

WITHDRAWN



B.A.T. (U.K. and Export) Limited
RESEARCH & DEVELOPMENT CENTRE

METHOD C.5.2 (N)
AMENDMENT 1

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DETERMINATION OF TOTAL PARTICULATE MATTER AND CARBON MONOXIDE
IN CIGARETTE SMOKE USING THE FILTRONA 300 SMOKING MACHINE
FITTED WITH THE ATCOM SYSTEM

SUMMARY

Cigarettes are marked for insertion and butt length, conditioned at 22°C/60% R.H., and total particulate matter (TPM) in the smoke determined using an automatic smoking machine. This method is applicable only when the Filtrona Model 300 smoking machine is used. It includes the setting-up procedure, the operation of the machine, and associated details concerning the Cambridge filter holder and pads. A Filtrona ATCOM (Automatic Carbon Monoxide Measurement) system attached to the smoking machine is used to collect the cigarette smoke vapour phase and subsequently to measure the carbon monoxide concentration using a non-dispersive infra-red analyser specially calibrated.

APPARATUS

Conditioning chamber at 22°C/60% RH.

Conditioned Smoking Environment, 22°C/60%RH

Filtrona Model 300 smoking machine fitted with the Filtrona ATCOM system (Note 1).

Vapour phase collection bags.

Small vacuum pump, to evacuate sample bags.

Thermometer.

Barometer.

44 or 55 mm Cambridge filter holders (LDPE labyrinth seal type).

44 or 55 mm Cambridge filter pads.

Silicone rubber washers for labyrinth seals.

Cambridge filter holder end caps (LDPE).

Thread, 100% cotton, designated 40 denier.

Puff volume indicator.

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Analytical Balance (readability 0.1 mg).

Temperature and Relative Humidity Measuring Instrument

N.B. For cigarettes with a TPM delivery of less than 25 mg per cigarette the 44 mm CF holders and pads are recommended. For cigarettes with higher yields the 55 mm holders and pads should be used.

A labyrinth seal consists of 3 silicone rubber washers held in a plastic cap which attaches to the front of the CF holder. They are assembled with the flat surface facing the CF pad.

The silicone washers are available with 3 sizes of orifice, designated A, B and C, to hold different sizes of cigarettes.

Washer size A - circumference range less than 24.3 mm

Washer size B - circumference range between 24.3 and 26.5 mm

Washer size C - circumference range greater than 26.5 mm

It is important that the correct size washers are used for the cigarettes to be smoked.

SAFETY PRECAUTIONS

CARBON MONOXIDE IS TOXIC! ALL GASES SHOULD BE VENTED INTO AN EXHAUST SYSTEM, PARTICULAR CARE BEING TAKEN WITH THE DISPOSAL OF THE STANDARD GASES.

REAGENTS

Detergent solution, e.g., 15% Teepol L in water, for use with the puff volume indicator.

Calibration gas mixtures - at least three standard gases of known concentration of carbon monoxide in nitrogen covering the range normally found in cigarette smoke. Suggested concentrations are 2%, 4% and 6% v/v. Gold Star mixtures in aluminium cylinders are recommended and a certificate of analysis must be obtained from the supplier.

N.B. Gold Star mixtures are expensive and supplied in medium sized cylinders only. Thus frequent replacements are necessary. As an alternative, a single Gold Star standard (usually the 4%) can be used to calibrate three secondary non-Gold Star standards at 2%, 4% and 6% v/v carbon monoxide in nitrogen which can be obtained in large cylinders. These latter are then used for the regular calibration of the ATCOM analyser. The calibration of the secondary standards should be carried out weekly.

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PROCEDURE

1. Preparation of the Cigarettes

1.1 Select, at random, from the laboratory sample, sufficient cigarettes to allow for smoking and a moisture test. This number should be a minimum of $(5n+2)+20$, where n = the number of smoking machine ports to be used for the particular sample. Take care to exclude cigarettes which are atypical of the sample, e.g., with damaged ends or torn cigarette paper.

1.2 Using a 5B pencil, mark the cigarettes for smoking as follows:-

1.2.1 First mark: 9 mm from the mouth end. This is the mark to which the cigarettes are inserted into the cigarette holding device on the smoking machine. For plain cigarettes ensure that this mark is made on the cigarette paper lap seam.

