ZAVOD ZA JAVNO ZDRAVSTVO
DR. ANDRIJA ŠTAMPAR
INSTITUTE OF PUBLIC HEALTH

Division of Environmental
and Occupational Health

Department of Environmental Protection and Health Ecology

Zagreb, August 29th 2014

Division of Environmental and Occupational Health

REPORT

on examination of one crude mineral oil sample and two soil samples after treatment with remediation agent for refinery and chemical waste

Sampling

As arranged on June 10th 2014 (with Mr. I. Rogan) one sample of crude mineral oil and two soil samples have been submitted for analysis July 2nd 2014, after treatment with remediation agent for refinery and chemical waste labeled as BCP35S / BioSurf.

Sample preparation and recording

All samples were extracted with an organic solvent (dichlorometane) and analyzed in the combined system of gas chromatograph - mass spectrometer (GC-MS system) in order to identify all mineral oil constituents and micro-organic contaminants present in treated soil samples.

A sample of crude mineral oil was prepared for GC-MS analysis by direct dissolution in dichloromethane, while the extracts of treated soil samples were prepared using ultrasonic bath and evaporation system under nitrogen stream.

Analysis was performed by direct injection of sample extracts into gas chromatograph. Standard conditions of temperature programming were: 5 min 40°C/10°C per minute/280°C 21 min, applied ionization method used electron impact energy of 70 eV. Full mass spectra of the separated compounds were recorded in the range from 45 up to 450 Da.

Identification of micro-organic contaminants present was conducted by comparing the full mass spectra with the mass spectra of known compounds from available databases (similarity index SI $\geq$ 85, Wiley, NIST, PMW databases). The presence of components from the group of linear and branched saturated hydrocarbons and the components from the group of lower substituted benzenes (group of BTEX: benzene, toluene, ethyl benzene and xylenes), C3 and C4 substituents was especially tested.

Analysis results

All of the recorded characteristic profiles for all samples were reviewed and verified and the identifications of the present components were made.
In the crude mineral oil sample used for contamination of soil samples, linear and branched hydrocarbons with C10 to C56 carbon atoms in the molecule dominated, with approximate share of 93%. Identified substances from the BTEX group (benzene, toluene, ethyl benzene and xylenes) were determinated with approximate share of 3.5%, further, identified C3-benzene substituents comprised approximately 2.5% and identified contents of the group C4 substituents of benzene made the remaining 1% in the total mass of raw mineral oil (Appendix 1).

After the review of the profiles of the recorded soil sample extracts, treated with the remediation agent, none of the extract samples contained ingredients identified like those in the analyzed solution of crude mineral oil (Appendix 2 and 3). Although, the presence of only traces of some crude mineral oil components was indicated. This may be the result of used solvent contamination.

Conclusion

Test results indicates a very high efficiency of remediation agent for refinery and chemical waste (labeled BCP35S / BioSurf).

Appendix

Characteristic profiles of TIC - chromatogram with mass fragments for a group of alkanes and for the groups of lower benzene substituents.

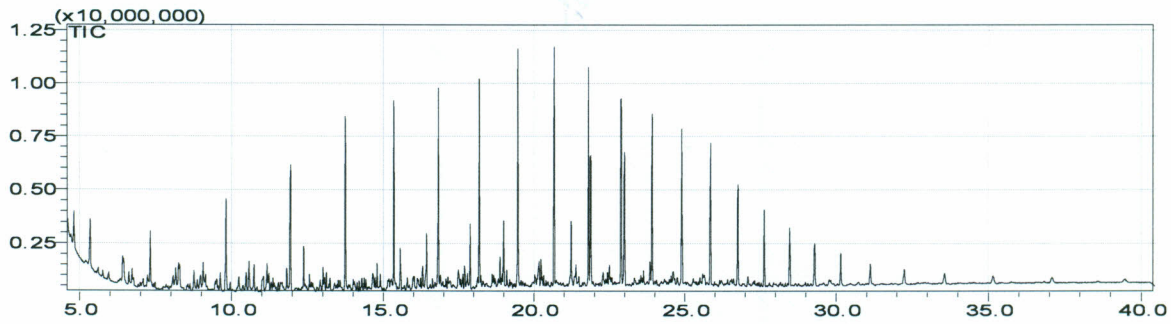
Head of Division:

Professor Zdenko Šmit, Ph. D., B. Sc.

