Executive Environment Agency, Ministry of Environment and Water, Bulgaria

Analytical problems by the determination of silt, clay and total phosphorus of soil samples

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Meeting of the Heads of the Laboratories
12.-13. October 2009 in Warsaw

Quantitative determination of soil fractions:
sand 2,00 - 0,063 mm

- Various sieves with different pore sizes
- Sieving by hand

Problem

- Big amount of contents clay and silt was determined as sand

Solution

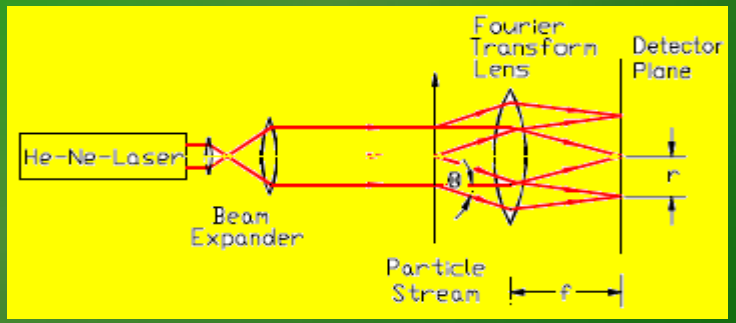
➢ Boiling and subsequent washing of the sample

Quantitative determination of soil fractions:

clay ≤ 2 µm

silt ≤ 2 - 63 µm in samples A,B and C

- **laser diffraction - equivalent to the conventional sedimentation method for analysis of silt and clay – ISO 11277**
- **The sample is analysed in form of soil suspension**



Problem:

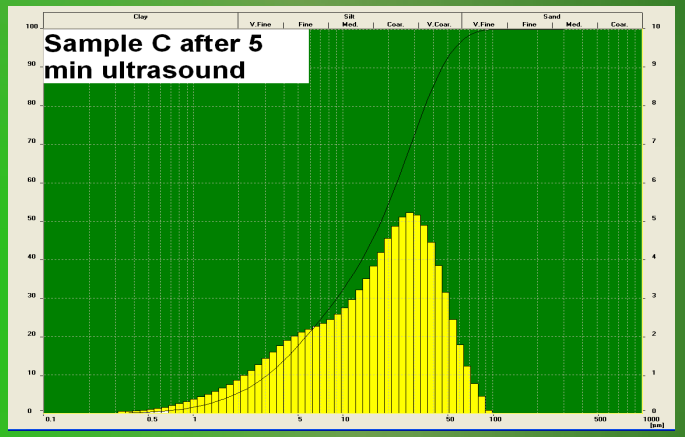
- **Instability of the sample**

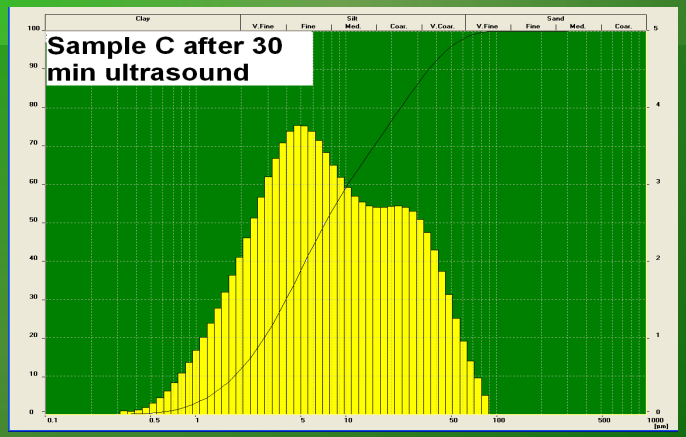
Reason:

- **Dispersion time is too short**
- **Sample is agglomerated**

Difficulty to disperse the samples

- **adhesion or cohesion forces – samples tend to stick**
- **magnetism**
- **coagulation…**





Solution for the problem:

