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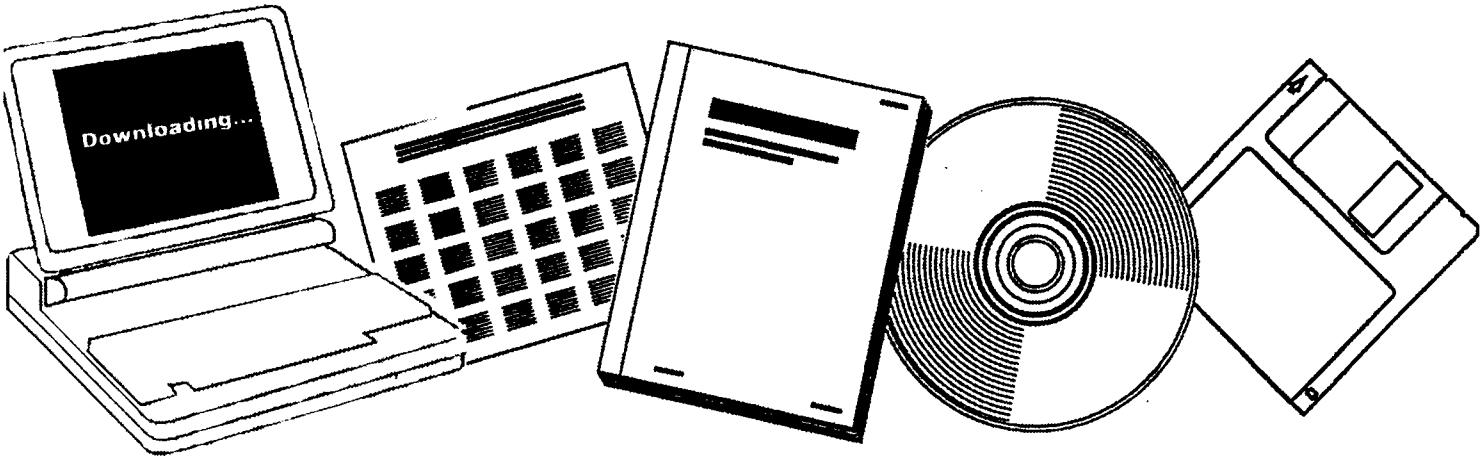

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By
David V. Nakles
Michael J. Massey
Albert J. Forney
William P. Haynes

Date Published - December 1975

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INFLUENCE OF SYNTHANE GASIFIER CONDITIONS ON EFFLUENT AND PRODUCT GAS PRODUCTION

by

David V. Nakles[1], Michael J. Massey[2]
Albert J. Forney[3], and William P. Haynes[4]

SUMMARY

Objectives and Experimental Procedure

During steam-oxygen gasification of coal by the Synthane Process and others, substantial quantities of foul condensate and sulfur-bearing char and tar are produced. Little is known quantitatively either about steady state rates of production of various gasifier effluents or about the relative effects of process variables in coal gasification on the types and rates of their production. Utilizing the Pittsburgh Energy Research Center's (PERC) 4-inch diameter Synthane gasifier, the present experimental program was initiated to study quantitatively the nature of effluent production and the various gasifier process variables which affect it. Included in the investigation were studies of:

(1) Rates of production of various gasifier effluents as a function of time from gasifier startup to shutdown;

(2) Effects of selected process variables on the rate of production of various effluents;

(3) Associated effects of changes in these variables on yields of total product gas and equivalent methane (methane plus twice the ethane production); and

(4) Impacts of changes in selected process variables on the composition and physical properties of char and tar produced.

To minimize experimental complications, non-caking North Dakota lignite coal was used exclusively in the 19 gasification tests conducted during this investigation. Process variables, e.g., fresh coal heatup rate, product gas residence time, reaction temperature, and the extent of gas-solid contacting, were varied by altering the fresh coal injection position and their impact on effluent production was monitored. During any given test, condensible hydrocarbons and contaminated water were separated from raw producer gas continuously;

[1]Chemical engineer, PERC and graduate student, Carnegie-Mellon University.
[2]Assistant Professor, Chemical Engineering and Public Affairs, Carnegie-Mellon University.
[3]Research Supervisor, PERC.
[4]Supervisory chemical engineer, PERC.

at 45 minute intervals, accumulated aqueous and hydrocarbon condensate were withdrawn from condensers, weighed, and sampled for analysis. Non-condensible product gas production and composition were monitored at 30 minute intervals throughout each run. Gasifier char was collected in a batch reservoir during each run and sampled once for analysis.

Major Findings

(1) Effluent production rates, viz., weight of condensed hydrocarbon "tars" and water soluble contaminants, vary significantly with time from gasifier startup to shutdown:

(a) With the exception of the free fall injection tests, effluent production rates are initially high, but decline rapidly, typically approaching steady state levels within 2 to 3 hours of reactor startup.

(b) In the free fall injection tests, steady state effluent production rates consistently exceeded startup rates, typically by factors of two or more.

(2) In shifting from free fall to shallow to deep bed-injection of coal into the gasifier, both steady state tar and measured aqueous effluent production rates, viz., phenol, chemical oxygen demand, total organic carbon, cyanide and thiocyanate concentrations (ppm) decrease by factors of as much as one thousand.

(3) Counterintuitively, neither total product gas nor equivalent methane production is affected by coal injection geometry, at least over the range of configurations investigated.

(4) Similarly, ammonia yields during gasification appear to be essentially independent of coal injection geometry.

(5) Sulfur contents of chars produced from free fall and shallow bed-injections of coal are consistently below the New Source Performance Standard (NSPS) of 1.2 lbs SO_2/MMBTU. Observed sulfur contents of deep bed-injection chars exceed the NSPS; however, problems with experimental conditions are believed to account for this discontinuity in the data.

(6) Regardless of coal injection geometry, the sulfur content of tar is consistently above the NSPS for liquid fuels of 0.8 lbs SO_2/MMBTU. However, limited data do indicate that this sulfur content declines significantly with gasifier operating time, apparently approaching a steady state level of 1.0 lbs SO_2/MMBTU or less.

(7) Over the range of coal-injection geometries studied, the bulk composition of condensed hydrocarbon "tars" are remarkably similar; specific gravities consistently exceed 1.0, and the initial boiling points of properly dehydrated samples average ~150°C indicating the absence of any light hydrocarbon fractions, particularly benzene, toluene, or xylene (BTX). It should be noted, however, that product gas was not analyzed for variations in BTX content.

General Conclusions

Three critical conclusions regarding effluent production and treatment in Synthane as well as other steam-oxygen gasification systems are apparent:

(1) Steady state effluent production rates differ dramatically from those during non-steady state gasifier operation, particularly reactor startup.

(2) Process variables influenced by coal-injection geometry, e.g., coat heatup rate, product gas residence time, reaction temperature, and gas-solid contacting have a significant impact on steady state gasifier effluent production.

(3) Neither total product gas nor equivalent methane yield are affected by significant variation in coal-injection location.

The first conclusion suggests that a substantial amount of existing data on effluent production during steam-oxygen gasification must be regarded as qualitatively accurate at best, since it was acquired largely by batch sampling effluents collected during startup and shutdown as well as during steady state operation of small-scale reactors. The second and third conclusions indicate strongly that effluent treatment by modified gasification reactor design represents at least a complement and quite possibly an alternative to large-scale treatment of gasifier effluents by conventional processing.

INTRODUCTION

Background

In support of its Synthane process development program, the Pittsburgh Energy Research Center (PERC) has been conducting coal gasification experiments in a 4-inch diameter fluid bed reactor since 1969. As part of this program, PERC has identified and measured a number of effluents produced during the gasification of three different coals*. Typical compositions of each of these coals is presented below:

Coal	Moisture wt %	Vol Matter wt %	Ultimate Analysis, wt %					
			C	H	O	N	S	Ash
North Dakota	12.2	37.0	56.4	5.2	28.6	0.8	0.8	8.2
Lignite	18.4	35.4	53.8	5.5	32.3	0.8	0.8	6.8
Illinois No. 6	8.0	37.0	65.1	5.2	14.3	1.2	3.5	10.7
	-	-	62.5	5.2	15.6	1.1	3.5	12.1
Pittsburgh Seam	2.5	31	68.4	4.7	9.3	1.2	1.3	15.1
	1.9	33.5	72.1	5.0	9.3	1.3	1.5	10.8

A complete summary of gasifier operating statistics for the 16 North Dakota lignite (non-caking coal), 64 Illinois No. 6 (highly caking coal), and 3 Pittsburgh Seam (highly caking coal) steam-oxygen gasification experiments in which effluent measurements have been made is presented here in Table I. Steady state product gas and various batch (collections from gasifier startup to shutdown) effluent production statistics for each of these tests are presented in Table II. Also noted in this table is the coal injection geometry utilized in each test; coal was either allowed to free-fall through the reactor's carbonization zone to the fluidized bed (procedure in the majority of the tests) or the carbonizer was bypassed and coal was injected directly into the top of the fluidized bed section of the reactor. In each of the experiments cited, three distinct effluent streams were monitored: (1) unreacted solids (char) containing carbon, ash, and variable amounts of sulfur; (2) condensible hydrocarbons, largely high molecular weight sulfur-bearing "tars" (2.9 to 184 lb/ton coal, MAF): and (3) aqueous condensate containing substantial quantities of phenol (3000-8000 ppm), ammonia (6000 to 32,000 ppm), chemical oxygen demand (COD from 9000 to 39,000 ppm). Note

*A brief report summarizing preliminary findings regarding tar production and composition, the types and quantities of contaminants in water condensate, and the disposition of heavy metals has been published.[1]

Table I. Operating Statistics, Past Synthane Steam-Oxygen Gasification Trials: North Dakota Lignite, Illinois No. 6, and Pittsburgh Seam Coal. Basis: Reactor Pressure, 40 atm.

Trial[a]	Date	Gasifier Reactor Feedrates, #/hr			Fluid Bed Temperature, °C			Reactor Operating Time, hrs		% Reactant Conversion		Partial Coal Anal., wt%	
		Coal	Steam	Oxygen	Bottom	Average	Top	Total	Steady State[b]	Steam	Carbon	H_2O	Ash
						NORTH DAKOTA LIGNITE COAL							
CHPL-1	03-27-73	36.97	20.16	4.72	864	848	736	3.50	2.50	37.9	59.0	23.0	7.2
CHPL-2	03-29-73	36.75	19.49	5.89	907	871	680	4.16	3.00	51.3	75.1	28.0	6.5
CHPL-3	06-11-73	36.17	29.85	6.36	890	870	661	4.25	2.00	44.1	70.7	11.4	9.2
CHPL-4	06-13-73	36.68	25.47	4.67	883	844	697	4.50	2.50	37.0	58.9	21.1	8.1
CHPL-5	06-15-73	36.09	25.02	5.12	876	846	734	4.50	2.00	50.6	67.3	21.0	8.5
CHPL-6	06-19-73	36.43	25.69	4.78	877	843	717	4.50	3.50	42.3	60.2	21.3	9.1
CHPL-7	06-22-73	32.37	25.19	4.14	870	855	735	4.41	3.00	51.4	73.5	20.3	10.2
CHPL-8	06-26-73	36.73	26.04	4.42	951	843	622	4.42	2.50	23.1	50.8	21.7	9.1
CHPL-9	06-29-73	36.90	26.04	5.89	877	850	691	4.42	3.00	46.0	71.0	20.6	8.3
CHPL-10	07-02-73	36.00	25.91	2.45	872	828	541	4.50	2.00	6.0	32.0	21.6	8.3
CHPFL-104	02-26-74	30.01	30.42	7.25	868	838	706	5.33	2.00	43.9	83.4	19.0	8.3
CHPFL-105	03-01-74	29.72	28.37	4.87	791	771	665	4.00	2.00	32.5	82.5	16.4	8.5
CHPFL-106	03-05-74	30.01	29.86	5.63	832	818	682	5.33	2.50	32.8	72.7	14.1	8.9
CHPFL-107	03-07-74	30.00	32.68	5.19	862	818	665	5.50	2.50	39.9	83.7	17.5	8.5
CHPFL-111	03-19-74	32.51	36.63	6.61	861	811	706	5.00	3.50	30.4	77.1	12.5	10.3
CHPFL-112	03-29-74	27.82	39.60	5.64	857	857	681	5.50	2.50	26.9	71.7	12.6	10.8
						ILLINOIS NO. 6 COAL							
CHPFI-2	04-24-72	19.40	20.10	5.66	872	913	862	3.33	1.50	--	78.4	3.5	14.5
CHPFI-3	04-26-72	20.40	20.10	4.58	952	929	883	5.00	2.00	--	70.1	4.7	11.9
CHPFI-10	05-12-72	22.70	25.10	3.70	839	906	871	4.67	1.50	17.5	57.2	7.2	11.6
CHPFI-16	05-31-72	17.70	20.10	3.96	950	885	818	3.67	1.00	--	53.0	8.0	11.9
CHPFI-17	06-05-72	18.90	22.60	4.85	857	914	880	5.50	2.00	19.2	59.5	7.9	12.2
CHPFI-19	06-09-72	17.00	22.60	3.32	883	893	863	3.00	.50	16.5	60.7	7.4	16.9
CHPFI-22	06-20-72	18.20	22.60	6.09	517	807	770	6.78	2.00	10.6	69.0	7.8	11.0
CHPFI-23	06-22-72	19.50	22.60	4.72	943	902	814	6.00	2.50	--	--	7.8	11.0
CHPFI-24	06-26-72	20.50	22.60	4.82	565	854	874	5.75	2.00	14.6	62.9	7.8	11.0

Footnotes:

(a) Abbreviations CHPL and CHPFL both refer to reaction in a high pressure fluidized bed equipped with a carbonization zone (See Figure 3).

(b) That period during which the product gas H_2 concentration was approximately constant.

Table I continued

Trial[a]	Date	Gasifier Reactor Feedrates, #/hr			Fluid Bed Temperature, °C			Reactor Operating Time, hrs		% Reactant Conversion		Partial Coal Anal., wt %	
		Coal	Steam	Oxygen	Bottom	Average	Top	Total	Steady State[b]	Steam	Carbon	H_2O	Ash
CHPFI-26	06-30-72	17.80	20.80	5.41	966	910	869	6.28	2.00	25.0	74.1	7.6	17.8
CHPFI-27	07-10-72	21.20	22.60	6.00	660	882	870	5.65	1.00	20.5	61.1	5.8	8.7
CHPFI-28	07-12-72	20.90	22.60	6.56	400	851	879	6.00	2.00	29.2	78.5	5.2	11.5
CHPFI-30	07-18-72	20.60	22.60	6.06	935	901	830	4.10	.50	18.5	60.4	2.8	12.5
CHPFI-34	08-16-72	20.60	22.60	5.36	945	898	841	6.00	1.50	19.7	61.0	3.0	12.3
CHPFI-38	09-07-72	18.50	22.60	6.59	490	793	795	5.00	1.00	13.2	62.8	8.9	12.0
CHPFI-41	10-13-72	20.10	22.60	4.78	448	830	810	5.33	1.00	20.8	68.6	5.3	13.3
CHPFI-42	10-17-72	19.90	22.60	6.60	965	931	808	4.67	2.50	26.7	82.4	3.2	13.6
CHPFI-43	10-19-72	23.10	22.60	5.27	967	928	777	5.67	2.00	22.9	70.7	3.2	13.6
CHPFI-45	11-06-72	15.30	22.60	6.52	932	897	705	5.00	1.00	21.2	84.2	2.7	16.6
CHPFI-47	11-13-72	23.80	22.60	4.35	753	870	862	5.00	1.00	18.6	61.0	5.3	14.3
CHPFI-49	12-21-72	21.50	22.60	4.95	953	891	701	5.00	2.00	11.8	53.9	5.3	13.8
CHPFI-50	01-09-73	19.99	22.60	4.59	988	887	660	4.33	1.00	--	36.9	6.9	10.0
CHPFI-53	01-17-73	20.00	22.60	3.41	968	897	665	4.67	1.00	6.7	36.2	6.4	10.7
CHPFI-54	01-09-73	18.10	22.60	5.41	505	772	753	5.00	2.00	24.8	80.1	6.5	11.6
CHPFI-55	01-23-73	21.40	22.60	6.69	850	874	729	3.25	1.50	19.2	72.1	5.6	14.5
CHPFI-56	01-29-73	23.30	22.60	7.14	543	774	695	3.00	1.00	10.1	60.2	5.6	15.4
CHPFI-59	02-15-73	20.70	22.60	5.71	872	881	728	3.75	1.00	21.1	65.3	6.1	11.4
CHPFI-61	02-26-73	22.10	22.60	5.81	940	889	748	5.00	1.50	16.9	54.7	1.1	11.4
CHPFI-62	02-28-73	21.60	22.60	5.13	941	891	740	3.33	1.00	14.2	56.1	3.5	11.3
CHPFI-63	03-02-73	25.03	22.60	5.92	941	883	779	5.00	1.50	13.1	51.3	2.6	11.7
CHPFI-64	03-06-73	26.30	22.60	6.20	938	877	747	5.33	2.00	20.6	60.3	3.1	16.0
CHPFI-65	03-08-73	22.70	22.60	6.00	934	879	758	5.50	2.50	22.8	61.4	3.0	13.6
CHPFI-70	04-02-73	26.90	22.60	5.91	945	877	730	5.00	1.00	19.3	52.2	2.4	15.7
CHPFI-73	04-24-73	24.20	19.42	5.10	947	915	731	5.00	3.00	24.0	49.0	2.5	18.3
CHPFI-74	04-27-73	25.70	18.91	5.10	946	912	766	5.00	3.00	27.8	56.0	4.5	19.5
CHPFI-75	05-03-73	25.60	19.82	5.50	940	903	758	5.00	2.50	23.7	47.0	3.4	13.7
CHPFI-76	05-10-73	26.50	17.83	5.33	925	869	746	3.00	3.00	21.2	46.0	2.9	13.6
CHPFI-77	05-23-73	22.70	21.89	5.66	941	904	737	5.92	2.00	32.3	61.0	3.0	14.1
CHPFI-78	05-30-73	24.10	29.93	5.95	953	911	755	5.00	2.50	25.0	54.0	2.7	12.6
CHPFI-79	06-01-73	25.60	27.42	5.40	940	896	727	5.00	2.50	17.0	42.0	3.7	12.2
CHPFI-80	07-05-73	26.80	23.90	5.10	940	895	744	5.00	2.00	13.7	42.0	8.2	11.3
CHPFI-81	07-11-73	23.50	27.74	6.15	929	892	738	3.50	2.00	10.2	45.0	8.1	12.1

