Successful Upgrading of Estonian Shale Oil Via Hydrotreating

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AS Narva Õlitehas

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Presentation Topics

- Introduction
  - Brief Introduction To Eesti Energia / Narva Õlitehas
  - 'Drivers' Leading To Shale Oil Pilot Testing Program
- Questions Addressed By Pilot Plant Testing
- Technical Results From Hydrotreating Pilot Plant
- Product Value Enhancement
- Forward Plans Of Eesti Energia / Narva Õlitehas

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Business Mix Of Eesti Energia

- Annual Production: ~ 14 million tons

- Shale Oil Retorts: 240,000 TPA Rated (1.5 million bbl/yr)
- Power Plants: 2,603 MW(e)+ 1,081MW(heat)

- Covers Whole Of Estonia: 5,000 km network

- Covers 92% Of Estonia Population

- 500,000 Customers
- Export Power Sales: Latvia, Lithuania, Finland

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The Narva Oil Factory, established in 1980, has almost 30 years of operating expertise

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'Drivers' For Upgrading Estonian Shale Oil Liquids

- **Oil Shale Is A Major Natural Resource Of Estonia**
- **Narva Õlitehas Is Already Processing Estonian Oil Shale Rock In Its Two Commercial TSK-140 Retorts [TSK = Recirculating Solid Heat Carrier Retort]**
- **Eesti Energia---Through AS Narva Õlitehas---Is Planning A Major Expansion To Its Oil Shale Retorting Operations**

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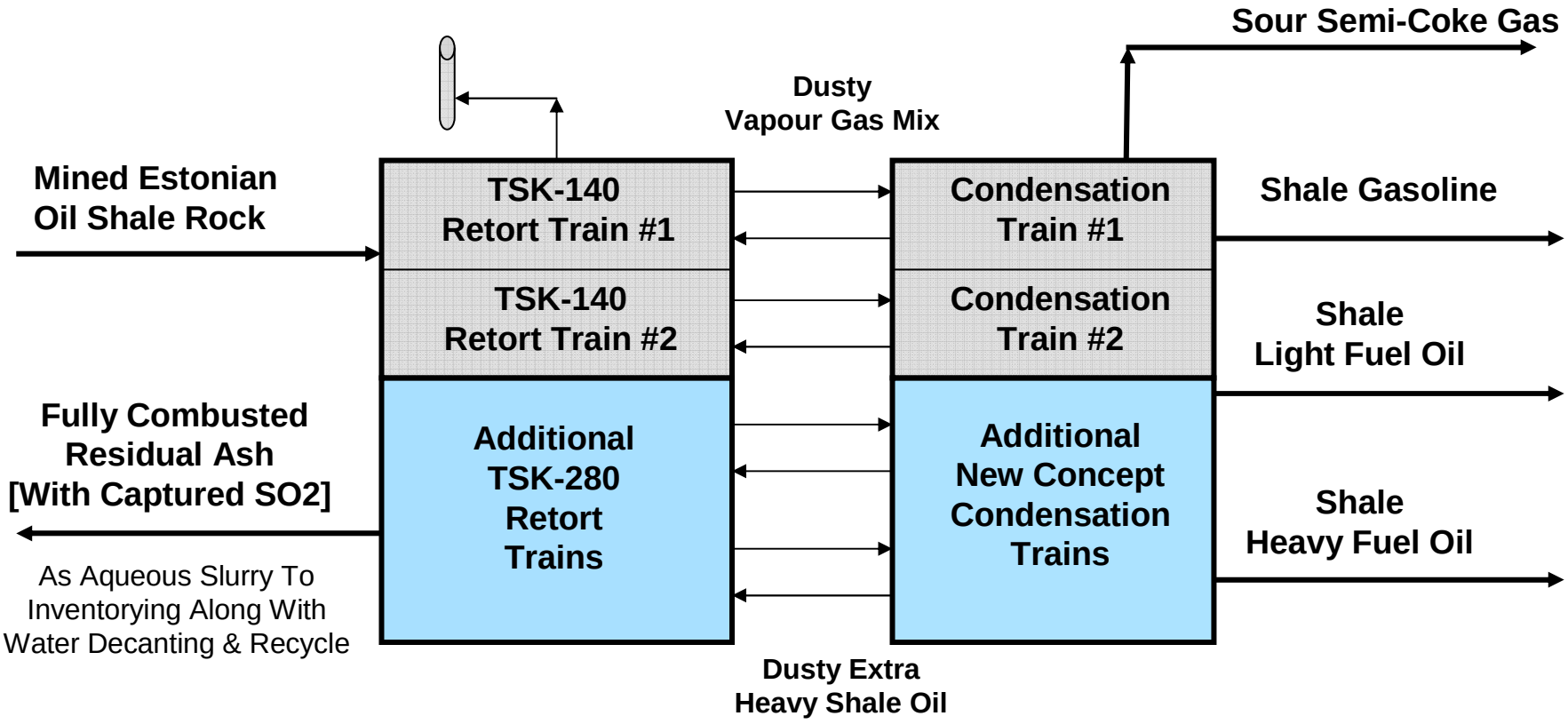
Expanding TSK Retorting Plant AS Narva Õlitehas



