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INFORMATION

OVERVIEW OF PROGRAM ON US–ESTONIAN SCIENCE AND TECHNOLOGY COOPERATION ON OIL SHALE RESEARCH AND UTILIZATION (STRATEGIC IMPORTANCE OF OIL SHALE STUDIES FOR ESTONIA AND USA)

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Introduction

Limitations on conventional petroleum resources are becoming well-known, and interest in unconventional hydrocarbon resources, especially oil shale and oil sands, is growing. The US and Estonia each contain vast quantities of unconventional oil shale resources. The Green River Formation deposits in Colorado, Utah and Wyoming contain more than 1,000 billion barrels of potentially recoverable kerogen oil. This amount is equivalent to the total world reserves of conventional petroleum and 50 times as much as the approximately 20 billion barrels of oil in the United States. There have been several attempts in the US to produce oil from this resource but none have survived to operate at an industrial scale.

Oil shale has been commercially produced in Estonia for over eighty years, resulting in long-standing traditions and extensive experience. Because Estonia contains no petroleum reserves, oil shale is Estonia's major energy resource. Approximately 65% of Estonia's energy needs come from its oil shale resources, with the remainder being imported in the form of finished petroleum products and natural gas. Today, three industrial plants produce shale oil; but the full chemical and economical potential of the oil has yet to be developed. Only the water-soluble phenols, about 2% of oil, are used separately as chemical feed-stock.

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The current production of shale oil causes serious environmental problems, especially the *Kiviter* process for which the semicoke of this process (organic matter content up to 10%) does not comply with hazardous waste norms of EU regulation. The problems arising from the large amounts of process dephenolized water (ca 140 kg/tonne of processed shale) plus leachates from semicoke and other deposited solid wastes need to be solved, if the industry is to remain viable over the long-run.

To improve the prospect of adding economic value to the kerogen oil, to prove a business model for application to US resources, and to provide supplemental revenues to help to solve key environmental issues, the US Department of Energy and the Ministry of Economic Affairs of Estonia conducted a joint research and development program. This paper summarizes the results of that program.

Value Enhancement Process Concept

Raw kerogen oil from Estonian shale contains high concentrations of oxygen compounds while US kerogen oil contains high concentrations of nitrogen compounds. These heteroatoms (O and N) are undesirable for refinery feedstocks and are costly to remove when manufacturing a petroleum substitute. If the kerogen oil is to be used solely to produce a substitute for petroleum, the final product value will be dictated by world crude oil prices.

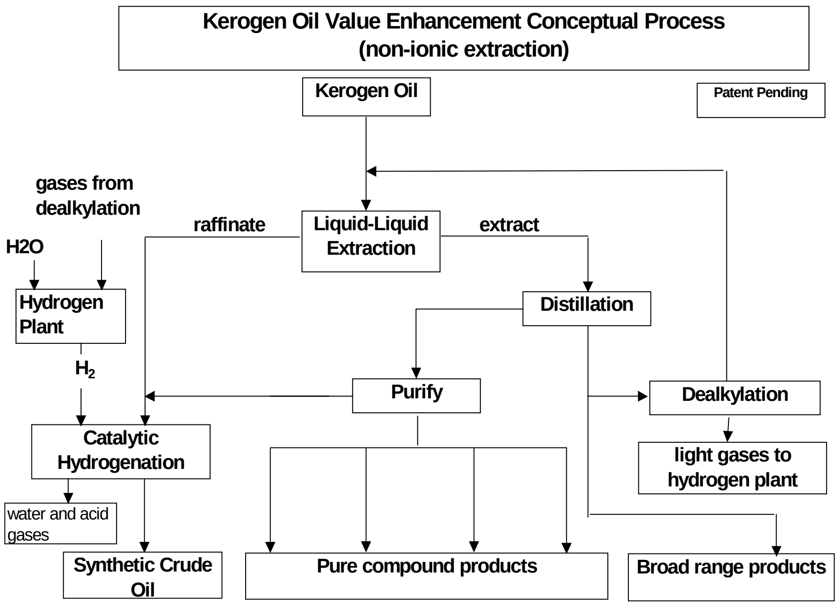

Fig. 1. Principal scheme of value enhancement process for treatment of kerogen oil based on separation by liquid-liquid extraction

However, these same heteroatom-containing compounds may prove of higher value if extracted from the oil to produce commodity and specialty chemicals. The prospect of producing high yields of high-value chemicals, which partially uncouples the revenue ceiling from world crude oil prices, helps mitigate risks of low oil prices, and enhances investment attractiveness.

