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The Characterization of Cigarette Smoke from Cytrel® Smoking Products+ and its Comparison to Smoke from Flue-Cured Tobacco

II. Semi-Volatile Phase Analysis*

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INTRODUCTION

Cigarette mainstream smoke consists of two principal fractions, the vapor phase and the particulate phase. Vapor phase components in the smoke from tobacco, Cytrel, and tobacco/Cytrel blend cigarettes were discussed in Part I of this series, which fully described the cigarettes being tested. The semi-volatile (SV) phase discussed in this article is that portion of the particulate matter which may be vaporized without appreciable decomposition at a defined temperature. Because of its volatility, the SV fraction includes a large proportion of the flavor and aroma contributing components of the smoke. A chromatographic analysis of the SV fraction is useful for characterizing and comparing the smoke from different cigarette blends.

Several techniques have been used by different researchers to obtain the semi-volatile fraction for analysis (1—6). Most of these procedures start by smoking cigarettes through glass fiber Cambridge filters to collect particulate matter from the smoke. The filters are then heated to vaporize the SV components, which are collected and injected into a gas chromatograph (GC) for separation and analysis. The SV fraction is defined according to the temperature used to vaporize the material from the Cambridge filter, typically a specific temperature between 100 and 200 °C.

Data presented in this report compare the semi-volatile components obtained from filtered cigarettes containing 100% tobacco, 100% Cytrel smoking material, and a 50/50 blend of tobacco and Cytrel. A capsule sampling technique (7, 8) was used to vaporize the SV fraction at 130 °C directly into a GC injector for separation and analysis. Graphic plots of the relative peak sizes demonstrate the effect of blending Cytrel with tobacco.

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EXPERIMENTAL

Apparatus

Smoking System: Weight-selected, conditioned cigarettes were smoked on a Keith-Newsome smoking machine (9). A Cambridge filter pad holder containing a 44 mm Cambridge No. 9—86 glass fiber filter pad (Phipps and Bird, Inc., Richmond, Va., U.S.A) was attached to the smoking machine for collection of particulate matter from the smoke. Single cigarettes were closely coupled to the inlet side of the filter with a thin latex sleeve.

Sampling System: A Perkin-Elmer Model MS-41 capsule sampling system was used to encapsulate portions of collected particulate matter for direct vaporization in the GC inlet. This system includes a special injector assembly for the chromatograph, a probe for inserting the sealed capsules into the injector, and a tool for sealing the capsules with a cold-welded seal. The test-tube-shaped aluminum capsules are three millimeters in diameter and seven millimeters long. A sealed capsule has an internal volume of about twenty microliters and is said to withstand internal pressures up to 35 atmospheres (10).

Chromatographic System: Chromatographic separations were made in a 100 ft. × 0.02 in. inside diameter stainless steel support coated open tubular column, coated with Carbowax 20M® liquid phase (Perkin-Elmer Corp., Norwalk, Conn., U.S.A.). The separating column was operated in a Perkin-Elmer Model 900 gas chromatograph equipped with a flame ionization detector (FID), a nitrogen-selective detector, and the MS-41 capsule sampling injection system. A short section of crimped capillary tubing was placed on the exit end of the column to allow operation with an inlet pressure of 46 psig. A minimum volume tee was coupled between

the crimped restrictor and the flame ionization detector, with the bottom of the tee connected to the mass spectrometer ion source vacuum system. A crimp in the bottom of the tee controlled the amount of eluent vapor drawn off for mass spectral analysis. The remainder passed on to the flame ionization detector to produce the general semi-volatile chromatogram. A Perkin-Elmer Model PEP-2 chromatographic data system monitored the output of the FID to measure areas under the chromatographic peaks and their retention times.

In an alternate configuration, the eluent vapor leaving the crimped restrictor was passed through a 50/50 splitter with half going to the FID to produce the general SV chromatogram. The other half went to the nitrogen-sensitive detector to produce a chromatogram showing nitrogen-containing SV components.