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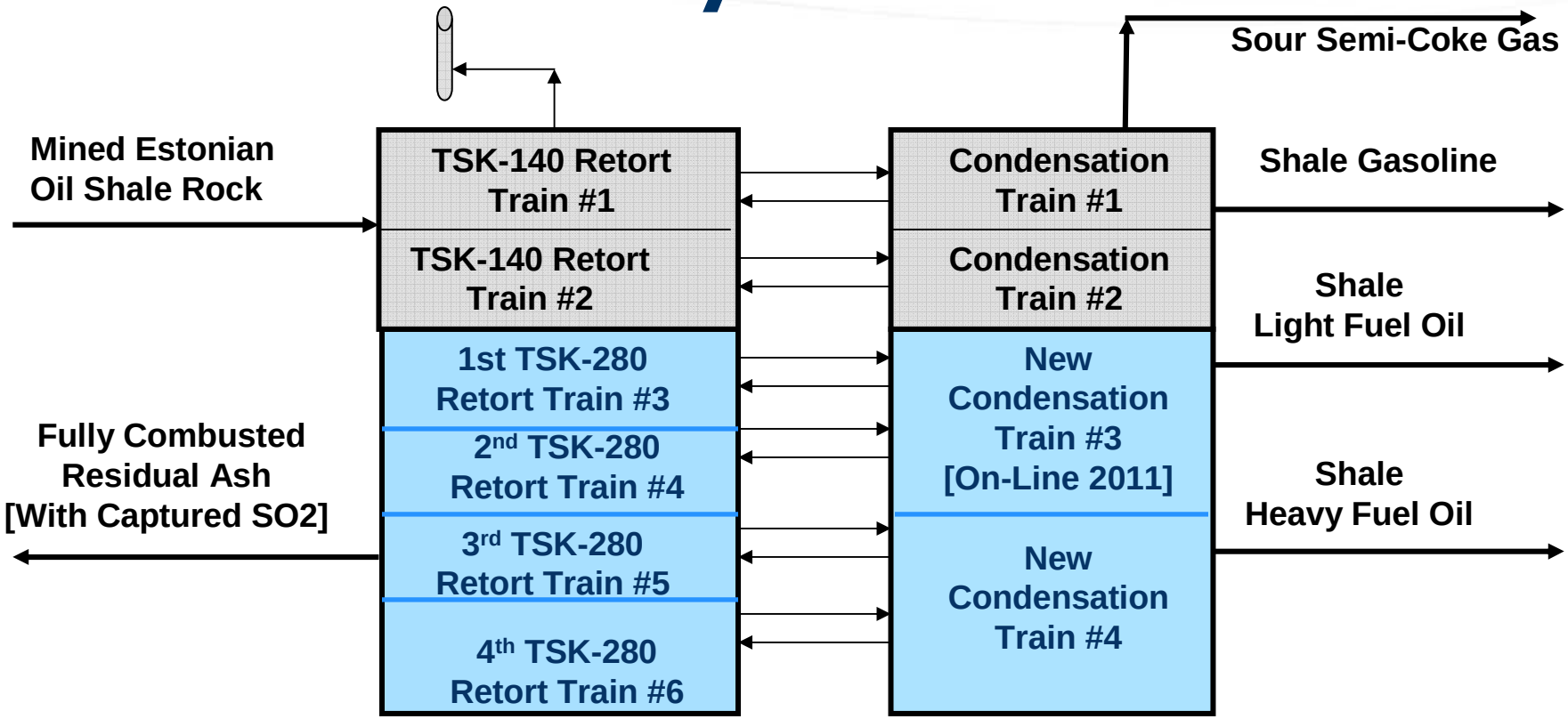
Narva TSK Retorting Plant By 2016