Historically, in Estonia, and recently through research conducted in the United States, a process concept has emerged that could enhance the economic margins for production of these resources and warrant capital investment even when world prices of crude oil are no greater than $20/bbl. Pure oxygen and nitrogen containing compounds manufacturable from kerogen oil are valued at prices greater than $400/bbl, which is many times the value of crude oil.

The process concept, called Value-Enhancement Processing, or VEP, involves the selective extraction of oxygen or nitrogen containing compounds and subsequently processing these extracts for their chemical values. A generalized process scheme of the VEP concept is given in Fig. 1. This concept has been partially proven on both US and Estonia kerogen oils.

VEP requires that research and development be performed to discover the processes and products that could constitute the basis for a viable commercial venture. Establishing a basis for a conceptual commercial process requires the application of sophisticated analytical techniques, laboratory research on extraction systems, conversion process research, purification and product formulation studies and market research.

US–Estonian Joint Program on Oil Shale Research and Utilization

To pursue the VEP concept discussions regarding a US – ESTONIA SCIENCE AND TECHNOLOGY CO-OPERATION ON OIL SHALE RESEARCH AND UTILIZATION were initiated in 1999. The on-going industry and extensive experience and commercial successes of Estonia would serve as the foundation. If the VEP concept researched in the United States were to be successful in processing of kukersite kerogen oil, which was being commercially produced, the investment hurdles would be considerably lower and the development time shorter in Estonia than in the United States, where no commercial production facilities exist. A profitable venture in Estonia would serve as a critical business model for use in the United States.

The synergism of this plan was even further enhanced by the fact that products produced in Estonia would be sold to different markets than products produced in the United States. This offered the possibility of both countries developing exclusive production facilities that could sell products into the world markets.

How it all began. President of the Estonian Academy of Sciences Jüri Engelbrecht (left) and Director of Naval Petroleum and Oil Shale Reserves, US Department of Energy Anthony P. Dammer signing the Roundtable Summary of Discussions in May 28, 1999 in Tallinn.

The discussions led to the signing on February 4, 2000, of a Joint Cooperation Agreement by Secretary Bill Richardson of the US Department of Energy and Minister Mihkel Pärnoja of the Estonian Ministry of Economic Affairs.

The Joint Cooperation Agreement called for a three-phase program to explore value-enhancement opportunities, to develop plausible methods for processing to high-value productions and to estimate the feasibility of the concept. The extensive experience in oil shale science and technology held in both countries allowed modest program costs to be leveraged for high impact. A prime objective was to attract private industry interest to help carry the project forward toward expanded commercialization in Estonia and first commercialization in the US.

- **Phase 1** was an Exploratory Research phase in which processes concepts were identified for their effectiveness in producing large yields of high value products.
- **Phase 2** was a Development phase in which process concepts and products were further examined to obtain quantitative yield and product quality data using commercially reasonable conditions. An office was to be established within Tallinn Technical University to manage intellectual property in anticipation of developing licensable technology.
- **Phase 3** was a Feasibility Analysis phase in which the technical, market and economic feasibility of the process concepts developed in phases 1 and 2 were evaluated and a development plan was prepared.

The research was performed on the Estonian kerogen oil from the *Kiviter* and *Galoter* retorts. Economically, the most attractive scale is for one VEP plant that accepts all of the oil production. Hence, the two sources of oil were combined in their production proportions, 65% *Kiviter* and 35% *Galoter*, to simulate a possible commercial feedstock.

Summary Technical Results of Program

The main results can be outlined as follows:

Oil Separation by Ionic Extraction

The scheme for the separation of total study oil into hydrocarbons, oil-soluble phenols and neutral oxygen compounds was designed. For this purpose 10% alkaline water solution is used to extract phenols from the oil and toluene is used to extract neutral oxygen compounds from the intermediate phenolates. Using the experimental results, unit operations and mass balances of a conceptual commercial process of a feed capacity of 3×10^5 metric tons/year were outlined. In addition, process schemes for recovering lower alkylresorcinols (LARs) from process waters were elaborated. Besides this extraction scheme, the resources of water-soluble phenols from process waters (including additional washing of oil with water) in Kiviter and Galoter processes were elaborated, resulting total yields 5.4 and 1.7 kg/tonne (of processed shale), respectively, with content of lower alkylresorcinols *ca* 90 and 40%, respectively.

