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A Smoking Machine Attachment for the Analysis of Carbon Monoxide, Carbon Dioxide and Nitric Oxide in the Vapour Phase of Cigarette Smoke*

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INTRODUCTION

The sampling of vapour phase of cigarette smoke has been carried out by several investigators (1 — 5). The use of specific analysers for the analysis of carbon dioxide, carbon monoxide and nitric oxide has also been described in some detail (6 — 8). However, the sampling methods described were found to be unsuitable for routine use. The major problems with these methods were contact of the vapour phase with silicone oil, puff-profile distortion, large dead volumes, lack of butt termination facilities, and a long hold-up time before analysis.

To overcome these problems, fundamental alterations to the cigarette smoking machine CSM 300 (Cigarette Components Ltd.) would have been necessary and, therefore, an eight-channel vapour-phase smoking attachment (VPSA) was developed. The attachment involves the interposition of a slave syringe (9) between the channel of the smoking machine and the Cambridge filter holder (Fig. 1). A puff taken by the smoking machine displaces the slave piston by 35 ml and the vapour phase is drawn into the syringe. Using a system of 3-way solenoids, the slave piston can be blown to the end of the syringe displacing that vapour phase into a suitable storage vessel, trap, or gas analyser. The butt termination system used on the CSM 300 is also modified for use with the VPSA.

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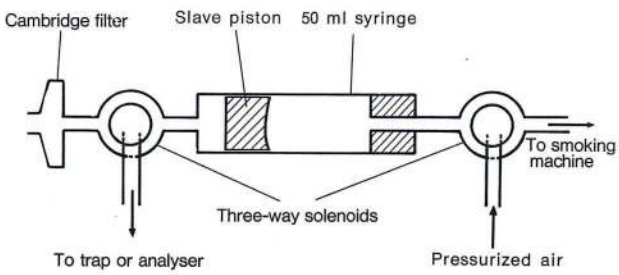
Figure 1.



APPARATUS

Figure 2 is a photograph of the apparatus for smoking eight cigarettes simultaneously. With valves V1 — V16 energized, the vapour phase is drawn into the eight syringes by eight channels of the CSM 300. Immediately the puff is completed, V1 — V16 are de-energized. Pressurized air tapped from the CSM 300 rotary pump passes into manifold M2 via V19 and V18 and pushes the slave pistons to the end of the syringes. This expels the vapour phase into syringe S9 via solenoid V17.

Immediately all the pistons reach the end of the syringes, V17 — V19 are de-energized. V17 then allows the gas analyser to sample from S9, V19 allows the pressurized air to vent, and V18 vents the manifold M2 to atmosphere. V1 — V16 are then energized for the next operation and the cycle is repeated.

Slave Syringes (S1 – S8)

These are standard CSM 300 smoking syringes with 30 mm cut from the end of the plunger to provide the slave piston. The cut edges of the piston are bevelled and polished.

Solenoid Valves (V1 – V19)

3-way solenoid valves (Skinner Europa No. V 53 DA 1075) fitted with suitable tube connections. The lower ports are labelled IN and CYL and are interconnected in the energized mode. In the de-energized mode CYL and the top port are interconnected. The positions of the valves are illustrated in Figure 2, the correct alignment of the valves is as follows:

V1 – V8	IN	ports towards holder bar,
V9 – V16	CYL	ports towards syringes,
V17	CYL	port connected to S9,
V18	CYL	port connected to M2,
V19	IN	port connected to IN port of V18.

Manifolds (M1 and M2)

The manifolds interconnect the top ports of V1 – V8 and V9 – V16 and are made of glass. M1 is constructed of 1.5 mm inside diameter, 6 mm outside diameter tubing to keep dead volume to a minimum.