- TSK 280 design ready by May 2009
- Operational 2011/2012
- 30,000 BPSD Shale Oil Liquids By 2016

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'Drivers' For Upgrading Estonian Shale Oil Liquids

- **Market Resistance Encountered For Current Products:**
  - **Shale Gasoline**
  - **Shale Light Fuel Oil**
  - **Shale Heavy Fuel Oil**
- **Market Resistance = Discounting**
- **Unconventional Qualities Are Holding Down Interest And Sales**

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Team Effort
Shale Oil Pilot Plant Tests

• Eesti Energia AS (Tallinn, Estonia)	Sponsor & Owner
• AS Narva Õlitehas (Narva, Estonia)	Operator Of TSK Retorts Shale Oil Producer / Supplier
• Jacobs Consultancy (Houston/London)	Program Planning, Organizing, Monitoring & Results
• Intertek PARC (Pittsburgh, PA, USA)	HTU Pilot Plant Facility Operations
• Haldor Topsoe (Lyngby, Denmark)	Hydrotreating Catalyst Supplier

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Key Questions Addressed By Hydrotreating Test Program

- **Can Estonian Shale Oil Liquids Be Upgraded Via *Refinery-Type* Distillate Hydrotreaters ?**

- **Will Hydrotreated Products Be Attractive To European Union Refiners ?**

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The Quality Problem
Naphtha-Distillates-VGO Σ C5 to 565°C

Quality		Raw Estonian Composite Shale Oil	Brent Crude Naphtha-Distillates-VGO
API Gravity	°API	15°	42°
Sulphur	Wt%	0.8	0.3
Nitrogen	Wt%	0.25	0.045
Oxygen	Wt%	6.1	< 0.15
Hydrogen	Wt%	9.8	13.3
Cetane In Diesel Cut	CI(D976)	~ 28	~ 49
Bromine Number	‘g Br2/100g	~ 45	< 2
UOP/Watson K	-	10.5±	11.9±

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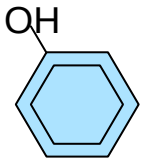
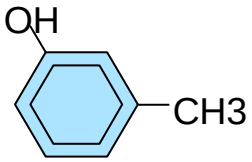
Heteroatom Oxygen Species Estonian Raw Shale Oil Liquids

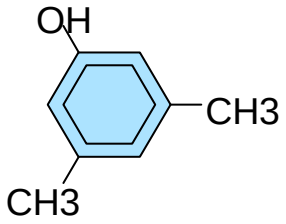
PHENOL



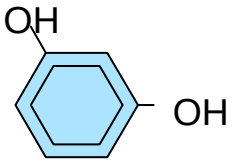
m-CRESOL



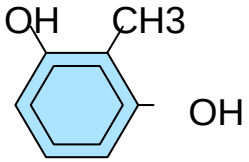
m-XYLENOL



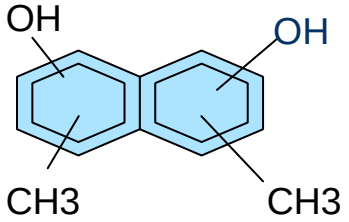
RESORCINOL



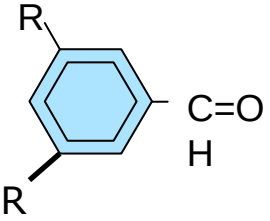
p-METHYL RESORCINOL



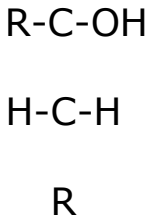
'PHENOLIC PNA'



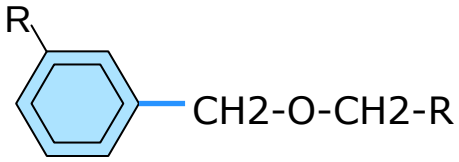
'ALDEHYDES / KETONES'