Mass Spectrometer System: Mass spectra for identification of separated SV components were obtained on a Varian MAT CH-5 mass spectrometer (MS) coupled to a Varian SS-100 data system. The mass spectrometer was coupled to the gas chromatograph splitter device with a 30-inch length of 1/4-inch outside diameter stainless steel tubing heated at 150–160°C. Electron bombardment spectra were produced at 70 eV with a 300 microampere ionizing current, using magnetic scanning and an electron multiplier detector. Spectra were scanned exponentially from m/e 24 to m/e 350. A new spectrum was digitized and stored on magnetic tape every seven seconds during the chromatographic separation.

Procedure

Cigarette Smoking: Cigarettes were conditioned at 74° ± 2°F and 60 ± 2% relative humidity for at least 48 hours. Conditioned, weight-selected cigarettes were smoked to collect Cambridge particulate matter, taking standard 35 ml puffs of two seconds' duration with an interval of 58 seconds between puffs. Cigarettes were smoked to a butt length of 23 mm, the length of the filter overwrap plus three millimeters. Five tobacco or tobacco blend cigarettes were smoked onto the Cambridge filter for analysis. Because of their low delivery of particulate matter, ten 100% Cytrel cigarettes were smoked to provide a sufficient sample for analysis.

Sample Preparation and Analysis: About two to twelve milligrams of collected particulate matter and glass fibers were peeled from the front surface of the Cambridge filter and sealed into an aluminum sample capsule. The filled, sealed capsule was weighed to the nearest 0.01 milligram prior to inserting it into a spring chuck on the probe-type injection rod.

As the loaded probe was inserted into the GC injector, surrounding air was flushed away by an auxiliary helium gas stream. The probe then passed through a pressure-tight gas lock into the GC injector block, which was heated at 130°C (for the 130°C SV fraction). After heating for one minute, the capsule was punctured by pressing the probe against a hollow spike in the injector. Components vaporized from the Cambridge filter material were passed directly into the carrier gas stream for separation and analysis. After flushing for two minutes, the punctured capsule was withdrawn through the gas lock to avoid continued elution of heavier components. The capsule was cooled and reweighed to estimate the amount of material vaporized into the separating column.

The separating column was initially at room temperature. One minute after puncturing the capsule, the GC oven door was closed. The column heated quickly to 70°C, then programmed at 2.5° per minute to a final temperature of 200°C. Helium carrier gas flow was ten ml per minute, measured at room temperature and atmospheric pressure. Eluent vapors from the separating column were split, drawing about one ml per minute into the mass spectrometer ion source and passing the rest into the GC flame ionization detector to produce the chromatogram. During the GC-MS analysis of semi-volatiles, GC peak data were acquired on a Perkin-Elmer PEP-2 data processor monitoring the flame ionization detector. Repetitive mass spectra were simultaneously acquired by a Varian SpectroSystem computer for later identification of the eluting components. Correlating the mass spectra, the GC retention times and peak areas, and the capsule weight loss provides a semi-quantitative comparison of individual components in the SV fraction. The amounts of a given SV component produced by different cigarette blends may be compared by this method. Information contained within the stored repetitive mass spectra may also be used to locate minor components that may be obscured by major components on the original FID chromatogram.

In an alternate instrument configuration, eluent vapors from the GC separating column were evenly split in the GC manifold. One portion went to the flame ionization detector to produce a conventional chromatogram, while the remainder went into a nitrogen-selective detector (11) to produce a chromatogram showing the nitrogen-containing SV components. These scans provide a qualitative comparison of SV nitrogen compounds from Cytrel and tobacco cigarettes.

RESULTS AND DISCUSSION

Semi-volatile components from the smoke of filtered cigarettes containing 100% Cytrel, 100% flue-cured tobacco, and a 50/50 blend of these were analyzed to characterize the SV fraction of Cytrel smoke, to compare it to the SV fraction of tobacco smoke, and to demonstrate the effect on individual components of blending Cytrel with tobacco. (Detailed descriptions and vapor phase analyses of these samples were reported previously in Part I.) Reproductions of the general SV and nitrogen-selective SV chromatograms are presented in Figures 1–3 for qualitative comparison of Cytrel and tobacco semi-volatiles. The FID chromatograms were obtained on sample sizes suitable for mass spectral analysis and were not chosen to represent deliveries on an equal cigarette basis. (The actual amounts of Cambridge pad material used to obtain the general SV chro-

Figure 1. Semi-volatiles from 100 % flue-cured tobacco cigarettes.

