Canada Border Services Agency | Agence des services frontaliers du Canada

People in the Historical Development of Ion Mobility Spectrometry in Canada

Canada

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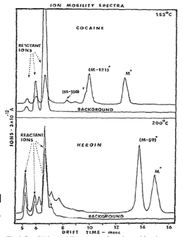
Professor Francis W. Karasek joined University of Waterloo, Ontario, Canada in 1968 and began an ion mobility spectrometry (IMS) research program in 1970, using the Beta VI IMS from Franklin GNO, a family-owned business founded by Henry Gibson. IMS was then known as plasma chromatography. Following Karasek's publications on the Beta VI, businesses of Franklin GNO picked up, with interests from analytical chemists. After Henry Gibson sold the business and Martin Cohen took charge of the company, Franklin GNO was renamed to PCP Inc.
DID YOU KNOW? PCP stands for "Plasma Chromatograph People".

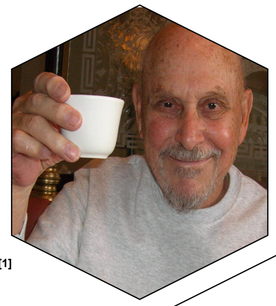
Prof. Karasek advanced IMS into an analytical detection method for trace organic compounds, including drugs, explosives and pesticides. He was the first person to demonstrate the capability of IMS to separate isomeric compounds and to develop mobility-mass correlation curves (known today as "trend lines"). His laboratory produced over 30 master and doctoral graduates in IMS and trace organic analysis. Both Prof. Gary Eiceman and Prof. Herbert Hill completed their post-doctoral fellowships at the Karasek Lab. On a personal note, Prof. Karasek, the father of seven sons, was gentle and sensitive to his students, especially foreign students with different cultures. Prof. Karasek retired from University of Waterloo in 1988 and he often travelled with his wife, Irma. He passed away in 2013.

Above: Howard Kim working on the Beta VI at Karasek's Lab, 1974

Left: Frank and Irma Karasek in Arizona, 2006. Irma passed away in 2012.

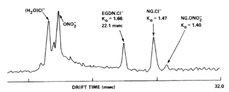


Mass-identified IMS spectra of cocaine and heroin from Beta VI [1]



Lorne Elias is a pioneer in trace explosives detection. After retiring from National Research Council of Canada (NRC), Lorne continued his explosives detection research under contracts with Transport Canada and Federal Aviation Administration. A genius in the laboratory, he built vapour generators (he did the glass blowing) and gas chromatographs (GC) from scratch. He designed and built air sampler, with parts purchased from Radio Shack. He is a strong proponent for standard testing protocols and quality control for evaluation of trace explosives detectors, many of which were based on IMS. Together with **Pavel Neudorfl**, they developed the technique of "Calibrated Explosive Thumbprints", they represented Canada at International Civil Aviation Organization (ICAO) technical meetings and they proposed 2,3-dimethyl-2,3-dinitrobutane (DMNB) to be used as explosive taggant. Lorne retired for a third time in 2011. He lives in Ottawa and is in good health. Pavel studied chloride chemistry and the effect of temperature to improve detection of thermally labile explosives and dinitro compounds by IMS [2,3], stemming from his philosophy on respecting the properties of target molecules (*Note*: Pavel was one of many who worked on dopant chemistry). Pavel dedicated most of his career to explosives detection, at NRC and at the Customs Lab (CBSA). He retired in 2014 and passed away in 2015.

Pavel Neudorfl and Lorne Elias, at Lorne's third retirement party, 2011



Detection of ethylene glycol dinitrate (EGDN) and nitroglycerin (NG) from headspace vapour of dynamite [2]

Right: Using IMS to detect drugs in marine containers (NDTA at Fort Huachuca). Below: an GC-IONSCAN used for drugs detection

André Lawrence worked on applications for IMS at NRC (1982-92) and at the Customs Lab (1992-2005). He published a series of articles on drug detection [4-8] and on wood species identification [9,10], using the PCP Phemto-Chem 100 IMS and the Phemto-Chem MMS-160 IMS-MS. André cherished his work with Pavel in the understanding of the interactions of ions with molecules [2,3], as one contribution to a subject studied by many IMS and MS scientists. **Pierre Pilon** worked on the development of IMS prototypes at the Customs Lab with scientists from Barringer. Before there was verification material, Pierre would check instrument performance using nicotine from cigar fragments. He has fond memories of technical discussions with Frank Kuja and Lucy Danylewych-May. His work also involved improving detection by studying the effects of concentration, desorption temperature and interferences [11], extensive field testing and equipment evaluation [12], including drug vapour detection from cargo containers [13,14]. Both André and Pierre were instrumental in fielding IMS equipment for customs applications. André retired in 2005 and Pierre retired in 2016.

