Transfer of Metallic Constituents of Cigarettes to the Main-Stream Smoke

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Tobacco leaf, in common with other plant tissue, contains a moderate portion of inorganic or mineral constituents. The residue left when tobacco is ashed consists largely of oxides and carbonates of calcium, potassium, magnesium, sodium, aluminum and iron, together with silica and combined phosphate, sulfate, and chloride. In addition, plant tissue normally contains trace amounts of boron, cobalt, copper, manganese, molybdenum, nickel and zinc. At least fourteen other metallic elements have been reported (4, 8) to have been found in plant material. These include arsenic, barium, chromium, lead, lithium, mercury, rubidium, selenium, silver, strontium, thallium, tin, titanium, and vanadium. Some of the latter elements may be accidental constituents or derived from special environments; certain of them, notably arsenic and lead, doubtless owe their presence chiefly to the application of inorganic insecticides.

When tobacco is burned in smoking, the bulk of its metallic components, as well as the greater part of the non-metals boron, silicon and phosphorus, remains in the ash. Small quantities, however, pass into the mainstream smoke and become included in the particulate phase. The appearance of metals in the smoke may arise in part from the vaporization of volatile compounds, e.g., metallic chlorides, and partly by mechanical entrainment of micro-fragments of ash or tobacco in the smoke stream. In order to estimate the extent to which each of these two mechanisms is responsible for the appearance of metals in cigarette smoke, quantitative data on the transfer of the various elements is needed. If the metals pass into the smoke solely by mechanical entrainment of solid particles, then to the degree that the tobacco is homogeneous and the process uniform, they should be present in the smoke in proportion to their relative abundance in the tobacco itself. On the other hand, the various elements can be expected to be present as compounds of widely differing degrees of volatility under the conditions obtaining in the combustion zone. If vaporization of volatile substances is the primary means by which the metals are introduced into the smoke stream, large differences in the transfer efficiencies of the individual elements should be observed. A knowledge of the efficiencies with which the metallic constituents are transferred from tobacco to smoke may be useful in further studies on the physico-chemical role these substances play in the smoking process and, in addition, may suggest possible mechanisms by which some of the other smoke constituents are introduced into the mainstream smoke.

The major purpose of this investigation was to determine the percentage transfer values of the chief metallic components in cigarette smoke. In previous studies on the composition of tobacco smoke, the organic components have held the interest of most investigators. Although it has long been recognized that tobacco contains more than a dozen metals in detectable amounts, little work has been devoted to the occurrence of these in smoke. Only arsenic, because of its physiological importance and the well-known volatility of its compounds, has received much attention in the scientific literature (1, 2, 5, 10, 13). The present investigation showed the presence of fourteen metallic elements in smoke from cigarettes of typical domestic brands. The concentrations of the most abundant of these elements have been determined, both in the smoke and in representative samples of the cigarettes from which the smoke was derived. Relative transfer efficiencies have been calculated.

Experimental

Collection and Preparation of the Smoke Samples.

The cigarettes used in this investigation, purchased during the summer of 1956, were the regular 70 mm. length type, without filters, and represented five of the more popular domestic brands. Before smoking, they were conditioned for several days at 58% relative humidity in a hygrostat over deliquescent sodium bromide. The moisture content of the cigarettes thus conditioned averaged 13.1%, as loss in weight at 110°C. for 20 hours. The cigarettes from one carton of each brand used were then weighed and segregated into groups matched by weight within 0.05 gram. Those of the highest and lowest weight categories were rejected.

The cigarettes were smoked with an automatic smoking apparatus (6) loaned by the Research Laboratory of the Liggett and Myers Tobacco Company. The smoking apparatus was designed to carry up to fifteen cigarettes, drawing from each cigarette in succession a uniform puff of 2 seconds duration with a 58-second interval between puffs. In these experiments, the cigarettes were smoked in groups of thirteen, leaving two positions on the smoker free for checking the puff volume while smoking was in progress. The puff volume was regulated at 35 ± 4 ml.; the average puff volume during the taking of a sample was 35 ml. Ten puffs were taken from each cigarette and, under these conditions, the average butt length after smoking was between 2.0 and 2.5 cm.

