Collaborative Study of the Determination of Tar and Nicotine in Cigarette Smoke: 1970 Study

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A collaborative study was made to evaluate the effects of (*1*) reducing the number of cigarettes per port, (*2*) using sleeves vs. dams for sealing the cigarette in the holders, and (*3*) pooling filter pads before water and nicotine analyses. None of these variables had a significant effect on the precision of the method. The statistical data obtained from this study agreed closely with those reported last year.

The smoking procedure used last year (1) for the determination of tar, water, and nicotine in cigarette smoke was modified in an attempt to improve the precision for high tar and nicotine delivery cigarettes. The precision with low and medium delivery cigarettes was satisfactory, but it diminished as the delivery levels increased in the upper range. Two possible causes suggested for this were: (*1*) the overloading of filter pads with loss of efficiency even though no breakthrough occurred and (*2*) the use of dams instead of the latex sleeves which had been recommended for holding nonfilter cigarettes in the filter holders. (The 2 high delivery samples were the only nonfilter cigarettes used in the study.)

This year's collaborative study was designed to determine whether either or both of these variables affected the precision. The experimental design required that the same lot of cigarettes be smoked, using both 4 and 5 cigarettes per port with both latex sleeves and rubber dams. In addition, pooling the filter pads from 2 or more ports for the water and nicotine analyses could effect a considerable saving in time and money. However, the effect of sample pooling on the precision of these determinations was unknown and was a necessary phase of these experiments. The same monitor cigarette (III, new monitor in 1969) was smoked again in order that the results obtained this year could be related more reliably to those from last year. The latest monitor cigarette (IV) was smoked so that collaborative data from this material would also be available.

Four samples were used in the study: 2 similar high delivery nonfilter cigarette samples and 2 monitor filter samples, the old (III) and the new (IV). Sample 1 was drawn from a commercial production line when it was judged under control and Sample 2 was drawn from another line at a much later date, again when it was judged to be operating properly. Samples 3 and 4 were old (III) and new (IV) monitor cigarettes, respectively, with medium tar and nicotine delivery.

The design of the study required that 2 ports be smoked on 3 days of one week and on 3 days of a following week for each combination of variables being tested (i.e., 4 and 5 cigarettes per port with both sleeves and dams). Thus, 16 ports were used each day for Samples 1 and 2. Two ports of each monitor cigarette were smoked on the remaining 4 ports each of the 6 days noted above, using 5 cigarettes per port, preferably with dams but optionally with sleeves. These collections were analyzed as described below. Two more ports of each monitor cigarette were smoked on a second run immediately following the first. The filter pads from these pairs of ports were pooled for the water and nicotine analyses.

The method was the same as that used last year with the exceptions noted above, i.e., sleeve vs. dam and 4 vs. 5 cigarettes per port, and an increase in shaking time from 20 to 60 min to insure complete solvation of water and nicotine into the isopropanol.

Results and Discussion

Results are reported as mg nicotine *per cigarette* and mg tar *per cigarette* in order to place

results from 4 and 5 cigarette samples per port on a comparable basis. All per cigarette results except variances (i.e., means, differences, standard deviations, confidence limits, etc., but not squared measures) can be converted to per port results by multiplying by 4 or 5, as appropriate. Mean squares and components of variance must be multiplied by 5^2 or 4^2.

Average values of nicotine and tar obtained by the cooperating laboratories for each sample by each treatment, i.e., by each combination of dental dam and rubber sleeve sealing apparatus with samples of 4 and 5 cigarettes smoked per port, are reported in Tables 1 and 2, respectively. Each result reported is the average of 12 determinations, 2 ports on 3 separate days in each of 2 weeks.

Rank tests on the means in Tables 1 and 2 and analyses of variance of individual laboratories (not reported) disclosed that no individual laboratory was so consistently more variable than the others as to need rejection, although the 3 laboratories using dental dam only did show somewhat more variability than the others. Accordingly, results from all 9 participating laboratories were used in further analyses, subject to limitations imposed by the lack of uniformity among laboratories as to treatments used.

