

Charge Number: Various
Program Title: Spectroscopy and Chromatography
Program Leader: D. C. Watson
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I. TOBACCO AND FILLER

- A. Bright/Burley Ratios - Competitive Brands
- B. Mixing Cylinder Test - Residual Ethanol

II. SMOKE

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III. ADDITIVES

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- B. Methoprene - Method Development
- C. Trinity - Preblend
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V. OTHER

- A. CO - Laser Perforator
- B. Model 283B Perkin-Elmer Infrared
- C. NMR - Lab Air Handling Modification
- D. Volatile Sweep - Disposable Cambridge Pad Holders
- E. Headspace - Method Improvement
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I. TOBACCO AND FILLER

A. Quantitation of bright and burley content of ET in selected competitor's brands was completed. The data also indicated (except for PM brands using DBC blend) the general use of thin grades in expansion processes. A summary of the data is shown below.

<u>MANUFACTURER</u>	<u>BRAND</u>	<u>% ET (Flootation)</u>	<u>% Bright ET</u>	<u>% Burley ET</u>
PM	Marlboro 85 (M/C)	11	100	0
	Marlboro 85 (Louis)	11	90	10
	Merit 85	11	98	2
Reynolds	Now 85	55	53	47
	Winston 85	12	39	61
	Vantage 85	15	39	61
Lorillard	Kent III 85	23	67	33
	Kent III 100	23	67	33
	Golden Lights 85	12	95	5
	Kent 85	12	99	1
	Newport 85	11	100	0
American	Carlton 85	47	55	45
	Tareyton 85	13	58	42
	Tareyton Lights 100	15	54	46
B & W	Viceroy 85	9	71	29
	Kool Super Lights 85	9	73	27
Liggett	Decade 85	28	54	46
	Lark 85	13	51	49

B. Residual ethanol levels were determined in filler just prior to, and at the exit of, the final mixing cylinder at the Manufacturing Center. The data are summarized below on an "as received" basis. The deviation is shown at 2σ for $N=12$.

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<u>Sample Point</u>	<u>mg Ethanol/g of filler</u>
Before mixing cylinder	1.65 ± 0.36
After mixing cylinder	1.28 ± 0.42

II. SMOKE

- A. A high efficiency vacuum pump is being installed to allow puff-by-puff analyses using the 100-meter gas cell with the TDL. Four different smoking assembly designs have been evaluated for application to the unit. Since dead volume in the system affects the puff profile shape, any system used must be characterized so that valid comparisons can be made between different cigarette types.

III. ADDITIVES

- A. An off-odor in a shipment of WS-14 was characterized using head-space analyses. The chromatograms showed considerable qualitative and quantitative differences between the volatiles collected from a control versus the "contaminated" sample.

The off-odor note was especially evident in the collected volatiles of the sample. No further characterization (i.e. gc/ms) was done.

- B. A draft of the method for the determination of methoprene in tobacco ($\mu\text{g/g}$ level) has been written. This method along with procedures for Assay of KABAT^R and technical grade methoprene and a list of all recommended instruments is being transmitted to QA personnel at our stemmerys.
- C. Trinity Preblend I is being compared on a lot-to-lot basis for conformity to laboratory batches using $(\text{gc})^2$. Preblend I components will be quantitated and the method for quantitation will be transmitted to QA personnel at the M/C.
- D. A method was developed for the determination of WS-14. Copies of the procedure (Analytical Methods Manual, E-55) were provided to QA personnel.

IV. CHEMOMETRICS

- A. The headspace collection and gc analysis has been completed for the 12 Trinity model's filler (6 x 85 mm and 6 x 100 mm). The chromatograms have been collated and are ready for application of

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chemometric techniques. Data will be generated for Cambridge to allow an over-all comparison of the headspace filler volatiles of: Kent III, Cambridge, Carlton (1 mg) and Carlton 100's.

- B. Data from the 17 samples previously studied (filler head space) were used with the SPSS discriminant program. Discriminant functions were calculated that led to 100% correct classification of the 3 brands, Carlton Box (1 mg), Carlton 100's, and Kent III.

When one of the 17 sample runs was omitted from the training set and used as a training set of one, however, the results were less than convincing.

IV. OTHER

- A. Infrared analyses showed no increase in CO levels in the vacuum exhaust (Hauni-Apollo Laser Cabinet) as compared to room air.
- B. The Model 283B Perkin-Elmer Infrared Spectrometer and data station was installed. Formal operator training will follow a period of familiarization with this microprocessor controlled instrument and its dedicated data system.
- C. Both NMR spectrometers were functional following a 3-week down-time. This was necessary for installation of the additional air handling capacity needed for optimum operation of their associated computer systems. The back-log of NMR experiments and requests that occurred during the down-time is being reduced.
- D. Volatile sweeps of the disposable Cambridge filter holders showed a loss of about 0.2 mg/holder of a phthalate. This amount is not enough to account for the approximately 1.5mg/holder observed in experiments by A. Kallianos.
- E. Refinements being made to tighten control of collection and analyses of head space volatiles include addition of appropriate internal standards.

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- F. The Varian Mat Statos 3308 printer/plotter was installed as a part of the Varian 200S GC/MS/Data System.
- G. A system for the automated determination of TPM, H₂O, nicotine, and tar was installed in PM facilities in Buenos Aires, Argentina. A similar, but up-dated, system will be set-up in the Maracay, Venezuela labs during May and a 5th such unit is scheduled for Quito, Ecuador.
- H. Routine and Semi-Routine Analyses included:
1. Purity scans of alternate sources of TA.
 2. Cation content of various suppliers' lots of WS-14.
 3. Cation content and level in Tipping papers.
 4. Pyrolytic products of materials proposed for use in our manufacturing facilities.
 5. Spectral/Structure correlations of synthesized samples (IR and NMR).
 6. Acetic acid, volatile fatty acids (C₂-C₅), and sugars content as a function of tobacco storage and of bruising.
 7. Formic and acetic acids, phenol, and catechol in TPM in ageing studies.
 8. Volatile fatty acids in CEL and finished sheet as a function of denitration processes.
 9. Residual air in liquid CO₂ storage tanks.
 10. Humectants, organic acids, plastilizers, and specific flavors in tobacco, filler, sheet, paper, and filters.
- I. Completion Report, Bennie W. Good, Milton E. Parrish, & D. R. Douglas, "Volatile Phase Profiling of Mainstream Smoke," April 17, 1980, ACC. No. 80-097.
- J. Methods Development
"WS-14 in Cigarettes, Filler, and Smoke", Analytical Division Methods Manual #E-55, Mary B. Buckner, J. R. Atkinson, and D. C. Watson.

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K. Significant Memos

1. L. C. Smith to Mr. Donald Keel, "Comparison of WS-14 Flavor Samples", April 1, 1980.
2. Tom Hill and Dot Clark to Mr. E. B. Stultz, "Sampling Laser Perforator Exhaust System", April 3, 1980.
3. Gunars Vilcins and Gary Forrest to Manuel Bourlas, "Atmospheric NO₂ Study", April 16, 1980.
4. C. S. Kroustalis to Mr. A. Kallianos, "Final Mixing Cylinder Test for Ethanol - M/C", March 25, 1980.
5. R. C. Esperdy to Andy Kallianos, "Headspace Analysis of Disposable Cambridge Filter Holders", March 27, 1980.

Deane Carter

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