'HIGHER ALCOHOLS'



'AROMATIC ETHERS'



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Quality Challenges
Estonian Raw Shale Oil Liquids

- **Very Aromatic & Unsaturated**
- **Uniquely High Content Of Organic Oxygen In All Fractions**
  - ► **Total Oxygen = 6.0-6.5 wt%**
- **Hydrogen Deficient**
- **As Quirks:**
  - **Shale Heavy Fuel Oil Is Low In Sulphur [S ~ 0.6 wt%]**
  - **Shale Gasoline Is Extreme In Sulphur, Nitrogen, Oxygen**

- Sulphur	=	14,000 wppm
- Nitrogen	=	600 wppm
- Oxygen	=	76,000 wppm
- Bromine Number	=	~ 60 g Br2/100 g oil

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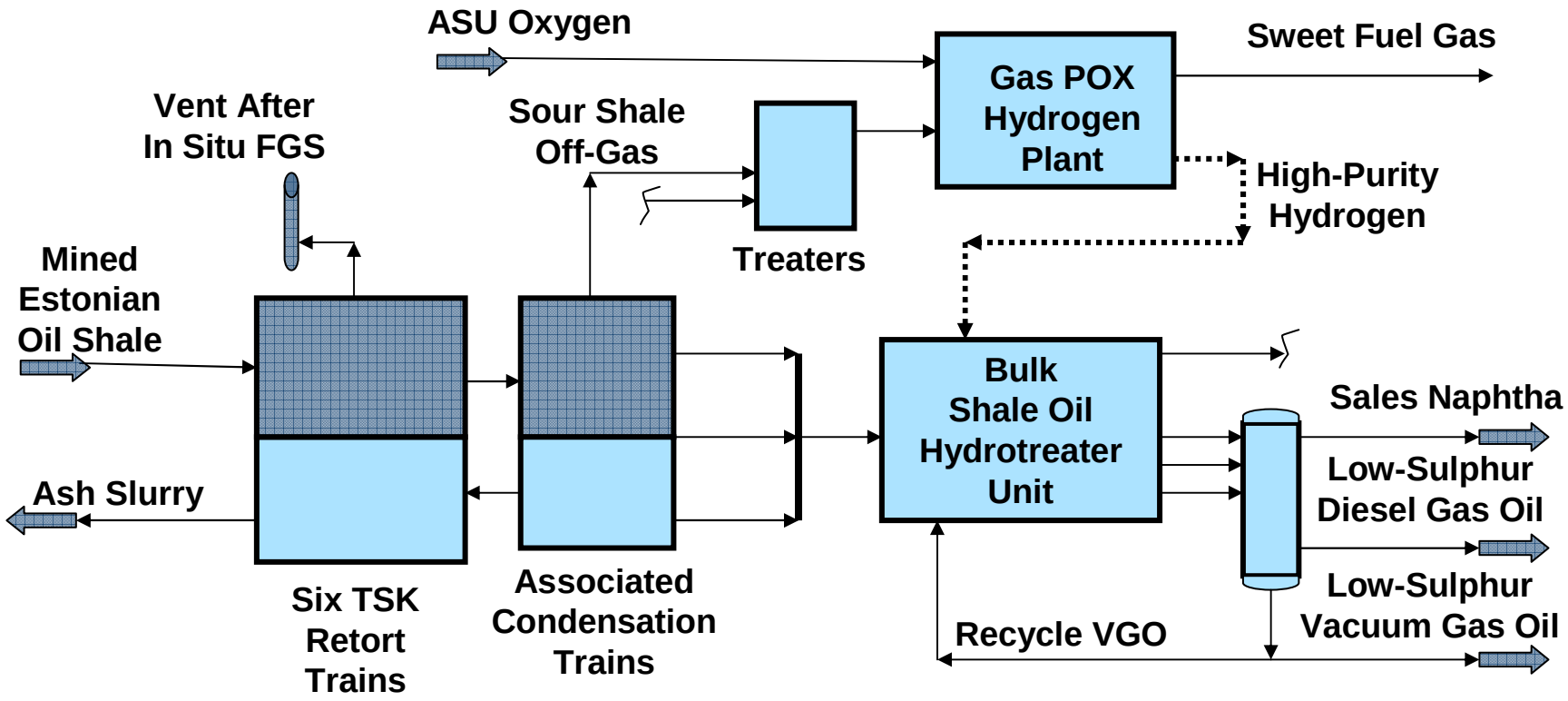
Proposed Solution To Quality Challenges