1.2.2 Second mark: according to the butt length regime applicable to the sample. Ensure the mark is made on the cigarette paper lap seam. For a regime which uses as a base line the edge of the overtipping (e.g., U.K.) then each cigarette should be marked individually. Alternatively, where the base line is the filter-tip length (e.g., ISO) then it is assumed that all of the tips are of the same length and all the cigarettes marked together.

N.B. All marks should be made using the minimum of pressure and every care taken to avoid puncturing the paper.

1.3 Transfer all of the cigarettes to the conditioning chamber and store at 22°C/60% R.H. (ISO 3402:1978, Condition A) for a minimum of 48 hours.

1.4 Transfer the cigarettes to sealed containers for transport and storage prior to smoking. This also acts as protection against accidental damage to the cigarettes.

1.5 Determine the moisture content (Method B.3.1) of the 20 cigarettes set aside for this purpose as a check on the conditioning stage.

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2. Preparation of the Smoking Machine

- 2.1 Lower the lighter bar and set the smoking machine and ATCOM controls to the following positions:-

Green keyswitch (known as the function keyswitch) to "AUTO"
"FREQUENCY IN SECONDS" to "060".

"COUNTER" switch to "OFF".

Set all puff counters to zero, by using the push-button immediately under each counter.

"BUTT LENGTH" switch to "IN".

"VACUUM" switch to "OFF".

ATCOM module "SAMPLE" switch to "OFF"

ATCOM module "COLLECT" switch to "OFF"

ATCOM module "VAC" switch to "OFF"

ATCOM module "PUMP" switch to "OFF"

The vacuum pump controlled by this switch is for use with the "false lips" type of cigarette holder which requires a vacuum to open the "lips" and allow insertion of the cigarette. The labyrinth seal type of holder does not require this facility. It has been found, however, that by converting the pump from vacuum to pressure (by changing over the outlet connection) it becomes a supply of compressed air useful for cleaning the cigarette lighters of pieces of burnt/unburnt tobacco.

- 2.2 Switch the smoking machine on by depressing the mains switch on the control console. Allow the machine to warm up on automatic cycling for at least 30 minutes.

During the warm up period prepare the CF Holders as detailed in the procedure 3.1 to 3.4.

- 2.3 After the warm-up period switch the function keyswitch to "MAN", the "BUTT LENGTH" switch to "OUT", the "COUNTER" switch "ON" and the ATCOM module "COLLECT" switch to "ON". Press and release the "TEST CYCLE" switch. The machine should now perform one cycle and come to rest. Observe the readings on the counters. The elapsed time indicator should display 02.0 (2 seconds) and each puff counter should be showing 0010. Repeat this test nine more times. The puff counter readings should increase by 10 units for each cycle, i.e., 0020, 0030, etc.

If any adjustment is necessary refer to the smoking machine handbook.

- 2.4 Depress the "LIGHTER" switch and make a note of the time taken for the lighters to reach red-heat (PREHEAT TIME). Subtract the Preheat time from 60. The resultant time is that of which the "LIGHTER" switch should be depressed when manually lighting the cigarettes.

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3. Preparation of Cambridge Filter Holders

- N.B. Surgical gloves should be worn at all times when handling CF holders and pads. Certain surgical gloves, however, must be avoided as they are coated inside and outside, with a white powder, probably talc. Handling the holders or pads with these gloves can introduce contamination.

CF pads and holders are conditioned by storing in the smoking room overnight, the moisture content will reach equilibrium with the ambient condition. When room air is puffed through the pads during smoking there should be no change in the moisture content due to the passage of this air.

While the smoking machine is warming up prepare the Cambridge filter (CF) holders as follows:-

- 3.1 Place a conditioned CF pad into each holder ensuring that the rough side faces the front (i.e., smoke inlet) of the holder.
- 3.2 Fit numbered caps to both front and back of each holder and weigh the holder plus caps. Record the weight.
- 3.3 Remove the numbered caps and store them such that they cannot be contaminated.
- 3.4 Prepare and attach the labyrinth seals. Check that the correct size is used

4. Puff Volume Check

- 4.1 On the smoking machine loosen the knurled screws on the top of the lighter bar and pull all the lighter elements back to the maximum extent. Lower the lighter bar and fit the weighed CF holders into place.
- 4.2 Switch "BUTT LENGTH" to "IN" (or "VOLUME CHECK/MICRO-SWITCH" to "MICRO-SWITCH").
- 4.3 Attach clip to the microswitch shaft of port 1 so that the microswitch is activated and the amber light for port 1 on the control console is extinguished. The ports not being tested at this stage are switched off to prevent unnecessary puffs of air being taken through them.

