

MEETING OF THE CORESTA SMOKE GROUPAT HAMBURG

24th May, 1962

Present:

Dr. J. Cuzin (SEITA - Chairman)  
 Dr. P. Waltz (F.T.R. - Neuchatel, Secretary)  
 Dr. K. Weber (V der C., Hamburg)  
 Dr. H. Kuhn (Austrian Tobacco Monopoly)  
 Dr. N. Carugno (Italian Tobacco Monopoly)  
 Dr. Libermann (SEITA)  
 Dr. Hausermann (F.T.R.)  
 Dr. L. Hjern (Svenska Tobaks Aktiebolaget, Stockholm)  
 Dr. Fouquet (Ste. Job.)  
 Dr. J. Bonnet (representing Ste Baumgartner)  
 Dr. Neurath (Reemstma)  
 Frl. Vogt (Hans Neuerburg)  
 Dr. G. Lipp (Brinkmann)  
 Dr. F. Seehofer (B-A.T., Deutschland)  
 Dr. Barkemeyer (B-A.T., Deutschland)  
 Hrn. Borowski (B-A.T., Deutschland)  
 Dr. Schmidt (Forchheim)  
 Dr. Wahl (Forchheim)  
 Prof. Kroning (Göttingen)  
 Dr. Rochus (Göttingen)  
 Dr. Artho (Burrus, Boncourt (Switzerland))  
 Mr. J.T. Williamson (Cigarette Components, Alperston)  
 Dr. Elmenhorst (V d. C., Hamburg)  
 Frl. Nügel (V d. C., Hamburg)  
 Dr. D.G. Felton (Southampton)

The meeting was arranged to coincide with the Quester-Colloquium for German speaking tobacco chemists, which was held this year at the Reemstma factory at Bahrenfeld, and members of the Smoke Group meeting mingled freely with others attending the Quester meeting.

Before the Smoke Group meeting on the Thursday, there took place a small dinner party on the previous evening. This was arranged by Dr. Weber and as this effectively decided the course of the main meeting, and indeed of the final recommendations, it will be described in detail.

100159195

WEDNESDAY EVENING, 23rd MAY

Present: Dr. Weber, Dr. Cuzin, Dr. Waltz, Dr. Carugno, DGF

Dr. Kuhn and Dr. Bonnet, who were also invited, did not attend.

Before dinner, Weber outlined some of the problems with which he was faced and which had bearing on the work of the Smoke Group. In Germany the Federal Republic was establishing a laboratory to deal with smoking and health problems and Weber feared that some of the recommendations of the R.C.P. report regarding the publication of tar and nicotine contents, filter efficiencies, etc. may receive Governmental enforcement. Moreover, a consumer's movement in Germany was being stimulated by a magazine "D. Mark", which had already published one article listing brands in order of tar yield and which was preparing a second publication. The first listings contained a number of errors, according to the members of the Verband (including the attribution of PETER STUYVESANT to B-A.T. !!), and this underlined the need for a standard CORESTA method for condensate measurement, to which Government laboratories and others could be referred.

The Neukomm-Bonnet process was by no means dead. The experimental work on which Neukomm staked his claims, including the bio-assay studies by Nicod and the chemical analyses by Matthey had now been published in Z. f. Präventiv-Medizin, a copy of which had been sent by Weber to Todd at the time, for his comment. There was a certain amount of pressure on the Verband to counter these claims. Cuzin added here that the French Minister of Health had put some pressure on Grimanelli and Waltz stated that he too was being urged to do something about the publication. It was felt that Neukomm was preparing another attack. Weber and Waltz believed that if no one else would answer these claims then CORESTA should do so. Waltz told me that he had not been entirely satisfied with Cuzin's chemical analyses. I understood that following the meeting in Paris in April 1961, Cuzin had discovered that his smoke collection device was retaining proportionately more benzpyrene than total condensate. He, therefore, repeated his series of experiments using cold traps and obtained on the whole similar results. Waltz had once more extracted tobacco with methylene chloride and had made carefully matched cigarettes from control and treated tobacco. These allowed for the improvement in fill of the tobacco and were matched as accurately as possible for numbers of puffs on burning. Analysis had then shown no diminution in the yield of benzpyrene. The sequel to these discussion is described later (see Interview with Dr. Bonnet).

Weber was also exercised about the possibility of there being new types of carcinogens in cigarette smoke. He cited the possibility of nickel carbonyl (Sundermann) and stated that B-A.T., Hamburg had demonstrated that nickel in some form was present in smoke condensate. The difficulty in proving that this was as nickel carbonyl was emphasised. Weber also mentioned that a "friend" (Dontenwill?) had shown that

100159196

nitrosamines were a group of substances which were exceedingly potent carcinogens, producing tumours all over animals, and were more active than benzpyrene and other aromatic polycyclics. He would like chemists to direct their attention to possible methods of analysis for these in smoke, or at least to demonstrating presence or absence. Weber said he believed that if they were present in smoke they could well be the "super carcinogen".