Table I continued

Trial[a]	Date	Gasifier Reactor Feedrates, #/hr Coal	Steam	Oxygen	Fluid Bed Temperature, °C Bottom	Average	Top	Reactor Operating Time, hrs Total	Steady State[b]	% Reactant Conversion Steam	Carbon	Partial Coal Anal., wt % H_2O	Ash
CHPFI-82	07-20-73	27.30	26.18	7.15	940	886	732	4.55	2.50	15.0	45.0	8.2	12.0
CHPFI-83	07-24-73	25.90	25.12	6.61	960	902	668	5.00	4.00	19.8	54.0	8.0	14.4
CHPFI-84	07-26-73	25.10	24.96	6.24	962	900	678	4.25	3.50	20.0	53.0	8.3	13.0
CHPFI-85	07-31-73	25.40	24.82	6.21	960	900	722	5.00	4.50	13.6	54.0	8.1	14.8
CHPFI-90	08-14-73	24.61	24.62	6.11	933	921	765	5.00	2.50	25.5	56.5	7.7	14.7
CHPFI-91	08-28-73	20.21	23.35	5.71	882	906	782	6.75	3.50	27.2	63.2	7.8	10.4
CHPFI-92	09-05-73	18.90	23.45	6.43	873	908	785	7.00	2.50	27.8	69.3	7.7	11.3
CHPFI-93	09-11-73	26.87	25.40	7.51	956	927	758	4.00	2.50	24.1	58.6	7.4	13.8
CHPFI-94	09-14-73	25.96	27.02	7.22	962	938	723	5.13	4.50	24.9	62.2	7.0	13.2
CHPFI-95	09-18-73	25.86	27.92	7.31	983	924	677	5.00	2.50	22.7	59.5	6.4	13.4
CHPFI-96	09-20-73	20.98	22.28	6.50	939	909	765	5.93	4.00	29.9	66.7	6.9	11.2
CHPFI-97	09-26-73	26.43	23.52	5.91	940	908	775	5.00	2.50	22.1	51.6	7.2	13.4
CHPFI-98	10-18-73	21.54	27.92	7.21	646	937	795	5.00	3.00	28.3	76.6	5.6	17.3
CHPFI-99	10-25-73	24.80	28.02	8.21	729	919	791	5.00	3.50	23.2	56.0	3.2	14.9
CHPFI-100	10-29-73	24.88	29.93	5.60	950	906	805	5.00	4.50	17.0	49.8	8.2	13.4
CHPFI-101	11-05-73	20.67	30.03	4.90	955	909	825	5.00	2.00	19.7	59.6	5.3	15.5
CHPFI-102	11-07-73	20.16	34.43	5.96	955	914	835	5.00	2.50	19.6	72.4	2.8	15.6
CHPFI-103	11-09-73	20.23	34.23	5.71	956	918	848	5.00	3.00	18.6	69.1	4.1	11.9
CHPFI-115	05-10-74	19.00	29.93	5.23	690	884	790	3.50	.50	15.7	57.0	1.8	13.2
CHPFI-116	05-14-74	19.62	26.52	6.61	825	893	718	5.00	1.50	9.7	51.8	4.3	12.2
CHPFI-117	05-16-74	18.42	27.70	7.51	953	921	826	5.33	2.50	20.0	62.1	2.8	12.3
CHPFI-118	05-21-74	16.67	23.38	6.46	952	908	760	5.50	1.50	15.6	62.0	6.0	12.8
						PITTSBURGH SEAM COAL							
CHPFP-86	08-02-73	20.30	24.86	7.29	675	903	751	4.67	3.00	23.7	57.0	2.5	15.1
CHPFP-87	08-06-73	26.40	22.32	5.50	915	895	720	5.00	2.00	6.5	35.0	1.9	11.0
CHPFP-88	08-08-73	22.60	27.12	7.31	932	912	797	5.00	3.00	19.7	50.0	1.9	10.8

Table II. Product Gas and Liquid Effluent Statistics, Past Steam-Oxygen Gasification Trials: North Dakota Lignite, Illinois No. 6, and Pittsburgh Seam Coal. Basis: Reactor Pressure, 40 atm.

Trial	Date	Coal Injection[a] Position		Coal Feed Rate, #/hr, MAF	Condensibles, #/ton coal, MAF		Partial Composition of Condensate, ppm					Gas Production, SCF/lb C Gasified	
		FFI	SBI		Condensate	Tar	Phenol	COD	NH_3	SCN^-	CN^-	Equiv CH_4[b]	Product Gas[c]
					NORTH DAKOTA LIGNITE COAL								
CHPL-1	03-27-73	x		25.81	1830	15.9	3300	22,000	8000	32	.100	6.23 ± .21	28.24 ± .63
CHPL-2	03-29-73	x		24.07	1721	47.6	3200	22,000	9000	22	.100	6.15 ± .22	31.91 ± .18
CHPL-3	06-11-73	x		28.72	1656	60.9	6600	39,000	7000	22	.100	5.24 ± .33	28.47 ± .64
CHPL-4	06-13-73	x		25.97	1872	15.8	--[e]	--	--	--	--	6.84 ± .67	30.84 ± 2.30
CHPL-5	06-15-73		x	25.44	1743	5.0	8000	11,000	8400	13	.100	7.53 ± .43	34.94 ± 2.30
CHPL-6	06-19-73		x	25.36	1807	27.9	3000	10,000	8000	16	≤.006	6.81 ± .22	31.51 ± 1.30
CHPL-7	06-22-73		x	22.50	1957	29.0	--	--	--	--	--	7.71 ± .65	33.92 ± 3.20
CHPL-8	06-26-73	x		25.42	2102	52.6	--	--	--	--	--	6.07 ± 1.05	26.04 ± 3.90
CHPL-9	06-29-73	x		26.24	1790	73.6	--	--	--	--	--	7.39 ± .30	29.22 ± .29
CHPL-10[g]	07-02-73	x		25.24	2564	72.7	--	--	--	--	--	5.11 ± .05	22.06 ± .19
CHPFL-104	02-26-74	x		21.82	2358	41.3	5000	22,000	7000	4	.400	7.46 ± 1.50	36.50 ± 8.70
CHPFL-105	03-01-74	x		22.32	2153	60.3	3325	15,000	6900	4	.040	3.41 ± .67	14.52 ± 3.40
CHPFL-106	03-05-74	x		23.11	2129	15.2	3250	17,000	7200	4	.060	7.32 ± .63	32.07 ± 2.40
CHPFL-107	03-07-74	x		22.20	2246	26.7	3900	15,000	5700	4	.003	6.87 ± .20	29.19 ± 1.10
CHPFL-111	03-19-74	x		25.10	2425	40.2	4120	16,000	31,900	4	.020	5.51 ± .08	25.84 ± 1.10
CHPFL-112	03-29-74	x		21.31	3035	14.2	--	--	--	--	--	6.01 ± .22	30.87 ± 1.20
					ILLINOIS NO. 6 COAL								
CHPFI-2	04-24-72	x		15.9	1760	121.9	--	--	--	--	--	6.63 ± .10	26.29 ± 1.96
CHPFI-3	04-26-72	x		17.0	1737	107.4	--	--	--	--	--	7.32 ± .13	25.60 ± .69
CHPFI-10	05-12-72	x		18.4	1981	122.3	--	--	--	--	--	6.07 ± .17	19.27 ± .14
CHPFI-16	05-31-72	x		16.1	2049	116.8	--	--	--	--	--	6.12 ± .05	18.68 ± .36
CHPFI-17	06-05-72	x		15.1	2409	106.0	--	--	--	--	--	5.88 ± .10	20.78 ± .90

Footnotes:

(a) FFI = Free Fall Injection; SBI = Shallow Bed-Injection (See Figure 3).

(b) Equiv. CH_4 = sum of SCF of CH_4 plus twice the SCF of C_2H_6.

(c) Product Gas = sum of SCF of CH_4, C_2H_6, H_2, and CO.

(d) Additives (e.g., limestone, dolomite, hydrated lime, & quicklime) blended with coal in an effort to enhance gasification activity.

(e) A dash in a data space indicates information was not collected during the trial.

(f) Product gas passed over a Co-Mo Catalyst prior to analysis.

(g) Gasification was completed with no bed accumulation (e.g. the entire reactor operated in a free fall mode).

Table II continued

Trial	Date	Coal Injection[a] Position FFI	Coal Injection[a] Position SBI	Coal Feed Rate, #/hr, MAF	Condensibles, #/ton coal, MAF Condensate	Condensibles, #/ton coal, MAF Tar	Partial Composition of Condensate, ppm Phenol	COD	NH_3	SCN^-	CN^-	Gas Production, SCF/lb C Gasified Equiv CH_4[b]	Product Gas[c]
CHPFI-19[d]	06-09-72	x		12.9	2645	78.1	--	--	--	--	--	5.26 ± 0.00	20.02 ± 0.00
CHPFI-22	06-20-72	x		15.3	2886	99.1	--	--	--	--	--	4.97 ± .43	21.31 ± 2.00
CHPFI-23	06-22-72	x		16.0	2521	107.7	--	--	--	--	--	6.60 ± .44	24.30 ± 1.30
CHPFI-24	06-26-72	x		16.7	2278	123.2	4600	20,764	--	188	.55	7.78 ± .20	26.50 ± .57
CHPFI-26[d]	06-30-72	x		13.3	2820	110.9	--	--	--	--	--	7.36 ± .35	26.76 ± .80
CHPFI-27	07-10-72	x		18.2	2275	124.0	--	--	--	--	--	7.56 ± .40	26.62 ± 1.40
CHPFI-28[d]	07-12-72	x		19.5	1884	109.2	--	--	--	--	--	6.77 ± .30	27.92 ± 1.50
CHPFI-30[d]	07-18-72	x		17.4	2420	84.6	--	--	--	--	--	6.80 ± .48	25.71 ± 2.70
CHPFI-34[d]	08-16-72	x		17.5	2199	30.4	--	--	--	--	--	6.87 ± .18	25.20 ± .56
CHPFI-38[d]	09-07-72	x		14.6	2936	109.3	--	--	--	--	--	6.98 ± .15	24.29 ± .72
CHPFI-41[d]	10-13-72	x		16.4	1989	111.5	4550	22,234	--	201	--	7.66 ± .10	31.42 ± .35
CHPFI-42[d]	10-17-72	x		16.6	2053	105.1	4800	25,142	--	145	--	6.82 ± .25	31.20 ± 1.28
CHPFI-43[d]	10-19-72	x		19.3	1815	107.5	--	--	--	--	--	6.61 ± .21	28.16 ± 2.30
CHPFI-45[d]	11-06-72	x		12.3	2871	102.4	--	--	--	--	--	6.87 ± .86	39.00 ± 4.70
CHPFI-47[d]	11-13-72	x		19.1	2134	101.0	--	--	--	--	--	5.54 ± .30	19.11 ± 1.40
CHPFI-49[d]	12-21-72	x		17.4	2628	140.0	--	--	--	--	--	6.59 ± .21	22.21 ± 1.30
CHPFI-50	01-09-73	x		16.7	2856	164.5	--	--	--	--	--	6.50 ± .24	19.25 ± 1.20
CHPFI-53	01-17-73	x		18.6	2338	102.5	--	--	--	--	--	6.04 ± .07	18.70 ± .71
CHPFI-54[d]	01-09-73	x		14.8	2736	93.4	--	--	--	--	--	6.55 ± .26	28.66 ± 1.80
CHPFI-55[d]	01-23-73	x		17.1	2681	71.6	--	--	--	--	--	6.75 ± 1.40	26.84 ± 4.70
CHPFI-56[d]	01-29-73	x		18.4	2917	81.6	--	--	--	--	--	6.07 ± .22	21.20 ± .16
CHPFI-59	02-15-73	x		11.0	3945	96.8	--	--	--	--	--	8.68 ± .06	29.58 ± .54
CHPFI-61	02-26-73	x		19.3	1925	93.3	--	--	--	--	--	7.71 ± .20	25.96 ± .68
CHPFI-62	02-28-73	x		19.2	2146	62.8	--	--	--	--	--	7.09 ± .21	26.43 ± .23
CHPFI-63	03-02-73	x		21.5	1938	93.2	--	--	--	--	--	7.63 ± .15	23.07 ± .70
CHPFI-64[d]	03-06-73	x		21.3	1745	112.8	--	--	--	--	--	6.71 ± .12	20.40 ± .22
CHPFI-65[d]	03-08-73	x		18.9	1862	109.9	--	--	--	--	--	8.34 ± .11	25.84 ± .49
CHPFI-70[d]	04-02-73	x		22.0	1722	151.2	--	--	--	--	--	7.43 ± .27	24.52 ± .74
CHPFI-73[d]	04-24-73	x		19.17	1717	132.6	--	--	13,300	--	--	7.89 ± .20	24.63 ± .29
CHPFI-74	04-27-73	x		19.53	1644	121.7	--	--	--	--	--	8.72 ± .18	26.27 ± .69
CHPFI-75	05-03-73	x		21.22	1638	137.1	6400	25,000	11,000	83	.600	8.16 ± .11	23.51 ± .58
CHPFI-76[d]	05-10-73	x		22.13	1381	127.2	--	--	--	--	--	4.38 ± .12	12.50 ± .57
CHPFI-77	05-23-73	x		18.82	1922	119.2	5000	22,000	10,000	102	.500	8.35 ± .24	26.31 ± .92

Table II continued

Trial	Date	Coal Injection[a] Position		Coal Feed Rate, #/hr, MAF	Condensibles, #/ton coal, MAF		Partial Composition of Condensate, ppm					Gas Production, SCF/lb C Gasified	
		FFI	SBI		Condensate	Tar	Phenol	COD	NH_3	SCN^-	CN^-	Equiv CH_4[b]	Product Gas[c]
CHPFI-78	05-30-73	x		20.41	2603	122.7	--	--	10,400	--	--	8.67 ± .20	25.88 ± .92
CHPFI-79	06-01-73	x		21.53	2301	144.2	--	--	--	--	--	8.32 ± .38	26.20 ± 1.60
CHPFI-80	07-05-73		x	21.57	2055	79.5	3000	15,232	8700	135	.458	7.29 ± .47	25.12 ± 1.00
CHPFI-81	07-11-73		x	18.75	2684	15.1	2000	11,000	7500	131	.700	9.42 ±1.90	36.16 ± 7.50
CHPFI-82	07-20-73		x	21.79	2229	86.9	3300	11,000	9000	133	.500	7.29 ± .11	24.30 ± .27
CHPFI-83[d]	07-24-73	x		20.10	2279	112.8	--	--	--	--	--	7.01 ± .05	25.25 ± .42
CHPFI-84	07-26-73	x		19.75	2285	112.4	--	--	--	--	--	6.77 ± .18	23.43 ± .26
CHPFI-85	07-31-73	x		19.58	2288	114.4	3500	22,000	15,000	200	.800	6.82 ± .25	22.86 ± 1.42
CHPFI-90	08-14-73	x		19.10	2071	106.0	--	--	--	--	--	7.62 ± .70	26.92 ± 2.80
CHPFI-91	08-28-73		x	16.53	2148	13.7	--	--	--	--	--	7.14 ± .37	34.91 ± 2.60
CHPFI-92	09-05-73		x	15.31	2587	2.9	--	--	--	--	--	7.20 ± .13	35.35 ± .40
CHPFI-93[d]	09-11-73	x		21.17	1998	105.4	--	--	--	--	--	6.16 ± .25	25.84 ± 1.20
CHPFI-94[d]	09-14-73	x		20.72	2194	121.5	--	--	--	--	--	6.70 ± .47	29.14 ± 2.80
CHPFI-95[d]	09-18-73	x		20.74	2336	116.3	--	--	9600	--	--	6.88 ± .31	27.98 ± 1.00
CHPFI-96[d]	09-20-73		x	17.18	1904	32.0	--	--	--	--	--	7.05 ± .42	29.45 ± 2.70
CHPFI-97[d]	09-26-73		x	20.99	1991	48.9	2300	11,000	9100	116	.300	7.91 ± 1.60	28.44 ± 6.10
CHPFI-98[d]	10-18-73	x		16.61	2748	126.1	3200	18,000	7000	102	.700	6.97 ± .36	31.62 ± .96
CHPFI-99[d]	10-25-73	x		20.31	2563	158.9	2300	20,000	10,000	125	.800	6.59 ± .31	25.64 ± 1.70
CHPFI-100[d]	10-29-73	x		19.51	2729	132.9	3300	21,000	7000	110	.300	7.42 ± .34	23.97 ± .66
CHPFI-101[d]	11-05-73	x		16.37	3106	121.8	2600	17,000	6000	83	.200	7.73 ± .14	26.73 ± .63
CHPFI-102[d]	11-07-73	x		16.45	3644	183.9	--	--	--	--	--	7.81 ± .19	26.81 ± .63
CHPFI-103[d]	11-09-73	x		16.99	3581	162.7	--	--	--	--	--	7.40 ± .14	26.94 ± .41
CHPFI-115	05-10-74	x		16.15	3094	49.1	1900	9000	6700	38	.230	6.46 ± .23[f]	27.66 ± 1.60
CHPFI-116	05-14-74	x		16.38	2620	80.3	3000	19,000	7600	104	.240	5.89 ± .23[f]	25.67 ± 1.80
CHPFI-117	05-16-74	x		15.64	2942	86.74	2500	21,000	30,500	61	.006	5.82 ± .05[f]	27.74 ± .43
CHPFI-118	05-21-74	x		13.54	2905	131.2	2550	15,000	6400	58	.040	6.13 ± .06[f]	24.74 ± .43
					PITTSBURGH SEAM COAL								
CHPFP-86	08-02-73	x		16.73	2385	94.8	--	--	--	--	--	7.27 ± .62	27.89 ± 2.30
CHPFP-87	08-06-73	x		20.88	1917	115.5	1700	19,000	11,000	188	.600	6.61 ± .19	22.60 ± .59
CHPFP-88	08-08-73	x		17.92	2558	112.9	--	--	--	--	--	7.39 ± .85	26.44 ± 2.60

that in each case, reported amounts of effluent represent material produced during reactor startup and shutdown as well as during steady state gasifier operation.