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- 4.4 Attach to correct size insert (see figure) for the labyrinth seals in the CF holder, to the 10 cm impedance in the volume indicator. Prepare the puff volume indicator by wetting the inside of the tube with the detergent solution to above the top graduation mark. Connect the indicator to the CF holder in port 1, adjust the position of the detergent film to the zero mark then operate the "TEST CYCLE" switch. The puff volume must be $35.0 \pm 0.3 \text{ cm}^3$. If the puff volume is less than 34.7 cm^3 check the CF holder for leaks. Adjust the puff volume if necessary by referring to the instrument operators instructions.
- 4.5 Repeat 4.3 and 4.4 for all remaining ports in turn.
- 4.6. Set ATCOM module "COLLECT" switch to "OFF".
Set ATCOM module "VAC" switch to "AUTO".
After a short pause the pump inside the ATCOM module is actuated to flush the vapour phase collection bags by a sequence of evacuate/fill steps. When the cycle is complete the "END" indicator light will glow.
Set ATCOM module "VAC" switch to "OFF".
Set ATCOM module "COLLECT" switch to ON.

5. Threading the Smoking Machine

- 5.1 Switch "BUTT LENGTH" to "IN" and the counters "ON".
- 5.2 Attach cotton thread to each microswitch. When lacing the thread always start at the left-hand side of the machine and work towards the right.
- 5.3 Take the thread over the top of the shaft via the groove and then wrap clockwise around it until, at the "9 o'clock" position, the cutaway diverts the thread out of the groove. Ensure that the cotton is trapped, under tension, between the end of the shaft and the dished washer. It is essential that the thread is diverted out of the groove and does not continue all the way round it otherwise there will be no "nipping" action from the dished washer to maintain the tension on the thread when it is burnt through on an adjacent port.
- 5.4 Continue looping, with a long continuous thread, all of the remaining switch shafts. As each switch is looped check that the amber channel light for the particular port is extinguished.

6. Positioning of Cigarettes and Lighters

- 6.1 Place the cigarettes into the correct CF holders so that the insertion mark is in line with the front washer of the labyrinth seal. The most effective seal is obtained by pushing the cigarette in just beyond the insertion mark and then pulling forward until the correct position is reached.

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- 6.2 Adjust the position of each CF holder to bring the butt mark on the cigarette in line with and just touching the cotton thread by adjusting the position of the CF holder carrier tube.
- 6.3 Raise the lighter bar carefully. Adjust each element in turn so that it is 1 mm away from the end of the cigarette. Lock each element in position by tightening the knurled screw on the top of the lighter bar.
- N.B. Never allow the lighter element to touch the end of the cigarette before or during heating up or while at red heat, otherwise the cigarette could be damaged and the life of the lighter element could be considerably shortened as a result of pieces of charred tobacco adhering to it.

7. Lighting and Smoking the Cigarettes

- 7.1 Reset all the puff counters to zero.
- 7.2 Switch the function key switch to "AUTO" and watch the elapsed time indicator.
- 7.3 When the indicator shows the value calculated in section 2.4, depress the "LIGHTER" button and hold down. Raise the lighter bar so that when the elapsed time indicator shows 58 seconds the lighter elements are 5-7 cm from the end of the cigarettes. Continue raising the bar so that it reaches its highest position (elements 1 mm from the end of the cigarettes) when the elapsed time indicator returns to zero and the puff cycle commences. Hold the bar in position until the end of the puff then release the "LIGHTER" button and lower the lighter bar.
- 7.4 If any cigarette fails to light, unlock all the lighter elements which lit cigarettes correctly. Pull them back to their fullest extent and lock in position. Re-zero the puff counters on all ports where the cigarette failed to light (Note 2) and check the adjustment, if necessary, of the lighter elements (wait 30 seconds from the end of the lighting puff to allow the element to cool down before repositioning otherwise the tobacco on the end of the unlit cigarette may start to smoulder). Repeat Procedure 7.3.
- N.B. On occasion a cigarette will not completely light over the exposed tobacco area. In this case the filter holder of the affected cigarette should be rotated so that the smouldering part of the cigarette is lowermost. If the entire exposed tobacco area is not smouldering by the time the second puff is taken, smoking on that port should be abandoned.

