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SIMULTANEOUS DETERMINATION OF TOTAL ALIPHATIC
ALDEHYDES, HYDROGEN CYANIDE, ACRYLEIN, PHENOL, NICOTINE
AND STEAM-VOLATILE ACIDS IN WHOLE SMOKE.

PROJECT NO. 0331-01

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ABSTRACT

A method has been developed for the simultaneous determination of acrolein, aldehydes, hydrogen cyanide, phenol, nicotine and steam volatile acids. Greater accuracy is obtained by applying the established procedures to aliquots of the whole smoke condensate from ten cigarettes, rather than by using the smoke condensate from a separate set of five cigarettes for each analysis.

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INTRODUCTION

In the previous standard methods for the determination of hydrogen cyanide, total aliphatic aldehydes, acrolein, total particulate matter (T.P.M.), nicotine and total phenols in cigarette smoke (procedure, November 19, 1965) a set of five cigarettes was smoked, TPM, nicotine and phenols being determined from Cambridge filters, and aldehydes, acrolein and HCN from the "vapour phase". It has since been shown that such determinations of vapour phase components are erroneous due to absorption in the particulate phase on the filter (1). Procedures have subsequently been developed for the determination of HCN (1), total aliphatic aldehydes (1) and acrolein (2) in whole cigarette smoke. It was felt that these, together with standard or modified methods for nicotine (3) and phenols (4) would give a useful alternative scheme to the above, except that the smoking of 15 cigarettes for each set of determinations would be necessary, compared with 5 previously. In addition the steam-volatile acids in whole cigarette smoke could be determined by smoking a further 5 cigarettes (5). In the present work a method has been developed by which hydrogen cyanide, aliphatic aldehydes, acrolein, phenol, nicotine and steam-volatile acids can be determined in whole cigarette smoke using the whole smoke condensate from only 10 cigarettes. An outline of this modified procedure is given in the appendix.

Comparative analyses using separate smoke condensates and a single condensate were obtained using conditioned 7 $\frac{1}{2}$ mm. plain Check cigarettes, selected at 1095 \pm 15 mg. The cigarettes were smoked under standard I.T. Co. conditions to a 25 mm. butt.

A. Determinations using Separate Smoke Condensates

The smoke from four sets of five cigarettes was trapped in the normal way, and the condensates used separately to analyse for the following components by the established procedures (a) total aliphatic aldehydes and HCN; (b) acrolein; (c) phenol and nicotine; (d) steam-volatile acids.

B. Determinations using a Single Smoke Condensate

The smoke from ten cigarettes was trapped in a cooled spiral, coated with 9:1 v/v methanol:water, and an aqueous methanolic solution of this condensate prepared. Aliquots of this solution were then analysed as described in the appendix.

RESULTS AND DISCUSSION

Check No. 7 cigarettes were used in the first study (Tables I and II), while for later analyses, Check No. 8 cigarettes were used (Tables III and IV). The standard deviations for individual analyses were smallest when aliquots of the same whole smoke condensate were used for each analysis. Thus although fewer cigarettes were smoked for the combined analyses, greater accuracy was obtained, possibly through the reduction of cigarette variability as a source of error.

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TABLE I

DETERMINATIONS IN SEPARATE SMOKE CONDENSATES FROM FIVE C.E. 7 CIGARETTES

Set No.	Acrolein µg/cigt.	Aldehydes µg/cigt.	Hydrogen Cyanide µg/cigt.	Phenol g/cigt.	Nicotine mg/cigt.	Steam-Volatile Acids mg/cigt.
1	87.2	1158.3	324.7	234	1.70	1.02
2	85.8	1230.8	305.0	240	1.69	1.11
3	80.8	1204.1	338.2	212	1.62	1.13
4	95.8		331.9	204	1.62	1.04
5			305.0			1.06
6			328.1			
7			353.2			
\bar{x}	87.4	1197.7	326.6	223	1.66	1.07
Standard Deviation	6.2	36.6	17.4	17.2	0.044	0.047
Coefficient of Variation %	7.14	3.06	5.3	7.73	2.63	4.39

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TABLE II
DETERMINATIONS IN ONE SHORE CONDENSATE FROM TEN C.K. 7 CIGARETTES

Set No.	Acrolein µgm/cigt.	Aldehydes µgm/cigt.	Hydrogen Cyanide µgm/cigt.	Phenol µgm/cigt.	Nicotine mg/cigt.	Steam-Volatile Acids µg/cigt.
1	79.7	1267	332.5	202	1.65	1.09
2	100.0	1348	339.7	210	1.70	1.02
3	90.7	1276	340.0	203	1.66	0.96
\bar{x}	90.1	1297	337.4	205	1.67	1.02
Standard Deviation	10.2	44.4	4.3	4.4	0.03	0.065
Coefficient of Variation %	11.3	3.42	1.26	2.13	1.58	6.37

