

MASTER

Contract No. DEAC01-78ET10325

CRYOGENIC METHANE SEPARATION/CATALYTIC  
HYDROGASIFICATION PROCESS ANALYSIS

Quarterly Report

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## I. OBJECTIVE AND SCOPE OF WORK

The objective of this extension of DOE contract No. DEAC01-78ET10325 (formerly ET-78-C-01-3044), "Cryogenic Methane Separation/Catalytic Hydrogasification Process Analysis", is to perform trade-off and optimization studies for the Rockwell/Cities Service Short Residence Time Hydrogasification (SRTTH) and the Exxon Catalytic Coal Gasification (CCG) processes in the acid gas removal and cryogenic separation areas. The contract extension is divided into nine (9) subtasks. Each subtask studies the effect of variation of a key design parameter on the treatment cost of the SNG produced. All subtasks will be conducted under the Task I scope of the original DOE contract No. ET-78-C-01-3044, which includes block flow sheet, overall heat and material balance, utility summary, four-line equipment description, investment and treatment cost summaries and final report writing in addition to monthly and quarterly reports.

The period of performance for the contract extension is nine (9) months, starting 21 July 1980.

## II. PROGRAM PLAN AND SCHEDULE

Since the submission of the contract extension proposal in the winter of 1979, both Exxon and Rockwell made a number of significant changes to the base-case design of their gasification processes. These changes made some of the subtasks in the original proposal for contract extension no longer entirely relevant to the development of Rockwell's SRTTH process and Exxon's CCG process.

After reviewing the scope of the work with Rockwell and Exxon, a revised program plan, reflecting the latest needs of Rockwell and Exxon, was developed and is shown in Attachment I. The overall intent, cost and schedule of the original scope are preserved. It is believed the revised plan will utilize DOE's funding in the most productive and cost effective manner. The program schedule was also updated and is shown in Attachment I. The revised Program Plan and Schedule were submitted to DOE in September, 1980 for review. Although APCI has not received DOE's formal approval for the proposed changes, work is proceeding in accordance with this plan.

## III. ROCKWELL/CITIES SERVICE SRTTH PROGRESS

A block diagram and mass balance for the acid gas removal and cryogenic methane separation modules developed by the Lummus Company were received from Rockwell on 30 September 1980 (Attachment II). The process scheme shown in the block diagram consists of an acid gas removal module followed by a cryogenic separation unit which produces SNG, recycle hydrogen, and light aromatics (benzene, toluene, xylene) streams. A methanation unit is located downstream of the cryogenic unit. This scheme is different from the one selected by APCI in the original contract study. In APCI's scheme, the BTX in the raw feed gas was removed in an oil scrub unit before the feed enters the acid gas removal unit and the methanation unit was placed upstream of the cryogenic unit.

For Subtasks 1 and 2 of this contract extension, APCI will accept Lummus' scheme as the base design case and perform an independent technical and economic assessment of the scheme.

Subtask 1 will begin in November 1980 and Subtask 2 will start in February, 1981.

#### IV. EXXON CCG PROGRESS

A block flow diagram for the integrated Selexol acid gas removal and cryogenic methane separation process is shown in Figure I. Stream numbers shown on the flow diagram are used in the tables of the overall material balance for the Tasks 3, 4, and 5.

##### A. Subtask 3 - Recycle Gas Purity

Heat and material balances and compressor train simulations for the cryogenic methane separation unit have been completed for two different  $H_2/CO$  recycle compositions. The percentage of methane in this  $H_2/CO$  recycle was reduced from the base 10% to 8% and 5%. Overall material balances for both cases are shown in Tables I and II. Both cases appear feasible; however, the 5% case approaches size limitations on the vacuum stage of the SNG compressor. A double-cased compressor is needed.

The colder temperatures required for better methane separation were obtained by lowering the J-T pressure of a portion of the SNG product. In addition, the 5% case required expansion of the high pressure  $H_2/CO$  recycle gas from 390 to 110 psia.

