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FOR PARTIAL OXIDATION OF CATTLE
FEEDLOT MANURE

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AICHE 84th National Meeting
Atlanta, Georgia

February 26 - March 1, 1978

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Thermochemical conversion of biomass offers an opportunity to increase the world's reserves of hydrocarbon resources (1, 2, 3). Biomass can be defined as solar energy stored in the form of plant matter and residues that have some positive value as a chemical resource. This is in contrast to wastes which have a negative value, e.g. - one must pay to dispose of wastes. Biomass is a very desirable hydrocarbon resource because it is renewable and thus, in theory, should provide an inexhaustable supply. The major obstacle to the implementation of biomass technology is that the energy density of biomass is very low compared to fossil fuels as shown below. Projections for silvaculture are that 8 tons/acre-yr. of wood could be produced. If

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one assumes that wood has a heating value of 6,000 BTU/lb, the energy density would be 92×10^6 BTU/acre.

$$\frac{8 \text{ tons}}{\text{Acre-yr}} \mid \frac{2000 \text{ lb}}{\text{ton}} \mid \frac{6000 \text{ Btu}}{\text{lb}} = 96 \times 10^6 \frac{\text{BTU}}{\text{Acre-yr}}$$

On the other hand, consider an oil reservoir that is ten feet thick, with a porosity of 25% and oil saturation of 65%. The energy density in this case is 53×10^9 BTU/acre.

$$\frac{10 \text{ ft}}{\text{Acre}} \mid \frac{43,560 \text{ ft}^2}{\text{Acre}} \mid \frac{0.25 \text{ ft}^3 \text{ void}}{\text{ft}^3 \text{ formation}} \mid \frac{0.65 \text{ ft}^3 \text{ oil}}{\text{ft}^3 \text{ void}} \mid \frac{50 \text{ lb oil}}{\text{ft}^3 \text{ oil}} \mid \frac{15000 \text{ BTU}}{\text{lb}} =$$

$$53 \times 10^9 \frac{\text{BTU}}{\text{Acre}}$$

One location where biomass can be found in large quantities is in a cattle feedlot. Feedlots containing 50,000 to 100,000 cattle are relatively common in some parts of the country, including West Texas (3). Each cow produces six to eight pounds per day of manure which means that a large quantity of manure is available in one location. This manure is currently used for fertilizer, but the cost of transporting and applying the manure restricts its use as fertilizer to areas near the feedlot.

In 1971, a project was initiated at Texas Tech University to develop methods to convert cattle manure to more valuable products that could be easily transported. The process which has emerged is thermochemical gasification (5). The process is designated the Synthesis Gas From Manure (SGFM) process. Construction of a 1/2 ton per day pilot plant to evaluate the SGFM process began in 1974 with the support of the U.S. Environmental Protection Agency, Pioneer Corporation and the Texas Cattle Feeders

Association. Operation of the pilot plant began in 1975. On September 1, 1976, the U. S. Energy Research and Development Administration contracted with Texas Tech to develop a data base for partial oxidation and pyrolysis of manure in the SGFM pilot plant that could be used as a basis for a commercial scale plant. This work was part of the Fuels From Biomass program, ERDA and was designated Phase I when the contract was extended on September 1, 1977. Phase II of the program is now in progress to develop a similar data base for wood and wood residue as well as investigation of other agricultural residues (sorghum stalks, corn stalks, etc.).

This paper presents the results of Phase I of the project now funded by the U. S. Department of Energy. A large amount of data is presented with very little explanation. Analysis of the data is in progress. One segment of Phase II will be to develop a kinetic model to correlate the results presented in this paper. A preliminary kinetic model has been presented previously (9) as has a detailed description of the SGFM process (10).

SGFM Process Overview

A schematic diagram of the pilot-scale processing apparatus is given in Figure 1. Solid feed is fed to the top of the reactor and falls counter-current to a gas mixture of steam, air, and product gases. Product gas from pyrolysis and partial oxidation exit (with input fluids) out the top of the reactor. The product gas is stripped of entrained solids, tar, and a water/organic mixture using a cyclone, impinger, and heat exchanger respectively. Char is removed from the bottom of the reactor through a center-port opening in the inlet gas distributor.

