

**ABSTRACTS OF PRESENTATIONS MADE AT THE  
1999 CORESTA JOINT MEETING OF THE  
SMOKE AND TECHNOLOGY STUDY GROUPS  
INNSBRUCK, AUSTRIA**

*(by alphabetical order of first authors)*

**BANYASZ J.L.**

CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST3

**The hydrophobic aspect of nicotine.**

Nicotine is a fundamentally hydrophobic molecule containing two amine functions which solubilize it in water. It falls into the class of amphiphilic solutes, *i.e.* molecules with both hydrophobic and hydrophilic attributes. The opposing tendencies result in complex aqueous solution behavior typical of amphiphilic molecules. The solution behavior of amphiphilic molecules such as alcohols and amines has been extensively studied. A brief summary is given. Thermodynamic properties of the nicotine-water system, are calculated from data available in the literature and are shown to be consistent with amphiphilic behavior. The activity coefficient of nicotine, calculated from vapor pressure data, exhibits a strong positive deviation from ideality. The azeotropic behavior of nicotine is a result of this. The excess thermodynamics of solution, calculated from vapor pressure data and calorimetric measurements, are dominated by a large negative entropy indicative of the strong structure making tendency of nicotine. The partial molar volume of nicotine, calculated from density data, shows a well defined minimum at a concentration of about two mole percent. This may be taken as evidence that nicotine is enclosed in a cage of about fifty water molecules. The cage structure breaks down at higher concentrations to yield a micelle-like state with a tendency for phase separation. This results in the well known circular phase diagram of nicotine in water. Unfortunately most of the available physical chemical data for nicotine are very old and measured with antiquated techniques. Nevertheless, they suffice to demonstrate the interesting complex solution behavior of nicotine which should be of interest not only to the physical chemist but to the biochemist. Hydrophobic interactions between amphiphiles and protein have long been recognized.

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**BASKEVITCH N.; LOUREAU J.M.; LE MOIGNE C.**

CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST35

**Paper combustibility: cigarette combustibility?**

Up to the 60's, cigarette paper commercial grades were simply described as "non combustible", "combustible" or "extra-combustible". It was assumed that combustibility of the paper itself was correlated with cigarette combustibility. Since then, the characteristics of cigarette paper, like natural porosity and burning additives, which influence truly cigarette combustibility, have been described by various authors and it became clear to cigarette designers that paper combustibility was not generally a significant factor governing cigarette combustibility. On the other hand, for Roll Your Own (RYO) wrapping papers, the ECPCI has proposed to the Coresta RYO Task Force to use paper combustibility, and specially the LCT test, to classify papers between low and high smoke delivery, and a new Task Force on paper combustibility was recently set up to develop a recommended method. Results demonstrating that for papers used on cigarettes, there is no general correlation between paper and cigarette combustibility. We will also show results confirming that, for the specific group of RYO booklet papers containing no burning additives, there is a correlation between paper combustibility and FCSA's combustibility/smoke delivery. This apparent discrepancy can be explained by reviewing the role played by the various characteristics of cigarette paper in governing cigarette combustibility.

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CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST5

**Modeling nicotine intake in smokers and snuff users using biological fluid nicotine metabolites.**

While urinary cotinine is the nicotine metabolite most commonly utilized to quantify nicotine intake, many additional nicotine metabolites have been identified and quantified in urine samples. Data from two studies involving smokers and snuff users are analyzed to address estimation of nicotine intake using urinary and salivary nicotine metabolites. Without precisely known dosage, nicotine intake is approximated as a linear combination of urinary nicotine and seven urinary nicotine metabolites. Comprehensive regression modeling is performed to determine what combinations of urinary nicotine metabolites provide better estimation of nicotine intake in these subjects than the predominant practice of analyzing only urinary cotinine. Variability, both within subjects (between measurements) and between subjects, is examined with regard to reliability of measurement and replicate sampling. Salivary cotinine models are compared to urinary metabolite models. Results suggest that estimation of nicotine intake is greatly improved by measuring urinary cotinine and additional metabolites (trans-3'-hydroxycotinine, and glucuronide conjugates) over measuring only cotinine. Analyses also indicate that replicate sampling on subject greatly improves the reliability of the measurement. Based on these data, a model to predict nicotine equivalents based on saliva cotinine was severely inferior to any of the urinary models, including that of urinary cotinine alone.

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**BRANTON P.**

CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST32

**Water vapour sorption on cigarettes and their components.**

Water vapour plays an important role in the quality of a cigarette, from the moistness of the tobacco, to spotting on the cigarette paper. It is thus of interest to establish new experimental techniques to measure a range of qualities such as the kinetics of adsorption and the degree of paper staining and to understand the way in which water interacts with the cigarette and its individual components. In order to achieve this we have conducted a number of experiments using a system comprising a microbalance with a temperature and humidity controller. The correct water content in tobacco is vital for consumer satisfaction. Results are presented describing the way in which water interacts with tobacco and also for the kinetics of wetting and drying of the tobacco. The sorption of water and other vapours have been used to study the properties of cigarette paper and its components i.e. fibre and filler. These include surface area and pore size, and the types of interactions involved with both polar and non-polar species. The thermodynamics of water sorption were studied by measuring isotherms at two or more different temperatures. Cigarettes were conditioned over a temperature range of 10 to 40°C and over the maximum humidity range. Following conditioning of the cigarette, a camera was focussed on the paper and imaging software used to measure the degree of staining on the cigarette paper.

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**COLLAZO H.; CROW W.A.; GARDNER L.; PHILLIPS B.L.; MARPLE V.A.; UTELL M.J.**

CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST15

**Application of inertial impactors for the measurement of aerodynamic diameters of cellulose acetate fibers.**

The aerodynamic diameters of particles and fibers greatly influence their site of deposition and toxicity in the respiratory tract. To characterize cellulose acetate fibers released from the filter end of cigarettes, we designed a multistage impactor to determine the aerodynamic diameters of large fibers ranging from 20-50 µm in diameter and 75-1000 µm in length. One hundred cigarettes were puffed directly into the impactor; 2-10 fibers/cigarette were released and their aerodynamic diameter

determined. In two studies, we found that the aerodynamic diameters were always greater than 22  $\mu\text{m}$ . Using standard lung deposition models, we concluded that the fibers are non-respirable, would deposit primarily in the oropharynx and are not likely to penetrate into the tracheobronchial region. Based on physical characteristics, shapes and aerodynamic diameters, there is a very low probability of penetration to the distal respiratory tract. Our findings, demonstrating release of only a small numbers of fibers with an extremely low likelihood of reaching the distal lung, indicate that the fibers should not represent a health concern.

