

INTERNAL CORRESPONDENCE

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FROM	<u>DR. R. M. MATULIS</u>	DATE	<u>SEPTEMBER 10, 1980</u>
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EXPLORATORY RESEARCH DIVISION

Ion Exchange Removal of Nitrate - Search continues for a salt other than chloride to enable simple one-step column regeneration. Mono- and polyvalent phosphate, citrate, and malate are ineffective. Currently, resin life is being determined using a more concentrated extract to get a better estimate of process costs.

Alternate Filter Materials - All polymers have been coated on alumina and pumped down under high vacuum. About a third of these have developed unacceptably high odor levels after two to three weeks in closed jars. This is believed due to solvent and monomer diffusing to the surface of the polymer coating. These samples are being pumped down a second time.

PJS Fill Value Improvement - To substantiate previous results showing fill value increase, further samples of R&D shredded PJS and Development Center cut PJS have been streamed and submitted for analysis. The extruder die, modified to reduce the wall thickness of PJS hollow tubes from 17 to 10 mils, was unacceptable. Roughness on the interior surface of the die caused the tubes to tear as they were extruded. A new die is being made.

CO Selective Filtration - We are in the process of determining the effect of resin aging in cigarettes on CO filtration. Diamond Shamrock high surface area polystyrene ES-861 is being used at two levels of PdCl₂/cupric salts.

Superabsorbents - An equilibrium moisture study with 1% Henkel 5025 superabsorbent on tobacco is in progress.

Cigarette Paper - Reducing cigarette paper thickness from 1.6 mil to 1.2 mil (same permeability) has no effect on CO/tar.

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ANALYTICAL RESEARCH DIVISION

Flavor Isolation and Identification

Amadori Compounds - The following compound preparations offered positive improvement on the smoking qualities over control cigarettes.

	<u>Rating</u>
1) fructose-valine, purified	good
2) fructose-lysine (I), purified (note: tentatively identified as α -di-fructose-lysine)	excellent
3) glucose/lysine reaction mixture	excellent
4) fructose-glutamic acid, purified	good
5) fructose-aspartic acid, purified	good
6) xylose/tryptophan reaction mixture	excellent

The following new preparations are being evaluated by Product Development: fructose-isoleucine, fructose-leucine, fructose-tryptophan, xylulose-isoleucine, xylulose-methionine, xylulose-4-aminobutyric acid, xylulose-proline, xylose/lysine reaction mixture in methanol, xylose/lysine reaction mixture in 95% ethanol, lactose/lysine reaction mixture in 95% ethanol, α -fructose-lysine, ϵ -fructose-lysine, and α , ϵ -di-fructose-lysine.

DIET Process - Roasted/Toasted - Using a new sample preparation technique, a second GC⁺-MS-DS analyses at Environmental Consultants was successful. A total of twelve pyrazines was identified and semi-quantitated using the total ion count from the mass spectrometer. The effect of the DIET process on pyrazine formation is seen below:

PYRAZINES IN DIET TREATED TOBACCO

<u>Pyrazine Compound</u>	<u>ET-1 Core Blend Control</u>	<u>Final DIET Treated</u>
Pyrazine	1.0	N.D.
2-Methyl	1.0	1.79
2,5-Dimethyl	1.0	12.83
2,6-Dimethyl	1.0	5.98
2,3-Dimethyl	N.D.	3344
2-Methyl-6-Ethol	N.D.	3601
2-Methyl-5-Ethol	N.D.	1503
Trimethyl	1.0	13.28
C ₄ Alkyl	N.D.	4802
Tetramethyl	1.0	2.75
2-Methyl-6-Vinyl	1.0	16.90
C ₅ Alkyl	N.D.	564

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This large increase in pyrazine levels is mostly responsible for the roasted-toasted odor of DIET treated tobacco.

Tip Volatile - No progress this month.

Data Base Management Computer System - Six weeks of successful computer system service to the cigarette physical testing lab has been completed. No major problems of any kind have been encountered and no indications of any significant future difficulties have been found. Continued application of the computer system in parallel with normal manual lab operations will allow a detailed evaluation of the system's performance. Minor adjustments to some of the computer programs have been made to optimize their function. These "fine tuning" efforts will continue as more system features are activated.

Several areas of the computer system have been completed and are nearing implementation. Programs to provide security back-up and restoration of data have been completed and successfully tested. Manual data entry programs are finished and testing is in progress. This feature will provide an efficient means of entering data from analytical methods that are not connected to the computer system.

Programs for the master scheduling function are done and testing is in progress. The master scheduling function will control the 24 hour per day, 365 day per year automatic operation of the computer system. Programs to allow entry of worklist and sample verification status are complete and testing is beginning.

The problem recently encountered with the PAMILA-MERLIN interface has been studied by a Hewlett-Packard systems engineer. The difficulty was traced to computer programs supplied by H-P. He is consulting factory specialists and should resolve the problem within the next few days. The PAMILA-MERLIN interface programs are nearing completion and testing should begin within two to three weeks.

Methods Development

Separation of Puffed Tobacco From Blend - A head-on test was carried out evaluating the heptane and liquid N₂ methods of determining puffed tobacco in cigarette blends. A statistical evaluation of this work is in progress.