FIGURE 1: FLOWSHEET OF SGFM EXPERIMENTAL UNIT

A typical temperature profile along the reactor length is shown in Figure 2. This graph shows the effect of cool solids entering the top of the reactor as well as the effect of combustion or partial oxidation in the lower part. The graph also demonstrates that severe heat losses are currently encountered in the reactor. This heat-loss feature was retained during Phase I until sufficient data were obtained to help explain an unexpected high concentration of hydrocarbon in the product gas.

Operation of the reactor system begins with heat-up to the desired operating temperature. Our results show that the best data (heat balance, mass balance, product composition) are obtained when the heat-up continues to about six (6) hours. At this time, the metal has reached a thermal steady-state. After reaching this thermal steady-state, feed is begun and another steady-state is reached after one to two hours. Only after the second steady-state, are data recorded. Data are generally obtained over a one to three hour period.

Phase I Results and Discussion

A. Mass Balance Data

Examples of mass balance data that have been obtained during Phase I are shown in Table I. These results show that mass balances in excess of 90% have been obtained on a regular basis. This improvement over early balances of 75-85% has been due, in large part, to extended durations of both the thermal (reactor metal) steady-state and the feed steady-state. These better mass balance data do not show any major shifts in yield(s) over previous results; but, the results can now be used with a greater degree of confidence in making economic estimates.

FIGURE 2: EXAMPLES OF REACTOR TEMPERATURE PROFILES

TABLE I

EXAMPLE MASS BALANCE DATA

(All data represent individual experimental measurements; product gas was calculated from experimental gas composition data and measured volumetric flow rate; each run preceded by one hour of operation with feed.)

	22	23	25	27	28	29	30	32
DURATION (min)	136	156	60	61.7	72	30	75	48.6
INPUT (Kg)								
1. Wet Manure	34.868	37.577	13.617	15.177	19.813	9.163	21.555	12.932
2. Air	6.028	8.396	2.200	2.812	3.279	1.406	9.702	5.566
3. Steam	7.111	15.331	5.987	6.387	5.443	2.790	5.697	3.679
4. Total Input	48.007	61.304	21.804	24.376	28.535	13.359	36.954	22.177
OUTPUT (Kg)								
1. Produce Gas	21.160	19.119	4.640	8.605	11.099	3.221	14.896	10.405
2. Water + Organics	20.185	26.649	9.149	8.039	8.931	3.875	8.251	5.615
3. Tar	1.510	1.565	0.839	0.352	0.805	0.363	0.718	0.943
4. Cyclone Fines	0.794	1.474	0.399	1.021	0.635	1.388	1.542	1.139
5. Char	9.594	10.251	5.216	4.921	5.352	3.973	3.425	2.041
6. Total Output	53.243	59.058	20.244	22.938	26.823	12.820	28.838	20.143
% MATERIAL BALANCE								
100 x (Output - Input) ÷ Input	+10.91	-3.66	-7.16	-5.9	-6.00	-4.04	-21.98	-9.20

The results still suggest that all of the mass output is not being collected. The discrepancy is estimated to reside in two measurement areas: (1) the product tar is difficult to collect and probably precipitates/condenses with other material on pipe walls; (2) gas composition analysis ignores acetylene and hydrocarbon greater than C2's.

A complete set of carbon balance are not available due to CHN-instrument problems. The limited data that are available do suggest that Figure 3 for earlier data are still valid.

B. Heat Balance Data

Heat balance data required additional studies beyond mass balance results because the reactor system heat losses had to be determined independently. The heat losses from the existing system are shown in Figure 4. These data were obtained using one to four electric heaters (no control) with air alone (•) or air plus steam feed (•). The total gas throughput was comparable to that observed during actual operation. During a run with manure feed, actual heat losses exceeded 3000 kcal/hr.

Figure 4 shows that these heat loss data are internally consistent because the data can be extrapolated to a zero-zero intercept. These same data were used to calculate a convective heat transfer coefficient after correcting for radiant losses. The average, convective heat transfer coefficient was estimated to be $0.93 \text{ cal/hr} \cdot \text{cm}^2 \cdot \Delta^\circ\text{C} \pm 16\%$ (approximately $1.9 \text{ BTU/hr} \cdot \text{ft}^2 \cdot \Delta^\circ\text{F}$) which is within acceptable engineering limits, considering the non-uniformity of insulation, estimated radiation losses, and conduction losses through connecting piping and superstructure. Hence, the heat loss data of Figure 4 are estimated to be accurate even though precision

FIGURE 3: DISTRIBUTION OF CARBON IN PRODUCTS.
SOLID LINES INDICATE DATA RANGE.