Top: General SV analysis, 0.18 mg sample, triacetin delivery 176 μg/cigarette.
Bottom: Nitrogen-sensitive SV analysis, 0.8 mg sample.

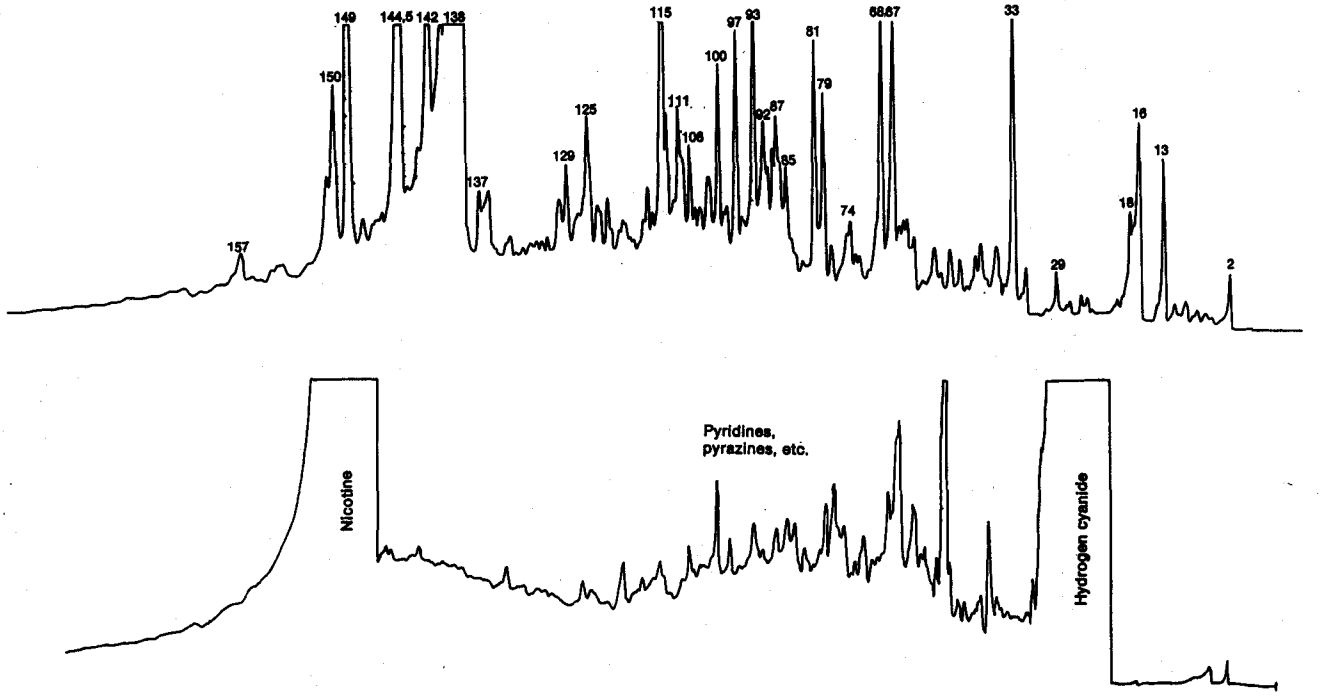
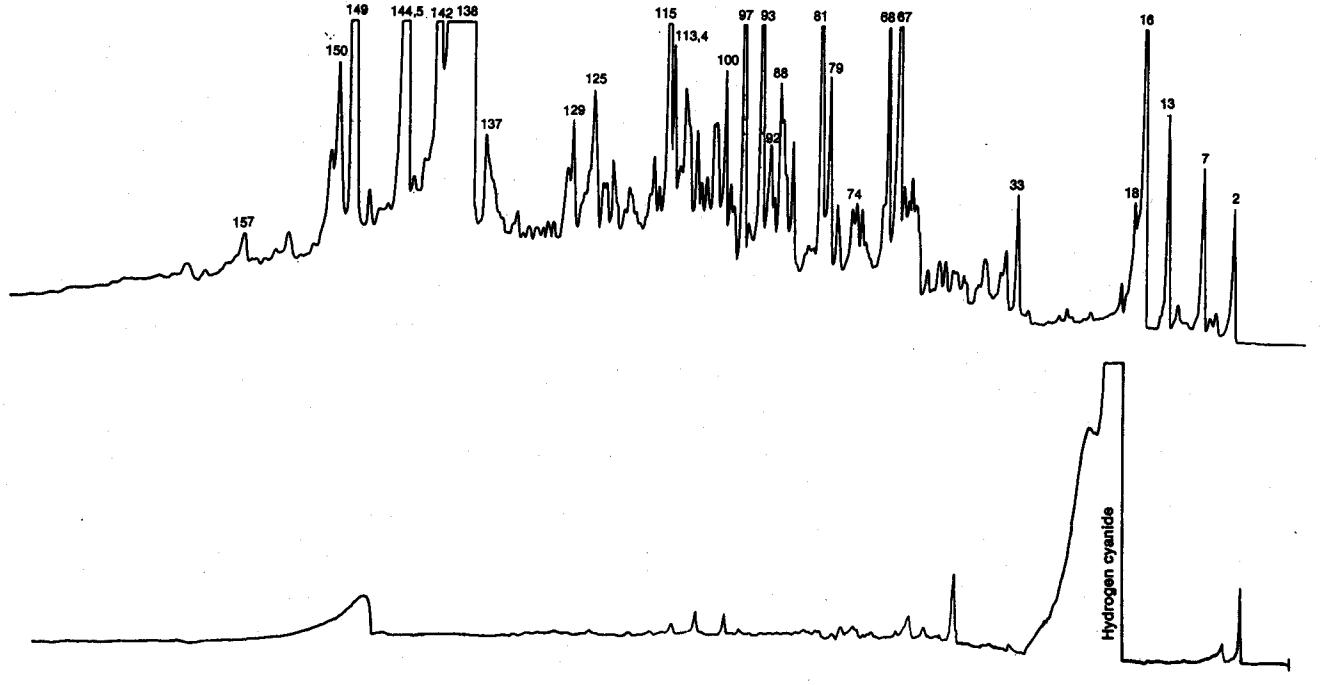


