

Spore  
HDA/DGF/FG/46D

28th July, 1958.

A.H. Faul, Esq.,  
W.D. & H.O. Wills (Australia) Ltd.,  
P.O. Box 145,  
Sydney, N.S.W.,  
Australia.

Dear Mr. Faul,

Following Mr. Hobson's cable to Mr. Foley, we have got together here some information for you on the equipment and methods which are available for the test smoking of cigarettes, and the determination of tar, nicotine and filter efficiency. These are:-

Cigarette Smoking Machines

- 1) "Mechanical Cigarette Smoking". 27th March, 1958. Amended 21st July, 1958. Ref. HDA/DGF/VC/46A.
- 2) "Ethel" Mark VI Smoking Machine. Brochure issued by Cigarette Components Ltd.

Tar and Nicotine Determination

- 3) "Determination of Nicotine and Smoke Condensables". 27th March, 1958. Ref. HDA/DGF/VC/46C.
- 4) "Spectrophotometric Determination of Nicotine". (Reprints).
- 5) Foster D. Snell Method (as used in Reader's Digest)
- 6) Unicam SP.500 Spectrophotometer. Brochure issued by Unicam Instruments Ltd.

Filtration Efficiency.

- 7) Letter to Dr. I. W. Tucker, 2nd July, 1958. Ref. HDA/VC/46B.

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8) Set of Pro Forms for recording filtration characteristics.

9) "Programme of investigations of Australian Cigarettes." 25th July, 1958. Ref. DGF/PE/46D.

We have now received the 27 brands of Australian cigarettes and are starting to investigate these at once. We enclose a detailed programme of the investigation which will be carried out. You will see that this entails a very heavy load of work and, in view of the urgency of the problem, we intend to report to you first the tar and nicotine figures for the smoke collected for each brand for

- i) selected cigarettes
- ii) cigarettes of high weight and draw resistance range.
- iii) cigarettes of low weight and draw resistance range

giving at the same time an indication of how these may be expected to reflect the average for unselected cigarettes. When this has been accomplished, we shall then turn to the remaining parts of the programme.

There is a number of methods currently in use for the determination of tar and nicotine, especially of tar. These do not all measure the same quantities and, for your information, we list them below. They are:-

1. E.-A.T. Method.

Smoke is collected by electrostatic precipitation. For "tar" estimations the smoke collected is dissolved in acetone, transferred to a tared flask and the solvent removed. The residue is heated at 100°C. for 15 minutes. For nicotine estimation the smoke is dissolved in acetone containing dilute hydrochloric acid; the acetone is removed by distillation under reduced pressure, and the residue, after the addition of alkali and sodium chloride, is steam distilled. The nicotine evolved is collected in dilute acid and estimated spectrophotometrically by the method of Willits et al (see attached photostat copies of papers).

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2. Foster D. Snell Inc. Method

This is described in detail in the accompanying photostat. It is the method employed in the Reader's Digest investigations. In this procedure smoke is collected by bubbling through acidified ethanol in solvent traps and the whole of the material collected is evaporated to dryness and then thoroughly heated (7 hours at 105-110°C.) before weighing the residue as "tar". Alternatively the material collected is steam distilled to remove volatile solvents, made alkaline and again steam distilled to liberate nicotine. The nicotine vapours are collected in acid and the alkaloid estimated by precipitation with silico-tungstic acid.

3. Consumer Union Method as used for the figures given by "Consumer Reports".

The smoke is collected by acidified ethanol in solvent traps as in the Foster D. Snell method. The combined solvents and washings are divided into two portions, one portion being steam distilled after basicification to liberate the nicotine which is collected in acid and estimated spectrophotometrically. The other portion is extracted by shaking with chloroform, the chloroform layer is separated, dried, and the chloroform evaporated. The residue is recorded as "tar".