Also, if some tobacco is pulled away from the cigarette when the lighter bar is withdrawn, smoking on that part should be abandoned.

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- 7.5 Allow the machine to continue cycling. As each butt mark is reached the cotton thread burns through releasing the microswitch which disconnects both the channel and the puff counter. Cut off the burning coal using sharp scissors, leaving the butt in position.
 - 7.6 When all the cigarettes have been extinguished record the number of puffs taken on each channel. Switch the function keyswitch to "MAN", "BUTT LENGTH" switch to "OUT", and the counters "OFF". Operate the "TEST CYCLE" button to take one clearing puff through each butt. Discard the butts.
 - 7.7 Smoke four more cigarettes through each port following the steps from Procedure 5. (Threading the Smoking Machine) to Procedure 7.6 for each cigarette.
8. Final Operations
- 8.1 Remove one CF holder at a time from the machine, remove its labyrinth seal holder and immediately cap front and back, taking care to see that the correct caps are used.
 - 8.2 When all CF holders have been capped, ensure that the "BUTT LENGTH" is "OUT" and the puff counters "ON" before clearing the vapour phase from the machine by depressing the "TEST CYCLE" switch five times, thus taking five more clearing puffs.
 - 8.3 Weigh each CF holder and caps and record the new weight.
9. Calibration of the ATCOM CO Analyser
- N.B. This operation must be performed just prior to the analysis of the collected vapour phase smoke.
- 9.1 Disconnect "PUMP INLET" from ATCOM module. Set analyser module "PUMP" switch to "ON" and purge the analyser with ambient air.
 - 9.2 Adjust the "ZERO" control until the digital display reads zero.
- N.B. There are two apparent zero positions on the ATCOM unit. At the correct zero, clockwise adjustment of the zero control results in a POSITIVE increase in the meter reading. In the second apparent zero setting the numerical reading increases, but the negative indicator is illuminated.
- 9.3 Evacuate completely one sample bag for each of the standard gas mixtures. Fill each bag with a gas mixture, re-evacuate, and re-fill with the same mixture.

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- N.B. Provided the bags are kept air-tight and used only for one calibration gas it is permissible, on the day of the test, to evacuate the remaining previous standard from the bag and refill with the same standard. This will prevent unnecessary wastage of the gases.

When new bags or standard gases are introduced follow the procedure detailed in 9.3 above.

- 9.4 Introduce the 4% standard gas mixture into the analyser by attaching the sample bag to the "PUMP INLET" port and adjust the "SPAN" control such that the digital display reads the known concentration of the standard.
- 9.5 Introduce the other standard gas mixtures in a similar manner and note the digital display reading. If all readings are within 0.1% v/v of the certified value then the instrument linearity is acceptable, otherwise refer to the instrument handbook.
- 9.6 Re-connect the "PUMP INLET" to the ATCOM module.

10. Measurement of Carbon Monoxide Levels

- N.B. Before measurement commences record the ambient temperature and atmospheric pressure in the smoking laboratory.
- 10.1 Set analyser module "PUMP" switch to "ON".
Set ATCOM module "COLLECT" switch to "OFF".
Set ATCOM module "SAMPLE" switch to "ON".
Each collection bag is now sampled in turn and the carbon monoxide concentration displayed and printed.
- 10.2 After the last bag has been sampled, set ATCOM module "SAMPLE" switch to "OFF" and analyser module "PUMP" switch to "OFF".
- 10.3 Set ATCOM module "VAC" switch to "AUTO" and on completion of cycle set to "OFF".

CALCULATION

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1. Total Particulate Matter

Calculate the delivery of TPM (mg/cigarette) from the formula:-

$$\text{TPM (mg per cig.)} = \frac{1000 (W_2 - W_1)}{N}$$

where, W_1 = weight (g) of clean CF holder and caps,

W_2 = weight (g) of CF holder, TPM and caps,

N = number of cigarettes smoked.