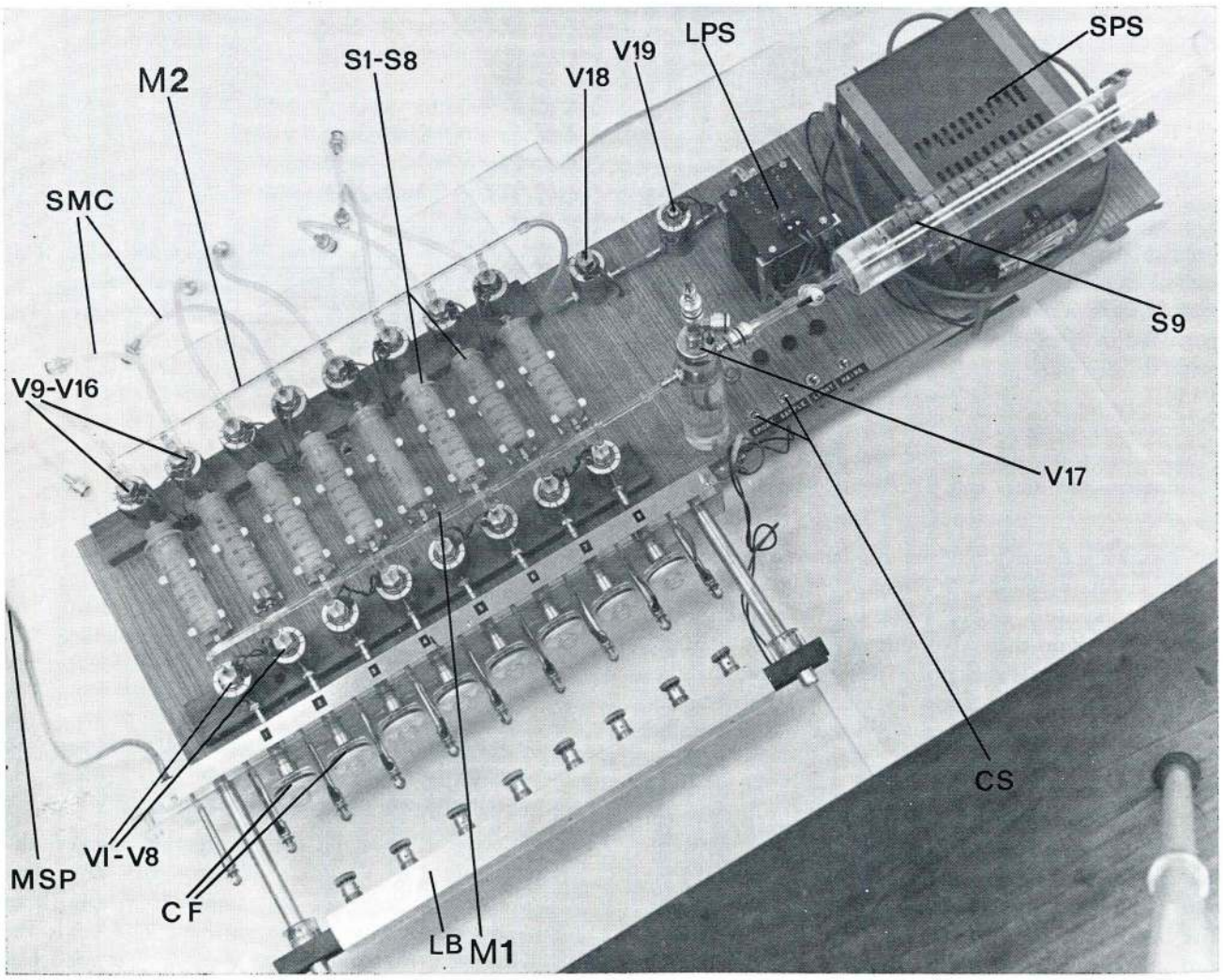
Power Supply

The solenoids require a 10 A 24 V supply. V1 – V16 are wired in parallel and energized using a 10 A biased toggle switch. V17 – V19 are wired in parallel and energized using a 5 A biased toggle switch (switches are biased to the OFF position). The lighters are wired in series to a 60 V supply fed via a suitable switch.

Filter Holder Bar and Termination Assembly

The VPSA described has one half of a microswitch/holder bar from a CSM 300 mounted in front of solenoids V1 to V8. Holders and microswitches of channels one and ten were removed and the remaining holes used to hold the lighter bar supports. The microswitches plug is interchangeable with the plug on the CSM 300 to which the VPSA is fitted and controls channels 2 to 9.

Figure 2. Plan view of attachment key.



SMC : Smoking machine connectors
LPS : Lighter power supply
SPS : Solenoid power supply
CS : Control switches
LB : Lighter bar
CF : Cambridge filter
MSP : Microswitch plug

Storage Syringe (S 9)

This is a specially made precision bore syringe of 500 ml capacity with a freemoving Perspex slave piston (supplied by Southern Scientific Suppliers, 87 Hare Street, Springs., Harlow, U.K.).