Using the preliminary process designs developed by the Process Engineering Department and the same economic criteria used in the previous estimate, the Economic Evaluation Department is evaluating the cost of gas treating for the two alternate cases. The result will be compared with the cost of the base design case documented in the final report of DOE contract No. ET-78-C-01-3044. This subtask will be completed in early November.

##### B. Subtask 4 - $H_2$ and CO in SNG

Heat and material balances and compressor train simulations for the cryogenic methane separation unit have been completed. The percentage of CO in the SNG product was raised to 0.5% by increasing the SNG stripper pressure and the cold flash temperature. The amount of hydrogen in the product remained negligible. The overall material balance for this case is shown in Table III.

Power costs are 1-2% lower than the base design, and cooling water flows are reduced 8%. Economic evaluation will begin soon.

#### C. Subtask 5-- CH<sub>4</sub> loss in Selexol

Process work has been completed for two alternate cases: Case 1 (0.54% CH<sub>4</sub> loss) and Case 2 (0.086% CH<sub>4</sub> loss). Overall material balances for both cases are shown in Tables IV and V. The purity of the treated gas has been kept almost the same for these cases as in the base case (0.32% CH<sub>4</sub> loss) in the final report of the original DOE work to facilitate comparisons.

Preliminary results indicate the Case 1 power savings are insufficient to pay for the additional lost methane, relative to the base case. In Case 2, the reverse was true and the CH<sub>4</sub> savings were not enough to cover increased power costs. Neither case shows any advantage over the base case in the preliminary results. This finding will be confirmed when the Economic Evaluation Department completes the cost analysis in November.

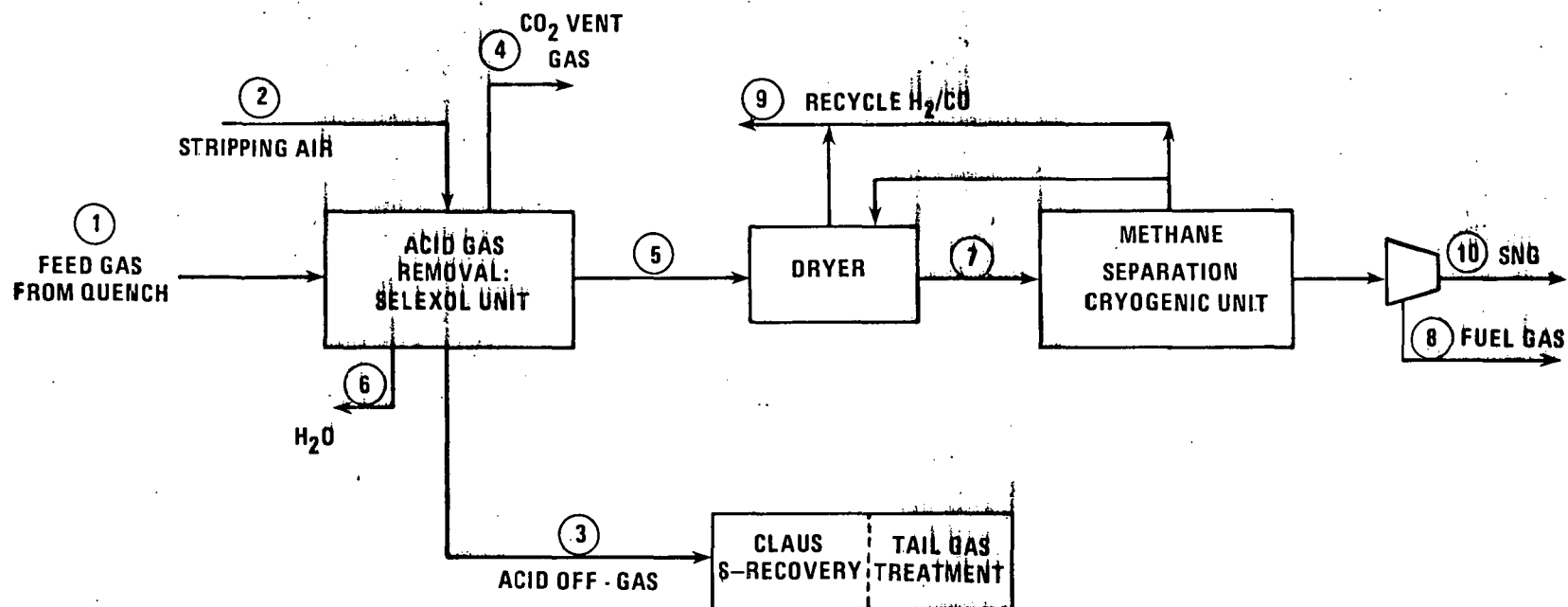
#### D. Subtask 8 - Cursory Evaluation for New Feed Condition at 250 psig

