PROJECT NO: FP6-508107

BEN-DET

AIR POLLUTION AND WORKER EXPOSURE BENZENE SPECIFIC GAS ANALYSIS AND MEASUREMENT INSTRUMENT- BEN-DET

Co-operative Research (Craft)

Horizontal Research Activities Involving SMEs

Publishable Final Activity Report

Date of issue of this report: Jan 2007

Start Date: 1st July 2004 Duration: 30 Months

Lead Contractor: Ion Science Ltd Version 01

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Project Information

PROJECT NO: FP6-508107107

CONTRACT NO: COOP-CT-2003-508107

TITLE OF PROJECT*:* AIR POLLUTION AND WORKER EXPOSURE BENZENE SPECIFIC GAS ANALYSIS AND MEASUREMENT INSTRUMENT- BEN-DET

COORDINATOR: Ion Science Ltd

SME EXPLOITATION MANAGER: Ion Science Ltd

***SME CONTRACTORS*:**
1 Ion Science Ltd
2 Braun Formenbau Gmbh
3 SRA Instruments
4 Sarantel
5 VIPEM Hackert
9 Euro-Index b.v.

***OTHER ENTERPRISE / END USER CONTRACTORS*:**
6 Shell International Bv

RTD PERFORMER CONTRACTORS:
7 Pera Innovation Ltd
8 Fraunhofer (ISC & IZM)

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Executive summary

This report covers the work carried out in the EC FP6 CRAFT project BEN-DET. The overall industrial objective of the project is to develop a hand-held, benzene specific, integrated measurement instrument based on a state of the art PID detector in combination with novel/innovative inert polymer-based micro, pre-concentrator and GC column. The micro GC column will be capable of selection and separation of benzene from other similar species hydrocarbons. The system will also include an intrinsically safe GPS tracking & Telecommunications module, along with the required pneumatic and electrical/electronic circuitry and an intrinsically safe rechargeable battery power source.

This proposal aims to improve the quality of life of all EU citizens through the advances in the control of benzene emissions which depend upon accurate detection and recording of emission levels in real time with mobile communication technology to ensure greater health, safety and ethics. This will significantly contribute towards reducing the reported deaths and injuries caused by benzene emissions every year in the workplace alone.

The technical work over the reporting period (1st July 2004 – – 31st Dec 2007) has been spread over the tasks in Work Packages 1, 2, 3 4 & 5: -

WP 1:	Extend S&T knowledge of Materials Science and Heating Technology
WP 2:	Proof of Principle of Resistive Element Heated Gas Chromatograph
WP 3:	Proof of Principle of Micro Capillary Tube Manufacture
WP 4:	Production and Testing of Proof of Principle Pneumatic Circuit, Micro Injector Valve, Activated Carbon Filter, Telemetry Module & Antennas & PID Sensor + Power requirements & specification of rechargeable battery
WP 5:	Integration of Individual Elements to Produce Prototype Benzene Specific Analysis and Measurement Instrument

Project Management, Co-ordination and Clustering and Dissemination & Implementation have been be on-going throughout the life of the project. The ‘Kick-off’ meeting was held at Ion Science on 26th August 2004, where the project goals, work plan and initial actions were successfully presented and agreed upon. During the duration of the project meetings were held approximately every 3 months hosted by various partners reviewing and carrying out trials for their relevant tasks

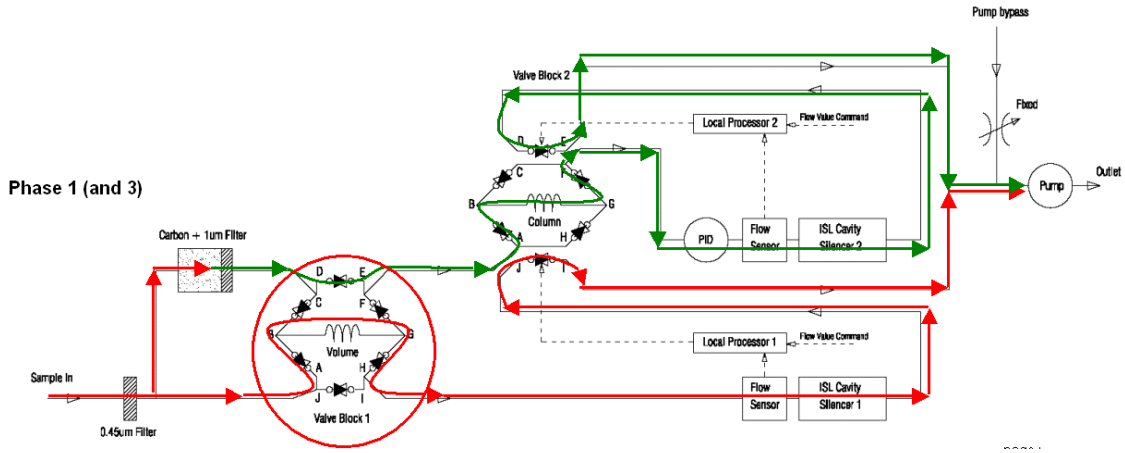
Several polymers were investigated for suitability as a moulding grade with high flow to mould 200µm features and a film suitable for hot embossing. It will be treated with solgel chemistry and used must have some surface polarity, also partial solubility of polymer can increase adhesion.