2. Carbon Monoxide

The recorded results for carbon monoxide concentration must be corrected for the dilution effect of the clearing puffs, as follows:-

$$\text{Corrected concentration} = \frac{C \times n}{L} \% \text{ v/v}$$

where, C = recorded concentration (% v/v) of carbon monoxide,

n = total number of puffs, (lit plus clearing, i.e., lit + 10),

L = total number of lit puffs.

Calculate the carbon monoxide delivery (mg/cigarette) from the formula:-

$$\frac{C \times 35 \times n \times P \times 273 \times 28}{N \times (T+273) \times 100 \times 760 \times 22.4} = \frac{C \times n \times P \times 0.157}{N \times (T+273)}$$

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where, C = recorded concentration (% v/v) of carbon monoxide,
35 = puff volume (cm³),
n = total number of puffs, lit plus clearing,
P = atmospheric pressure (mm mercury),
273 = 0°C in °K,
28 = molecular weight of carbon monoxide,
N = number of cigarettes smoked,
T = laboratory temperature (°C),
760 = atmospheric pressure (mm mercury) at NTP,
22.4 = volume (litre) occupied by gramme molecular weight carbon monoxide.

NOTES

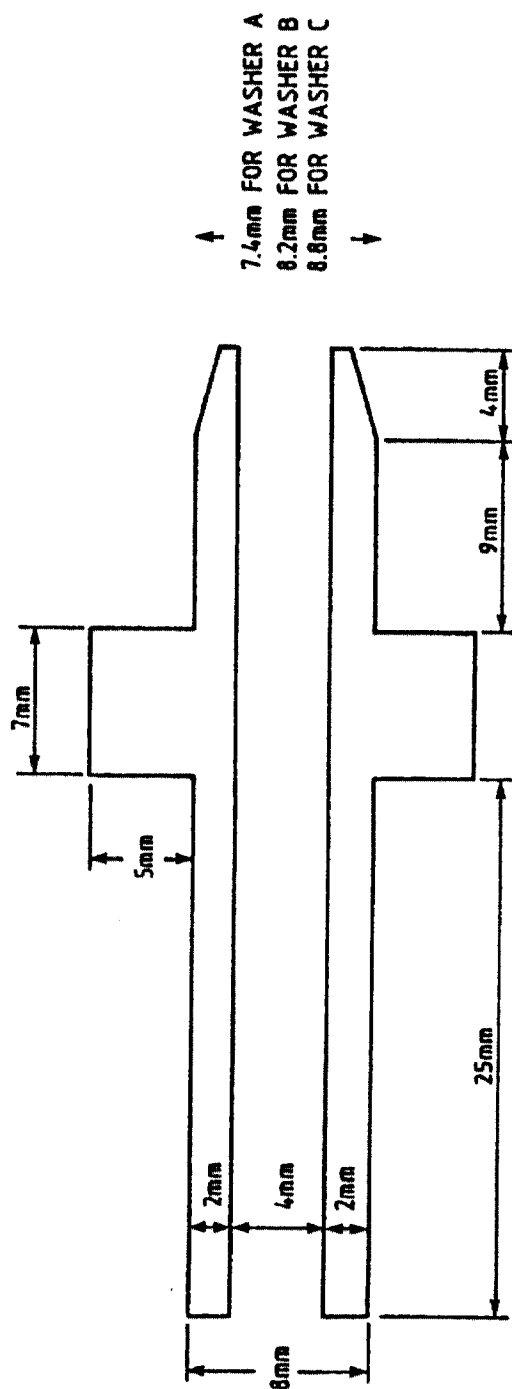
1. The air-flow through the smoking machine tri-duct system should be set at 46 linear metres (150 linear feet) per minute for the two smaller outside ducts and 76 linear metres (270 linear feet) per minute for the larger central duct. These settings give the correct air-flow over the cigarettes of 1.2 - 2.1 linear metres (4 - 7 linear feet) per minute.

If possible the ATCOM unit should be left on continuously, 24 hours a day for improved stability.

2. The puff taken when a cigarette fails to light should be included in the carbon monoxide calculations as an unlit clearing puff.

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INSERT FOR USE DURING PUFF VOLUME ADJUSTMENT



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