

Patent
Cross-Division

November 5, 1964.

Mr. J. A. Grove, Asst. to Vice Pres.,
The American Tobacco Co.,
111 Fifth Avenue,
New York, N. Y.

Dear Sir:

We are reporting the results of the study of cigarettes which were treated with Ethylene Oxide.

I feel a great deal of personal satisfaction in making this report. The quality of the work is excellent. Each experiment was duplicated with very close agreement. Of course, the details of procedure by which these smoking experiments were done have been perfected over a period of several years, but the actual experiment itself required about three weeks for completion. I venture to say that in another laboratory which was not specially equipped for the purpose, and lacking in our background and experience in handling tobacco, this investigation would have required from six months to a year, and the experimenter would have been mightily fortunate if results were anything like as reliable as these. Without some measure of control of the atmosphere in the laboratory and the special facilities which we have been accumulating, with your approval, during the last two years, it would have been impossible to perform an experiment of this caliber. I feel a great deal of pride in the efficiency and thoroughness with which the men in this Department can solve problems of this sort.

In this experiment a thorough analysis was made of the cigarettes before treatment, after fifteen minutes' treatment, and after thirty minutes' treatment. Both main and side stream smoke were analyzed for Nicotine, Ammonia, and Acids. In addition to this the Ammonia and Nicotine were determined in the butts of the cigarettes so that we could determine the total percentage of Nicotine recovered, or indirectly the percentage destroyed. In the attached tables are given the complete analyses of the tobacco and the smoke.

Briefly, this experiment shows conclusively that Nicotine is not destroyed nor driven off from the tobacco. The Nicotine enters into chemical combination with Ethylene Oxide producing a compound which is sufficiently stable to resist standard analytical methods of separation. It is not sufficiently stable, however, to resist the heat of smoking, and consequently a considerable proportion of the Nicotine which did not appear in the analysis of the tobacco did appear in the analysis of the smoke after it had been liberated by the heat of the burning cigarette. This is shown by the total recovery of Nicotine from the three lots of cigarettes which was as follows:

<u>Untreated</u>	<u>15 Min. Treated</u>	<u>30 Min. Treated</u>
65%	83%	112%

In other words, there is an actual reduction of Nicotine in the smoke of the cigarettes but it is far less than would be predicted from the analysis of the tobacco. In line 17 the indicated reduction in Nicotine due to the fifteen minute treatment is 33%, and due to the thirty minute treatment 57%. In the main stream smoke, in which we are chiefly interested, reduction by comparison is 16% and 30%. In the side stream smoke the reduction is 24% and 31%. The reduction in Nicotine from the smoker's standpoint is, therefore, much more apparent than real.

This is also shown in line 18 which I am recapping below. This gives the percentage of total Nicotine in the tobacco which is transmitted to the smoke. In order to make the picture complete and account for all the Nicotine present, it was necessary for us to analyze the butts and make the proper correction. They also are given in the table together with the percentage recovery. Obviously the percentage of Nicotine not recovered in the smoke or the butts has been destroyed.

	% Nicotine in the Tobacco which is Transmitted to the		<u>% Butts</u>	<u>% Recovery</u>
	<u>Smoke</u>			
	<u>Main Stream</u>	<u>Side Stream</u>		
Untreated Cigs.	22	40	8	65
15 Min. Treated Cigs.	29	48	6	83
30 Min. Treated Cigs.	35	65	12	112

These percentages are based on the Nicotine content of the actual weight of tobacco smoked.

A brief discussion of the significance of the analysis of the tobacco is given below:

NICOTINE AND TOTAL VOLATILE BASES (1 & 2)

The effect upon the Nicotine has been discussed. The Total Volatile Bases, which we have found by experiment and observation to approximately measure the strength of tobacco, are, of course, reduced since they include the Nicotine. They are reduced only by the amount of the Nicotine, and other bases, chiefly Ammonia, have not been affected.