APCI received a simplified block flow diagram and material balance from Exxon on 4 September for the case of gasifier operation at 250 psig (Attachment II). The ratio of CO<sub>2</sub> to H<sub>2</sub>S remains at about 25; the same as that in the base design case when the gasifier operates at 500 psig. However, the partial pressure of the total acid gas is only about one-third of that in the base design case. The low acid gas partial pressure would make some of the chemical absorption processes more attractive. APCI will perform a cursory evaluation, comparing the gas treating cost of the base case with low pressure alternatives. The effect of feed pressure on the performance of the cryogenic methane separation unit will also be studied. This task will begin at the end of November.

#### V. SCHEDULE FOR BALANCE OF PROGRAM

Due to a late start and some scope changes, the program fell two to three weeks behind schedule during the first quarter. However, since Rockwell and Exxon have both agreed upon the proposed changes and have provided the necessary process information for the revised tasks, it is expected that the program will be brought back on schedule in the next quarter.

DOE/EXXON CCG  
FIG. 1: SELEXOL AND CRYOGENIC INTEGRATION SCHEME





**TABLE I**  
**DOE/EXXON CCG**  
**OVERALL MATERIAL BALANCE**  
**METHANE SEPARATION UNIT**  
**CASE 1 - 8% CH<sub>4</sub>**

STREAM NO.		7	8	9	10
STREAM NAME		TREATED GAS	FUEL GAS	RECYCLE H <sub>2</sub> /CO	SNG PRODUCT
PRESS, PSIA		411	45	600	1,015
TEMP., °F		4.8	162.5	173.3	120
LB-MOLE/HR	CO	10,505.0	2.1	10,464.4	38.5
	H <sub>2</sub>	37,671.5		37,671.5	
	CH <sub>4</sub>	35,536.4	1,610.7	4,601.3	29,424.4
	C <sub>2</sub> H <sub>6</sub>	4.0	2		3.8
	N <sub>2</sub>	3,585.6		3,585.3	.3
	CO <sub>2</sub>	4.5	2		4.3
	H <sub>2</sub> O				
	TOTAL	87,307.0	1,613.2	56,222.5	29,471.3
TOTAL, LBS/HR		1,040,982	25,913	641,713	473,356

**TABLE II**  
**DOE/EXXON CCG**  
**OVERALL MATERIAL BALANCE**  
**METHANE SEPARATION UNIT**  
**CASE 2 - 5% CH<sub>4</sub>**

STREAM NO.		7	8	9	10
STREAM NAME		TREATED GAS	FUEL GAS	RECYCLE H <sub>2</sub> /CO	SNG PRODUCT
PRESS, PSIA		411	45.0	600.0	1,015
TEMP., °F		4.8	162.4	293	120
LB-MOLE/HR	CO	10,505.0	2.5	10,453.8	48.7
	H <sub>2</sub>	37,671.5		37,671.5	
	CH <sub>4</sub>	35,536.4	1,610.3	2,751.8	31,174.3
	C <sub>2</sub> H <sub>6</sub>	4.0	2		3.8
	N <sub>2</sub>	3,585.6	.02	3,585.3	.3
	CO <sub>2</sub>	4.5	20		4.3
	H <sub>2</sub> O				
	TOTAL	87,307.0	1,613.2	54,462.4	31,231.4
TOTAL, LBS/HR		1,040,982	25,913	513,359	501,710