Figure 4: Experimental Heat Loss Data for SGFM Reactor

cannot be considered to be any better than $\pm 5\%$ (± 200 kcal/hr at 4000 kcal/hr loss rate).

A summary of the heat balance data using cattle feedlot manure as feed are shown in Table II. It should be noted that the data represented here also include the mass balance of Table I. The total ΔH_r (= Input - Output) are shown in Figure 5 as a function of air feed rate.

These results indicate that the overall reaction with manure feed is mildly exothermic with $\Delta H_r = -122 \pm 55$ cal per gram of dry, ash-free material converted ($\Delta H_r = -220 \pm 99$ BTU/lb dry, ash-free material converted). An average value of $-\Delta H_r = 94$ cal/gram was obtained if the heat of combustion equivalent to $C + O_2 \longrightarrow CO_2$ was subtracted from each of the runs in Table II. The ΔH_r values given here are based on a temperature of 25°C.

It must be appreciated that the $(-\Delta H_r)$ value is considered to be only an order-of-magnitude estimate. The value represents only 5-15% of the total heat input or output. In other words, the differences between heat input and heat output that were used to calculate the $(-\Delta H_r)$ to approach the error limits of the individual values in each run. This limitation is particularly true at the lower reaction rates. A value of approximately 120 cal/gram is thought to be sufficiently close to zero to be consistent with other data reported in the literature.

The heat balance results do show, in general, that the SGFM partial oxidation can be considered to be autothermal if proper and efficient heat exchange can be accomplished between the inlet and exit streams with minimum heat losses. This summation is illustrated in Table III for 80% exchange between the inlet and outlet streams with 10% heat losses. The excess heat

TABLE II
HEAT BALANCE RESULTS

RUN NO.	25	27	28	29	30	32
INPUT(a)						
1. Manure, as-received	0	0	0	0	0	0
2. Air	508	686	718	512	1389	1070
3. Steam	16380	17315	12742	15375	12707	12022
4. Helium	0	0	0	0	0	0
5. Heaters	<u>20404</u>	<u>28566</u>	<u>27729</u>	<u>23884</u>	<u>23761</u>	<u>27630</u>
INPUT TOTAL	37292	46567	41189	39771	37857	40722
OUTPUT(a)						
1. Losses(b)	15300	19000	19500	16750	16300	18800
2. Steam	20395	21350	16910	20100	17195	17188
3. Product Gases	4050	10029	11352	8670	17439	22033
4. Char	1324	1429	1331	2018	696	496
5. Fines	139	350	201	876	418	533
6. Tar	984	404	766	804	664	1438
7. Liquids	1621	293	1331	500	487	2511
8. Helium	<u>1367</u>	<u>2734</u>	<u>1467</u>	<u>1531</u>	<u>1647</u>	<u>1983</u>
OUTPUT TOTAL	45180	55589	52858	51249	54846	64982
% Loss = $\frac{\text{Loss}}{\text{Input}} \times 100$	74.99	66.51	70.32	70.13	68.60	68.04
TOTAL ΔHr = INPUT - OUTPUT	-7888	-9022	-11669	-11478	-16989	-24260

(a) 25°C REFERENCE; BTU/HR

(b) FROM CALIBRATION DATA

(c) 8.9% MOISTURE EXCEPT RUN 30 WHICH WAS 21.7%

(d) $C + O_2 \longrightarrow CO_2$

FIGURE 5: ESTIMATE OF HEAT OF REACTION

TABLE III
EXAMPLE CALCULATIONS SHOWING AUTOTHERMAL
CHARACTER OF SGFM TECHNOLOGY

	Run 25	Run 32
INPUT (BTU)		
Manure (Wet; 10% H ₂ O)	0	0
Air	508	1070
Steam	<u>16380</u>	<u>12022</u>
OUTPUT (BTU)		
Steam	20395	17188
Gases	4050	22035
Char + Fines	1463	1029
Tar + Liquids	<u>2605</u>	<u>3949</u>
10% Heat Losses of Output	(2851)	(4420)
80% Efficiency of Output	(5703)	(8840)
Net Output	19959	30941
Net Output - Input	3071	17849
(ΔH) Water @ Conditions	1238 BTU/LB	1238 BTU/LB
Potential Added Water	2.48 lb	14.4 lb
Potential Moisture Content	23%	57%

for the calculations show that moisture content of the feed could be as high as 50% even with the assumed losses.