- TSK Retort Plant Expansion

- New Shale Oil Upgrader Plant



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Technical Objectives
HTU Pilot Plant Program

- Prove Estonian Shale Oil Liquids Can Be Upgraded Using *Refinery-Type* Hydrotreaters And Catalysts, *Despite*:
  - Extremely high organic oxygen content
  - Uncertainty whether poly-nuclear aromatic cores in shale oil will hydrogenate easily
- Characterize Derivative Oil Liquids
  - How Much Do Qualities Improve?
- Verify Chemical Hydrogen Requirements

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Key Results
Kinetic Performance HTU Pilot Plant

• **Hydro-De-Sulfurization**	=	**> 98 %**
• **Hydro-De-Nitrification**	=	**~ 90 %**
• **Hydro-De-Oxygenation**	=	**~ 96 %**
• **Bromine Number Reduction**	=	**> 98 %**
• **Selectivity**		**~ 94.5 wt % C5+** **~ 107 vol % C5+**
• **∆ API Uplift [Shale Oil Composite]**		**+20° ∆** **[15° → 35°]**
• **Chemical Hydrogen**		**~ 3.4 wt%** **2160± SCF per barrel**

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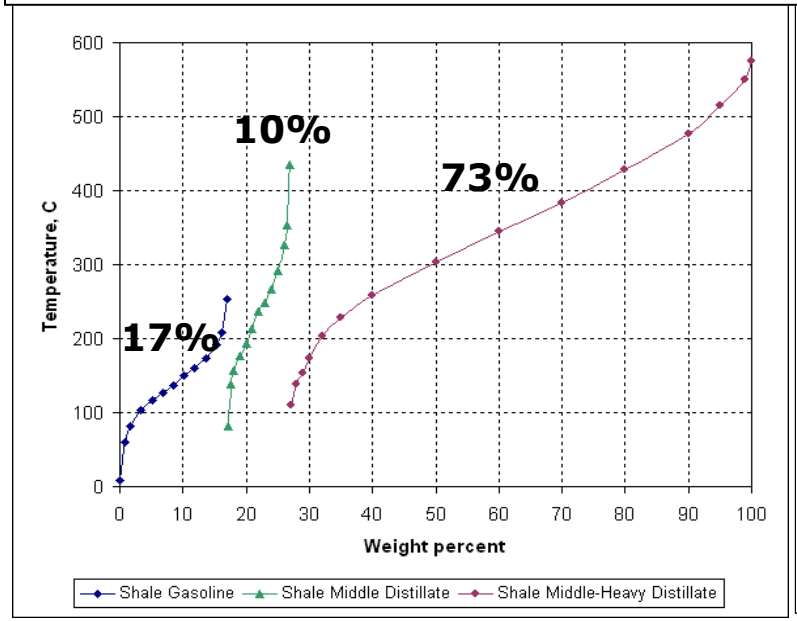
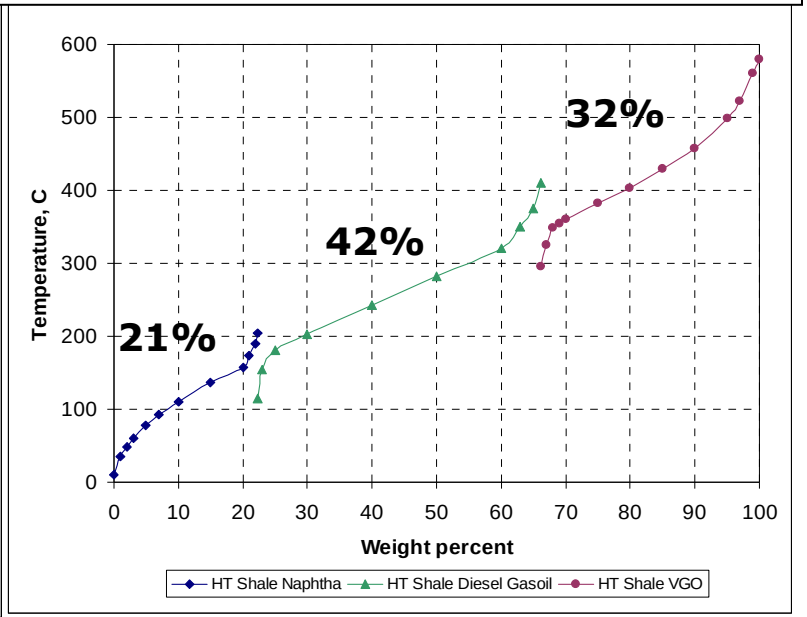
Changes Found Across Shale Oil Hydrotreater