OPERATION

The VPSA described was constructed to fit the bed of a CSM 300 and is attached as follows: The IN ports from V 9 — V 16 are plugged into channels 2 to 9 and pressurized air from the CSM 300 vacuum pump is attached, via a flow controller to the CYL port of V 19. The left hand microswitch plug is removed and the plug from the VPSA fitted in its place. The analysers are connected to the top port of V 17 using standard gas couplings.

The CSM 300 is operated normally and V 1 — V 16 are energized a few seconds before the puff cycle. On completion of the puff cycle V 1 — V 16 are de-energized and V 17 — V 19 energized. The pistons will automatically return to the end of the syringes forcing the vapour phase (or air during puff-volume checking) into the storage syringe S 9. V 17 — V 19 must be de-energized immediately the last piston returns to zero, failure to do so may result in dilution of vapour phase by air forced past the slave pistons due to pressure increase in M 2. Once V 17 — V 19 are de-energized the gas analyser(s) will draw the vapour phase from S 9 via V 17.

Having completed puff-volume checks and analyser calibration the VPSA is loaded with the cigarettes to be analysed. Even lighting is essential during the first puff cycle. Two switching operations are required for each puff until all the channels have terminated, the clearing puffs are taken by over-riding the micro-switches.

ANALYSIS

The following gas analysers were coupled to the VPSA:

(1) Carbon monoxide: Grubb Parsons IRGA 20 CO Dual Range 0—3 % and 0—15 %
(2) Carbon dioxide: Grubb Parsons IRGA 20 CO_2 Dual Range 0—3 % and 0—15 %
(3) Nitric oxide: BOC huminox Model 860100 Multi-range 1—10,000 VPM.

Each analyser was coupled to a chart recorder and operated according to the manufacturer's instructions.

Calibration Mixtures: The following gas mixtures (with test certificates) were obtained from BOC Special Gases Ltd.:

(1) 100 VPM NO, 2 % CO, 5 % CO_2 balance gas nitrogen,
(2) 500 VPM NO, 5 % CO, 8 % CO_2 balance gas nitrogen,
(3) 1000 VPM NO, 8 % CO, 12 % CO_2 balance gas nitrogen.

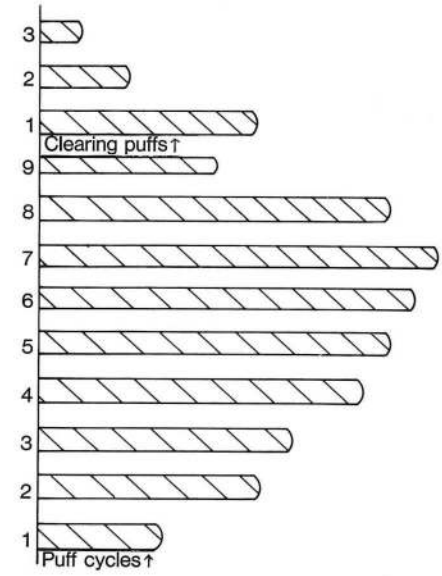
Carbon monoxide has been found to react with steel cylinders forming iron carbonyl which interferes with the nitric-oxide readings. If combined standard gases are used, calibrate the nitric-oxide analyser with the convertor on to breakdown the iron carbonyl. On no account use the convertor for measurement of nitric oxide in the smoke vapour phase.

Analyser Calibration and Vapour-Phase Sampling: Connect the analysers to the top port of V 17. Fill S 9 to 280 ml with calibration gas through the IN port of V 17, adjust the sampling rates of the analysers to give optimum response, and empty S 9 in about 30 seconds. Draw calibration graphs from the recorder peaks obtained from the three calibration mixtures.

The last one or two puffs during smoking can produce less gas phase due to butt termination of some channels during the final puff cycles. This volume of vapour phase may be inadequate for optimum analyser response. Therefore determine the minimum sample volume required by the analyser by filling S 9 with varying volumes of a calibration gas. Reference to the individual channel puff counts will indicate which cycles should be eliminated from the calculation of component concentration.

Using the analysers described for the simultaneous measurement of carbon monoxide, carbon dioxide and nitric oxide puff cycles producing a less than 90 ml were ignored. However, no correction is made to the total puff count figure which is used to calculate weight of gas delivered. Figure 3 shows a typical recorder profile from each analyser coupled to the VPSA. One clearing puff has been found suitable for carbon monoxide and carbon dioxide when analysed individually. Small quantities of nitric oxide however, appear to be absorbed by the particulate matter and apparatus tubing, therefore three clearing puffs were taken.

