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Oil Yield and Gas Compositions of Oil Shale Samples at Variable Time and Temperature



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Indications of Oil Shale Sensitivity to Temperature and Time

- Prolonged drying of oil shale at 150°C reduces Fischer assay oil yield.
- Prolonged drying of lignite coal at 150°C significantly reduces Fischer assay oil yield.
- Shale exposure to 200°C, as in the ATP preheat zone, results in some odorous gases.
- Shale exposure to 275°C for short periods results in condensate containing hydrocarbons.
- Certain shales have a faint bituminous smell when crushed indicating the presence of hydrocarbons.



Overall UMATAC Test Program to Evaluate Oil Shale Sensitivity to Time and Temperature

Test Series	Apparatus Used	Vapour Discharge Orientation	Duration of Test	Temperature of Test (°C)	Sample Used
A	ATP Batch Unit	Side	Various	150, 225, 300, 350, and 400	5 Oil Shales
В	Slow Soak	Тор	50 – 70 days	150 – 560	5 Oil Shales
С	ATP Batch Unit	Side	Various	400	5 Oil Shales
D-1	Slow Soak	Bottom	1 day	20 – 560	Utah and Jordan Shales
D-2	Slow Soak	Bottom	5 – 20 days	20 – 560	Utah and Jordan Shales







The ATP Processor in Gladstone, Australia



211 t/h ATP Processor at the Stuart Oil Shale Demonstration Facility



Characteristics of the Five Oil Shales Tested

ltem	Jordan	Utah	Fushun	Australia	Estonia
Sample Age (years)	11	3	5	14	8
Crushed Top Size (mm)	12	14	14	18	12
Moisture (wt%)	3-7	2-4	3-7	25-30	10-20
Modified Fischer Assay (LT0M C_4 +)	135	140	90	125	110
C ₆ + Oil Specific Gravity (g/mL)	0.95	0.94	0.89	0.90	0.94
Modified Fischer Assay (kg/t 0M)	125	130	80	112	103
Ultimate Analysis (dry basis)					
C (wt%)	19.60	18.90	13.60	19.24	17.14
H (wt%)	1.89	1.90	2.10	2.72	1.68
N (wt%)	0.39	0.50	0.81	0.55	0.36
S (wt%)	3.27	0.61	0.61	1.52	2.62
O (diff) (wt%)	9.85	10.9	5.76	5.27	9.75



Characteristics of the Five Oil Shales Tested

Typical UMATAC – ATP Batch Unit (per 1000 kg feed)

ltem	Jordan	Utah	Fushun	Australia	Estonia
C ₆ + Oil (kg)	114	121	86	110	114
C ₆ - Gas (m ³)	36	33	37	32	36
Main Feature	High Sulfur Oil	High Limestone	Lowest Oil Content	High Moisture	Phenolic Oil



Crushed Jordan Oil Shale Sample





ATP Batch Unit – 2500 g Oil Shale Feed Capacity





Test Series A – Test Results for Jordan Oil Shale

Jordan Oil Shale Feed	150°C	225°C	300°C	350°C	400°C
Oil (mL)	0.0	0.0	0.0	4.6	30.0
Water (mL)	0.0	0.0	0.0	4.4	1.8
H ₂ S (vol %)	Trace	Trace	1%	30%	60%
CO ₂ (vol %)	1%	2%	5%	15%	12%
C ₆ + (vol %)	Present	0.5%	1.5%	2.0%	1.5%
$C_{1} - C_{5}$	No Peaks	Slight	All	All	All
H ₂ (vol %)	0.0	Trace	Trace	0.5%	2.0%
Odour	None	Musky	Faint HC	H_2S	Strong H ₂ S
Feed LOI* (wt%)					25.0
Product Solids LOI* (wt%)					19.6
LOI* Loss (wt%)					5.4

*LOI is Loss on Ignition at 600°C



Slow Soak Test Units



Pictured on the left are stainless steel containers used for the slow soak tests

Pictured on the right are the insulated and assembled stainless steel test units used for the slow soak tests



Gas Analysis

Shown on the right is a tedlar gas bag, pump, and gas meter used during the analysis



Shown on the left are UMATAC's gas chromatographs – one is used to measure refinery gas and the other for hydrogen and methane