Figure 2. Semi-volatiles from 50/50 blend of tobacco and Cytrel.

Top: General SV analysis, 0.28 mg sample, triacetin delivery 75 μg/cigarette.
Bottom: Nitrogen-sensitive SV analysis, 0.8 mg sample.

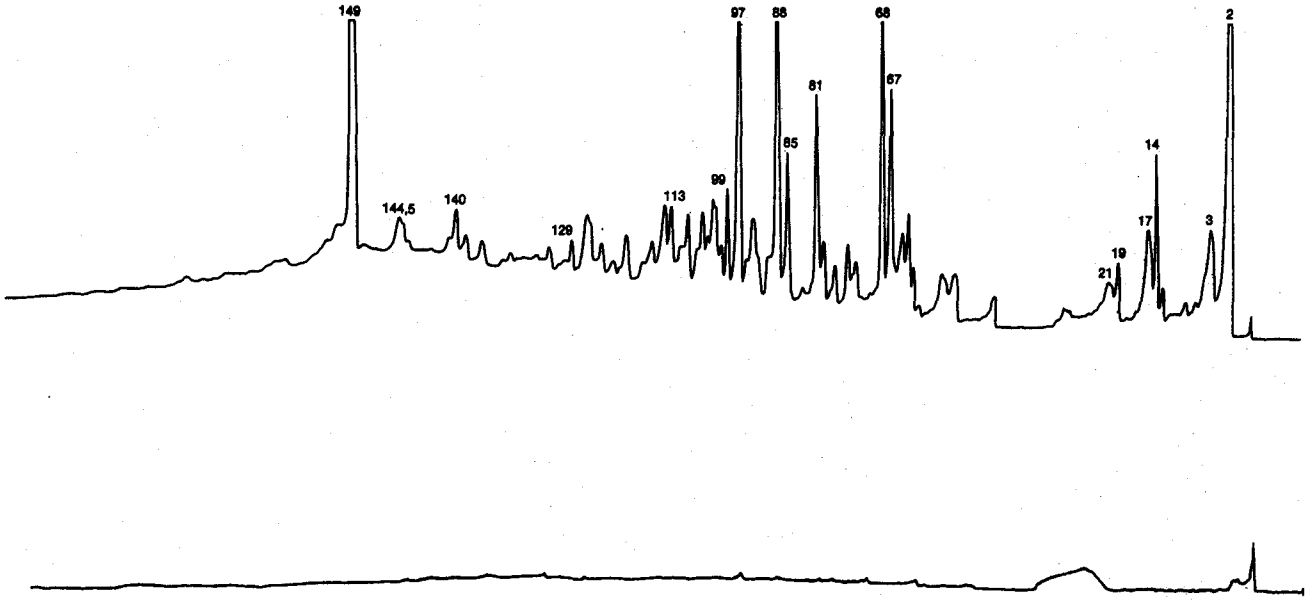


matograms in Figures 1—3 were: 2.3 mg from the tobacco sample, 4.0 mg from the blend, and 13.3 mg from the Cytrel sample. The amounts of SV fraction vaporized from these at 130°C were 0.18, 0.28, and 1.59 mg, respectively.) Similarly, the nitrogen-selective chromatograms were obtained using approximately equal weights of semi-volatile material for both Cytrel and tobacco. In both cases the Cytrel chromatograms represent a significantly greater number of cigarettes than the corresponding tobacco scans. The principal SV component from Cytrel at 130°C is water, which cannot be detected by flame ionization and does not appear on the recorded chromatograms, but does contribute to the measured weight loss from the sample capsule.

At its present stage of development, the capsule sampling technique does not provide absolute dry tar delivery data, although direct comparisons of SV components can be made on a relative basis within a given set of

Figure 3. Semi-volatiles from 100 % Cytrel, Type 361, Lot 227.

Top: General SV analysis, 1.59 mg sample, triacetin delivery 29 μg/cigarette.
Bottom: Nitrogen-sensitive SV analysis, 0.9 mg sample.



samples. The major difficulty in making these relative comparisons is in relating the amount of SV material injected into the GC to the tar delivery of the individual cigarettes. However, triacetin, which appears in the SV fraction as a contaminant from the cigarette filters, can be determined independently and used as an internal standard. The actual triacetin delivery, in micrograms per cigarette, was determined for each sample by solvent (2-propanol) extraction of the particulate matter from a Cambridge filter, followed by analysis of the extract on a GC column calibrated with standard solutions. Except for the syringe injection, the column and conditions were the same as for the SV analyses.