Results from a brass cylinder fill value test shows no significant difference in fill value of samples of G-13 either treated with liquid N₂ or not exposed to liquid N₂.

Special samples were analyzed by the liquid N₂ technique; BARCLAY shifts 1 and 2 (8/19/80) D. Frank (4) Brands and J. F. Nall (8/28/80)

Firmness Integrator Standard - Three samples of silicone rubber were received from SWS silicones with a sample of silicone oil to be used as a softening agent. These rubbers are all from temperature vulcanizing materials.

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Three samples of a PVC type elastomer were received from the M.F. Manufacturing Company in Texas. The PVC material is all moderate temperature vulconizing plastic.

Sample firmness standards are being cast from the materials received from both suppliers in two different sized molds.

FTC Tar by Colorimetry - Sufficient routine data has been logged to permit the construction of a calibration curve. This will allow future routine data to be expressed directly in tar delivery units.

Estimation of Dry Tar From Wet Tar - A follow-up memorandum has been written on the utility of a statistical estimation of dry tar from wet tar weights without the need to measure TPM water. The new technique is shown to give results that are well within the 95% confidence limits of tar estimation by the normal FTC method.

HPLC Analysis for Individual Sugars - Work on this project has been completed. The methodology is being typed.

Improved Acrolein Analysis - We received the Waters Radical Compression column system last month and planned to start on this project early this month. However, the columns supplied were not able to be used and we are waiting for a new shipment of replacement columns.

Automated Methods

Smoke Nicotine - A new system has been developed and tested which is less complex than the existing procedure, but can conservatively operate at a 50% faster rate of analysis and with a ten-fold increase in analytical sensitivity. The method will be implemented upon purchase and receipt of new autoanalyzer equipment.

Infralyzer/Blend Composition - Technicon Industrial Systems has agreed to carry out a preliminary study on the feasibility of using the Infralyzer for the determination of blend composition. They are now formulating the experimental design.

STATISTICS

Predicting tar from wet tar - Estimation model refined to predict tar better in low tar range.

Review an experimental design - Southhampton DIET Experimentation. Statistical comments given in hand written form.

Macon High Speed Fabrication Machines Capacity Study - Sample size options given. Resolution in Speed Test and Sampling Scheme for Manufacturing trial are provided.

Liquid N₂ G-13 Flotation Method Study - The results show that the Liquid N₂ method is better than the heptane in % G-13 floated and in method precision.

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Strip bulking Trials - Wilson GLT sampling design to test bulker effect on particle size distribution was given.

Macon Dry Weight Blending at Grade-Bin Area - The results from simulation study showed that the deviation from target weight for each grade depends on the tobacco moisture variation at exit of conditioning cylinder.

Grade-Bins Bulker Study - Preliminary analysis shows that use of grade bins bulkers reduces between hogshead variability and the number of alkaloid classes needed. Investigating problem of hogshead misclassification.

Sensory and Preference Research - Doing preliminary research on possible methods of analysis and waiting for data.

TECHNICAL SERVICES

Key Points

Total number of analyses performed during July and the previous three months are shown below:

<u>Month</u>	<u>Number of Analyses</u>
May	76,982
June	80,171
July	67,173
August	69,849

We plan no further work on the improvement in determining wet TPM from ultra low tar cigarettes. A "blank" adjustment is necessary to assure getting the most accurate wet TPM results. However, we plan to follow the FTC procedure, which does not utilize the "blank".

Agreement with CQA has been obtained on the installation of the flavor inspection program at the Petersburg Branch. The need for facilities, equipment and staff are under review. We expect implementation sometime during the 1st or 2nd quarter of 1981.

Incoming Materials Monitoring - While continuing to carry out development work in our incoming flavors inspection program, we analyzed many flavors for BW/BAT Export as well as for project GEMINI.

B&W Product Monitoring - Our major smoking effort during August was devoted to the BARCLAY cigarettes. The 100 mm version has been near its tar target; the King Size version has been well above its tar target. Adjustments are being made in production.

Tar deliveries of ARCTIC Lights 85 (7.5 mg/cig.) and KOOL Super Lights 85 (5.7 mg/cig.) are below their respective manufacturing targets (9 and 7 mg, respectively).

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Services to Other Departments - We have completed our work on characterization of SIR WALTER RALEIGH. The data for this work are now under analysis.

Q.A. Procedures in Development Center - We have begun to compare data obtained in Development Center at the time of manufacture with data obtained in R&D just prior to shipment. Our conclusions will be reported in October.

Analytical Methods/Equipment - Nicotine analysis of ultra low tar cigarettes using the GC nitrogen/phosphorus detector is progressing well. We are now working to interface the GC with the PAMILA computer.

Initial cross-check results with Wilson and Lexington redryer plants showed that Wilson results were low. More recent results show good agreement. In addition to alkaloid cross-checks, our plans are to add moisture cross-checks with Wilson.

We have begun experimentation with the computer SAS program for control charting firmness results. Retrieval and data analysis will be facilitated by this program.

Competitive Brand Monitoring - Reporting of competitive brand results is now on schedule. We plan to continue emphasis on competitive brands which will allow timely reporting.

R. M. M. 

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