4. Cigarette Components Method. (as now used by Scientific Control Laboratory, Liverpool).

Smoke is collected by electrostatic precipitation in a weighed glass tube. The tube and smoke precipitate is immediately reweighed to give the weight of "wet tar". The smoke condensate is then dissolved in dry methanol and the water present is estimated by titration with Karl Fischer reagent. The water content so found is used to correct the "wet tar" figure to "dry tar". Liverpool do not at present measure nicotine in smoke.

5. I.T.Co.(Canada) Method. (for whole tar only)

Smoke is collected in helical traps surrounded by solid carbon dioxide (dry ice). The traps are washed into beakers using a mixture of methylene chloride-methanol (85:15); the solution is filtered into a standard flask and made to 100 ml., ensuring all tar is washed from the filter and paper. Aliquots of 2 ml. are pipetted into weighing bottles and the solvent is evaporated by

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suspending the bottle above a warm (35°C.) hot plate and blowing air across the surface. When all the solvent appears to have been evaporated, the bottles are placed in a vacuum desiccator until successive weighings are within  $\pm 0.1$  mg.

6. I.T.Co.(Bristol) Method

This is in the process of being altered; two methods have been employed in the past.

a) Smoke is collected by electrostatic precipitation and the condensate is washed from the tubes by acetone and made to a standard volume. The concentration of smoke is then measured colorimetrically by a Hilger "Spekker" absorptiometer using neutral filters. This is a comparison method only.

b) Smoke, collected by electrostatic precipitation, is washed from the tubes by a mixture of ether and hydrochloric acid. The layers are separated and the ether layer washed with more dilute hydrochloric acid and then dried (sodium sulphate). The solvent is removed and the residue of basic-free tar is weighed. The acid solution and washings are used for nicotine estimation, the solution is basified, steam distilled, and the nicotine is estimated spectrophotometrically by the Willits method.

We are in the middle of compiling a list of the known methods, including others beside those listed above, for measuring smoke condensables, with critical comments about the properties they determine, and this we will send to you as soon as it is prepared. We are also at the moment in the process of comparing the Foster D.Snell procedure with our own. Initial results suggest that the Foster D.Snell figures are a little lower due to the much greater period of heating employed by them. We think the B.-A.T. method for determination of nicotine described above is as accurate and simpler to use than the silico-tungstic method which Foster D.Snell employ. There are also some indications that a very little smoke escapes collection by the solvent absorption train. By placing our electrostatic precipitators after solvent traps we have found signs of this.

This comparison will enable us to report results in our absolute values and to transpose these to what would very probably be the corresponding Foster D.Snell figures.

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We have given much thought to the apparatus and methods which you yourselves should set up in your Laboratory for the routine testing and evaluation of these same figures.

a) Conditioning and Selecting Cigarettes.

We do not know how you are placed as regards accommodation in air conditioned surroundings. Ideally, all sorting and selection of cigarettes should be done in a R.H. of approximately 55-60% and at 65-75°F. If you have a constant R.H. room then this will be ideal. If not, you will need a humidity cabinet for reconditioning your cigarettes and for storing the selected ones. For weight selection it is possible to weigh cigarettes individually on a balance. We use a Holins Automatic Individual Cigarette Weigher adjusted so that the "efficiency" range covers  $\pm 20$  mg. either side of the selection weight. An assistant watches the machine and removes those cigarettes which fall within the range, although eventually we hope to have the equipment modified for automatic selection. Weight selected cigarettes are then selected for draw resistance and for this we use home-made pressure drop machines which incorporate a pump for suction, a ballast tank, a flowmeter (Rotameters or capillary flow meters are suitable) and a manometer. Cigarette Components Ltd. make a pressure drop tester which you may already have for filter plug evaluation.