- Specific Gravity = 0.966 → 0.865
- Organic Sulphur = 0.8 wt% → 0.015 wt%
- Organic Oxygen = 6.1 wt% → 0.3 wt%

TBP Distillation Curves --- Before And After





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Before And After

Raw Shale Oil

Hydrotreated Shale Oil

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After Quality Comparison
Naphtha-Distillates-VGO Σ C5 to 565°C

Quality		Raw Shale Composite	Hydrotreated Shale Composite	Brent Naphtha-Distillate-VGO
Volume Balance	Percent	100 %	107± %	-
API Gravity	°API	15°	35°	42°
Sulphur	Wt%	0.8	0.015	0.3
Nitrogen	Wt%	0.25	0.025	0.045
Oxygen	Wt%	6.1	0.3	0.15
Hydrogen	Wt%	9.8	12.9+	13.3
Cetane In Diesel	CI(D976)	~ 28	~ 47	~ 49
Bromine No.	`g Br2	45	1	< 2
UOP/Watson K	-	10.5±	11.7±	11.9±

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Conclusions

- **Operating Pressure Must Be Similar To Diesel Hydrotreater Units Making Ultra Low Sulphur Diesel Gas Oils:**
  - **To deal with high oxygen + nitrogen+ sulphur in front end**
  - **To gain marketability via hydrogenation of PNA cores**
- **Haldor Topsoe Catalyst Performed Very Well**
  - **No indications of deactivation**
  - **But only short duration tests**
- **High Percentages Of Organic Oxygen Can Be Removed**
- **Hydrotreated Oil Liquids Become Similar To Crude Oil Cuts**

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Challenges And Lessons Learned

- **Shale Heavy Fuel Oil Is Subject To 'Aging' and 'Chronic Coking'. Commercial HTU Must Mitigate Via:**
  - **Direct transfer from TSK Retort Trains**
  - **Latest generation oil feed filtration**
  - **Hydrogen spiking as soon as possible**
  - **Liquid phase dilution within HTU reactor**
- **Diluent Oil Must Boil In Vacuum Gas Oil Range**
  - **Diluent oil is essential to control ΔT rises in reactor**
  - **ULS diesel gas oil proved too volatile**

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Product Value Enhancement

- **Very Large Uplifts In API Gravity And Cetane Index Occur In Diesel Gas Oil Fraction. Hypotheses On Why:**
  - **Release Of Polar Compaction From Oxygen Species**
  - **Intentional Hydrogenation Of Poly-Nuclear Aromatics**
- **Large Volumetric Expansions Found. This Offsets 'Mass Loss' From Removing Hetero-atoms [Organic S / N / O]**
- **Modeling Work Vis-A-Vis European Union Refineries Indicates All Three 'Hydrotreated Shale Products' Should Sell At Prices Similar To Marker Brent Crude**