A thermal model of the heater system using thin film heater technology was defined. The resistance required for the copper foil was calculated and a lab based model trialled. Using hot copper foil embossing (MID) technology successful trials were carried out using 50µm copper foil on samples of the selected polymer films. An initial concept for a 2 dimensional heater pattern has now been developed with tracks of 200µm and when completed will be tooled and tested. A cost efficient method to measuring the temperature without using temperature sensors and additional circuitry has

been developed by Ion Science

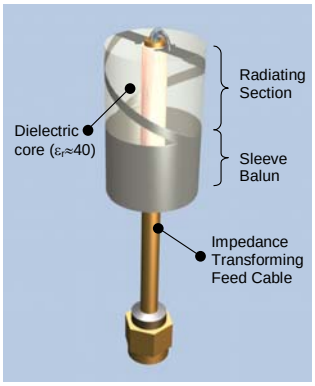
Initial chromatography trials from SRA have been carried out using different columns and Ion Science's modified prototype PID. The results showed excellent analytical data, however there are concerns regarding achieving those temperatures and flows in the BEN-DET device. For the separation of BTEX molecules stationary phases of PDMS type were synthesized via a sol-gel process as adherent coatings on polymeric substrates or as powders for filling steel tubes (packed column). Experiments at Ion Science with a short packed column showed a good chromatographic separation of benzene and toluene at ambient temperatures, initial field trials to be arranged at Shell.

A polymer column design was developed using fine moulded features on CAD following the column characteristics researched from Ion. The design was developed to fit with the same envelope as the initial design 30x10x3mm. Taking the best theoretical design as a basis the column layout was re-designed to give the best possible packing efficiency while also being possible to tool and manufacture. A CAD model was successfully developed of the mould tool insert and the tool was produced using spark erode technique. Sample polymer column have been made and sealing and pressure and flow tests successfully investigated.



A concept design of the pneumatic logic and specify control elements required to route the gas sample and carrier gas between the carbon filter, micro injector valve, pre-concentrator and GC column has been developed. Flow diagrams have been produce showing the flow paths required for each stage of the operation of the instrument detailing interconnections between the sub-components. Successful trials have been carried out using IZM micro-injection valve with high and low flows. New low flow valves are to be made using Fraunhofer's clean room fabrication process and Individual prototype housings to test the new valves are being developed. The construction and fabrication of the pneumatic manifold which will carry all fluidic and most electronic components of the system is currently being developed and use fine moulded features technology.

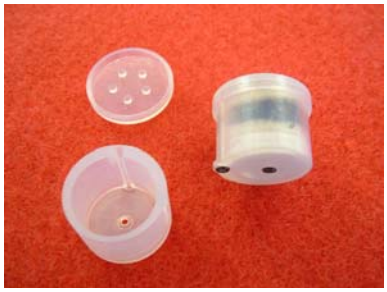
The concept design for the telemetry module is being developed around a 16 bit processor for control. Trials using Sarantels GeoHelix GPS Antenna and Wifi/Bluetooth antennas have shown excellent performance when combining a GPS system and a Wifi/Bluetooth communication device into a compact device.