- **Longer ultrasound**
- **The surface forces can be reduced by adding a dispersion agent like sodium diphosphate (sodium pyrophosphate: $Na_4P_2O_7$) or polysalts in concentrations of approx. 1%**

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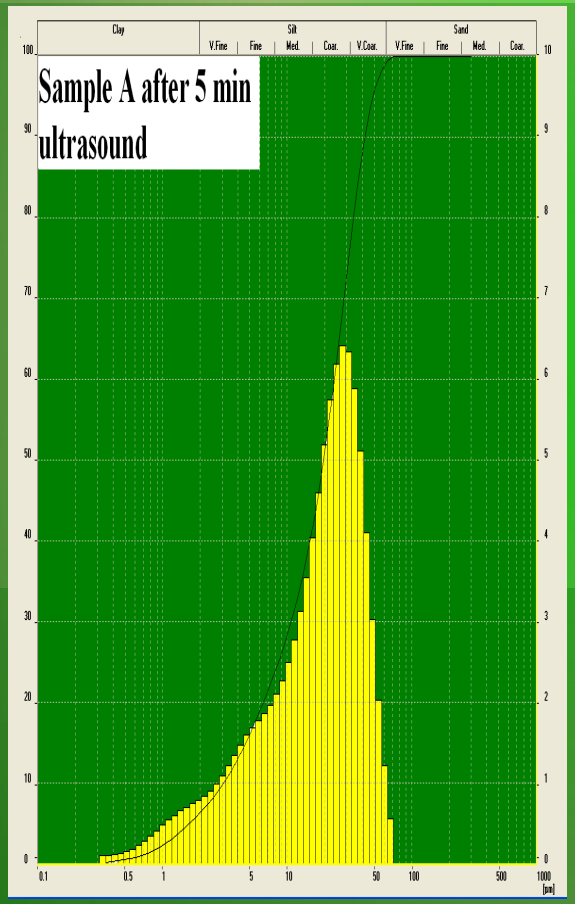
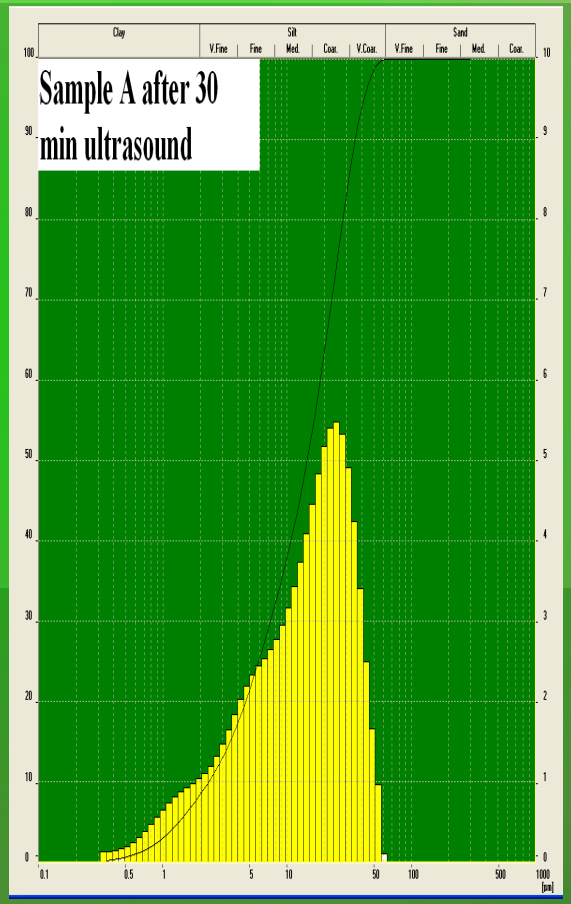
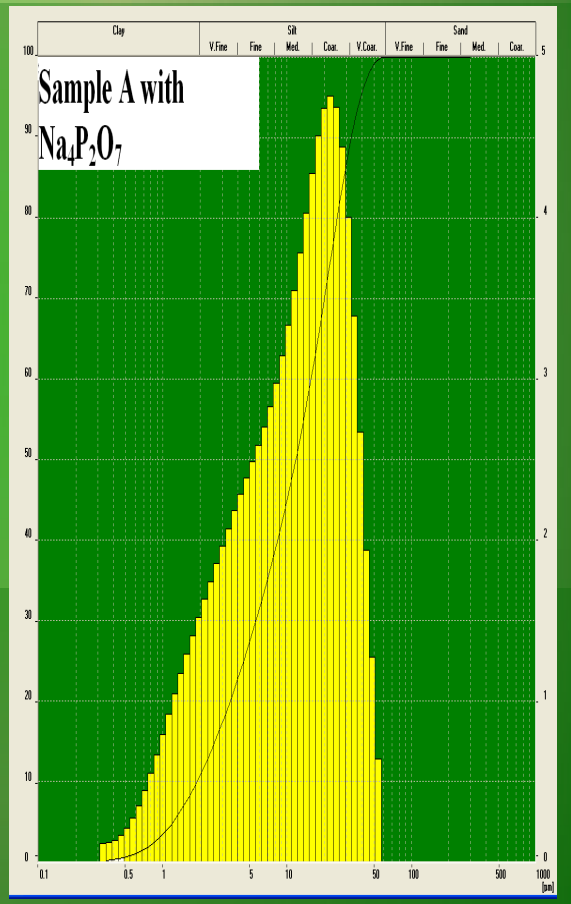
Distribution of soil fraction of sample A