These estimates do not mean that external heat would not be necessary. Instead the results suggest that external heat duty could be kept to a minimum. Hence, overall process thermal efficiency should be comparable to those reported for coal processes, 60-70%.

C. Solid Feed and Product Data

It is realized that the above heat and mass balance data can only be put in their proper context when the feed is characterized. Figures 6 and 7 show examples of typical product size distribution and heat content. In Figure 6, the results show that the overall heat content of the as-received feed decreases with decreasing particle size. However, on a dry, ash-free basis, the results of Figure 7 suggest that the reactive material in the manure feedstock does not change drastically as a function of particle size, i.e., the heat of combustion is relatively constant. Furthermore, the results also suggest that both "fines" and larger particles exhibit a reasonable degree of reaction, approximately 30% of the material in the fines converted and up to 90% of the bulk feed were converted in runs 25 thru 32. Overall conversion of reactive material ranged from 65% to 90%, based on ash data for these runs.

Complete results on the C, H, N, O, S content of the solid feed and product are not yet available. Current results show, however, that feed material was comparable to that reported earlier: 35-45% C, 5-6% H, 2.5-3.0% N, 0.5-1.5% S, and 24-28% O on a dry basis with 15-30% ash.

Figure 6: Gross Heat of Combustion Data For Cattle Feedlot Manure Feedstock

Figure 7: Heat of Combustion Data for Cattle Manure on Dry, Ash-Free Basis

The same data also illustrate that particle size of the solid does not change much during the course of reaction. There does, however, appear to be a small shift towards smaller particle sizes as illustrated in Table IV. The approximate solid conversion (based on ash content) for each particle size range is also shown in the same table and suggests that particles > 2.0 mm (> 0.10 inch) should be minimized in a full-scale process. Additional data, particularly for Runs 30 and 32 at higher, overall conversion levels, on conversion versus particle size remain to be summarized and will be reported.

Additional data on overall, fractional conversion of solid material is given in Table V. These results help demonstrate that a relatively high degree of solid conversion has been achieved during investigations using cattle feedlot manure. The 93% solid conversion is expected to be approaching the upper limit because the entrainment of fines (< 0.01 inch or 0.03 cm) during reaction.

D. Gas Yield and Composition

The data base developed on the conversion of cattle feedlot manure in the SGFM reactor is given in Table VI. This table illustrates the range of operating conditions as well as product gas data. An overall interpretation of these data is given later relative to total conversion and synthesis gas generation.

Internal reactor gas compositions were also obtained for four of these runs (19 thru 23). These results were obtained to develop a basic understanding of the partial oxidation-pyrolysis reactions and fluidization characteristics occurring within the reactor and provide data for developing scale-up principles and/or guidelines.

TABLE IV
COMPARISON OF PARTICLE SIZE DISTRIBUTION
OF FEED AND CHAR

ITEM	Screen Diameter, mm				
	0-0.7	0.71-1.0	1.01-1.41	1.42-2.0	>2.0
Wt % in Feed	36.8	13.4	14.0	16.2	19.6
Wt % in Char, Run 22	35.5	16.8	16.4	17.1	14.3
Wt % in Char, Run 23	32.9	15.5	15.4	21.5	14.9
Approximate Solid Conversion					
Run 22	0.70	0.65	0.59	0.54	0.37
Run 23	0.70	0.66	0.59	--	0.45

TABLE V
FRACTIONAL CONVERSION OF REACTIVE MATERIAL IN SOLID FEED

<u>Run Number</u>	<u>% Ash in Feed</u> (α)	<u>% Ash in Char</u> (X_c)	<u>% Ash in Fines</u> (X_f)	<u>lb fines per lb char</u> (a)	<u>Fractional Conversion</u> (f)
25	0.1886	0.4590	0.6700	0.0781	0.7424
27	0.2024	0.5395	0.6715	0.2081	0.8024
28	0.2144	0.4195	0.6180	0.1167	0.6529
29	0.1918	0.5870	0.6028	0.3472	0.8358
30	0.1957	0.7090	0.6735	0.4552	0.8946
32	0.1876	0.8047	0.6488	0.4745	0.9261