Experiments at ION SCIENCE with a short (some cm) commercially available packed column (stationary phase: TCEP on Chromosorb PAW) showed a good chromatographic separation of benzene and toluene at ambient temperatures. The stationary phase was characterising by some measurements. A sol-gel preparation procedure of this stationary phase should give further possibilities for modifications and applications as film or coating on diverse substrates (polymers, steel tubes etc.). This was done via hydrolysation and condensation (catalyzed by hydrochloric acid) of tetramethoxysilane (TMS) in a common solution with TCEP in methanol (gel entrapment). After gelation the product was dried and cured up to 120 °C to glassy granules, which may be used for production of a packed column.

New low flow valves were made using a clean room fabrication process based on silicon micro machining by wet chemical deep etching (KOH) as well as reactive ion etching (RIE) and silicon fusion bonding. Individual prototype housings to test the new valves have been developed, IZM assembled the ceramic plates and NF8-valves which were connected by wire bond, and the surface and bond wires were passivated with silicone and successful trials were carried out.

A Micro GC Column was developed using the stainless steel tube which was pre-formed into a spiral pattern and over moulded with the thermoplastic compound using a prototype mould tool. The top edge of the over-moulding has a radius feature to facilitate the application of a self adhesive Thermofoil Heater circuit which will be initially used to test thermal requirements.



An activated carbon filter has been designed to enable any type of activated carbon which is an ideal material to scrub air for use as a carrier gas in packed column gas chromatography. For the prototype filters the outer casing and cover was produced using SLA rapid prototyping.

A pneumatic assembly that as two main parts of circuit sampling flow circuit and probe flow circuit with different flow rate and leak flow requirements two different valve types and two flow different sensors which also integrates the prototype Micro GC Column, Active Carbon Filter and PID sensor has been designed and prototyped. The assembly needed to be as compact as possible and able to be integrated to the BenDet control circuitry which will be housed in the BenDet working device. The concept of a small thin manifold component was developed which included a series of micro channels where the sample gasses would flow on the top side and be controlled by the micro valves and micro flow sensors which would be fitted to the underneath side, each with a ceramic plate to provide electrical connections. The prototype Micro GC Column, Active Carbon Filter and PID sensor would be fitted to the manifold component securely but not a permanent fitting as these are serviceable components. Micro-machining technology was used to produce the micro channels and features in the prototype manifold component. The low flow valves that were specifically fabricated for the BEN-DET device were fitted and sealed accurately in place and wire boned to their contact ceramic plates. After assembly all the devices were successfully electrically tested (valves: capacitance and motion sound, sensors: resistivity).

The control system has been developed which will be a fully integrated prototype capable of controlling all the critical elements of the instrument and operate the device during the validation trials. The device will be 'bluetoothed' for all communication and IRDA and USB may be designed into the base for direct PC communication. The control PCB will be a serviceable part along with the PID sensor, carbon filters, GC columns, pump and manifold components.

Initial field trials were carried out at Shell Pernis/Moerdijk using the BEN-DET: 1st prototype to test the initial proof of principle. These results provided much important data and good results. The next prototype will include the new mini PID sensor and prototype pneumatic components which will be included in the BEN-DET device.

The development of a concept symmetrical case design has been completed that is small and light, able to be easily held with either the left or right hand or worn in a top pocket, which may use a joystick with soft menu functionality with space for warning LED's and sounders. The device would last for at least 10hrs, to satisfy SHELL's requirement and will include the largest acceptable display. A SLA rapid prototype model has been produced to assemble the individual elements of the system and the detector has been successfully evaluated. This will be used to carry out the final validation trials and field tests.

There has been much progress achieved in the development of the various sub-components of the BEN-DET device which has overcome many technical complex challenges and third party supply problems. The consortium have reviewed the actions required to get the development to the pre-production phase. An action plan to build and trial between 5 and 10 prototypes by June 2007 was agreed and approved by all:

A PID detector has been designed that has a large signal to noise ratio in a clean and stable environment, which is a more sensitive detector. The minimum and maximum flow requirements have been determined through the detector to achieve the best possible signal to noise ratio. The whole detector package has been redesigned with consideration for 'Designed for Manufacture'. This module has high voltages generated to excite the lamp and polarise the electrodes within the cell. The design is fully compliant with the European ATEX safety standards for instruments to be used within hazardous gaseous environments.

The development of the BenDet Mini PID detector has now been complete; fully researched and tested and trials by the partners have been very successful. The results prove that the target levels of detection have been achieved.