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CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST14

**Development of inertial impactors for measurement of aerodynamic diameters of man-made organic fibers.**

It is widely accepted that the aerodynamic diameter of a particle is one of the main factors which determine particle deposition into the human respiratory system. The determination of aerodynamic diameter of spheres or near spherical objects is routinely accomplished using impactors. The aerodynamic diameter of fibers, on the other hand, have not traditionally been measured by impactors, because fibers of the same cross section can have different lengths and a variety of shapes (straight, curve, etc.) for each length. Thus, the aerodynamic size of the fibers is a function of fiber orientation. Nevertheless, fibers do have an aerodynamic diameter, albeit a function of orientation, and will be collected in an impactor if the aerodynamic diameters are larger than the cut size of the impactor. Single and multiple stage impactors have been developed, calibrated and validated specifically for the determination of the aerodynamic diameter of large fibers with physical diameters of 20-50  $\mu\text{m}$  and lengths between 75 and 1000  $\mu\text{m}$ . The performance characteristics are evaluated by round particles of known diameters, fibers of known length and diameter, and computational fluid dynamic calculations. Our methodology has shown that inertial impactors can be used to experimentally determine the aerodynamic diameter of large cellulose acetate fibers.

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CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST21

**Transfer of semi-volatile components from tobacco to smoke.**

Information on the transfer rates of semi-volatile components from tobacco to smoke is of importance to tobacco scientists. However, previous studies in this area have generally focused on experimental nonfilter or king-sized filter cigarettes. There is little information in the literature on other cigarette designs that are used commercially or on analytes other than nicotine and menthol. In the early nineties, scientists at the American Tobacco Company developed a GC method for the determination of glycerol, menthol, and propylene glycol as well as nicotine and five other compounds in the 2-propanol extract generated from the FTC smoke method. In 1993, they applied this methodology to over 160 brand styles taken from the marketplace. The sample set included a wide variety of menthol and nonmenthol products ranging in nominal length from 70 mm to 120 mm and in nominal circumference from 17 mm to 25 mm. In addition, the sample set included products with a range of humectant and menthol levels. Transfer rates have been calculated for glycerol, propylene glycol, nicotine and menthol, where applicable. In particular, it was found that the transfer rates for propylene glycol were more affected by filter ventilation than were those for glycerin. This finding as well as other findings on transfer rates will be discussed in terms of cigarette designs, humectant levels, and product moistures.

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**DE VOS J.**

CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST31

**Lamina puffing in an HF field.**

High frequency (HF) heating is the term applied to the generation of heat in non-conducting materials by their dielectric losses when subjected to an alternating field of high frequency. In vegetable materials the captured HF power brings the fluid in the cell tissue to the boil and the vapor pressure developed expands the cell structure. For cut cigarette tobacco this is a low cost and environmentally acceptable method of lamina puffing. The only emission is the steam escaping from the tobacco tissue. After conditioning and pre-heating, the tobacco is expanded by passing it through a filament-reinforced epoxy resin tube placed between two electrodes across which the HF voltage is applied. The paper reviews the main performance parameters of the expansion step: permissible working stress or the tube material for the desired service life; dielectric loss properties of the tube material for determination of the HF voltage to be applied to the electrodes; effect of power input on expansion pressure; residence time for satisfactory expansion without excessive tobacco moisture loss. To progress the lamina puffing application to the industrial stage we have tentatively scheduled a 3-phase R&D program: a laboratory phase to quantify the principal process engineering and design data, including production of samples for initial filling power and smoking tests. A pilot plant phase where we check out the operating conditions for the expansion of tobacco blends in a 100 kg/h demonstration plant; the commercialization phase where we license industrial scale (1500 kg/h) tobacco expansion projects through an engineer/contractor.

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**DEUTSCH L.J.; JEFFORDS A.L.**

CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST11

**The impact of filter design on cigarette performance. Part I: vapor phase.**

Cigarette filters began to achieve widespread acceptance in the 1950's. In the United States today, 98% of all factory-produced cigarettes have filters, and worldwide, more cigarettes are filtered than not. The common aspect of all filters is that they are designed to enhance the performance of the cigarette, and by doing so, provide added satisfaction to the smoker. Most parameters utilized in filter design modify the tar yield of the cigarette by altering the filter's retention of the particulate phase. These design parameters have little impact on the vapor phase of cigarette smoke. An exception to this is the use of ventilation, which affects both the particulate and vapor phases via dilution. Apart from ventilation, changes in the delivered vapor phase of cigarette smoke by means of the filter are generally induced via chemical interaction of the vapor phase with the filter components. Unlike particulate phase modification, which is primarily dependent on the physical composition of the filter, vapor phase modification depends mostly on the filter's chemical composition. The most obvious example is the charcoal filter, which is specifically intended to moderate vapor phase deliveries. However, the choice of primary filter material (cellulose acetate, paper, polypropylene, viscose, etc.) will also have a significant impact on the vapor phase and must be considered in filter design. This study investigated the influence of filter composition on vapor phase deliveries. The effect of filter material and filter additives on vapor phase components will be discussed.

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**DJORDJEVIC M.; BARR W.H.; BRANCIFORTE S.; BURTON H.R.; JAFFE J.H.**

CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST9

**Reduced levels of 4-(methylnitrosamino)-1-(3-pyridyl)-1-butanol in smokers of cigarettes produced from nitrosamine-free tobacco.**

Model studies on lung cancer in animals have shown that adenoma and adenocarcinoma are specifically elicited by nicotine-derived tobacco-specific N-nitrosamines (TSNA), such as 4-(methylnitrosamino)-1-(3-pyridyl)-1-butanone (NNK). Lowering or entirely eliminating TSNA in tobacco and in cigarette smoke might, in theory, reduce a large portion of the cancer-causing potential of these products. Recent technologic advances have led to the production of a tobacco that is essentially free of TSNA. To examine the effects of smoking cigarettes produced from such modified tobacco on carbon monoxide (CO) burden, and nicotine uptake and metabolism, and to verify whether uptake and metabolism of NNK is indeed reduced, we determined the CO levels in expired air and analyzed urine specimens from 11 smokers who initially smoked full-flavored American blended cigarettes (FTC nicotine yield:  $\geq 1$  mg/cigarette) and then switched for 9 days to an experimental cigarette CigRx<sup>TM</sup> containing only Virginia tobacco with minimal or no TSNA and no added flavorings, but the same level of nicotine as their usual brands. Switching to the CigRx<sup>TM</sup> cigarette lowered CO levels by 40% and reduced urinary levels of the major NNK metabolites, 4-(methylnitrosamino)-1-(3-pyridyl)-1-butanol (NNAL) and its glucuronic acid conjugate, by 50-70%. Cotinine, the biomarker of nicotine exposure, remained at the levels determined when subjects were smoking their usual brands. Urinary NNAL was not completely eliminated after switching to the TSNA-free cigarette, but this finding is in line with others showing NNAL persistence well over 120 days after complete cessation of smoking. In summary, smoking cigarettes produced from TSNA-free tobacco significantly reduced the body burden of the lung carcinogen NNK without increasing the exposure to CO.