**TABLE III**  
**DOE/EXXON CCG TASK 4—**  
**METHANE SEPARATION UNIT**  
**OVERALL MATERIAL BALANCE**

STREAM NO.		7	8	9	10
STREAM NAME		TREATED GAS	FUEL GAS	RECYCLE H <sub>2</sub> /CO	SNG PRODUCT
PRESS, PSIA TEMP., °F		411 4.8	45 105	600 158	1,015 120
LB-MOLE/HR	CO	10,505.0	8.0	10,357.9	139.1
	H <sub>2</sub>	37,671.5		37,671.5	
	CH <sub>4</sub>	35,536.4	1,604.8	5,857.1	28,074.5
	C <sub>2</sub> H <sub>6</sub>	4.0	2		3.8
	N <sub>2</sub>	3,585.6	.1	3,584.1	1.4
	CO <sub>2</sub>	4.5	2		4.3
	H <sub>2</sub> O				
	TOTAL	87,307.0	1,613.3	57,470.6	28,223.1
TOTAL, LBS/HR		1,040,982	25,977	560,445	454,560

**TABLE IV**  
**DOE/EXXON CCG TASK 5 - SELEXOL**  
**ACID GAS REMOVAL**  
**CASE 1 - 0.54% METHANE LOSS**  
**OVERALL MATERIAL BALANCE**

STREAM NO.		1	2	3	4	5	6
STREAM NAME		FEED GAS TO SELEXOL	STRIPPING AIR	ACID OFF-GAS TO CLAUS	CO <sub>2</sub> VENT	TREATED GAS	CONDENSATE
PRESS., PSIA TEMP., °F		450 120	14.7 60	25 120	16 3	411 8	25 120
LB-MOLE/HR	CO	10,510.0		4.2	4.7	10,501.1	
	H <sub>2</sub>	37,671.0		1.8	0.4	37,668.8	
	CH <sub>4</sub>	35,657.0		53.1	139.8	35,464.1	
	C <sub>2</sub> H <sub>6</sub>	6.0		0.1	1.0	3.9	
	N <sub>2</sub>	3,585.0	18,813.1		18,813.1	3,585.0	
	O <sub>2</sub>		5,008.5		5,008.5		
	CO <sub>2</sub>	21,633.0	7.1	1,546.0	19,989.3	4.1	
	CDS	2.0		0.4	1.6	< 0.8 ppm	
	H <sub>2</sub> S	878.0		877.9	0.1	< 0.3 ppm	
	H <sub>2</sub> O	416.0	381.3	191.0	22.3	2.6	581.5
TOTAL		110,357.0	24,210.0	2,774.5	43,980.8	87,230.2	581.5
TOTAL, LBS/HR		2,032,494	694,699	106,793	1,570,162	1,639,767	10,471

**TABLE V**  
**DOE/EXXON CCG TASK 5 - SELEXOL**  
**ACID GAS REMOVAL**  
**CASE 2 - 0.086% METHANE LOSS**  
**OVERALL MATERIAL BALANCE**