Time series plots of product gas and equivalent methane (methane plus twice ethane production) production rates for each of the lignite runs listed in Table I are presented in Figures 1a and 1b. Overall, in the 83 gasification experiments cited in Table I (Lignite, Illinois No. 6, and Pittsburgh Seam coals), average total product gas production during a run varied from 12.5 to 39.0 SCF/lb of carbon gasified; average equivalent methane yields ranged from 3.4 to 8.7 SCF/lb of carbon gasified. In considering these statistics it is important to note that a shift has been made here from the conventional product gas reporting basis of SCF/lb of coal, MAF to that of SCF/lb of carbon gasified. Both coal feedrates (on a moisture, ash-free basis -- see Table II) and percent carbon conversion (see Table I), varied significantly among the gasification tests cited. The specified yield reporting basis of SCF/lb of carbon gasified was defined to permit direct comparison of experimental results where each of these feed conditions varied from run to run. Where system mass balances can be adequately closed, pounds of carbon gasified can be determined either directly on the basis of measured carbon in recovered products or as a weight difference between measured feed coal carbon and residual carbon in the char. In the absence of a reliable mass balance, the fresh coal-char carbon difference approach is the more meaningful method of calculation. Data reported here were calculated using the latter method.

Treatment of Effluents from Synthane Steam/Oxygen Gasification

Under contract to the Environmental Protection Agency, Exxon has studied potential environmental impacts associated with effluents from a number of coal gasifiers including Synthane[2] and concluded that:

(1) To insure compliance with Federal New Source Performance Standard, SO_2 scrubbers will be required for at least those Synthane plant boilers fueled with tars and chars produced during the gasification of caking coals, e.g., Illinois No. 6 and Pittsburgh Seam; and

(2) To meet either anticipated water quality regulations or process restrictions for reuse of water, extensive treatment of aqueous condensates will be required, including NH_3 recovery, H_2S stripping, phenol recovery or destruction, biological oxidation of thiocyanate and cyanide, removal of dissolved solids, and disposal of biological sludge.

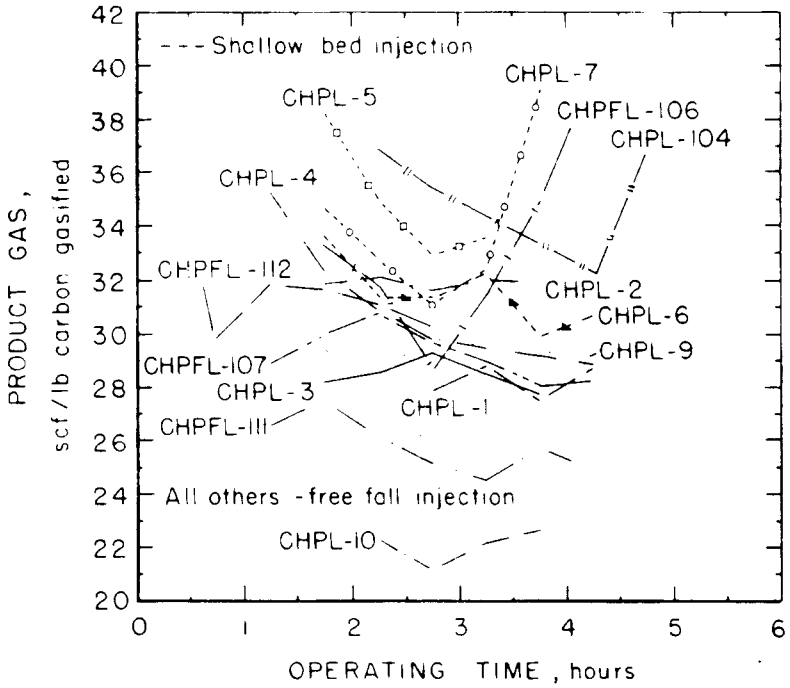

Figure 1a - Product gas production versus time: past free fall and shallow bed - injections of North Dakota lignite.

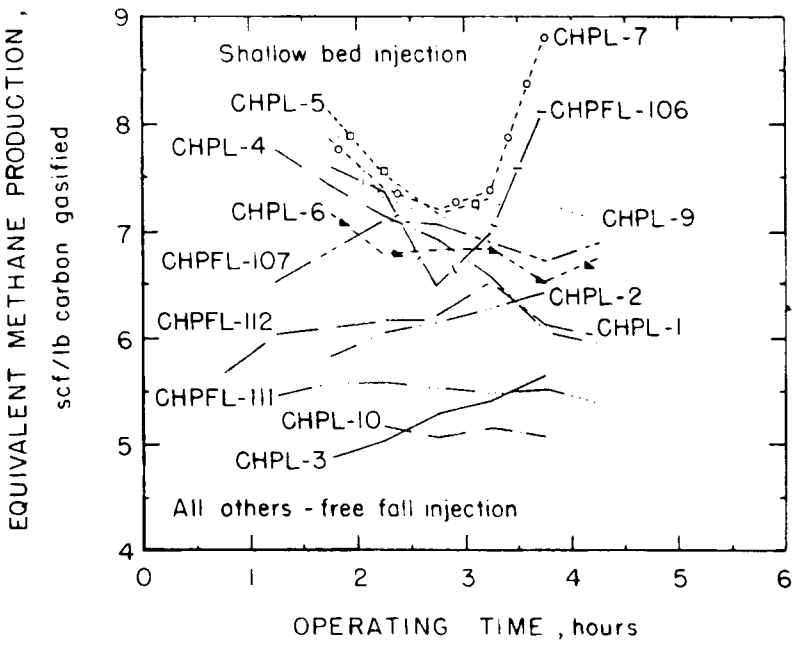

Figure 1b - Equivalent methane production versus time: past free fall and shallow bed - injection of North Dakota lignite.

These conclusions were based upon available batch effluent data of the type presented in Table II. Relying upon the same data base but working independently, staff at PERC have investigated overall design requirements and costs for various segments of the anticipated treatment system required for the Synthane process. For example, initial estimates are that capital costs for equipment to treat aqueous condensates from a standard plant (250 MMSCFD of synthetic natural gas) for recycle to the Synthane gasifier would be $13.2 million (1972 dollars); annual operating costs (excluding credits for by-product production, e.g., NH_3) would be about $3 million.(3)

Although preliminary, the environmental control assessments for the Synthane process conducted by Exxon and PERC complement one another in indicating that:

(1) extensive treatment of Synthane gasifier effluents is required to comply with environmental regulations;

(2) necessary processing involves a significant number of handling and processing steps; and

(3) capital and operating costs even for aqueous effluent treatment alone (excluding SO_2 scrubbing required if char and tar combustion emissions exceed Federal limits) are not negligible.

Present Experimental Program

The present experimental program was designed to fulfill two basic purposes: (1) to improve and extend the available data base on effluent production rates during Synthane coal gasification, and (2) to investigate the feasibility of utilizing the gasifier as an effluent decomposition reactor, thereby reducing or possibly eliminating the need for one or more currently envisioned effluent treatment processes.

(1) Improvement of Existing Effluent Data Base

Two distinct problems exist with the current Synthane gasifier effluent data base. First, as is evident in Table II, there is substantial scatter in reported production rates among apparently similar runs. Secondly, as a result of the batch sampling procedure employed in the acquisition of these data, it is not possible to isolate steady state production rates from those of reactor startup and shutdown. In an effort to correct for these inadequacies, three groups of gasification experiments were conducted. In each group of experiments, process variables and operating procedures were monitored closely to minimize variations in system operating conditions which might result in fluctuations in effluent production rates. To distinguish effluent production during steady state operation from that during startup, samples of condensed tars and contaminated water condensate were collected at regular intervals throughout each run and analyzed.

(2) Feasibility of Using the Gasifier for the Destruction of Effluents

At typical reactor operating conditions, viz., high temperature (1600-1800°F) and high partial pressures of both H_2 and H_2O, essentially none of the effluents measured in past experiments and summarized in Table II (except NH_3) are thermodynamically stable. Methods available for enhancing the approach of these compounds to chemical equilibrium within the gasifier include increasing fresh coal heatup rates, increasing reaction temperature, extending product gas residence time, introducing a catalyst, and improving gas-solid mixing. In the present experimental program, various sets of gasifier operating conditions were employed to test the feasibility of utilizing the gasifier itself for the partial or complete destruction of effluents. It is important to note that, as with various measured effluent production rates, typical Synthane gasifier methane yields (Table II) also are substantially in excess of thermodynamic equilibrium values. Since high gasifier methane yields are desirable, modifications in gasifier operating conditions introduced to enhance effluent decomposition also could adversely affect methane yields. Careful measurements of both equivalent methane and total product gas yields were made during each test to determine the extent to which this occurred.

EXPERIMENTAL EQUIPMENT AND PROCEDURE

Gasifier Configuration and Effluent Sampling Apparatus

A flow diagram illustrating the basic equipment train for PERC's 4-inch diameter laboratory scale Synthane gasifier is presented in Figure 2. Fresh coal is injected into the gasifier under pressure (40 atm) at the rate of about 25 lbs/hr; reactant gases (steam and oxygen) are introduced at the base of the gasifier. Unreacted solids (char) are discharged from the base of the reactor to a batch receiver while raw product gas is withdrawn from the top of the reactor. Following coarse particulate removal, this gas is cooled in a series of two water-jacketed condensers where high molecular weight hydrocarbons and contaminated water are condensed and collected. Prior to venting, the composition and flowrate of non-condensible gases leaving the second condenser are measured.

As is illustrated in Figure 3, the Synthane gasifier is divided into two sections, an upper carbonization zone (~6 ft) through which fresh coal free-falls and/or unreacted solids disengage from product gases, and a lower fluidized bed zone (~6 ft). In the present experimental program, fresh lignite coal was injected into the gasifier at one of three locations: (1) the top of the carbonizer (free-fall injections); (2) approximately 1-1/3 ft into the fluidized bed (shallow bed-injections); and (3) approximately 4-1/2 ft into the fluidized bed (deep bed-injections). Although some variation in gasifier temperature profile with coal injection position occurs (see Figure 4), typical reactor temperatures range from an average of 400 to 500°C at the top of the carbonizer to 800 to 900°C at the base of the fluidized bed.

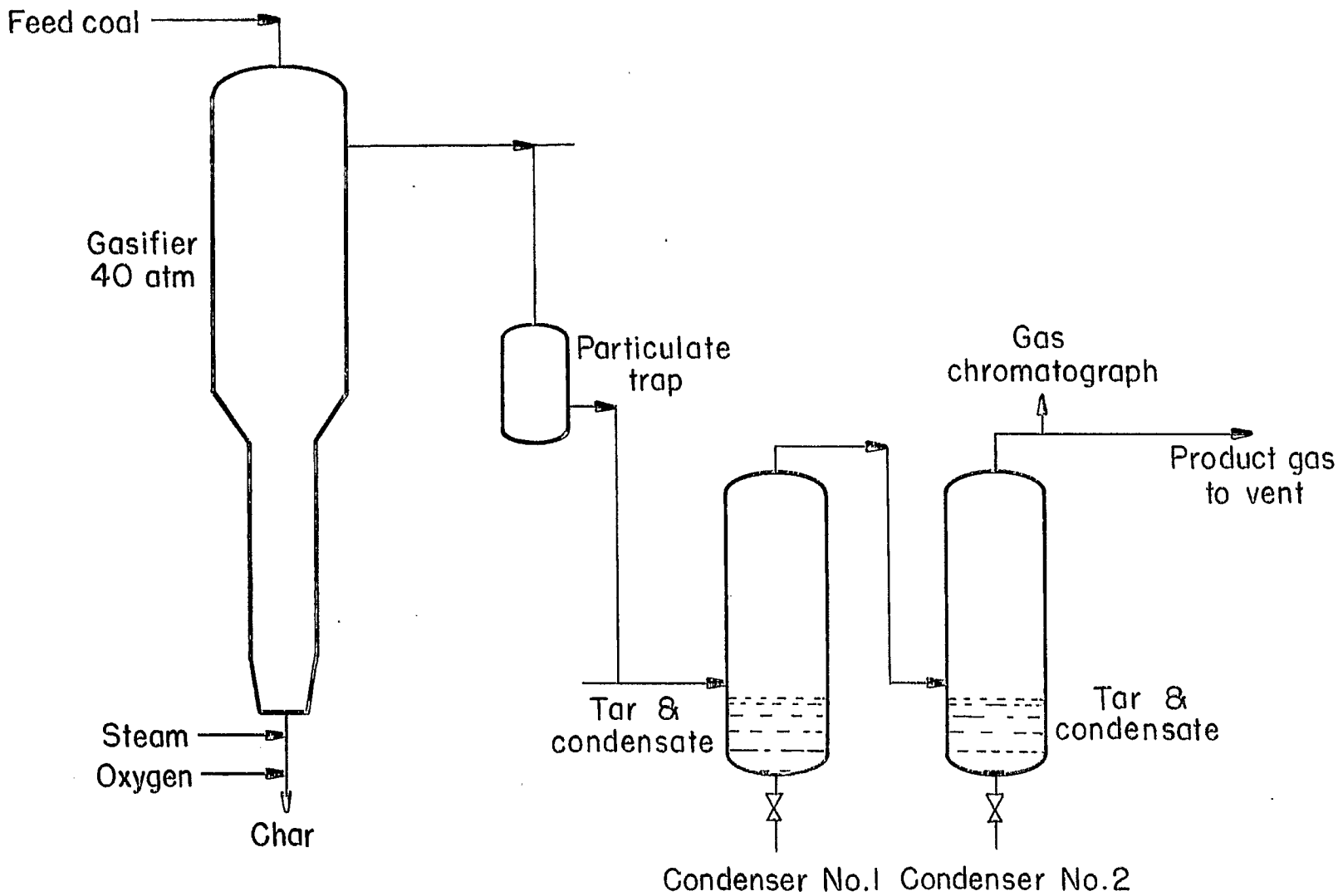

Figure 2 - Basic laboratory scale equipment train for the batchwise collection of aqueous and liquid hydrocarbon effluents from the Synthane gasifier