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CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST2

**The effect of ingredients added to tobacco in a commercial Marlboro Lights cigarette on FTC nicotine yield, 'smoke pH', and Cambridge filter trapping efficiency.**

The objectives of this study were to determine the effect of added ammonia compounds and other ingredients, in levels utilized in a commercial cigarette, on: (1) FTC nicotine yield; (2) "smoke pH"; and (3) Cambridge filter trapping efficiency for nicotine. Five cigarette models were examined ranging from commercial Marlboro Lights (CML) to a CML with no ingredients added to the tobacco filler. The five cigarettes and the Kentucky Reference 1R4F were tested for "soluble ammonia" in the filler and smoke ammonia, "filler pH" and "smoke pH", FTC nicotine and "tar" yield, and the trapping efficiency of the Cambridge filter for nicotine. The results from the five cigarette models indicate the following: significant increases in filler "soluble ammonia" (0.12 to 0.31% DWB) and smoke ammonia (6.1 to 11.9  $\mu\text{g}/\text{cig.}$ ) observed for those cigarettes with added ammonia compounds; small increases in "filler pH" (5.6 to 5.8) observed for those cigarettes with added ammonia compounds; no significant difference in "smoke pH" (5.3-5.4) and FTC nicotine yield (0.74-0.81 mg/cig.) in cigarettes with and without ingredients; and greater than 99% trapping efficiency of the Cambridge filter independent of the "ammonia" content of filler and smoke. The results of these studies suggest that ingredients, including ammonia compounds, added to tobacco in a commercial Marlboro Lights cigarette do not increase "smoke pH" and FTC nicotine yield or reduce the trapping efficiency of nicotine on the Cambridge filter.

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CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST20

**Experimental investigation of formation of aromatic hydrocarbon from pyrolysis of cellulosic materials.**

The major chemical pathway to produce aromatic hydrocarbons in the pyrolysis followed by flaming combustion of biomass is high temperature secondary reactions of the primary pyrolysis products. The second and less explored route that is more pertinent to lower temperature smoldering combustion, is the evolution of aromatics from the remaining char as it is further aromatized with increasing pyrolysis temperature. In this study, the pyrolysis of cellulosic materials with emphasis on the formation of aromatic hydrocarbons from the second route was investigated. Experimental work was mostly carried out in a TG/DSC/MS analyzer. In order to reduce the formation of hydrocarbons from the secondary reactions of the primary pyrolysis products, small sample size, *i.e.*, 2-10 mg, high helium flow rate of 150 ml/min, and heating rates of less than 100°C/min were used. Identities and relative quantities of masses detected by TG/DSC/MS analyzer was confirmed by GC/MS using a CDS Pyroprobe. Under slow heating conditions which is typical of thermal analyzers, hydrocarbon products begin to evolve above 350°C, where the primary decomposition of cellulosic materials has completed and the remaining char has began the carbonization / aromatization process. Detectable products above 600°C in our thermal analyzer are methane, benzene, and hydrogen. Some kinetic parameters for the formation of hydrocarbon products are deduced from the experimental findings and discussed in this presentation.

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CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST19

**Quantitative characterization of tobacco pyrolysis products using TG/FTIR.**

A TG/FTIR instrument that combines thermogravimetric analysis with evolved products analysis by Fourier transform infrared spectroscopy is a powerful thermo-analytical technique providing a quantitative time-resolved analysis of major pyrolysis products of any bio-originated material including tobacco. In this work, we demonstrate how the time-resolved spectra of TG/FTIR along with the target factor analysis (TFA) can be used to identify and then quantify the pyrolysis products. This technique was employed for the characterization of up to twenty major pyrolysis products of Burley and blended tobaccos. It was shown that tar is the primary and most abundant product of tobacco pyrolysis. Other oxygenated products such as aldehydes, acids, alcohols, and light hydrocarbons were also quantified. In order to practically use the data generated from this instrument, a non-isothermal method of determining kinetic parameters was also used to estimate the kinetic parameters of evolved products during tobacco pyrolysis. Furthermore, the application of these estimated kinetic parameters in predicting the products yields from a burning cigarette yielded a reasonable result.

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**HAMPL V.**

CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST38

**Use of pigment blends to control cigarette paper porosity.**

The porosity of cigarette paper is its most important physical property because of its impact on cigarette static burn rate, puff count, ventilation, tar deliveries and ultimately taste. The papermaker's main tools for controlling paper permeability are fiber refining and pigment level. A newly developed method for controlling permeability is to use blends of pigments, which have a dissimilar size. This method complements the existing means for permeability control and offers additional flexibility in

the paper manufacturing process. Inorganic pigments create porosity in paper by wedging themselves between fibers and preventing fiber-fiber bonding. The pore volume created depends on the number of pigment particles, their size and shape. Generally, as the particle size increases, the pore volume increases and hence porosity also increases. The new method takes advantage of this principle by blending two (or more) pigments which have different particle sizes. For example, in order to increase porosity the fraction of the larger size particle in the pigment blend is increased while the fraction of the smaller size particle is decreased. The total pigment level can be changed or kept constant. Laboratory evaluations followed by trials on a paper machine demonstrated that paper porosity could be controlled over a fairly wide range by varying the ratio of two dissimilar size pigments in a blend. The addition rate of a pigment can be changed quickly and thereby yield a faster response in porosity adjustments than changing fiber refining. Cigarette paper made by this technique is similar to paper made by conventional papermaking techniques, but there are some differences such as a slightly higher variability in paper opacity. This paper discusses further how blending of pigments affects cigarette paper properties and consequently cigarette performance.

