difference in our routine with respect  
to others. This is really something important because  
it has something to do with corrosion. I will first  
give the recipe: we add some sodium-sulphite in the  
stop bath and correlatively we increase the amount of



Vanderhaeghe : acetic acid to reach the reasonable pH value of about  
(cont.) 4.8. We are convinced that the presence of the sulphite in the stop bath is really very important because the stop bath has, in principle, a double role: firstly, to stop the development, and that is achieved by dropping the pH and the temperature; secondly, to destroy and wash out the developer. But usually a part of the developer remains in the emulsion. If you do not take care, the remaining Amidol will be oxydized and this is definitely a cause of corrosion, not the normal corrosion of the silver by hypo, but some more complicated process which has been called "catastrophic corrosion". I recall here an accident we had in Brussels, using the usual stop bath without sulphite. We were trying to improve the stop bath, having in mind to wash out the Amidol. We changed the stop baths three times, and we had a fantastic corrosion! Fortunately we kept the solutions. The three stop baths were of quite different colour: the first one was still colourless; the second one was slightly pink; and the third one was completely red. I remember we discovered this with Bonetti, and we understood then that, trying to wash out the developer from the emulsion we removed most of the sulphite, whereas a fraction of the Amidol remained absorbed by the emulsion. Of course, this Amidol was easily oxydized and we had corrosion. So I insist very much on this point.

Gottstein : Thank you. Shall we discuss this last point first, perhaps? Not all people use stop baths with sodium-sulphite, and not all people get this terrible corrosion.

Heckman : What is your recipe?

Vanderhaeghe : It is about 12 g/litre of sodium-sulphite; and about 1% of acetic acid, just to get a pH value of about 4.8.

Teucher : I just want to add something about the acetic acid concentration. I was asked this morning why I put only one in one thousand, that means 0.1%. This 0.1% I took from Occhialini's last formula for the handling of K5 emulsions, but I must admit that in our old routine, ten years ago, we started by 2% and gradually it went down to about 0.5 to 0.1%. I do not think that this concentration is extremely important because most of the stopping effect comes from the cooling-down, I would say.

Dahl-Jensen : When you are buying sodium-sulphite it is not supplied 100% pure by most manufacturers. You may have only 85% of real sodium-sulphite, the remaining 15% being oxydized to sodium-sulphate. Therefore, if you want to have a very constant sulphite concentration, you should make a chemical analysis of your sulphite and it usually comes out around 85%.

Gottstein : How serious is it to leave the stop bath out altogether? I mean, the pH change is also brought about by the fixer and the stopping is done by lowering the temperature, so I am wondering what will happen if you leave it out completely.

Dahl-Jensen : We have tried to avoid the stop bath and the argument is very simple. You have a developer of pH about 6.4, and you then introduce a stop bath in which one-half per cent acetic acid will give you about 3.8 pH. Most laboratories are using fix baths having a pH of about 4.7. As Gottstein told us, the development is stopped

Dahl-Jensen (cont.) : by lowering the temperature: so why introduce this acid stop bath when we are going to an acid fix bath immediately afterwards? We have tried to omit the stop bath several times for small batches of plates, especially spurious scattering plates, and we have found that it has no effect either on the quality of the processing or on the spurious scattering.

Vanderhaeghe : We also tried the same thing, and the result has been the same as regards the spurious scattering. But the plates were slightly less transparent, and this again means that the stop bath has a washing effect.

Gottstein : But did you get this really with two plates from the same batch, everything being done in the same way except for the difference of the stop bath?

Vanderhaeghe : Yes.

Gottstein : Are there any more comments on the whole developing procedure up to the stop bath, inclusive?

Finney : I would just like to make one comment on the pH value of the developer. In Durham we found when trying to develop emulsion at 15°C, using a developer with boric acid, that the quality of the development depended critically on the pH value of the developer. We also found, by accident, that the distillation unit was producing distilled water with a low pH, and the subsequent developer using this water did not, in fact, produce development. Using a buffer solution to control pH we found that, using ordinary tap water, we could get very good developments. This would indicate that one would need to be careful when using ion exchange water of low pH if one was not using

Finney (cont.) : a developer which did not contain boric acid, otherwise the pH of the developer may vary sufficiently to stop development.