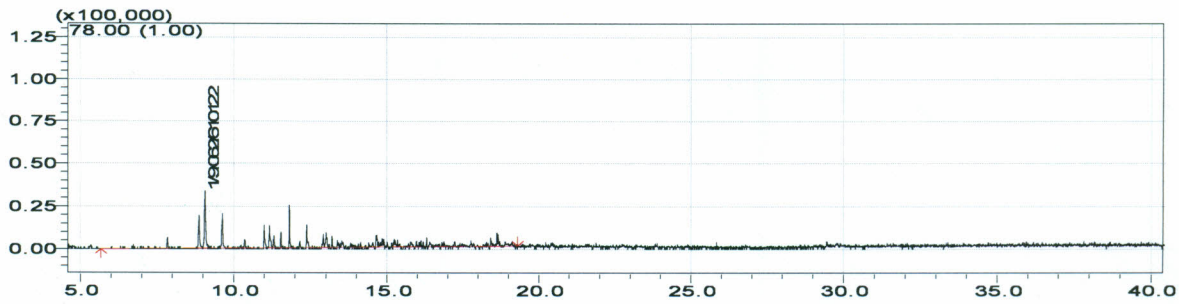
Appendix 1. **Waste Abr.346std1 - btex** 1uL/10mL = 20 mg = 2ug (= 30min 50°C)

40°C 5'/10°C/ 280°C 21'_Pc=1,45 mLHe/min _Dv=1,45 kV BTEX3start=4,6'