STREAM NO.		1	2	3	4	5	6
STREAM NAME		FEED GAS TO SELEXOL	STRIPPING AIR	ACID OFF-GAS TO CLAUS	CO <sub>2</sub> VENT	TREATED GAS	CONDENSATE
PRESS., PSIA TEMP., °F		450 120	14.7 60	25 120	16 0	411 8	25 120
LB-MOLE/HR	CO	10,510.0	-	0.5	0.7	10,508.8	
	H <sub>2</sub>	37,671.0	-	0.2	0.4	37,670.4	
	CH <sub>4</sub>	35,657.0	-	8.0	22.6	35,626.4	
	C <sub>2</sub> H <sub>6</sub>	6.0	-	0.4	0.6	4.0	
	N <sub>2</sub>	3,585.0	20,789.4	-	20,789.4	3,585.0	
	O <sub>2</sub>	-	5,526.3	-	5,526.3	-	
	CO <sub>2</sub>	21,633.0	7.9	781.0	20,855.8	4.1	
	CDS	2.0	-	0.3	1.7	-	
	H <sub>2</sub> S	878.0	-	878.0	-	< 0.3 ppm	
	H <sub>2</sub> O	416.0	421.2	123.6	20.9	2.2	690.5
TOTAL		110,357.0	26,744.8	1,792.0	47,218.4	87,400.9	690.5
TOTAL, LBS/HR		2,032,494	767,406	66,679	1,678,222	1,642,559	12,440

## A T T A C H M E N T I

- PROGRAM PLAN
- PROGRAM SCHEDULE

## PROGRAM PLAN

The following tasks describe the study for the Rockwell Hydro-gasification Process and the trade-off and optimization study for the Exxon process in the acid gas removal and cryogenic separation areas. All subtasks will be conducted under current Task I scope of DOE contract DEAC01-78ET10325 (formerly ET-78-C-01-3044). Task I scope includes block flow sheet, overall heat and material balance, utility summary, four-line equipment description, investment and treatment cost summaries and final report writing in addition to monthly and quarterly reports.

### Rockwell

#### Subtask 1 - Cryogenic Separation Evaluation (Tentative: Subject to DOE Approval)

A Task 1 type cursory evaluation will be performed using Rockwell's revised feed gas. The revised feed gas is at about 1,000 psig and contains benzene. The Rockwell/Lummus process block configuration will be accepted, with the cold box producing SNG, recycle hydrogen and benzene. Methanation will be considered downstream of the cold box. This evaluation will provide an independent technical and economic assessment of the Rockwell/Lummus separation requirement.

#### Subtask 2 - Acid Gas Removal Evaluation (Tentative: Subject to DOE Approval)

A Task 1 type cursory evaluation will be performed using Rockwell's revised feed gas, as in Task 1. The potential systems for acid gas removal will be reviewed and a gas treatment cost estimated. This evaluation will provide an independent technical and economic assessment of the Rockwell/Lummus acid gas removal requirement.

### Exxon

#### Subtask 3 - Recycle Gas Purity

A Task 1 type evaluation will be performed of cryogenic separation with the recycle gas purity increased by changing from the base 10% CH<sub>4</sub>, to tentatively 8% and 5%. This evaluation would allow Exxon to consider the tradeoff in cold box compression costs vs. fuel, thermal efficiency, and yield in the gasifier as a function of CH<sub>4</sub> content in the recycle gas.

#### Subtask 4 - H<sub>2</sub> and CO in SNG

A cursory study of the effect of permitting up to 5% H<sub>2</sub> and 0.5% CO in the SNG product to evaluate the potential for cryogenic savings will be made. A descriptive report would be submitted.

#### Subtask 5 - CH<sub>4</sub> Loss in Selexol

A Task 1 type evaluation of Selexol acid gas removal with the CH<sub>4</sub> (syngas) loss higher and lower than the base loss, 0.32%, will be made. Studies of 0.5 and 0.1% CH<sub>4</sub> losses are planned. This evaluation will allow APCI to show the tradeoff in power consumption vs. CH<sub>4</sub> loss.

#### Subtask 6 - Fuel Gas Regeneration of Adsorbers

Per Exxon's request, the adsorber reactivation scheme would be revised to replace electric preheat with gas fired preheat. Summary utility requirements and economics will be reported.

#### Subtask 7 - Two Train Design

The original APCI Task 1 design for acid gas removal and cryogenic separation will be scaled from four to two trains. Summary economics and technical comments will be reported. This will allow Exxon to merge APCI economics with their own overall economics developed on a two train basis.