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#### **HASELGRÜBER T.; FRÜHWIRTH H.**

CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST37

#### **Qualitative and quantitative analysis of mineral fillers in tipping paper by FTIR and FT-Raman spectroscopy.**

A method was developed which allows the qualitative and quantitative analysis of cigarette tipping paper for mineral fillers that comply with the German Tobacco Ordinance, i.e. calcium carbonate (calcite), kaolinite, talcum, titanium dioxide (anatase), aluminum hydroxide, hydrated iron oxide and amorphous calcium silicate. FT-Raman spectroscopy is employed for titanium dioxide, FTIR spectroscopy for all other fillers. The spectroscopic interference of cellulose and other organic paper constituents such as wet strength and sizing agents with the fillers necessitates the rigorous removal of organic substances prior to analysis. Paper ash was used for the analysis of talcum, calcium carbonate and amorphous calcium silicate. As for the analysis of fillers whose characteristic, completely resolved absorptions bands are thermally labile a novel sample preparation step which removes organic material by oxidation in aqueous medium but preserves the chemical and crystal structure was developed. Spectroscopic interactions between the individual fillers were studied and calibration curves were plotted. The qualitative and quantitative results from tipping papers and filler mixtures with known filler composition and content demonstrate the applicability of the developed procedure.

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#### **HWANG K.J.; RHEE M.S.; RA D.Y.**

CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST26

#### **Effect of the types of room air cleaners on the removal of ETS components.**

This study was conducted to evaluate the ability of room air cleaners to remove gases, vapor and particles from closed rooms contaminated with environmental tobacco smoke (ETS). The types of room air cleaners selected were paper filter, fiber filter, carbon filter, static precipitator and anion generator. The ETS measurements covered total suspended particle (TSP), ultraviolet particulate matter (UVP), fluorescent particulate matter (FPM), solanesol, and the following gases and vapor: carbon dioxide, carbon monoxide, nicotine, and 3-ethenylpyridine. Tobacco smoke was generated and mixed in a closed room in which the airflow rates were in the range of 0.00 - 0.03 m/s. As the room air cleaners were started, the decay rates of the ETS components were measured. After the use of the room air cleaners, excluding the paper filter cleaner, over 90% of the solid components of ETS, such as TSP, UVP, FPM and solanesol were removed, and about 50% of the vapor phase components of ETS, such as nicotine, 3-ethenylpyridine (3-EP) were eliminated. However, even after the use of the

room air cleaners, with the exception of the anion generating cleaner, the decreases in carbon dioxide and carbon monoxide concentration were insignificant compared to a control situation. Our results indicate that the removal efficiency depends on the types of filter materials in the air cleaners, and also that the reduction of the particulate and vapor components of ETS were greatest. However, there was no significant effect on reduction of gaseous components of ETS. Thus, it is possible to increase the air quality in a room contaminated by ETS if the air cleaner is carefully chosen, operated and maintained.

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**Opportunities for the use of RF heating in the tobacco industry.**

Radio frequency (RF) heating is a very long established technique widely used in the food, textile, paper and plastics industries, there are many thousands of installations. Its uses include drying, thawing, moisture conditioning and heat treatment. Products processed range from biscuits and breakfast cereals to frozen blocks of meat and 500 kg bales of wool. RF is sometimes confused with microwaves but, although it has some similarities, it is very different in many respects. Most people's experience of microwave is the domestic oven which is thought to be a means of cooking volumetrically. In fact the penetration depth in a potato in microwave oven is only about 1 cm and cooking largely depends on conduction. Industrial experience of microwave confirms that uneven heating can be a significant problem. RF and microwave are from adjacent sections of the electromagnetic spectrum with RF having significantly lower frequencies. Both heat water rather within a host material i.e. cellulose, although the mechanism of heating differ. Because RF has lower frequencies (13 and 27 Mhz) it has greater penetration depths than microwave (900 and 2450 Mhz) and it produces a much more uniform distribution of power within a product being processed resulting in more uniform heating. It is very unusual for an equipment manufacturer to offer both RF and microwave and consequently most will attempt to sell what they have available when in reality one is likely be better for a process than the other. New RF technology which uses remote generators with transmission lines has allowed RF to be installed on equipment and in situations where it was previously impracticable. This technology has already made possible significant developments in the food and chemical industries. The paper will describe work currently being undertaken by an equipment manufacturer who supplies both RF and microwave. It will cover projects which are currently self funded and which are being monitored by a number of tobacco companies. These projects include 1. Non toxic infestation control; 2. Moisture profiling of cigars; 3. On line curing of cellulose acetate filters; 4. The rapid identification of coring in cellulose acetate filters; 5. Preconditioning of die cut leaf prior to overrolling in cigar manufacture; 6. Conditioning of tobacco in the primary section; 7. On line expansion.

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CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST27

**Practical modeling of cigarette ventilation rate.**

A quantitative predicted model describing the effects of wrapping materials on the levels of filter ventilation was developed and evaluated. The development of the model was expressed in terms of a quadratic polynomial relationship which was validated with experimental measurements for numerous porosity of plug wrap and tipping paper, unencapsulated pressure drop of filter plug and cigarette column and vent position. Five independent factors were chosen for their effects on the various responses. Forty-six trial numbers were obtained as a results of using by Box-Behnken design, and it was analyzed by multiple regression analysis with forward stepwise in STATISTICA/pc. Practical cigarette ventilation was affected linearly by porosity of tip paper (5.62),

vent position of tip paper (3.43), pressure drop of cigarette column (2.12), porosity of plug wrap (2.06) and filter pressure drop (0.12) in the decreasing order, and also the pressure drop of cigarette column and vent position of tip paper interaction F ratio among five factors had a P-value of 0.082, indicating comparatively higher interaction and significant at the level of 0.1 (a) between these factors. Based on the analysis of variance, the model fitted for ventilation was significant at 1% confidence level and the coefficient of determination (0.83) was the proportion of variability in the data fitted for by the model.