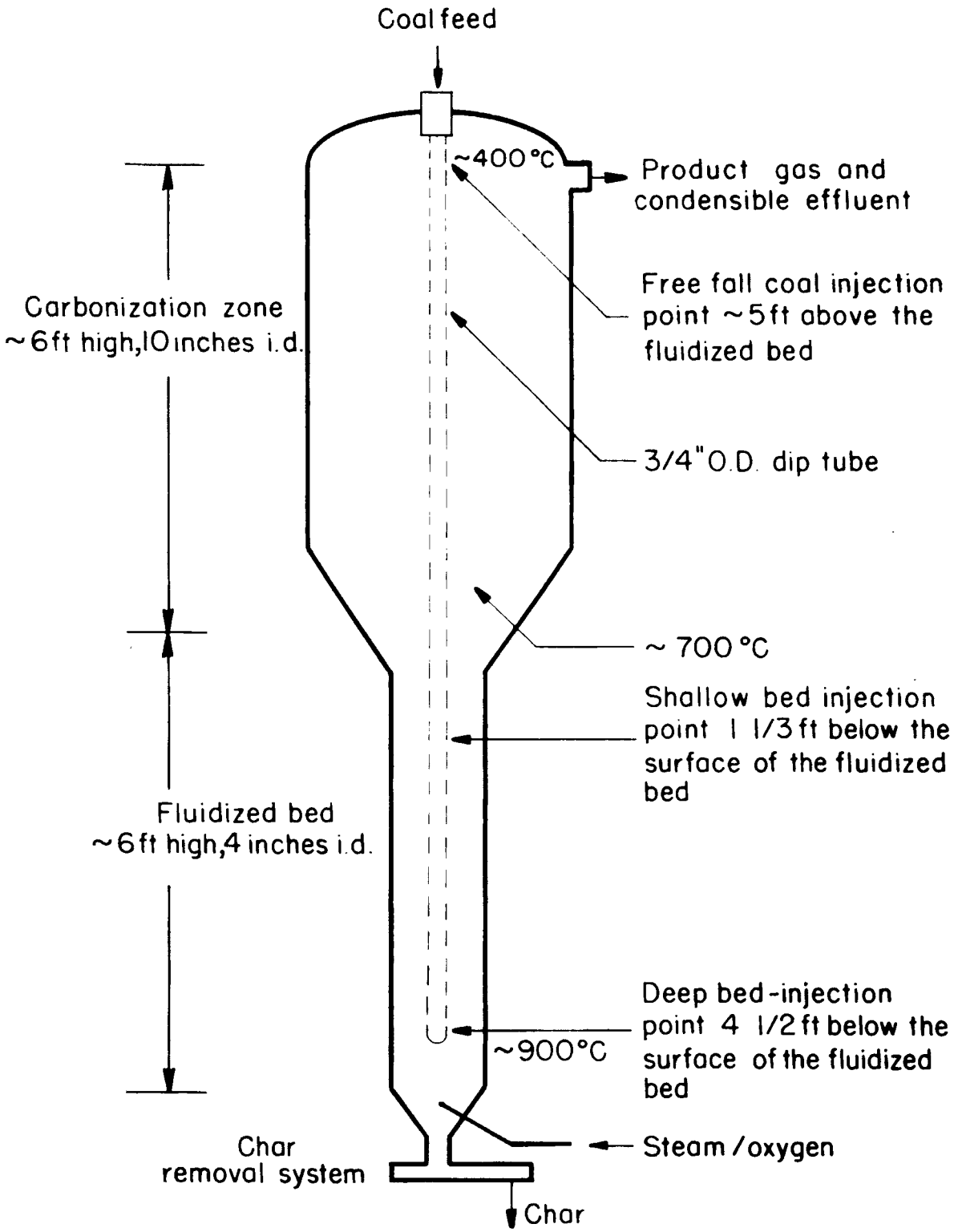

Figure 3 - Basic configuration and coal feed locations of Synthane gasifier

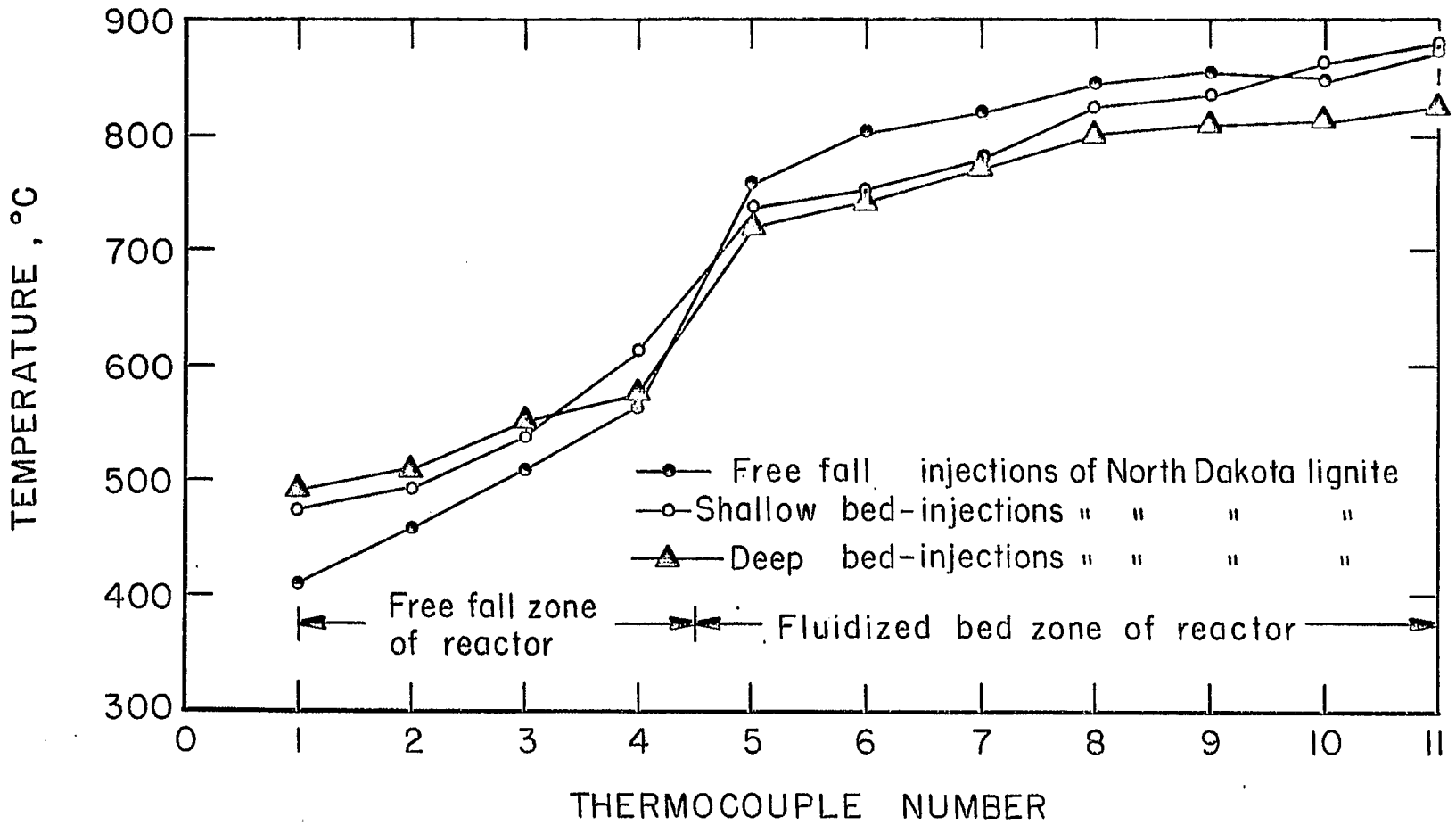

Figure 4 - Variations in gasifier axial temperature profiles with coal injection location.

To permit periodic sampling of hydrocarbon and water condensates during a run, the batch condensate collection equipment shown in Figure 2 was modified as shown in Figure 5. Small reservoirs were attached to the base of each condenser. At regular intervals, these reservoirs were pressurized with nitrogen and filled with accumulated condenser condensate. Subsequently, each reservoir was depressurized and the contents were drained, weighed, and sampled for analysis. Gases vented during depressurization of these reservoirs were collected during one run and analyzed to determine whether condensate fractions, particularly light hydrocarbons such as benzene were being vaporized and lost. Mass spectrographic analysis of blowdown gases indicated the presence of largely N_2 and CO_2 and essentially no light hydrocarbons.

Experimental and Analytical Procedure

With the exception of the coal feeding technique, experimental procedure was standardized as much as possible from run to run. At start-up, N_2 was employed as the initial fluidizing agent and reactor heat was supplied electrically. Gradually, steam and oxygen flows were increased, nitrogen flow was decreased and reactor heat requirements were met increasingly by exothermic gasification reactions. Following an initial accumulation of solids in the reactor's fluid bed, char extraction rates were varied to maintain a desired bed height. Bed temperatures were maintained largely by varying the flowrate of oxygen to the reactor. Note that both oxygen flowrate and char extraction rate were varied manually. A certain amount of the observed fluctuation in gasifier performance during a run is believed to be related to this manual control.

In each experiment, a measured quantity of cold coal was discharged at a known rate from a pressurized reservoir to the gasifier through a star feeder. Typical size distributions for coals fed in the various injection geometries are presented below:

	wt % Above Specified Mesh Size	
Mesh Size (U.S. Standard Sieve)	Free Fall and Shallow Bed-Injections (10 mesh by zero)	Deep Bed-Injections (20 mesh by zero)
20	10.5	0.0
50	39.8	41.4
100	15.7	18.1
140	17.5	17.4
200	6.4	7.3
325	5.2	9.7
fines	4.9	6.1

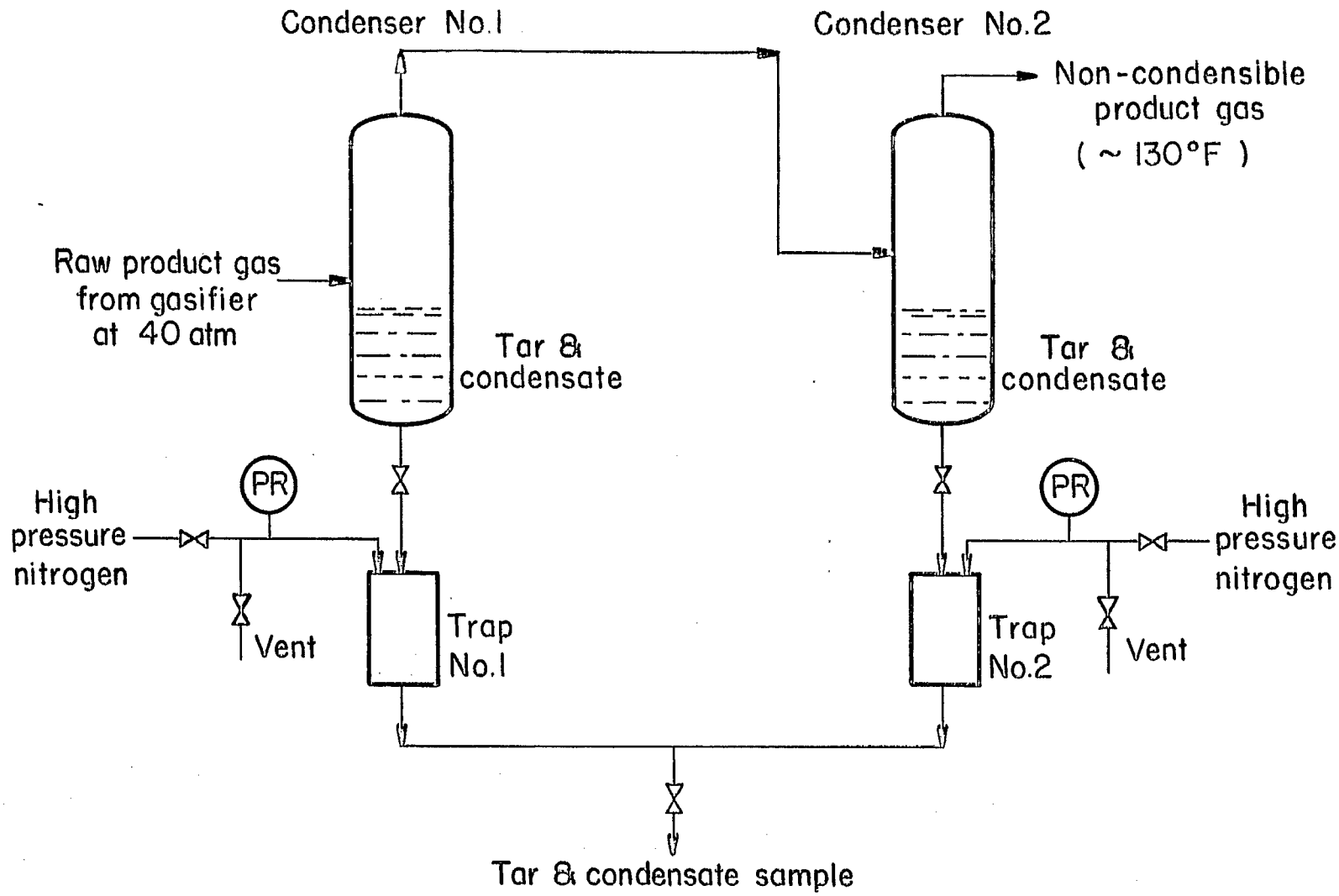

Figure 5 - Apparatus for intermittent sampling of tar and condensates produced during the gasification of coal.

In the free-fall injection runs, coal was allowed to fall by gravity from the top of the gasifier. In the shallow and deep bed-injection runs, nitrogen was utilized as a propellant to transport the coal through a standpipe from the top of the reactor to the appropriate point of injection within the fluidized bed.

From reactor startup to shutdown (typically about 6 hours), product gas samples were collected every 30 minutes. Condensates (hydrocarbon and aqueous) were collected for 1-1/2 hours during reactor startup and at 45 minute intervals for the remainder of the run. Product gas was analyzed routinely by three independent techniques -- on-line gas chromatography, mass spectroscopy and laboratory gas chromatography -- and the results were averaged. Condensates first were separated and weighed. Samples of water then were withdrawn and routinely analyzed for phenol, cyanide, thiocyanate, chemical oxygen demand, and total organic and inorganic carbon. Ultimate analyses, true boiling point curves and specific gravities were determined for a limited number of hydrocarbon condensate samples to characterize tar composition.

Unreacted solids (char) were discharged from the gasifier through an extractor to a pressurized receiver where they accumulated throughout a run. At the end of each run, this receiver was emptied and a batch sample of the char was collected for ultimate analysis.

Following each gasification test, hydrogen, carbon, and oxygen mass balances were determined on the basis of the known composition of the feed coal, measured steam, oxygen, and nitrogen feedrates, and the weights and compositions of collected product gas, gasifier char, and hydrocarbon/water condensates.

EXPERIMENTAL RESULTS

Five free fall, eight shallow, and six deep bed-injection gasification tests were conducted on North Dakota lignite. Relevant operating statistics for each of these tests are presented in Table III. Time series plots of product gas and equivalent methane production rates as a function of time for individual tests are presented in Figures 6 and 7, respectively. Carbon, hydrogen, and oxygen mass balances for each test are summarized in Figure 8. As is apparent from this figure, material was consistently lost during each experiment conducted. The closest approximation to closure of a mass balance occurred during the six deep bed-injection tests.

Table III. Operating and Product Gas Statistics, Series of Steam-Oxygen Gasification Trials: Free Fall, Shallow and Deep Bed-Injections of North Dakota Lignite Coal

Trial	Date	Gasifier Reactant Feedrates, #/hr			Fluid Bed Temp, °C			Reactor Operating Time, hrs		% Reactant Conversion		Partial Coal Anal, wt %		Gas Production SCF/lb C Gasified	
		Coal	Steam	Oxygen	Bottom	Average	Top	Total	Steady State[a]	Steam	Carbon	H_2O	Ash	Equiv CH_4[b]	Product Gas[c]
FREE FALL INJECTION OF LIGNITE COAL															
CHPFL-131	08-15-74	24.14	39.41	7.40	857	834	804	5.30	2.0	33.7	84.2	23.4	7.6	5.42 ± .36	34.94 ± 1.60
CHPFL-132	08-20-74	23.10	39.56	6.77	856	827	799	6.00	2.5	28.8	84.9	23.9	7.0	5.84 ± .29	32.97 ± .87
CHPFL-133	08-26-74	23.10	39.10	6.77	871	823	769	6.00	3.0	24.5	90.2	12.2	9.0	3.99 ± .24	25.11 ± .80
CHPFL-134	08-28-74	26.96	39.10	7.67	875	832	753	5.75	2.5	29.6	82.1	22.2	7.3	6.24 ± .09	30.62 ± 1.70
CHPFL-135	08-30-74	25.70	39.10	7.22	873	823	717	5.00	3.0	24.4	70.8	19.7	7.5	5.33 ± .38	34.56 ± 1.08
SHALLOW BED-INJECTION OF LIGNITE COAL															
CHPFL-123	07-02-74	27.02	36.10	6.33	839	801	742	4.97	3.5	33.3	72.2	12.7	8.5	6.45 ± .48	32.24 ± 2.10
CHPFL-124	07-08-74	26.73	36.10	6.77	794	790	731	5.33	4.0	34.0	74.6	18.4	6.8	6.57 ± .56	32.70 ± 3.10
CHPFL-125	07-17-74	26.29	40.46	6.95	844	784	718	5.50	3.5	30.0	76.1	14.4	7.5	6.20 ± .14	31.21 ± .91
CHPFL-126	07-19-74	26.38	39.71	6.95	831	784	729	5.50	3.5	28.5	77.7	15.9	7.5	6.08 ± .41	29.44 ± 1.80
CHPFL-127	07-23-74	24.40	39.41	6.60	836	774	703	6.00	3.5	24.2	81.2	12.2	8.2	5.78 ± .23	26.38 ± 1.30
CHPFL-136	09-04-74	25.60	39.21	8.38	858	794	733	6.00	3.0	32.8	80.8	13.0	7.9	5.68 ± .17	31.37 ± 1.80
CHPFL-137	09-06-74	23.80	39.51	7.75	877	796	714	6.00	3.0	23.4	87.3	22.7	7.3	5.12 ± .11	28.84 ± .75
CHPFL-138	09-12-74	22.46	38.36	7.13	868	792	712	6.00	3.5	24.1	84.1	18.5	8.1	5.39 ± .75	26.30 ± 3.30
DEEP BED-INJECTION OF LIGNITE COAL															
CHPFL-143	10-20-74	23.40	39.11	7.67	784	759	716	3.42	1.5	15.6	68.9	23.1	7.4	6.72 ± 1.60	41.27 ± 2.70
CHPFL-144	10-16-74	24.50	39.11	7.84	827	787	731	4.40	3.0	30.8	73.8	16.2	8.0	5.66 ± .29	33.78 ± 2.70
CHPFL-146	11-04-74	25.52	39.16	9.00	809	781	735	5.00	3.0	30.3	67.9	16.1	8.7	5.71 ± .30	36.98 ± 1.60
CHPFL-147	11-06-74	24.80	39.11	9.27	819	775	717	5.30	2.5	28.2	68.9	21.8	8.6	5.26 ± .54	36.56 ± .98
CHPFL-148	11-08-74	25.70	39.11	9.45	804	775	729	5.00	3.0	29.4	64.3	18.7	8.4	5.93 ± .58	37.04 ± 1.50
CHPFL-149	11-13-74	25.70	39.11	9.18	776	758	711	5.00	2.5	26.9	66.0	16.9	8.5	5.48 ± .45	32.96 ± .49

Footnotes:

(a) That period during which the product gas H_2 concentration was approximately constant.