Dahl-Jensen : The question of ion exchange water has been raised, and in my opinion it has no influence on the developer because the ion-capacity in distilled water is very, very inferior to the ion-capacity of the chemicals you are putting in.

Another point I should mention is that if you rely on ion exchange water for the presoak, then it can easily have a very low pH which will completely remove the latent image, whereas if you add chemicals which are supposed to buffer the developer, the initial water you use can be extremely acid without affecting the pH of the subsequent solution.

Gottstein : Now we might go to the next step, which is the fixing.

Vanderhaeghe : About the fixing, I have first one question to Teucher. He did not give the pH of the fixing bath this morning. What is your routine with pH? I suppose it is about 6 or 6.5?

Teucher : Yes.

Vanderhaeghe : We have the tendency to use a somewhat lower pH in order to get more transparent plates and to avoid refixing. But we know that this is also dangerous because the lower the pH, the more easily the corrosion occurs. So we usually make a compromise and have a pH of 5 to 5.5. It is not only a question of transparency and clarity of the emulsion, but also of the reduction of the swelling.

Vanderhaeghe : The isoelectric point being about 4.8, it is better to (cont.) keep up as near as possible to this value in order to reduce the swelling. As we fix at a temperature of about 10°C (we cannot go lower for technical reasons), we have to be very careful about swelling.

Now I am coming to a point of disagreement. Teucher recommended this morning to fix at a temperature as low as possible. I think that it is a bit dangerous. As Dahl-Jensen said, this might be a cause of chopping. If you go too low, around 0°C, some crystals could be formed inside the emulsion. So I do not recommend a very low temperature. I think the best is probably around 6°C.

Teucher : Of course it was not my intention to recommend fixing at -1° or something like that. I did not mean anything lower than 5°, and I have never tried to do it below 5°. So the meaning of my "as low as possible" was, if you can do it, do it at 5°. I have developed stacks at 8° and 9°, and this works sufficiently well, too.

Vanderhaeghe : I forgot something about the silver-bromide in the fixing bath. I think it is a good recommendation to start with some silver-bromide although, according to our present experience, it is not absolutely essential, especially if you start without agitation for a few hours; you will then have enough silver in the fixing bath to avoid the surface corrosion. Only one detail about the preparation of the silver-bromide: you have to wash it to remove the  $KNO_3$ .

One word about sodium-sulphate in the hypo to reduce the swelling. It can be used in case of blistering or some catastrophe of this kind, but it does not help very much.

- Vanderhaeghe : Recently, we have had a very bad experience with  
(cont.) blistering and I added some alcohol to the fixing bath,  
just to shrink the emulsion a little bit, and this worked  
very well. You can put 10-20% of alcohol; it helps  
very quickly and very well, without any trouble.
- Teucher : There are several possibilities for building a fixing  
apparatus. You always need some circulation of your  
hypo which is rather important, otherwise the old hypo  
will just collect on the surface of your emulsion and  
this is pretty bad.
- Heckman : Much has been said about agitation throughout the whole  
process. Vanderhaeghe said that leaving hypo in the  
stationary state for the first few minutes would be  
helpful to raise the silver content, then to employ  
agitation. We have always started agitation almost  
immediately without necessarily adding any silver bromide.
- Teucher : I do think that if you apply a dry-hot stage you need  
agitation only in the fixing bath, and you do not need  
any agitation in all the other steps. This might probably  
be different for a wet-hot stage, because in order to  
get uniformity of the temperature during the hot stage,  
you probably need some agitation. As we heard from  
Dahl-Jensen, he has his pumps in his tank. For the  
wet development we are now planning to introduce agita-  
tion, too, in a somewhat different manner. But I think  
for the fixing it is absolutely essential whatever you  
try to do. That means you need a rather gentle stream  
of hypo, and there are several way how to do it.\*)

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\*) Editorial Note : The description of specific processing installations  
and equipment (e.g. fixing tanks) has been omitted  
from this report of the discussion.

