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IMPROVED METHODOLOGY FOR THE DETERMINATION OF PUFF-BY-PUFF DELIVERIES OF TPM, WATER AND NICOTINE

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SUMMARY:

Several methods of determining puff-by-puff PMWNF deliveries have been developed. However, all use smoke filter holders made of Perspex (Lucite) and hence the holders change weight with temperature and humidity. This weight instability is a source of error and variability in puff-by-puff determinations, wherein much less particulate phase is collected in a holder than in a corresponding determination of cigarette total PMWNF.

This report describes how a Borgwaldt rotary smoking machine can be easily made to accept lightweight non-hygroscopic filter holders (Gelman) which in turn enable a more accurate method of determining water in TPM to be employed.

Improvement in data acquisition and handling are also discussed and the use of the Borgwaldt automatic holder changer in puff-by-puff determinations is described.

INDEX TERMS:

Mainstream Smoke
Puff-by-puff Deliveries
TPM
PMWNF
Tar
Water
Heinr. Borgwaldt
Isolation Procedures
Nicotine
Low Delivery Cigarettes
Ultra Low Delivery Cigarettes
Coulometric Water Analysis
Gelman Sciences
Smoking Machines
Apple PC

INTRODUCTION

As cigarettes become more sophisticated, for example, in having novel filters, it is increasingly important to be able to measure deliveries of smoke components on a puff-by-puff basis. Using such measurements, theories of smoking mechanics can be devised or verified and the effectiveness of new developments, such as controlled delivery profile cigarettes, can be confirmed.

Several procedures have been devised in BATUK&E involving the use of different smoking machines, including the BAT 6-port machine (1), the Filtrona 300/302 machines (2), the Mason Rotary machine (3-5) and the Borgwaldt RM20C/S (6).

The procedure devised in R&DC using the Borgwaldt smoking machine (6) involved the use of the standard Borgwaldt puff-by-puff holders and determined mean puff-by-puff deliveries of TPM from 15 cigarettes. The balance used to weigh the filter holders was linked to an Apple microcomputer which was programmed to plot TPM against puff number. Standard extraction and analysis procedures were used to determine water (7) and nicotine (8) in the TPM to give mean PMWNF deliveries on a per-puff basis.

This report describes how these procedures were improved to enable more accurate and precise determinations of puff-by-puff deliveries of TPM, water, nicotine and hence PMWNF to be obtained. In addition, a personal computer was programmed and interfaced to the smoking machine as well as to the balance, which results in much improved data acquisition, handling and presentation.

DISCUSSION

(a) Improved Determination of Puff-by-Puff TPM Deliveries

The 44 mm filter holders supplied by Borgwaldt for puff-by-puff determinations weigh ca. 80 g and are made of Perspex. Our previous work had proved that conditioned* 44 mm Perspex holders supplied for the Filtrona smoking machines and weighing about 50 g contain more than 36 mg of water which can all be lost (from the outer surfaces only) by desiccation at room temperature and pressure. Loss of some of this water during the smoking procedure is responsible for the inaccuracy and high variability of TPM and PMWNF determinations using Perspex holders (9).

In order to determine whether the Borgwaldt puff-by-puff holders are susceptible to similar problems, five conditioned holders, sealed with rubber serum caps and rubber bungs at front and rear respectively, were weighed, then stored in a desiccator over freshly-dried silica gel (removing them only for weighing). The changes of the holders with time in the desiccator are recorded in Table 1.

*Wherever 'conditioned' is mentioned in this report, the conditions are 21°C and 60% R.H.

TABLE 1

WEIGHT CHANGES OF FIVE SEALED BORWALDT 44 MM PUFF-BY-PUFF FILTER HOLDERS WITH DESICCATION AT ROOM TEMPERATURE

Weight of Conditioned Holder (g)	WEIGHT LOSS (mg)			
	After 24 Hours	After 48 Hours	After 72 Hours	After 241 Hours
80.7591	33.4	45.9	57.6	81.9
81.3327	34.4	47.2	57.0	82.6
81.4921	34.6	46.7	57.1	82.2
81.4774	34.9	46.9	56.3	82.3
79.9977	35.1	47.9	56.4	83.4
Mean Weight Loss (mg)	34.5	46.9	56.9	82.5

The results show that sealed, conditioned Borgwaldt puff-by-puff holders contain an average of >82 mg of water which can be lost, via the outer surfaces only, at room temperature and pressure. The rubber seals have previously been shown to be substantially non-hygroscopic (9). These findings, coupled with the undesirability of determining weight changes of a few milligrams against a 'background' tare of 80 g, render the Borgwaldt holders unsuitable for accurate puff-by-puff TPM and PMWNF determinations.