For calculation purposes, the 100% tobacco sample was used as the reference for comparison. Multiplying the sample/reference triacetin delivery ratio by the reference/sample triacetin SV peak area ratio provides a "triacetin factor" for a given sample. Multiplying the measured peak area of any component in that sample by this factor normalizes its value to the delivery of that component in the reference material on an equal number of cigarettes basis. These calculations are shown below:

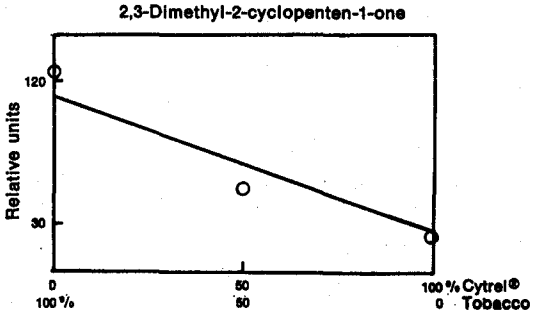
[1] Triacetin (TA) factor =

$$= \frac{\text{TA delivery (sample)}}{\text{TA delivery (reference)}} \times \frac{\text{TA peak area (reference)}}{\text{TA peak area (sample)}},$$

[2] Peak area (sample) × TA factor = Peak area related to corresponding peak in the reference material on an equal number of cigarettes basis.

This method requires equivalent filters on each cigarette in the set being compared. In this way a numerical comparison of relative SV component deliveries on an approximate per cigarette basis may be obtained. Data normalized by this calculation are presented in Table 1, comparing the relative amounts of major SV components delivered by cigarettes containing 100% Cytrel, 100% flue-cured tobacco, and a 50/50 blend of these. Figure 4 displays this information graphically for a selected SV component, clearly showing the effect of blending Cytrel with tobacco. Other components may be plotted the same way from the data given, although low-boiling components eluting in the first twenty minutes do not necessarily show a straight-line relationship. The previously described vapor phase analysis provides a more accurate measure of these low-boiling compounds.

