2007 CORESTA JOINT STUDY GROUPS MEETING
SMOKE SCIENCE & PRODUCT TECHNOLOGY

WORKING PROGRAMME

MONDAY 1 OCTOBER 2007

SESSION 1: **Cigarette Design**

Chairman: Y. SAINT-JALM

8:40 – 9:00		**Welcome**
9:00 – 9:20	SSPT 01	**Current profile of the tobacco industry in Korea.** RHEE Moonsoc; LEE Dong-Wook *KT&G Central Research Institute, 302 Shinseong-Dong, Yuseong-Gu, Daejeon, Rep. of Korea*
9:20 – 9:40	SSPT 02	**Market survey: study on the cigarette designs used in Europe.** LE MOIGNE C.(1); RAVERDY-LAMBERT D.(1); TARDIF N.(1); LOUREAU J.M. (2); LE BOURVELLEC G.(2) 1. *LTR Industries, Usine Le Mans, 72702 Allonnes Cedex, France* 2. *Papeteries de Mauduit, 29393 Quimperlé Cedex, France*
9:40 – 10:00	SSPT 03	**A prediction model for flow parameters and smoke yields of cigarettes smoked under arbitrary smoking regimes.** EITZINGER B. *Delfortgroup, Dr.-Franz-Feurstein GmbH, Fabrikstrasse 20, A-4050 Traun, Austria*
10:00 – 10:20		COFFEE

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SESSION 2: **Cigarette Paper**

Chairman: K. MIURA

10:20 – 10:40 SSPT 04 **Influence of ionic additives on the pyrolysis behavior of cigarette paper.**

STADLMANN K.(1); EBERHERR W.(1); KLAMPFL C.W.(1); KÖLL B.(2); ZEMANN A.(2)

1. *Institute of Analytical Chemistry, Johannes-Kepler-University Linz, Altenbergerstrasse 69, A-4040 Linz, Austria*
2. *Papierfabrik Wattens, Member of Delfortgroup, Ludwig-Lasslstrasse 15, A 6112 Wattens, Austria*

10:40 – 11:00 SSPT 05 **Paper tools to reduce formaldehyde and other carbonyl compounds in cigarette mainstream smoke.**

LOUREAU J.M.; JOYEUX T.; LE BEC L.; LE BOURVELLEC G.; LE MOIGNE C.

Papeteries de Mauduit, 29393 Quimperlé Cedex, France

11:00 – 11:20 SSPT 06 **Systematic studies on cigarette paper: simplex lattice experimental designs applied to cigarette paper components and the effects on paper characteristics and sidestream yields.**

CASE P.D.; KIMPTON H.; COBURN S.; COTTE V.

British American Tobacco, Group R&D Centre, Regents Park Road, Southampton SO15 8TL, UK

11:20 – 11:40 SSPT 07 **Influence of the origin of the cigarette paper cellulose on the smoke chemical composition and "*in vitro*" toxicity tests on mainstream cigarette smoke.**

TROUDE V.(1); ACHARD S.(1); DESTRUHAUT S.(1); HAOND C.(1); SARABIA J.(2)

1. *Altadis - Research Centre, 4 rue André Dessaux, 45404 Fleury-les-Aubrais, France*
2. *Altadis, Eloy Gonzalo 10, 28010 Madrid, Spain*

11:40 – 11:50 **Task Force Low Ignition Propensity (LIP)**

CRUMPLER L.

R.J. Reynolds Tobacco Company, R&D, Winston-Salem, NC, USA

12:00 – 13:30 LUNCH

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SESSION 3: **Agrochemicals**

Chairman: P. CASE

13:30 – 13:50	Report	**Agrochemical Advisory Committee (ACAC)** PRAT M. *JT International Germany GmbH, Diedenhofener Strasse 30, D-54294 Trier, Germany*
13:50 – 14:10	Report	**Sub-Group Pesticides** PRAT M. *JT International Germany GmbH, Diedenhofener Strasse 30, D-54294 Trier, Germany*
14:10 – 14:30	IG 01	**Trends in crop protection agent residue levels by tobacco types over time: an evaluation of the VdC database.** EBERHARDT H.-J.(1); CZECHOWICZ M.(2) 1. *Verband der Cigarettenindustrie, Neustädtische Kirchstr. 8, D-10117 Berlin, Germany* 2. *AMC Consulting, ul. Marcinkowskiego 7/5, PL-48-300 Nysa, Poland*
14:30 – 14:50	SSPT 08	**Determination of carbamates and imidacloprid residues in tobacco by LC-MS-MS.** LIU Huimin; XIE Fuwei; WANG Sheng; GONG Wei *Zhengzhou Tobacco Research Institute of CNTC, Zhengzhou, Henan, China*
14:50 – 15:10	SSPT 09	**The determination of multiple agrochemical residues in tobacco by gas chromatography-triple quadrupole mass spectrometry.** LEE Jeong-Min; JANG Gi-Chul; HWANG Keon-Joong *KT&G Central Research Institute, 302 Shinseong-Dong, Yuseong-Gu, Daejeon, , Rep. of Korea*
15:10 – 15:30		TEA

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SESSION 4: **Poster Session / Tobacco Analysis**

Chairman: J. MENDAZA

15:10 – 16:30 TEA & POSTER SESSION (See next page)

16:30 – 16:40 Report **Sub-Group Environmental Protection in Production**

WICKENDEN C.

Imperial Tobacco Group PLC, P.O. Box 244, Southville, Bristol BS99 1LQ, UK

16:40 – 17:00 SSPT 10 **The content analysis of Cr, Cd, Hg and other inorganic elements in tobacco.**

HU Qingyuan(1,3); LI Li(2); CHEN Zaigen(1)

1. *China National Tobacco Quality Supervision & Test Center, Zhengzhou 450001, China*
2. *Technical Research and Development Center, China Tobacco Chuanyu Industrial Corporation, Chengdu 610066, China*
3. *Anhui Institute of Optics and Mechanics, Chinese Academy of Sciences, Hefei 230031, China*

17:00 – 17:20 SSPT 11 **Investigation of the mechanical extraction properties of the raw materials of reconstituted tobacco sheet.**

SUNG Yong-Joo; HAN Young-Lim; BAEK Shin; KIM Kun-Soo; RHEE Moonsoo

KT&G Central Research Institute, 302 Shinseong-Dong, Yuseong-Gu, Daejeon, Rep. of Korea

17:20 – 17:40 SSPT 12 **The analytical method development of measuring ammonia gas in the headspace of Swedish style snus tins.**

VELLA J.; STEWART-JONES D.

British American Tobacco, Group R&D Centre, Regents Park Road, Southampton SO15 8TL, UK

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SESSION 4: **Poster Session**

15:10 – 16:30

SSPTPOST 01 **Impartial monitoring tool for the ASTM test E 8187-02b - Standard test method for measuring the ignition strength of cigarettes.**
ROSE N.
Borgwaldt KC GmbH, D-22525 Hamburg, Germany

SSPTPOST 02 **Potential sources of measurement error when measuring the permeability of Low Ignition Propensity (LIP) papers to ISO 2965.**
VINCENT J.H.; HANKEY J.; TINDALL I.
Cerulean, Rockingham Drive, Linford Wood East, Milton Keynes, MK14 6 LY, UK

SSPTPOST 03 **A possible test piece for size and ovality correlation in inter-laboratory trials.**
VINCENT J.H.; HANKEY J.; RUSHWORTH J.; TINDALL I.
Cerulean, Rockingham Drive, Linford Wood East, Milton Keynes, MK14 6 LY, UK

SSPTPOST 04 **Observations on the effects of air flow on smoking yields as applied to linear and rotary smoking machines.**
TINDALL I.; MASON T.
Cerulean, Rockingham Drive, Linford Wood East, Milton Keynes, MK14 6 LY, UK

SSPTPOST 05 **Adsorption and thermal release of menthol by zeolite.**
CAO Yi (1); WANG Ying(1); ZHU Jian Hua(1); LIU Chuan(2)
1. *School of Chemistry and Chemical Engineering, Nanjing University, Nanjing 210093, China*
2. *British American Tobacco, Group R&D Centre, Regents Park Road, Southampton SO15 8TL, UK*

SSPTPOST 06 **Analysis of acrylamide in mainstream cigarette smoke and effects of reducing sugars on acrylamide content.**
KIM Ick-Joong; LEE John-Tae; MIN Hye-Jeong; KIM Hyo-Keun; HWANG Keon-Joong
KT&G Central Research Institute, 302 Shinseong-Dong, Yuseong-Gu, Daejeon, Rep. of Korea

SSPTPOST 07 **Development and validation of an HPLC/UV method for simultaneous determination of 9 organic compounds.**
KA Mi-Hyun; KIM Mi-Ju; CHO Sung-Eul; KIM Yong-Ha; MIN Young-Keun
KT&G Central Research Institute, 302 Shinseong-Dong, Yuseong-Gu, Daejeon, Rep. of Korea

SSPTPOST 08 **Evaluation of the behavior of the anion in RECON extract by various adsorbents.**
HAN Young-Rim; SUNG Yong-Joo; BAEK Shin; KIM Kun-Soo; RHEE Moonsoo
KT&G Central Research Institute, 302 Shinseong-Dong, Yuseong-Gu, Daejeon, Rep. of Korea

SSPTPOST 09 **Studying effect of additives on chemical and taste characteristics of Caspian pipe tobacco.**

MOHSENZADEH R.; MORADI G.R.; SHAMEL ROSTAMI M.T.; YAZDANPANAH A.A.
Tirtash Tobacco Research & Education Center, P.O. Box 48515-155, Behshahr, Iran

SSPTPOST 10 **Study of the pyrolysis pattern and the transfer rate of the organochlorine pesticide in tobacco.**

MIN Hye-Jeong; JANG Seok-Su; KIM Ick-Joong; KIM Yong-Ha; MIN Young-Keun
KT&G Central Research Institute, 302 Shinseong-Dong, Yuseong-Gu, Daejeon, Rep. of Korea

SSPTPOST 11 **Characteristic of analytical techniques for hydrogen cyanide in mainstream smoke.**

LEE John-Tae; KIM Hyo-Keun; HWANG Keon-Joong; LEE Kyung-Gu; JANG Seok-Soo
KT&G Central Research Institute, 302 Shinseong-Dong, Yuseong-Gu, Daejeon, Rep. of Korea

SSPTPOST 12 **ISO 17025 accreditation for *in vitro* toxicology tests on cigarette smoke products.**

COOKE L.; COWIN R.; LANGFORD R.; DELVES S.; GARCIA-CANTON C.; JONES S.; BLACK S.
Advanced Technologies (Cambridge) Ltd., 210 Cambridge Science Park, Milton Road, Cambridge CB40WA, UK

SSPTPOST 13 **Toxicity assessment of gas phase cigarette smoke using cell-free method.**

PARK Chul-Hoon; SOHN Hyung-Ok; SHIN Han-Jae; LEE Hyoung-Seok; HYUN Hak-Chul
KT&G Central Research Institute, 302 Shinseong-Dong, Yuseong-Gu, Daejeon, Rep. of Korea

SSPTPOST 14 **Smoking-related changes in biomarkers of effect in exhaled breath condensate (EBC): results of a pilot study.**

SCHERER G.; HAGEDORN H.W.; URBAN M.; ENGL J.; KAVVADIAS D.
ABF Analytisch-Biologisches Forschungslabor GmbH, Munich, Germany

SSPTPOST 15 **A ring trial test of nicotine uptake determinations by multiple laboratories.**

BYRD G.; OGDEN M.
R.J. Reynolds Tobacco Company, Human Studies Division, Winston-Salem, NC, USA

SSPTPOST 16 **Adsorption behavior of propylamine on activated carbon fiber surfaces as induced by oxygen functional complexes.**

KIM Byeoung-Ku; RA Do-Young; KWAK Dae-Keun; OH In-Hyeog; JO Si-Hyung
KT&G Central Research Institute, 302 Shinseong-Dong, Yuseong-Gu, Daejeon, Rep. of Korea

SSPTPOST 17 **Development of filter material selectively decreasing B(a)P yields in mainstream cigarette smoke.**

CHENG Zhangang; CHEN Yikun; WANG Jianxin; ZHANG Chu-an; HU Suxia
Technology Center of Wuhan Tobacco (Group) Co. Ltd., Wuhan 430012, China.

SSPTPOST 18 **A study to assist in the determination of tolerance values to be applied to the analysis of fine-cut smoking articles.**

DYMOND H.F.(1); WILKES E.B.(1); KOTZIAS D.(2); HEBERGER K.(3); KEPHALOPOULOS S.(2)

1. *Consultant, European Smoking Tobacco Association, c/o 45, Monarch Way, West End, Southampton SO30 3JQ, UK*
2. *European Commission, DG Joint Research Centre, Institute for Health & Consumer Protection, Physical & Chemical Exposure Unit, T.P. 281, I-21020-Ispra (VA), Italy*
3. *Chemical Research Center, Hungarian Academy of Sciences, P.O. Box 17, H-1525 Budapest, Hungary*

TUESDAY 2 OCTOBER 2007

SESSION 5 **Smoke Analysis (1)**

Chairman: A. ZEMANN

8:20 – 8:40 SSPT 13 **Fast on-line tobacco smoke analysis with Photo Ionisation - Time-of-Flight-Mass Spectrometry - Part 1: Scientific background and basic principles.**

ZIMMERMANN R.(1,2,3); MÜHLBERGER F.(2); MITSCHKE S.(1,2); STREIBEL T.(1,2); ROSE N.(4)

1. *Analytical Chemistry, Institute of Physics, University of Augsburg, D-86159 Augsburg, Germany*
2. *Institute of Ecological Chemistry, GSF - National Research Centre for Environment and Health GmbH, D-85764 Neuherberg, Germany*
3. *BIfA - Bavarian Institute of Applied and Environmental Research and Technology GmbH, Environmental Chemistry, D-86167 Augsburg, Germany*
4. *Borgwaldt KC GmbH, D-22525 Hamburg, Germany*

8:40 – 9:00 SSPT 14 **Fast on-line tobacco smoke analysis with Photo Ionisation - Time-of-Flight-Mass Spectrometry - Part 2: Machinery.**

ROSE N.(1); MITSCHKE S.(2,3); ZIMMERMANN R.(2,3,4)

1. *Borgwaldt KC GmbH, D-22525 Hamburg, Germany*
2. *Analytical Chemistry, Institute of Physics, University of Augsburg, D-86159 Augsburg, Germany*
3. *Institute of Ecological Chemistry, GSF - National Research Center for Environment and Health GmbH, D-85764 Neuherberg, Germany*
4. *BIfA - Bavarian Institute of Applied and Environmental Research and Technology GmbH, Environmental Chemistry, D-86167 Augsburg, Germany*

9:00 – 9:20 SSPT 15 **Fast on-line tobacco smoke analysis with Photo Ionisation - Time-of-Flight-Mass Spectrometry - Part 3: First measurements with a Smoke Analyzer Prototype system.**

MITSCHKE S.(1,2); ROSE N.(3); ZIMMERMANN R.(1,2,4)

1. *Analytical Chemistry, Institute of Physics, University of Augsburg, D-86159 Augsburg, Germany*
2. *Institute of Ecological Chemistry, GSF - National Research Center for Environment and Health GmbH, D-85764 Neuherberg, Germany*
3. *Borgwaldt KC GmbH, D-22525 Hamburg, Germany*
4. *BIfA - Bavarian Institute of Applied and Environmental Research and Technology GmbH, Environmental Chemistry, D-86167 Augsburg, Germany*

9:20 – 9:40 SSPT 16 **Real-time measurement of flavour release and persistence from cigarette smoke using APCI-MS.**

COTTE V.M.E.(1); PRASAD S.K.(1); WAN P.H.W.(1); LINFORTH R.S.T.(2); TAYLOR A.J.(2)

1. *British American Tobacco, Group R&D Centre, Regents Park Road, Southampton SO15 8TL, UK*
2. *Division of Food Sciences, University of Nottingham, Sutton Bonington Campus, Loughborough, UK*

9:40 – 10:00	SSPT 17	**Multiple component analysis of mainstream cigarette smoke using Terahertz spectroscopy, comparison with standard chemical analytical methods.** CUISSET A.(1); BIGOURD D.(1); BOCQUET R.(1); HINDLE F.(1); MOURET G.(1); CAZIER F.(2); DEWAELE D.(2); NOUALI H.(2) 1. *Laboratoire de Physico-Chimie de l'Atmosphère CNRS UMR 8101, Bâtiment MREI2, Université du Littoral Côte d'Opale, 189A Ave. Maurice Schumann, 59140 Dunkerque, France* 2. *Centre Commun de Mesures, Bâtiment MREI1, Université du Littoral Côte d'Opale, 145 Ave. Maurice Schumann, 59140 Dunkerque, France*
10:00 – 10:20		COFFEE

TUESDAY 2 OCTOBER 2007

SESSION 6: **Smoke Analysis (2)**

Chairman: T. PASCHKE

10:20 – 10:30 Report **Sub-Group Routine Analytical Chemistry**
CRUMPLER L.
R.J. Reynolds Tobacco Company, R&D, Winston-Salem, NC, USA

10:30 – 10:40 Report **Sub-Group Cigar Smoking Methods**
MENDAZA J.
Altadis S.A., R&D Centre, Eloy Gonzalo 10, 28010 Madrid, Spain

10:40 – 11:00 SSPT 18 **Puff profile simulator for tobacco smoke particle diameter and mass measurement.**
McAUGHEY J.(1); REAVELL K.(2); FROST B.; McGRATH C.(1); DAILLY C.(2)
1. *British American Tobacco, Group R&D Centre, Regents Park Road, Southampton SO15 8TL, UK*
2. *Cambustion Ltd., J6 The Paddocks, 347 Cherry Hinton Road, Cambridge, CB1 8DH, UK*