(b) Equiv. CH_4 = sum of SCF of CH_4 plus twice the SCF of C_2H_6.

(c) Product Gas = sum of SCF of CH_4, C_2H_6, H_2, and CO.

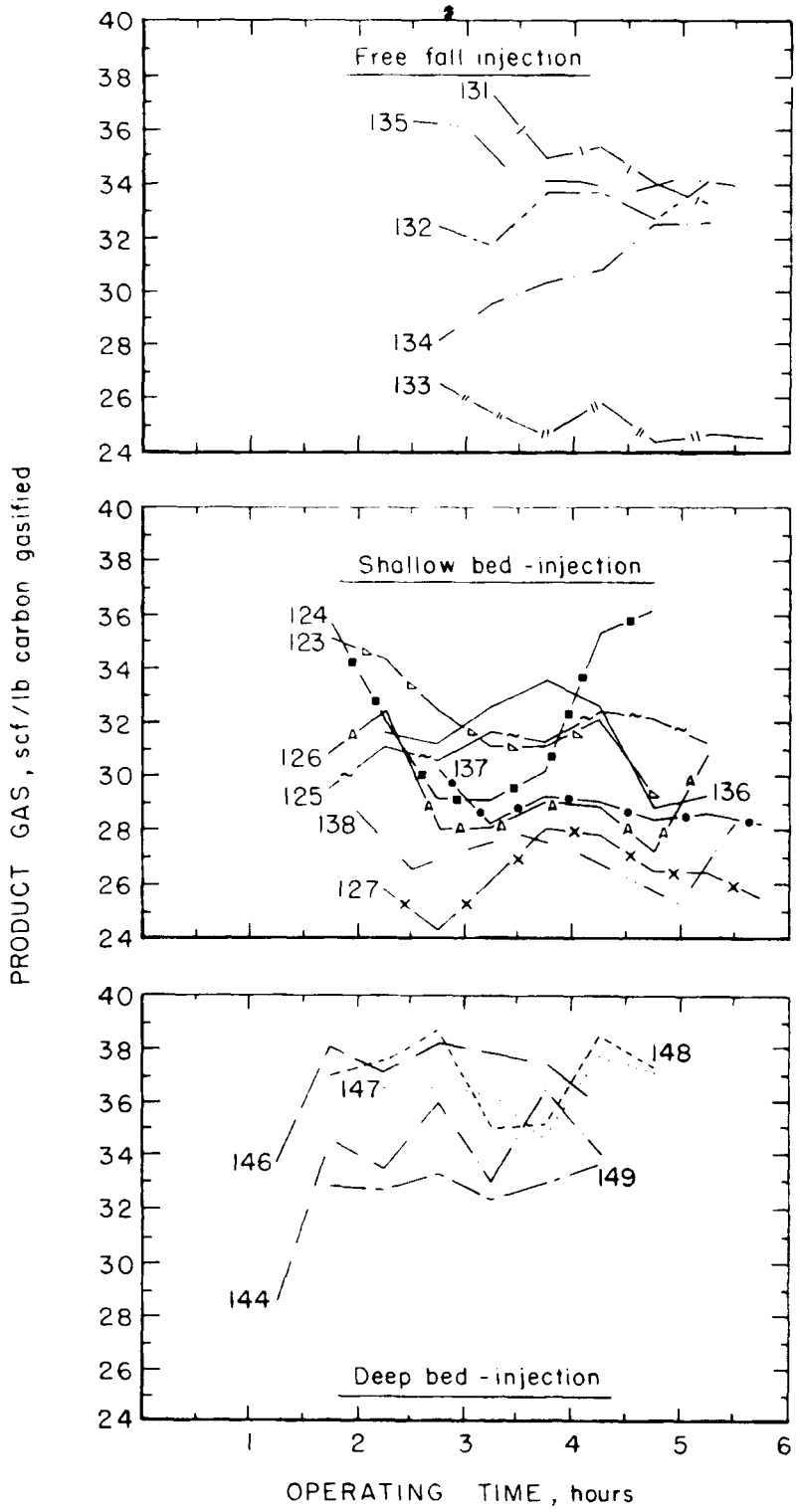

Figure 6 - Product gas production versus time : gasification of North Dakota lignite

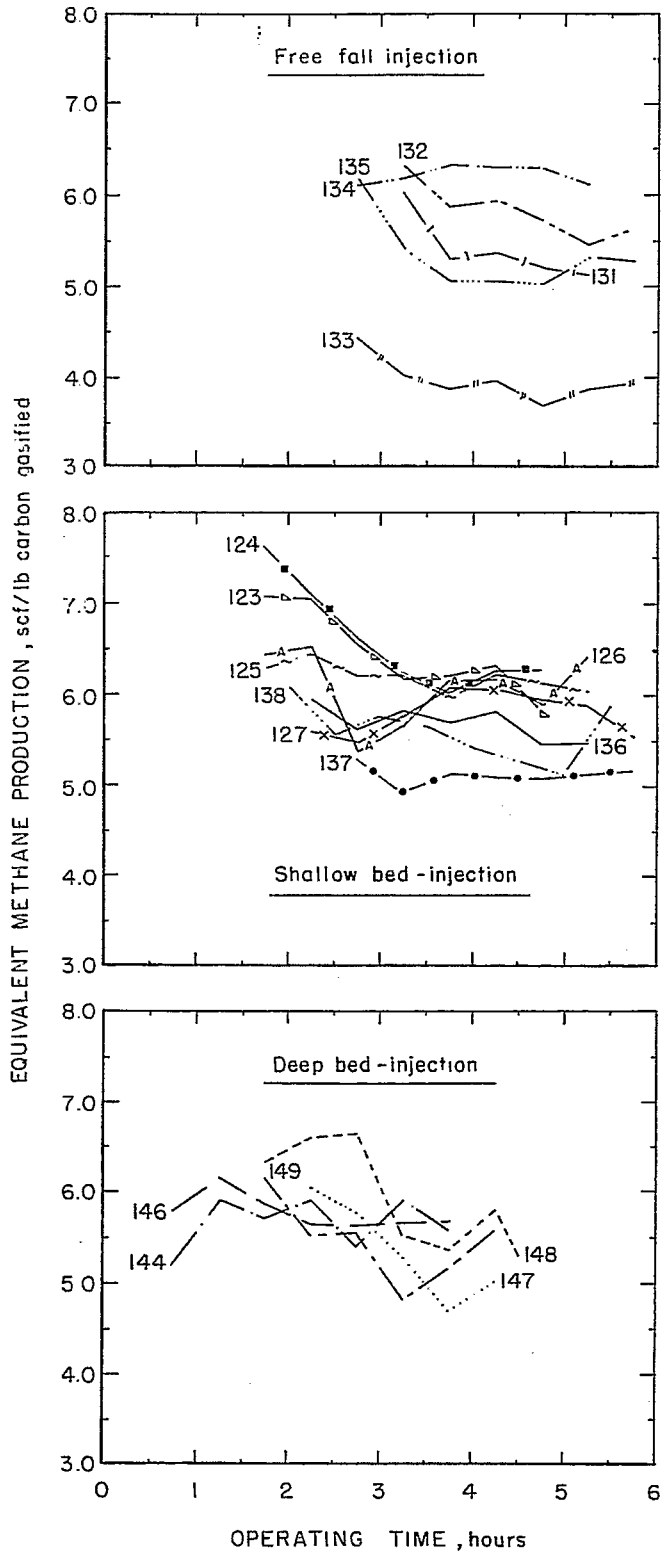

Figure 7 - Equivalent methane production versus time: gasification of North Dakota lignite

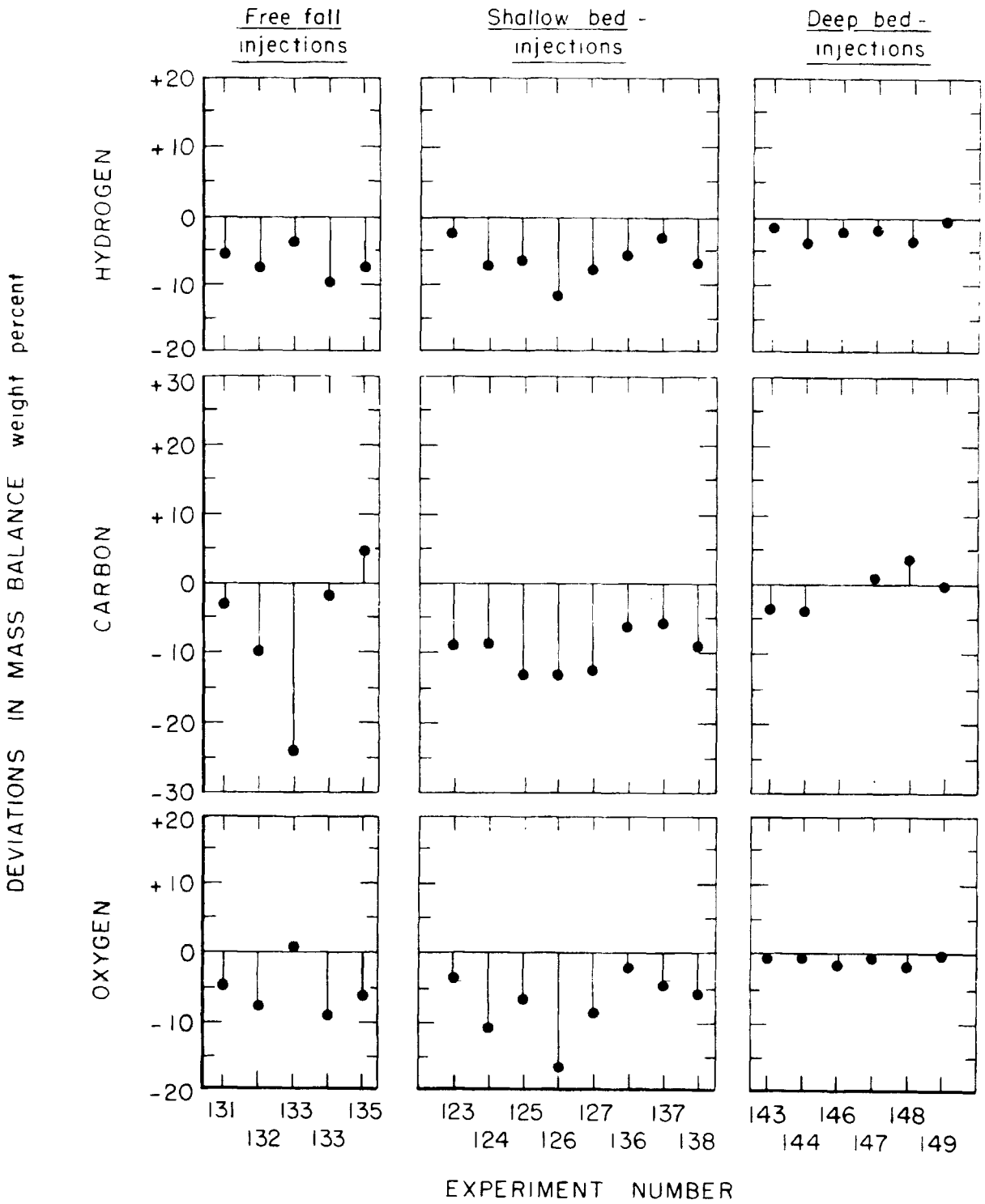

Figure 8 - Hydrogen, carbon, and oxygen mass balances : gasification of North Dakota lignite.

Aqueous Effluent Production

Time series measurements of phenol, chemical oxygen demand, and total organic and inorganic carbon contents of aqueous condensates are presented in Figures 9 through 12, respectively. Similar measurements of thiocyanate concentrations are presented in Table IV; cyanide measurements, though taken routinely, rarely exceeded 0.01 ppm and therefore were not tabulated. Data in each figure are grouped according to coal injection geometry, viz., free fall, shallow, and deep bed-injection. Note that, with the exception of inorganic carbon measurements, all the data reported indicate major reductions in effluent production as a function of both time and depth of coal injection. Consistent inorganic carbon contents were anticipated; they reflect the presence of dissolved CO_2 in basic (pH 9) condensate. At steady state, measured phenol production rates range from a high of 12 lbs/ton coal, MAF (free fall injections) to a low of < 0.5 lbs/ton coal, MAF or equivalently less than 30 ppm (deep bed-injections). Similarly, chemical oxygen demands range from a high of ~80 lbs/ton coal, MAF to a low of ~2.5 lbs/ton coal, MAF and total organic carbon contents range from a high of ~21 lbs/ton coal, MAF to a low of ~2 lbs/ton coal, MAF. Thiocyanate production rates range from a high of ~0.045 lbs/ton coal, MAF (free fall injections) to a low of less than 0.017 lbs/ton coal, MAF (deep bed-injections), the limiting sensitivity of the measurement technique employed.

Recent analytical work carried out at PERC on aqueous condensates from a range of Illinois No. 6 gasification tests[4] reflect the trends toward decreased phenol production with increased depth of fresh coal injection reported here in Figure 9. Concentrations of selected compounds measured in condensates from two free fall and three shallow bed-injection tests with Illinois No. 6 coal and reported in reference 4 are summarized below:

Compound Present in Condensate	Free Fall Injections, ppm		Shallow Bed-Injections, ppm		
	CHPFI-49	CHPFI-55	CHPFI-80	CHPFI-96	CHPFI-97
Phenol	3,400	2,660	1,300	1,270	1,000
Cresols	2,840	2,610	530	890	930
C_2-phenols	1,090	780	140	270	330
C_3-phenols	110	100	20	50	50
Dihydrics	250	540	60	20	20
Benzofuranols	70	100	30	40	50
Indanols Acetophenones	150	100	40	50	60

In contrast to the considerable scatter evident in previously reported effluent production data (Table II), note that in the present series of experiments, individual data for runs at constant coal injection geometry are substantially reproducible. Also, note that in the particular case of free fall injection tests, steady state production rates of all the effluents measured actually exceed those at gasifier startup. This result was not

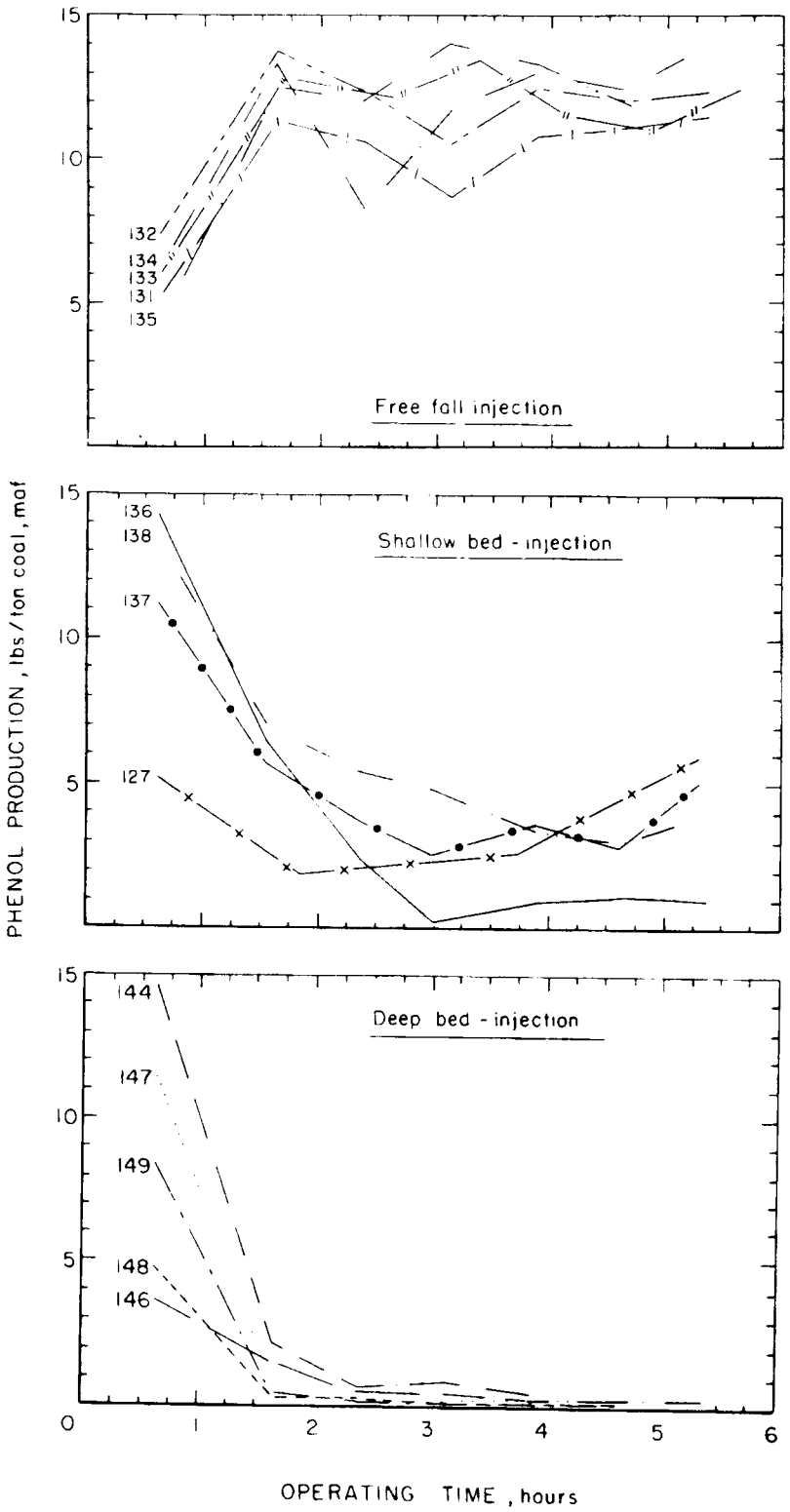

Figure 9 - Phenol in by-product water versus time : gasification of North Dakota lignite