Results obtained with each treatment and/or type of cigarette in Study 1 (1) are treated by analysis of variance and reported in Tables 3 and 4 for nicotine and tar, respectively.

Results from combined analyses of variance of the 6 laboratories smoking all 4 combinations of dental dam and rubber sleeves with 4 and 5 cigarettes per port are reported in Table 5 for nicotine and tar. Combined analyses of variance of 5 laboratories reporting comparable results with all treatments of high tar cigarettes and monitors and of 3 laboratories reporting comparable results with samples of 4 and 5 cigarettes (using dental dam only) and monitors are reported in Table 6 for nicotine and tar.

Comparisons of variability found in the 1969 and the 1970 studies are reported for nicotine in Table 7 and for tar in Table 8.

Table 1. Mean nicotine values[a] (mg per cigarette) by study, cigarette class, apparatus, number of cigarettes, laboratory, and sample

		Study 1						Study 2	
		High Tar				Monitor		Monitor	
		Dams		Sleeves		Dams	Sleeves	Dams	Sleeves
Lab.	Sample	4	5	4	5	5	5	5	5
1	1	1.62	1.63	1.70	1.69		1.16		1.20
	2	1.50	1.51	1.59	1.57		1.18		1.22
4	1	1.62	1.57	1.63	1.60		1.15		1.17
	2	1.52	1.51	1.52	1.46		1.14		1.20
8	1	1.69	1.70	1.72	1.66		1.17		1.20
	2	1.63	1.57	1.58	1.59		1.17		1.16
19	1	1.83	1.82	1.76	1.76		1.24		1.14
	2	1.68	1.68	1.60	1.61		1.29		1.14
24	1	1.77	1.77	1.79	1.76		1.21		1.26
	2	1.69	1.68	1.62	1.63		1.24		1.32
2	1	1.69	1.67	1.70	1.72	1.21		1.23	
	2	1.62	1.61	1.63	1.60	1.23		1.27	
13	1	1.86	1.84			1.26		1.26	
	2	1.70	1.68			1.24		1.25	
15	1	1.73	1.68			1.23		1.24	
	2	1.63	1.59			1.24		1.26	
21	1	1.68	1.73			1.18		0.92	
	2	1.58	1.62			1.19		0.91	
Av., Labs. 1, 2, 4, 8, 19, 24	1	1.705	1.693	1.716	1.698				
	2	1.606	1.593	1.590	1.576				
Av., Labs. 1, 4, 8, 19, 24	1	1.707	1.697	1.720	1.695		1.185		1.192
	2	1.604	1.591	1.583	1.571		1.204		1.206
Av., Labs. 13, 15, 21	1	1.741	1.732			1.220		1.163	
	2	1.631	1.624			1.224		1.172	

[a] Means of 12 determinations—2 weeks, 3 days per week, 2 ports per day per sample, and treatment.

From Tables 1, 2, 5, and 6 it is evident that there are no universal effects of the various combinations of dental dam and rubber sleeve with numbers of cigarettes per port (4 or 5) on the levels of tar and nicotine found. There is evidence, however, that laboratories differ with respect to results obtained with the different treatments; *see* entries for T′ × L in Table 6 and T × L in Table 5 and means for Laboratories 1 and 19, especially, in Tables 1 and 2.

The best evidence on whether the combinations of sealing technique and number of cigarettes per sample change the variability of the procedure is contained in the analyses of variance of the 4 treatment combinations summarizing results from the 6 laboratories (1, 2, 4, 8, 19, and 24) that evaluated all 4 combinations; *see* Tables 3 and 4, top sections. These results offer no consistent evidence that any of the combinations either decrease or increase the variance. In fact, tests for homogeniety of variance reveal that at no line, in either the nicotine or tar analyses, is there a significant difference among the 4 mean squares. Furthermore, there is no tendency for any treatment to have higher or lower mean squares over the several lines of an analysis. Under these circumstances it would seem fair to conclude that neither method of sealing nor number of cigarettes per sample was the cause of excessive variability in high tar cigarettes reported by Ogg and Schultz (1) for the 1969 study. Since the 4 treatments have similar variances, they were treated by combined analyses of variance over the 6 laboratories as reported in Table 5.