11:00 – 11:20 SSPT 19 **Simultaneous on-line analysis of gas phase and particulate phase of cigarette mainstream smoke.**
ADAM T.(1,2); MCAUGHEY J.(3); MITSCHKE S.(1,2); MCGRATH C.(3); MOCKER C.(1,2); ZIMMERMANN R.(1,2,4)
1. *Analytical Chemistry, Institute of Physics, University of Augsburg, 86159 Augsburg, Germany*
2. *Institute of Ecological Chemistry, GSF - National Research Center for Environment and Health, 85764 Neuherberg, Germany*
3. *British American Tobacco, Group R&D Centre, Regents Park Road, Southampton SO15 8TL, UK*
4. *BIfA - Bavarian Institute of Applied and Environmental Research and Technology GmbH, Environmental Chemistry, 86167 Augsburg, Germany*

11:20 – 11:40 SSPT 20 **Sidestream smoke analysis with Single Photon Ionisation/Time-of-Flight Mass Spectrometry (SPI-TOFMS).**
MITSCHKE S.(1,2); ADAM T.(1,2); BAKER R.R.(3); ZIMMERMANN R.(1,2,4)
1. *Analytical Chemistry, Institute of Physics, University of Augsburg, D-86159 Augsburg, Germany*
2. *Institute of Ecological Chemistry, GSF - National Research Center for Environment and Health GmbH, D-85764 Neuherberg, Germany*
3. *British American Tobacco, Group R&D Centre, Regents Park Road, Southampton SO15 8TL, UK*
4. *BIfA - Bavarian Institute of Applied and Environmental Research and Technology GmbH, Environmental Chemistry, D-86167 Augsburg, Germany*

11:40 – 11:50	Report	**Sub-Group Monitoring and Maintenance of Physical Test Methods** ROSE N. *British American Tobacco (Germany), Weiherstraße 26, D-95448 Bayreuth, Germany*
12:00 – 13:30		LUNCH

WEDNESDAY 3 OCTOBER 2007

SESSION 7: **Smoke Analysis (3)**

Chairman: L. CRUMPLER

8:20 – 8:40 SSPT 21 **Determination of "Hoffmann Analytes" in cigarette mainstream smoke. The CORESTA 2006 collaborative study.**

INTORP M.(1); PURKIS S.(2); WHITTAKER M.(3); WRIGHT W.(4)

1. *Imperial Tobacco Group, Albert-Einstein Ring 7, 2761 Hamburg, Germany*
2. *Imperial Tobacco Limited, P.O. Box 525, Southville, Bristol BS99 1LQ, UK*
3. *Statistical Consultant to Imperial Tobacco Limited, Bristol, UK*
4. *Labstat Inc, 262 Manitou Drive, Kitchener, Ontario N2C1L3, Canada*

8:40 – 9:00 SSPT 22 **Determination of nitrogen oxides in cigarette mainstream smoke by ion chromatography.**

WU Mingjian; HE Zhihui; LIANG Wenliu

Technical Center of Hunan Tobacco Group, Changsha 410001, China

9:00 – 9:20 SSPT 23 **The effect of blend type on mainstream and sidestream Hoffmann analyte machine yields.**

WINTER D.; CASE P.; COLEMAN M.; DITTRICH A.; WARREN N.

British American Tobacco, Group R&D Centre, Regents Park Road, Southampton SO15 8TL, UK

9:20 – 9:40 SSPT 24 **Study on puff-by-puff deliveries of the volatile and semi-volatile organic acids in TPM of mainstream cigarette smoke.**

ZHAO Xiaodong; XIE Fuwei; LU Ximei; LIU Huimin

Zhengzhou Tobacco Research Institute of CNTC, Zhengzhou, Henan, China

9:40 – 10:00 SSPT 25 **Artifact formation during the analysis of tobacco specific nitrosamines in sidestream smoke.**

GLAZIER M.; VAN HEEMST J.D.H.

British American Tobacco, Group R&D Centre, Regents Park Road, Southampton SO15 8TL, UK

10:00 – 10:20 TEA

WEDNESDAY 3 OCTOBER 2007

SESSION 8: **Smoke Analysis (4)**

Chairman: A. ZEMANN

10:20 – 10:40 SSPT 26 **Determination of major semi-volatile compounds in mainstream cigarette smoke.**

XIE Fuwei; ZHAO Ge; ZHAO Le; WANG Sheng; XIA Qiaoling

Zhengzhou Tobacco Research Institute of CNTC, Zhengzhou, Henan, China

10:40 – 11:00 SSPT 27 **Investigation of free radicals in cigarette smoke by Electron Spin Resonance.**

GHOSH M.(1); MCAUGHEY J.(1); IONITA P.(2)

1. *British American Tobacco, Group R&D Centre, Regents Park Road, Southampton SO15 8TL, UK*
2. *Institute of Physical Chemistry, 202 Spl. Independentei, Bucharest 060021, Romania*

11:00 – 11:20 SSPT 28 **Analysis of vapor and particulate phase free radicals in mainstream cigarette smoke.**

TAKANAMI Y.; MORIYAMA T.

Japan Tobacco Inc., Tobacco Science Research Center, 6-2, Umegaoka, Aoba-ku, Yokohama, Kanagawa 227-8512, Japan

11:20 – 11:40 SSPT 29 **The scavenger effects of various antioxidants in cigarette filters on the free radicals in mainstream smoke.**

PARK Jin-Won; KIM Soo-Ho; KIM Jong-Yeol; KIM Chung-Ryul; RHEE Moonsoo

KT&G Central Research Institute, 302 Shinseong-Dong, Yuseong-Gu, Daejeon, Rep. of Korea

11:40 – 12:00 SSPT 30 **Investigation of the thermal decomposition and pyrolysis behavior of monomenthyl succinate by non-isothermal TGA and on-line PyGC/MS.**

YANG Liu(1); MIAO Mingming(1); WU Yiqin(1,2); XIANG Nengjun

1. *R&D Center of Hongta Tobacco (Group) Co., Ltd., Yuxi 653100, China*
2. *College of Chemical Science and Engineering, Yunnan University, Kunming 650091, China*

12:00 – 13:30 LUNCH

WEDNESDAY 3 OCTOBER 2007

SESSION 9: **Filters (1)**

Chairman: L. CRUMPLER

13:30 – 13:50	IG 02	**An effective product evaluation strategy for PREP cigarettes.** DOOLITTLE D.J.; COOK C.J. *R.J. Reynolds Tobacco Company, R&D, Winston-Salem, NC, USA*
13:50 – 14:10	SSPT 31	**Filters for ultra-slim cigarettes: flow rate and pressure drop performance.** RASMUSSEN G.T.; WATTS A.S. *Eastman Chemical Company, P.O. Box 511, Kingsport, TN 37662, USA*
14:10 – 14:30	SSPT 32	**Effects of triacetin on the retention of acetate filters with dispersed additives.** SCHÜTZ E. *Rhodia Acetow GmbH, Engesserstr. 8, D-79108 Freiburg, Germany*
14:30 – 14:50	SSPT 33	**The adsorption of various smoke compounds by activated carbon.** TAYLOR M.J.; WALKER J. *Filtrona Technology Centre, Shaftesbury Avenue, Jarrow, Tyne and Wear, NE32 3UP, UK*
14:50 – 15:10	SSPT 34	**The influence of cigarette design on the ageing of carbon filters.** PETERS G.(1); MUELLER C.(1); WALKER J.(2); TAYLOR M.J.(2) 1. *Imperial Tobacco Group, Albert-Einstein-Ring 7, 22761 Hamburg, Germany* 2. *Filtrona Technology Centre, Shaftsbury Avenue, Jarrow, Tyne and Wear, NE32 3UP, UK*
15:10 – 15:30		TEA

WEDNESDAY 3 OCTOBER 2007

SESSION 10: **Filters (2) / Pyrolysis**

Chairman: P. CASE

15:30 – 15:50	SSPT 35	**Physical properties of carbons prepared from a coconut shell by steam activation and chemical activation and the influence of prepared activated carbon on the delivery of mainstream smoke.** KO Dong-Kyun; SHIN Chang-Ho; LEE Young-Taek; JANG Hang-Hyun; RHEE Moonsoo *KT&G Central Research Institute, 302 Shinseong-Dong, Yuseong-Gu, Daejeon, Rep. of Korea*
15:50 – 16:10	SSPT 36	**The effect of activated carbon characteristics on adsorption efficiency of VOCs in cigarette smoke.** SASAKI T.; YAMASHITA Y. *Japan Tobacco Inc., Cigarette Materials R&D Division, Yokohama, Japan*
16:10 – 16:30	SSPT 37	**The activity of different carbon weights in a cigarette filter and the effects of triacetin.** WALKER J.; TAYLOR M.J. *Filtrona Technology Centre, Shaftesbury Avenue, Jarrow, Tyne and Wear, NE32 3UP, UK*
16:30 – 16:40	Report	**Task Force Plasticisers in Charcoal Filters** TAYLOR M. *Filtrona International Ltd - Tech. Centre, Shaftsbury Avenue, Jarrow, Tyne & Wear NE32 3UP, UK*
16:40 – 17:00	SSPT 38	**Comparison of pyrolysis pattern of different tobacco leaves by double-shot pyrolysis-GC/MSD method.** LEE Chang-Gook; LEE Jae-Gon; JANG Hee-Jin; KWON Young-Ju; LEE Jang-Mi; KWAG Jae-Jin; RHEE Moonsoo *KT&G Central Research Institute, 302 Shinseong-Dong, Yuseong-Gu, Daejeon, Rep. of Korea*
17:00 – 17:20	SSPT 39	**Volatile products from pyrolysis, oxidative pyrolysis and combustion of tobacco.** NAPPI L.; LIU Chuan *British American Tobacco, Group R&D Centre, Regents Park Road, Southampton SO15 8TL, UK*
17:20 – 17:40	SSPT 40	**The effect by the side chain structure of amino acids on the generation of aromatic amines in tobacco pyrolysis.** YOSHIDA S.; UWANO Y.; KUSAKABE T. *Japan Tobacco Inc., Tobacco Science Research Center, 6-2, Umegaoka, Aoba-ku, Yokohama, Kanagawa 227-8512, Japan*

THURSDAY 4 OCTOBER 2007

SESSION 11: *In vitro* Technology

Chairman: E. MASSEY

8:20 – 8:40	SSPT 41	**Comparison of the *in vitro* exposure methods for cytotoxicity assessment of cigarette mainstream smoke.** SHIN Han-Jae; SOHN Hyung-Ok; PARK Chul-Hoon; LEE Hyeong-Seok; MIN Young-Keun; HYUN Hak-Chul *KT&G Central Research Institute, 302 Shinseong-Dong, Yuseong-Gu, Daejeon, Rep. of Korea*
8:40 – 9:00	SSPT 42	**Mutagenicity of cigarette smoke vapour phase in the AMES test.** WIECZOREK R.; RÖPER W.; KAHL E. *Imperial Tobacco Group, Reemtsma Cigarettenfabriken GmbH, Corporate Responsibility, 22761 Hamburg, Germany*
9:00 – 9:20	SSPT 43	**Detection of cytotoxicity in the water-insoluble fraction of cigarette smoke gas vapor phase using a whole smoke exposure system.** NISHINO T.; NARA H.; FUKANO Y. *Japan Tobacco Inc., Tobacco Science Research Center, 6-2, Umegaoka, Aoba-ku, Yokohama, Kanagawa 227-8512, Japan*
9:20 – 9:40	SSPT 44	**The effects of nitric oxide in the *in vitro* toxicity of cigarette smoke condensate.** REID J.R.; LEVERETTE R.D.; BENNETT M.B.; MISRA M.; HAMM J.T. *Lorillard Tobacco Company, A.W. Spears Research Center, Greensboro, NC 27405, USA*
9:40 – 9:50	Report	**Task Force *In Vitro* Toxicity Testing of Tobacco Smoke** MASSEY E. *British American Tobacco, Group R&D Centre, Regents Park Road, Southampton SO15 8TL, UK*
10:00 – 10:20		COFFEE

THURSDAY 4 OCTOBER 2007

SESSION 12: **Biomarkers / Smoking Behaviour**

Chairman: W. RÖPER

10:20 – 10:40 SSPT 45 **Gene expression analysis of human lung cells treated with cigarette smoke.**

ACHARD S.; VERRON T.; SEVESTRE O.; DUMERY B.

Altadis - Research Centre, 4 rue André Dessaux, 45404 Fleury-les-Aubrais, France

10:40 – 11:00 SSPT 46 **Results from CORESTA yield-in-use ring trial.**

NELSON P.R.(1); BURTON S.(2)

1. *R.J. Reynolds Tobacco Co., R&D, P.O. Box 1487, Winston-Salem, NC 27102-1487, USA*
2. *Arista Laboratories, 1941 Reymet Rd., Richmond, VA 23237-3723, USA*

11:00 – 11:20 SSPT 47 **A study design to investigate the influence of ISO tar yield and tar band switching on cigarette smoke dose as determined by filter analysis and biomarkers of exposure.**

SHEPPERD C.J.; McEWAN M.

British American Tobacco, Group R&D Centre, Regents Park Road, Southampton SO15 8TL, UK

11:20 – 11:30 Report **Task Force Nicotine Uptake**

SCHERER G.

ABF Analytisch-Biologisches Forschungslabor GmbH, Munich, Germany

11:30 – 11:50 SSPT 48 **Biomarkers for the tobacco smoke-related exposure to acrylonitrile.**

SCHERER G.; URBAN M.; HAGEDORN H.W.

ABF Analytisch-Biologisches Forschungslabor GmbH, Munich, Germany

11:50 – 12:10 SSPT 49 **Performing clinical trials to good clinical practice for biomarker assessments to good laboratory practice.**

SHAW I.W.

Covance Laboratories Ltd., Otley Road, Harrogate HG3 1PY, UK

2007 CORESTA JOINT STUDY GROUPS MEETING
SMOKE SCIENCE & PRODUCT TECHNOLOGY

LIST OF ABSTRACTS

IG 01

Trends in crop protection agent residue levels by tobacco types over time: an evaluation of the VdC database.

EBERHARDT H.-J.(1); CZECHOWICZ M.(2)

1. *Verband der Cigarettenindustrie, Neustädtische Kirchstr. 8, D-10117 Berlin, Germany.*
2. *AMC Consulting, ul. Marcinkowskiego 7/5, PL-48-300 Nysa, Poland.*

The VdC database for collecting information on crop protection agent residues on raw tobaccos has grown considerably in size over time. The most recent version (No. 4) contains over a million entries. The database is regularly and intensively used by the participating companies and the Agro-chemical Advisory Committee (ACAC) of CORESTA, both as a source of information on the present state of residues in certain tobacco-growing countries and as a basis for evaluating the use of crop protection agents on tobacco according to the rules of GAP (Good Agricultural Practices). The amount of data available on 333 different pesticides from 88 countries of origin form a sound basis for drawing up an overview of pesticide use worldwide according to the GAP standards. Different parameters can be used and combined to evaluate the data, such as the crop protection agent itself, or origins, tobacco types or crop years.

The more than one million data contained in the most recent version of the VdC database cover a total of 7 tobacco types such as Virginia flue-cured (54.2%), Burley (23.8%), dark air-cured (3.9%), Kentucky fire-cured (1,5%), Oriental / Semi-Oriental (15,5%), sun-cured (0.9%) and Maryland (0.2%). The documentation of all pesticide residues measured over time and over a sequence of crop years within the different tobacco types shows the continuous decrease in residue-containing samples starting in the nineties up to 2005.

An exception is sun-cured tobacco, which reveals only minor variations due to the relatively short period of residue data collection (2001-2005). Approximately 80% of the entire tobacco production worldwide can be attributed to 11 tobacco-growing countries. These are, among others, China, USA, Brazil, India, Argentina and, in the EU, Italy and Greece. Taking into account the most important tobacco-growing countries and the tobacco types produced there, a survey of the residue situation in these countries over the period crop years under review is drawn up on the basis of the data available. Factors possibly involved in the country- and/or tobacco-specific developments following the use of the crop protection agents will be discussed.

IG 02

An effective product evaluation strategy for PREP cigarettes.

DOOLITTLE D.J.; COOK C.J.

R.J. Reynolds Tobacco Company, R&D, Winston-Salem, NC, USA.

Cigarette smoking has been shown to substantially increase the risk of a number of diseases; including lung cancer, pulmonary disease and heart disease. There is agreement among many stakeholders that the development of potentially reduced exposure cigarette products (PREPs) may offer the opportunity to reduce the risk of disease for smokers. The appropriate testing strategy for evaluating PREPs for potential to reduce the risk of smoking-related diseases is an ongoing subject of discussion within the scientific community. R.J. Reynolds Tobacco Company (RJRT) has developed an effective product evaluation strategy for PREPs that includes both stewardship and reduced exposure components. Product stewardship is a tiered approach focused on ensuring that the proposed product modifications do not increase the inherent biological activity of smoke from the modified cigarette. Stewardship studies may include smoke chemistry, *in vitro* toxicology and animal toxicology studies. Evaluation of reduced exposure may include data from multiple types of studies, including yield data under multiple machine regimens, yield-in-use data, and biomarker data from smokers. In conformance with the IOM guidelines it is critically important to demonstrate the biological plausibility of a PREP. Biological plausibility requires developing a sufficiently compelling argument based on scientific data to support the conclusion that the demonstrated reduction in exposure would be anticipated to result in a measurable reduction in morbidity and/or mortality in subsequent clinical or epidemiology studies. Approaches that demonstrate biological plausibility include biological and chemical evidence from preclinical toxicology studies, biomarkers of exposure and effect in smokers, and quantitative risk assessment. Finally, the scientific evidence developed in support of a potential PREP should be reviewed and the PREP conclusion verified by an external scientific body.

SSPT 01

Current profile of the tobacco industry in Korea.

RHEE Moonsoo; LEE Dong-Wook

KT&G Central Research Institute, 302 Shinseong-Dong, Yuseong-Gu, Daejeon, Rep. of Korea.

Korea is a unitary nation located in Far East Asia. The total population of the Republic of Korea is about 48 million. Based on data from 2006, total adult smoking prevalence has remained relatively constant at about 27% (male rate is 48% and female rate is 6.4%, respectively) since 2005 despite a decrease by 10% point from 2000 (37%).

Cigarettes, white stick ones, are dominant in tobacco consumption in Korea, and the market is dominated by sales of filter cigarettes. Total cigarette consumption in 2006 was about 87.6 billions units of which 72% were KT&G's products. Korea produces Burley and flue-cured leaf tobacco, and the former is grown in the western part of Korean peninsular and the latter in the eastern regions. The production of leaf tobacco was 22,000 tons in 2006, and ha decreased dramatically (47,500 tons in 2002).