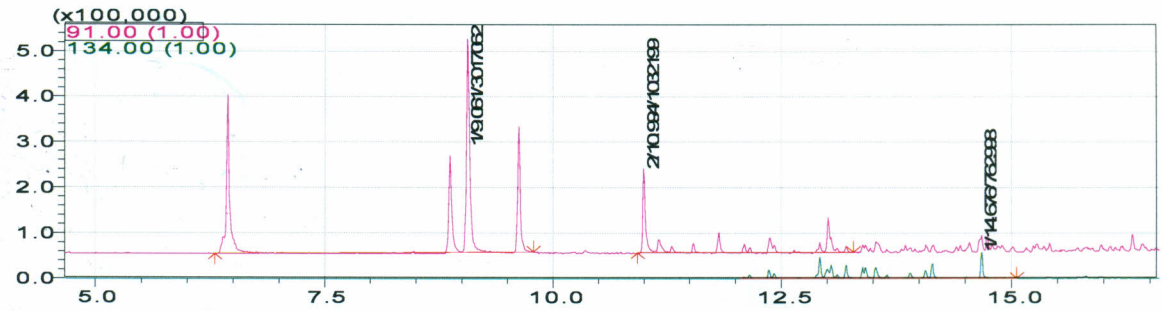
T I C



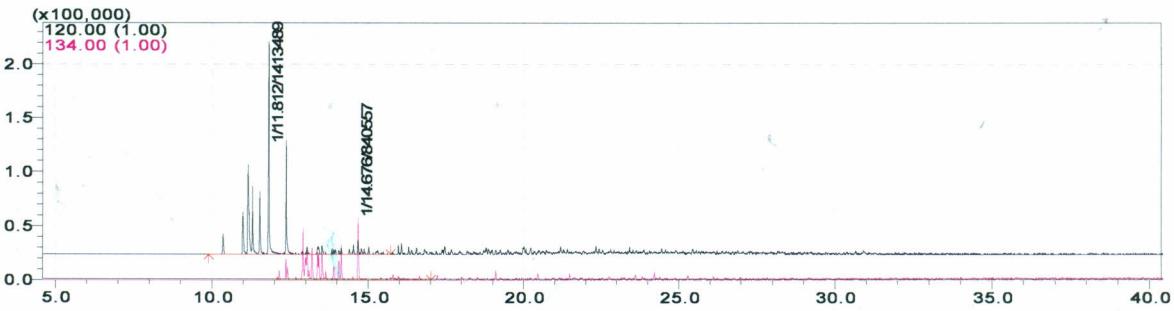
m/z 78 PK = 610



m/z 91 Pk = 3017 + 1032 m/z 134 Pk= 763



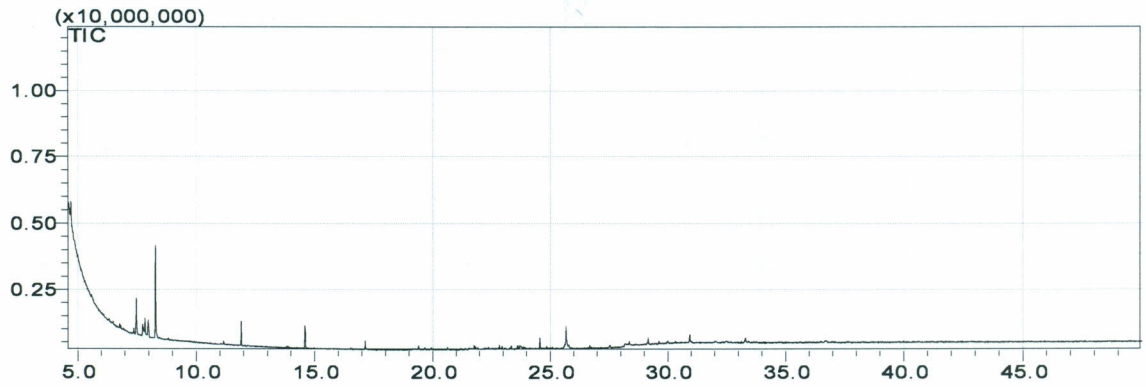
m/z 120 Pk = 1413 i m/z 134 Pk = 841



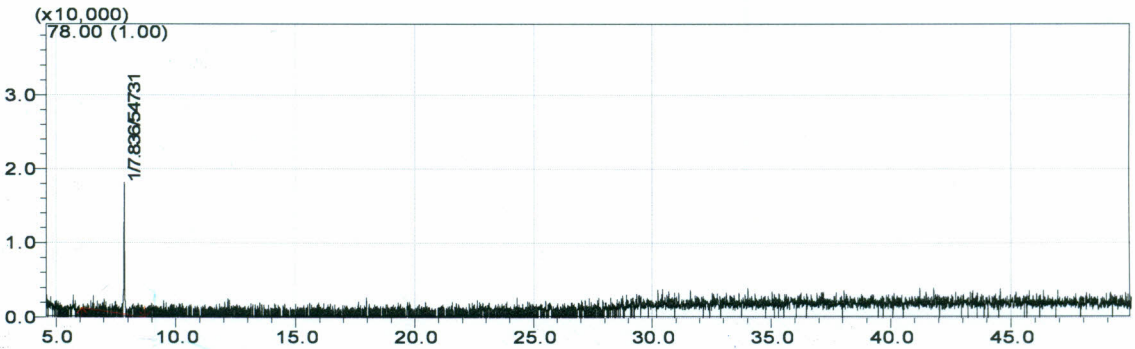
Appendix 2. Waste - soil Abr.347 - btex 1uL/10mL = o,5g

40°C 5'/10°C/ 280°C 21'_Pc=1,45 mLHe/min _Dv=1,45 kV BTEX3start=4,6'