Further examination of Tables 3 and 4, middle and bottom sections, indicates that the port variance of the high tar cigarette is significantly greater than that of the monitor. It also indicates that the 3 laboratories using dams only, when treated as a single class, were more variable than the other laboratories. These results can also be seen in Tables 7 and 8.

Although port variances by lots or types of cigarettes were calculated for nicotine and tar in the 1969 study, they were not conclusive in and of themselves and were not reported. They are reported now (converted from per port to per

Table 2. Mean tar values[a] (mg per cigarette) by study, cigarette class, apparatus, number of cigarettes, laboratory, and sample

		Study 1						Study 2	
		High Tar				Monitor		Monitor	
		Dams		Sleeves		Dams	Sleeves	Dams	Sleeves
Lab.	Sample	4	5	4	5	5	5	5	5
1	1	23.9	24.5	25.5	25.9		17.6		17.8
	2	21.7	21.2	23.2	22.4		17.5		17.2
4	1	25.4	25.5	26.0	25.1		18.9		18.7
	2	22.8	22.9	23.2	22.8		18.1		18.4
8	1	25.4	25.3	26.0	25.8		18.0		18.1
	2	23.7	23.2	23.5	23.5		17.0		17.1
19	1	25.4	25.6	24.5	24.6		16.7		16.7
	2	22.6	22.6	21.3	21.2		16.6		16.6
24	1	25.9	25.9	26.2	25.8		18.1		18.6
	2	24.1	23.6	22.6	22.9		17.7		18.2
2	1	24.8	24.8	24.8	25.3	17.8		17.8	
	2	22.6	22.4	22.3	22.8	17.4		17.5	
13	1	27.0	26.5			17.5		17.2	
	2	23.5	23.7			16.6		16.6	
15	1	25.4	25.0			18.0		18.3	
	2	22.3	22.3			17.8		16.3	
21	1	24.6	25.4			18.0		19.3	
	2	21.8	22.6			17.6		17.6	
Av., Labs. 1, 2, 4, 8, 19, 24	1	25.13	25.27	25.50	25.42				
	2	22.92	22.65	22.68	22.62				
Av., Labs. 1, 4, 8, 19, 24	1	25.20	25.37	25.64	25.44		17.87		17.98
	2	22.97	22.70	22.75	22.57		17.36		17.48
Av., Labs. 13, 15, 21	1	25.44	25.44			17.79		18.13	
	2	22.55	22.74			17.37		17.01	

[a] Means of 12 determinations—2 weeks, 3 days per week, 2 ports per day per sample, and treatment.

Table 3. Mean squares from combined analyses of variance of nicotine results, Study 1

Variation	DF[a]	Study 1					
		High Tar				Monitor	
		Dams		Sleeves		Dams	Sleeves
		4	5	4	5	5	5
		Laboratories 1, 2, 4, 8, 19, 24					
Samples (S)	1	0.3500	0.3550	0.5675	0.5426		
Laboratories (L)	5	0.1553	0.1610	0.0492	0.0864		
Weeks (W) in L	6	0.0204	0.0285	0.0339	0.0292		
Days (D) in W in L	24	0.0154	0.0101	0.0158	0.0177		
S × L	5	0.0063	0.0061	0.0088	0.0052		
S × W in L	6	0.0027	0.0078	0.0090	0.0082		
S × D in W in L	24	0.0079	0.0088	0.0085	0.0045		
Ports	72	0.0069	0.0074	0.0068	0.0090		
		Laboratories 1, 4, 8, 19, 24					
S	1	0.3172	0.3370	0.5644	0.4612		0.0104
L	4	0.1942	0.2012	0.0612	0.1048		0.0552
W in L	5	0.0244	0.0340	0.0363	0.0345		0.0144
D in W in L	20	0.0178	0.0118	0.0175	0.0208		0.0047
S × L	4	0.0071	0.0056	0.0050	0.0065		0.0041
S × W in L	5	0.0032	0.0090	0.0099	0.0076		0.0018
S × D in W in L	20	0.0075	0.0079	0.0086	0.0049		0.0023
Ports	60	0.0076	0.0078	0.0060	0.0080		0.0032
		Laboratories 13, 15, 21					
S	1	0.5232	0.5365			0.0000	
L	2	0.2650	0.1960			0.0250	
W in L	3	0.1505	0.1113			0.0095	
D in W in L	12	0.0340	0.0227			0.0141	
S × L	2	0.0139	0.0180			0.0012	
S × W in L	3	0.0188	0.0098			0.0081	
S × D in W in L	12	0.0246	0.0144			0.0084	
Ports	108	0.0139	0.0132			0.0081[b]	