Korea is one of the most intensive countries in anti-smoking campaign. The WHO FCTC was ratified and took effect in 2005. All tobacco products sold in Korea require the labeling of tar and nicotine yields on the pack together with a health warning. Smoking is restricted in most public places including large buildings, hospitals and trains and traffic facilities, and the restriction areas are steadily being expanded. There is no regulation on ingredients. In this paper, the current tobacco industry profile in Korea will be presented.

SSPT 02

Market survey: study on the cigarette designs used in Europe.

LE MOIGNE C.(1); RAVERDY-LAMBERT D.(1); TARDIF N.(1); LOUREAU J.M. (2); LE BOURVELLEC G.(2)

1. *LTR Industries, Usine Le Mans, 72702 Allonnes Cedex, France.*
2. *Papeteries de Mauduit, 29393 Quimperlé Cedex, France.*

Two market surveys were done, in 2000 and in 2005, which consisted in analyzing the major brands sold in 15 Western European countries. The principle was to analyze for each selected country a number of brands representing 60 to 70% of the domestic consumption. About 70% of the total Western Europe consumption was analyzed in the 2000 study and included 188 brands, 63% of the Western Europe consumption was covered in the 2005 study.

The general design and the smoke yields for each brand have been analyzed: the main characteristics determined on cigarettes were cigarette dimensions, standard and encapsulated pressure drops, tobacco type and density, hardness, filter type, filter and paper ventilations, permeability of the cigarette paper, tipping paper and porous plug wrap. The smouldering of the cigarettes have been determined as well as the smoke yields.

Because of the change in regulation between 2000 and 2005, from 12 mg maximum tar to the existing 10 mg tar/1 mg nicotine/10 mg CO maxima, comparative analysis of the two studies allowed us to follow the evolution of the cigarette designs to cope with this regulation. The major evolutions in cigarette design to adapt to the 10/1/10 regulation will be presented and compared with the theoretical tools to reduce tar and CO/tar.

SSPT 03

A prediction model for flow parameters and smoke yields of cigarettes smoked under arbitrary smoking regimes.

EITZINGER B.

Delfortgroup, Dr.-Franz-Feurstein GmbH, Fabrikstrasse 20, A-4050 Traun, Austria.

The effect of alternative smoking regimes on smoke yields has, in the past, been thoroughly investigated by experiments. Furthermore, current smoking machines are able to reproduce arbitrary smoking regimes, for example, human smoking profiles. Mathematical prediction models, however, have not yet achieved this level of flexibility, as they are almost always limited to specific smoking regimes.

In this study a mathematical model is presented which predicts flow parameters such as filter ventilation, rod ventilation and draw resistance as well as 'tar', nicotine and carbon monoxide for arbitrary smoking regimes. In contrast to other models the smoking regime is not fixed, but can be freely defined. It can, for example, be set equal to a measured human smoking profile. Furthermore the model offers a puff-by-puff resolved prediction of the above quantities.

The model requires the cigarette construction, the desired smoking regime and the ISO smoke yields as input. First, a physical flow model is used to predict the flow velocity and pressure inside the cigarette during each puff. These results are then used to predict the smoke yields by taking dilution, filtration in the tobacco rod, diffusion through the cigarette paper and filter retention into consideration. The model is evaluated twice, once for the ISO smoking regime and once for the alternative smoking regime. A final comparison of predicted ISO smoke yields with the input data allows to improve the prediction for the alternative regime.

Despite its complexity the model can be evaluated within a few seconds on a standard desktop computer, which offers an advantage over advanced methods such as computational fluid dynamics. While a comparison of the predictions with experimental data shows a good agreement, further improvements are still possible, depending on the availability of reliable experimental data.

SSPT 04

Influence of ionic additives on the pyrolysis behaviour of cigarette paper.

STADLMANN K.(1); EBERHERR W.(1); KLAMPFL C.W.(1); KÖLL B.(2); ZEMANN A.(2)

1. *Institute of Analytical Chemistry, Johannes-Kepler-University Linz, Altenbergerstrasse 69, A-4040 Linz, Austria.*
2. *Papierfabrik Wattens, Member of Delfortgroup, Ludwig-Lassl-Strasse 15, A 6112 Wattens, Austria.*

Pyrolysis of cigarette paper causes a cascade of reaction products that can be characterized using GC/MS. Main pyrolysis products include hydroxyl and carbonyl compounds, furane and pyrane derivatives, phenols, and anhydro sugars. The composition of the pyrolyzate, however, can be manipulated by adding metal salt ions and salts of organic acids, respectively. In particular metal ions cause a substantial change not only in the distribution of the various pyrolysis product classes, but of the total amount of pyrolysis products. The effects increase with the valence and concentration of the metal salts (alkaline metal salts > earth alkaline metal salts) respectively. The influence when various organic acids are used

is less significant compared to metal ions, however, the pyrolysis pattern again depends on the valence of the organic acids (acetate/lactate vs malate/malonate/succinate vs citrate). The pyrolysis experiments were carried out using a pyrolyzer and a GC/MS instrument. Multivariate data analysis was used in order to interpret the pyrolysis data.

SSPT 05

Paper tools to reduce formaldehyde and other carbonyl compounds in cigarette mainstream smoke.

LOUREAU J.M.; JOYEUX T.; LE BEC L.; LE BOURVELLEC G.; LE MOIGNE C.

Papeteries de Mauduit, 29393 Quimperlé Cedex, France.

Carbonyl compounds and in particular formaldehyde are major oxidation by-products of combustion processes of tobacco during smoking. These compounds are generated in both mainstream and sidestream smoke. Several studies showed that the amount of formaldehyde formed during a puff is dependent on the heating rate, the amount of tobacco and char consumed, the temperature of the tobacco prior to the puff, the tobacco moisture, etc. Other studies focused on precursors in tobacco like carbohydrates : cellulose is one of the major precursors of formaldehyde.

Filter ventilation and cigarette paper contribution to formaldehyde and other carbonyl compounds yields in cigarette smoke was studied.

After measuring the large contribution of the lighting and first puff to total formaldehyde yield of the cigarette, the impact of filter ventilation and cigarette paper characteristics will be detailed: natural air permeability, basis weight, filler content, fibre weight, filler weight, combustion salt content, paper ventilation (uniformly distributed added air permeability and discrete added air permeability).

Finally, paper tools will be proposed to reduce formaldehyde and other carbonyl compounds yields in cigarette smoke.

SSPT 06

Systematic studies on cigarette paper: simplex lattice experimental designs applied to cigarette paper components and the effects on paper characteristics and sidestream yields.

CASE P.D.; KIMPTON H.; COBURN S.; COTTE V.

British American Tobacco, Group R&D Centre, Regents Park Road, Southampton SO15 8TL, UK.

Sidestream smoke is an area of interest for cigarette designers and for regulatory bodies in various parts of the world. The role of changing cigarette paper characteristics on sidestream yields is therefore of interest to the tobacco industry.

The objective of this exercise was to undertake a systematic experimental design on cigarette paper, more specifically cigarette paper was considered as a mixture and a four component simplex lattice design was utilised. The study involved examining the effect of varying the levels of cellulose fibre, calcium carbonate, magnesium oxide, and tri-potassium citrate. The levels of the variables chosen were in the range that satisfactory samples could

be physically manufactured. All the samples incorporated an approximately constant level of natural paper permeability.

Machine made cigarettes were produced with these papers utilising a constant cigarette construction in terms of tobacco column density, blend type, and filter format. Sidestream smoking was undertaken and analyses for NFDPM, nicotine, carbon monoxide, carbon dioxide and puff number were undertaken.

Data analysis, on both the resulting paper properties and sidestream yields was undertaken. The resulting effects of variation in the paper parameters illustrated how mechanical properties such as tensile strength are increased by increasing the quantity of fibre and decreased by varying the quantity of filler and where interactions between these variables become significant. The study also illustrated that it was possible to predict the sidestream yields from the papers in question anywhere within the experimental space outlined by the defined variables.

Comment will also be made as to the applicability of the approaches used, and the limitations on the design and interpretations will be explained.

SSPT 07

Influence of the origin of the cigarette paper cellulose on the smoke chemical composition and "*in vitro*" toxicity tests on mainstream cigarette smoke.

TROUDE V.(1); ACHARD S.(1); DESTRUHAUT S.(1); HAOND C.(1); SARABIA J.(2)

1. *Altadis - Research Centre, 4 rue André Dessaux, 45404 Fleury-les-Aubrais, France.*
2. *Altadis, Eloy Gonzalo 10, 28010 Madrid, Spain.*

Nowadays, cigarette paper is made out of cellulose coming from different sources. Wood cellulose is currently very often used, but cellulose coming from different textile plants like hemp, flax, etc. were used primarily in the past and are still present in commercial cigarette papers. This study was undertaken to determine if the origin of the cigarette paper cellulose has an impact on the chemical composition of mainstream smoke and on the results of classical *in vitro* toxicity tests performed on smoke.

Two different cigarette models, including different blends and designs were used. In both cases, cigarettes were produced with cigarette papers made out of either pure wood cellulose, or pure plant cellulose or a mixture of wood and plant cellulose. All Hoffmann analytes with the exception of trace metals were measured in the smoke of these cigarettes. The B[a]P determination was completed with qualifications of others PAHs: Pyrene and Dibenzopyrenes.

The three *in vitro* toxicity tests recommended by CORESTA were also performed namely: Ames and micronucleus assay on TPM; neutral red uptake test on both gas and TPM phases. The cell-free "GSH depletion" test was also used to assess the toxicity of the gaseous phase.

After statistical treatment, no significant differences were found between terms of comparison for all Hoffmann analytes and with the *in vitro* tests. The origin of the cigarette paper cellulose was not demonstrated to have an influence on the properties of mainstream cigarette smoke.

SSPT 08

Determination of carbamates and imidacloprid residues in tobacco by LC-MS-MS.

LIU Huimin; XIE Fuwei; WANG Sheng; GONG Wei

Zhengzhou Tobacco Research Institute of CNTC, Zhengzhou, Henan, China.

A method for the simultaneous determination of 11 carbamates and imidacloprid residues in tobacco by liquid chromatography-mass/mass spectrometry was developed. The pesticides were extracted from ground tobacco by a mixture solvent of acetone/water (80/20, V/V) for one hour. The extract was diluted with water and partitioned into dichloromethane. The organic phase was evaporated to dryness in a water-bath, and then the residue was dissolved in dichloromethane. After cleaning up by NH_2-SPE column, the elute was analyzed by LC/MS/MS in the multiple reaction monitoring mode, and with D7-carbaryl as the internal standard. The recovery studies were performed at 0.05, 0.2, 0.5 μg/g fortification levels for each pesticide with the recoveries at ranges of 69.2% to 121.0%. The limit of detection for 12 pesticides range from 0.16 to 4.0 μg/kg with 1.0%~12.0% of relative standard deviation in the overall test procedure. The impacts of tobacco type on matrix effects were discussed.

SSPT 09

The determination of multiple agrochemical residues in tobacco by gas chromatography-triple quadrupole mass spectrometry.

LEE Jeong-Min; JANG Gi-Chul; HWANG Keon-Joong

KT&G Central Research Institute, 302 Shinseong-Dong, Yuseong-Gu, Daejeon, Rep. of Korea.

Traditionally agrochemical residues in tobacco have been analyzed with multi-residue methods by using gas chromatography with either selective detectors (ECD, NPD, FPD) or mass spectrometry. Because many modern agrochemicals are either not amenable to GC or not detectable at sufficiently low levels, more rapid and sensitive analytical methods are needed to quantify the residues. This study was carried out for two main purposes: to give a more rapid and accurate sample preparation, and to use the high selectivity and sensitivity of analyte detection by triple quadrupole mass spectrometry (MS/MS). We compared the three sample preparation methods of agrochemical residues in tobacco such as the conventional liquid-liquid extraction (LLE), pressurized liquid extraction (PLE) and QuEChERS (quick, easy, cheap, effective, rugged and safe) methods. These methods were validated for listed agrochemicals in the CORESTA ACAC guide that were amenable to GC-MS/MS determination. LLE with acetonitrile, and PLE with acetone were followed by solid phase extraction (SPE), the SPE fractions were analyzed by GC-MS/MS in CI and EI mode. In the QuEChERS method, the effects of adsorbent and analyte dependent matrix for the tobacco types were investigated. MS/MS acquisition provided higher specificity and selectivity for agrochemicals and lower limit of detection and quantification than selective detectors and MS. The QuEChERS method did not need a complex clean-up procedure and can be used for the rapid and sensitive analysis of agrochemicals and as an alternative to LLE and PLE methods.

SSPT 10

The content analysis of Cr, Cd, Hg and other inorganic elements in tobacco.

HU Qingyuan(1,3); LI Li(2); CHEN Zaigen(1)

1. *China National Tobacco Quality Supervision & Test Center, Zhengzhou 450001, China.*
2. *Technical Research and Development Center, China Tobacco Chuanyu Industrial Corporation, Chengdu 610066, China.*
3. *Anhui Institute of Optics and Mechanics, Chinese Academy of Sciences, Hefei 230031, China.*

A rapid method of microwave assisted digestion-inductively coupled plasma mass spectrometry was established to simultaneously determine 24 inorganic elements in tobacco leaves, including Na, Mg, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Sr, Mo, Ag, Cd, Sn, Cs, Ba, Hg, Tl, Pb and Th. The relative standard deviations (RSD) of test procedure for analyzed elements are from 1.28 to 9.18%. The recoveries and limits of detection (LOD) are in the range of 85.16~114.7% and 0.3979 ng/L~1.725 μg/L, respectively. Applying this method, 85 kinds of tobacco samples from different regions in China were measured. It was shown that the levels of the 24 elements in the tobacco leaf vary more significantly according to type and region.

SSPT 11

Investigation of the mechanical extraction properties of the raw materials of reconstituted tobacco sheet.

SUNG Yong-Joo; HAN Young-Lim; BAEK Shin; KIM Kun-Soo; RHEE Moonsoo

KT&G Central Research Institute, 302 Shinseong-Dong, Yuseong-Gu, Daejeon, Rep. of Korea.

The various wastes produced by the tobacco industry such as stems, scraps, winnowers, dust, etc., have been transformed into a valuable material, called a reconstituted tobacco sheet (RECON). Especially, the stem is one of the most important components of the RECON. More than 50 w.t.% of the RECON is composed of the tobacco stem.

In this work, the solid/liquid extraction of the stem was evaluated. The mechanical extraction of the stem has some unique features, such as that the stems are the compressible materials, the extract is originated not only from the outside of the stems but also from the inside of the stems, and the actual mechanical pressing is performed with a constant speed, not a constant pressure, by using a screw press. In order to evaluate the mechanical extraction properties of the stem more practically, the novel expression analyzer which could simulate the mechanical pressing of the screw press in a RECON mill was developed.

The results showed the higher temperature of the stock during the mechanical extraction process could decrease the efficiency of the extraction process by increasing the compressibility of the stem, which could lead to more clogging in the press hole and greater reduction in the pathway between the stem fibres. The effects of the pre-treatment process, such as the pulping process, on the mechanical extraction process were also evaluated.

SSPT 12

The analytical method development of measuring ammonia gas in the headspace of Swedish style snus tins.

VELLA J.; STEWART-JONES D.

British American Tobacco, Group R&D Centre, Regents Park Road, Southampton SO15 8TL, UK.

A method has been developed to accurately quantify the level of ammonia in the headspace of a tin of snus. Traditional Swedish style snus can have a strong aroma of ammonia, while this can be detected by sensory methods it is useful to be quantified and rate snus products in order of ammonia content. When a tin of snus is opened the headspace within the tin is immediately diluted with the atmospheric air, to measure this would be inaccurate, therefore a method was developed to establish ammonia exposure at the instant the tin is opened. A method had to be developed where the ammonia was trapped efficiently and it was possible to extract and analyse the resultant ammonia. We developed a method where the unopened tin was sealed inside a Tedlar bag, a known volume of nitrogen was added and the tin then opened inside the bag. The tin was allowed to infuse within the bag for 3 hours which allowed for equilibrium to be achieved between the snus and the gas inside the bag. The resultant gas mixture was drawn through a sorbent tube. The tube contents were analysed by ion chromatography with conductivity detection. With this method it is possible to measure samples equivalent to 0.1 µg/mL of ammonium ions up to an ammonium concentration of 5 µg/mL.

This method delivers a sufficient throughput in order to be a routine method and we have been able to differentiate between different snus samples.

SSPT 13

Fast on-line tobacco smoke analysis with Photo Ionisation - Time-of-Flight-Mass Spectrometry - Part 1: Scientific background and basic principles.

ZIMMERMANN R.(1,2,3); MÜHLBERGER F.(2); MITSCHKE S.(1,2); STREIBEL T.(1,2); ROSE N.(4)

1. *Analytical Chemistry, Institute of Physics, University of Augsburg, D-86159 Augsburg, Germany.*
2. *Institute of Ecological Chemistry, GSF-National Research Centre for Environment and Health GmbH, D-85764 Neuherberg, Germany.*
3. *BIfA-Bavarian Institute of Applied and Environmental Research and Technology GmbH, Environmental Chemistry, D-86167 Augsburg, Germany.*
4. *Borgwaldt KC GmbH, D-22525 Hamburg, Germany.*

Mass spectrometry (MS) with soft ionisation (*i.e.* non fragmenting ionisation) allows a direct on-line monitoring of organic compounds in complex gaseous matrices such as tobacco smoke. Photo ionisation (PI) methods are particularly well suited for this purpose as they are not sensitive to matrix effects. In principle multi- and single photon processes (REMPI and SPI) can be utilized for the soft ionisation of organic species. The respective selectivity and ionisation efficiency depends on the wavelength, spectral bandwidth and intensity of the UV/VUV-light source (i.e. laser technologies or electron pumped excimer lamps - EBEL) used for PI. The sensitivity and particularly the achievable measurement speed are determined by the used mass analyzing technique (Quadrupole -, Time-of-Flight - or Ion trap - mass analyzer).