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CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST13

**Computational analysis of flow through perforations in tipping paper.**

Filter ventilation is an essential technique to reduce tar and nicotine in cigarette smoke and an estimate of filter ventilation is required to design cigarettes. There are several mathematical systems that calculate filter ventilation based on pressure drop about the axial direction of cigarette. Air flow through perforations in tipping paper enters the cigarette in a radial direction. It is necessary to obtain not only the axial property of a cigarette but also the three dimensional property of flow around the perforations. First, three dimensional numerical analysis of flow was performed on models of measuring permeability of laminates with tipping paper and outer and inner plug wrappers. Calculated permeabilities were compared and found to agree approximately with the experimental values. The effectiveness of the method was confirmed and it suggested that the method could estimate permeability on various combinations of materials. Second, the numerical analysis carried out on cylindrical models simulated a portion of a tip of a cigarette with a dual filter. Properties of flow from the analysis suggested that using high permeability paper as the outer plug wrapper makes higher filter ventilation than locating high permeability paper as an inner wrapper. This effect was confirmed by measuring the permeability of laminates with tipping paper and two plug wrappers and filter ventilation on actual cigarettes.

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CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST23

**An automated method for determination of "organic" potassium in tobacco.**

During the CORESTA meeting in Budapest in 1993, Nicolas Baskevitch and James Horler presented a new chemical index related to tobacco combustibility: "organic" potassium expressed as  $K_2O$  and defined as the total potassium minus potassium which is theoretically combined to chlorides and sulphates. It is called "organic" because it corresponds to the potassium combined with anions of organic acids such as malate, oxalate or citrate. Over the years, this index has been used to predict the combustibility of single grades of tobacco, to adjust the burn rate of reconstituted tobacco and as a blending tool to optimise the burning rate of cigarettes. This index has shown to be particularly useful in countries producing slow burn rate tobaccos and cigarettes. The traditional chemical methods to measure potassium, chloride and sulphate content needed to calculate the "organic" potassium index are time consuming as they require several preparation steps such as calcination, acidification and filtration. This paper describes the development of a continuous flow analysis for the simultaneous determination of potassium, chloride and sulphate in tobacco in order to save time. An interference of phosphates on sulphates measurement was detected. A method using calcium nitrate was set up to eliminate phosphate prior to the analysis. The interference problem being solved, the conclusion is that the auto analyser method is efficient. It is more accurate than the traditional chemical methods and it allows to increase the number of analyses from six to hundreds of samples per day.

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CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST8

**The development of low TSNA air-cured tobaccos. II. Effects of curing conditions and post-curing drying on TSNA formation.**

As a part of the project to produce dark air-cured tobaccos with low TSNAs and other acceptable characteristics, studies were conducted to evaluate the effect of varying curing conditions. Very low levels of TSNAs were observed when tobaccos were cured under roofed, open-sided sheds. Similar levels were obtained from tobaccos cured under the small, plastic covered, portable frame structures used for curing mechanically harvested burley tobacco. In contrast, the TSNA levels were much greater in tobaccos cured in old, log, flue-cured barns. Several experiments in which tobacco was misted with an ascorbic acid solution during curing as a possible means of reducing nitrite and TSNA levels will be discussed. Results of a study on the effect of post-curing drying conditions suggest temperature is very important in determining TSNA levels. The importance of certain curing conditions on the production of low TSNA tobaccos will be discussed.

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**LOUREAU J.M.; HAMPL V.; BASKEVITCH N.; DEBERLY S.; LE MOIGNE C.**

CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST34

**The role of cigarette paper in sidestream reduction.**

The paper will first review how visible sidestream smoke reduction can be evaluated, either during puffing or during smouldering, and expressed on an amount of "tar" per cigarette basis or on a rate of generation basis. Different results can be expected if puff number varies. We will then review the three main mechanisms by which sidestream smoke can be reduced: either modifying mass balance by reducing the amount of tobacco burnt between puffs, or by trapping sidestream smoke inside the tobacco rod, or finally by a more complete combustion of sidestream smoke. Various techniques to reduce sidestream delivery will be shown, including modification of cigarette design and/or using cigarette papers with specific characteristics. In each case, an attempt will be made to attribute the observed reduction of sidestream "tar" delivery to one or more of the three mechanisms. All measurements of sidestream smoke presented were made using the fish tail collection method proposed by Proctor (1998).

*Papeteries de Mauduit, Quimperlé, France.Schweitzer-Mauduit International, U.S.A.*

**MIURA K.; NAGAO A.**

CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST33

**Heat emission from a burning cigarette.**

The heat of combustion released during char oxidation can influence the different cigarette properties, such as burning rate and smoke formation. Heat emission of burning cigarettes has been studied to estimate the heat transfer rates during cigarette smoking. Heat flux around a burning cigarette for each position was determined by measurement with various sensors. Heat emission from the periphery and/or end of the cigarette was measured with heat flux sensors. The temperature of the solid phase was measured by an infrared thermometer in conjunction with an optical fiber. Heat of radiation, heat of convection and heat of conduction could be evaluated separately. Heat balance of the burning cigarette has been discussed. It was considered that total heat evolved in the cigarette should be larger than combustion heat of tobacco char. Most of the heat is given off to the vicinity of the cigarette, especially as heat of radiation from the surface of the cigarette paper ash. A small quantity of heat is conducted through the tobacco column.

*Japan Tobacco Inc., Tobacco Science Research Laboratory, Yokohama, Japan.*

**OGDEN M.; HAQUE A.; HEAVNER D.; DAVIS R.; RICHARDSON J.; TWIFORD K.**

CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST10

**LC/MS/MS method for determining NNAL in smokers' urine.**

A sensitive LC/MS/MS method was developed for the determination of two urinary metabolites of 4-(methylnitrosamino)-1-(3-pyridyl)-1-butanone (NNK) - a tobacco specific nitrosamine. Extraction of the two metabolites, 4-(methylnitrosamino)-1-(3-pyridyl)-1-butanol (NNAL) and NNAL-glucuronide in urine samples was accomplished by solid phase extraction (SPE). A simple, ion exchange, SPE method was developed for preparing urine samples (3-10 ml) using iso-NNAL as the internal standard (IS). NNAL-glucuronide was assayed indirectly from NNAL concentrations after treating urine samples with  $\beta$ -glucuronidase. NNAL and its IS were separated on a C18 reversed phase column (15 cm x 3 mm) by gradient elution (5% acetonitrile to 70% acetonitrile in 10 mM ammonium acetate buffer) with a flow rate of 0.8 ml/min and run time of 10 min. Both electrospray (ES) and atmospheric pressure chemical ionization (APCI) interfaces were investigated for optimum sensitivity and matrix interferences. The metabolites were determined by APCI tandem mass spectrometry by monitoring the transition ions,  $m/z$  210 @180 and  $m/z$  210 @162, for NNAL and IS, respectively. The recoveries of both NNAL and its IS were greater than 85% by SPE. Linearity of the method was demonstrated ( $r > 0.99$ ) for NNAL in the range from 100-10,000 pg/ml. Merits of this method include (a) small sample size, (b) rapid sample clean-up, (c) reduced analysis time, and (d) excellent sensitivity.