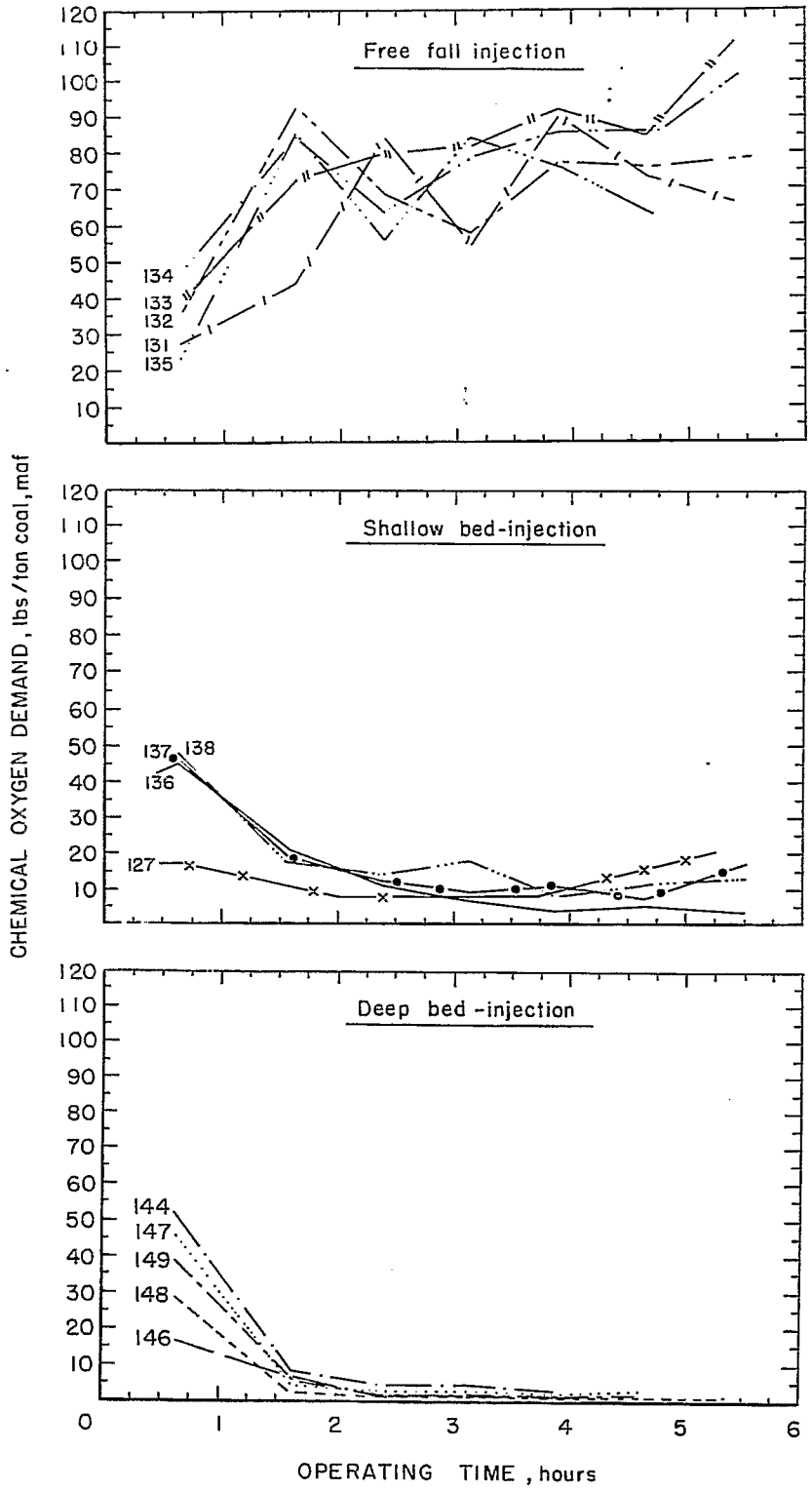

Figure 10 - Chemical oxygen demand in by-product water versus time : gasification of North Dakota lignite.

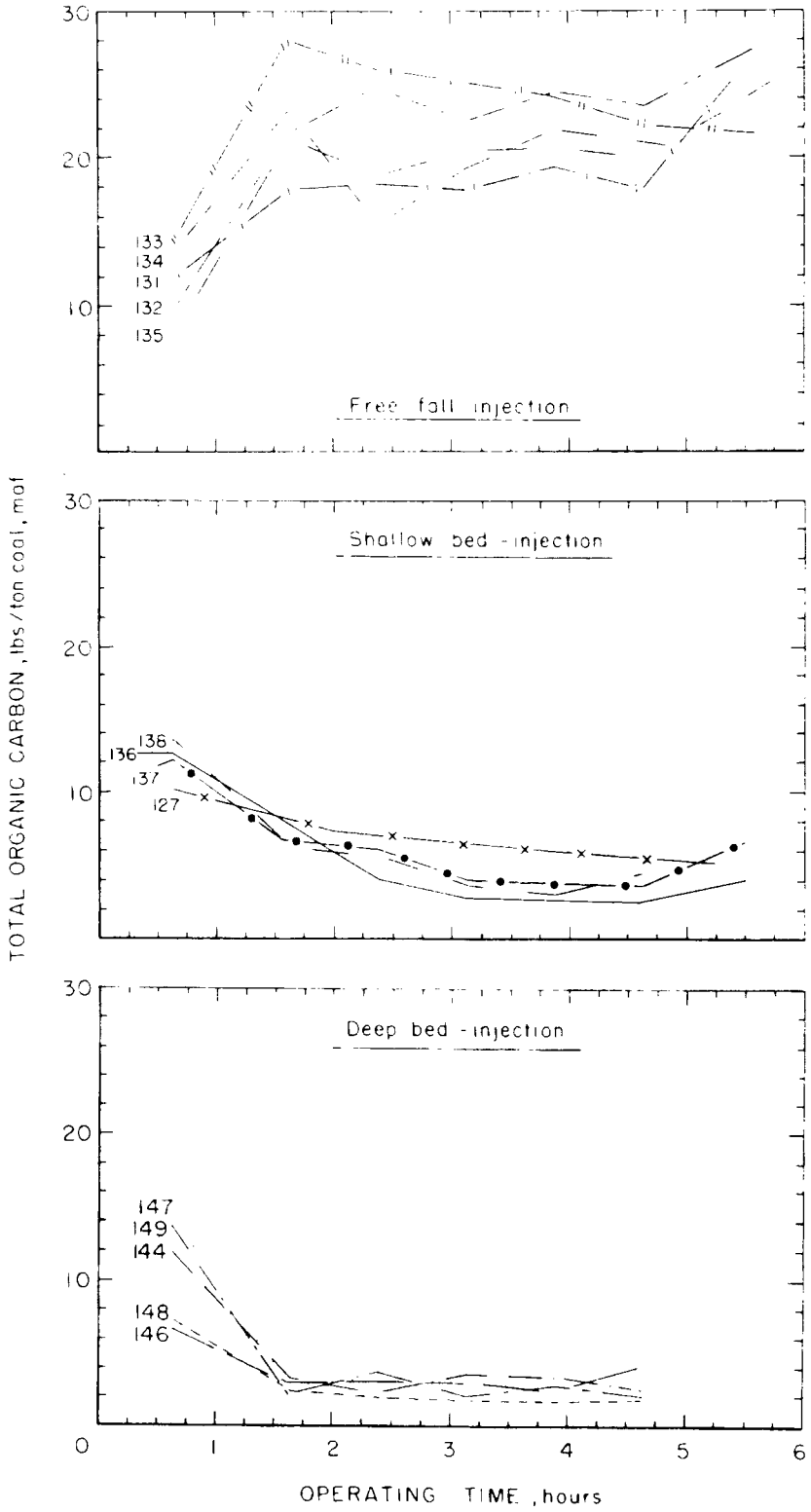

Figure 11 - Total organic carbon in by-product water versus time gasification of North Dakota lignite

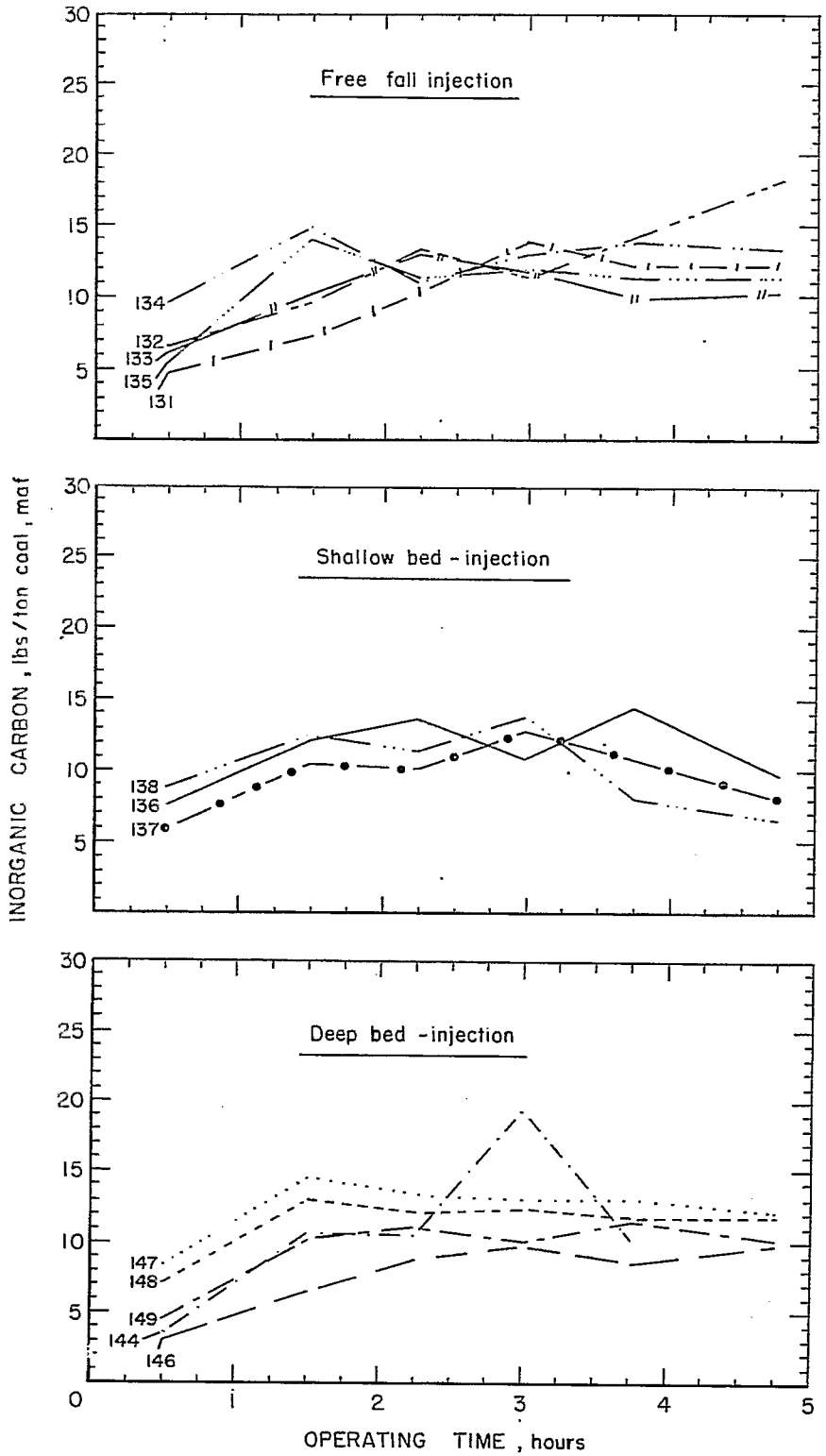

Figure 12- Inorganic carbon in by-product water versus time: gasification of North Dakota lignite.

Table IV. Time Series Measurements: Thiocyanate Content of Aqueous Condensates Collected During the Steam-Oxygen Gasification of North Dakota Lignite: Free Fall, Shallow, and Deep Bed-Injections

Trial	Aqueous Thiocyanate Concentrations, ppm						
	1-1/4 hrs	2 hrs	2-3/4 hrs	3-1/2 hrs	4-1/4 hrs	5 hrs	5-3/4 hrs
Free Fall Injection Tests							
CHPFL-131	15	16	15	12	18	7	15
CHPFL-132	≤5	≤5	≤5	≤5	≤5	≤5	≤5
CHPFL-133	11	9	12	11	11	7	7
CHPFL-134	20	18	20	21	20	20	18
CHPFL-135	25	25	30	24	22	15	4
Shallow Bed-Injection Tests							
CHPFL-127	Values consistently ≤5 ppm						
CHPFL-136							
CHPFL-137							
CHPFL-138							

	Trap #1[a]	Trap #2[a]	Trap #3[a]
CHPFL-123	≤5	≤5	12
CHPFL-124	≤5	≤5	7
CHPFL-125	≤5	≤5	≤5
CHPFL-126	≤5	--	--

Deep Bed-Injection Tests					
CHPFL-143	17		7		
CHPFL-144					
CHPFL-146					
CHPFL-147	≤5	consistently ≤5 ppm	≤5	≤5	≤5
CHPFL-148					
CHPFL-149					

Notes

(a) Batch sampling of condensate was carried out for four experiments. In these cases,
- Trap #1 contains condensate collected in condenser #1 (Figure 1) over the course of the entire run.
- Trap #2 contains overflow condensate from condenser #1.
- Trap #3 contains condensate collected in condenser #2 (Figure 1) over the course of the entire run.

Note that significant concentrations of thiocyanate are detectable only in Trap #3. This is consistent with the fact that water was condensed at a lower temperature in condenser #2.

anticipated; an initial surge in effluent production followed by a decline toward a reduced production rate at steady state had been expected. The expected pattern of production with time did occur in the shallow and deep bed-injection tests.

Ammonia Production

Time series data on ammonia production rates were not collected in the present experimental program. However, at least one measurement of ammonia content of aqueous condensate was made during all but two of the tests run and these results are reported in Table V. For purposes of comparison, the ammonia yields for past experiments on North Dakota lignite and Illinois No. 6 coals are presented in Table VI in terms of lbs/ton coal, MAF (these data are also presented in Table II in the form of concentration, ppm). In contrast to the patterns of other major constituents of gasifier condensate, ammonia production is not significantly influenced by variations in coal injection geometry. Within the accuracy of the data, ammonia production appears to vary from 15 to 20 lbs/ton coal, MAF for lignite and average about 20 to 22 lbs/ton coal, MAF for Illinois No. 6 coal. This yield is substantially greater than the approximately 6 to 8 lbs NH_3/ton, coal MAF typically produced in by-product coking.[7]

Char Production and Composition

Statistics on char production and composition are presented in Table VII. Included in this table are: (1) production rates, lbs/ton coal, MAF, (2) ultimate analyses, including sulfur content, (3) estimated high heating values, BTU/lb, and (4) combustion emission rates, equivalent lbs SO_2/MMBTU. These data are compiled for PERC's 16 previous lignite gasification tests (Tables I and II) as well as for the present series of 19 experiments. Note first that char production rates vary widely among both past and present experiments. This is a direct reflection of gasifier operating practice; char extraction rate is varied manually to maintain a consistent fluid bed height. Unfortunately, variations in extraction rate result in widely varying degrees of coal carbon conversion (see ultimate analyses of chars in Table VII) complicating the problems of experimental analysis.* Secondly, note that although chars produced in the three shallow bed-injection and many of the 13 free fall injection tests conducted previously by PERC exceeded the Federal New Source Performance Standard for combustion emissions of 1.2 lbs SO_2/MMBTU[4], all of the present free fall and shallow bed-injection tests produced chars well within compliance. Finally, note that in the present series of experiments, although char combustion emission levels are within Federal limits for free fall and shallow bed-injection tests, they are consistently exceeded in the deep bed-injection tests. This is an unexpected result. However, the following summary of the char statistics available in Table VII appears to provide a possible explanation:

*The product gas reporting basis of SCF/lb carbon gasified utilized in this report was developed specifically to compensate for variations in coal carbon conversion.