Michel Asselin's group at the Defence Research Establishment Valcartier (now Defence Research and Development Canada, DRDC, Valcartier) was the first government lab in Canada to have an IMS-MS – a PCP Phemto-Chem 100. Lorne Elias and Pavel Neudorfl envied Michel and would drive five hours from Ottawa to Valcartier "*to drool over the IMS-MS*". Michel studied the ionization of ethylene glycol dinitrate (EGDN) vapours from dynamite, the explosive of interest during 1970s, using IMS [15].

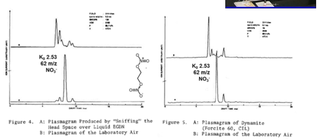
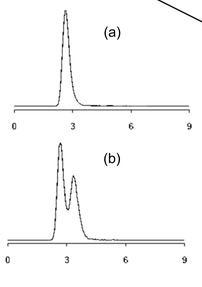


Figure 4. A: Plasmagram Produced by "Sniffing" the Head Space over Liquid EGDN
B: Plasmagram of the Laboratory Air

Figure 1. A: Plasmagram of Dynamite (Forcite 60, CIL)
B: Plasmagram of the Laboratory Air

Paul D'Agostino, DRDC Suffield, uses IMS as one of the tecniques for chemical warfare agent (CWA) detection. A FAIMS-MS method was developed to detect and identify CWA and CWA hydrolysis products in food products [16]. TWIMS-MS/MS (Waters Synapt HDMS) was used to analyze CWA from headspace samples, collected with solid phase microextraction (SPME) fibers [17,18].

Analysis of aqueous extracts from an old chemical rocket (left), suspected to contain sarin, showed no signal for hydrolysis products of sarin (isopropyl methyl phosphonic acid and methylphosphonic acid) using LC-MS; whereas analyses by Synapt HDMS clearly detected both (a) methlphosphonic acid [MH]$^+$ and (b) isopropyl methylphosphonic acid [MH]$^+$.



Paul Kebarle contributed significantly to gas phase ion chemistry used to ionize sample in IMS and chemical ionization mass spectrometry, helping both IMS and mass spectrometry communities in understanding the fundamentals of gas phase reactions [31,32]. His early work on water clustering contributed significantly to fixing operational parameters for IMS early in its development. He considered his paper with Udo Verkerk on electrospray to be his most important work [33,34]. Originally from Bulgaria, he studied mass spectrometry at University of British Columbia, conducted post-doctoral work at the NRC, and he was a professor at University of Alberta from 1958 until retirement.
John A. Stone, Professor Emeritus from Queen's University, Kingston, Ontario, also plays a prominent role in the advancemt of gas-phase ion-molecule chemistry in IMS. He collaborates extensively with the Eiceman research group.

Barringer booth at ISIMS conference in Buxton, England, 1999. From left: Andy Rudolph (Barringer), Jun Xu (Oak Ridge National Laboratory), Steve Rigdon (U.S. Coast Guard)

Barringer Research Ltd., Toronto, Ontario, began reseach on using IMS for trace detection in the 1980s. The IONSCAN 100 was launched in 1990, utilizing dopant chemistry. Then came IONSCAN 250, 350, 400 and the iconic 400B – which became the gold standard in trace explosives and drugs detection. In 2001, Barringer was acquired by **Smiths Detection**. IONSCAN 500DT, with simulateneous trace explosives and drugs detection, was introduced in 2005; the IONSCAN 600, launched in 2015, employs non-radioactive ionization.

Prototype, circa 1986

Model 100, 1990

Model 250, 1991

Model 350, 1993

Model 400, 1995

Model 400B, 1998

Model 500DT, 2005

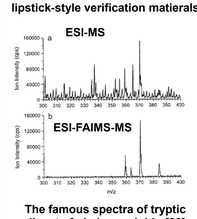
Model 600, 2015

DID YOU KNOW? The afternoon before a ministerial visit to the Customs lab in 1989, the IONSCAN demo unit was not functioning properly. **Lucy Danylewych-May**, a scientist at Barringer, offered to drive the unit to the Toronto Barringer lab, fix it and drive five hours back to Ottawa the following morning. Pierre Pilon was sure he would lose his job. On the morning of the ministerial visit, true to her words, Lucy May delivered a perfectly working IONSCAN. This showcased the dedication and confidence of Barringer scientists in their IMS.