[a] DF = degrees of freedom.
[b] Ports degrees of freedom = 36; only 2 ports per sample per treatment instead of 4 as in high tar cigarettes.

cigarette basis) in Tables 7 and 8 for nicotine and tar for the high tar and monitor cigarettes. One may observe the fairly good agreement in estimates of port variance for 1969 and 1970 if one omits from the 1970 work the 3 laboratories (13, 15, and 21) that did not use sleeves. This is true for both nicotine and tar for both high tar and monitor cigarettes. One may also observe (again omitting Laboratories 13, 15, and 21) that the analyses of the high tar cigarettes for both nicotine and tar showed that the mean squares for "Laboratories" and "S × L (Samples × Laboratories)" were larger in 1969 than in 1970. This was not true of the monitor cigarettes. Such behavior might indicate that the closer attention given by the laboratories to the procedure in 1970 enabled them to more nearly duplicate each other's results with the highly variable high tar cigarettes in 1970 than in 1969.

Analysis of variance combining results from high tar and monitor cigarettes in one analysis, after demonstrating that these have different variances, is not recommended. However, such analyses were performed in this study in order to observe whether the large sources of variability in the procedure are still the same ones as reported last year. All of the analyses reported in Tables 5 and 6 corroborate the statement made last year that variability in results can be attributed largely to 3 generalized sources:

(*1*) The basic underlying determination error of the procedure, or "within laboratory" variability—*see* bottom lines of analyses of variance, Tables 5 and 6.

(*2*) Differences among laboratories, or "among laboratory" variability—including Laboratory × Material interaction—*see* lines for Laboratories, Laboratories × Treatments, and Laboratories × Lots in Tables 5 and 6.

(*3*) Variability among samples of material, or

Table 4. Mean squares from combined analyses of variance of tar results, Study 1

Variation	DF[a]	Study 1					
		High Tar				Monitor	
		Dams		Sleeves		Dams	Sleeves
		4	5	4	5	5	5
		Laboratories 1, 2, 4, 8, 19, 24					
Samples (S)	1	176.446	248.850	286.173	282.800		
Laboratories (L)	5	13.221	9.992	11.686	8.296		
Weeks (W) in L	6	1.774	1.009	1.898	0.654		
Days (D) in W in L	24	2.013	1.228	2.883	1.309		
S × L	5	0.996	1.216	1.577	1.870		
S × W in L	6	0.827	0.934	1.479	1.386		
S × D in W in L	24	1.168	1.078	2.210	0.837		
Ports	72	1.012	1.410	1.293	1.059		
		Laboratories 1, 4, 8, 19, 24					
S	1	148.518	213.066	249.985	247.107		7.854
L	4	15.854	11.511	12.727	10.353		11.208
W in L	5	2.118	0.968	2.257	0.560		1.395
D in W in L	20	2.260	1.325	3.111	1.494		1.148
S × L	4	1.239	1.462	1.769	2.134		1.119
S × W in L	5	0.932	0.913	1.319	1.401		1.179
S × D in W in L	20	1.086	1.054	2.144	0.783		0.315
Ports	60	1.002	1.393	1.220	1.184		0.585
		Laboratories 13, 15, 21					
S	1	354.694	278.333			3.380	
	2	52.831	27.008			5.021	
in L	3	2.502	6.801			0.167	
D in W in L	12	3.978	1.743			3.904	
S × L	2	0.994	0.144			0.721	
S × W in L	3	0.185	1.076			0.216	
S × D in W in L	12	1.537	2.649			0.867	
Ports	108	1.709	1.646			0.991[b]	

[a] DF = degrees of freedom.
[b] Ports degrees of freedom = 36; only 2 ports per sample per treatment instead of 4 as in high tar cigarettes.

sampling variability—*see* lines for Samples in Tables 5 and 6.