Three samples of smoke were collected. For the first two, 117 and 130 cigarettes respectively of two separate brands were smoked. The third sample, consisting of the smoke from 780 cigarettes represented a composite of five brands for which 156 cigarettes of each brand were used.

The smoke was condensed in a series of cold traps. The cold-trap train consisted of three cylindrical flasks, 9 x 28 cm., each having a volume of 1.7 liters, with inlet tubes reaching nearly to the bottom; these

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Table I. Weight of Cigarettes and Smoke.

Sample No.	Brand	Number of Cigarettes Smoked	Weight of Cigarettes Smoked[a]	Av. Wt. of Cigarette[a]	Temperature and Relative Humidity[b]	Weight of Smoke (uncorr.)	Weight of Smoke (corr. for atmospheric moisture)	Weight of Smoke per Cigarette (corr.)	Weight of Smoke per puff (corr.)
			g.	g.		g.	g.	mg.	mg.
1	A	117	126	1.08	25°C.,25%	9.11	8.5	73	7.3
2	B	130	137	1.05	26°C.,20%	10.54	10.0	77	7.7
3	A	156	173	1.11					
	B	156	164	1.05					
	C	156	158	1.01					
	D	156	163	1.04					
	E	156	161	1.03					
		780[c]	819[c]	1.05	29°C.,57%	64.59[c]	55.7[c]	72	7.2

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[a] *Conditioned at 58% relative humidity.*
[b] *Average values during sample collection.*
[c] *Total for sample No.3.*

were followed by three traps in spiral form. The six units of the train were connected in series by ground glass joints, and immersed in cooling baths. The first flask was surrounded by an ordinary ice bath at approximately 0°C; the remaining five traps were at dry ice-alcohol temperature at about −72°C. The empty traps were weighed before assembling into the train, and at the end of the sample collection they were allowed to come to room temperature and reweighed.

The train of cold traps was backed by two flasks, approximately 500-ml. and 125-ml. volume respectively, which were uniformly packed with glass wool. These filters were weighed before and after taking the sample to determine the amount of filterable smoke escaping condensation in the cold traps. The smoke caught by the filters was not included in the sample analyzed. The first filter trapped 4.0% of the total weight of smoke collected; the second, 0.1%. Since the second filter caught only a negligible amount, it may be assumed that the quantity of particulate phase smoke unaccounted for was insignificant, and that the portion caught in the cold traps and analyzed represented approximately 96% of the total smoke. The first three units of the cold-trap train retained approximately 87% of the smoke; the spiral precipitators, about 9%.

The smoke was washed from the traps by rinsing them alternately with redistilled acetone, 0.06 *N* hydrochloric acid in 90% acetone, and water. When the smoke had been completely removed from the traps, the latter were rinsed in succession with a 100 ml. portion of concentrated nitric acid. This acid was reserved as part of that required for the subsequent digestion of the sample. The smoke solution was concentrated by evaporation until the acetone had been removed.

Collection of the Tobacco Samples. For each group of thirteen cigarettes smoked on the automatic smoking apparatus, one cigarette from the same weight category was reserved for preparation of the control tobacco sample. The control cigarettes of each individual brand were opened, the tobacco removed and thoroughly mixed. The tobacco sample corresponding to the composite smoke, sample No. 3, consisted of the equivalent of one average cigarette including one paper per cigarette of each of the 5 brands. For the tobacco samples accompanying smoke samples Nos. 1 and 2, which represented smoke from cigarettes of individual brands, the equivalent of 4 cigarettes of the particular brand was used.