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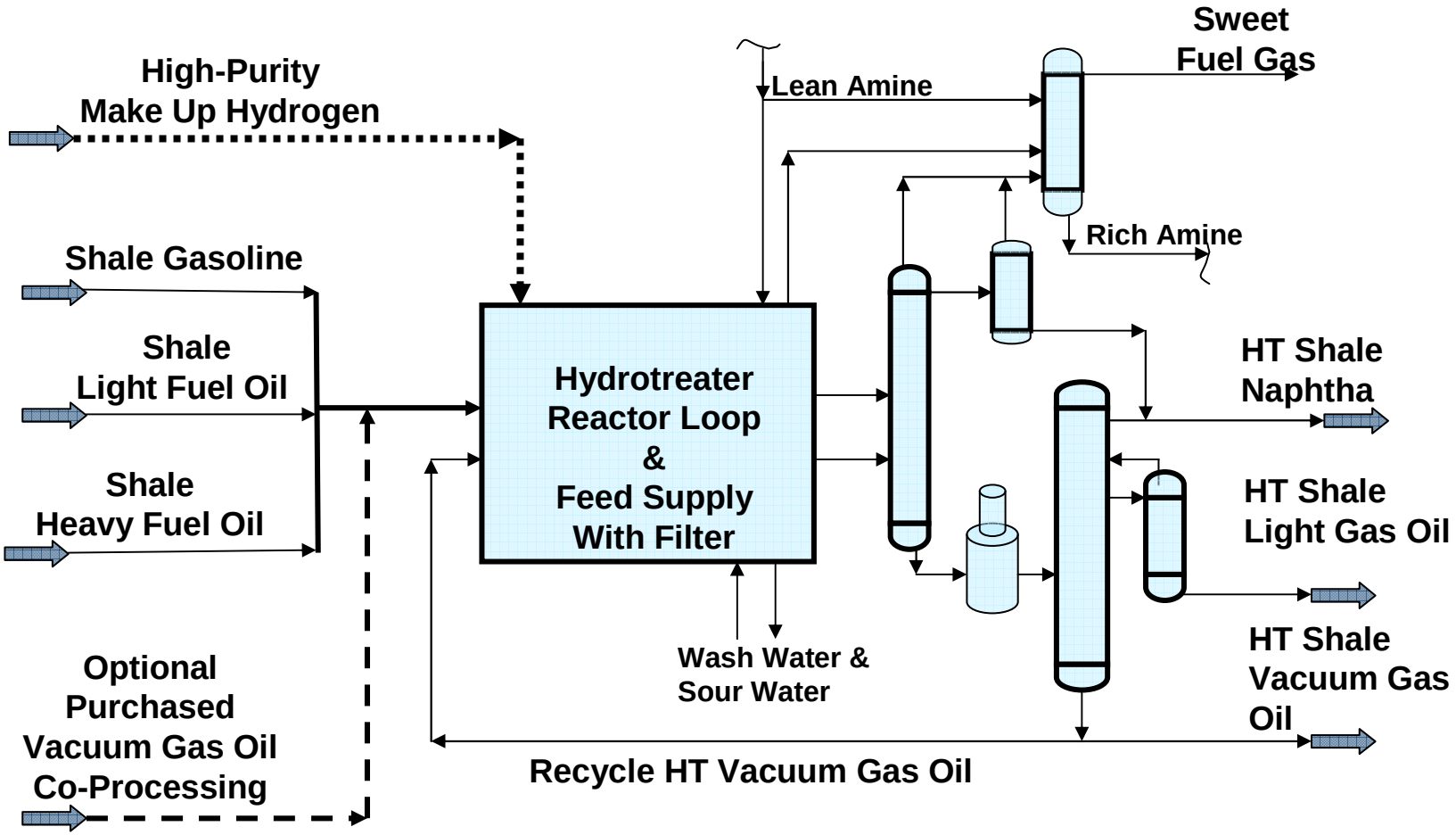
Commercial-Scale
Bulk Shale Oil Hydrotreater Unit



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Forward Plans

- **Update Strategic Plans For:**
  - **New TSK Retorts & Condensation Trains**
  - **Bulk Shale Oil Hydrotreater Unit**
  - **Semi-Coke Gas POX Hydrogen Plant**
  - **Sulphur Block**

- **Structure And Run Confirming HTU Pilot Plant Testing:**
  - **Explore Catalyst Deactivation**
  - **Develop Complete Assays For HT Shale Co-Products**

- **Prepare 'Front End Engineering Design'**

- **Confirm 'Capital Investments'**

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Acknowledgment

Eesti Energia AS / AS Narva Õlitehas

- Priit Ploompuu
- Kirill Kaidalov
- Rikki Hrenko

Jacobs Consultancy

- George Grey
- David Whittaker
- Mitch Sapp
- Shaun Boardman
- Anna Rozenberg

Haldor Topsoe

- David Juist
- Lars Skyum

Intertek PARC

- John Brophy
- Robert Absil

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Questions?

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