The use of non-hygroscopic holders, such as the Gelman Smoke Filter which weighs less than 6 g, has been found to be satisfactory in such situations (9). PTFE adaptors were therefore made to connect the front of the Gelman holder to the Borgwaldt RM20C/S segment behind the puffing port, and to connect the rear of the Gelman holder to the pneumatic module (Figures 1 and 2 respectively). These adaptors were designed so that the holder could be changed within six seconds, as required by the earlier procedure (6).

The use of the Gelman holder with these modications proved entirely successful.

(b) Improved Extraction of TPM Prior to Water and Nicotine Determination

Conventional methods of extracting water (7) and nicotine (8) from small quantities of TPM have been shown to have significant drawbacks which were overcome by the development of an 'in situ' extraction procedure (10). This procedure was devised for non-hygroscopic filter holders - specifically the Gelman filter. There would be little point in applying it to hygroscopic holders such as the Borgwaldt puff-by-puff type because of the possibility of water exchange between holder and solvent.

The 'in situ' extraction of TPM in Gelman holders with specially-dried isopropanol (5 ml) is therefore much more suitable for puff-by-puff work than standard methods of extraction.

The procedures are fully described elsewhere (11) and were adopted with no problems as part of the revised puff-by-puff methodology.

(c) Improved Determination of Water and Nicotine in TPM Extracts

It has been shown (10) that coulometric determination of water in TPM has several advantages over the widely used gas chromatographic procedures (7), especially when small quantities of TPM (and hence of water) are involved.

Nicotine determinations can be carried out using established auto-analyser (8), or gas chromatographic techniques (12).

Full details, including comparisons of the effects of standard and revised procedures on PMWNF deliveries are given in an earlier report (10).

(d) The Borgwaldt 'Twin-Filter' Smoke Trap

Borgwaldt have developed an automatic filter pad/holder changer ('twin filter' smoke trap, Part No. R25.00) which enables puff-by-puff determinations to be carried out on a fully-loaded 20-port Borgwaldt machine (Figure 3).

The device consists of a motor-driven carousel with two sockets for Cambridge filter holders. One holder collects mainstream smoke and, if a bag is attached, vapour phase, while the other is idle or being changed ready for insertion into the smoke stream while the smoking machine is indexing between ports. Therefore, almost a minute is available to change filter holder assemblies rather than the six seconds in the previous method.

By changing pads twice per revolution of the smoking machine, it is possible to perform puff-by-puff TPM and vapour phase determinations on two different types of cigarettes during the same smoking run. If the same two filters are used it is possible to determine total TPM and vapour phase on two different types of cigarettes during the same run. The Borgwaldt Twin Filter Smoke Trap would need to be modified to accept non-hygroscopic holders for TPM determinations, and its cost may not be justified unless a high sample throughput is envisaged.

(e) Improved Automated Data Acquisition and Data Handling

An earlier report (6) described the capabilities of software (prepared by Vlassek) coupled with a R&DC Basic Programme which enables an Apple II Microcomputer to calculate the mean weight of TPM-per-puff when interfaced to a fast automatic balance (e.g. the Mettler AK160). This data handling package is limited and may give rise to errors. At the start of smoking, the operator is required to enter the number of cigarettes (usually 15) and the number of puffs to be accommodated in the run (usually a higher puff number than expected is entered). The programme was designed to cater for whole puffs and divides the total TPM collected per revolution of the carousel by the number of cigarettes loaded at the start of the run. Towards the end of the smoke run, some cigarettes would be only part-puffed or not puffed at all as their butt mark is reached. If the run is not terminated before this occurs, the tabulation and plot of TPM *vs* puff number emerging from the printer will contain errors.

For most cigarettes, the TPM-per-puff increases with increasing puff number and consequently these later puffs or part puffs make a significant contribution to the total TPM delivered. Therefore, if the smoke run is stopped as soon as one cigarette reaches the last puff before its butt mark, in order to avoid erroneous tabulation or plotting by the programme, then the data generated cannot be related to any total TPM/cigarette yield which may have been determined on these cigarettes using standard procedures (13, 14, 15).

The last puffs and part puffs may be important in formulating or verifying theories of smoking mechanics, or for validating the effectiveness of filters designed to produce a level profile. The limitations of the previous programme are such that this vital information cannot be obtained.