In this contribution the advantages and limitations of different light source - mass analyzer combinations for on-line smoke analysis are analyzed. Furthermore benefits and disadvantages of alternative soft ionisation technologies such as chemical ionisation (CI) or field ionisation (FI) for smoke science applications are discussed. Finally the technology concept chosen for a commercial fast PI-MS smoke analyzer is introduced.

SSPT 14

Fast on-line tobacco smoke analysis with Photo Ionisation - Time-of-Flight-Mass Spectrometry - Part 2: Machinery.

ROSE N.(1); MITSCHKE S.(2,3); ZIMMERMANN R.(2,3,4)

1. *Borgwaldt KC GmbH, D-22525 Hamburg, Germany.*
2. *Analytical Chemistry, Institute of Physics, University of Augsburg, D-86159 Augsburg, Germany.*
3. *Institute of Ecological Chemistry, GSF-National Research Center for Environment and Health GmbH, D-85764 Neuherberg, Germany.*
4. *BIfA-Bavarian Institute of Applied and Environmental Research and Technology GmbH, Environmental Chemistry, D-86167 Augsburg, Germany.*

The chemical properties of tobacco smoke change very rapidly during the smoking process. A real time analysis is required to better understand the chemistry of smoking. Puff-resolved analysis of tobacco smoke can be done by conventional off-line analysis methods, but can be difficult and time-consuming. For fast on-line, real-time measurements of the tobacco smoking process, time-of-flight mass spectrometry (TOFMS) is the optimal method, as only TOFMS can record simultaneously a range of masses at the required very high measurement rates and precision. The theoretical background of the functionality is described in Part 1: "Scientific background and basic principles".

Based on the theoretical background of the functionality measurement method given in Part 1, this presentation gives a review of the components used in the collection of measurements. It also describes the potential issues of coupling an analyser directly in-line with the mainstream smoke path as well as possible solutions. Such are specific solution, measurement point and hysteresis effects.

But also the influence of the specific solutions to the measurement results are investigated and discussed.

In conclusion a practical configuration will be presented to provide in-line measurement of mainstream smoke.

First results with this instrument and expectations for the future will be presented in Part 3 of this presentation: "First measurements with a Smoke Analyser Prototype system".

SSPT 15

Fast on-line tobacco smoke analysis with Photo Ionisation - Time-of-Flight-Mass Spectrometry - Part 3: First measurements with a Smoke Analyzer Prototype system.

MITSCHKE S.(1,2); ROSE N.(3); ZIMMERMANN R.(1,2,4)

1. *Analytical Chemistry, Institute of Physics, University of Augsburg, D-86159 Augsburg, Germany.*
2. *Institute of Ecological Chemistry, GSF-National Research Center for Environment and Health GmbH, D-85764 Neuherberg, Germany.*
3. *Borgwaldt KC GmbH, D-22525 Hamburg, Germany.*
4. *BIfA-Bavarian Institute of Applied and Environmental Research and Technology GmbH, Environmental Chemistry, D-86167 Augsburg, Germany.*

In the past few years several studies were undertaken investigating mainstream and sidestream smoke with modern on-line mass spectrometric methods with soft ionisation methods, such as single-photon ionisation (SPI) and resonant multi photon ionisation (REMPI) time-of-flight mass spectrometry (TOFMS). These modern techniques have proven to be powerful analytical methods providing a series of information, *e.g.* a simultaneous measurement of many different compounds and the investigation of their dynamics during the smoking cycle. In this matter exceptionally high amounts of various unsaturated hydrocarbons were discovered during the first puff, as well as changes in the chemical composition of the different puffs. However, the past analysing-systems were mostly custom-made, laser based systems, which required intense training and therefore were not suited for large-scale routine analysis. First measurements done with a user-friendly prototype are presented in this paper. The system was designed in co-operation with Borgwaldt KC GmbH, Hamburg, Germany and is equipped with an ultra compact TOFMS system (TOFWERK AG, Thun, Switzerland) and a novel vacuum-ultraviolet (VUV)-lamp system (Coherent, Dieburg, Germany) as presented in Part 2: "Machinery". The presented results show a characterisation of the instrumental setup and demonstrate its capabilities for routine puff resolved on-line analysis of smoke, possible future analytical applications as well as research and development tasks.

SSPT 16

Real-time measurement of flavour release and persistence from cigarette smoke using APCI-MS.

COTTE V.(1); PRASAD S.K.(1); WAN P.H.W.(1); LINFORTH R.S.T.(2); TAYLOR A.J.(2)

1. *British American Tobacco, Group R&D Centre, Regents Park Road, Southampton SO15 8TL, UK.*
2. *Division of Food Sciences, University of Nottingham, Sutton Bonington Campus, Loughborough, UK.*

The real-time measurement of cigarette smoke was carried out using APCI-MS to understand the effects of tobacco types and smoker interactions on the flavour release and persistence of cigarette smoke in the nose. Five tobacco types and one blend were made into cigarettes which were smoked by up to four volunteers following a standard smoking protocol and also by using a single port smoking machine (ISO smoking method). During smoking, five ions were monitored in the nosespace using APCI-MS in SIM mode.

The different temporal release (puff by puff) profiles obtained between machine and human smoking from the six cigarettes used in this study can be explained according to the properties of tobacco types (composition, filling values, burn rate), smoking behaviour (puff volume, puff duration, puff interval), breathing patterns (depth, flow, duration, breath-hold), physiology aspects and the properties of smoke aerosol and its behaviour during respiration.

The short-term persistence (or aftertaste) of cigarette smoke was also monitored in between each puff separated by a 1 minute interval. The smoke aerosol monitored in the nosespace during smoking showed different decay rates on a puff by puff and compounds (ions) basis. The trend showed that the decay rates increased in function of the puff number.

It is possible to monitor flavour release and persistence from complex matrices such as cigarette smoke in real-time using APCI-MS. This method may be useful to develop cigarettes generating optimum flavour release in the nose during smoking and flavour persistence post-smoking. The limits of the method reside in the use of SIM mode allowing the measurement of few ions at a time in order to maintain a high sensitivity. Many parameters influence the flavour release and persistence of tobacco smoke in the nose. Therefore, in order to remove the release variations from different smoking behaviours and breathing patterns, the use of a Smoking Behaviour Analyser and partial body plethysmograph attached to a spirometer is recommended in future studies using APCI-MS.

SSPT 17

Multiple component analysis of mainstream cigarette smoke using Terahertz spectroscopy, comparison with standard chemical analytical methods.

CUISSET A.(1); BIGOURD D.(1); BOCQUET R.(1); HINDLE F.(1); MOURET G.(1); CAZIER F.(2); DEWAELE D.(2); NOUALI H.(2)

1. *Laboratoire de Physico-Chimie de l'Atmosphère CNRS UMR 8101, Bâtiment MREI2, Université du Littoral Côte d'Opale, 189A Ave. Maurice Schumann, 59140 Dunkerque, France.*
2. *Centre Commun de Mesures, Bâtiment MREI1, Université du Littoral Côte d'Opale, 145 Ave. Maurice Schumann, 59140 Dunkerque, France.*

The Terahertz (THz) frequency domain is a transition region lying between the millimetre wave and the infrared portions of the electromagnetic spectrum. The submillimetre wavelengths and the far-infrared region extend from 1 mm to 30 µm, corresponding to frequencies from 300 GHz to 10 THz. This frequency range was considered as a "spectral gap" due to the technological difficulties in producing a tunable source with sufficient power. Therefore the pioneer applications of the THz radiation were dedicated to the linear absorption spectroscopy. Terahertz radiation allows the pure rotational transitions of small polar compounds or the low-frequency vibrational modes of larger molecular systems to be probed. Linear absorption THz spectroscopy is now currently being pursued in various scientific domains such as astrophysics, atmospheric chemistry and biochemistry.

The THz radiation offers the possibility to obtain information in environments contaminated by fog, rain, dust or smoke, which are opaque at visible wavelengths. In order to study the potential of the THz radiation in order to probe realistic media, THz spectroscopy has been used for the first time for the detection and the quantification of small polar species in mainstream cigarette smoke. Two THz sources, one pulsed and one continuous, have been used alongside. The pulsed THz radiation allowed to obtain a rapid and simultaneous detection of several pure rotational transitions of hydrogen cyanide (HCN) and carbon monoxide (CO) in realistic conditions of pressure and temperature. The narrow spectral purity of the continuous wave terahertz source produced by photomixing permitted the concentrations of these molecules to be measured at pressures of tens of hPa. Moreover, at lower pressure, traces of formaldehyde (H_2CO) have been unambiguously identified at frequencies above 1 THz. A comparison with standard chemical analytical methods has been completed for each molecule highlighting the advantages of the direct measurement by THz spectroscopy.

SSPT 18

Puff profile simulator for tobacco smoke particle diameter and mass measurement.

McAUGHEY J.(1); REAVELL K.(2); FROST B.; McGRATH C.(1); DAILLY C.(2)

1. *British American Tobacco, Group R&D Centre, Regents Park Road, Southampton SO15 8TL, UK.*
2. *Cambustion Ltd., J6 The Paddocks, 347 Cherry Hinton Road, Cambridge, CB1 8DH, UK.*

This paper describes a system for cigarette testing measuring fresh TPM (total particulate material) mass, median particle diameter, and particle number concentration, with any desired flow profile. This allows measurements at conditions representative of human smoking or for regulatory pre-defined machine smoking profiles. The data are collected at 10

Hz time resolution with cumulative number and TPM mass measurement in real time on a puff by puff basis.

The system is designed to be used with real-time aerosol instruments such as DMS-type fast electrical mobility spectrometers to provide continuous measurement of the aerosol inhaled from the cigarette during smoking. The flow through the cigarette is metered with an orifice-Δp type flow sensor and controlled to follow a specified profile at 12.5 Hz. To follow highly dynamic puff profiles a feed-forward type controller is used. The complete smoking of a cigarette with a different profile for each puff can be reproduced.

The flow drawn through the cigarette is diluted with filtered air close to the filter holder to halt agglomeration processes. The system operates with a constant total diluted flow to minimise errors in the measurement of total mass emissions from the cigarette. A dilution ratio signal is provided to allow calculation of the undiluted concentrations if desired.

The system has been tested with standard machine profiles and those measured from human smokers. Control of cigarette flows down to approximately 1 cc/s is possible, with a dynamic range of at least 30:1. The typical error in the integrated volume of a puff is around 1%.

Puff by puff were measurements carried out on a series of 1- and 4-mg yielding products using a 35 ml puff of 2 s duration every 60 s, using an ISO puff profile, and normalised to 7 puffs. These data were compared on a puff by puff basis with gravimetric measurements.

Count median diameters (CMD) were measured puff by puff and ranged from 163 - 247 nm, depending on the puff number and yield of each puff. Individual puffs were measured from 0.18 to 1.05 mg TPM and correlated well with the equivalent gravimetric data ($r^2 = 0.88$).

SSPT 19

Simultaneous on-line analysis of gas phase and particulate phase of cigarette mainstream smoke.

ADAM T.(1,2); McAUGHEY J.(3); MITSCHKE S.(1,2); McGRATH C.(3); MOCKER C.(1,2); ZIMMERMANN R.(1,2,4)

1. *Analytical Chemistry, Institute of Physics, University of Augsburg, 86159 Augsburg, Germany.*
2. *Institute of Ecological Chemistry, GSF - National Research Center for Environment and Health, 85764 Neuherberg, Germany.*
3. *British American Tobacco, Group R&D Centre, Regents Park Road, Southampton SO15 8TL, UK.*
4. *BIfA-Bavarian Institute of Applied and Environmental Research and Technology GmbH, Environmental Chemistry, 86167 Augsburg, Germany.*

Tobacco smoke is a complex and dynamic matrix consisting of gaseous and particulate material, in which about 4800 constituents have been identified. The chemical composition and partition between phases of the smoke can change continuously and is strongly influenced by time, temperature, chemistry and dilution of smoke.

We present an experimental set-up consisting of gas phase and particulate phase on-line instrumentation for comprehensive analysis of mainstream tobacco smoke.

Cigarettes comprising Burley, Virginia or Oriental tobacco at 3 filter ventilation levels were smoked, with particle diameter and concentration measured by electrical mobility (Model DMS-500, Cambustion, Cambridge, UK). Chemical composition was characterised on-line by two soft photoionisation techniques; resonance-enhanced multi-photon ionisation (REMPI) and single photon ionisation (SPI) techniques, both coupled to time-of-flight mass spectrometry (TOFMS).

Count median diameter (CMD) averaged over the cigarette varied from 182 - 260 nm and increased with increasing filter ventilation and lower puff flow rates; a consequence of increasing smoke residence time and coagulation within the rod. Puff-by-puff data showed increasing particle concentration and decreasing diameter as the tobacco was consumed and the coagulation period decreased.

Mass spectrometry data show that most smoke constituents feature a continuous increase from the first to the last puff. However, there are some substances, in particular unsaturated hydrocarbons *e.g.* butadiene, isoprene, and propyne, which show a completely different behaviour by having the highest amounts in the first puff. This is likely to be related to the different combustion and pyrolysis conditions when the cigarette is lit.

SSPT 20

Sidestream smoke analysis with Single Photon Ionisation/Time-of-Flight Mass Spectrometry (SPI-TOFMS).

MITSCHKE S.(1,2); ADAM T.(1,2); BAKER R.R.(3); ZIMMERMANN R.(1,2,4)

1. *Analytical Chemistry, Institute of Physics, University of Augsburg, D-86159 Augsburg, Germany.*
2. *Institute of Ecological Chemistry, GSF-National Research Center for Environment and Health GmbH, D-85764 Neuherberg, Germany.*
3. *British American Tobacco, Group R&D Centre, Regents Park Road, Southampton SO15 8TL, UK.*
4. *BIfA-Bavarian Institute of Applied and Environmental Research and Technology GmbH, Environmental Chemistry, D-86167 Augsburg, Germany.*

Recently several studies dealing with online analysis of mainstream tobacco smoke utilising photo ionisation mass spectrometry (PI-MS), namely single photon ionisation (SPI) and resonance-enhanced multi photon ionisation (REMPI) time-of-flight mass spectrometry (TOFMS) have been published revealing the puff resolved dynamics of cigarette smoking. In this matter standard cigarettes, as well as single tobacco grade cigarettes, were investigated. In this study the chemical composition of the gas-phase and particulate phase of sidestream smoke emissions of these cigarette types is characterised using a laser-based SPI-TOFMS instrument. Sidestream smoke is generated from various cigarette types (2R4F research cigarette; Burley, Oriental and Virginia single tobacco type cigarettes) smoked under ISO conditions on a Borgwaldt single port smoking machine and collected using a fishtail chimney device.

Like in mainstream smoke the concentration profiles of various substances in sidestream smoke can be categorised into several groups, either depending on the occurrence of a mainstream puff or uninfluenced by the changes in the burning zone during puffing. In general, substances which are influenced by the puff show a decrease in concentration during the two second puff duration followed by an increase of approximately ten seconds. Changes in the chemical composition of the different emission stages were analysed utilising Principal Component Analysis (PCA) after variable reduction by the calculation of Fisher-Ratios. The same statistical approach was applied to distinguish the emissions of different single type cigarettes. The sidestream emissions occurring directly after a mainstream puff is drawn from the cigarette strongly resemble the composition of mainstream smoke. Additionally, like in mainstream smoke, the chemical composition of the sidestream smoke is strongly affected by the tobacco type.

SSPT 21

Determination of "Hoffmann Analytes" in cigarette mainstream smoke. The CORESTA 2006 collaborative study.

INTORP M.(1); PURKIS S.(2); WHITTAKER M.(3); WRIGHT W.(4)

1. *Imperial Tobacco Group, Albert-Einstein Ring 7, 2761 Hamburg, Germany.*
2. *Imperial Tobacco Limited, P.O. Box 525, Southville, Bristol BS99 1LQ, UK.*
3. *Statistical Consultant to Imperial Tobacco Limited, Bristol, UK.*
4. *Labstat Inc, 262 Manitou Drive, Kitchener, Ontario N2C1L3, Canada.*

Regulatory authorities are currently discussing the measurement of and imposition of ceilings on certain smoke analytes, the so called "Hoffmann" analytes. However, as a pre-requisite, the measurement methods and the tolerances around the measurements firstly need to be established.

In 1999, CORESTA responded to regulatory interest by setting up a Task Force to deal with analytical methodology for measuring Hoffmann analytes under ISO smoking and to work towards the standardisation of methods. This paper describes the output from a 2005-6 collaborative study, set up within the Task Force, to obtain data on most Hoffmann analytes, from reference cigarettes (2R4F and 1R5F), collecting data according to the existing methods used by the nineteen participating laboratories, in order to describe the within and among laboratory variability.

The approach used to determine the current method performance was to see if statistical differentiation could be achieved between 1R5F and 2R4F cigarettes when applying the different methodologies. It was felt that if the methods could not well differentiate these products of differing tar yield then there were significant weaknesses in the method application.

Results indicate that Hoffmann analyte data is generally more variable both within and among laboratories than NFDPM; nicotine and carbon monoxide. Accordingly, tolerances around methods adopted for regulatory purposes will need to be proportionately higher.

Methods on benzo[a]pyrene (BaP) and tobacco-specific nitrosamines (TSNAs), already taken to Recommended Methods through this Task Force, give some of the most reproducible results, showing the value of this process. However, this data strongly suggests that the among-laboratory variability (R) is much higher for BaP and TSNAs than for NFDPM, nicotine and CO. Based on the only two available one point in time studies, the indications are that BaP tolerance values even for the higher yielding 2R4F cigarette should be at least mean ± 35% from the 2003 CORESTA collaborative data and mean ± 45% from the current study. Similarly, for four individual TSNAs, tolerances should be at least in the range mean ± 35 to 55% from the 2005 CORESTA collaborative data and mean ± 26 to 42% from the current study.

The collected data is useful to participating laboratories for internal method validation and accreditation purposes and data comparisons with others allow laboratories to identify strengths and weaknesses in their current methods.

SSPT 22

Determination of nitrogen oxides in cigarette mainstream smoke by ion chromatography.

WU Mingjian; HE Zhihui; LIANG Wenliu

Technical Center of Hunan Tobacco Group, Changsha 410001, China.