Data on the samples taken and weights of smoke collected are given in Tables I and II. The average weight of smoke obtained, after correction for atmospheric moisture at the temperature and relative humidity prevailing during smoking, was 74 mg. per cigarette, or 7.4 mg. per puff. This weight is somewhat larger than is often reported, ~5 mg. per puff, but this is to be expected as the collection procedure used effectively traps some gas phase constituents which, once condensed with the particulate phase of the smoke, are not readily volatilized in the smoke collection procedure used in this investigation.

Digestion of the Samples. The or-

Table II. Weights of Tobacco Samples.

Sample	Brand	Weight of Average Cigarette[a]	Weight of Tobacco Taken[a]	Weight of Papers[a]	Total Weight of Sample[a]	Equivalent No. of Average Cigarettes
		g.	g.	g.	g.	
1	A	1.08	4.129	0.175	4.304	3.98
2	B	1.05	3.952	0.170	4.122	3.93
3	A	1.11	1.070	0.045	1.115	1.00
	B	1.05	1.009	0.043	1.052	1.00
	C	1.01	0.973	0.048	1.021	1.01
	D	1.04	1.006	0.042	1.048	1.01
	E	1.03	0.985	0.048	1.033	1.00
Sample No. 3 Totals:			5.043	0.226	5.269	5.02

[a] *Conditioned at 58% relative humidity.*

Table III. Concentrations of Metals in Tobacco and Smoke.

Element	Sample No.	Concentration Tobacco p.p.m.[a]	Concentration Smoke p.p.m.	Micrograms per Cigarette Tobacco	Micrograms per Cigarette Smoke	Micrograms per Puff	Relative Per cent Transfer
Potassium	1	24800	1840	26800	134	13.4	0.50
	2	25800	1790	27100	138	13.8	0.51
Sodium	1	805	126	870	9.2	0.92	1.06
	2	850	90	890	7.1	0.71	0.80
Calcium	3	26100	nil (<6)	27500	nil (<0.4)	<0.04	
			nil (<3)		nil (<0.2)	<0.02	<0.001
Magnesium	3	4750		5000			
		4950		5200			
		Av. 4850	1	Av. 5100	0.07	0.007	0.001
Aluminum	1	860	–	930	–	–	–
	2	630	<2	660	<0.15	<0.015	<0.03
	3	600	0.03	630	0.02		
			0.03		0.02		
			Av. 0.03		Av. 0.02	0.002	0.003
Iron	1	560	–	605	–	–	–
	2	415	∼2.5	435	∼0.2	∼0.02	∼0.05
	3	525	1	550	0.085		
		505	1	530	0.066		
		Av. 515	Av. 1	Av. 540	Av. 0.075	0.0075	0.014
Manganese	3	175		185			
		185		195			
		Av. 180	nil (<0.6)	Av. 190	nil (<0.04)	<0.004	<0.02
Copper	1	36		39			
		38		41			
		Av. 37	–	Av. 40	–	–	–
	2	20	4.6	21	0.36		
		19	4.9	20	0.38		
		Av. 20	Av. 4.7	Av. 21	Av. 0.37	0.037	1.7
	3	17	1	18	0.08		
		17	1	18	0.08		
		Av. 17	Av. 1	Av. 18	Av. 0.08	0.008	0.4

[a] *Conditioned at 58% relative humidity.*

ganic matter in the smoke and tobacco samples was destroyed by wet digestion with nitric and perchloric acids in Vycor beakers. The digestion was begun with somewhat diluted nitric acid, ca. 4 to 1. After evaporation to a small volume successive additions of concentrated nitric acid, with intervening evaporations, were made until the samples were completely liquefied. The solutions were then treated with a mixture of equal volumes of water, concentrated nitric acid and 72% perchloric acid, and evaporated to fumes of the latter acid. Evaporation with further portions of the acid mixture was carried out until colorless or light yellow solutions resulted, at which point the samples were evaporated nearly to dryness. The residues were taken up in hot dilute hydrochloric acid, the precipitated silica filtered off, and the resulting solutions made up to definite volume.