This raised the question of co-carcinogenesis and the possibility that phenols could be implicated. Most members of the Smoke Group had been represented at the Eastman Colloquium at Zug or knew of the disclosures by Touey and by Wynder. This tended to influence their thinking on the means of condensate measurement to be adopted.

Weber was also concerned in that Wynder had arrived suddenly in Hamburg. He appeared at Reemtsma while the Smoke Group was meeting but did not attend the sessions. He was present during luncheon and next day visited the B-A.T. laboratories with Seehofer.

During the evening, the Agenda suggested for the meeting was discussed at length. It soon became clear that there were two reasons why a CORESTA standard method was required and that a single method would not satisfy the requirements. The reasons were:-

- (a) for routine purposes for rating brands in terms of condensate and of nicotine, for which purpose condensate was defined in terms of total particulate matter.
- (b) for absolute research and reference purposes, when the method of collection should take account of all constituents in the smoke other than permanent gases.

Waltz, in particular, wanted a method employing Cambridge filters, which he already used. He was particularly concerned with the butt-electrification effect, which he had observed, and with the general hazards of leaking charges.

Cuzin believed that this could be successfully overcome by attention to the geometric design of the electrostatic precipitator unit. I argued that I.M.S.C. had adopted a method of collection using Cambridge filters and although the method of assay was different, I could only see good in the adoption internationally of a common collection device. Alternative methods of assay could be compared in this way without conflicting ideas as to how much and what was collected.

For the absolute method, there was general agreement that a cold-trap method operating at  $-80^{\circ}\text{C}$ . would be needed, especially if future interest turned to the phenols apparently present in the vapour phase. It was important, in Cuzin's opinion, that in neither case should the methods of collection and assay (and consequently, the

100159197

definitions of smoke condensate adopted) carry any implication of a health context, and it was agreed that this should be put to the meeting at the outset.

After considerable discussion, a draft "protocol" was drawn up by Cuzin and Waltz and this in fact was the final document agreed by the meeting next day, with minor alterations.

During the drafting session, Weber referred, in conversation to me, to the experiments on PCL cigarettes which were being carried out by Dontenwill in Munich, repeating those made earlier by Druckrey. I understood him to say that so far no tumours at all had appeared and that the animals were healthy. Burgan will be seeing Dontenwill with Weber during June.

The need for a standard method for benzpyrene was then discussed. Weber suggested that as a number of methods (Cuzin, Waltz, Barkemeyer, Grimmer) all gave about the same quantity of benzpyrene in smoke, that CORESTA should not issue a Standard protocol. Cuzin agreed with this. It seemed to me that everyone was relieved that they did not have to equip themselves with new apparatus in order to follow other peoples' methods. In particular, there was no enthusiasm to adopt the benzpyrene tracer technique for estimating losses which we use at R. & D.E., because no one possessed radioactive counting facilities.

#### THURSDAY, 24th MAY

Following the meeting on the previous evening, the actual proceedings of the CORESTA Smoke Group seemed an anti-climax. The course of the discussion was smoothly guided by Cuzin along the lines planned.

Cuzin opened by stating that an outline protocol had been circulated and that a definition of smoke condensate would depend upon the means of collection and assay chosen. He suggested that the definition should be without reference to health, but that it should be wide enough to embrace any future possible interests in this context (the implication being phenols or other volatile components).

Seehofer and Barkemeyer then made the point that two definitions were required:-

1. for routine purposes, which should be simple and rapid.
2. a standard reference method for research purposes.

Cuzin accepted these, only adding that a routine method for total particulate matter should lead as nearly as possible to a definition of the actual structure.

It was then agreed that an estimation of total wet weight of smoke was required, partly because some of the water in smoke may arise by chemical reaction between smoke constituents, and may play a role in promoting bio-chemical reactions between smoke and the

100159198

organism. This could then be followed by an estimation of the amount of water present, e.g. by Karl Fischer reagent. Cuzin mentioned that, in a series of collaborative tests between Hamburg and SEITA (Paris) in which the moisture content of the tobacco and the ambient relative humidity were carefully controlled, the standard deviation was smaller for wet T.P.M. than for dry T.P.M., using the Karl Fischer estimation. Hjern gave figures supporting this viewpoint.

Cuzin then listed the requirements for both types of method and the individual types of collecting devices which were put forward by the meeting, e.g.