Oil Separation by Non-Ionic Extraction

Using polar organic solvents an oxygen-rich extract (11.3% oxygen, average molecular weight 220) was recovered from total kerogen oil. The yield was 16%, but contained more than 90% of the LARs present in the feedstock oil. This resorcinol-rich concentrate can be subsequently separated and processed to recover pure compounds. Broad range concentrates can also be produced from the extract. The use of organic solvents affords high selectivity and relatively straightforward solvent recycle; however, the overall yields of polar oxygenates is lower than with ionic extraction.

Oil Hydrogenation

Catalytic hydrogenation of ~100 g charges of total raffinate (and other oil components and fractions as well) was performed in a flow reactor at various conditions (e.g. temperature 340–360 ºC, pressure 8–11 MPa, space velocity 4 h^{-1}). Material balances were compiled (typical yield of refined oil was about 90%) and preliminary economic calculations were performed. The results show the feasibility of hydrogenation process in improving quality of the refined oil. Additionally, gas and gas naphtha are transformed *via Zeoforming* process and used as components of petrol and diesel fuels (plus some amount of propane-butane fraction).

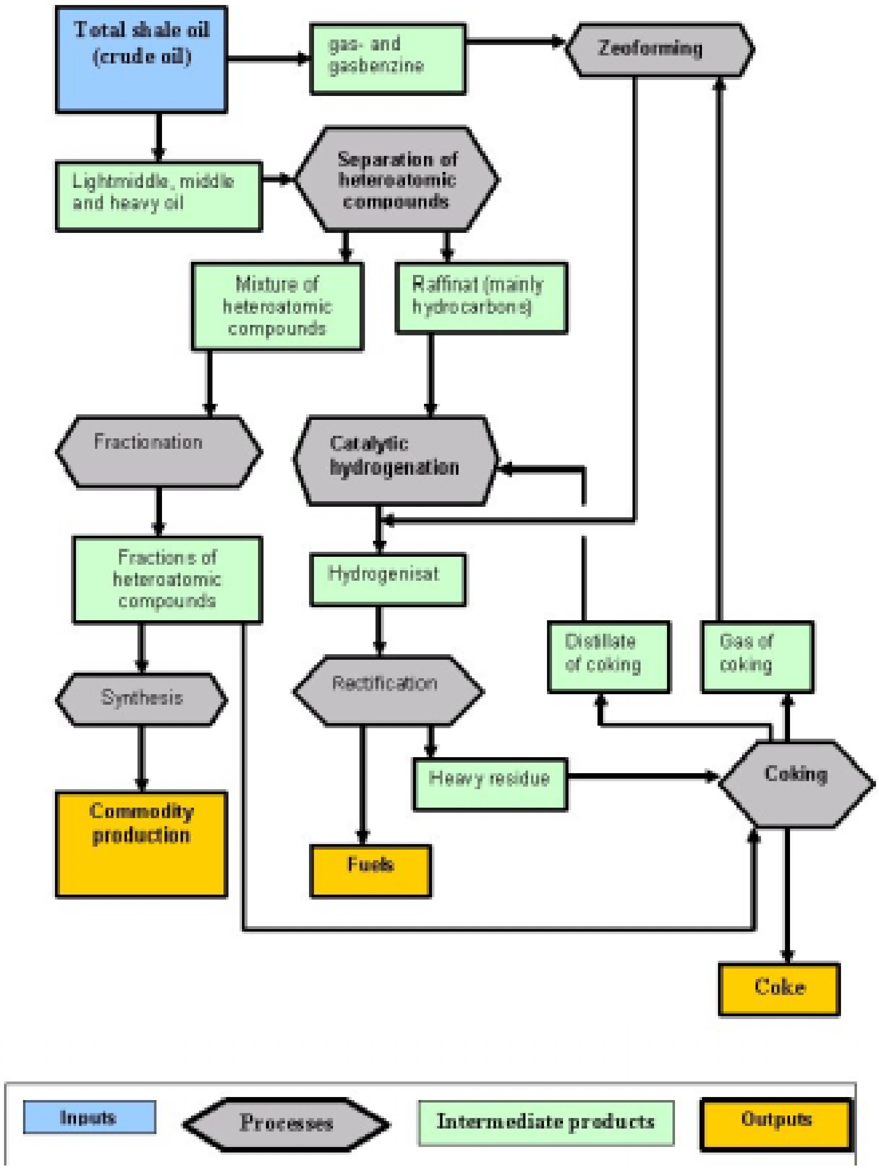

Fig. 2. Common scheme of shale oil treatment

Development of New Commodity Products from the Separated Groups

Specific Estonian shale oil products:

- pure compounds including phenol, cresols, resorcinol, orcinol and higher alkyl mono- and di-hydric phenols
- light-thermal stabilizer for the technical rubber industry, produced from water-soluble phenols
- fungicide for building materials, lubricants and paints, produced from oil-soluble phenols
- fuel additives produced from specified oil fractions
- many other modified products developed in prior research on the bases of water-soluble phenols (individual crystalline alkylresorcinols, epoxy-resins, clues, leather tannides, backfill and curing resins, etc.). The basis for present production is easily expanded
- petrol, diesel and heavy fuels can be produced using processes of petrochemistry, i.e. catalytic hydrogenation/zeoforming, and use of additives
- bitumen and coke

General Scheme of Shale Oil Processing

Based on results given above and previous experience of cooperation partners a common scheme of treatment of shale oil was developed (Fig. 2). The central process of the treatment is extraction. According to patent search this scheme is on the state of art level workout where the main attention was given to complexity of developed process scheme and uniting all steps into one big unit.

The key features of this scheme are the separation of the total oil into three components, the catalytic hydrogenation of the hydrocarbon component and the fractionation of the heteroatomic component. For the case of Estonian shale oil, the latter comprises oxygen compounds. The outputs of the scheme are fuels, commodity products and coke. In principle, the yield of the latter depends on the “horizontal and vertical” extent of the hydrogenation process, i.e. which oil components are hydrogenated to varying extents. In development of oil shale processing the attention must be given that gases cleaned from sulphur can be used in economic way to produce energy.

Process Waters Treatment

Process water treatment was improved for both phenolic compounds and nitrogen containing compounds; experiments were carried out in a continuous flow mode laboratory unit. Biological aerobic treatment reduces the di-basic phenol content (mainly LARs) below the concentrations required to comply with current regulations (15 mg/L). The nitrogen content (ca 500–550 mg/L, so far not regulated for chemical plants in Estonia, but regulated for urban-waters at 10 mg/L) was cut in half in a combined anaerobic/aerobic treatment. For further nitrogen removal it is recommended that ozonation be included in the aerobic oxidation stage. The results are useful in the designing of semicoking process waters purification for US retort waters which are richer in organic nitrogen compounds.

Recommendations for Treating Solid Wastes

In principle only retorting methods that burn semicoke and fusses to organic-free ash are environmentally acceptable for solid waste disposal. Increasing oil prices are giving more options for improved solid waste disposal and reclamation through revegetation. It is quite unrealistic to retrofit existing retorting for gasification of semicoke, and the main focus should be on proper disposal technology. Using semicoke as a fuel for steam or heat generation is feasible if the moisture content is below about 10% by weight.

Results of Market Research and Business Studies

Possible applications, key trends and expectations, competitive products, major consumers and producers for products, market capacities and geography for shale oil products were examined. These data are essential for the proper forecast of the future and forms the basis for business studies of the possible processing unit.

Five groups of products were selected for market studies.

Existing products for which expansion of the market segment is sought include:

- Alkylphenols and alkylresorcinols in mixture as well as separated in pure form (5-methyl-, 2,5-dimethyl-, 5-ethylresorcinol) – from shale oil and process-waters – raw material for foundry resins, tanning chemicals, plugging resins, and feedstock for synthesis of fine chemical products.
- Epoxy resins synthesized from alkylresorcinols originated from shale oil – used for preparation of wood adhesives, laminating glues, rigid plastics, building composites.

New possible commercial products examined for their specifications, but which will require new investment include:

- Diesel fuel additives (modifiers) – based on the hydrocarbon part of the shale oil. The additives are added in small quantities to fuel to improve the burning and lubricating properties of that.
- Biocides based on alkylresorcinols separated from shale oil – used as components of building materials like paints and lacquers for protection from biodegradation.
- Light- and thermostabilizers based on alkylresorcinol-based benzo- or acetophenones – used for additives in plastics and rubber compositions as

UV blockers, antioxidants, to protect from degradation under heat and light.

It was found that the growing markets for chemicals will create good opportunities for shale oil products of different kinds, and the main chemical and market potential have water-soluble shale oil phenols and products as their base. The most promising market for alkylphenols and alkylresorcinols is chemical feedstock market, the straightforward conclusion is to develop synthesis chemistry on their base and sell more expensive end-products. The separation of individual compounds (5-methylresorcinol) is also a very good prospect.