Because of the large quantities of acetone, ca. 2000 ml., and acids required in the preparation of the smoke sample, a blank consisting of approximately the quantities of solvents used in eluting the smoke from traps was prepared and carried through the digestion. The acetone, and hydrochloric and nitric acids employed were redistilled in Pyrex before use.

Colorimetric Determination of the Metals. The solutions resulting from the wet-digestion of the smoke and tobacco were analyzed by adaptations of established photometric procedures (9,11). In some cases the existing procedures were reduced in scale because of the limited amount of smoke sample available and the low concentrations of the elements being determined.

Sodium and potassium were determined by flame photometry, employing the Perkin-Elmer Model 52-C instrument, with lithium as an internal standard. Calcium was determined by precipitation as calcium oxalate, followed by the colorimetric determination of the oxalate by the ceric sulfate-iodide method. For the estimation of magnesium, the Titan yellow

Table III. (Continued).

Element	Sample No.	Concentration		Micrograms per Cigarette		Micrograms per Puff	Relative Per cent Transfer
		Tobacco p.p.m.[a]	Smoke p.p.m.	Tobacco	Smoke		
Lead	1	20		22			
		18		20			
		Av. 19	14	Av. 21	1.0	0.10	4.8
	2	80	43	84	3.3		
			42		3.2		
			Av. 43		Av. 3.3	0.33	3.9
	3	40	26	42	1.9		
		37	26	39	1.9		
		Av. 39	Av. 26	Av. 41	Av. 1.9	0.19	4.6
Zinc	1	33		36			
		33		36			
		Av. 33		Av. 36	–	–	–
	2	24	0.3	25	0.02		
			1		0.08		
			Av. 0.7		Av. 0.05	0.005	0.2
	3	29	2.5	31	0.18		
		28	2.5	30	0.18		
		Av. 29	Av. 2.5	Av. 31	Av. 0.18	0.018	0.6
Nickel	3	1.9	–	2.0	–	–	–
Chromium	3	1.3	–	1.4	–	–	–
Arsenic	1	7	4	7.8	0.31	0.031	
		6		6.4			
		Av. 7		Av. 7.1			4.4
	2	25	18	26.5	1.4	0.14	
		25		26.2			
		Av. 25		Av. 26.4			5.3
	3	12	6	13.0	0.46	0.046	
		13	6	13.4	0.45	0.045	
		Av. 13	Av. 6	Av. 13.2	Av. 0.46	Av. 0.046	3.5

[a] *Conditioned at 58% relative humidity.*

colorimetric procedure was employed. Aluminum was determined colorimetrically with aluminon; and iron, by the thiocyanate reaction. Copper, lead and zinc were isolated and measured colorimetrically by extraction with dithizone. Arsenic was separated as arsine and determined by the molybdenum blue colorimetric technique. Nickel was isolated by extraction into chloroform with dimethylglyoxime and subsequently determined colorimetrically with the same reagent. The estimation of chromium was made by oxidizing it to chromate with hypobromite in alkaline solution and applying the diphenylcarbazide color reaction.

Spectrographic Analyses. Aliquots of smoke sample No. 3 and the corresponding tobacco and blank samples were submitted for semi-quantitative spectrographic analysis to the testing laboratories of W. B. Coleman and Company, Philadelphia, Pennsylvania.

Analytical Results

The concentrations of the various metals found in the smoke and in representative samples of cigarettes from which the smoke was derived are shown in table III. The values in column 4, for concentrations in parts per million parts of smoke, are based upon the corrected weights of smoke given in table I. Columns 5 and 6 show the number of micrograms of each element present in one whole cigarette of average weight, and in the smoke derived from one cigarette using 10 puffs per cigarette. The relative percentage transfer values are calculated from the data of columns 5 and 6, and are thus based upon the weight of the particular element in one whole cigarette rather than on the portion, about two-thirds, burned in the smoking or during the puff time. Uncertainty as to the exact amount of tobacco burned during puffing led to the use of *relative* percentage transfer figures rather than *actual* percentage transfer. In addition to the metals determined colorimetrically and listed in table III, the spectrographic examination showed the presence in smoke of titanium and strontium, and the non-metal silicon. The non-metal boron was detectable in the tobacco, but not in the smoke. In all cases, the concentration levels indicated by the semi-quantitative spectrographic analysis agreed well with those found by the colorimetric

Table IV. Arsenic-to-Lead Ratio in the Cigarettes.