Lucy Danylewych-May invented the lipstick-style verification matierals

Roger Guevremont, receiving the Lossing Award, presented by Michael Siu from the Canadian Society for Mass Spectrometry, 2009

Roger Guevremont started working at NRC after obtaining his PhD in analytical chemistry from University of Alberta. He worked on mass spectrometry at Professor Richard Yost's lab at University of Florida during his sabbatical. He developed Field Asymmetric Ion Mobility Spectrometry (FAIMS) based on differential mobility spectrometry with cylindrical geometry, which was originally developed by the Gorshkov's group at the Institute of Thermo Physics of the Siberian Academy of Science [23] and improved upon by the Field Ion Spectrometry (FIS) team at Mine Safety Applicances (MSA) [24-26]. Matthew Matyjaszczyk recalled handcarrying a FIS instrument to NRC and training Roger. Roger created the term "ionogram" for FAIMS-MS experiments where the compensation voltage is scanned while using the mass spectrometer as a detector [27]. He formed Ionalytics Corporation in 2000 and commercialized FAIMS as an ion filter for mass spectrometers. On July 1, 2005, Roger was hit by a car head-on, while he was riding a motorcycle. Despite having lost a leg and much memory, he has conducted an active life of skiing, horse-riding, parasailing, swimming, scuba diving, and of course, painting. Ionalytics was acquired by Thermo Electron in August 2005.

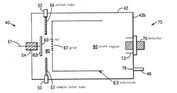


The famous spectra of tryptic digest of pig hemoglobin [28]

Many scientists worked with Roger Guevremont in the development of FAIMS, including Matthew Matyjaszczyk, Randy Purves and Govindanunny (Unny) Thekkadath. Randy and Unny were also part of the Ionalytics team. Matthew, who previously worked at Barringer (IONSCAN) and MSA (FIS), continued to work on trace explosives detection at Implant Sciences (Quantum Sniffer), Transportation Security Laboratory, and is now consulting on IMS, FAIMS and on FIS-based breathalyzers. Randy is a professional research associate at the University of Saskatchewan. His recent project uses FAIMS-MS in the analysis of medically relevant compounds in faba bean seeds [29]. Unny worked on IMS development at CPAD, IDS and Scintrex Trace. He is the inventor of the "Cup" IMS [30], which uses solely geometry to establish the electric field. He is currently a Senior Technology Adviser at the Canadian Air Transport Security Authority.

Randy Purves

Unny Thekkadath



Electric field established by geometry [30]

In 1986, Colin D. Corrigan founded **CPAD Holdings**, developing detection technology for explosives and landmine detection. Mariusz Rybak acquired CPAD in 1995 and renamed it **Intelligent Detection Systems** (IDS); IDS acquired **Scintrex Limited** in 1998. CPAD and Scintrex Limited were acquired by Control Screening (now **Autoclear**) in 2001. CPAD/ IDS/ Scintrex developed and produced GC-IMS based explosives and drugs detectors – the CPAD EDSS General Purpose Scanner, the IDS Orion, the IDS Northstar, and the current E5000 and EN3300.

IDS Orion

CPAD EDSS General Purpose Scanner

IDS Northstar

E5000

Corrigan

EN3300

Rybak

Colin Corrigan was born in Northy Bay, Ontario. He studied engineering, business and mathematics. His career began as a Merchant Marine, and eventually became an entrepreneur, founding CPAD Holdings. He passed away in 2010.

Mariusz Rybak emigrated to Canada from Poland in early 1980s. He was a visiting professor in environmental sciences at Brock University in St. Catharines, Ontario. He left IDS in 2000.

Pylon Electronics Inc. developed IMS-based portable drug detector, from 1986 to 1988, under a contract with NRC (Lorne Elias and André Lawrence). Additional funding was also obtained from the Ontario Technology Fund to commercialize IMS for drugs and explosives detection. Five different IMS designs were built: stacked ring designs, glass and/or ceramic tubes with either resistive deposits or metal electrodes located on the external tube surfaces [32,33]. Pylon's IMS was to provide a quantitative capability to complement other GC detectors. This quantitative aspect for multiple target ions during a GC run was presented at the 1992 ISIMS conference [34]. The IMS project was discontinued around 1993 due to lack of commercial interests. Today, Pylon Electronics provides repair and calibration services for test and measurement instruments [35].

Teknoscan Systems Inc., located in Vaughan, Ontario, was founded in 2008 by Sabatino Nacson, who previously worked at Barringer and Smiths Detection. Teknoscan manufactures benchtop GC-IMS system, mostly for explosives and drugs applications.

SCIEX, a mass spectrometry company founded by Barry French and others from University of Toronto in 1970, collaborated with Sionex to develop a planar DMS-MS in 2007. Similar to the development pathway of FAIMS, DMS with planar geometry also originated from the Soviet Union [36,37]. SelexION was commercialized in 2011, where the addition of DMS adds the capability of ion-filtering to improve signal-to-noise ratio and instrument selectivity to MS. Polar modifiers were introduced in 2012 to increase DMS resolution – harnessing the separation capacity of the technique by clustering with neutral molecule in low field conditions and declustering in high field conditions [38,39]. SCIEX is based in Concord, Ontario.

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