#### Subtask 8 - Cursory Evaluation for New Feed Condition at 250 psig

Exxon's revised CCG feed gas at about 250 psig will be considered for acid gas removal and cryogenic separation. In the base case, the raw feed gas will be compressed to about 450 psig (corresponding to the CCG reactor at about 500 psig) before treatment with Selexol and cryogenic methane separation. Alternative schemes of treatment at 250 psig will then be evaluated. This cursory evaluation will compare the gas treating cost of the base case with the low pressure alternatives. The effect of an intermediate pressure will also be discussed in the report.

#### Subtask 9 - Technical Support of Exxon PDU and Report Writing

Technical support will be provided as needed for the start-up of the cryogenic separation unit at Exxon's 1 TPD PDU at Baytown. Air Products' process and start-up experience will provide technical consultation and start-up assistance to troubleshoot the cryogenic unit of the PDU. Preparation of Technical Progress Reports and the Final Report will be performed.

# PROGRAM SCHEDULE

MONTHS	0	1	2	3	4	5	6	7	8	9
DATE	23 JUL. 1980	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN 1981	FEB.	MAR.	23 APR. 1981

SUBTASK NO.

1



2



3



4



5



6



7



8



9

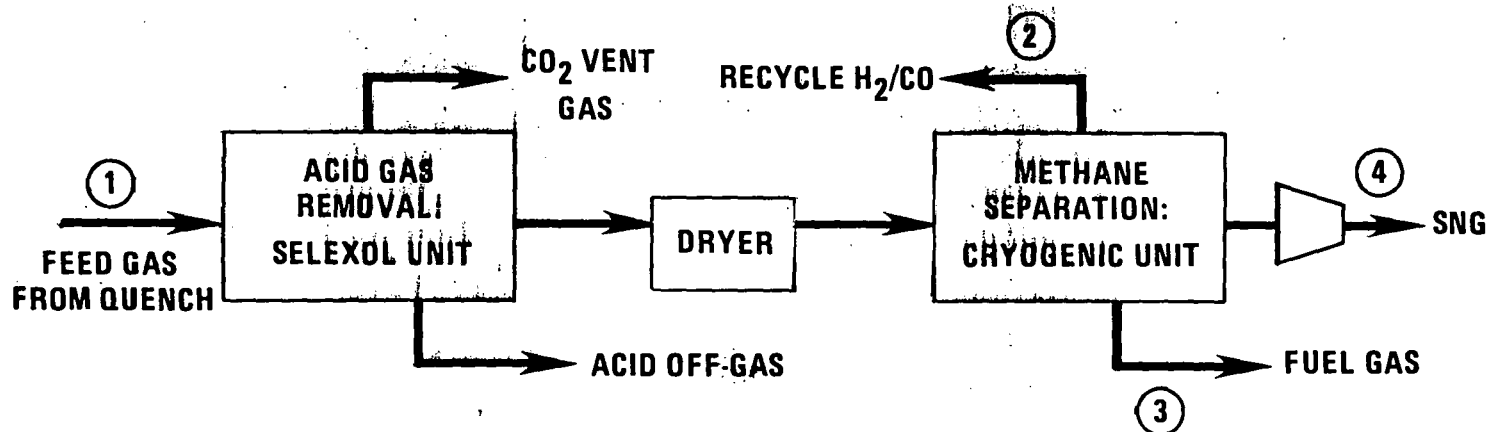


## **ATTACHMENT II**

- NEW MATERIAL BALANCE FOR EXXON CCG AT 250 PSIG**
- REVISED MATERIAL BALANCE FOR ROCKWELL/CITIES  
SERVICE SRTH AT 1,000 PSIG**