b) Smoking Machine

We think that the Patent Cigarette Components smoking machine is best fitted to your task as it combines the greatest flexibility in duration of puff, number of puffs and size of puff with reasonable reliability. We are ordering one of these machines at once and it should be in your hands by early October. The best delivery date they can quote is eight weeks. We understand from Mr. Hobson that it would be desirable if this equipment were at Sydney when Sir Charles Ellis visits you and, in these circumstances, the only solution would appear to be air freighting the machine from England to Australia. If you wish the shipment to be made by air freight, would you please let us know by return so that Engineering Department at Hillbank can make the necessary arrangements and inform you of full details which will be required for import licence etc. This smoking machine includes an electrostatic precipitator for the collection of smoke, and we suggest that you adhere to this method which is superior, in

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our opinion, to the Foster D. Snell wet collection. If you desired to copy exactly the Pader's Digest method, you may do so with this same smoking machine.

c) Oven

Both the B.-I.T. method and the Foster D. Snell method use an oven for heating the tar under standard conditions. For this purpose we have found that a standard Pearson moisture test oven is suitable. You will perhaps have such an oven available. It is not easy, however, that this oven should be kept entirely for drying out the tar samples; it will not be free for standard moisture tests. If this condition cannot be met, then you will either need another Pearson or a similar thermostatically controlled electric oven fitted with a fan for forced ventilation. Should you require assistance in getting one of these quickly, please let us know.

d) Spectrophotometer

We recommend the use of a spectrophotometer for nicotine determination and on your behalf have already made enquiries for a Unicam SP.500 (brochure enclosed) which we think is the most reliable, robust and easy to use apparatus of its kind. Unicam have an agency in Australia: Electronic Industries Imports Ltd., 139-143 Bourke Street, Melbourne, and they should be able to supply ex stock. In this way you will avoid import duties, import licence formalities, and will be able to obtain installation and service facilities. If we were to obtain the instrument for you, we should have to disguise the fact that its ultimate destination was Australia. We therefore advise you to approach them as soon as possible.

e) Glassware

In this work we use exclusively glassware with ground glass standard taper joints supplied in England by Quickfit & Quarts. Probably you will have little difficulty in obtaining similar equipment in Australia. We are currently investigating the use for nicotine steam distillation of the Markham still, which is available from Quickfit. By the time your other equipment is ready, we should be in a position to send you full details of the use of this piece of apparatus.

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A.H. Paul, Esq.

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The meaning we attach to filtration efficiency is clearly explained in Item 7, but considerable confusion is found among different authorities by the definitions which are chosen for this term of filter efficiency. We think if you will adhere to our definition, measuring both the efficiency of the filter and that of the tobacco rod which it replaces, you will not only be able to understand and interpret the results, but will also be able to compare them with other authorities' figures no matter how they have expressed them.

We incline to think that the most pertinent figure, at the moment, is the amount of tar (or nicotine) produced per gram dry weight of tobacco smoked. This enables all blends to be compared on an equal basis very easily and figures such as the amount of smoke reaching the smoker's mouth may be determined from a knowledge of the weight of the cigarette, amount of butt, and the filtration efficiency of tobacco or filter. This figure is not a total amount of smoke (mainstream and sidestream) coming from a gram of tobacco, but is, of course, only the mainstream, and it would be more illuminating to know both quantities. As it is, the mainstream smoke is dependent upon the rate of smoulder and the number of puffs per minute. This is why it is advantageous to settle in one's smoking technique for a standard procedure, and we would suggest that you follow the Foster D. Noil method of 35 ml. 2-second puffs once a minute, which seem very close to human smoking habits.

If there is any further information which you would like, or if anything in this letter or enclosures is not completely clear to you, please cable or write and we will do our best to send you additional information.

I should like to add that we are very happy to think we are assisting you and intend to do all we can to provide you with the information you require.

With best wishes and kindest regards.

Yours sincerely,

*HDA*

Encs: 9

M.C. D.L.L. Hobson, Esq. (all encs. except No.2 and 5)  
Sir Charles Ellis (all encs. except No.2)  
Engineering Dept. London (No enclosures)

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