Figure 4. Effect of blend level on a typical semi-volatile compound.



SUMMARY

Major semi-volatile components from Cytrel cigarette smoke have been characterized and compared to the smoke from flue-cured tobacco cigarettes. Significantly

Table 1. Relative deliveries of semi-volatile components in smoke.

Peak*	Retention time	Component	Sample		
			100 % tobacco	50 % Cytrel	100 % Cytrel
1	3.1	Carbon dioxide	1	T**	1
2	4.5	Acetaldehyde	42	22	44
3	5.6	Propanal	8	4	15
5	6.0	2-Methylpropanal	14	4	H+
7	6.5	Acrolein	12	27	2
8	6.8	Methylfuran	5	H	T
10	7.2	Methanol	20	2	2
11	7.6	Methyl ethyl ketone	7	–	1
12	7.9	Allyl ethyl ether	16	8	T
13	8.5	Methyl vinyl ketone	92	31	2
14	9.0	2,3-Butanedione	8	T	8
15	9.5	Methyl isopropyl ketone	H	H	13
16	10.0	Hydrogen cyanide	190	106	–
17	10.4	Methyl propyl ketone	H	H	T
18	10.6	Ethyl vinyl ketone	84	3	T
19	11.4	Crotonaldehyde	14	11	4
21	12.0	2,3-Pentanedione	H	H	8
23	13.3	C_7-Alkene	11	2	1
24	14.4	1-Penten-4-one	7	1	–
26	14.5	Xylene	4	1	–
27	14.7	1-Methylpyrrole	T	2	1
28	14.9	2-Penten-4-one	H	T	1
29	15.2	1-para-Menthene	32	2	1
30	17.1	Unknown, mol. weight 136	22	1	–
31	17.4	Cyclopentanone	10	T	–
33	17.8	Limonene	211	17	T
34	18.2	Pyridine	H	H	–
38	18.8	Methylpyridine	H	18	–
39	18.9	N,N-Dimethylaminoethane nitrile	H	1	–
40	19.0	C_3-Alkylbenzene	68	T	–
42	19.6	Unknown, m/e 41, 69, 112	13	18	4
43	19.9	Unknown, m/e 41, 55, 57, 79	46	–	T
45	20.3	Cyclooctatetraene	32	2	–
47	20.8	C_4-Alkylbenzene	6	–	–
49	21.2	C_4-Alkylbenzene	23	6	6
51	21.2	C_3-Alkylbenzene	H	H	H
53	21.8	C_3-Alkylbenzene	30	1	–
55	22.4	Unknown, m/e 42, 43, 67, 68, 83, 98, 120, 121, 136	10	6	9
57	22.8	Tridecane	43	8	–
58	23.6	Dimethylpyrazine	10	5	1
60	24.0	Methylhexadiene (?)++	28	5	3
61	24.4	Unknown, mol. weight 110	39	15	6
62	24.7	C_3-Alkylbenzene	36	11	8
65	24.9	Tridecene	43	18	–
66	24.9	Unknown, mol. weight 96	H	H	H
67	25.2	2-Cyclopenten-1-one	216	71	17
68	26.0	2-Methyl-2-cyclopenten-1-one	203	64	17
70	27.4	C_3-Alkylphenol	17	6	–
72	27.7	Trimethylpyrazine	14	8	3
74	28.0	Tetradecane	37	10	1
75	28.2	3-Furfural	40	13	4
76	29.1	Unknown, m/e 82, 96, 124	25	11	4
79	29.6	Unknown, m/e 43, 86	126	33	4
80	29.9	2-Cyclohexen-1-one (?)	H	H	H
81	30.1	2-Furfural	143	53	14
82	30.6	Methylfurfural	T	1	–

Table 1 (continued)