*R.J. Reynolds Tobacco Co., R & D Department, Winston-Salem, NC, U.S.A.*

**PAINE J.B. III**

CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST4

**The oxidation of nicotine with sodium hypochlorite. Identification of some of the products formed, and a rationalization of the pathways by which they arose.**

The oxidation of nicotine with sodium hypochlorite was studied. Autoanalyzer-monitored reactions showed rapid consumption of about 8 molar equivalent of oxidant. Further oxidation was slow, incomplete, and pH-dependent. Simultaneous monitoring of pH showed a rapid increase, as hypochlorite was reduced, then a long gradual decline. Since nicotine oxidation could not be driven to complete "combustion", it was necessary to identify the products formed that resisted further oxidation. Reaction extracts were examined by GC/MS. Products and mechanistic pathways were identified or inferred using a mass-spectral library and chemical logic. Ultimate products persisted in reactions with excess oxidant. Intermediates formed early in the oxidation process were found in reactions with low proportions of oxidant, which had been exhausted before all vulnerable intermediates could be further oxidized. Nicotine could be understood to generate four principal families of products, depending upon the initial site of attack on the nicotine molecule by oxidant: C-2', C-5', N-methyl, or the pyridine-pyrrolidine bond. Minor other products (chlorinated nicotinic acids, pyridones) derived from initial chlorination or oxidation of the pyridine ring. Initial attack at C-5' affords cotinine (persistent), thence on to 3-acetylpyridine (quite persistent), 3-chloroacetylpyridine and (via the haloform reaction) to nicotinic acid and chloroform (both persistent). Oxidation of the N-methyl group leads to N'-formylnornicotine (persistent), or, if the carbon is lost during the oxidation, to myosmine (vulnerable), which via inferred chlorinated pyrroles ultimately affords 3-cyanopyridine (persistent) and 3,3-dichloroacrylic acid (vulnerable). Oxidation of C-2' leads briefly to nicotyrine, thence onward to 3,3-dichloroacrylic acid and to N-methylnicotinamide. Chlorinative cleavage of nicotine affords 3-chloropyridine (stable) and N-methylpyrrolidinone. Apparent nicotine dimers were found in reactions deficient in oxidizing power, probably artifacts from intermediates not normally surviving oxidation. Also observed were N'-nicotinoylnornicotine, 3-dichloromethylpyridine, 1-(3-pyridyl)-1,2-propanedione, and cyclopropyl 3-pyridyl ketone. The formation of a number of chlorinated byproducts argued against the use of hypochlorite to destroy nicotine commercially.

*Philip Morris USA, Research Center, Richmond VA, U.S.A.*

**PARTHENOPOULOS D.**

CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST28

**Metrology at Philip Morris Europe.**

The importance of the metrology function at Philip Morris Europe, an organisation having over 40 production centres in the European, Middle Eastern and African Regions is presented. Standardisation of test methods and equipment as well as the traceability of calibration gauges to the same reference gauge are essential in order to obtain comparable results among the various production centres. The metrology function as well as the qualification of instruments and the editing of test and calibration operating procedures for this region are conducted or co-ordinated by the Research and Development Department in Neuchâtel - Switzerland. In this paper the metrology function within PM Europe shall be presented based on the measurement of the resistance to draw for which the PME R&D laboratory is accredited (EN 45001) as both a calibration and a testing laboratory. This parameter is measured according to ISO 6565:1998 using automated instruments that are calibrated by means of glass multicapillary gauges. This presentation will address issues such as the traceability of calibration standards to national standards; comparison of results among manufacturing centres; the choice, the budget as well as the computation of uncertainties. Furthermore some practical aspects related to the calibration and use of the glass multicapillary gauges will be discussed.

*Philip Morris Europe R & D, CH-2003 Neuchatel, Switzerland.*

**PEELE D.M.; GENTRY J.S.**

CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST 6

**Formation of tobacco specific nitrosamines in flue-cured tobacco.**

The nitrosation of naturally occurring tobacco alkaloids is commonly accepted as the mechanism of tobacco specific nitrosamine (TSNA) formation in tobacco. As minimal-to-no TSNA is present in green tobacco, TSNA formation and accumulation is generally considered to occur during curing. Many hypotheses have centered on microbial reduction of nitrate to nitrite and other NxOy compounds that react with tobacco alkaloids to form TSNA in cured leaf. This natural microbial process seems to be the primary source of TSNA formation in cured burley tobacco. It has been common practice to flue-cure certain tobacco types, such as Virginia tobacco, in barns using a flue-curing process. Modern day bulk tobacco barns burn propane gas in direct-fired burners. Data from 1998 and 1999 research will be presented that demonstrate the significance of direct-fired curing methods as the primary source of TSNA formation in flue-cured tobacco.

*R.J. Reynolds Tobacco Co., Merry Hill, NC, U.S.A.*

## **SCHADE K.**

CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST 25

### **Use of the CORESTA monitor test piece as a troubleshooting tool.**

The use of monitor cigarettes and control charts has become standard in nearly every smoking lab. But what can be done when the smoking results of the monitor cigarette reach control limits? There are many factors that can affect the smoking results, sometimes making troubleshooting a long, drawn out process. Thus it is helpful to know how deviation from a parameter can influence the smoking results of the monitor cigarette. In practice, small parameter deviations are particularly difficult to recognize; this is why changes in the parameters that are just barely outside of the permissible limits have been examined here. Smoking tests on a Smoking Machine RM 200 were done with the CORESTA Monitor CM 2. The following parameters were changed:

<b>Parameter</b>	<b>1st setting</b>	<b>Standard setting</b>	<b>2nd setting</b>
Puff volume	34 ml	35 ml	36 ml
Puff duration	1.8 s	2.0 s	2.2 s
Puff frequency	1 puff per 58 s	1 puff per 60 s	1 puff per 62 s
Butt length	31 mm	30 mm	32 mm
Air flow	15 mm/s	20 mm/s	25 mm/s
Conditioning humidity	50% R.H.	60% R.H.	70% R.H.
Lab humidity	50% R.H.	60% R.H.	70% R.H.