We decided not to estimate variance components from this year's work for the following reasons: (*1*) the ranges of nicotine and tar values studied this year are much less than last year; (*2*) fewer laboratories can be analyzed together this year than last; (*3*) the inclusion of treatments as well as lots or sources of cigarettes and unequal numbers of determinations in the 2 pairs of samples makes the isolation and estimation of components much more complex and difficult this year than last; (*4*) examination of the mean squares in this year's analyses reveals essentially the same large sources of variability as last year, with the mean squares roughly in the same ratios as last year, thus indicating component values similar to last year. We believe that the statements made last year about sample size based on

Table 5. Mean squares from combined analyses of variance; 6 laboratories (1, 2, 4, 8, 19, and 24), smoking all combinations of sleeves and dams with 4 and 5 high tar cigarettes but no monitors

Variation	DF[a]	Nicotine, mg	Tar, mg
Laboratories (L)	5	0.3948	23.898
Weeks (W) in L	6	0.0907	2.936
Days (D) in W in L	24	0.0254	3.470
Treatments (T)	3	0.0111	0.434
Samples (S)	1	1.7922	985.698
S × L	5	0.0131	2.672
S × W in L	6	0.0106	2.388
S × D in W in L	24	0.0106	1.595
T × S	3	0.0076	2.857
T × L	15	0.0191	6.432
T × W in L	18	0.0071	0.800
T × D in W in L	72	0.0112	1.321
T × S × L	15	0.0045	0.995
T × S × W in L	18	0.0057	0.746
T × S × D in W in L	72	0.0064	1.233
Ports	288	0.0075	1.194

[a] DF = degrees of freedom.

Table 6. Mean squares from combined analyses of variance

Variation	5 Labs. (1, 4, 8, 19, 24)[a]			3 Labs. (13, 15, 21)[b]		
	DF	Nicotine, mg	Tar, mg	DF	Nicotine, mg	Tar, mg
Laboratories (L)	4	0.5261	32.153	2	0.3952	43.857
Weeks (W) in L	5	0.1133	3.830	3	0.0913	1.390
Days (D) in W in L	20	0.0301	4.037	12	0.0235	4.948
Cigarette lots (C)	1	19.5622	4011.280	1	12.7980	2504.224
Samples (S) in C	2	0.8337	428.997	2	0.5298	317.036
Treatments (T′) in C	3	0.0125	0.836	1	0.0030	2.068
S′ × T′	3	0.0077	2.847	1	0.0001	2.311
L × S in C	8	0.0071	2.046	4	0.0160	1.450
S × W in L × C	10	0.0063	1.577	6	0.0081	0.255
S × D in W in L × C	40	0.0064	0.847	24	0.0158	1.497
C × L	4	0.0217	6.601	2	0.0453	33.765
C × W in L	5	0.0070	1.089	3	0.0846	2.372
C × D in W in L	20	0.0044	1.235	12	0.0180	2.939
T′ × L	12	0.0230	7.633	2	0.0453	7.590
T′ × W in L	15	0.0079	0.794	3	0.0015	0.568
T′ × D in W in L	60	0.0127	1.356	12	0.0097	1.623
T′ × S′ × L	12	0.0048	1.211	2	0.0013	1.578
T′ × S′ × W in L	15	0.0063	0.864	3	0.0207	0.970
T′ × S′ × D in W in L	60	0.0062	1.231	12	0.0160	2.060
Ports	300	0.0066	1.077	252	0.0128	1.580

[a] Smoked monitors and all 4 combinations of sleeves and dental dams with 4 and 5 high tar cigarettes.
[b] Smoked monitors and 4 and 5 high tar cigarettes with dams only.

components are as true now as they were then. They may be a little conservative if laboratories can reduce the "between laboratory" variability, as was indicated might have happened when the high tar cigarettes were evaluated in 1970. However, since the high tar cigarettes were from the same source both last year and this year, the possibility exists that the finding of greater variability in high tar cigarettes is a characteristic of this particular source of cigarettes rather than the reaction of the procedure to high tar lots in general. This should be investigated.