Peak*	Retention time	Component	Sample		
			100 % tobacco	50 % Cytrel	100 % Cytrel
83	30.9	Methylindan	13	2	–
84	31.1	Unknown, m/e 68, 96, 124	H	1	1
85	31.9	3,4-Dimethyl-2-cyclopenten-1-one (?)	74	19	10
86	32.3	Indene	51	11	H
87	32.5	Methyl furyl ketone	147	47	26
88	32.7	2-Propyl-2-cyclopenten-1-one	H	H	H
90	33.0	2-Methyl-3-pentanone	45	8	T
91	33.3	Methylindan	H	H	1
92	33.3	Pentadecane	127	28	–
93	33.9	3-Methyl-2-cyclopenten-1-one	187	57	11
94	34.1	Benzaldehyde	H	H	H
96	34.6	Unknown, mol. weight 124	31	8	1
97	35.0	2,3-Dimethyl-2-cyclopenten-1-one	126	52	22
98	35.6	Unknown, m/e 42, 82, 110	19	6	–
99	35.8	2-Ethyl-3-methyl-2-cyclopenten-1-one	16	10	5
100	36.1	5-Methylfurfural	96	26	2
102	36.6	Unknown, m/e 96, 124, 138	56	32	9
103	36.8	Bicyclo(3,3,1)nonane	H	H	H
104	37.0	3-Ethyl-2-cyclopenten-1-one	H	10	2
105	37.3	Methylindene	H	–	7
106	37.3	Dimethylindan (?)	15	–	–
107	37.6	Methylindene	8	4	1
108	37.9	Unknown, m/e 41, 69	40	11	–
109	38.3	Methylindene	102	32	8
110	38.3	Benzonitrile	H	H	H
111	38.5	Unknown, m/e 41, 69	H	H	–
112	38.9	Methylindene	9	10	H
113	39.2	3-Propyl-2-cyclopenten-1-one	H	H	5
114	39.2	gamma-Butyrolactone	45	22	–
115	39.5	Furfuryl alcohol	221	62	8
116	39.9	Unknown, mol. weight 110	H	H	H
117	40.2	Unknown, m/e 41, 69, 112	10	5	–
118	40.5	Acetophenone	38	16	5
120	43.0	Dimethylindene	31	14	2
121	43.6	2-Methyl-2-penten-1-al	30	16	–
122	43.6	Dimethylindene	H	H	3
123	43.6	2-Methyl-1-penten-3-one	H	H	–
124	44.0	Dimethylindene	H	H	–
125	44.2	(E)-Solanone	150	54	7
127	44.5	Dimethylindene	H	H	1
129	45.5	Naphthalene	59	18	2
130	46.0	Nonadecene	46	17	2
131	46.7	aryl-Methylacetophenone	7	2	T
132	47.1	Unknown, m/e 79, 93, 136, 116, 129, 144	7	3	2
133	47.4	Unknown, m/e 145, 159, 160	7	2	–
134	49.0	Unknown m/e 91, 93, 136, 145, 159, 160	18	6	2
135	49.8	Trimethylindene	3	1	1
136	50.3	6-Methyl-3,5-heptadien-2-one (?)	76	12	–
137	50.9	Methylnaphthalene	36	23	3
138	52.	Nicotine	2196	1199	–
139	51.9	Dimethylphenol	H	H	2
140	52.5	Methylnaphthalene	H	H	5
141	53.0	1-Phenylpyrrole	H	H	T
142	54.0	Neophytadiene	409	151	–
143	55.4	3-Methyl-1-indanone	48	11	1
144	55.9	Phenol	744	142	6
145	55.9	Cresol	H	H	H
146	56.9	Biphenyl	29	1	T

Table 1. Relative deliveries of semi-volatile components in smoke (contd.).

Peak*	Retention time	Component	Sample		
			100 % tobacco	50 % Cytrel	100 % Cytrel
147	58.1	1-Indanone	23	7	3
149	59.0	Triacetin	479	204	78
150	59.9	Cresol	{ 168	{ 51	–
151	60.0	Dimethylphenol	{ H	{ H	1
152	60.4	Benzyl alcohol (?)	92	25	1
153	61.5	Ethylnaphthalene	6	1	T
154	63.1	Dimethyl benzyl alcohol (?)	22	10	{ 2
155	63.5	Dimethylphenol	11	4	{ H
156	63.8	C_3-Alkylphenol	13	2	–
157	65.7	Ethylphenol	57	17	T
159	84.1	Diethylphthalate	9	3	5
160	86.3	p-Ethylbenzyl ether (?)	43	15	1
161	92.4	Indole	10	5	–
162	99.6	Methylindole	18	8	–