Table V. Ammonia Yields: Free Fall, Shallow, and Deep Bed-Injection Gasifications of North Dakota Lignite

Trial No.	Ammonia Yield, lbs/ton coal, MAF		
	Free Fall Injection	Shallow Bed-Injection	Deep Bed-Injection
	PRESENT NORTH DAKOTA LIGNITE TRIALS		
CHPFL-131	21.1	-	-
CHPFL-132	22.3	-	-
CHPFL-133	14.6	-	-
CHPFL-134	18.7	-	-
CHPFL-135	20.6	-	-
CHPFL-123	-	15.7	-
CHPFL-124	-	10.7	-
CHPFL-125	-	17.6	-
CHPFL-126	-	17.2	-
CHPFL-127	-	19.1	-
CHPFL-136	-	16.5	-
CHPFL-137	-	N.D.[a]	-
CHPFL-138	-	N.D.	-
CHPFL-143	-	-	22.5
CHPFL-144	-	-	N.D.
CHPFL-146	-	-	16.7
CHPFL-147	-	-	21.3
CHPFL-148	-	-	23.9
CHPFL-149	-	-	17.8
Average Yield, lbs NH_3/ton coal, MAF	19.5±3.0 (5)	16.1±2.9 (6)	20.4±3.1 (5)

Notes
(a) N.D. indicates "no data".

Table VI. Ammonia Yields, Past Synthane Gasification Trials: North Dakota Lignite and Illinois No. 6 Coals

PAST NORTH DAKOTA LIGNITE RUNS

Trial No.	lbs NH_3/ton coal, MAF: Free Fall Injection	lbs NH_3/ton coal, MAF: Shallow Bed-Injection
CHPL-1	14.7	-
CHPL-2	15.5	-
CHPL-3	11.6	-
CHPL-4	N.D.[a]	-
CHPL-5	-	14.7
CHPL-6	-	14.5
CHPL-7	-	N.D.
CHPL-8	N.D.	-
CHPL-9	N.D.	-
CHPL-10	N.D.	-
CHPFL-104	16.5	-
CHPFL-105	14.9	-
CHPFL-106	15.3	-
CHPFL-107	12.8	-
CHPFL-111	77.4[b]	-
CHPFL-112	N.D.	-
Average Yield lbs NH_3/ton coal, MAF.....	14.5 ± 1.7 (7)	14.6 ± 0.1 (2)

PAST ILLINOIS NO. 6 RUNS

Trial No.	lbs NH_3/ton coal, MAF: Free Fall Injection	lbs NH_3/ton coal, MAF: Shallow Bed-Injection
CHPFI-73	22.9	-
CHPFI-74	N.D.	-
CHPFI-75	18.1	-
CHPFI-76	N.D.	-
CHPFI-77	19.2	-
CHPFI-78	27.1	-
CHPFI-79	N.D.	-
CHPFI-80	-	17.9
CHPFI-81	-	20.1
CHPFI-82	-	20.1
CHPFI-83	N.D.	-
CHPFI-84	N.D.	-
CHPFI-85	34.3	-
CHPFI-90	N.D.	-
CHPFI-91	-	N.D.
CHPFI-92	-	N.D.
CHPFI-93	N.D.	-
CHPFI-94	N.D.	-
CHPFI-95	22.4	-
CHPFI-96	N.D.	-
CHPFI-97	18.1	-
CHPFI-98	19.2	-
CHPFI-99	25.6	-
CHPFI-100	19.1	-
CHPFI-101	18.6	-
CHPFI-102	N.D.	-
CHPFI-103	N.D.	-
CHPFI-115	20.7	-
CHPFI-116	19.9	-
CHPFI-117	89.7[b]	-
CHPFI-118	18.6	-
CHPFI-10	19.4	-
CHPFI-30	19.6	-
CHPFI-34	29.2	-
CHPFI-38	22.3	-
CHPFI-41	28.4	-
CHPFI-42	17.8	-
CHPFI-49	12.9	-
CHPFI-55	3.8[b]	-
CHPFI-56	12.5	-
CHPFI-61	16.6	-
Average Yield, lbs NH_3/ton coal, MAF ...	21.0 ± 5.1 (23)	19.4 ± 1.3 (3)

Notes
(a) N.D. indicates "no data".
(b) Measurement not included in the computation of average ammonia yield.

Table VII. Selected Analyses of Chars and Tars Produced During the Gasification of North Dakota Lignite: Free Fall, Shallow, and Deep Bed-Injection of Coal.

Trial	Type of Coal Injection[a] FF	SB	DB	#/ton coal MAF	Time of Sample Collection, hrs	Ultimate Analysis, wt % C	H	S	O	N	Ash	Est'd HHV BTU/lb[b]	Equivalent lbs SO_2/MMBTU[c]
NORTH DAKOTA LIGNITE GASIFICATION CHARS													
Previous Trials													
CHPL-1	x			646	Char collected only at the end of each trial.	71.7	1.3	0.4	1.8	.5	24.3	11,108	0.720
CHPL-2	x			525		58.9	1.1	0.6	1.9	.4	37.1	9,123	1.315
CHPL-3	x			545		60.0	1.3	1.4	0.0	.4	35.6	9,587	2.921
CHPL-4	x			691		68.3	1.1	1.0	1.9	.3	27.4	10,506	1.904
CHPL-5		x		597		50.9	5.7	1.2	8.5	.7	33.0	8,426	2.848
CHPL-6		x		649		66.9	1.1	1.0	2.2	.3	28.5	10,280	1.946
CHPL-7		x		595		65.3	1.1	1.6	0.0	.3	30.9	10,242	3.124
CHPL-8	x			899		71.8	1.5	0.6	4.7	.5	20.9	11,030	1.088
CHPL-9	x			471		58.9	0.9	2.0	0.0	.2	38.6	9,203	4.346
CHPL-10	x			1074		74.3	1.8	1.0	3.9	.6	18.4	11,658	1.716
CHPFL-104	x			531		66.4	1.3	0.5	3.0	.4	28.4	10,249	0.976
CHPFL-105	x			475		68.7	1.7	0.6	4.1	.5	24.4	10,750	1.116
CHPFL-106	x			498		72.2	1.3	0.4	2.5	.3	23.3	11,127	0.719
CHPFL-107	x			481		50.5	0.8	1.1	0.0	.3	48.7	7,883	2.791
CHPFL-111	x			357		55.8	1.1	0.5	1.5	.4	45.8	8,700	1.149
CHPFL-112	x			446		56.0	1.0	0.3	2.1	.4	40.2	8,612	0.348
Present Trials													
CHPFL-131	x			445		58.2	1.4	0.5	3.8	.5	35.6	9,056	1.104
CHPFL-132	x			423		52.5	1.1	0.3	3.1	.4	42.6	8,088	0.743
CHPFL-133	x			311		54.0	1.4	0.2	5.0	.5	38.9	8,340	0.480
CHPFL-134	x			493		52.5	1.1	0.2	---	3.0	42.4	8,324	0.481
CHPFL-135	x			956		44.8	1.0	0.3	3.6	.4	49.9	6,867	0.874

Footnotes:

(a) FF = Free Fall Injection; SB = Shallow Bed-Injection; DB = Deep Bed-Injection.

(b) Calculated using the DuLong formula[5]: HHV = 145.4C + 620.3 (H - O/8) + 40.5S where C, H, S and O represent weight percent of the respective element.

(c) The EPA New Source Performance Standard for sulfur emissions during combustion of solid fuels is 1.2 lbs SO_2/MMBTU. The limit for liquid fuel combustion is 0.8 lbs SO_2/MMBTU[6].

Table VII. continued

Trial	Type of Coal Injection[a] FF	SB	DB	#/ton coal MAF	Time of Sample Collection, hrs	Ultimate Analysis, wt % C	H	S	O	N	Ash	Est'd HHV BTU/lb[b]	Equivalent lbs SO_2/MMBTU[c]
CHPFL-123		x		600	Char collected only at the end of each trial.	67.7	1.3	0.3	2.9	.4	27.4	11,031	0.544
CHPFL-124		x		506		73.9	1.4	0.3	4.6	.4	19.3	11,269	0.532
CHPFL-125		x		517		65.4	1.3	0.5	4.2	.5	28.1	10,010	0.999
CHPFL-126		x		507		63.2	1.2	0.3	3.5	.4	31.4	9,674	0.620
CHPFL-127		x		434		56.7	1.3	0.3	4.5	.4	36.8	8,714	0.689
CHPFL-136		x		520		62.3	1.3	0.3	3.8	.4	31.9	9,582	0.626
CHPFL-137		x		554		45.4	1.2	0.3	2.9	.4	49.8	7,133	0.841
CHPFL-138		x		438		55.8	1.4	0.4	4.0	.5	37.9	8,688	0.921
CHPFL-143			x	705		65.8	1.5	0.6	3.2	.6	28.3	10,274	1.168
CHPFL-144			x	674		60.5	1.3	0.5	4.2	.5	33.0	9,298	1.076
CHPFL-146			x	744		61.2	1.4	1.1	0.8	.5	35.8	9,749	2.257
CHPFL-147			x	667		65.5	2.0	0.7	6.0	.6	25.2	10,327	1.356
CHPFL-148			x	871		66.6	2.1	0.7	6.0	.6	24.0	10,549	1.327
CHPFL-149			x	714		68.9	2.7	0.9	8.1	.8	18.6	11,101	1.621

NORTH DAKOTA LIGNITE GASIFICATION TARS[d]

									Present Trials				
CHPFL-131A	x			116.00	3/8	70.0	5.9	1.25	--[e]	--[f]	--[g]	13,346	1.873
CHPFL-135A	x			33.60	3/8	62.4	8.1	0.98	--	--	--	13,594	1.442
CHPFL-147A			x	13.53	3/8	71.1	5.9	1.80	--	--	--	13,528	2.661
CHPFL-133C	x			87.80	2-5/8	73.9	8.4	0.90	--	--	--	15,449	1.165
CHPFL-138B		x		23.50	1-5/8	73.2	6.3	1.70	--	--	--	14,077	2.415
CHPFL-147C			x	4.44	2-3/8	--	--	4.70	--	--	--	--	--
CHPFL-131F	x			18.00	4-5/8	67.8	8.1	0.82	--	--	--	14,373	1.141
CHPFL-133E	x			88.80	4-1/8	70.0	8.6	0.79	--	--	--	15,002	1.053
CHPFL-135E	x			88.00	3-7/8	71.2	8.3	0.83	--	--	--	14,992	1.107

Footnotes:

(d) Tar samples listed were taken at various times during a trial run. Samples collected early in a trial run represent unsteady state conditions. Samples taken late in a run approximate a steady state condition. These samples were not dehydrated prior to determination of their ultimate analysis. They may contain as much as 15% H_2O.

(e) No measurements were taken. In previous tar analyses oxygen contents of approximately 7 percent have been recorded.

(f) No measurements were taken. A typical value in previous analyses is about 1 percent.

(g) No measurements were taken. It must be assumed that the bulk of the unaccounted-for tar composition is ash and water.

Item	Free Fall Injection	Shallow Bed-Injection	Deep Bed-Injection
Char Yield, lbs/ton coal, MAF.	418 ± 77	510 ± 55	729 ± 75
Ultimate Analysis, wt%			
C	52.4 ± 4.8	61.3 ± 8.7	64.8 ± 3.3
H	1.2 ± 0.2	1.3 ± 0.1	1.8 ± 0.5
S	0.3 ± 0.1	0.3 ± 0.1	0.8 ± 0.2
O	3.9 ± 0.8	3.8 ± 0.7	5.5 ± 1.9
N	0.5 ± 0.1	0.4 ± 0.0	0.6 ± 0.1
Ash	41.9 ± 5.3	32.4 ± 9.0	27.5 ± 6.3
Estimated HHV, BTU/lb . .	8135 ± 796	9513 ± 1344	10,216 ± 628
Combustion Equivalent lbs SO_2/MMBTU	0.74 ± 0.27	0.72 ± 0.18	1.47 ± 0.43

From free fall to shallow to deep bed-injections, there is a distinct trend towards increased volumes of char having progressively higher carbon, hydrogen, oxygen, sulfur, and nitrogen contents, lower ash contents, and higher heating values. It appears that with deepening injection, increasing amounts of feed coal exit prematurely through the char extractor after undergoing only partial reaction. In a properly modified reactor, deep bed-injection char sulfur contents would not be expected to differ dramatically from those for free fall and shallow bed-injections.

Hydrocarbon Condensate (Tar) Production

Tar production rates for each of the 19 experiments conducted are presented as a function of time in Figure 13. Grouped according to coal injection geometry, these data indicate that: (1) with the exception of free fall coal injection runs, tar production declines with time from an initially high rate to a reduced rate at steady state; and (2) steady state tar production levels fall rapidly as coal is injected deeper into the fluidized bed portion of the reactor. As with the previously cited aqueous effluent production data, steady state tar production levels in the free fall injection tests actually exceed those observed during gasifier startup. Measured steady state tar production levels range from a high of ~100 lbs/ton coal, MAF (free fall injections) to a low of ~5 lbs/ton coal, MAF (deep bed-injections). In contrast to previously reported batch data on tar production during gasification (Table II), present time series data, particularly those for shallow and deep bed-injection tests, are highly reproducible from run to run.

Tar Composition

Ultimate analyses (including sulfur content), estimated high heating values (BTU/lb), and approximate sulfur combustion emission rates (equivalent lbs SO_2/MMBTU) are presented in Table VII for a limited number of tar samples

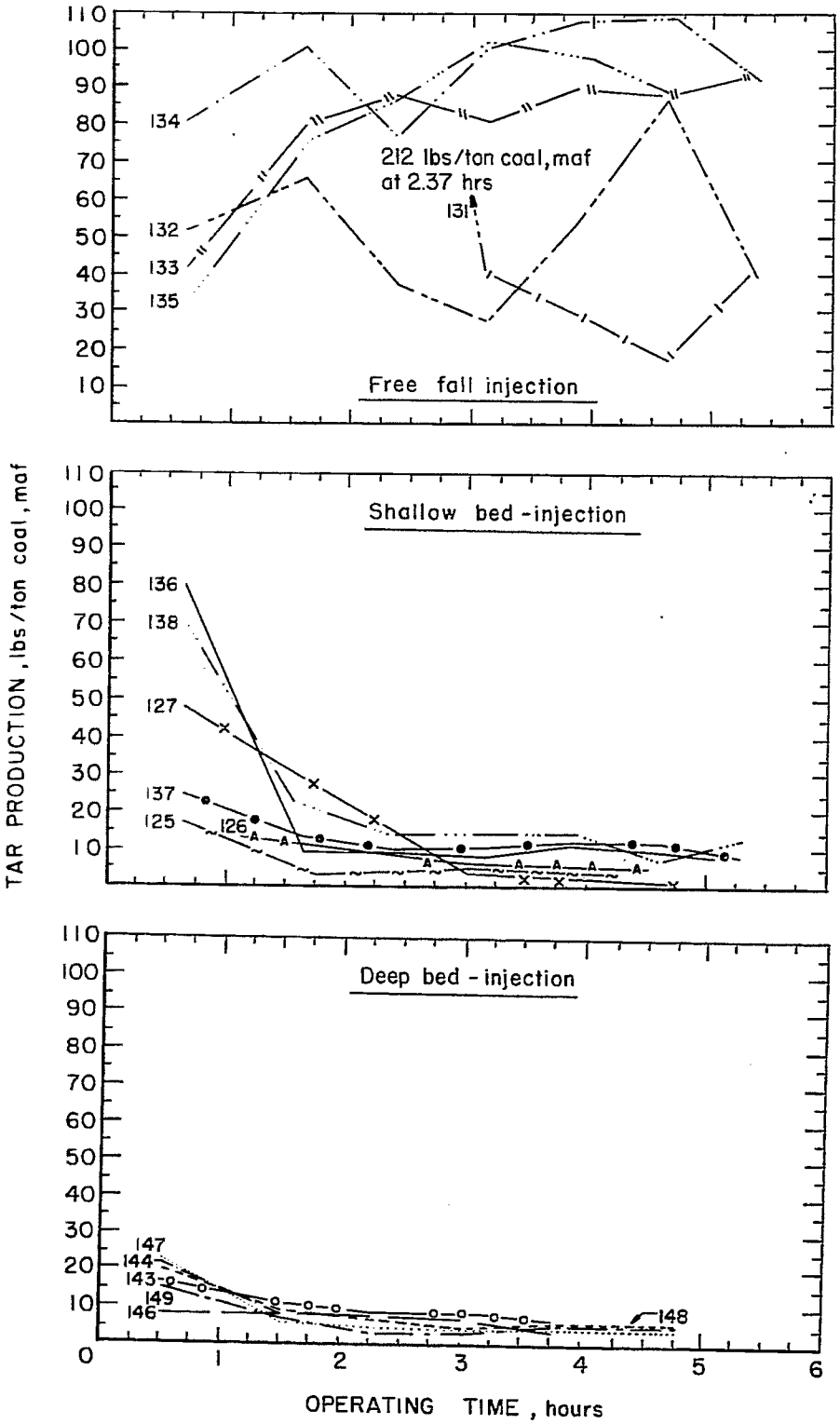

Figure 13 - Tar production versus time : gasification of North Dakota lignite

collected at different times during several gasification tests. Designations A through F were assigned to samples to distinguish the relative time of sampling during a run; A corresponds to a sample early in the run while F corresponds to a sample taken late in the run.