Notes: (a) All values on a dry basis

(b) $1 - f = \alpha(1 - \beta)/\beta(1 - d)$

$\beta = (a X_f + X_c)/(1 + a_1)$

TABLE VI
OPERATING CONDITIONS AND PRODUCT GAS DATA

Operating Conditions	Run Number						
	1	6a	6b	7	8	9	10
Manure Feed Rate, lb daf/hr ^(a)	11.5	15.9	35.6	28.6	29.2	28.0	18.2
Manure Feed Rate, lb ar/hr	17.1	23.3	52.0	41.8	39.8	39.7	25.9
Air Feed Rate, SCF/hr	40.5	60.0	52.5	67.2	49.5	150	27.0
Steam Feed Rate, lb/hr	12	10	10	8	6.8	6	8.2
Average Temperature, °C	670	655	600	575	590	630	590
Product Gas Data ^(b)							
Total Dry Gas, SCF/lb daf ^(c)	19.1	(10.7)	9.3	6.5	7.3	(11.5)	5.1
Heat Value (HHV), BTU/SCF	321	328	426	380	396	295	376
Gas Composition, Vol %							
H ₂	25.2	22.2	20.0	28.2	17.4	15.1	20.9
N ₂	14.6	27.8	15.1	23.2	26.7	36.8	24.2
CH ₄	12.8	7.7	12.6	9.2	14.1	8.9	11.7
CO	11.6	15.3	21.3	16.4	21.2	20.3	22.4
CO ₂	30.8	20.7	22.1	15.4	14.1	14.2	14.8
C ₂ H ₄	4.7	6.4	8.5	4.9	5.8	4.2	5.5
C ₂ H ₆	0.3	0.5	0.4	2.7	0.7	0.5	0.5

(a) daf = dry, ash-free; ar = as-received

(b) All data are average values from at least two samples. Individual gas samples were analyzed on the gas chromatograph using at least two injections.

(c) Values in parentheses are back-calculated values using a nitrogen balance.

TABLE VI (continued)

Operating Conditions	Run Number										
	19	20	21	22	23	25	27	28	29	30	32
Manure Feed Rate, lb daf/hr ^(a)			24.54	23.57	23.31	22.18	23.65	26.05	29.75	23.94	26.03
Manure Feed Rate, lb ar/hr	33.3	32.9	33.4	33.9	31.86	30.0	32.56	36.4	40.4	38.02	35.17
Air Feed Rate, SCF/hr	66	63.6	70.2	72.6	74	60.2	74.7	77.9	26.6	212.0	188
Steam Feed Rate, lb/hr	10	14	10.6	12.5	14	13.21	14.1	12	12.3	10.0	10
Average Temperature, °C			596	629	577	517	619	629	533	573	636
Product Gas Data ^(b)											
Total Dry Gas, SCF/lb daf ^(c)			10.2	12.60	10.25	6.20	12.35	13.36	8.10	19.67	19.66
Heat Value (HHV), BTU/SCF	321.8	325.3	351.1	335.8	338.9	270.9	309.6	300.7	326.9	265.31	268.6
Gas Composition, vol %											
H ₂	24.75	26.44	21.04	23.78	17.66	3.68	21.87	24.60	30.0	30.93	32.92
N ₂	21.59	20.55	17.78	18.84	23.16	22.65	22.35	24.00	16.10	23.00	21.23
CH ₄	9.11	9.04	9.34	9.82	10.69	8.15	8.27	8.76	9.20	6.27	6.19
CO	11.69	13.07	13.23	14.56	13.56	10.03	13.84	14.57	13.0	11.63	11.05
CO ₂	27.02	25.41	30.72	27.14	28.00	39.56	27.87	23.79	27.0	25.13	25.57
C ₂ H ₄	5.20	4.86	6.98	5.25	6.13	4.37	5.0	3.61	3.34	2.13	2.26
C ₂ H ₆	0.65	0.63	0.88	0.611	0.79	1.56	0.8	0.67	1.36	0.91	0.78

(a) daf = dry, ash-free; ar = as-received

(b) All data are average values from at least two samples. Individual gas samples were analyzed on the gas chromatograph using at least two injections.