- Gottstein : I should like to ask Professor Teucher a question. This morning you said that one should avoid changing the fixing bath during fixing. So one should start with enough fixing solution. So what do you consider enough fixing solution for, say, 1 litre of emulsion? Obviously you do not want to have your hypo too much saturated with silver at the end.
- Teucher : During all our last experiences we never went above 12 g of silver per litre of solution, and the amount of hypo was adjusted to this figure. This included the 2 g initially put into the hypo. But, of course, this is a matter of taste; other people consider even 10 g dangerous because of bubble formation, and in the old days even 17 or 18 g were allowed.
- Vanderhaeghe : We tried to keep below 10 g per litre of silver at the end. That means that sometimes, when we have many pellicles in the same tank, we have to replace a part of the hypo before the end of the fixing.
- Dahl-Jensen : We have built an apparatus in Copenhagen [J.Sci.Instr. 37, 360 (1960)] with which we can keep the silver concentration in the fixing baths constant, so that when the silver concentration goes too high a magnetic valve opens and fresh hypo is added to the tanks, and when the lower limit is reached the valve will close again. The reason why we built it was the following: it was believed that corrosion was prevented by keeping the silver concentration up. We believe that if we keep the silver concentration at about 3 g per litre throughout the fixation, we should be safe. At least we have had no catastrophic experience.

Dahl-Jensen (cont.) : We have been using sodium-sulphate in the washing stage, and I do not understand why you want to put it in the fixing stage where you already have a very high ion concentration. If you add sulphate to your washing water, you can prevent the emulsion from swelling at this stage, but at some stage you must wash out the sodium-sulphate and then the thickness goes up again to almost the same extent; so in the last year we have left out the sodium-sulphate from the wash-water.

Another point is that Heckman mentioned that they go up to 95% alcohol in the drying. Teucher said that it should not be necessary to go so high. What we have found is that about 78% alcohol will have no further drying effect on the emulsion. So if you go higher than that you are only trying to make the emulsion thinner than required, and you will have a hardening of the surface as Teucher mentioned. Therefore it must be recommended that the final drying bath should not exceed about 80% alcohol.

Gottstein : Thank you. Any more comments?

Vanderhaeghe : I have one suggestion to avoid large swelling during the washing. It has been recommended to add sodium-sulphate at this stage to reduce the bubbling effect, because the sulphate has a slight tanning effect, but it does not help very much. I think alcohol is better. Another way to achieve this very easily is just to add a few per cent of acetic acid. If you keep the pH low, you do not get this raise. I am not sure that we have to wash necessarily with pure water at the end.

Dahl-Jensen : Another possibility must be to add a compound you want to have in your emulsions after they have been processed,



- Dahl-Jensen (cont.) : namely glycerine. It should have the same effect and you do not have to remove it afterwards.
- Gottstein : I should like to ask a question. Is really the smaller space which you need for a wet-hot stage the main consideration, or perhaps the only consideration, or do you also believe that you really get better results? May I ask the CERN people. Do you plan in the near future to replace your machine by a wet-hot stage?
- Vanderhaeghe : I do not think that we are going to spend a lot of money to transform completely our processing plant in the near future. We can use our plant as it is for the wet-hot stage, and it goes very well. We do not have the facility for agitation, but that does not seem to be really essential. The first attempt, on a large scale, has been made last month by Levi-Setti rather successfully. So, for the moment we have no special reason to change the plant completely. I must say that, for the moment, I am not at all expert in the wet-hot stage, but I do not see any strong argument against it.
- Teucher : I would like to say that even the big Chicago dry-hot stage plant was used a few months ago for a wet-hot stage. So most of the dry-hot stage plants can be used for wet-hot stages if someone likes to do it. For more than 10 years I was a strong believer in dry-hot stages, but now for the sole reason of not having enough space, I have to build a wet-hot stage. The problem of spurious scattering and dry- and wet-hot stage is extremely complex, as we know from the Copenhagen and Lausanne conferences.

Gottstein : So, am I correct in summarizing that so far the result of the wet-hot stage is not better and not worse than the result of the dry-hot stage as far as the emulsions go? Does everybody agree with that?

Dahl-Jensen : Our wet-hot stage of course has some disadvantages as well. One is that in most of the processings we also get a silver deposit on the emulsion surfaces that has to be wiped off afterwards. Another thing is that when you study a pellicle after processing, you will find a large number of very tiny grains which have highest concentration in the top and lower in the bottom. It does not affect measurements and visibility in the plates, because they are so extremely small that you have to look for quite a while to see them, but it is not perfect. However, I should like to ask the dry-hot plate people about the gradient in the emulsions, because Occhialini's argument for introducing the dry-hot stage was to avoid a gradient. I have been measuring in a few dry-hot stage pellicles, and I have found a gradient in all of them. Of course, in our own processing we also have a gradient, but it is generally much less than 5%, which is the maximum drop we have found at the surface and bottom of a pellicle. Dry-hot stage pellicles that I have seen show gradients appreciably greater than this.