<u>Routine Method</u>	<u>Reference (Research) Method</u>
Electrostatic precipitator	Cold trap at $-80^{\circ}\text{C}$ .
Cambridge filter	Solvent trap at $80^{\circ}\text{C}$ .
Filtration on wadding (Wahl trap)	Centrifugation
Cold traps at ? temperature	
Solvent traps	
Smoke densitometry/nephelometry	

After considerable discussion, the meeting settled on

(a) Reference method - cold trap at  $-80^{\circ}\text{C}$ .

(b) Routine method

(i) Electrostatic precipitator

(ii) Cambridge filter

For the reference method, Cuzin proposed that the trap should be allowed to warm to room temperature and then be purged with dry nitrogen until constant weight was achieved.

The design of the electrostatic precipitator would be left to a small sub-committee. There was considerable discussion regarding the size of Cambridge filter to employ. Several people, who had used the smaller size designed by the American Tobacco Company, had found trouble through blockage with consequent rise in pressure drop. Cuzin asked me for our experience and I referred to the 2nd Edition of T.M.S.C. Research Paper 4, which gave full design details of the larger unit. I was asked from where we obtained our units and, on replying that they were made in our workshops, I was asked whether we could supply them. I mentioned that there was the possibility of making them by extrusion moulding, which we were investigating but that the adoption of this technique depended upon a suitably large order leading to a reasonable price. Cuzin then asked the meeting to leave it to negotiations

100159199

between Weber, Cuzin and myself before the details of the Cambridge Filter were agreed.

[Subsequently, in conversation with Williamson, I have learnt that Cigarette Components also have an interest in Cambridge Filters and that although at present they use the smaller size, they are giving consideration to the larger size. It is possible that Cigarette Components would be interested either in buying extrusion moulded holders from B-A.T. or in becoming general suppliers of the larger size holders, i.e. they would absorb the initial cost of manufacturing the mould. They might also be interested in supplying the filter discs. At present the matter has only been discussed informally.]

The meeting then discussed the possibility of the same method being used for the determination of nicotine in smoke. One group, headed by Cuzin, believed that, in the smoke of fermented tobacco especially, nicotine existed in the vapour state. Hjern represented another group, who had no evidence for this. This may possibly be explained as due to the estimation of volatile pyridine bases as nicotine. It was agreed to adopt the same collection methods for the nicotine estimation.

The design of smoking machine, smoking parameters, etc. were agreed as those already used by CORESTA in the filter efficiency method.

The question of adequate sampling was discussed and Cuzin pointed out the difficulty in ensuring that a given sample is representative of a brand. Waltz suggested that the only solution was a repetitive sampling over a period of weeks or months until the variation between samples was known. This is the same solution to the problem as that for TARNIK or TRAFALGAR. It was suggested that the question of the size of sample (between 200 - 1000 cigarettes) should be the responsibility of the person calling for the analysis and will represent a given sample at a given time.

In conclusion, Cuzin made the point that it was the aim of CORESTA to provide standard methods approved by the industry to which a State Authority could be referred if it wished to publish tar and nicotine results.

During the later session, the draft protocol (attached as Appendix A) was circulated and approved.

The meeting also took note of the results to date of the collaborative trial of the CORESTA methods for filter efficiency determination. Copies were distributed (see Appendix 2). Results are still to come from one laboratory, but a partial statistical analysis had been carried out. It is surprising to note from this that determination of total nicotine alkaloids for the control and filter tip cigarettes yields a higher variance ( $\frac{s}{m}$ ) than do determinations of either wet or dry tars; but that there is little to choose between the variances of the filter efficiencies calculated in any of the three indices. It is possible that this is owing to one or more laboratories being unable

100159200

to analyse for nicotine satisfactorily.

The CORESTA method for filter efficiencies, it will be remembered, is based upon the comparison of smoke yields from a filter tip cigarette and a control cigarette of equal total length.

The need for a CORESTA standard method for 3:4-benzpyrene was questioned by Cuzin and the meeting agreed that this was probably unnecessary.