Analogues to oil-soluble phenols on the world market were not found. However, the potential resources of oil-soluble phenols (higher alkyl phenols) are much bigger then water-soluble phenols (lower alkyl phenols). There is a great challenge for scientists and technologists to find useful applications and market opportunities for higher alkyl phenols.

Also the new commercially valuable products based on the shale oil hydrocarbon portion, like diesel fuel additives, could be developed. It is assumed that the conventional petroleum refining industry is large enough that hydrotreated raffinate could be sold directly to these markets, provided that specifications are within reasonable limits for refinery feedstocks.

There are three groups of products (diesel additive, biocide and light-thermo-stabilizer), which have passed initial specification tests and need to be valuated for product development. The end customers of these products are **fuel and plastics-rubber industries respectively**. These industries might be interested in the abovementioned products as raw material for their production or they might want to produce the stabilizer themselves if results of price-benefit analysis would be favorable.

To help accomplish the objective of making the program results attractive to investment it was important to establish a framework for managing Intellectual Property (IP) generated during the program. In the Office of Research and Development of Tallinn University of Technology (TUT) was set the order to manage IP and make it available for licensing on commercially-reasonable terms. Preliminary analysis of the value-enhancement venture indicated considerable economic attractiveness and it raises the value of results of the Program.

As result of the Joint Research Program, the IP-Portfolio includes:

1. Reports of market studies of five product groups;
2. Scientific studies reports;
3. Business analysis report;
4. Patent research reports on the novelty examination of biocides and light-thermo-stabilizers.
5. And any additional reports, articles and information, created during or after the end of Program based on Program results.

In the past ten years, the existing patents claim solutions mainly for cleaning of different oils – shale oil is mentioned as one possible use without special tests on shale oil. There is no special solution for shale oil only. There are only very few complex solutions for oil treatment. In conclusion it can be said that this scheme is on the contemporary world level technical standing. A process patent application based on the scheme shown in Fig. 1 is pending in both the US and Estonia.

Patents in the field of biocides were searched in order to evaluate the patentability of the biocide composition developed by TUT. Subject matter of the described invention was the use of oil-soluble phenolic compounds as biocide, i.e. as antibacterial and antifungal agent. Use of phenolic compounds (including alkyl-phenols and -resorcinols) for antibacterial and antifungal purposes in wood, paints, fuels, construction materials and as fungicides and stabilizers of plastic materials is well known. Several patent opportunities were revealed and patents are being pursued.

A patent search was conducted on light- and thermo-stabilizers in order to evaluate patentability of the light- and thermo-stabilizers developed by TUT. Use of benzophenones as light- and thermo-stabilizers is well known. In principle, the use of shale oil resorcinols for synthesis of light-thermostabilizers is possible. There was no information on patents that would be infringed by using Estonian oil shale products as light- and thermo-stabilizers. A number of patent opportunities were revealed and patents are being pursued.

Conclusions of Technical Program

The results confirmed the original premise of the program; that is, large quantities of oil soluble phenolics may be selectively extracted using processes adapted from the petroleum and chemical industries. The extracted phenolics can be manufactured into pure compounds, valued at $400/bbl, or more, and broad range concentrates, valued at $100/bbl, or more.

The markets for these products are generally established. Pure compounds are used as feedstocks for manufacture of foundry resins, wood adhesives, laminating glues, rigid plastics, UV blockers, antioxidants, and feedstocks for manufacturing of fine chemical products. Broad range concentrates are expected to find markets as industrial surfactants, industrial antimicrobials, moldicides, solvents, oil-field chemicals, fuel additives, asphalt stability enhancers and other possible products that can utilize the properties of phenolic functionality. The main high-volume product is a low-sulfur fuel, expected to compete favorably with petroleum-derived fuels.

Excellent success was achieved in the environmental tasks. Process conditions for decontaminating process water to class 3 standards (industrial recycle) have been identified. The current phenolic water processing systems might be upgraded at acceptable costs with the goal of approaching zero discharge for new facilities, while reducing the discharge for current facilities. Energy balances on the utilization of semi-coke, along with projected economics, show that new processes could avoid the current environmental problems at acceptable costs, and legacy problems may be remediated under certain conditions.