(c) Values in parentheses are back-calculated values using a nitrogen balance.

A qualitative summary of the internal compositions is given in Figure 8. An interpretation of these data have indicated that:

- Approximately 50-75% of the pyrolysis reactions occur in the lower one-third of the reactor. This was somewhat unexpected because previous "projections" led us to conclude that most of the pyrolysis would occur in the upper one-half. Heat transfer to the larger particles may explain part of this observation. The results suggest that it may be possible to reduce the required reactor volume using somewhat smaller feed particle size and/or providing a better match of temperature profiles.
- Liquid (tar) generation was greatest in the middle of the reactor volume (region of higher temperatures). However, little liquid tar was observed in the very top and bottom of the reactor. These results indicate, along with the individual composition data, that tars should be injected into the middle of the reactor.
- Solid density was highest in the lower part of the reactor. This was an expected result. This observation and the above on liquid formation were qualitatively established by the amount collected in the sample probes.
- Data analysis showed that, at the existing concentration, as much as 40% of the ethylene could be decomposing within the reactor. This value is high, but would be much higher if steam were not present. The presence of steam as a diluent

Figure 8: Internal Net Rates of Production

reduces the rate of decomposition by a factor of 3.5 based on concentration alone and also reduces the residence time at temperature by a factor of 2. Knowledge of the decomposition of ethylene and similar hydrocarbons is important because these components, even in dilute concentrations, are important, e.g.,

$$\text{HHV ethylene/HHV Hydrogen} = 4.94$$

$$\text{HHV ethylene/HHV Methane} = 1.58$$

Or, 5% ethylene is equivalent to 24.7% hydrogen or 7.91% methane in terms of overall heat content of the gas. One goal of thermochemical conversion should, accordingly, be to minimize the decomposition of these hydrocarbons.

- Gas velocity increased by a factor of approximately 2 in the lower part of the reactor (22 cm/sec to 42 cm/sec) and then decreased about 50% in the upper portion of the reactor. Gas phase residence time in the reactor was estimated to be 7.6 seconds. These results were developed at the conditions of run 3, but would apply to the others in this series (19-23). Gas velocity changes would be more dramatic at higher conversion levels, such as runs 30 and 32. Gas velocity changes in the absence of steam are indicated by the nitrogen concentration profile in Figure 9. The higher velocities help minimize decomposition of valuable hydrocarbons as discussed above.

The overall results of these internal composition data are still under review because they provide a firm contribution to the understanding

Figure 9: Nitrogen Concentration Along Axial Reactor Length

of the reactions and events that are occurring. These data and others will be used to help develop a reactor design model in Phase II.

E. Conversion Efficiency

The heating value of the produced gas as a function of conversion is shown in Figure 10. The conversion shown is an indication of thermal efficiency and is given as a ratio of the total heat content of gas produced to the heat content of the dry, ash-free feed, based on higher heating value. The early data (Runs 1, 6b, 7, 8, 10) are not as reliable as the later values due to poor material balances. Figure 11 shows the same data if the CO_2 is removed from the gas. With CO_2 removal, the gas produced has a HHV that is consistently greater than 300 BTU/SCF.

Conclusions

Results of the pilot plant tests on cattle manure demonstrate the technical feasibility of the SGFM process. A subcontract has been awarded to the Bechtel Corporation to perform a preliminary process design and economic evaluation of the SGFM process to produce an ammonia synthesis gas or an ammonia synthesis gas plus ethylene from 1000 tons per day of oven-dry cattle feedlot manure. This study will be completed by September 1, 1978 and should provide a good assessment of the economic viability of thermochemical conversion processes.

Acknowledgements

The authors wish to thank the U. S. Department of Energy (Contract No. EY-76-S-04-3779), the Pioneer Corporation and the Texas Cattle Feeders Association for supporting this project.

Figure 10: Heating Value of Raw Gas As A Function of Conversion

Figure 11: Heating Value of CO_2 -Free Gas as a Function of Conversion

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