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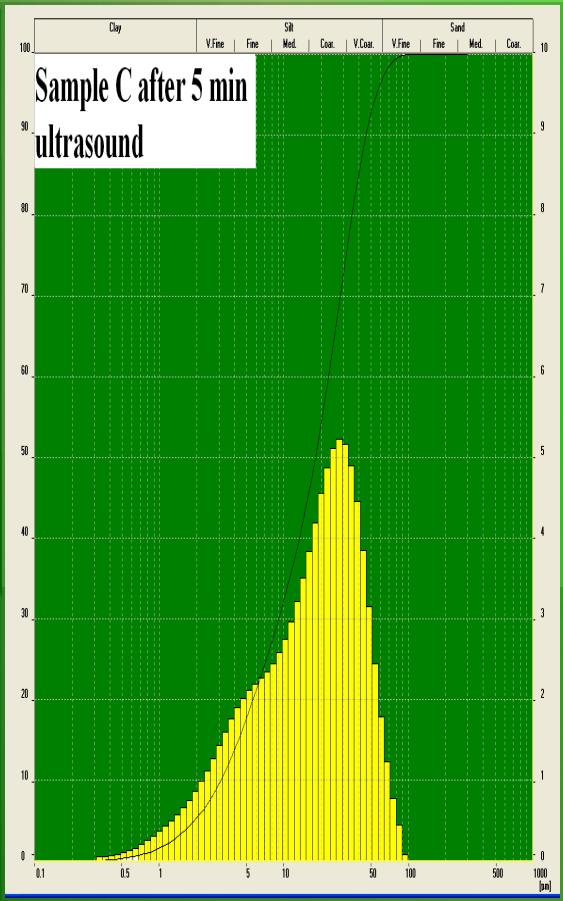
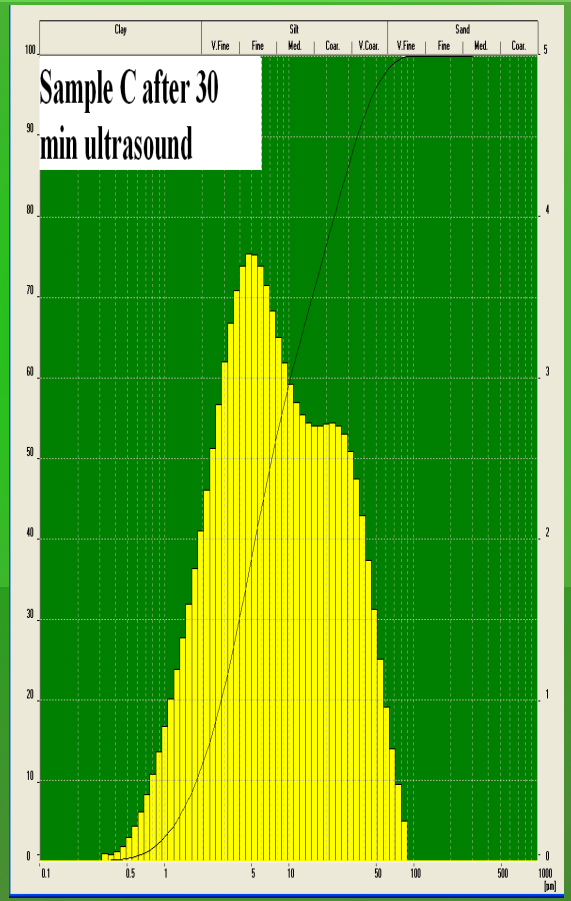
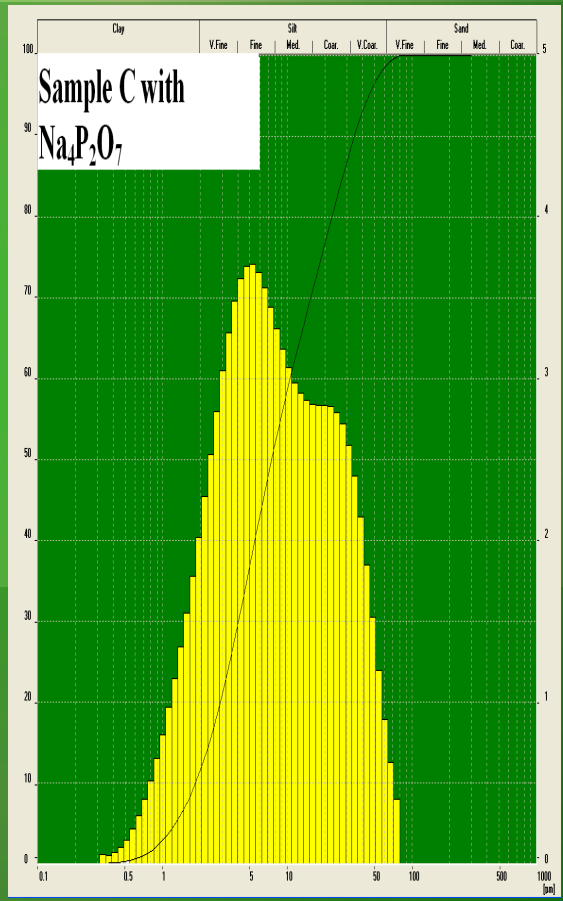
Distribution of soil fraction of sample C







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Analytical problems by the determination of total phosphorus in soil samples

Method for extraction:

- **ISO 11466**
- **1,5 g. air dried soil sample is extracted with aqua regia - (3:1 – 12 Mol/l HCl : 15 Mol/l HNO3).**

Instrument determination of total phosphorus:

International Co-operative Programme on Assessment and Monitoring of Air Pollution Effects on Forests– update 06/ 2006

- **colorimetric method**
- **ICP**

Method 1 - yellow phosphorus

Extract +
Vanadate – Molibdate reagent

410 nm

- **50% enhancement of the results**
- **The forest soil are rich on organic matter**
- **extraction of huminic acid.**

Sample	Results F29	6 th intercomparison
A	504.3	276.5
B	33.8	43.1
C	192.9	115.1
D	1463.4	748.6
E	1980.3	947.7

Method 2 - blue phosphorus

antimony-phosphate – molybdate complex + ascorbic acid

880 nm

Sample	Results F29	6 th intercomparison
A	365	276.5
B	49	43.1
C	139	115.1
D	743	748.6
E	961	947.7

Thank you for your attention!