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TABLE III

DETERMINATIONS IN SEPARATE SMOKE CONDENSATES FROM FIVE C.K. 8 CIGARETTES

Set No.	Acrolein $\mu\text{g}/\text{cigt.}$	Aldehydes $\mu\text{g}/\text{cigt.}$	Hydrogen Cyanide $\mu\text{g}/\text{cigt.}$	Phenol $\mu\text{g}/\text{cigt.}$	Nicotine $\text{mg}/\text{cigt.}$	Steam Volatile Acids $\text{mg}/\text{cigt.}$
1	102.8	1267.5	520.1	250	1.69	1.27
2	139.8	1378.2	468.8	222	1.57	1.17
3	125.8	1240.7	545.7	206	1.68	1.18
4	118.0	1308.2	541.3	254	1.69	1.19
5	108.0	1058.0	490.6	270	1.74	1.27
6	107.8	1344.0	522.8	278	1.72	1.19
7	127.8	1260.0	525.0	286	1.72	
8	120.0	1316.0	562.5	289	1.72	
9	104.8				1.72	
\bar{x}	117.2	1271.6	522.1	257	1.69	1.21
Standard Deviation	12.5	97.6	30.3	29.7	0.026	0.046
Coefficient of Variation %	10.6	7.67	5.79	11.5	1.51	3.80

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TABLE IV

DETERMINATIONS IN ONE SMOKE CONDENSATE FROM TEN C.K. 8 CIGARETTES

Set No.	Acrolein $\mu\text{g}/\text{cigt}$	Aldehydes $\mu\text{g}/\text{cigt}$.	Hydrogen Cyanide $\mu\text{g}/\text{cigt}$.	Phenol $\mu\text{g}/\text{cigt}$.	Nicotine mg/cigt .	Steam Volatile Acids mg/cigt .
1	124.0	1488	528	212	1.75	1.22
2	126.7	1565	557	246	1.78	1.22
3	114.6	1420	538	248	1.77	1.27
4	105.2	1418	530	262	1.71	1.27
5	119.0	1435	567	236	1.72	1.33
6	116.0	1445	565	248	1.73	1.27
\bar{x}	117.6	1462	547.5	242	1.74	1.26
Standard Deviation	7.6	38.1	17.6	16.9	0.028	0.04
Coefficient of Variation	6.49	2.60	3.22	6.98	1.61	3.17

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APPENDIX

PROCEDURE FOR DETERMINATION OF ACROLEIN, ALDEHYDES

HYDROGEN CYANIDE, PHENOL, NICOTINE AND STEAM-VOLATILE

ACIDS FROM ONE SMOKE CONDENSATE

1. Smoke ten cigarettes, each conditioned and selected at their average or target weight ± 15 mg. to a 23 mm. or specified butt length under standard or specified smoking conditions.
2. Collect the smoke in a spiral trap which is internally coated with a 1:9 (v/v) water:methanol mixture and cooled by immersion in solid carbon dioxide and acetone.
3. Wash the spiral, containing the smoke solution, successively with several portions of a 1:9 (v/v) water:methanol mixture, transfer the washings to a 100 ml. volumetric flask, and make up to volume with water:methanol mixture.

Determination of Aldehydes and Hydrogen Cyanide

4. Pipette a 25 ml. aliquot, (paragraph 3), into a dry, clean Claisen flask and steam distil.
5. Collect approximately 180 ml. of distillate in a 200 ml. volumetric flask containing 20 ml. of a 1:9 (v/v) water:methanol mixture, and adjust to the correct volume.
6. Use aliquots (4 ml.) for the aldehyde and hydrogen cyanide determinations following the established procedures.

Determination of Acrolein

7. Transfer 20 ml. of 95% ethanol into a 200 ml. volumetric flask and reserve as distillate receiver.
8. Pipette a 25 ml. aliquot, (paragraph 3), into a dry, clean Claisen flask.
9. Connect the Claisen flask to the distillation apparatus, start the ethanol vapour generator and distil, collecting about 180 ml. of distillate and adjusting to volume.
10. Use aliquots (1 ml.) for the determination of acrolein following the established procedure.

Determination of Phenol and Nicotine

11. Transfer 10 ml. of saturated sodium bicarbonate solution into a 200 ml. volumetric flask, and use as distillate receiver.
12. Pipette a 25 ml. aliquot, (paragraph 3), into a clean Claisen flask containing 20 ml. of 5% sodium hydroxide solution.
13. Add 10 ml. of 20% sulphuric acid solution. Connect immediately to the distillation apparatus and steam distil, collecting about 190 ml. and adjusting to the mark. Do not discard distillate after distillation.

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14. Determine the phenol in a 25 ml. aliquot of distillate by the established procedure.
15. Cool the phenol distilland (paragraph 13) in ice water and add 10 ml. of a solution which consists of 30% sodium hydroxide in saturated sodium chloride solution.
16. Repeat steam distillation, collecting approximately 480 ml. of distillate in a 500 ml. volumetric flask containing 18 ml. 1.2N aqueous hydrochloric acid, and adjust to volume with water.
17. Determine the nicotine by the established procedure.
18. Determine a phenol blank in the same apparatus, by distilling 25 ml. of 1:9 (v/v) water:methanol mixture, to which 20 ml. of 5% sodium hydroxide and 10 ml. of 20% sulphuric acid solution have been added.

Steam-Volatile Acid Determination

19. Transfer a 20 ml. aliquot (paragraph 3) into a 125 ml. separatory funnel.
20. Add 60 ml. of peroxide-free ether and 10 ml. of 0.02 N sodium bicarbonate. Shake and allow the layers to separate.
21. Transfer the aqueous layer into a 50 ml. Erlenmeyer flask.
22. Repeat the extraction with 5 ml. of 0.02 N sodium bicarbonate.
23. Proceed with the acid determination on the combined aqueous layers using the established procedure.

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