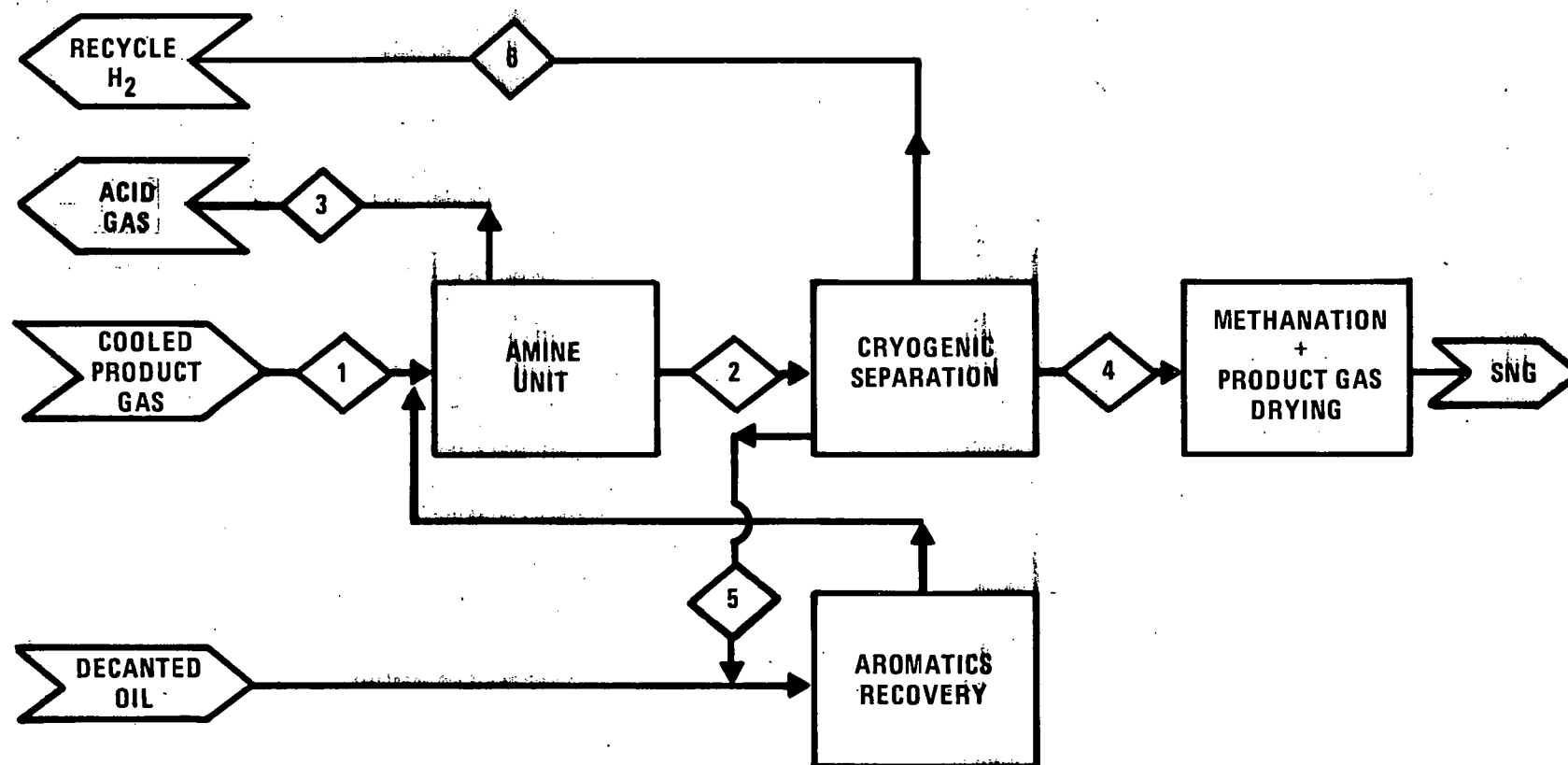


# **EXXON CCG** **PROCESS INFORMATION FOR 250 PSIG CCG** **GASIFIER CASE EVALUATION**



STREAM NUMBER		1	2	3	4
TEMPERATURE, °F		120	175	105	120
	PRESSURE, PSIA	220	345	45	1,015
FLOWS: (LB-MOLES/HR.)	CO	15,893	15,857	2	28
	CO <sub>2</sub>	21,656	59	—	—
	H <sub>2</sub>	57,225	57,225	—	—
	CH <sub>4</sub>	38,653	8,731	1,499	28,292
	C <sub>2</sub> H <sub>6</sub>	5	—	—	3
	H <sub>2</sub> S	878	—	—	—
	N <sub>2</sub>	5,494	5,494	—	—
	H <sub>2</sub> O	416	16	—	—
TOTAL		140,220	87,382	1,501	28,323