* Peak numbers are in ascending order, but are not necessarily consecutive. Numbers are keyed to chromatograms in Figures 1 — 3.
** T = Trace amount, no area count.
\+ H = Hidden by larger peak (braces indicate area count including this peak).
++ (?) = Questionable identification.

greater numbers of cigarettes are required to produce the Cytrel scans due to the very low tar deliveries of these cigarettes. Even so, the Cytrel scans are far simpler than those from tobacco-containing samples. Using triacetin delivered from the cigarette filters as an internal standard, numerical data have been derived to compare semi-volatile components from 100 % Cytrel with 100 % flue-cured tobacco and with a 50 % blend with tobacco on an approximately equal cigarette basis. Of the 128 semi-volatile components compared, 37 were found only in tobacco-containing samples and 66 others were present in significantly greater amounts in tobacco than in Cytrel. No components were found in Cytrel semi-volatiles that were not also present in tobacco smoke. Glycerol, which is present in Cytrel smoke, does not appear in the 130°C semi-volatile fraction obtained by the capsule sampling technique.

ZUSAMMENFASSUNG

Die hauptsächlichen halbflüchtigen Inhaltsstoffe des Rauches von Cytrel-Cigaretten wurden untersucht und mit dem Rauch von Cigaretten aus „flue-cured"-Tabak verglichen. Zur Herstellung der Chromatogramme aus Cytrel-Cigaretten ist eine wesentlich größere Zahl von Cigaretten erforderlich, da deren Kondensatausbeute sehr klein ist. Dennoch sind die Chromatogramme dieser Cigaretten weitaus einfacher als die der Tabak enthaltenden Proben. Unter Verwendung des aus den Filtern stammenden Triacetins als inneren Standard wurden auf der Basis jeweils etwa gleicher Cigaretten Zahlenwerte erhalten, die einen Vergleich ermöglichen zwischen den halbflüchtigen Inhaltsstoffen von Cigaretten aus 100 % Cytrel, 100 % „flue-cured"-Tabak und aus einer Mischung von 50 % Cytrel und 50 % Tabak. Von den 128 verglichenen halbflüchtigen Inhaltsstoffen fanden sich 37 nur in den Tabak enthaltenden Proben, und weitere 66 kamen im Tabak in wesentlich größeren Mengen vor als in Cytrel. Bei den halbflüchtigen Inhaltsstoffen des Cytrel-Rauches wurden keine Verbindungen beobachtet, die nicht auch im Tabakrauch vorhanden waren. Das im Cytrel-Rauch vorkommende Glycerin erscheint nicht in der halbflüchtigen Fraktion, die bei 130 °C durch das Kapsel-Probeeingabeverfahren erhalten wurde.

RESUME

Les composés semi-volatils principaux ont été déterminés dans la fumée de cigarettes Cytrel, et comparés à ceux contenus dans la fumée de tabac «flue-cured». Les cigarettes Cytrel produisant très peu de condensat, il faut en utiliser un nombre beaucoup plus important pour obtenir un chromatogramme. Celui-ci reste toujours beaucoup plus simple qu'un chromatogramme venant d'un tabac pur. On a utilisé la triacétine venant des filtres comme étalon interne, et les données numériques dérivées ont permis de comparer, sur la base de cigarettes approximativement égales, les composés semi-volatils produits par des cigarettes 100 % Cytrel, 100 % tabac «flue-cured», et un mélange 50 %—50 %. Sur les 128 composés semi-volatils considérés, on en a trouvé 37 uniquement dans les échantillons de tabac, et 66 autres en quantités nettement plus importantes dans le tabac que dans le Cytrel. On n'a trouvé dans les fumées de Cytrel aucun composé semi-volatil qui ne soit également présent dans la fumée de tabac. Le glycérol, qui est présent dans la fumée de Cytrel, n'apparaît pas dans la fraction semi-volatile de 130 °C lorsque l'on utilise l'échantillonnage par capsule.

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