A new method for determining nitrogen oxides in mainstream cigarette smoke by ion chromatography was developed. Impingers equipped with a fritted glass bubbler containing 5% triethanolamine solution were used to collect nitrogen dioxide in mainstream smoke (Nitrogen monoxide was firstly oxidized to nitrogen dioxide by chromium trioxide in oxidizing tube). And then NO_2^- and NO_3^- in absorbing solution were directly determined by ion chromatography simultaneously without any pretreatment due to the proper gradient elution procedure. The ions had good linear responses in the range of 0.5 mg/L-10 mg/L. The relative standard deviations were less than 2%. The detection limits were found to be 0.04 mg/L and 0.05 mg/L for NO_2^- and NO_3^- respectively. Recoveries of NO_2^- and NO_3^- were found to be ranged between 97% and 101%. The simple, rapid and accurate method was suitable for the determination of nitrogen oxides in mainstream cigarette smoke. Several brands of cigarettes including Kentucky Reference cigarettes (1R5F and 2R4F) were determined with this method. The results indicated that the NOx yields in mainstream smoke of Virginia cigarette that are most popular in the China market are lower than in the American blended cigarette.

SSPT 23

The effect of blend type on mainstream and sidestream Hoffmann analyte machine yields.

WINTER D.; CASE P.D.; COLEMAN M.; DITTRICH A.; WARREN N.

British American Tobacco, Group R&D Centre, Regents Park Road, Southampton SO15 8TL, UK.

The potential relationships between various tobacco blend components and Hoffmann analyte yields were examined. Three distinct lamina tobacco blend styles of Virginia, Burley (uncased), Oriental and a 1:1 mix of the Virginia/Burley tobaccos were used. A uniform cigarette design of 80 mm Water Gauge filter pressure drop, 35% on-machine laser ventilation and a cigarette paper permeability of 35 CORESTA units was selected.

For each blend style, various blend components *e.g.* total nitrogen, cellulose were measured and mainstream/sidestream Hoffmann analyte yields determined.

The following were observed for both mainstream and sidestream smoke. Correlations indicated possible relationships between blend chemistry components and Hoffmann analyte yields. A statistical multivariate technique called 'clustering' was used to classify similar correlations into groups. This showed that certain blend components might be possible precursors for various Hoffmann analytes and groupings of various Hoffmann analytes may have a common blend precursor. Linear regression analysis identified blend components that could predict certain Hoffmann analyte yields in smoke. These analysis techniques illustrated that Hoffmann analyte yields could be correlated to more than one blend measurement but they did not imply 'cause and effect', in that a specific blend component was definitely a precursor of a particular Hoffmann analyte.

SSPT 24

Study on puff-by-puff deliveries of the volatile and semi-volatile organic acids in TPM of mainstream cigarette smoke.

ZHAO Xiaodong; XIE Fuwei; LU Ximei; LIU Huimin

Zhengzhou Tobacco Research Institute of CNTC, Zhengzhou, Henan, China.

A method for the simultaneous determination of volatile and semi-volatile fatty acids in TPM of mainstream cigarette smoke was developed. The organic acids in TPM of mainstream cigarette smoke were extracted with dichloromethane in an ultrasonic bath, then were derivertised with N,O-Bis(trimethylsilyl)trifluoroacetamide. The derivatives of 20 organic acids were analyzed by selective ion monitoring GC/MS with the recoveries at the range of 74.3% to 101.8%, and R.S.D in overall analysis procedure was less than 9.8% for all organic acids. Applying this method, the puff-by-puff deliveries of the 20 volatile and semi-volatile organic acids in TPM of mainstream cigarette smoke were investigated. Puff-by-puff smoking was performed on a modified smoking machine. The TPM of individual puff of 20 cigarettes was collected on a Cambridge filter according to the ISO method. The yields of 20 organic acids in TPM of the seven puffs were determined quantitatively. The results indicated that there is an ascending trend that is similar to TPM in which the organic acids delivery increases with increasing puff count. However, the puff-by-puff delivery profile of volatile and semi-volatile organic acids is dissimilar.

SSPT 25

Artifact formation during the analysis of tobacco specific nitrosamines in sidestream smoke.

GLAZIER M.; VAN HEEMST J.D.H.

British American Tobacco, Group R&D Centre, Regents Park Road, Southampton SO15 8TL, UK.

As well as in mainstream smoke, tobacco specific nitrosamines (TSNA) are present in high amounts in sidestream smoke. A method was developed to measure and quantify the amounts of these compounds using LC-MS/MS. Sidestream smoke was led through a BAT design fishtail. Initially, a number of different approaches were used to trap the TSNA in the sidestream smoke. All these approaches yielded different results for the amounts of TSNA in mainstream smoke, especially for NNK and NNN. When ascorbic acid was used in various stages of the procedure, much lower TSNA amounts were observed suggesting the involvement of this compound in the suppression of the formation of TSNA after the smoke leaves the cigarette. Although TSNA may still be formed naturally in the sidestream smoke, the way the sidestream smoke is captured during the analysis causes the smoke to be much more concentrated than it would be naturally. Therefore, it is not likely that this formation happens naturally and it can be attributed to being an artifact of the analysis. Hence, to get a true representation of amounts of TSNA in sidestream smoke, this artifact formation should be suppressed.

SSPT 26

Determination of major semi-volatile compounds in mainstream cigarette smoke.

XIE Fuwei; ZHAO Ge; ZHAO Le; WANG Sheng; XIA Qiaoling

Zhengzhou Tobacco Research Institute of CNTC, Zhengzhou, Henan, China.

A new method has been developed for the quantitative analysis of major semi-volatile compounds in both particulate phase and vapour phase of mainstream cigarette smoke. The vapour phase was collected with a XAD-4 cartridge instead of cold solvent trap, and the particulate phase of mainstream smoke was collected on a Cambridge filter. The filter and resin were extracted with methanol containing 0.01% triethylamine, then the extract was analyzed using selective ion monitoring (SIM) GC-MS technique. 20 semi-volatile compounds including pyridine, styrene and substituted pyridines can be identified in particulate and vapour phase with the recoveries at the range of 83% to 116%. The limit of detection for 20 semi-volatile compounds range from 4.2 to 34.8 ng/cigarette. The repeatability of the technique is good, with average 3.9% R.S.D and 6.3% R.S.D in overall analysis procedures for all of the analyzed compounds in particulate phase and vapour phase respectively. 2R4F, 1R5F Kentucky reference cigarette and 11 brands of commercial cigarette were measured.

SSPT 27

Investigation of free radicals in cigarette smoke by Electron Spin Resonance.

GHOSH M.(1); McAUGHEY J.(1); IONITA P.(2)

1. *British American Tobacco, Group R&D Centre, Regents Park Road, Southampton SO15 8TL, UK.*
2. *Institute of Physical Chemistry, 202 Spl. Independentei, Bucharest 060021, Romania.*

Tobacco smoke is a complex carbon-based, dynamic liquid droplet aerosol, suspended in an equally complex organic vapour mixture. The particulate phase contains several exceptionally long lived radical species principally a mixture of quinone, semiquinone and hydroquinone functionalities held in a polymeric matrix. In marked contrast to the tar-phase radicals, the gas-phase of the tobacco smoke contains radicals that are too reactive to be detected directly.

In this work we have identified and quantified particulate and gas-phases free radicals from the combustion of tobacco by Electron Spin Resonance spectroscopy (JEOL-FR30EX). The particulate phase free radicals have been investigated by placing a cellulose acetate filter rod containing the smoke condensate directly into the ESR cavity. Spin trapping method in conjunction with ESR was used to investigate free radicals in the gas phase of cigarette smoke. We then applied the methods to compare radical concentrations in three base tobacco blends (namely Virginia, Burley, Oriental). All the tests were optimised and normalised using the University of Kentucky 2R4F reference cigarette. The radical concentration in particulate phase was found to be ca. 10^{13} - 10^{14} spins per cigarette. The gas phase radical concentration was in the range of 10^{14}-10^{15} spins per cigarette. A linear correlation was observed between the concentrations of gas phase carbon centred radicals and nitric oxide measured for the three base blends.

SSPT 28

Analysis of vapor and particulate phase free radicals in mainstream cigarette smoke.

TAKANAMI Y.; MORIYAMA T.

Japan Tobacco Inc., Tobacco Science Research Center, 6-2, Umegaoka, Aoba-ku, Yokohama, Kanagawa 227-8512, Japan.

Cigarette smoke contains various types of free radicals. Generally, the radicals are categorized into vapor phase radicals, particulate phase radicals and reactive oxygen species, which are analyzed by separate methodologies. In this report, the analytical conditions of the vapor and particulate phase radicals are investigated in detail to find analytical methods with robustness. A 20-port rotary smoking machine is used for smoke collection. For the vapor phase radical analysis, the cigarette smoke passes through a Cambridge filter and is bubbled with a PBN spin-trapping agent in benzene, and then the ESR spectrum of the solution is measured. For the particulate phase radical analysis, the tar collected on the Cambridge filter is extracted with benzene and the ESR spectrum of the solution is measured. The amount of the radicals is determined using calibration curves obtained from the ESR spectra of TEMPO. The method validation including the stability and linearity is examined. To improve the stability of the data, the aging effect of the radicals must be considered by the analytical conditions. To obtain results stable enough for evaluation of the vapor phase radicals, the timing of the analytical steps between smoking and ESR analysis is kept the same for each analysis. The particulate phase radicals are relatively stable and the aging effect is suppressed for a certain duration by cooling the Cambridge filter until the extraction. Good linearity is found between the amount of the vapor phase radicals and the number of cigarettes smoked. The precision of the amount of the particulate radicals is good; however, the linearity of the amount of the radicals and tar concentration is limited, especially for tar extracts with low concentrations. Further studies for obtaining the linearity of the analytical data will be needed, and the analytical results of some cigarette samples will be discussed.

SSPT 29

The scavenger effects of various antioxidants in cigarette filters on the free radicals in mainstream smoke.

PARK Jin-Won; KIM Soo-Ho; KIM Jong-Yeol; KIM Chung-Ryul; RHEE Moonsoo

KT&G Central Research Institute, 302 Shinseong-Dong, Yuseong-Gu, Daejeon, Rep. of Korea.

Cigarette mainstream smoke is a complex mixture of approximately 4,000 chemical constituents. It is well known, for example, that the cigarette smoke can contain high concentrations of free radicals that may bind to DNA and could lead to damage of cells.

This study was conducted to evaluate the effect of antioxidants, used for free radical reduction and added to cigarette filters, on the delivery of free radicals in MS by ESR (electron spin resonance). Also, we analyzed Hoffmann's analytes and *in vitro* mutagenicity and cytotoxicity. The total amount of tar and vapor phase free radicals in MS are decreased to 16~40% and 14~24%, respectively.

The inactivation of tar and vapor phase free radicals by natural antioxidants is more effective than that using synthesized antioxidants. With regard to the correlation between total delivery,

amount of NO and free radicals in MS at the same level of tar, we obtained a poor correlation factor.

Based on the results for the Hoffmann's analytes, filters treated with ascorbic acid gave lower deliveries of PAHs, isoprene and quinoline in MS in comparison to the other antioxidants.

In the t-test of significance on the difference for the mutagenicity and cytotoxicity of various antioxidants, there are no significant differences at the 95% confidence level.

Those results indicated that the antioxidants were useful for reducing free radicals in MS because of the fast reaction between antioxidant and free radical in MS.

SSPT 30

Investigation of the thermal decomposition and pyrolysis behaviour of monomenthyl succinate by non-isothermal TGA and on-line PyGC/MS.

YANG Liu(1); MIAO Mingming(1); WU Yiqin(1,2); XIANG Nengjun

1. *R&D Center of Hongta Tobacco (Group) Co., Ltd., Yuxi 653100, China.*
2. *College of Chemical Science and Engineering, Yunnan University, Kunming 650091, China.*

The thermal decomposition and pyrolysis behaviour of a natural flavorous compound, monomenthyl succinate, were investigated by non-isothermal thermogravimetry (TGA) and on-line pyrolysis gas chromatography/mass spectrometry (PyGC/MS). The kinetic model of monomenthyl succinate in the main reaction zone of thermal decomposition process was established by TGA, and important kinetic parameters were obtained. PyGC/MS was used for the qualitative and semi-quantitative analysis of the pyrolysis products of monomenthyl succinate at 300, 400, 500, 600, 700, 800 and 900°C. 75 pyrolysis products, including menthol, menthene and succinic acid were identified. The results indicated that flavorous and refreshing substances, such as menthol, *p*-menth-3-ene and 3-methyl-6-isopropyl-cyclohexene, were released from monomenthyl succinate under 700°C. However, above this temperature, flavours were not found in the pyrolysis products. Moreover, with the rise of pyrolysis temperature, complicated pyrolysis products appeared, and the content of harmful substances, such as benzene, anthracene and fluoranthene were also increased. According to the relative content and category of the pyrolysis products, the pyrolysis mechanism of monomenthyl succinate was further discussed, which concluded that the pyrolysis of monomenthyl succinate may take place via 5 different approaches. This investigation gave an exemplification for the transfer behaviour of tobacco essence in the cigarette burning process, and provided a reliable theoretical foundation for the perfume reinforcement technology in tobacco products, which contribute to the development of cigarette products with better aroma and taste.

SSPT 31

Filters for ultra-slim cigarettes: flow rate and pressure drop performance.

RASMUSSEN G.T.; WATTS A.S.

Eastman Chemical Company, P.O. Box 511, Kingsport, TN 37662, USA.

Ultra-slim cigarettes have been available for twenty years, and in some regions, the popularity of ultra-slim cigarettes is growing dramatically. Based on a survey of brands from North America, Asia, and Europe, typical filter lengths for ultra-slim cigarettes fall in the range

of 27 to 30 millimetres. Pressure drop targets for these filters are generally comparable to those for filters of conventional circumference. However, with a circumference near 17 millimetres, ultra-slim cigarettes have half the face area of cigarettes made at a conventional circumference. The small circumference of ultra-slims influences their performance significantly, requiring unique filter materials to maintain acceptable product characteristics. Pressure drop measurement protocols and smoke-testing regimes specify volumetric flow rates. By contrast, pressure drop and filtration efficiency depend fundamentally on the velocity of the air or smoke. Reducing the face area of a filter by half at constant volumetric flow rate doubles the linear velocity of the fluid. Without a compensating change in filter material, filter pressure drops, even at constant filter density, would also double. Typically, filter materials with larger fibres are employed to maintain acceptable filter pressure drops. Even with specialized filter materials, small changes in filter weight may cause relatively large changes in filter pressure drop. Ultra-slim filters are also unique in the relative contributions of viscous and inertial flow to filter pressure drop. Measurements over a broad range of flow rates show that the contribution of inertial flow to pressure drop in ultra-slim filters is more than twice that for filters of conventional circumference. An understanding of the factors influencing the performance of ultra-slim filters enables the cigarette manufacturer to make effective design and material selection choices for these products.

SSPT 32

Effects of triacetin on the retention of acetate filters with dispersed additives.

SCHÜTZ E.

Rhodia Acetow GmbH, Engesserstr. 8, D-79108 Freiburg, Germany.

Acetate filters with dispersed active carbon granules are more and more used in cigarettes when a high retention of volatiles is desired. These so called dalmatian filters are constructed with very different Filter Tow items and different types of charcoal granules. Dalmatian filters are used for the whole range of cigarettes from low up to high tar deliveries.

While the retention performance with respect to volatiles is the predominant aim of charcoal in cigarette filters, there is also a strong effect on the removal of particulate matter.

Both the retention of volatiles and the retention of particulate matter by charcoal filters are affected by several factors; these include triacetin, shape and size of the charcoal granules.

In this context the purpose of our paper is to present studies that investigate the effect of the granule shape and of triacetin on the removal of particulate matter and of volatiles by Dalmatian filters. In particular:
- a model for the prediction of particulate matter retention as a function of triacetin content;
- a correlation of the retention performance of volatiles and of particulate matter
are presented.

SSPT 33

The adsorption of various smoke compounds by activated carbon.

TAYLOR M.J.; WALKER J.

Filtrona Technology Centre, Shaftesbury Avenue, Jarrow, Tyne and Wear, NE32 3UP, UK.

The ability of a porous material to adsorb compounds from gas or liquid streams is often related to a number of properties of the material such as surface area, pore size distribution and to some extent the nature and surface functionality of the material. A number of methods are used by carbon suppliers to measure the overall adsorption capacity or activity of carbon by measuring the adsorption of compounds such as carbon tetrachloride, butane, iodine or molasses. Commonly for carbons used in gas applications the carbon tetrachloride or butane activity would be quoted and for carbons meant for liquid applications iodine number of molasses number used. How do such measurements that often rely on adsorption only relate to activity in cigarette smoke? In such a complex mixture as cigarette smoke carbon activity is often measured as the average retention of a range of vapour phase compounds. But how does the chemistry and volatility of compounds in smoke affect the adsorption by carbon?

Measurements of adsorption isotherms for coconut shell carbon have been carried out for a range of smoke compounds to see how these compounds are adsorbed by carbon. Data will be presented on the adsorption of a range of compounds and how the volatility and chemical nature of the compounds affects the levels of adsorption. In particular the adsorption and possible chemisorption of acetic acid by carbon will be discussed.

SSPT 34

The influence of cigarette design on the ageing of carbon filters.

PETERS G.(1); MUELLER C.(1); WALKER J.(2); TAYLOR M.J.(2)

1. *Imperial Tobacco Group, Albert-Einstein-Ring 7, 22761 Hamburg, Germany.*
2. *Filtrona Technology Centre, Shaftsbury Avenue, Jarrow, Tyne and Wear, NE32 3UP, UK.*

Coconut shell based activated carbon is still the most powerful tool for retaining the various constituents from mainstream cigarette smoke. Several authors have reported on the reduction of carbon activity as a function of storage time and conditions. Previous work indicates that the formation of acetic acid by hydrolysis from triacetin plays only a minor role but a higher storage temperature leads to a faster de-activation of those micropores which can adsorb the smaller gas phase components. This is most likely due to the increasing mobility of compounds originating from the tobacco blend and the packaging material.

