

PRIVATE & CONFIDENTIAL

Research & Development Establishment,  
British-American Tobacco Co. Ltd.,  
SOUTHAMPTON.  
12th February, 1958

VISIT TO I.T.Co., RESEARCH DEPARTMENT, BRISTOL

11th February, 1958

Persons seen: Dr. H. R. Bentley  
Mr. J. G. Burgan

1. SUPPRESSION OF BORSTAL FORMATION BY AMMONIUM SULPHAMATE

The Research Department of I.T.Co. have been approached informally both by I.C.I. Ltd., fairly recently, and about a year ago by Albright & Wilson, with the offer to supply ammonium sulphamate for the treatment of cigarette paper. In both cases, the reply made by I.T.Co. was that they were not interested.

The rejection was based on the results of experiments designed to test the claims of the Rand Development Corporation process as it applies to cigarettes wrapped in treated paper.

The BORSTAL content was the same for smoke from 500 cigarettes made from untreated rag wrapped in untreated paper and from 500 cigarettes from untreated rag wrapped in paper loaded with 4½% (w/w) ammonium sulphamate. A reduction of 40-50% in the BORSTAL content of smoke was observed for cigarettes made from rag treated with 5% ammonium sulphamate and wrapped in untreated paper. At that time, I.T.Co. were looking for 90% reduction in BORSTAL content and other compounds were equally promising.

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Rand's method of burning paper by stuffing it into a glass tube was rejected as unrealistic and the claim for an extensive reduction in BORSTAL production under these conditions was not checked.

I.T.Co. studied BORSTAL formation from cigarette paper in two other ways:

- (i) Paper "cigarettes" yielded 10 g/500 g. paper
- (ii) In conjunction with Fletchers, they burned cigarette paper directly from a bobbin by passing it in a continuous feed over a platinum spiral, heated electrically to approx. 800°C. The tars and gases were aspirated away through a collection train. In the presence of excess air, 5 g./500 g. was detected, whilst in minimum air, none was found. This is rather the reverse of what was expected, but I.T.Co. did not enlarge on this.

I.T.Co. appear to have covered thoroughly the case which would interest cigarette manufacturers most closely. Reduction of BORSTAL production under conditions which could not possibly obtain in cigarette smoking, would seem highly irrelevant.

The work was submitted to the I.M.S.C. as a draft paper for publication, but was later withdrawn.

## 2. GALLAHER-I.T.Co. JOINT EXPERIMENT ON SMOKING PARAMETERS

At present the I.T.Co. Autosmoker, which is used for all the smoking experiments at Bristol and for the production of fractions of smoke for biological examination by outside workers, operates with 4 puffs per minute each of 15 ml. volume and 2 sec. duration. It was felt that this

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was an unrealistic approach to the average conditions of human smoking and an effort was made to see whether the opposite extreme conditions gave a smoke of different composition.

Gallaher's Research Department at Belfast employ an autosmoker very similar to the Cigarette Components Machine Ethol Mark IV, i.e., it operates at constant pressure instead of constant volume, employs solenoid-operated valves and is adjustable over the range 20-60 ml. puff once or twice a minute. A point worth noting for R.& D.E. is that they find it necessary to employ an electrostatic precipitator unit operating at 18-20 Kv. in order to ensure complete precipitation of smoke at the much higher airflows encountered with the larger puff volumes. For this experiment the Callaher machine was run at 1 puff per minute of 2 sec. duration and 35 ml. volume.

Cigarettes were made from two Canadian flue-cured pipe grades at Bristol and after conditioning, these were weight-selected and then draw-selected. Cigarettes for the two series of experiments were then drawn from these at random.

Five series of estimations were made on the smoke from each of these batches of cigarettes, following the I.T.Co. standard methods of smoke analysis. These were -

- a) Total Nitrogen (TN)
- b) Total Volatile Nitrogen (TVN)
- c) Nicotine Nitrogen (Nic.N)
- d) Total Acids (TA)
- e) Total Volatile Acids (TVA)

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For each estimation, six replicate smokings and analyses were carried out, using five cigarettes each time.

The experiments at Belfast were carried out by workers from Bristol.

The results were expressed in terms of the ratios:

$\frac{TVM}{TH}$ ,  $\frac{M.C.H.}{TH}$ ,  $\frac{TA}{TH}$ ,  $\frac{TVA}{TH}$ , and the combined blocks were treated statistically using the Analysis of Variance. The differences found between smoking conditions were shown to be different for each of the grades for the first three ratios with significances of 0.3%, 0.1% and 0.1%, respectively, i.e., this difference would only be encountered by chance in three cases, one case and one case out of 1000, respectively. The ratios  $\frac{TVA}{TH}$  were not significantly different.

Dr. Bentley and Mr. Dargan were at pains to point out that this was only a ranging type of experiment, but it has interested them sufficiently to lead them to begin to build a smoker like the Callaher machine and to suggest to the I.M.S.C. a study of human smoking conditions.

I was shown a typescript of a letter to Mr. Baumann of Cigarette Components in which the results were detailed and was informed that a copy was being sent to B.A.T. to Mr. Hobson. I abstracted the main details, given below.