Sample No.	Pb/cig.(ug)	As/cig.(ug)	Ratio As/Pb
1	21	7.1	0.34
2	84	26.4	0.31
3	41	13.2	0.32
		Av.	0.32
Theoretical ratio in $PbHAsO_4$			0.36

and flame photometric measurements. Strontium, and barium if present, would count as calcium in the oxalate method employed for the colorimetric determination of the latter element, but the spectrographic analysis indicated that the concentration of strontium, both in tobacco and smoke, was less than one one-hundredth that of calcium. Barium was not found in the spectographic examination. Titanium is often present in plant material in concentrations of 10 to 30 p.p.m., a level which agrees well with that indicated in the tobacco by the spectrographic tests. The indicated titanium content of the smoke was below 0.06 micrograms per cigarette. Chromium was determined colorimetrically to have a concentration of 1.3 p.p.m. in the tobacco; it was not analyzed for in the smoke. The spectrographic results showed its presence in the smoke, but at a level of less than 0.06 micrograms per cigarette. Nickel was not found spectrographically in either tobacco or smoke. Colorimetric determination of nickel in the tobacco showed a concentration of 1.9 p.p.m.

The following elements were checked but not found, in either tobacco or smoke, by the spectographic procedure: antimony, barium, beryllium, bismuth, cadmium, cobalt, columbium, gallium, germanium, gold, molybdenum, platinum, silver, tellurium, tin, tungsten, vanadium and zirconium.

Discussion

From the data of table III, it can be seen that the transfer efficiencies of the various metallic constituents present in tobacco vary over a quite wide range. This shows that vaporization is almost completely responsible for the appearance of metals in smoke. Three of the metals determined, namely calcium, magnesium and aluminum, under the conditions of basic fusion existing in the combustion zone should be readily converted to difficultly reducible and extremely non-volatile oxides. The carry-over of these elements to the small extent observed is probably due largely to mechanical entrainment of material in the smoke stream. The transfer efficiencies of these metals average less than 0.002%, and this can be taken as an approximate upper measure of the extent to which mechanical transfer contributes to the metallic content of smoke. The metals found in the tobacco totaled about 62 mg. per cigarette. A transfer of 0.002% would thus correspond to approximately 1.2 micrograms per cigarette, or less than one per cent of the total quantity of metals (ca. 150/cig.) found in the smoke. Evidently any solid particles that may be swept or explosively ejected into the smoke stream at the burning tip of a cigarette are quite effectively filtered out by the column of unburned tobacco even in the short butt length remaining at the end of smoking.

Since the hottest zone of a cigarette probably consists of a complex mixture of fused salts and carbonaceous material, the transfer of the metallic elements cannot, with the data at present available, be conclusively pictured in terms of the formation of any particular compounds. Vaporization of metallic chlorides, however, appears to be the most likely explanation, and the transfer data are in line with this hypothesis. The temperature in the burning cone of a cigarette has been determined as in the range of 850° to 920°C. (14). The alkali metal chlorides are known to be appreciably volatile at temperatures well below their boiling points which for sodium chloride is 1413°C., for potassium chloride approximately 1550°C, and lead chloride, b.p. 950°C, vaporizes rapidly above 550°C. (3). It is of some interest that a partial pressure of KCl of a few tenths of a millimeter would account for the potassium found. The vapor pressure of pure KCl is given as 1 mm at 821°C. (12). Both the chloride and the oxide of trivalent arsenic vaporize below 200°C., and the metal itself sublimes at about 600°C. With the easily reducible metals of low boiling point, viz. arsenic, lead, zinc, reduction by the carbonaceous material and vaporization in the elemental state may occur. The fact that volatility is a controlling factor in the case of transfer of the metallic constituents has certain inferences for other components of the smoke. including organic materials. It would seem that volatilization will probably play an essential role in determining what substances can get into the smoke stream whatever their nature and thus substances with extremely low vapor pressures at temperatures of 950°C. and below may not be expected in quantity in the smoke. Conversely one may expect to find in the collected main-stream smoke at least some of all components of tobacco which can develop even a low vapor pressure at thermal levels below their decomposition temperature. In addition there may be present degradation and reaction products of the various tobacco components that are produced in the pyrolysis-combustion process providing such products have reasonable volatility.