The results of the tests demonstrate which parameters require special attention when attempting to account for and eliminate deviations of the smoking results for the monitor cigarette.

*Borgwaldt Technik GmbH, Schnackenburgallee 15, D-22525 Hamburg, Germany.*

## **SCHERER G.**

CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. IG1

### **Is there compensation when switching to lower yield cigarettes?**

Compensation means a change in smoking behaviour in order to adjust the smoke uptake to the different yield of the new cigarette. In quantitative terms, complete compensation (100 % compensation or compensation index (CI) = 1) means that the smoke uptake is equal while smoking the old and the new cigarette. No compensation (0 % or CI = 0) means that the relative change in uptake is equal to the relative change in yield between the old and the new cigarette. The vast majority of studies on compensation are experimental brand-switching studies, during which the subjects are supplied with cigarettes, different in yield compared to their usual brand. Most of these investigations supply evidence for "partial" compensation ( $0 < CI < 1$ ), suggesting that cigarettes with lower yields are smoked more intensively than those with higher yields. These studies also show that changing the daily number of cigarettes smoked is not a common mechanism for compensation. Compensation indices tend to be higher (meaning more complete compensation), when changing to cigarettes with higher yields than when changing to cigarettes with lower yields. The role of nicotine in cigarette smoking has been investigated by different approaches. These include nicotine supplementation studies, studies with experimentally induced increase or decrease of the renal nicotine excretion and studies in which nicotine antagonists were applied prior to smoking. The data available provide some evidence that a subgroup of smokers might regulate their nicotine uptake. The regulation, however, is imprecise. In only a few studies, the role of other factors on compensational smoking was investigated. There is some limited evidence that "tar", taste, irritating properties of the smoke and the draw resistance of the cigarette might play a role in compensational smoking. In conclusion, the data available suggest that smokers partially compensate for a different smoke yield.

However, the factors responsible for compensational smoking have not yet been unequivocally identified.

*Analytisch-Biologisches Forschungslabor (ABF), München, Germany.*

### **SEEMAN J.I.; FOURNIER J.A.; PAINE J.B. III**

CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST 18

#### **Aspects of the thermal behavior of nicotine and its salts with carboxylic acids.**

Nicotine transfer to the gas phase from pure nicotine, nicotine carboxylic acid salts (*i.e.*, protonated nictines), and Burley tobacco was studied by thermogravimetric/differential thermal analysis/mass spectroscopy (TG/DTA/MS). Under conditions used in this study, transfer of nicotine to the gas phase occurred maximal at circa 110-125°C for both nicotine and nicotine acetate, circa 160-210°C for nicotine malates, circa 195-210°C for nicotine bis (hydrogen-(2*R*,3*R*)-tartrate), and circa 160-220°C for burley tobacco. Separate thermolysis experiments (at heating rates of about 400°C/min under a flow of air) afforded 92-94% yields of recovered nicotine from its salts with carboxylic acids. Under the same conditions, pure nicotine was transferred in yields of ca. 97%. It is concluded that nonprotonated nicotine and salts of nicotine with natural tobacco carboxylic acids will transfer nicotine to smoke with comparable yields and efficiencies during the smoking process. Co-pyrolysis of both nicotine and these salts with urea had little effect on nicotine recoveries. In contrast, co-pyrolysis of the nicotine salts with diammonium phosphate significantly reduced the yields of recovered nicotine. Most pyrolysis experiments reported in the literature involve inserting the substrate(s) into a preheated region. Such experiments have been used over the years as models for cigarette smoke formation. The experimental design herein involves heating the substrates from ambient to higher temperatures under gas flow. This allows any volatile products to escape pyrolytic destruction by distillation into a cooler environment before exposure to higher temperatures, a process in closer similarity to the behavior within a puffing cigarette. The temperatures required to transfer nicotine to the gas phase from its protonated forms, such as found in tobacco, are lower than the decomposition temperature of the nicotine superstructure. In contrast the superstructure of protonated forms of some other alkaloids decompose thermally at temperatures lower than the temperatures required to effect their deprotonation and/or evaporation.

*Philip Morris USA, P.O. Box 26583, Richmond, Virginia 23261-6583 U.S.A.*

### **STOTESBURY S.J.**

CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST17

#### **A method for assessing changes to smoke from cigarette additives based on Pyrolysis-GC-MS.**

Last year at the CORESTA Congress in Brighton a method was presented for modelling the smoke chemistry in a cigarette using pyrolysis-GC-MS. The purpose of this talk is to present some new information to demonstrate the level of confidence in relating the pyrolysis results to smoke chemistry. Components were identified in smoke from cigarettes spiked with unlabelled additives and additives labelled with <sup>18</sup>O and <sup>13</sup>C and compared with the results from pyrolysis of the same additives. The extent of degradation was reliably predicted by the pyrolysis data, and the major degradation products were also identified for each of the additives under consideration. The presentation will show how the results from pyrolysis can be used alongside other analytical methods in the assessment of potential cigarette additives. This will include a systematic approach for providing cross checks between pyrolysis data and smoke chemistry.

*British American Tobacco, Regents Park Road, Southampton SO15 8TL, U.K.*

#### **TEUFEL E.**

CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST16

#### **Application of the Laser-Doppler technique to measure the tow velocity during filter tow blooming.**

The Laser-Doppler Technique is known as an instrument for the contactless measurement of velocities in industrial environments. This principle has been applied to evaluate the dynamic behaviour of Filter Tow and fibres in of the tow as a whole, this technique also allows the detection of the speed dispersion of individual filaments at each point of the process. The resulting speed profiles describe the crimping and stretching status of the Tow throughout the entire blooming. The impact of machine design, as well as the influence of machine settings (pretension force, stretching ratio) and machine configurations (roller profiles and diameters) can be examined. The obtained speed profiles permit the development of a simplified model to describe the different stages of blooming. The model supplies the basis to optimize the blooming process with respect to yield, pressure drop variations, and tow fly (lint)

*Rhodia Acetow AG, Freiburg, Germany.*

#### **TIGAU M.; NEGOMIREANU L.; ANGHEL-POPESCU D.**

CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST24

#### **Reduction of the "tar" content of plain cigarettes: design considerations.**

Plain cigarettes have higher tar and nicotine deliveries in their smoke condensates than filter cigarettes. Nevertheless, a number of consumers choose plain cigarettes because they get more satisfaction from them. 60% of the cigarettes produced by the Roumanian National Company are plain cigarettes. The very moderate price of plain cigarettes is certainly a purchasing argument for popular classes of the country. In these conditions, our Company took a series of measures in the interest of consumer protection, reducing the "tar" of these cigarettes. The measures included the selection of an appropriate cigarette paper, the study and implementation of tobacco blends resulting in lower tar and nicotine deliveries, and eventually a reduction of the smoke yield of the products was obtained without altering their taste. Consumers were informed of the quality improvements of the products and preservation of their flavor and aroma qualities. Following these measures, tar deliveries of plain cigarettes were diminished by 4-5mg/cig and nicotine deliveries by 0.3-0.5 mg/cig.