The development of a more versatile programme which overcomes these problems and which has been designed to operate with or without the Borgwaldt pad changer has been developed and is available on request.

CONCLUSIONS

The procedures developed for the determination of puff-by-puff deliveries produce results which are more accurate and precise than earlier techniques. Mean puff-by-puff deliveries of TPM, water, nicotine and hence PMWNF can now be determined on cigarettes delivering from 0.1 to more than 3 mg TPM/puff at puff volumes ranging from 10 to 130 ml. With some modifications puff durations of up to 4 seconds can be accommodated. Furthermore, meaningful puff-by-puff PMWNF determinations on single cigarettes (delivering more than ca. 10 mg/cig) are now feasible.

In addition to the improvements made to the analytical procedures, improvements have been made to the data acquisition, data handling and data presentation.

The procedures have been used extensively in R&DC and are now available as a standard analytical method.

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15. Determination of TPM Deliveries from Ultra-Low Delivery Cigarettes using the Filtrona Model 300 Smoking Machine, BATUKE R&DC Standard Method C.1.4.

FIG. 1 T. 202

PTFE ADAPTOR FOR PUFF-BY-PUFF COLLECTION OF PARTICULATES USING THE BORGWALDT RM 20 C/S SMOKING MACHINE

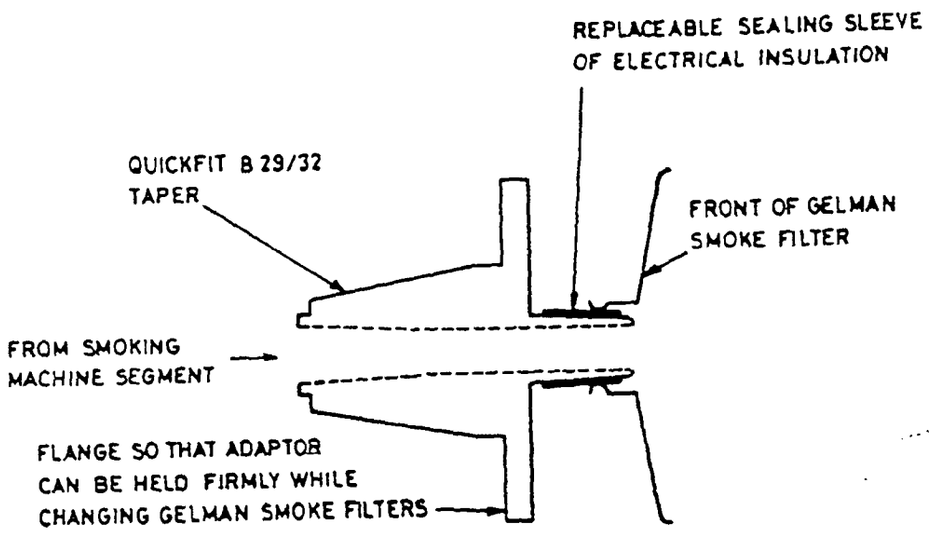

FIG.2

PTFE ADAPTOR FOR CONNECTING BACK OF GELMAN SMOKE FILTER TO THE BORGWALDT PNEUMATIC MODULE

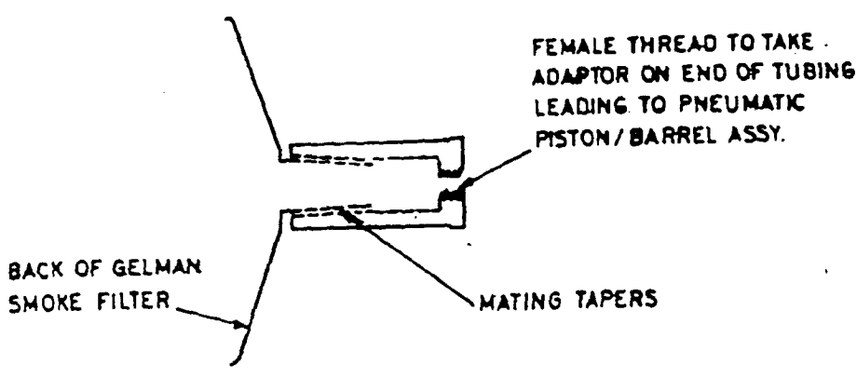

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FIGURE 3

BORGWALDT AUTOMATIC HOLDER CHANGER (R 25.C0)
MOUNTED ON AN RM 20/CS SMOKING MACHINE