Vanderhaeghe : At least for what we call a good processing -- and I think we have done a few of them in recent years -- we essentially have had only some 50% fading over the first 20  $\mu$  both sides of the emulsion. It is fading, it is not a gradient.

Teucher : What Dahl-Jensen calls the gradient is, in fact, the fading at the top and the bottom. I must say I really do not

Teucher  
(cont.)

: think that it is as bad as this curve. It could be the case if you are extremely careless, but if you are careful it does not happen like that. So from this point of view I do not think that I would make a strong argument for building a wet-hot stage.

Heckman

: I have a question, not necessarily on the uniformity of development, but I should say that our experience has been quite good as far as the variation of sensitivity or development of the emulsion is concerned. Our experience in dry-hot stage is very small, and so we have really no comparison in our own laboratory on the relative merits of the uniformity of development. On the question of cleaning your emulsions by rubbing, Teucher mentioned that you could wipe off the silver deposits after the stop bath, when it is quite easy to rub off. The other alternative is to wait until the processing is completed and you have gone to your alcohol and drying, and then we will remove the silver. I do not know if there is any real evidence which way is the better. I can only cite one example. Some years ago when we did the collaborative experiment on antiprotons, one of the techniques we used to measure multiple scattering of very steep particles was a method which we call 'surface angle' which was essentially measuring the angle that a particle makes when it enters into the surface of each emulsion. As it dives deeply into the emulsion, each cell length was, in fact, one emulsion plate. There were several stack exposed, but processed in different fashions and particularly rubbed off in different fashions. We were getting quite good results with our noise. We had about a tenth of a degree noise on measuring the entrance angle to each plate. The plates which Birge and Perkins worked on had, I think, perhaps twice or three times that noise, and one of

Heckman (cont.) : the suggestions was that this was perhaps a result of different treatment of rubbing. We waited to the very end of the process, finishing off at 95% alcohol. The surface hardened considerably, so that rubbing was probably less damaging to the very surface than when you rub it even in the stop bath, which is the method they used. They cleaned it off in the stop bath; we cleaned it off as the last thing, and there was a considerable difference in the distortion of the angle at the entrance of the top surface. This has led us to believe that it is perhaps better to rub the silver deposit off at the very end of the process, when the emulsion is really hardened and no serious distortion can be introduced.

Fletcher : Is there any real reason for alcohol drying as opposed to air drying, or is it just a matter of convenience?

Vanderhaeghe : Certainly alcohol drying is much simpler in many respects. First, you do not have such a serious edge effect which occurs at the beginning of the air drying. Secondly, it goes much faster, and thirdly, as mentioned by Teucher, you avoid automatically the trouble with bacteria which occurs sometimes with air-drying. According to me, these are all very good reasons. I do not think that anybody has really investigated completely the problem of distortion, but it is certainly not much better with air-drying than with alcohol.

Pelosi : What is the correlation between the various kinds of processing you mentioned and general distortion?

Teucher : I do not think that this can be answered in a few sentences because there are, of course, many ways in which one can introduce these distortions. Certainly you can

Teucher  
(cont.) : introduce distortion in the hot stage if you go to too high a temperature. You can introduce distortion if you do not keep your plates in a horizontal position but a vertical direction during fixing. You can introduce distortions by changing the pH too rapidly or if you are careless during the drying stage, especially in the air-drying, if you kept islands of water on your plate, and so on. There are so many reasons that it is really very difficult to give a short answer.

Winzeler : If one adds alcohol to the hypo, would it not be worth while to add it right at the beginning of the fixing, because in the case that bacteria are one reason for bubble formation, this would lower the possibility of bubble formation?

Vanderhaeghe : It has nothing to do with bacteria.. I never trusted this hypothesis of bacteria making bubbles in the hypo, just because one of the best killers of bacteria is silver. Ionic silver is a strong antiseptic. I do not recommend putting alcohol in the fixing solution every time because it is quite expensive.

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