Figure 3. An example of the puff profiles obtained from the recorder.



CALCULATION

For each gas, the individual puff height values (including that of the clearing puff) were totalled and the result divided by the number of puff cycles measured (not including the clearing puff).
The percentage by volume of carbon monoxide (% CO) and carbon dioxide (% CO_2) and the average concentration by volume of nitric oxide in volumes per million (VPM NO) were read from the respective calibration graphs.

Then

$$\text{CO mg/cig.} = \frac{\%\,\text{CO} \times \text{TC} \times 0.01965 \times \text{P}}{\text{T}}$$

$$\text{CO}_2\ \text{mg/cig.} = \frac{\%\,\text{CO}_2 \times \text{TC} \times 0.03088 \times \text{P}}{\text{T}}$$

$$\text{NO}\ \ \mu\text{g/cig.} = \frac{\text{VPM NO} \times \text{TC} \times 0.00211 \times \text{P}}{\text{T}}$$

where,

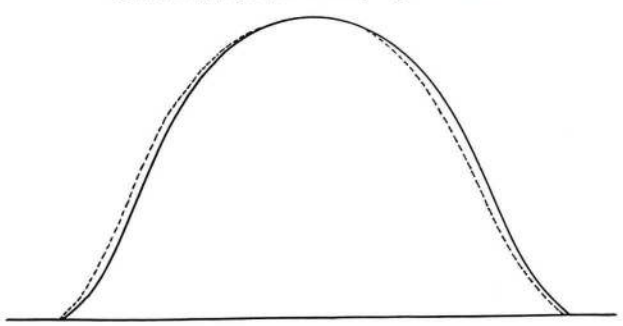
TC = total no. of puff counts for 8 cigarettes,
T = temperature (°K) during analysis,
P = atmospheric pressure (mmHg) during analysis.

RESULTS AND DISCUSSION

The VPSA eliminates the major problems involved in sampling cigarette vapour phase.
Simultaneous analysis for NO, CO and CO_2 will give very accurate results providing each analyser draws enough sample for optimum response. Single component analysis may be necessary if a gas analyser requires large sample volumes. An accurate total puff-count figure is essential in the determination of vapour-phase yields, and the microswitch assembly provides a very accurate puff count and termination facility. In order to achieve consistent puff counts it is necessary to prevent draughts during smoking.
Puff volumes set at 35.5 ml on the CSM 300 gave volumes of between 34.7 ml and 35.1 ml on the VPSA giving a small change in the puff profile (Figure 4). Only minor maintenance should be necessary, over one hundred determinations were performed on the VPSA before the syringes were cleaned. Routine puff-volume checks and visual inspection of the slave pistons during operation will show any tendency on the part of the pistons to stick. The VPSA can also be used for trapping other vapour-phase components by replacing the storage syringe S 9 with adsorbant, liquid and cold trapping systems.

Figure 4.

———— CSM 300
— — — — CSM 300 with gas-phase sampling attachment.



SUMMARY

An eight-channel smoking attachment for the collection of the vapour phase from cigarettes has been developed. The utilisation of the device for the simultaneous puff by puff measurement of carbon monoxide, carbon dioxide and nitric oxide is described. The attachment has been constructed for use with the CSM 300 smoking machine but could readily be adapted for use with other smoking machines.

ZUSAMMENFASSUNG

Es wurde eine Abrauchzusatzvorrichtung mit einem Rauchkopf für acht Cigaretten entwickelt, mit dem die Dampfphase des Cigarettenrauches aufgefangen werden kann. Die Verwendung des Gerätes für die gleichzeitige Pro-Zug-Messung von Kohlenmonoxid, Kohlendioxid und Stickoxid wird beschrieben. Die Vorrichtung wurde für die Rauchmaschine CSM 300 konzipiert, kann aber auch anderen Rauchmaschinen leicht angepaßt werden.

RESUME

On a conçu un accessoire à huit canaux, permettant le recueillement de la phase vapeur de la fumée de cigarette. L'utilisation de cet instrument est décrite. Il permet la mesure simultanée, et bouffée par bouffée, du monoxyde et du dioxyde de carbone ainsi que de l'oxyde d'azote. L'accessoire a été construit pour la machine à fumer CSM 300, mais pourrait facilement être adaptée à d'autres machines à fumer.

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Note: An agreement has been reached with Cigarette Components Limited, U.K., for them to manufacture and sell this instrument.

Acknowledgement

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