**FIGURE 1. CITIES SERVICE/ROCKWELL HYDROGASIFICATION PROCESS:  
HYDROGASIFIER EFFLUENT GAS PROCESSING SCHEME**



E. OR J. NO. \_\_\_\_\_  
 DATE \_\_\_\_\_  
 SHEET 1 OF \_\_\_\_\_  
 BY \_\_\_\_\_

TABLE 1  
 ROCKWELL/CITIES SERVICE SRTH  
 THE LUMMUS CO.  
 MATERIAL BALANCE  
 SIMPLIFIED OVERALL MATERIAL  
 BALANCE - SCHEME 3  
 (TASK 5.9)

STREAM COMPONENT	1		2		3		4		5 (NOTE 1)		6	
	COOLED GAS		GAS AFTER AGR		ACID GAS FROM AMINE UNIT (NOTE 1)		CH <sub>4</sub> RICH GAS		BTX RECOVERED FROM CRYOGENIC UNIT		RECYCLE H <sub>2</sub>	
	LB-MOLE HR.	MOL. %	LB-MOLE HR.	MOL. %	LB-MOLE HR.	MOL. %	LB-MOLE HR.	MOL. %	LB-MOLE HR.	MOL. %	LB-MOLE HR.	MOL. %
H <sub>2</sub>	165145.4	83.31	165145.4	83.88			7450	21.22			157695.4	97.73
CH <sub>4</sub>	25428.	12.83	25428.	12.88			24510	69.81			918.0	.87
C <sub>2</sub> H <sub>6</sub>	88.8	.04	88.8	.04			88.8	.25			—	
CO	4140.0	2.09	4140.0	2.10			2413.3	6.87			1726.7	1.07
CO <sub>2</sub>	175.8	.09	9.7	49 PPM	166.1	19.43					9.7	.01
N <sub>2</sub> + Ar	1595.0	.80	1595.0	.81			648.0	1.85			947.0	.59
NH <sub>2</sub>												
H <sub>2</sub> S	643.2	.32	1.0	5 PPM	642.2	75.14	—				1.0	—
C <sub>6</sub> H <sub>6</sub>	750.0	.38	735.0	.37	15.0	1.76			733.0	76.98	2.0	—
C <sub>7</sub> H <sub>8</sub>	3.5	—	3.5	—					3.1	0.33	0.4	—
C <sub>8</sub> H <sub>10</sub>	73.7	.04	73.7	.04					71.6	7.52	2.1	—
C <sub>10</sub> H <sub>8</sub>	0.7	—	0.7	—					0.7	.07	—	—
H <sub>2</sub> O	198.0	.10	198.0	.10	31.4	3.67			143.8	15.10	54.2	.03
TOTAL	198242.1	100.00	197418.8	100.00	854.7	100.00	35110.1	100.00	952.2	100.00	161356.5	100.00
PRESSURE (PSIA)	942		932		26		1070		920		1200	
TEMP. (°F)	100		100		100		390		45		150	
MW	5.07		5.03		36.19		14.15		—		2.54	

FIGURE 12

NOTE (1) THESE STREAMS WILL CONTAIN SMALL QUANTITIES OF H<sub>2</sub>, CH<sub>4</sub> & CO  
 HOWEVER FOR THIS BALANCE THESE QUANTITIES HAVE BEEN NEGLECTED.

RECEIVED BY TIC

DEC 8 1980