This study looks at a set of product parameters such as blend style, filter design and tar level of the test cigarettes. Packed samples have been stored at room temperature and at 40° Celsius. To assess the effect of aging on carbon activity a set of 20 vapour phase constituents and some semi-volatile smoke components, *e.g.* phenols, have been measured under ISO smoking conditions over a period of 6 months. Additionally the rate of plasticizer hydrolysis has been measured as a function of time by analysing the acetic acid contents in the filter tips.

The present investigation demonstrates that a set of selected vapour phase compounds is sufficient for monitoring the activity of carbon in cigarette filters over a period of time. This study also confirms the influence of storage conditions on deactivation of active carbon but also demonstrates that the effect of aging can be kept at a tolerable level by choosing an appropriate set of product properties

SSPT 35

Physical properties of carbons prepared from a coconut shell by steam activation and chemical activation and the influence of prepared activated carbon on the delivery of mainstream smoke.

KO Dong-Kyun; SHIN Chang-Ho; LEE Young-Taek; JANG Hang-Hyun; RHEE Moonsoo

KT&G Central Research Institute, 302 Shinseong-Dong, Yuseong-Gu, Daejeon, Rep. of Korea.

Several activated carbons with different specific surface areas were prepared by steam and chemical activation of coconut shell. Products were characterized by BET (N_2) at 77K, and probed to reveal a highly specific surface area of 1580 m^2/g and pore volume that increased with activation conditions. We have analyzed the adsorption efficiency of the vapor phase components in cigarette mainstream smoke in order to evaluate the relationship between the smoke components and the physicochemical properties of activated carbons. As a result of this study, the delivery of mainstream smoke was directly affected by the specific surface area and the pore size of activated carbon.

The activated carbon prepared by steam activation exhibited better adsorption efficiency on the vapor phase components in mainstream smoke compared with activated carbon prepared by $ZnCl_2$, due to the higher micropore area of 66%. The adsorption efficiency of semi-volatiles such as phenolic components in mainstream smoke by the mesoporous activated carbon prepared by zinc chloride is more effective. From these results, we can conclude that the increase of specific surface area by the micropore area increased the adsorption efficiency of activated carbon on vapour phase components, but semi-volatiles or particulate matter are affected by the ratio of mesopore area to total specific surface area.

SSPT 36

The effect of activated carbon characteristics on adsorption efficiency of VOCs in cigarette smoke.

SASAKI T.; YAMASHITA Y.

Japan Tobacco Inc., Cigarette Materials R&D Division, Yokohama, Japan.

In the present study, the effect of pore structure (volume, pore size, etc.) of activated carbon on the adsorption efficiency of VOCs is examined, and the prediction of adsorption efficiency on VOCs in cigarette smoke is discussed.

Ten kinds of charcoal with various pore characteristics were used in this study. The relative concentration of each VOC was analyzed by gas chromatograph. The adsorption efficiencies (E) of VOCs increased with the amount of charcoal (w). Accordingly, the penetration efficiencies ($1-E$) ought to decrease in proportion to w. The relationship between w and $1-E$ could be described by a logarithmic penetration equation, $\ln(1-E)=-Kw$ (where, K:constant). The values of K on all VOC enabled estimation in a same manner. The value of K can be predicted from measurement of a micropore volume and a pore size distribution with a high degree of accuracy, by utilization of the general adsorption theories for a single component vapor.

Consequently, it is recognized that the adsorption efficiency of charcoal for various VOCs is predicted by analyzing the physical properties (volume, pore size).

SSPT 37

The activity of different carbon weights in a cigarette filter and the effects of triacetin.

WALKER J.; TAYLOR M.J.

Filtrona Technology Centre, Shaftesbury Avenue, Jarrow, Tyne and Wear, NE32 3UP, UK.

It is known that during storage the activated carbon in a cigarette filter can undergo a reduction in adsorption capacity. The factors contributing to this change in activity are complex and numerous however previous work has shown the critical role of storage time and temperature for both filter rods and assembled cigarettes. For a complex mixture such as cigarette smoke a good measure of the activity of carbon is the retention of vapour phase compounds. Thus the activity of varying coconut shell activated carbon weights in a cigarette filter towards vapour phase and semi-volatile compounds in cigarette smoke is presented. The initial capacity & hence weight of carbon per filter is another key factor in deactivation over time. The influence of plasticiser loading & its ratio to carbon in deactivation is not yet fully understood. Studies are presented on the differing performance of carbon towards a wide range of smoke compounds for carbon pre and post triacetin loaded for a range of triacetin loadings and carbon weights. Also comparisons are made of the retention characteristics of smoke compounds with different chemical properties and volatilities.

SSPT 38

Comparison of pyrolysis pattern of different tobacco leaves by double-shot pyrolysis-GC/MSD method.

LEE Chang-Gook; LEE Jae-Gon; JANG Hee-Jin; KWON Young-Ju; LEE Jang-Mi; KWAG Jae-Jin; RHEE Moonsoo

KT&G Central Research Institute, 302 Shinseong-Dong, Yuseong-Gu, Daejeon, Rep. of Korea.

Thermal pyrolytic behaviour of different types of tobacco leaves, such as flue-cured, Burley and oriental tobacco were investigated using double-shot pyrolysis-GC/MSD methods. Three grades of flue-cured tobacco and Burley tobacco, and three origins of Oriental tobacco were pyrolyzed over seven discrete temperature ranges with an interval of 50°C, from 100°C to 450°C with a heating rate of 50°C/min. Total ion chromatograms of pyrolysate of each of the temperature ranges showed different pyrolytic patterns due to different tobacco types and grades. Pyrolysis of temperature range from 150°C to 200°C and subsequent from 200°C to 250°C gave a distinct result among different types of tobacco leaves. In the case of higher temperature ranges, from 300°C to 350°C and subsequent temperature range from 350°C to 400°C, there were less distinct results between different tobacco types. With thermally evolved components such as nicotine, neophytadiene, furaneol, hydroxymethylfurfurole, megastigmatrienone, palmitic acid, and so forth, the principal component analysis gave different statistical distances among different types of tobacco leaves depending on the tobacco types, grades and temperature ranges.

SSPT 39

Volatile products from pyrolysis, oxidative pyrolysis and combustion of tobacco.

NAPPI L.; LIU Chuan

British American Tobacco, Group R&D Centre, Regents Park Road, Southampton SO15 8TL, UK.

Pyrolysis and oxidative pyrolysis of virgin Burley tobacco were carried out using a thermogravimetric analyser (TGA) under a ramped temperature programme. The same tobacco was also pyrolyzed under pure nitrogen in a furnace and was used to carry out combustion tests using the TGA. Heating rates in the range 5 and 80°C min^{-1} and different carrier gas (pure nitrogen, 5%, 10% and 21% oxygen in nitrogen) were used. Advanced kinetic analyses without assuming kinetic models were applied in order to calculate the activation energy and the pre-exponential factor values as a function of the overall rate of reaction progress. Selected volatile products generated by the TGA were analysed by a Fourier Transform Infrared Spectrometer (FTIR). The identification of the volatile species were conducted off-line using a multivariate (target factor) analysis software. The obtained information was used to differentiate between pyrolysis, oxidative pyrolysis and combustion of tobacco in terms of volatile products.

This study mainly highlighted that: (1) some thermal degradation processes are affected by the presence of oxygen and (2) the charring process is strongly dependent on the experimental conditions, and the preparation of the tobacco char has an effect on its combustion.

SSPT 40

The effect by the side chain structure of amino acids on the generation of aromatic amines in tobacco pyrolysis.

YOSHIDA S.; UWANO Y.; KUSAKABE T.

Japan Tobacco Inc., Tobacco Science Research Center, 6-2, Umegaoka, Aoba-ku, Yokohama, Kanagawa 227-8512, Japan.

2-Aminonaphthalene (2-AN) and 4-aminobiphenyl (4-ABP), which constitutes a part of aromatic amines in cigarette smoke, are listed as Group1 "human carcinogens" on IARC monograph. It has been reported that the amount of nitrogenous compounds in tobacco, especially protein which is a polymer of amino acids (AAs), contribute to the yields of aromatic amines in tobacco pyrolysis. The aim of this study is to examine the effect of AAs on the generation of 2-AN and 4-ABP by focusing on the side chain structure of AAs.

For preparation of the sample, aqueous solutions of asparagine, proline, alanine, and phenylalanine were sprayed onto cut flue-cured tobacco (5% w/w respectively). The control sample was prepared by adding water to the same tobacco as mentioned above. Individual AAs and five tobacco samples were pyrolyzed respectively by an infrared furnace in nitrogen atmosphere up to 800°C. 2-AN and 4-ABP collected on Cambridge pad were determined by GC/MS after solid phase extraction and derivatization.

The results showed that; 1) Addition of asparagine, proline, and alanine to tobacco increased yields of 2-AN and 4-ABP, although pyrolysis of three AAs yielded quiet small amounts of aromatic amines. 2) Addition of phenylalanine to tobacco increased the generation of 4-ABP higher than that of the other AAs, but increased 2-AN generation only

1.5-fold, although pyrolysis of phenylalanine yielded both aromatic amines 150-fold, an amount that was as large as that of the other AAs.

Considering the results; a) Yields of 2-AN in tobacco pyrolysis were only slightly affected by the side chain structure of AAs, and estimated that it depends mainly on the nitrogen content of AAs. b) There is a possibility that the amount of 4-ABP in tobacco pyrolysis may be affected by phenyl structure in the side chain of AAs.

SSPT 41

Comparison of the *in vitro* exposure methods for cytotoxicity assessment of cigarette mainstream smoke.

SHIN Han-Jae; SOHN Hyung-Ok; PARK Chul-Hoon; LEE Hyoung-Seok; MIN Young-Keun; HYUN Hak-Chul

KT&G Central Research Institute, 302 Shinseong-Dong, Yuseong-Gu, Daejeon, Rep. of Korea.

Generally *in vitro* biological studies on the activity of cigarette smoke have been conducted on the total particulate matter (TPM) or gas/vapor phase (GVP) prepared by bubbling through in buffer solution (indirect exposure method). Recently the whole smoke exposure system for the direct exposing cell at air-liquid interface to freshly generated cigarette smoke has been used to test the toxicity of cigarette smoke (direct exposure method). In this study we compared the *in vitro* cytotoxicity of cigarette mainstream smoke using both direct and indirect exposure methods. To compare the sensitivity and specificity of these exposure methods, we used the Kentucky Reference Cigarette 2R4F and two cigarette types which have control carbon filter and new carbon filter, separately. The cytotoxic activity of cigarette smoke was assessed by neutral red uptake assay using mouse embryo BALB/c 3T3 cells. According to EC_{50} values of direct and indirect methods, the cytotoxicity of new carbon filter cigarette was less active than the other cigarettes. On the other hand, we calculated the toxicity index of three cigarette types from the EC_{50} value of selected constituents (acetaldehyde, acrolein, formaldehyde, catechol, o-cresol, hydroquinone) known as high contribution to toxicity of cigarette smoke. Also, we compared these toxicity indexes with *in vitro* cytotoxicity results obtained by the above methods. The results suggest that the direct exposure method may be more useful for the assessment of cigarette smoke cytotoxicity.

SSPT 42

Mutagenicity of cigarette smoke vapour phase in the AMES test.

WIECZOREK R.; RÖPER W.; KAHL E.

Imperial Tobacco Group, Reemtsma Cigarettenfabriken GmbH, Corporate Responsibility, 22761 Hamburg, Germany.

For more than 30 years the Ames test is a widely used *in vitro* method for the determination of cigarette smoke condensate mutagenicity in bacteria. Recent Canadian regulation requires cigarette testing according to Health Canada method T 501 with five tester strains of *Salmonella typhimurium*, TA98, T100, TA102, TA1535 and TA1537. It is well known from literature that these strains respond differently to cigarette smoke condensates. The mutagenic effects of fresh whole smoke and vapour phase in this bacterial system are less well known.

In the present study the mutagenicity of fresh whole smoke and its vapour phase was tested. All five tester strains were directly treated with freshly generated whole smoke and vapour phase from a 13 mg tar American Blend cigarette. Smoke of up to 12 cigarettes was bubbled through 12 ml of buffered bacteria suspension. The smoke exposed suspensions were further processed using standard Ames plate incorporation techniques.

In comparison to the other tester strains, TA98 showed the highest response to fresh whole smoke, whereas vapour phase mutagenicity could only be detected with strain TA100.

In order to further investigate vapour phase mutagenicity, TA100 only was used for tests with different experimental cigarettes, namely American Blend style with three different filters and tar yields as well as three single grade tobacco styles, Burley, Virginia and Oriental cigarettes. Mutagenicity was expressed per cigarette and a per mg tar, respectively. Mutagenicity of fresh whole smoke was compared to effects of vapour phase and those measured with Cambridge filter condensates. It is obvious that only part of the fresh whole smoke is retained by the bacterial suspension. In order to get an estimate of the particulate matter trapped optical density was measured at 370 nm.

Fresh whole smoke mutagenicity in TA100 was higher than vapour phase mutagenicity. On the other hand, on a per mg particulate matter basis, Cambridge filter condensate mutagenicity was lower than whole smoke mutagenicity, possibly caused by some lack of vapour phase compounds in tar or different mutagenic mechanisms.

Significant differences were also found between cigarettes with different tar yields and tobacco types, Burley exhibiting the most pronounced effects, confirming the data from previous condensate studies.

SSPT 43

Detection of cytotoxicity in the water-insoluble fraction of cigarette smoke gas vapor phase using a whole smoke exposure system.

NISHINO T.; NARA H.; FUKANO Y.

Japan Tobacco Inc., Tobacco Science Research Center, 6-2, Umegaoka, Aoba-ku, Yokohama, Kanagawa 227-8512, Japan.

When biological evaluations of cigarette smoke are conducted, the particulate phase (PP) and gas vapor phase (GVP) of mainstream smoke are collected separately and exposed to cultured cells. GVP is generally collected in phosphate buffer saline (PBS) by bubbling method. However, using this methodology for collecting GVP means that water-insoluble fraction of the GVP will not be collected. Therefore, the toxic potential of the water-insoluble fraction of the GVP has hardly been investigated. The objective of this study is to research toxic effects of water-insoluble fraction of GVP using a whole smoke exposure system such as CULTEX® system that enables cultured cells to be exposed to PP and GVP of native cigarette smoke including water-insoluble fraction.

For the research on the water-insoluble fraction of the GVP from mainstream smoke of research cigarette K2R4F, we placed a piece of Cambridge filter and 6 impingers connected in tandem with each other between the cigarette ports and the cylinder in the smoking machine VC10. The PP of K2R4F was removed by the Cambridge filter and the GVP was forwarded to the 6 tandem impingers containing PBS to remove the water-soluble fraction. The water-insoluble fraction passing through the impingers was directly exposed to Chinese hamster ovary cells (CHO-K1). After the exposure, we performed neutral red uptake assay (NR uptake assay) to research the cytotoxicity of the water-insoluble fraction of the GVP. To confirm the removal of the water-soluble components, we analyzed cytotoxic aldehydes as markers using High Performance Liquid Chromatography (HPLC) and found that no aldehydes

were detected after the 6th impinger. As a result, the cytotoxicity was detected even from the water-insoluble fraction of the GVP.

SSPT 44

The effects of nitric oxide in the *in vitro* toxicity of cigarette smoke condensate.

REID J.R.; LEVERETTE R.D.; BENNETT M.B.; MISRA M.; HAMM J.T.

Lorillard Tobacco Company, A.W. Spears Research Center, Greensboro, NC 27405, USA.

Nitric Oxide (NO) has a physiological role in normal signaling processes, but it is also thought to play a role, alone or through interactions with other chemical species, in the etiology of several disease conditions. Apart from its normal regulatory functions in biological systems, NO has been demonstrated to play cytoprotective as well as a cytotoxic role that can occur in inflammatory responses. Additionally, NO alone, or through interactions with oxygen and nitrogen radical species, can potentially lead to mutagenic, genotoxic, and inflammatory effects in biological assay systems. NO is found in mainstream cigarette smoke at levels around 300 ppm. The objective of this study was to study the possible effect of exogenous nitric oxide on cigarette smoke condensate-mediated cytotoxicity, mutagenicity, free radical generation, and inflammatory responses in cultured cells. Results from our studies with *Salmonella* strain TA100 (S9-) gave low levels of activity separately for the NO-donor diethylamine-NONOate (DENO) and condensate. However, when exposed simultaneously, the specific activity increased significantly, demonstrating a possible synergism. The measured rate of NO release from DENO was found to increase in the presence of cigarette smoke condensate (CSC), suggesting that CSC-induced mutations were augmented by the increased levels of released NO. Results were confirmed by the use of additional tester strains YG1024 and YG1029, known to be more sensitive to nitrogenous compounds. Studies utilizing both NO-donor compounds and NO-scavengers will be presented. Conversely results from Neutral Red Uptake cytotoxicity studies with CSC-DENO mixtures show a reduction in the cytotoxicity in cells. Additionally, the NO-CSC-mediated dose and time-dependent cellular release of the inflammatory cytokine IL-8 will be presented.

SSPT 45

Gene expression analysis of human lung cells treated with cigarette smoke.

ACHARD S.; VERRON T.; SEVESTRE O.; DUMERY B.

Altadis - Research Centre, 4 rue André Dessaux, 45404 Fleury-les-Aubrais, France.

Gene expression profiling is considered as a promising tool that may identify more sensitive, mechanism-based biomarkers. Such an experimental approach should provide a better understanding of the impact of chemical compounds on the intracellular mechanisms leading to different pathologies.

The purpose of this study was to evaluate and to compare the impact of mainstream cigarette smoke from two commercial blends, on the modulation of gene expression in A549 cells, a type II alveolar epithelial human cell line.

Cells were exposed to cigarette smoke at various concentration levels for 20 h. After treatment, RNA was extracted, cDNA synthesized and gene expression analyzed on a cDNA macroarray spotted with 288 toxicologically relevant genes (Human Oligo GEArray®, membrane EHS 401).

Global gene expression analysis was conducted in three steps using R-Statistical computing. Initially, a normalization based on the "quantile method" was used to produce comparable arrays. In a second step, a discriminate analysis was performed to select the most relevant genes for differentiating the effect of smoke from the respective cigarettes. A third step consisted in confirming the significant differences in gene expression with two statistical analysis : SAM and PAM (Significant and Predictive Analysis Methods).