*Soc. Nationala Tutunul Romanesc, Bucharest, Rumania.*

#### **VOLGGER D.**

CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST36

#### **The aroma of cigarette paper - characterisation with gas chromatography and principal component analysis.**

Sensory evaluation is an extremely important feature in the production of cigarettes. Beside the tobacco also the cigarette paper has an influence on the aroma of the cigarettes. Therefore a new analytical method for the determination of the aroma of cigarette paper was developed. In this work samples of cigarette paper and plug wrap paper were analysed by Gas Chromatography equipped with a Mass Spectrometric Detector (GC-MSD) in combination with Sniff Detection to study differences in the aroma. The obtained results were compared with other methods known in the literature, like Electronic Nose, Likens - Nickerson - Extraction, Solid Phase Micro Extraction (SPME), and Head Space. Samples were taken daily directly from the paper machines. The sample preparation was performed with Thermo - Extraction, which has the advantage, that no organic solvent must be used and that volatile compounds, independent from the chemical properties, can be extracted. For this purpose the paper is heated at a defined temperature under a defined flow of helium. The components, desorbed from the paper were collected on a adsorption material, in the most case Tenax TA. Glass tubes, containing this adsorption material were put directly into the injector of the gas chromatograph,

which is equipped with a Thermo - Desorption - System (TDS) and a Cold - Injection - System (CIS). For the interpretation of the chromatograms odorous regions were identified with the Sniff - detector and the individual peaks were then identified and quantified by the MSD. With the calculated concentrations a principal component analysis was performed and it could be shown, that it is possible to differentiate paper samples with regard to the used pulps and burning additives for the production. Furthermore the calculated models are suitable for a continuous quality control of the produced paper.

*Papierfabrik Wattens, Wattens, Austria.*

**WAHLBERG I.; LONG R.C.; BRANDT P.; WIERNIK A.**

CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST7

**The development of low TSNA air-cured tobaccos. I. Effects of tobacco genotype and fertilization on the formation of TSNA.**

A project having the objective to evaluate tobaccos suitable for smokeless products was initiated in 1989. In the course of this project, a broad range of genotypes was grown under a regimen of heavy nitrogen fertilization (250 lbs N/A) near Reidsville, NC, USA. All tobaccos were air-cured and evaluated with respect to selected physical, chemical and sensory characteristics. The chemical measurements included levels of nicotine, sugar, nitrate, nitrite, and tobacco-specific nitrosamines (TSNA). This presentation will deal with the results obtained with respect to tobacco genotype and TSNA formation. Our results confirm previous findings that bright tobacco varieties tend to have lower levels of TSNA than burley and dark tobaccos after air-curing. In addition, we observed that the TSNA levels are, in general, lower in bright genotypes than in cigar and other traditionally air-cured varieties and in Oriental varieties. Experiments have also been conducted to study the effects of fertilization level, type of fertilizer, and fertilization scheme on the formation of TSNA. The results obtained will be presented. Factors that are likely to influence the formation of TSNA in a given tobacco will be discussed. These include levels of precursors, *i.e.* alkaloids and nitrosating species, levels of potential inhibitors and relative rate of moisture loss from the tobacco leaf during air-curing.

*Research & Analysis, Swedish Match, SE-118 85 Stockholm, Sweden*

**YANG S.S.; SMETENA I.**

CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST1

**Analytical approaches for the determination of tobacco alkaloids.**

Tobacco alkaloids of interest include nicotine, nornicotine, myosmine, anabasine and anatabine. In the past, several analytical approaches were tested for the analysis of these compounds, involving the use of HPLC, capillary electrophoresis, and GC or GC/MSD with the combination of a solventless sampling technique, solid-phase microextraction. The experimental parameters and analytical results of these methods will be briefly reviewed as background information for the audience. The focus will remain on an improved GC-NPD (nitrogen-phosphorous detector) method, which is currently used for the determination of alkaloids in tobacco. Tobacco samples were treated with a small quantity of aqueous ammonium hydroxide solution to 'loose' tobacco tissue and to control pH, followed by extraction using organic solvent. The selection of extraction solvent influences the recoveries of alkaloids, particularly nornicotine, as well as the other problems such as carry-over at the injection liner and 'quenching' at the NPD detector. The question of whether a packed injection liner (e.g., with Carbowax/KOH on Chromsorb) is needed to resolve the carry-over problem was studied. The quenching problem of a NPD detector was reduced by increasing the split ratio, which resulted in a loss of sensitivity. Thus, a narrow bore capillary column was applied to improve sensitivity and to speed up GC analysis. More detail information along with statistical data from method validation and sample analysis will be presented.

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**YOKOYAMA T.; KANO T.; SATO T.; SATO K.; OKUMOTO Y.**

CORESTA Meet. Smoke-Techno Groups, Innsbruck, 1999, abstr. ST30

**The development to the changing arrangement technique of cut filler in cigarettes.**

Generally, the arrangement of the cut filler parallels the longitudinal direction of cigarette. We have already found that we can make the cigarette harder with randomizing the arrangement of the cut filler, because the internal stress is isotropic, and the void ratio of the cigarette increases, when the cut filler is changed to the randomized arrangement. We have developed a cigarette making machine that can change the arrangement of the cut filler in the cigarette with 8000 cigarettes per minute. And we have been able to produce cigarettes with the randomized cut filler. Moreover we have confirmed that the randomized cigarettes were harder than the cigarettes produced by the present machine. We can, therefore, decrease the amount of used cut rag by 1.8% with this machine, at the same cigarette hardness, and the other characteristics (cf. loose-ends, particle size distribution, etc.) of cigarettes are nearly equal to the present. In the future, we will apply this new machine to the new brands.

*Japan Tobacco Inc., Yokokawa, Tokyo, Japan.*