Grade	<u>I.T.Co. Conditions</u>		<u>Callaher Conditions</u>	
	HF1	HF2	HF1	HF2
Average Ratio				
$\frac{TVM}{TH}$	60.0	58.9	55.0	57.9
$\frac{M.C.H.}{TH}$	33.5	41.6	32.0	38.7
$\frac{TA}{TH}$	230.4	-	265.3	-
$\frac{TVA}{TH}$	60.4	-	61.5	-

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I.T.Co. have also recorded weights of smoke collected but, since they use cold traps for smoke collection and smoking is not carried out under conditions of constant humidity, they have not analysed these figures statistically and could not say whether there were any significant differences. Their "smoke collected" figures refer to samples containing variable amounts of water. They do not heat their samples to constant weight, which is the procedure at R. & D.F., nor do they estimate water in tar by the Karl Fischer reagent. They are not interested in tar as such.

### 3. ARGENTINIAN FILTERED CIGARETTES

A number of points were taken up with Mr. Burgan on this subject. It was suggested that expressing results in terms of dry weight tobacco analysed or dry weight tobacco smoked would be advantageous in rendering comparisons between groups more simple. He pointed out in reply that their general procedure was evolved for their studies of smoke quality and in these cases they make their own cigarettes from straight grades etc. on their Molins "Minimaker". These cigarettes are manufactured to Class B standards and are then weight selected for  $\pm 2\%$  of average weight (not formula weight) and then selected for  $\pm 4\%$  draw resistance. This means that, in general, their cigarettes are all about the same, but he agreed that this was not the case with the Argentinian cigarettes and admitted that there was no reason why the dry weight basis should not be used.

The leaf analysis figures, in fact, are in terms of dry weight tobacco and represent the average of duplicate analyses. The smoke analysis figures are the mean of six replicate analyses. Standard deviations and 95% confidence limits are computed for all analyses of smoke.

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The difficulties in analysis which have apparently been encountered at R. & D.E. in the first attempts so far made were discussed. Mr. Burgan was not at all surprised that the initial attempts were abortive. A discussion of the methods employed ensued. Smoke analysis was dealt with in I.T.Co. Research Reports R.034 and R.035 and copies have been sent to B.A.T. The micro-Kjeldahl method employed was demonstrated and has proved very rapid and reliable, but extreme precautions must be employed. A separate laboratory from which ammonia fumes can be excluded completely is essential and blanks of reagents and apparatus are necessary. The various pieces of equipment are available commercially. The analyses and statistical calculations are conducted very accurately by a team of four girl assistants, average age 19-20, supervised by a woman of H.N.C. (Chemistry) standard. The calibre of these girl assistants appeared to be very high.

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Some of the Argentinian cigarettes were supplied to Dr. Cook of the H.N.C. Smoke Analysis Laboratory at Exeter, who has told I.T.Co. that he has had some interesting findings. He requested a further supply of the cigarettes, which I.T.Co. have sent. Cook wishes to talk over his results when <sup>he</sup> has completed his experiments, and Bentley and Burgan intend to visit him when he has sent them his results. Bentley broached the possibility of the writer accompanying them, but nothing more transpired on this subject.

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#### 4. OTHER OBSERVATIONS

##### a) Gas Chromatography

I.T.Co. now have their home-made equipment working on model mixtures of esters in a series of experiments designed to evaluate the best conditions of temperature and column packing for separation. They then intend to employ it to analyse the acid fraction of smoke by conversion to a mixture of methyl esters by means of diazomethane. Their apparatus was demonstrated and separated an eight-component mixture of esters in 10 minutes. It employs a Martin Gas-Density Balance detector, the output of which is fed directly to a Sunvic recorder (1.6 sec. response time) modified to give a full scale deflection for 100 microvolts input and with a chart speed of 12 inches per hour. The Martin Gas-Density Balance gives a stable base line and is very easily set up. It possesses the advantage of being independent of flow rate and does not give the spurious peaks which certain hot wire Katharometers do. A Perspex model of this Balance was kindly loaned to the writer by Mr. Burgen in case R.& D.E. should consider constructing one in the future.

##### b) Infra-red Spectrometer

At present this is being employed in attempts to study alkaloid degradation products in the basic fraction of smoke. A number of pure reference samples have been synthesised, e.g., myosmine, nor-nicotine and certain 3-pyridyl ketones. My impression was that the fractionation of the basic fraction of smoke had not progressed very far.

##### c) Aldolides in smoke

Some attempts were being made to set up a scheme of analysis

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for aldehydes in smoke. The mixture of aldehydes and ketones are converted into their 2:4-dinitrophenylhydrazones, which are then separated by paper chromatography. The individual spots are then eluted and examined by means of ultra-violet spectrophotometry. Aldehyde and ketone dinitrophenylhydrazones of various fundamental skeletons give characteristic U.V. spectra, as is well known. This project has not yet progressed very far.

d) Flowmeters

I.T.Co. have recently acquired a flowmeter from Cigarette Components at a cost of \$51 and are very impressed by the speed and accuracy of working possible with it. However, they do not like the C.C. cigarette holder and still prefer to use Veridia tubing. They, too, have observed a variation in draw resistance depending on the depth the cigarette is pushed into the holder, but apparently had not considered whether paper porosity could be responsible. Their draw resistance measurements are all done in an air conditioned room.

DEF/VC/46D

*D. L. Seltzer*

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