Comparison of process efficiencies between the two operating technologies (*Galoter* and *Kiviter*) shows specific benefits for each. The scale of a conceptual commercial operation is large enough to utilize all of the oil currently being produced in Estonia. Preliminary analysis of the economics of a value-enhancement venture shows considerable attractiveness, particularly at a maximum scale of operation. Interest by industry is growing and relationships with the Joint Cooperation Agreement are being developed.

Recommendations for Future Research and Development

The results of the research showed promising directions to concentrate future research and development efforts:

- specification of the extent of the hydrogenation process and optimizing the scheme with introduction the most modern technologies for extraction
- search for new, more selective separation techniques for both hydrocarbon part and heteroatoms containing part
- development of new products and new processing technologies for higher alkyl, oil-soluble phenols
- identify and develop a process for dealkylation of higher alkyl phenols
- adjusting the parameters for coking process and expanding the Value Enhancement Process concept for shale oil coke

Further developments must contain testing and specification of parameters on pilot units for all stages of the proposed general scheme of shale oil processing (in the future including shale oils from different sources, possible joint retorting of shale oil and organic waste). For promotion of the results of USA-Estonia Joint Research program, additional work could be done by the Estonian or USA federations of the chemistry industry, who could communicate and provide informational support to the companies looking for new R&D opportunities. The role of such federations could be to regularly update the product development matrix and import newly released data from manufacturers and end-users. Companies are financially interested to promote their own products, processes and intellectual property. The federation could issue and maintain a comprehensive database of products from different companies thereby increasing the value of the database for any interested body.

Program Accomplishments

The original motivation for exploiting the prior experience of both USA and Estonia researchers to accomplish the objectives of the program at low cost has been validated by this work. Within four years the program was formed, tasks were delineated, laboratory experimentation was conducted, results were integrated, and a package of intellectual property was produced.

The general goals achieved in Program are:

A. Created new product manufacturing opportunities through value-enhancement processing.

B. Developed the science and technology approach for improved mitigation of environmental impacts relative to solid waste and water effluents.

C. Established a mechanism for technical cooperation between the two Countries.

D. Coordinated the various oil shale expertise and interests of both Countries including Government, Industry and Academia.

E. Advanced the prospects of technology application including technology licensing for use in other countries.

The Program gave specific benefits for both of countries. For the United States a foundation has been established for a commercial model for replication in the US, and certain technology has been further reduced to practice that may have export value. The Program opened lines of communication and established a working relationship between the two countries on integration of common interests in unconventional oil shale development together with related databases of industrial activities, product manufacture and market opportunities.

The Program has had very positive impact on Estonian oil shale studies and helped to preserve a body of expertise in Estonia, and additionally to establish an awareness of the value of Intellectual Property as the basis for advancing economies. The Program had a major influence on implementing formal policies and procedures within TUT for capturing, preserving and developing ideas of potential market value, as well as the establishment of formal personnel agreements that detailed the responsibilities, obligations and management of inventions.

With this program the Estonian side enhanced its technology and intellectual property position and framed a new opportunity for investment in Estonia's most important natural resource. Results enhanced the capability of making efficient and environmentally responsible use of their oil shale together with electricity generation, which contributes to nuclear-free energy independence for the entire Baltic region. Valuable are results from exploration of processes and products that minimize current and prior environmental impacts.

The joint benefits are:

- Cooperation on oil-shale research resulted in a significant confluence of unique scientific and technical abilities, private U.S. investment, potential for profitability in both Estonia and U.S. and furtherance of national security interests.
- The market potential from Estonian oil shale derivatives could be of particular interest to U.S. investors.
- Established a scientific exchange program between experts as a means of enhancing economic development of the oil shale resources in both Estonia and the U.S.
- Identified commercially plausible processes to produce high value products from Kukersite (Estonia) and Green River (U.S.) shale oil and carry out related R&D.
- Examined scientific and technical means for improving the efficiency of oil production, process water treatment and waste disposal.
- Constructed a development plan to attract private interest and investment in commercial development

The main attention and constant cooperation between scientists and engineers must be directed to improvement of known products and finding new markets for them. The development of shale oil industry is a realistic way to meet future demands for energy and organic raw material. There must be a dialog between oil shale industry and politics, especially on environmental issues, where scientists must play the balancing role taking into consideration economical needs.

It is hoped and expected that this Program will serve as the initial activity of further technical cooperation between the countries of Estonia and the U.S.

Member of the Joint Coordinating Committee Mr. Hugh D. Guthrie (left) and Program Coordinator Ph.D. Mihkel Koel.

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