LABORATORY GENERATED BITUMEN FUMES UNDER STANDARDIZED CONDITIONS AND EMISSION LEVELS FOR VOC, SEMI-VOLATILE AND PARTICULATE PAH AND PASH. ION TRAP GC-MS ANALYTICAL METHOD DEVELOPMENT AND CLEAN-UP SCHEME

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Concern regarding the hazardous health effects from exposure to chemicals in bitumen fumes has been expressed and there are a great deal of interests for data on profile of individual compounds in the gas and particulate phases. The purpose of the study was to evaluate a method for the determination of volatile organic compounds (VOC), semi-volatile and particulate polycyclic aromatic compounds (PAH) and sulfur polyheterocylic (PASH) compounds in bitumen fumes. The methylated derivatives of PAH are of particular interest since many of them exhibit carcinogenic activities while the parent compound does not. Bitumen fumes were laboratory generated at 170°C using a fume generator with controlled characteristics. Sampling of bitumen fumes was carried out with XAD-2 Orbo tubes for the gas phase chemicals and semi-volatile PAH or PASH. Glass Fiber GF/B were used to sample the fumes aerosol particulates at a flow rate of 1.2 L/min

PAH profiles and PASH in bitumen fumes were determined by GC-MS further to a multi-step sample treatment and automatic clean-up procedure. The internal standard was added to 0.1 to 0.3 g of raw bitumen or bitumen fumes filter samples which were then extracted twice with 25 mL toluene by sonication for 10 min each. The reduced extract was partitioned between cyclohexane and dimethylformamide before elution on a SiO₂ micro column. The eluent was reduced and fractionated by HPLC. The PAH fraction of interest which also contained PASH was separated by capillary column GC-ion trap MS using the single ion storage mode for optimized sensitivity. Quantification was made relative to the respective calibration curves between compounds of interest and the internal standard.

The semi-volatile SVPAH and SVPASH were determined by separate desorption of the two distinct parts of the XAD-2 by 5mL CS₂ and sonication for 30 min. Microliters of the combined filtered extract were then injected into a GC column and quantified by ion trap MS. Standard reference compounds were used for comparisons of respective spectra.

Summary of results

VOC: Aliphatic alkanes from C6 to C17 amount to several hundreds mg per equivalent gram of particulate fumes collected on the filter. Most of the other volatile present are benzene derivatives and total VOC amounts to 42 mg/g of fumes. Surprisingly, no benzene was detected in all the samples.

PAH: Methylnaphthalene substitutes are quantitatively predominant. The well known trend is observed with light semi-volatile PAH found mainly in the gas phase. The trend is being reversed in the particulate phase from the methylphenanthrene and the four ring PAH to heavier molecular weight PAH. The naphthalene and its derivatives account for 93% of the total (both gas and particulate) PAH from 2 to 7 ring PAH.

Methylchrysene derivatives are at higher levels than benzo[a]pyrene (BaP) in the fumes. The 6-7 rings PAH are not detected.

PASH: The carcinogenic benzo[b]naphtho[2,1-d]thiophene is found in the fumes in higher amount than its two other isomers but less than the predominant dibenzothiophene. The sum dibenzonaphthothiophenes represents 16% of PASH analysed in the filter, gas phase not included.

In conclusion, our analytical procedure is valid and robust enough for the determination of PASH and PAH in bitumen fumes with a relatively low uncertainty (<35% for light PAH and < 15% for higher MW PAH. New data are now available with the numerous methylnaphthalene derivatives and methylchrysene derivatives. VOC data and profile may be of interest for the interpretation of irritation effect The data may be useful for emission modelling purposes.

Keywords: Bitumen fumes, volatile organic compounds, polycyclic aromatic hydrocarbons, sulfur polyheterocyclic compounds, analysis.





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