Series B – Jordan Oil Shale Slow Soak Testing

Jordan – 58 Day Soak – Oil and Gas Accumulations

Jordan – 58 Day Soak – Off Gas Compositions





Series B – Utah Oil Shale Slow Soak Testing

Utah – 70 Day Soak – Oil and Gas Accumulations

Utah – 70 Day Soak – Off Gas Compositions





100

90

80

70

30

20

10

0

Series B – Fushun Oil Shale Slow Soak Testing

Fushun – 61 Day Soak – Oil and Gas Accumulations

Fushun – 61 Day Soak – Off Gas Compositions

CO₂

10

15

20

FMG Slow Soak C₆+, H₂S, CO₂, C₁, & H₂

Soak Temperature

C₁

30 35 40 Soak Time (days) 45

50

55

60

65 70

H₂S

25





Series B – Estonia Oil Shale Slow Soak Testing

Estonia – 48 Day Soak – Oil and Gas Accumulations

Estonia – 48 Day Soak – Off Gas Compositions







Series B – Australia Oil Shale Slow Soak Testing

Australia – 62 Day Soak – Oil and Gas Accumulations

Australia – 62 Day Soak – Off Gas Compositions





Series B – Gas Chromatograph Traces

Utah Oil Shale Slow Soak Top Discharge Gas Chromatograph





Series B – Slow Soak Tests Oil Yield Results

	C ₆ +	% Recovery Slow Soak		
	Fischer	ATP Batch	Slow Soak	(referenced to Fischer)
Jordan	125	114	21	17%
Utah	127	121	46	37%
Fushun	80	86	20	23%
Australia	112	110	24	22%
Estonia	103	114	19	17%



Series B – Slow Soak Tests Gas Yield Results

	Gas Volume (m³/t)				
	Fischer	ATP Batch	Slow Soak	Ratio Referred to ATP Batch	
Jordan	Not measured directly but included in the overall weight loss	36	72	2.0	
Utah		33	108	3.3	
Fushun		37	56	1.8	
Australia		32	73	2.3	
Estonia		36	61	1.7	

Series B – Slow Soak Tests Specific Gravity of Oil Produced

	C ₆ + Oil Specific Gravity (g/mL)				
	Fischer	ATP Batch	Slow Soak		
			325°C	375°C	
Jordan	0.96	0.95	0.82	0.81 - 0.85	
Utah	0.94	0.93	0.80 - 0.76	0.78 - 0.88	
Fushun	0.89	0.87	0.77	0.78	
Australia	0.90	0.89	0.78 - 0.80	0.92	
Estonia	0.94	0.92	0.85	0.90	

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Jordan Oil Shale Batch Tests - Oil Yield (g/kg, dry basis) 120 110 100 90 Total Oil Yield 80 □ Oil Yield @ 400°C 0il Yield (g/kg) 00 20 20 550°C 40 30 20 400°C 10 0 Ramp Run to 1 1/2 hour 3 hour soak @ 5 hour soak @ 7 hour soak @ 18 hour soak 44 hour soak 60 hour soak 560°C soak @ 400°C 400°C then to 400°C then to 400°C then to @ 400 C then @ 400°C then @ 350°C then then to 560°C 560°C 560°C 560°C to 560 C to 560°C to 560°C



Test Series C

Jordan oil shale 400°C soak to accelerate alteration reaction on kerogen then rapid heating to 560°C to observe oil remaining.

Test Series C

Utah oil shale 400°C soak to accelerate alteration reaction on kerogen then rapid heating to 560°C to observe oil remaining.

Note – Fushun, Estonia, and Australia results are similar





Top Vapour Discharge on Slow Soak Test Units





Bottom Vapour Discharge on Slow Soak Test Units



Test Series D tests were carried but with vapour bottom discharge to eliminate effects of oil condensing and refluxing back into hot oil shale charge. Only Jordan and Utah oil shales tested to date.

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Alberta Taciuk Process (ATP) Technology



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Alberta Taciuk Process (ATP) Technology



Conclusions to Date Based on Results of UMATAC Tests

- 1) Jordan, Utah, Estonia, China, and Australia oil shales have very similar time, temperature, product composition characteristics.
- 2) Measurable quantities of oil and gas released at 300°C.
- 3) Kerogen decomposition becomes rapid beyond 320°C.
- 4) Kerogen decomposition accelerates as temperature is increased.
- 5) Inefficient removal of oil and gas products (oil reflux) remarkably reduces oil yield.

Conclusions to Date Based on Results of UMATAC Tests

- 6) During kerogen decomposition the oil quality and the off gas composition trends are remarkably similar for different oil shales.
- 7) During soak time secondary alteration reactions occur which tend to reduce oil yield tending towards ultimate coking.
- 8) Long term soak periods of 60 day duration with reflux reduce oil yield by 50 to 80% referenced to Modified Fischer assay.



Comments by Author

UMATAC's technical paper searches regarding this type of slow soak research has turned up very little information on oil yields, gas yields and product comparison under our test conditions. The presentation of our test methods, our test data, and findings is to stimulate discussion with other researchers in aid of understanding the complex nature of oil shale kerogen.

Comments by Author

The author recognizes that UMATAC's test programs were performed under conditions using a prepared crushed oil shale and operating at atmospheric pressure. Based on the conclusions reached in this paper regarding kerogen alternation, reaction and the effect of reflux, the author has a much better appreciation of the complexity of modeling, designing and operating a large scale in-situ type oil shale operation where the additional factors of pressure, viscosity, bed permeability, heat transfer co-efficient, potential for multiple refluxing of oil products, catalytic action and potential for gasification are at least some of the additional considerations.

The oil resources are there in the oil shales but collectively we need to develop means to recover this oil in a safe, environmentally friendly, and economic fashion.



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