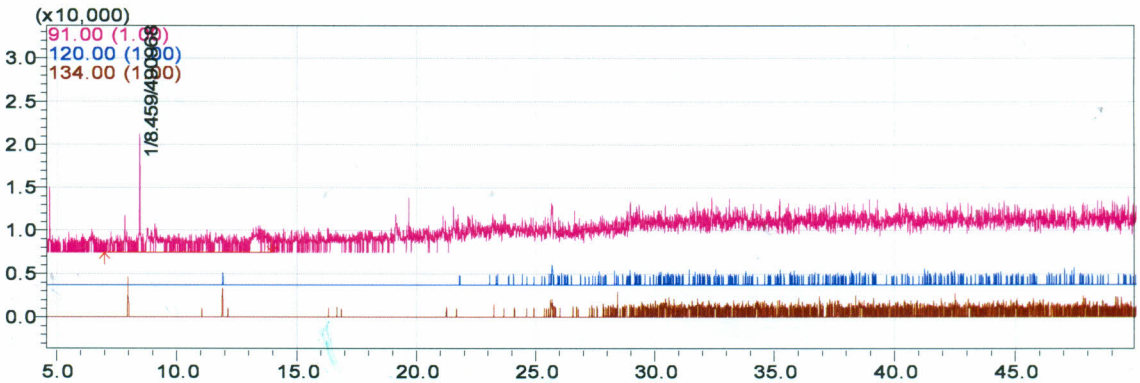
T I C



m/z 78 Pk = 54,7



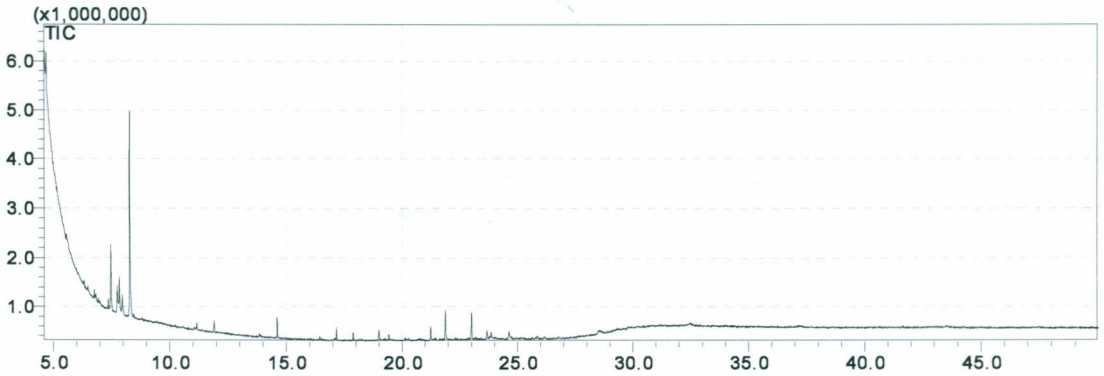
m/z 91 Pk = 491, m/z 120 Pk = ND m/z 134 Pk = ND



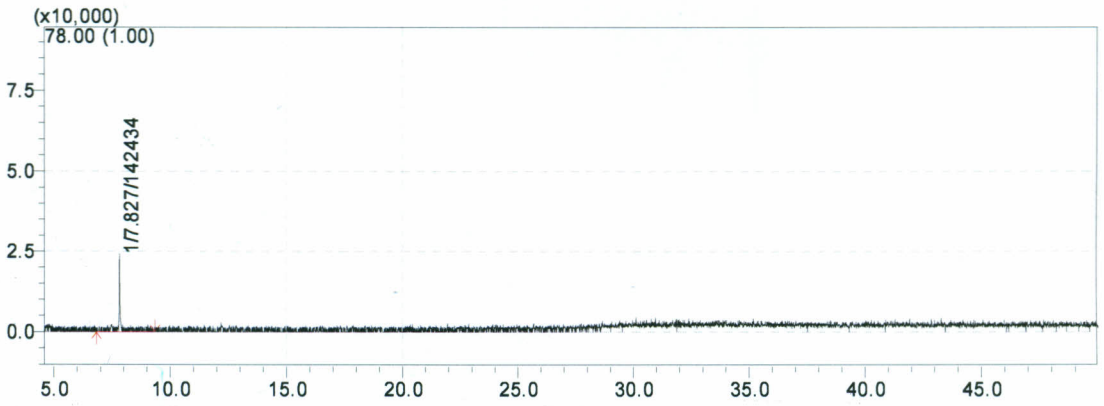
Appendix 3. Waste - soil Abr.348 - btex 1uL/10mL = 1,0g

40°C 5'/10°C/ 280°C 21'_Pc=1,45 mLHe/min _Dv=1,45 kV BTEX3start=4,6'

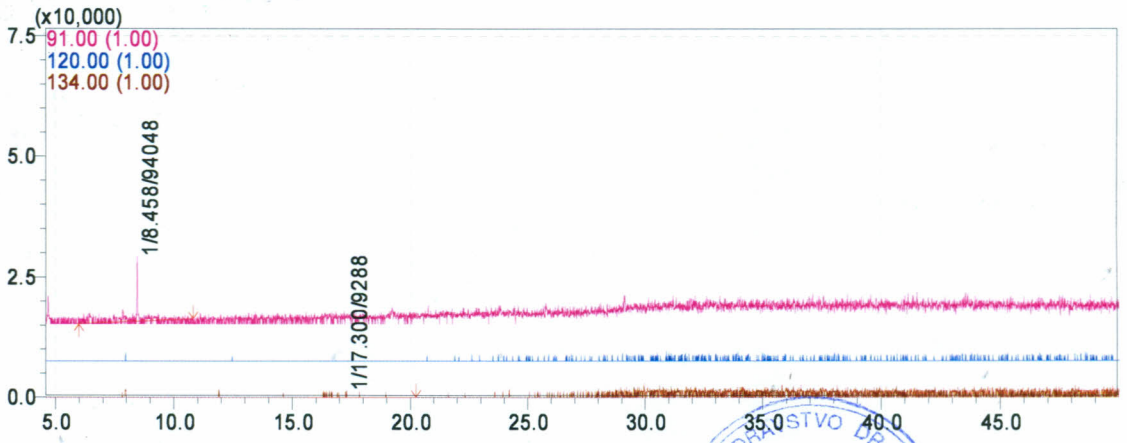
T I C



m/z 78 Pk = 142



m/z 91 Pk = 94 m/z 120 Pk = ND m/z 134 Pk = 9,3



Head of Division:

Professor Zdenko Šmit, Ph. D., B. Sc.