Cigarette smoke significantly modulated the expression of some genes. As an example, the genes allocated to the weakest TPM-concentration are either associated with cell cycle regulation or transcription factors: TP53, PCNA, and SAFB. Discrimination between blends was observed with those genes. In the higher TPM-concentration, no genes involved in DNA damage were modulated; however, some genes involved in metabolism such as GAL3ST1, GPX1, and PON3 were modified.

In conclusion, several genes were identified as potential biomarkers of early biological effects caused by mainstream cigarette smoke exposure in our *in vitro* experimental conditions.

SSPT 46

Results from CORESTA yield-in-use ring trial.

NELSON P.R.(1); BURTON S.(2)

1. *R.J. Reynolds Tobacco Co., R&D, P.O. Box 1487, Winston-Salem, NC 27102-1487, USA.*
2. *Arista Laboratories, 1941 Reymet Rd., Richmond, VA 23237-3723, USA.*

Cigarette yield-in-use (YIU) provides an increasingly popular, non-invasive method for determining how much smoke passes through a cigarette into the mouth of a smoker. Many laboratories worldwide use YIU measures to better understand the interaction of smokers and their cigarettes. Within the Smoking Behavior Sub-Group of CORESTA, there is also an interest in understanding differences among YIU methods and the impact that they might have on the comparison of results from different labs. In 2006, the Sub-Group conducted a ring trial to examine the accuracy and precision of nine different YIU measures. Data were obtained from a well characterized set of cigarette filters which were analyzed in nine labs, according to each lab's unique protocol.

To compare results among methods, each participating lab analyzed the filters from cigarettes that were machine smoked and had "tar" and nicotine yields determined by standard methods. Three types of cigarettes were smoked at two regimes that were expected to provide yields near the middle and low ends of those expected from human smokers. Each laboratory analyzed the "unknown" filters using its own in-house extraction, calibration and analysis methods. The results were then reported back to a central location for collation and statistical analysis.

Despite the differences in YIU methods, the results among the nine participating labs were remarkably consistent. The methods used may be divided into three broad groups. For "tar", the overall accuracy was 6%, 15 and 28% for each group of methods. For nicotine, the accuracy was 3%, 7 and 33% for the three method groups. Within-lab precision CV ranged from 3-9% with a CV of 21% among labs.

SSPT 47

A study design to investigate the influence of ISO tar yield and tar band switching on cigarette smoke dose as determined by filter analysis and biomarkers of exposure.

SHEPPERD C.J.; McEWAN M.

British American Tobacco, Group R&D Centre, Regents Park Road, Southampton SO15 8TL, UK.

Two methodological approaches have previously been used to achieve estimates of human smoke exposure, namely filter analysis and biomarkers of exposure.

Filter analysis estimates the maximum amount of smoke that exits the cigarette filter and is taken into the mouth (mouth level exposure). Conversely, biomarkers of exposure use the level of biomarkers of specific smoke components in biological media (urine, blood or saliva for example) to estimate smoke uptake into the body.

Past studies have shown both good and poor correlations between mouth level and biomarker exposure estimates, depending on the smoke component in question [Rickert *et al.* 1981, Russell *et al.* 1982]. Studies using filter analysis have shown that smokers of lower tar yield products have lower mouth level exposure of 'tar' and nicotine than smokers of higher yield products [Shepperd & Mariner, 2001]. However, data from biomarker studies are less conclusive. Some have shown a dose response for nicotine and metabolites, but for other smoke components such as pyrene and NNK there appears to be no significant differences in the levels of biomarkers of exposure found in the body fluids in groups of cigarette smokers over a range of tar yields [Benowitz *et al.*, 2005, Hecht 2005].

Since these studies have not taken into account smoke retention or metabolism differences between individual smokers, the correlation between mouth exposure and biomarkers may be partially dependent on these aspects. Therefore, by incorporating smoke retention measures and tar band switching into a filter analysis/biomarkers of exposure study, a design has been developed to further investigate the level of correlation between these exposure measures.

The design includes comparison of the level of estimated human cigarette smoke exposure in smokers of a range of ISO/FTC tar bands and non-smokers as well as the influence of switching to a lower tar band.

SSPT 48

Biomarkers for the tobacco smoke-related exposure to acrylonitrile.

SCHERER G.; URBAN M.; HAGEDORN H.W.

ABF Analytisch-Biologisches Forschungslabor GmbH, Munich, Germany.

Acrylonitrile is a reactive volatile compound, which is cytotoxic, mutagenic and possibly carcinogenic to humans. A major source for the non-occupational exposure to acrylonitrile is tobacco smoke. Mainstream and sidestream smoke yields of cigarettes, when smoked according to CORESTA/ISO/FTC smoking regimes, were reported to be 3 - 15 and 76 - 85 µg/cigarette, respectively. Environmental tobacco smoke (ETS)-related acrylonitrile concentrations in indoor air were found to amount to 0.6 - 0.8 $\mu g/m^3$. Environmental exposure to acrylonitrile is low, except near factories and waste sites. There is some exposure to acrylonitrile from the residual monomer in commercial polymeric material in fibres and food package materials.

A suitable long-term biomarker of acrylonitrile exposure is the 2-cyanoethylvaline (CEVal) hemoglobin adduct. Smokers were found to have 15 - 50-fold higher CEVal adduct levels than non-smokers. Adducts levels in smokers are strongly associated with the smoking dose.

The major urinary metabolite of acrylonitrile in humans is 2-cyanoethylmercapturic acid (CEMA), representing a suitable short-term biomarker of acrylonitrile exposure. We have developed and validated an LC-MS/MS method (liquid chromatography with tandem mass spectrometry) using deuterated CEMA as an internal standard. Urinary excretion of CEMA was found to be, on average, > 100-fold higher in smokers compared to non-smokers. CEMA was also strongly correlated with the smoking dose. Our results show that CEMA is a specific short-term biomarker for the tobacco smoke-related exposure to acrylonitrile.

SSPT 49

Performing clinical trials to good clinical practice for biomarker assessments to good laboratory practice.

SHAW I.W.

Covance Laboratories Ltd., Otley Road, Harrogate HG3 1PY, UK.

Conducting clinical trials for the collection of biological samples and biomarker assessments is an established research tool for the tobacco industry. With the renewed possibility of FDA regulation applied to assess reduced risk tobacco products, the need for appropriate controls and standards in the conduct of these clinical trials is paramount. A recognised set of standards are provided by Good Clinical Practice and by Good Laboratory Practice.

This paper will highlight key factors in the execution of regulatory compliant clinical trials in smokers and non-smokers for the tobacco industry to ensure the application of these controls and standards. The presentation will draw upon experiences gained in the conduct of a number of clinical trials performed in typical clinical research centres in Europe on smoking and non-smoking volunteers.

It will consider the implications for maintaining Good Clinical Practice throughout these studies and will review the challenges encountered in the conduct of studies in this population. Examples influencing ethical review and approval and the recruitment of volunteers will be discussed. The implications for then maintaining Good Laboratory Practice in the conduct of related activities including sample management, data management and collection and handling and analysis of biological samples will also be discussed.

POSTER ABSTRACTS

SSPTPOST 01

Impartial monitoring tool for the ASTM test E 8187-02b - Standard test method for measuring the ignition strength of cigarettes.

ROSE N.

Borgwaldt KC GmbH, D-22525 Hamburg, Germany.

To prevent fatal fires generated by lit cigarettes dropped onto beds or upholstered furniture a test method for the propensity of a cigarette to ignite soft furnishings is established in many states of the USA.

The test requires that more than 75% of a batch of 40 cigarettes is self extinguishing before reaching the tipping paper while placed on ten layers of Whatman No.2 substrate.

The test requires the individual doing the test to assess visually both: if the cigarette has self extinguished and the level of charring caused to the substrate. As with all processes requiring individual assessment, results are subjective to each individual and so inherently more variable than a measured test.

The poster will introduce a new technology to automate the assessment of the cigarette status, aimed specifically at providing repeatable results for the ASTM test.

Additionally this new application will also discuss the additional information that can be gained during the automated test, such as burn time, temperate profile and conduction to the contact surface.

In conclusion the data of manual and sensor controlled testing will be compared to provide a clear indication as to the potential improvements possible from automating the ASTM test.

SSPTPOST 02

Potential sources of measurement error when measuring the permeability of Low Ignition Propensity (LIP) papers to ISO 2965.

VINCENT J.H.; HANKEY J.; TINDALL I.

Cerulean, Rockingham Drive, Linford Wood East, Milton Keynes, MK14 6 LY, UK.

The measurement of permeability of cigarette envelope papers used in the manufacture of Low Ignition Propensity (LIP) cigarettes presents a number of practical problems:

- Measurements must be made on both the conventional and low permeability band regions.
- The low permeability band must be measured using a small orifice (15 x 2 mm^2 compared to the normal 20 x 10 mm^2) with a resulting flow as low as 1 ml/minute.
- Leakage from the periphery of the paper - both between the paper and the clamp face and laterally through the paper - is more significant as the orifice to perimeter ratio increases (smaller orifice size), which can offset permeability values obtained on the same paper when different orifice sizes are used.

The effect of measuring cigarette papers using 2 orifice sizes has been quantified by combining two orifices into a single clamp and enabling both vacuum and pressure measurements. Through measurement without needing to invert papers this procedure has enabled a direct measurement of flow asymmetry for banded papers, which was found not to be significant.t

To provide a means of validating measurement accuracy for the two orifice sizes, aluminized plastic with a fine pin hole was used to provide a constant flow without peripheral leakage. Non-banded envelope papers were measured using both large and small orifices to determine the fraction of flow due to the area and the periphery for each orifice. As much as 20% of the measured flow from the small orifice could be ascribed to the periphery.

The use of rubber clamp faces to reduce the peripheral leakage was investigated and significant reductions were obtained.

Recommendations are made to reduce inconstancies in comparisons between paper types and to enable more reliable permeability measurements of LIP papers.

SSPTPOST 03

A possible test piece for size and ovality correlation in inter-laboratory trials.

VINCENT J.H.; HANKEY J.; RUSHWORTH J.; TINDALL I.

Cerulean, Rockingham Drive, Linford Wood East, Milton Keynes, MK14 6 LY, UK.

Measurement of circumference (or, more strictly, periphery) and ovality (maximum minus minimum diameter) of cigarette and filter rods is a key production and quality control requirement in the industry, not only where "out of round" is an undesirable property but also where ovality is a design characteristic for particular markets.

Over many years CORESTA has organized inter-laboratory trials of measurement equipment but a convenient, consistent and robust check piece for ovality measurement has not been available. Rapid prototyping manufacturing methodology has enabled a variety of rods with arbitrary cross-sections to be manufactured at low cost in rugged plastic. This set of check rods has been produced with nominal periphery of 18, 21 and 24 mm with ovalities from 0 to 1 mm. These included nominally elliptical and 'D' shaped cross-sections commonly seen in cigarette manufacture, where the overlap is flattened.

These test pieces have then been evaluated for their suitability as standards, particularly in terms of surface finish and robustness in a number of different measurement instruments. Nominal repeatability and reproducibility's obtained with these test pieces is here reported.

Results are reported that support the use of such check pieces in future inter-laboratory trials.

SSPTPOST 04

Observations on the effects of air flow on smoking yields as applied to linear and rotary smoking machines.

TINDALL I.; MASON T.

Cerulean, Rockingham Drive, Linford Wood East, Milton Keynes, MK14 6 LY, UK.

It has been long established that air flow is one of the key factors that effect smoking yields in routine smoking machines. This is acknowledged in ISO 3308 by a requirement to measure and set air flow as part of the smoking criteria. However it is becoming clear that the magnitude alone of the air flow is not sufficient to obtain consistent results between machine types.

Current commercially available machine types have small but measurable differences in mean yields as shown through collaborative studies of monitor cigarettes.

This paper shows that changing air flow direction, whilst maintaining the magnitude of flow, on linear machines with some products can show changes in CO yield of up to 9% per cigarette. It is postulated that the slight but measurably higher CO yields obtained for monitor cigarettes on rotary machines are in part due to flow direction. This theory is tested by building a rotary smoking machine with linear smoking machine characteristics and then comparisons made for established monitor cigarettes.

On a small sample of data there is evidence that CO yields in this configuration more closely match those of linear machines on those monitors tested. This appears to hold true for a range of cigarette types from low yield to high yield. The effect upon NFPDM and nicotine yields is also described.

This paper explores the causes of CO yield differences with particular reference to air flow and poses some questions as to how these might be minimized through attention being paid to design of air flow.

SSPTPOST 05

Adsorption and thermal release of menthol by zeolite.

CAO Yi (1); WANG Ying(1); ZHU Jian Hua(1); LIU Chuan(2)

1. *School of Chemistry and Chemical Engineering, Nanjing University, Nanjing 210093, China.*
2. *British American Tobacco, Group R&D Centre, Regents Park Road, Southampton SO15 8TL, UK.*

Zeolites are known to exhibit selective adsorption and catalysis of volatile compounds in the gas stream, therefore various attempts have been made to use them to reduce carcinogenic compounds for environmental and health protection. On the other hand, the pores and channels of zeolites may be employed to accommodate desirable volatile species, for example, menthol. The potential benefits include enhanced stability during storage and modified release of the substance during smoking. In this study the adsorption of menthol at ambient temperature was carried out on a group of zeolites by adding them onto tobacco rod. The release of menthol took place when the hot coal of the burning cigarette approached the zeolite host. Finally, the adsorption efficiency on nitrosamines and PAHs in cigarette smoke by the treated zeolites was evaluated.

The thermal release of menthol from zeolites was measured by a temperature- programmed-desorption (TPD) method. Most zeolites studied in this work showed a main desorption peak below 500 K with the exception of NaY, which displayed two desorption peaks around 545 and 650 K. In addition, the zeolite additives reduced about one third of nitrosamines in the sidestream smoke.

SSPTPOST 06

Analysis of acrylamide in mainstream cigarette smoke and effects of reducing sugars on acrylamide content.

KIM Ick-Joong; LEE John-Tae; MIN Hye-Jeong; KIM Hyo-Keun; HWANG Keon-Joong

KT&G Central Research Institute 302 Shinseong-Dong Yuseong-Gu, Daejeon, Rep. of Korea.

Acrylamide has been found in many foods in our everyday life. Acrylamide levels in foodstuffs were analyzed by a GC/MS after bromination of acrylamide and by a LC/MS for underivatized acylamide. In these methods, various clean up procedures are applied for the purification of the extract.

In this study, a simple and fast method for analysis of acrylamide in mainstream cigarette smoke, without the clean up step, was developed and the effects of reducing sugars on acrylamide content were observed. The analysis of acrylamide in mainstream cigarette smoke started by collecting TPM from smoking and extracting with 0.1% acetic acid solution and then detecting by liquid chromatography tandem mass spectrometry using electrospray in the positive mode. The recovery of acrylamide in 2R4F reference cigarette was 97.7% and the reproducibility was 2.5% and the limit of detection was 6.4 ng/cigarette.

Reducing sugars are considered to be the main precursors of acrylamide in foodstuffs. Cut tobacco contains substantial amounts of the reducing sugars, which may explain the occurrence of acrylamide in mainstream cigarette smoke. The effects of reducing sugars was studied in an experiment with a range of tobacco grades. The result indicated that level of reducing sugars in cut tobacco was linearly correlated to acrylamide content in mainstream cigarette smoke.

SSPTPOST 07

Development and validation of an HPLC/UV method for simultaneous determination of 9 organic compounds.

KA Mi-Hyun; KIM Mi-Ju; CHO Sung-Eul; KIM Yong-Ha; MIN Young-Keun

KT&G Central Research Institute 302 Shinseong-Dong Yuseong-Gu, Daejeon, Rep. of Korea.

Humectants and preservatives are added to cigarettes to delay microbiological, enzymatic and chemical deterioration and to extend shelf life. This study was conducted to establish a simple and rapid method for determining preservatives that can be used in the cigarette manufacturing process. Nine preservatives were examined: dehydroacetic acid, sorbic acid, benzoic acid, methylparaben, ethylparaben, isopropylparaben, propylparaben, isobutylparaben and butylparaben. The preservatives were separated and detected simultaneously by reversed-phased high-performance liquid chromatography (HPLC)/UV-visible spectrophotometer (UV) methods. The samples were extracted with methanol and centrifuged and then filtered. The clear filtrate was analyzed by the HPLC. The analysis was carried out by isocratic elution (acetonitrile: methanol: 0.005 M CTA buffer=15: 35: 50) at pH 4.6 and wavelength of the detector was set at 254 nm. Under these conditions, nine preservatives were separated within 40 min and this method was applied to the determination of preservatives in cigarettes. The limit of detection (LOD) and limit of quantification (LOQ) were 0.12~1.20 μg/g and 0.23~5.83 μg/g, respectively. Linearity, recovery rate, repeatability and reproducibility of this method were also validated. As compared with Health Canada Method (T-313), this developed method can be used to simultaneously determine the nine preservatives and offers a simplified pre-treatment procedure, fast analysis time and excellent validation results.

SSPTPOST 08

Evaluation of the behaviour of the anion in RECON extract by various adsorbents.

HAN Young-Rim; SUNG Yong-Joo; BAEK Shin; KIM Kun-Soo; RHEE Moonsoo

KT&G Central Research Institute 302 Shinseong-Dong Yuseong-Gu, Daejeon, Rep. of Korea.

The objective of this study is to evaluate the behaviour of anions in RECON extract by various adsorbents and several ion exchange resins using ion chromatography. The determination of ionic species in solution is a classical problem with a variety of solutions. Conventional wet-chemical methods such as titration, photometry, gravimetry, turbidimetry and colorimetry are all labour-intensive, time-consuming, and occasionally troublesome. In contrast, ion chromatography offers the following advantages: speed, sensitivity, selectivity and simultaneous detection. The isocratic ion chromatographic method to separate and determine main ions in RECON extract was applied. The method allows the separation of anions on Dionex Ion Pac AS18 column by KOH elution and conductometric detection. The anions such as fluoride, chloride, nitrite, sulfate, bromide and nitrate were separated within 20 minutes. In this study, various adsorbents such as activated carbon, wood charcoal and ion exchanger resins were applied. . The experimental parameters such as amount of adsorbent, contact time, initial anion concentration and extract concentration were tested. The composition of ions was changed depending on the properties of adsorbents. The main anions adsorbed by the activated carbon resulted in decreases of 10% in chloride ions, 10% in sulfate ions and 25% in nitrate ions. The concentration of ions was also affected by the addition amount of adsorbent and concentration of RECON extract. The optimal condition of adsorption, especially for the nitrate anion was discussed in terms of the amounts of the adsorbent addition and concentration of RECON extract.