The rest of the meeting was occupied by scientific communications by members, including a number left over from the Quester colloquium. Brief summaries follow:-

1. Barkemeyer gave an account of his modified method for the estimation of benzpyrene employing column chromatography in place of thin-layer chromatography with detection by fluorescence spectra based on the work of Sawicki.
2. Borowski described experiments to measure the smoke temperature gradient as the cigarette butt got shorter. Measurements were taken by a germanium semi-conductor electronic thermometer and recorder embedded in the filter or tobacco stub 8 mm. from the mouth end. Burning temperatures were simultaneously measured by a platinum/platinum-rhodium thermocouple. The dependence of temperatures upon moisture content of the cigarettes was studied, but few conclusions were drawn from the work.
3. Kuhn gave brief summaries of papers published in the recent issue of Fach. Mitt. Oesterr. Tabak. on the estimation of low molecular weight carbonyls in smoke and of the measurement of burning temperatures by the fusion of crystals of high melting salts.
4. Barkemeyer demonstrated a modification of the vibrating smoke collection trap suitable for quantitative collection for analytical purposes. The collection efficiency was claimed to be greater than 99%.
5. Hjern, Barkemeyer and Lipp discussed recent work on the estimation of glycols in tobacco by gas chromatography.
6. Cuzin then raised the problem of Nickel carbonyl in cigarette smoke as a possible subject for consideration by the smoke group. Barkemeyer reported that he had demonstrated the presence of nickel in some form in smoke. Fouquet stated that cigarette paper was free from nickel, which therefore presumably came from the tobacco. The most interesting contribution on this came from Kuhn who stated that he had a project in Vienna in which lung tumours from smokers and non-smokers were to be assayed for nickel

100159201

by activation analysis. This was being conducted in the University of Vienna.

#### THE QUESTER COLLOQUIUM

In conversation with members of the colloquium I gathered that most of the topics discussed were of limited interest. The colloquium had ended with a discussion about ways of measuring filling power. The meeting had eventually decided that there was no simple definition of what was meant by filling power, since this could be influenced by many factors, e.g. moisture content, width of cut, additives, etc. It will be brought up again next year, when the colloquium will be held at Orleans under the auspices of SEITA.

I also learned that Hjern had seen a F.P.I. Tester at B-A.T. Hamburg last February and that he had ordered one from Imperial at Bristol, which he hoped to receive at Stockholm shortly.

#### INTERVIEWS WITH DR. BONNET

Despite the interest shown in the Neukomm-Bonnet process at the unofficial dinner party already described, it was not mentioned during the formal meeting. It was, therefore, greatly interesting to me to be approached by Bonnet after the meeting.

He told me then that he and Neukomm no longer saw eye to eye on the subject of exploiting the process. He, Bonnet, believed at the time he made his analyses that his results were correct. Subsequent work appeared to have proved him wrong. Neukomm, however, insisted on attributing great significance to these and to the Nicod biological tests. Bonnet did not accuse Neukomm of dishonesty, but came very near to doing so. He explained that, contrary to my belief, Neukomm was not Director of the Lausanne Institute, although he insisted on behaving as if he were. As long as the Chairman of the Board was a friend of Neukomm this was alright, but this was no longer the case and Neukomm was manoeuvring to maintain his hold of the Institute. Bonnet no longer works for him, but has interests of his own on air pollution. He is still employed by the Institute and is merely a consultant for Ste Baumgartner. If Neukomm wins the battle at the Institute, then Bonnet will find his own position untenable and will seek a post in industry, but probably not with Baumgartner. He mentioned that Neukomm is reopening his work on tobacco, suggesting that a further attack will be forthcoming; and he implied that Neukomm is now misapplying Institute funds to further the investigations by SASMOCO. This company has changed hands once again and is now owned by someone who is a stranger to Neukomm.

Subsequently, Bonnet sought me out in the Departure Lounge at the airport on the Saturday. He had attended a meeting with Cuzin and Weber

100159202

the previous evening (Waltz had been with me at Seehofer's house that evening until he left Hamburg by train and so had not participated). Bonnet urgently wished to bring me up-to-date on developments. It had been decided that Weber should go and see Nicod and confront him with a report by a Statistician retained by Weber to examine the Nicod report. Weber had shown me this report on the Wednesday night. It is along similar lines to the Todd/D.G.F. report to T.M.S.C. and calls attention to all the anomalies of vanishing mice, imperfect statistics, etc.

It had further been decided that CORESTA should publish (in the Bulletin?) a full and detailed refutation of the process based on the chemical analyses by Cuzin (both series) and by Waltz and the short-term biological tests by the French Regie. This would be reinforced by the statistical criticisms.

Bonnet seemed to look on this as a weapon in his battle with Neukomm and suggested that the publication would guard against Neukomm keeping hold of the Lausanne Institute. Bonnet offered to keep in touch with me and to send me the full issue of the Swiss Journal<sup>n</sup> which was published the details of the Neukomm-Bonnet process. (He subsequently did this.) He admitted that the machine for treating tobacco was useless and that Neukomm knew it could not be incorporated in a production line. In reply to a direct question he told me that they had never employed a catalyst, nor investigated the suitability of compounds for this purpose. As at Paris last year, he once again admitted that the second patent mentioned catalysts purely in order to give novelty for patent purposes.

100159203