The question of whether the amount of chemical evaluation work (determination of water and nicotine) might be reduced by compositing the results from 2 or more ports before making single determinations was investigated in Study 2 with monitor cigarettes only. In this study, each of the monitor samples was smoked on 2 ports at any smoking. The 2 filters were placed together in larger flasks with twice as much reagent as for single filters. Samples were withdrawn for duplicate evaluation of water and nicotine. Evaluation of the variation among such duplicates will give an estimate of how much of the variability of "ports" under the present system of one evaluation per filter (i.e., one evaluation for each sample of 5 cigarettes smoked on a single port) is due to the chemical evaluation process and how much is due to other factors, such as variability among the individual ports of the smoking machine and variability among the samples of 5 cigarettes presented to the machine.

Evaluation is difficult because 2 laboratories either made only single readings or reported average readings in Study 2. However, comparison of the pooled value of all mean squares for duplicates in Study 2 and the pooled port mean squares for monitors in Study 1 gives the following pairs of estimates of mean squares: nicotine—port mean square = 0.004758 with 108 degrees of freedom and determination mean square = 0.001281 with 84 degrees of freedom; tar—port mean square = 0.726343 with 108 degrees of freedom and determination mean square = 0.045893 with 84 degrees of freedom.

If the duplicate determination mean square is accepted as the basic component of variance and subtracted from the port mean square, the remaining difference may be taken as the component of variance due to ports cleared of determination error. Thus we have the following pairs of components of variance

Nicotine: $\hat{\sigma}^2_{\text{detn}} = 0.001281$, $\hat{\sigma}^2_{\text{ports}} = 0.003477$

Tar: $\hat{\sigma}^2_{\text{detn}} = 0.045893$, $\hat{\sigma}^2_{\text{ports}} = 0.680450$

From the estimates of components of variance

Table 7. Comparison of variability in nicotine analysis in 1969 and 1970

Variation	1969—8 Labs.[a]		1970—6 Labs.[b]		1970—3 Labs.[c]	
	DF[d]	MS[d]	DF	MS	DF	MS
High Tar Cigarettes						
Samples (S)	1	1.7416	4[e]	0.4538[e]	2[f]	0.5298[f]
Laboratories (L)	7	0.4453	20	0.1130	4	0.2305
Weeks (W) in L	8	0.0194	24	0.0280	6	0.1309
Days (D) in W in L	32	0.0133	96	0.0148	24	0.0283
S × L	7	0.0108	20	0.0066	4	0.0159
S × W in L	8	0.0043	24	0.0069	6	0.0143
S × D in W in L	32	0.0096	96	0.0074	24	0.0195
Ports	96	0.0080	288	0.0075	216	0.0135
Monitor Cigarettes						
S	1	0.0057	1	0.0125	1	0.0000
L	7	0.2792	5	0.0468	2	0.0250
W in L	8	0.0277	6	0.0121	3	0.0095
D in W in L	32	0.0279	24	0.0046	12	0.0141
S × L	7	0.0032	5	0.0033	2	0.0012
S × W in L	8	0.0096	6	0.0016	3	0.0081
S × D in W in L	32	0.0018	24	0.0023	12	0.0084
Ports	96	0.0051	72	0.0030	36	0.0081

[a] Labs. 1, 2, 4, 8, 15, 19, 21, and 24.
[b] Labs. 1, 2, 4, 8, 19, and 24.
[c] Labs. 13, 15, and 21.
[d] DF = degrees of freedom; MS = mean squares.
[e] Pooling analyses of all 4 combinations of dams and sleeves with 4 and 5 cigarettes, Table 3.
[f] Pooling analyses, using 4 and 5 cigarettes but with dams only, Table 3.