SSPTPOST 09

Studying effect of additives on chemical and taste characteristics of Caspian pipe tobacco.

MOHSENZADEH R.; MORADI G.R.; SHAMEL ROSTAMI M.T.; YAZDANPANAH A.A.

Tirtash Tobacco Research & Education Center, P.O. Box 48515-155, Behshahr, Iran.

The tobacco leaf is the chief source of flavour and aroma in any tobacco product but additive supplements are necessary to help maintain a consistency in taste and aroma. An experiment was conducted at the Tirtash Research & Education Center, Iran, in 2006, to evaluate the effect of additives (3 types of sidestream flavours & two types of smootheners), single and combination (12 treatments) on the taste and chemical characteristics of Caspian pipe tobacco. The samples of pipe blended were grades of Burley (34%), Virginia (56%) and Oriental tobacco leaves (10%) and cut-width 1.8-2 mm. Burley tobacco cased (licorice, cocoa, glycerin, water, invert sugar, ethanol alcohol) and then toasted at 80-90°C for three hours. Then after 6 months aging, samples were evaluated through a taste test (taste, irritant and harshness, aroma, burning rate) and chemical factors *e.g.* pH, nicotine, sugar, and compared with unsupplemented samples by pipe smokers (weight and moisture per package was 40±2 gr and 15-16% respectively). Percentages of nicotine and sugar blend were 1.41±0.04, 7.5±0.5 respectively and pH=5. Results showed that the application of a combination of additives can improve smoke flavour, taste, impact, combustibility and acidic number, and can reduce harshness, irritation and volatile base salts of Caspian pipe tobacco.

SSPTPOST 10

Study of the pyrolysis pattern and the transfer rate of the organochlorine pesticide in tobacco.

MIN Hye-Jeong; JANG Seok-Su; KIM Ick-Joong; KIM Yong-Ha; MIN Young-Keun

KT&G Central Research Institute 302 Shinseong-Dong Yuseong-Gu, Daejeon, Rep. of Korea.

GRLs (Guidance Residue Levels) are recommended by the CORESTA ACAC guide. In the GRLs list, organochlorine group is one of pesticide commonly used on tobacco cultivation. In this model study, the quantitative correlation between transfer rate of pesticide residue into tobacco smoke and pyrolysates was investigated by spiking cigarettes with organochlorine pesticides. The spiking concentration referred to the range of GRLs list and the organochlorine pesticides in mainstream smoke were analyzed by GC-MS and gas chromatography with selective detector (ECD). To understand the composition variation versus temperature, the behaviour of pesticides were investigated by pyrolysis-gas chromatography-mass spectrometry (Py-GC-MS). In this study, the transfer rate of pesticide residue into tobacco smoke at each spiking concentration and the composition of pyrolysates were analyzed differently. At low concentrations, pesticides in smoke were not detected by pyrolysis. At high concentrations, organochlorine pesticides were transferred into tobacco smoke in 2~10% each of component and most of pesticides were pyrolyzed during smoking. It was found that the decomposition compounds from organochlorine pesticides were composed of oxygenous and nitrogenous compounds. This study could estimate that transfer rate of pesticides into tobacco smoke at the range of GRLs concentration recommended by the CORESTA ACAC is a very small amount.

SSPTPOST 11

Characteristic of analytical techniques for hydrogen cyanide in mainstream smoke.

LEE John-Tae; KIM Hyo-Keun; HWANG Keon-Joong; LEE Kyung-Gu; JANG Seok-Su

KT&G Central Research Institute 302 Shinseong-Dong Yuseong-Gu, Daejeon, Rep. of Korea.

Hydrogen cyanide (HCN), formed from pyrolysis of various nitrogenous compounds such as protein, amino acids and nitrate in tobacco, is present in both the particulate phase and vapor phase of cigarette smoke. Typically the determination of HCN in cigarette smoke has been done through colorimetric and electrochemical techniques, such as fluorescence spectrometry, spectrophotometry (UV), continuous flow analyzer (CFA), capillary GC-ECD and ion chromatography (IC). The general procedure for determining HCN in cigarette smoke involves the collection of mainstream smoke through impingers filled with trapping solution and Cambridge filter pad. Most of these techniques are time-consuming and some lack specificity or sensitivity. The available results from both internal testing and reported literatures for 2R4F Kentucky reference cigarette, smoked under ISO conditions, showed a relatively wide variation ranging from 100 to 120 µg/cig of HCN. Especially, the precision and accuracy of the analytical result of HCN tended to get worse in low tar cigarettes and under intense smoking conditions.

This paper suggests an optimized analytical method including the modification of previously used methods to obtain lower detection limits and to improve accuracy and precision and therefore is applicable for a wide range of tar levels in cigarettes under ISO and intense

smoking conditions. This method includes improved sample collection and quantification systems such as the number of absorption tubes, the type of extractant and reaction time for colour development. The pH of collection solution is over 11 to avoid volatilization loss of HCN and using a cooled trap to collect HCN were the recommended conditions to analyze HCN in mainstream smoke. Between seven and twelve minutes is the best effective reaction time from the addition of the colour reagent to the formation of cyanogens chloride almost reaching a maximum.

SSPTPOST 12

ISO 17025 accreditation for *in vitro* toxicology tests on cigarette smoke products.

COOKE L.; COWIN R.; LANGFORD R.; DELVES S.; GARCIA-CANTON C.; JONES S.; BLACK S.

Advanced Technologies (Cambridge) Ltd., 210 Cambridge Science Park, Milton Road, Cambridge CB40WA, UK.

One of the British American Tobacco Group of companies, Advanced Technologies (Cambridge) Ltd (ATC), performs 'duty of care' *in vitro* toxicology testing on tobacco products, to examine the potential harmful effects of cigarette smoke on DNA.

Genotoxicity tests (eg Ames, *In Vitro* Micronucleus) identify compounds that have the potential to induce genetic damage. Neutral Red assays identify cytotoxicity. These tests are accepted by OECD[1] and NICEATM[2] as valid methods for the assessment of the genotoxic and cytotoxic potential of novel compounds.

In the wider scientific environment, the majority of these tests are performed as part of the non-clinical safety evaluation testing of novel compounds in accordance with Good Laboratory Practice regulations. Laboratories testing products outside this scope (*e.g.* the tobacco industry) cannot apply to be part of the MHRA GLP programme. ATC decided therefore to perform their testing to an international standard, ISO 17025, to confirm the reliability and reproducibility of its results.

Generally, ISO 17025 accreditation is undertaken by chemical and physical laboratories who perform tests where the parameters are readily defined and quantitative. Such systems are not applicable to many biological tests (*e.g. in vitro* toxicology testing), because the tests are qualitative and imprecise.

One of the problems faced is the absence of standards to demonstrate validity of the measurements, therefore the testing needs to be performed under rigorously-controlled conditions. Assessment to the OECD Guidelines and ISO 17025 shows that the laboratory is technically competent, operates robust measurement traceability and internal quality control systems that enable it to accurately analyse materials that are inherently variable.

The desire to achieve ISO 17025 accreditation presented several challenges for both ATC and the accrediting body which were successfully overcome, resulting in ATC becoming the first laboratory in Europe to gain ISO 17025 accreditation for *in vitro* toxicology studies on smoke condensates.

[1] Organisation for Economic Co-operation and Development
[2] National Toxicology Program Interagency Center for the Evaluation of Alternative Toxicological Methods

SSPTPOST 13

Toxicity assessment of gas phase cigarette smoke using cell-free method.

PARK Chul-Hoon; SOHN Hyung-Ok; SHIN Han-Jae; LEE Hyoung-Seok; HYUN Hak-Chul

KT&G Central Research Institute 302 Shinseong-Dong Yuseong-Gu, Daejeon, Rep. of Korea.

In vitro toxicity tests such as cytotoxicity, mutagenicity and genotoxicity assay are useful for evaluating the relative toxicity of smoke or smoke condensates from different cigarette configurations. A major disadvantage of these tests as toxicity screening methods for tobacco product test and development is that they are relatively time-consuming and expensive. Recently, a cell-free glutathione consumption assay (GCA) as a rapid and simple screening method for the toxicity assessment of smoke has been reported by Cahours X. *et al.* (CORESTA, 2006). This study was performed to assess the capability of GCA to predict the toxicity of gas phase cigarette smoke (GVP) and to further identify individual compounds responsible for the GSH consumption. Three types of cigarettes such as 2R4F, charcoal filter cigarette (CFC), and new charcoal filter cigarette (NCFA) were evaluated by using GCA method and Neutral Red Uptake assay. The carbonyl compounds, which may contribute to toxicity, were also measured and the GSH consumption by these compounds was individually observed. The overall order of toxicity using GCA method was 2R4F > CFA > NCFA, which was consistent with the result of Neutral Red Uptake assay. The levels of carbonyl compounds of NCFA were lower than those of 2R4F and CA, indicating that GSH consumption was associated with carbonyl compound yields. A major toxicant under current study is acrolein, which contributed to more than half of the GSH consumption. Collectively, the toxicity of GVP determined by GCA method may be mainly attributed to acrolein.

SSPTPOST 14

Smoking-related changes in biomarkers of effect in exhaled breath condensate (EBC): results of a pilot study.

SCHERER G.; HAGEDORN H.W.; URBAN M.; ENGL J.; KAVVADIAS D.

ABF Analytisch-Biologisches Forschungslabor GmbH, Munich, Germany.

Exhaled breath condensate (EBC) is a non-invasively obtainable biofluid which is regarded as reflecting the composition of the bronchioalveolar lining fluid. The effect of tobacco smoking on inflammation biomarkers in EBC has been investigated in a number of studies, with controversial results up until now.

The purpose of our investigation was to establish, validate and apply methods for collecting EBC and measuring EBC biomarkers in order to determine the suitability of this technique for the evaluation of PREPs.

Analytical methods for the following EBC constituents were established and validated: nitrite, nitrate, Na^+, K^+, Ca^{2+}, Mg^{2+}, urea, alpha-amylase, 3-nitrotyrosine, aldehydes, total proteins, interleukine-6, tumour necrosis factor alpha, adenosine, and cadmium. Nitrogen oxide in exhaled breath (NOex) was also determined. These biomarkers were applied to a semi-controlled pilot study with 12 volunteers (6 non-smokers, 6 smokers), who supplied 11 EBC samples each during the course of the study. All EBC markers measured in the pilot study showed high intra- and inter-individual variations. In addition, there was significant interference from background contamination. Nitrite and NOex were inversely associated with smoking, whereas nitrate was not affected by smoking. 3-Nitrotyrosine tended to be

elevated in smokers and showed a weak association with the smoking dose. Acrolein, crotonaldehyde and hexanal were, on average, significantly higher in EBC of smokers compared to non-smokers. The effect of smoking was weaker and not significant for heptanal and malondialdehyde and almost absent for nonanal.

Aldehydes (in particular crotonaldehyde) and 3-nitrotyrosine show some potential as biomarkers of smoking-related effects in the lung. NOex is a suitable medium- to long-term biomarker for smoking related suppression of the alveolar NO synthetase (NOS) activity. EBC technology requires further improvements to be suitable for application in testing of modified tobacco products.

SSPTPOST 15

A ring trial test of nicotine uptake determinations by multiple laboratories.

BYRD G.; OGDEN M.

R.J. Reynolds Tobacco Company, Human Studies Division, Winston-Salem, NC, USA.

Total nicotine uptake by smokers is best determined by measuring nicotine and its major metabolites (usually five) in 24-h urine samples. Of additional interest is the primary metabolite cotinine in saliva since it is also related to total nicotine uptake. CORESTA recently coordinated a ring trial test among several laboratories for performance testing of nicotine uptake determinations in urine and saliva.

Uniform sets of urine and saliva samples were prepared and shipped to 10 participating labs. Within each set were embedded calibration standards (seven in triplicate) prepared by adding known amounts of each analyte to a non-smoker matrix; the remaining samples (seven in triplicate) were authentic samples from smokers. Participating labs analyzed all samples as unknowns using their own calibration standards and methods and returned the data, including raw area counts and response equations, to the coordinator.

For urine calibration standards, all the means for reported concentrations of individual analytes (nicotine, cotinine, 3'-hydroxycotinine and their respective glucuronide conjugates) were ±20% of prepared concentrations; for total nicotine equivalents (sum of the six individual analytes), reported concentrations were ±10%. Thus, general accuracy for total nicotine equivalents was quite good. For some analytes such as nicotine-N-glucuronide and 3'-hydroxycotinine-O-glucuronide, however, mean reported concentrations varied greatly by lab. Inter-lab CV's were 10-25% for the aglycons and approximately twice that for the glucuronides; total nicotine equivalents CV's ranged 11-15%. For authentic urine samples, agreement with mean reported and embedded calibration curve-corrected concentrations was within 20% for all analytes and less than 10% for total nicotine equivalents, which suggests that procedures used by the laboratories were generally appropriate. Use of the embedded calibration curve reduced inter-lab CV's by half. Saliva cotinine samples showed good agreement in general for both standards and authentic samples. Reference material inconsistency may be a factor in some comparisons, especially for the glucuronides.

SSPTPOST 16

Adsorption behaviour of propylamine on activated carbon fibre surfaces as induced by oxygen functional complexes.

KIM Byeoung-Ku; RA Do-Young; KWAK Dae-Keun; OH In-Hyeog; JO Si-Hyung

KT&G Central Research Institute 302 Shinseong-Dong Yuseong-Gu, Daejeon, Rep. of Korea.

In this study, the surfaces of activated carbon fibres (ACFs) were modified by nitric acid to introduce surface oxygen complexes and to observe the influence of those complexes on the propylamine adsorption of the ACFs. It was found that the oxygen complexes including carboxylic and phenolic groups were predominantly increased, which resulted in the increase of total surface acidity. However, the specific surface areas (S_{BET}) and the total pore volumes (V_t) of the modified ACFs were decreased by 5-8% due to the increased blocking (or demolition) of micropores in the presence of newly introduced complexes. Despite the decrease of textural properties, it was found that the amount of propylamine adsorbed by the modified ACFs was increased by approximately 17%. From the XPS results, it was observed that propylamine reacted with strong or weak acidic groups, such as COOH or OH, on the ACF surfaces, which resulted in the formation of pyrrolic-, pyridonic-, or pyridine-like structures.

SSPTPOST 17

Development of filter material selectively decreasing B(a)P yields in mainstream cigarette smoke.

CHENG Zhangang; CHEN Yikun; WANG Jianxin; ZHANG Chu-an; HU Suxia

Technology Center of Wuhan Tobacco (Group) Co. Ltd., Wuhan 430012, China.

In order to selectively decrease B(a)P in the total particulate matter of mainstream cigarette smoke, a kind of macromolecule polysiloxane was synthesized by two kinds of siloxanes, catalyst and other additive reagents. The cavity and polarity of macromolecules can be adjusted by changing the ratio of two kind of siloxanes. When the cavity is 2-3 nm, the B(a)P yields in mainstream cigarette smoke can be decreased by about 35% with the macromolecule filter, while the yields of nicotine, water and other polar compounds in mainstream smoke were maintained at the previous level. The macromolecule material still has good effect after long time storage.

Meanwhile, the technology of adding the macromolecular polysiloxanes to the filter was studied. The powder macromolecular material was formed as granule with the size of 0.25 mm by mixing with viscidity reagent. By adding 2 mg/mm macromolecule granule in the composite filter and changing the tobacco blend, the B(a)P yields can be decreased by 58% without a change in sensory quality.

SSPTPOST 18

A study to assist in the determination of tolerance values to be applied to the analysis of fine-cut smoking articles.

DYMOND H.F.(1); WILKES E.B.(1); KOTZIAS D.(2); HEBERGER K.(3); KEPHALOPOULOS S.(2)

1. *Consultant, European Smoking Tobacco Association, c/o 45, Monarch Way, West End, Southampton, SO30 3JQ, UK*
2. *European Commission, DG Joint Research Centre, Institute for Health & Consumer Protection, Physical & Chemical Exposure Unit, T.P. 281, I-21020-Ispra (VA), Italy*
3. *Chemical Research Center, Hungarian Academy of Sciences, P.O. Box 17, H-1525 Budapest, Hungary*

The European Smoking Tobacco Association (ESTA) in conjunction with the Joint Research Centre of the European Union (JRC) agreed to a joint experiment to determine the variability of data from routine smoking of fine-cut smoking articles.

The study was conducted in four phases, each phase taking approximately six months to complete. Each phase consisted of the analysis of three commercially available brands of fine-cut tobacco. The tobacco and tubes for each phase were distributed to each participating laboratory by the manufacturing company.

As a result of the co-operation 19 laboratories began this study by taking part in phase 1. Five of these laboratories are directly responsible for, or contracted to provide, analysis of tobacco products for five national Governments within the European Union. Two laboratories were independent contract laboratories and the remainder were associated with the tobacco industry. As a result of company mergers and takeovers or laboratory closures, the number of laboratories that completed all four phases was reduced to 14.

The procedures outlined in method ISO 125592-3 were followed throughout. At each phase, each laboratory was required to make four samples according to the protocol using two papers at each of the two weights specified in the method. One hundred FCSAs of each sample were smoked and analysed for each phase using the smoking machine normally used in that laboratory.

Each laboratory analysed the samples for NFDPM and nicotine yield. As not all laboratories were able to analyse carbon monoxide, this was not a requirement of the protocol for the study but those laboratories that were able to do so were invited to submit data. The data was analysed on completion of each phase and in total at the completion of the study.

The poster will provide details of the protocol for the study and provide the main data for NFDPM and nicotine with limited data for CO. The conclusions of the study suggest that a higher tolerance will be required for the analysis of FCSAs than for manufactured cigarettes. Nevertheless, the study suggests that the analysis outlined in the ISO/CORESTA method is viable. Proposed values will be presented and discussed.