RATIO NICOTINE TO TOTAL VOLATILE BASES (3)

We have found this ratio in tobacco useful in characterizing different types of tobacco. For example, the better grades of Burley which we use have a ratio of 0.45. Bright tobacco has a much higher ratio, approximately 0.70 to 0.75, while Turkish, due to its very low Nicotine content, has a much lower ratio than Burley. It is curious that the thirty minute treated tobacco has a ratio very much like that of Turkish tobacco, which, of course, does not mean that it has an aroma similar to Turkish tobacco, but it does indicate that the smoke from this tobacco would have a Nicotine and Ammonia content somewhat like that of Turkish tobacco.

AMMONIA (4, 5, & 6)

There is a negligible change in the Ammonia content of the tobacco and this is borne out in the analysis of the smoke. The Total Ammonia content is made up of what we describe as "free" Ammonia, which is very easily dislodged, and "hydrolyzed" or liberated Ammonia. We think that hydrolyzed Ammonia plus the Free Ammonia gives us a very good value for the Ammonia strength of the tobacco. It will be noted that the Free Ammonia is very, very slightly reduced, whereas the liberated Ammonia has not changed at all.

SUGARS (DEXTROSE) (7, 8, & 9)

The Sugars were determined to establish the uniformity of the blend, and the Sugars After Inversion indicate that the percentage of casing in each lot is in close agreement.

TOTAL NITROGEN - PROTEIN NITROGEN (10, 11)

These two values indicate that there has been no elimination of nitrogenous constituents from the tobacco.

ACIDS (12, 13)

The Total Volatile Acids and the Formic Acid indicate us close agreement as can be expected by analysis of these constituents. In short, there has been no effect upon the Acid constituents of the tobacco. You will recall that the patent claims a reduction in acidity. This is true to some extent as is indicated by the pH (14) which shows a slight increase in alkalinity or a reduction in acidity of the tobacco. This is not due, however, to any change in the Acids themselves, but evidently is due rather to a change in the character of the Nicotine which makes it a stronger base or alkali. This view is further substantiated by the fact that the percentage of Oxalic Acid (15) has not changed.

ASH (16)

This determination was made merely to confirm again the uniformity of blend in these three lots of cigarettes. The indications are that they are in close agreement.

In the analysis of the smoke, both main stream and side stream, it is interesting to note that there has been absolutely no change in the Ammonia content; on the contrary, there is indication of a slight increase. This increase is not sufficient to compensate for the reduction in Nicotine, and the Total Volatile Bases here which in general measure the strength of the smoke actually show a reduction amounting to about 5% in the fifteen minute treated and 14% in the thirty minute treated cigarettes. This compares to an indicated reduction of 21% and 33% respectively based on the analysis of the tobacco.

With respect to the Acids in the smoke, both main and side stream, it is very interesting to us that for all practical purposes there is absolutely no change in the amount of Acids produced. We have found this repeatedly to be the case in the blending of tobaccos. The Acids, regardless of the type of tobacco used, are subject to very little change under normal smoking conditions. Since they do not change the smoke is influenced mainly by a variation in the Nicotine and Ammonia content of the tobacco, and hence of the smoke.

There is nothing in these data to indicate that the burning quality of the tobacco has been affected by the treatment, all effects observed being directly attributable to the reaction of Ethylene Oxide with the Nicotine to produce a more stable compound.

It will probably be interesting to you to note the difference between the main stream and side stream smoke. You will observe, for example, in an untreated cigarette that about 22% of the Nicotine from the tobacco actually consumed in smoking is transmitted to the main stream, whereas nearly double this amount escapes in the side stream. You will

November 5, 1934.

also note that the Ammonia content of the smoke in the main stream is about 0.04%, whereas the Ammonia content of the side stream is about 1.10%. In other words, the Ammonia produced in the side stream smoke is between 25 and 30 times as great as that produced in the main stream smoke. As you know, the side stream smoke is extremely obnoxious, irritating to the eyes, and harsh when accidentally inhaled. Because the side stream smoke is so irritating and at the same time so strongly ammoniacal, we were first led to study Ammonia in its relation to mildness in cigarette smoke.

In addition to the experimental data here given, the actual nature of the compound formed between Nicotine and Ethylene Oxide has been pretty well established in the laboratory.

Very truly yours,

H. R. Hamner
RESEARCH DEPARTMENT

HRR:WPH