Project Goals

- To reach 10 ppb of Isobutylene at real time measurement
- To put this in a Series 4 mini-body
- Fully ATEX and IECEx approved
- Easily serviceable parts
- Operate under Low Power 3V and 30mA
- Noise equivalent to 10ppb
- 4.5 decade Linearity 10ppb to 500pp To become the BenDet detector

Technology

- Patented Detected Cell
- Gives status of contamination
- Pneumatic sealing
- Used at non atmospheric pressures

- Unique Lamp Drive circuit
- Will always power up and find resonance under any environmental conditions

The BenDet Mini PID detector is now available for initial production volumes.

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Overview of Project Objective

The overall project objective is the development of a hand-held, benzene specific, integrated measurement instrument based on a state of the art PID detector in combination with novel/innovative inert polymer-based micro, pre-concentrator and GC column where the main areas of required are:

- The micro GC column will be capable of selection and separation of benzene from other similar species hydrocarbons.
- An intrinsically safe GPS tracking & Telecommunications module that can automatically record and encrypt the exact latitude and longitude of the sampling location using Assisted GPS.
- Pneumatic and electrical/electronic circuitry and an intrinsically safe rechargeable battery power source.

The key operation targets for the system have been reviewed periodically through-out the project and the final specification is agreed as below:

- Manufacturing cost less than €2250
- Minimum detecting limit - 0.1ppm (this is an improvement on current legislation and will become a new milestone for future measurements)
- Selectivity – must be capable of separating BTEX group of VOC's containing Benzene, Toluene, Ethylbenzene, m-Xylene, p-Xylene and o-Xylene. ? Other volatiles
- Resolution – minimum detection limit
- Repeatability – +/- 5% of reading.
- Positional data - accurate to 10 meters
- Thermal control of micro GC column & pre-concentrator +/- 1 C
- Response time - Total time for measurement – near real time near < 3 minutes aim to nearer 1 minute
- Maintenance – PID lamp to operate for one month without cleaning whilst detecting BTEX
- Compliant to ATEX Directive 94/9/EC (category II 2 G EEx ib) concerning equipment/protective systems intended for use in potentially explosive atmospheres

Relation to Current "State of the Art

Measuring benzene in the atmosphere is currently done using a pumped sampling method on a sorbent cartridge followed by a gas chromatographic determination in a laboratory. It should be noted that the current directive does not specify the detection type. The most common methods of analysis are GC/PID, GC/FID (Flame Ionization Detection) & GC/MS. Other methods that are used for measurement & analysis are:

- Colormetric Gas Detection Tubes:
- Chip Measurement Systems;
- Catalytic Sensors;
- Thermal Conductivity Gas Detectors (TCD);
- Electrochemical Detectors;
- Semiconductor/Solid State/Metal Oxide Sensors (MOS);
- Surface Acoustic Waves Gas Sensors (SAWS);
- Infra-red Detectors;
- Ultraviolet/Visible Detectors (IR);
- Photo Ionisation Detectors (PID);
- Flame Ionisation Detectors (FID);
- Ion Mobility Spectrometry (IMS);
- Mass Spectrometry (MS).

The potential of these technologies were highlighted as a possible basis for a portable or hand held benzene concentration in air measurement system in combination with GC. Only PID was considered for use in development of the new technology, as the other methods sensitivity capability was greater than the required resolution. Also the ability of the methods to be an ignition source in potentially explosive situations was a major concern. An overriding concern was the cost of the instruments with many being greater than €5200, which makes them expensive/inaccessible to less wealthy companies.

While being state of the art, many of the technologies are either not available or still emerging. Current methods for detecting benzene levels are time consuming & expensive. Many cannot be used in potentially explosive situations without endangering lives, thus the negative aspects have led to scientific research/development into other techniques, but specifically into making technologies portable, hand held & cost effective.

Thus the development of a hand-held, benzene specific, integrated measurement instrument based on a state of the art PID detector in combination with novel/innovative inert polymer-based micro, pre-concentrator and GC column.

- The micro GC column will be capable of selection and separation of benzene from other similar species hydrocarbons.
- An intrinsically safe GPS tracking & Telecommunications module that can automatically record and encrypt the exact latitude and longitude of the sampling location using Assisted GPS.
- Pneumatic and electrical/electronic circuitry and an intrinsically safe rechargeable battery power source.

Final Plan for Using and Disseminating the Knowledge

See Deliverable D47: Dissemination and User Plan