Table 8. Comparison of variability in tar analysis in 1969 and 1970

Variation	1969—8 Labs.[a]		1970—6 Labs.[b]		1970—3 Labs.[c]	
	DF[d]	MS[d]	DF	MS	DF	MS
High Tar Cigarettes						
Samples (S)	1	19.776	4[e]	248.567[e]	2[f]	316.514[f]
Laboratories (L)	7	54.917	20	10.799	4	39.920
Weeks (W) in L	8	1.618	24	1.334	6	4.652
Days (D) in W in L	32	1.815	96	1.858	24	2.860
S × L	7	5.231	20	1.415	4	0.569
S × W in L	8	1.333	24	1.156	6	0.630
S × D in W in L	32	2.185	96	1.323	24	2.093
Ports	96	1.400	288	1.194	216	1.678
Monitor Cigarettes						
S	1	13.653	1	8.653	1	3.380
L	7	7.071	5	8.967	2	5.021
W in L	8	0.333	6	1.197	3	0.167
D in W in L	32	1.131	24	1.032	12	3.904
S × L	7	1.058	5	0.912	2	0.721
S × W in L	8	1.021	6	0.990	3	0.216
S × D in W in L	32	0.587	24	0.307	12	0.867
Ports	96	0.551	72	0.594	36	0.991

[a–f] See footnotes, Table 7.

and costs, the optimum number of determinations per port, as reviewed by Schultz (2), is given by the following equation

$$n' = \sqrt{(\sigma_S^2/\sigma_L^2) \times (C_L/C_S)}$$

where n' = optimum number of smaller units per larger unit, i.e., number of determinations per port; σ_S^2 = variance component due to smaller units, i.e., determinations; σ_L^2 = variance component due to larger units, i.e., ports; C_L = cost of

additional larger unit, i.e., cost of obtaining an additional filter collection; and C_S = cost of *additional* smaller unit, i.e., cost of evaluating an additional filter of collected material. If n' is fractional, $1/n'$ ports should be composited and evaluated once.

The two ratios of σ_S^2/σ_L^2 are 0.001281/0.0033477 and 0.045893/0.680450 for nicotine and tar, respectively, or approximately ⅓ and 1/15. Assuming that collecting the material on the filter costs 10 times as much as, the same as, or 1/10 as much as the evaluation procedure, i.e., C_L/C_S = 10, 1.0, or 0.1, the optimum numbers of determinations per filter would be estimated to be 2, 0.6, and 0.2 for nicotine and 0.8, 0.3, and 0.08 for tar. Thus, for most of the likely range of costs, it would seem that some compositing of samples would be in order.

Tables 12 and 13 of the 1969 study gave estimated differences not expected to be exceeded more often than 5% of the time for various schemes of sampling and evaluation. The ratios of the variance components for determinations and ports reported in this study (⅓ and 1/15 for nicotine and tar, respectively) were applied to the port variances reported last year, and new estimates of differences not expected to be exceeded more often than 5% of the time were computed for the same schemes, but 2 ports were composited for single evaluations of water and nicotine. All such estimates increased slightly. However, no such difference for nicotine increased more than 0.06 mg *per port* and no such difference for tar increased more than 0.2 mg *per port*. The number of duplicate samples (10 cigarettes) necessary to have a 90% chance of identifying a genuine 5 mg per port difference in tar and a 0.5 mg difference in nicotine between 2 lots utilizing compositing (if single ports on different days can be composited by holding the first filter in isopropanol-ethanol solution until the second is available) was estimated to be 23 for tar and 21 for nicotine. These can hardly be considered different from the 23 and 20 reported last year. These results indicate a very small loss in precision from compositing 2 ports before single evaluations are made.

It is recommended that the tar and nicotine method studied this year, including the option of pooling 2 filter pads for water and nicotine analyses, be adopted as official first action.

References

(1) Ogg, C. L., and Schultz, E. F., Jr., *JAOAC* **53**, 659–672 (1970).

(2) Schultz, E. F., Jr., *Proc. Amer. Soc. Hort. Sci* **66**, 421–433 (1955).