The major part of the arsenic and lead found in the cigarettes was probably derived from lead arsenate applied to the tobacco as an insecticide. The constancy of the arsenic-to-lead ratio in the tobacco samples, as shown in table IV, supports this conclusion. The common lead arsenate, or "acid lead arsenate", has a composition approximating $PbHAsO_4$, with a theoretical arsenic-to-lead ratio of 0.36. The average ratio in the tobacco samples is 0.32; in the smoke samples, it is 0.35. The transfer efficiencies found for the two metals on the individual samples differ slightly, but the average transfer efficiencies of both are identical (4.4%). Hence, the two metals were probably present in combination on the surface of the tobacco and may have been volatilized in combination or simultaneously through reduction of the lead hydrogen arsenate.

The transfer of arsenic in cigarette smoking has been the subject of several studies (2,5,13). These investigators used only a few cigarettes for their smoke samples and experienced difficulty in obtaining consistent values because of the non-uniformity of arsenic content, even among cigarettes taken from the same package (2). In the present investigation at least one hundred cigarettes were smoked to obtain each sample. The cigarettes used for samples Nos. 1, 2 and 3 differed widely in average arsenic content, and the range of values for arsenic transferred to the smoke, 0.31 to 1.4 micrograms per cigarette, reflects this variation. In another series of samples of smoke prepared in this laboratory in 1954 by Miss Elizabeth Williams (15) for the determination of arsenic only, the values of arsenic in the smoke ranged from 0.43 to 0.99, and averaged 0.65 micrograms of arsenic per cigarette smoked. No data were obtained on the arsenic content of the cigarettes used to produce these smoke samples. The weighted average of the ten samples analyzed in this and the Williams study, representing in aggregate the smoke from 1727 cigarettes, was 0.60 micrograms of arsenic per cigarette. The average arsenic figure found by Miss Williams is somewhat higher, 0.65 vs 0.46, than reported in the present investigation on the composite sample and may result from the present trend away from use of arsenicals as insecticides.

The eleven elements analyzed for in the smoke include all of the major metallic constituents of the tobacco from which the smoke was derived. The data, in summation, therefore represent a reasonably complete determination of the metal content of smoke. From these data, it may be concluded that the total quantity of metallic elements in the smoke from one average cigarette, when smoked under the conditions described, is in the neighborhood of 150 micrograms. Of this, approximately 90% is potassium and about 5% is sodium.

Summary

Using conditions approximating human smoking about 150 micrograms of metallic constituents per cigarette were found in the mainstream smoke of five representative brands of cigarettes on the domestic market. Of this quantity approximately 90% was potassium and 5% sodium. The remaining metallic constituents, at least twelve in number, include traces of aluminum, arsenic, calcium, chromium, copper, iron, lead, magnesium, manganese, strontium, titanium and zinc. Vaporization of the more volatile compounds of the metals or of the volatile reduced metal itself appear to be the essential mechanisms of transfer from the cigarette to the smoke. Mechanical transfer by erosive or related processes probably accounts for less than one per cent of the transferred materials. The inference of these findings for other smoke components is discussed.

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