A cursory review of the available data in Table VII indicates that emissions from the combustion of sampled tars consistently exceed the Federal New Source Performance Standard for liquid fuels of 0.8 lbs SO_2/MMBTU[6]. However, it is also apparent from these data that, at least in the case of the free fall tests, tar approaches a steady state composition slowly and that its sulfur content consistently declines with time as steady state is approached:

Trial	Sample Time, hrs	wt% S in Tar	Equivalent lbs SO_2/MMBTU
131	0.63	1.25	1.87
	4.63	0.82	1.14
133	2.38	0.90	1.17
	4.13	0.79	1.05
135	0.63	0.98	1.44
	3.88	0.83	1.11

While a similar trend is expected for shallow and deep bed-injection tars, sufficient data have not been gathered to confirm this. A major reason for this is a lack of sufficient sample to conduct a reliable analysis. As indicated earlier, tar production rates fall precipitously as coal is injected deeper into the gasifier.

In the course of determining tar composition, a serious problem of oil-water emulsion was encountered. As is evident from the data present in Table VIII some tar samples contain significant amounts of water and its presence influences such physical properties as tar density significantly. To avoid such complications, an analytical procedure was developed for the dehydration of tars. Raw tars were selectively distilled and overhead water was checked by gas chromatography for the presence of light hydrocarbon carryover. When the distillation is conducted properly, essentially total water removal is achieved and hydrocarbon carryover is negligible (see Table VIII).

Two analyses were conducted on a select set of dehydrated tars: (1) specific gravities and (2) true boiling point (TBP) curves. As is apparent from Table VIII, the specific gravities of all measured tar samples, regardless of coal injection position or relative time of sample collection, exceed that of water (1.0). Although the data are limited, specific gravity does appear to increase with both run time (e.g., 138D versus 138A) and the depth of coal injection (e.g., 149B versus 138A versus 131D). As has been noted before, problems

Table VIII. Specific Gravities of Tars Produced During the Gasification of North Dakota Lignite: Free Fall, Shallow, and Deep Bed-Injection of Coal.

Sample	Time of Sample Collection, hrs	Specific Gravity of Tars, $60^{o}F/60^{o}F$ "As Rec'd"[a]	"Dehydrated"[b,c]	H_2O in Sample "As Rec'd", wt%	Hydrocarbons in H_2O-Phase, wt%
Free Fall Injection of Coal					
CHPFL-131D	3-1/8	1.085	1.089	15.5	1.64
CHPFL-132D	3-1/8	1.066	1.081	13.3	3.29
Shallow Bed-Injection of Coal					
CHPFL-137A	3/8	--[d]	1.087	--[d]	--[d]
CHPFL-137B&C	3/4	1.135	1.101	13.4	2.62
CHPFL-138A	3/8	1.212	1.115	11.8	1.20
CHPFL-138D	3-1/8	1.178	1.139	~ 0	~ 0
Deep Bed-Injection of Coal					
CHPFL-149B	1-1/8	1.223	1.397	17.0	0.50

Footnotes

(a) "As Rec'd" samples contain a certain amount of water due to an oil-water emulsion.

(b) Water-bearing samples were dehydrated by light end distillation. Overhead product was checked for hydrocarbon content by gas chromatography.

(c) Note that the A.P.I. gravities of each of the dehydrated tar samples are negative:

$$\text{API Gravity} = \frac{141.43}{\text{Sp.Gr.}60^{o}F/60^{o}F} - 131.4$$

(d) Water content too high for accurate measurement of specific gravity.

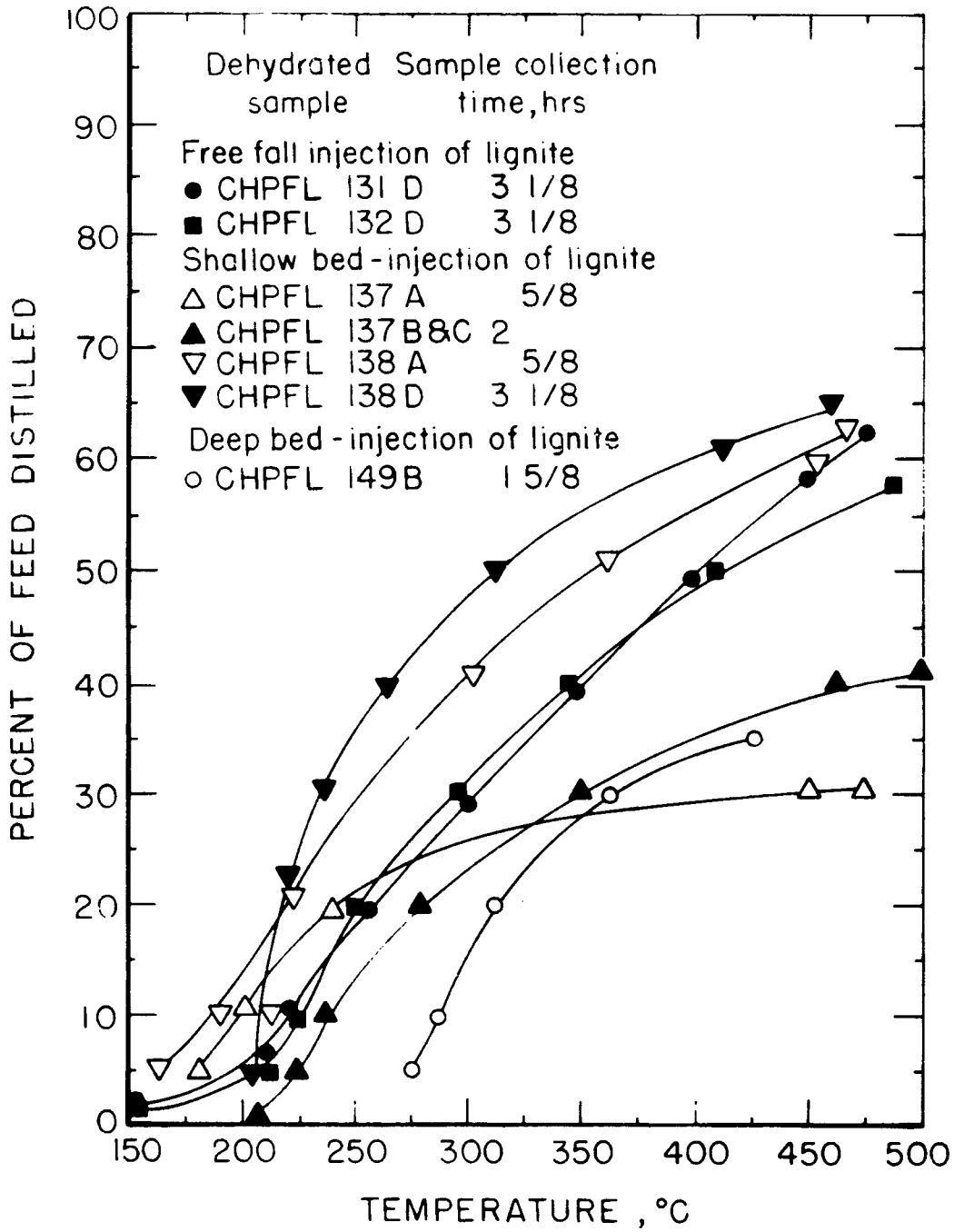

Figure 14 - True boiling point determinations of tars produced during the gasification of North Dakota lignite: Free fall shallow and deep bed - injections of the coal.

were encountered in obtaining samples of shallow and deep bed-injection tar. Production rates in these tests were so low (see Figure 13) that, for example, only one of the deep bed-injection samples collected was large enough to yield a workable quantity of tar following dehydration.

True boiling point (TBP) distillation curves for the dehydrated tar samples listed in Table VIII are presented in Figure 14. Note first that, regardless of coal injection geometry, the initial boiling point of each of the samples analyzed is substantially in excess of 100°. This suggests the presence of essentially no low molecular weight hydrocarbons, in particular benzene, toluene and xylene (BTX). Secondly, note that, depending upon the sample considered, from 30 to 60 percent of the hydrocarbons present in the tar boil at temperatures in excess of 500°C. No trend in TBP curves with coal injection geometry is discernible from the limited data available in Figure 14. Curves for two shallow bed-injection samples (138A and 138D) lie above the curves for the free fall injection samples (131D and 132D), and two (137A and 137B&C) lie below; the curve for the one available deep bed-injection sample lies below that of the free fall sample. A trend in the shape of TBP curves with reactor operating time is suggested for individual gasification runs. Tars collected late in a gasification run (samples 137B&C and 138D) have higher TBP curves, i.e., contain a slightly larger fraction of lower molecular weight hydrocarbons, than tars collected early in a run (samples 137A and 138A). However, interestingly, observed shifts toward lower molecular weight tars do not give rise to increases in BTX production, at least as measured by their presence as constituents in the tar. The BTX content of the product gas was not monitored.

DISCUSSION OF RESULTS

Time Series Analyses of Effluent Production and Composition

As had been expected, individual effluent production rates were found to vary significantly with time from reactor startup to shutdown. Available data indicate that individual production rates are typically high during reactor startup, and decline with time, typically approaching steady state levels after about 2 to 3 hours of reactor operation. Unexpectedly, effluent production rates in the free fall injection trials ran counter to this trend; production rates consistently increased with time following reactor startup. However, regardless of the coal injection geometry involved, it is significant to note that, for a given mode of gasifier operation, effluent production rates are consistently reproducible, a result not achieved previously where batch sampling techniques were employed for effluent collection (see Table II).

Interestingly, certain effluent compositions as well as production rates vary significantly with time. For example, the sulfur content of tar declines substantially during the first 3 to 5 hours of gasifier operation. Although not monitored in this experimental program, the composition of char, in particular its sulfur content, is expected to vary similarly with time.

On the basis of the extensive time series analyses presented here, it is evident that not only steady state production rates but in certain cases the composition of individual effluents must be known to assess accurately

the types and sizes of process equipment needed for satisfactory effluent treatment. Effluent data obtained from the analysis of batch samples collected over an entire run, e.g., past PERC data presented here in Table II, while qualitatively helpful in characterizing the effluent potential of a particular coal or reactor operating procedure, are not sufficiently precise to form a basis for the detailed design of an effluent treatment system.

Effect of Coal Injection Geometry on Gasifier Steady State Effluent Production and Composition

(1) Effluent Production Rates

The 19 tests conducted in this experimental program amply demonstrate that gasifier operating practice, specifically the coal injection position within the gasifier, strongly influences steady state rates of production of the full range of reactor effluents. A statistical summary of steady state values of the 7 indicators of effluent production monitored throughout this study is presented in Table IX. For purposes of comparison, similar data also are presented (where available) for weak ammonia liquor typical of a steel industry coke plant effluent. Note first that phenol production rates and chemical oxygen demand (COD) are more than a factor of 10 greater for the free fall gasification of lignite than for the manufacture of blast furnace coke. However, cyanide and thiocyanate production are more than a factor of 10 lower. Secondly, note that, although dramatic reductions in gasifier effluent production are achieved by shifting from free fall to deep bed-injection of lignite, residual phenol and COD levels are still roughly equivalent to those of untreated coke plant weak ammonia liquor. Tar production levels are greatly reduced from free fall to deep bed-injection tests -- by more than a factor of 10. However, notice that the largest percentage reduction in gasifier tar production, viz., 86.4 percent, resulted from the shift from free fall to shallow bed-injections of lignite. Increasing the depth of injection of lignite from 1-1/2 to 4-1/2 ft in the fluidized bed portion of the gasifier (deep bed-injections) resulted in an additional reduction of only 37.6 percent. Similar trends in chemical oxygen demand (COD) and total organic carbon (TOC) of aqueous effluents are apparent; COD's are reduced by 84.2 and 69.5 percent, TOC's by 78.2 and 43.8 percent, respectively. Interestingly, the above pattern does not hold for phenol production. Shifting from free fall to shallow bed-injections of lignite results in a 70.4 percent reduction in phenol production; however, shifting from shallow to deep bed-injections of lignite results in a further reduction in phenol production of 85.7 percent. Such evidence strongly suggests that different mechanisms may be responsible for observed reductions in various steady state effluent production rates with changes in fresh coal injection geometry.

On the basis of data presented here, it appears that with additional residence time, i.e., injection of coal into a deeper fluidized bed, and/or increased reaction temperature, essentially zero rates of production of the major effluents - tar, phenol, and COD - could be achieved. Consolidation Coal Company experience with its CO_2-Acceptor pilot plant in Rapid City, South Dakota tends to support this supposition. There, essentially no hydrocarbons heavier than methane are detected in gas leaving a fluid bed reactor operated at 1400 to 1500°F with a gas residence time of about 20 seconds[10].

Table IX. Comparative Steady State Effluent Production Rates: By-Product Coking, Free Fall, Shallow, and Deep Bed-Injections of North Dakota Lignite

Pollutant	By-Product Coke Plant[a]	Gasification of North Dakota Lignite: Free Fall Injection	Shallow Bed-Injection	Deep Bed-Injection
Tar, lbs/ton coal, MAF	93	74.1 ± 27	10.1 ± 5	6.3 ± 2.2
Phenol, lbs/ton coal, MAF	0.86 - 0.97	11.9 ± 1.3	3.5 ± 1.9	0.5 ± 0.6
Chemical Oxygen Demand, lbs/ton coal, MAF	4.0 - 5.5	77.7 ± 14.4	11.8 ± 5.4	3.6 ± 2.4
Total Organic Carbon, lbs/ton coal, MAF	1.60 - 1.96	22.0 ± 3.3	4.8 ± 1.3	2.7 ± 0.7
Inorganic Carbon, lbs/ton coal, MAF[c]	N.D.	12.5 ± 2.4	11.0 ± 2.3	11.4 ± 2.4
Cyanide, lbs/ton coal, MAF	0.018 - 0.054	Negligible	Negligible	Negligible
Thiocyanate, lbs/ton coal, MAF	0.31 - 0.35	0.045 ± 0.083	≤0.016 ± 0.002[b]	≤0.017 ± 004[b]

Notes
(a) Sources: References 7, 8, and 9.
(b) Measurements at or below the limiting sensitivity of the analytical method employed.
(c) The relatively constant inorganic carbon production levels reported for different lignite injection positions are expected and reflect the presence of a saturated amount of CO_2 in basic (pH of 9) condensate.

(2) Tar Production

Although tar production rates drop precipitously with the shift from free fall to deep bed-injection of lignite, the overall composition of condensible hydrocarbons remains remarkably similar (see true boiling point curves in Figure 14). This is an unexpected result. It had been presumed that reductions in the yield of heavy tar would be paralleled by a greatly increased production of light hydrocarbons including benzene, toluene, and xylene (BTX). In fact, little, if any BTX was found in free fall tars, and no significant percentage increase in light hydrocarbons was observed as overall tar production declined. This observation may be important in view of various independent reports of high yields of BTX in bench-scale experiments in which coal is heated rapidly, exposed to high partial pressures of hydrogen for a short time (typically less than 10 sec) and subsequently quenched.[9-11] While coal heatup rates were necessarily relatively low in all the free fall injection tests (most of the length of the carbonizer was heated to only about 400°C; see Figure 4), they were quite rapid in both the shallow and the deep bed-injection tests. At the same time, gas residence times at the elevated temperatures in the fluid bed portion of the gasifier were relatively short, less than 7 seconds even in the deep bed-injection tests. The essentially complete lack of BTX production (i.e., condensed BTX; quenched product gas was not analyzed for BTX) under these conditions as well as the consistently high molecular weight of residual tar stand in sharp contrast to smaller-scale laboratory results referenced above. While hydrogen partial pressures were significantly lower in the present experiments (approximately 180 versus 1000 psig or more) and there was a significant partial pressure of water in each of the present tests, the relevance of these differences is unknown.

Effect of Coal Injection Geometry on the Distribution of Sulfur in Gasifier Effluents

It has been noted that all the chars produced in both the free fall and the shallow bed-injection tests met the New Source Performance Standard (NSPS) for SO_2 emissions during combustion; deep bed-injection chars consistently were out of compliance. In contrast, tar sulfur contents exceeded the NSPS regardless of coal injection geometry. However, with increasing depth of injection, total tar production diminishes rapidly. This raises a significant question: is the partitioning of feed coal sulfur in the gasifier between eventual boiler fuels (tar and char) and raw product gas influenced by coal injection geometry? As can be seen by the calculated ratios of sulfur in char and tar relative to total feed sulfur presented in Table X, partitioning of sulfur between raw product gas and the combined char and tar streams appears to be essentially independent of coal injection geometry.

Table X. Distribution of Coal Sulfur Among Tar, Char and Product Gases: Free Fall, Shallow, and Deep Bed-Injections of North Dakota Lignite

Trial No.	$\frac{\text{lbs S in Char \& Tar}}{\text{Total lbs S in Feed Coal}}$	
Free Fall Injections		
CHPFL-131	0.75	Average --- 0.57 ± 0.23
CHPFL-132	0.24	
CHPFL-133	0.71	
CHPFL-134	0.42	
CHPFL-135	0.72	
Shallow Bed-Injections		
CHPFL-123	0.57	Average --- 0.56 ± 0.09
CHPFL-124	0.45	
CHPFL-125	0.54	
CHPFL-126	0.47	
CHPFL-127	0.47	
CHPFL-136	0.61	
CHPFL-137	0.69	
CHPFL-138	0.69	
Deep Bed-Injections		
CHPFL-143	0.58	Average --- 0.54 ± 0.09
CHPFL-144	0.44	
CHPFL-146	0.63	
CHPFL-147	0.43	
CHPFL-148	0.51	
CHPFL-149	0.63	

Further, even though there is considerable scatter in the data, on the average slightly more than half of the sulfur in the feed coal reports to char and/or tar and therefore is eventually emitted as SO_2 during combustion of these materials as fuel for power generation. Apparently, modified gasifier design does not offer significant prospects for decreased sulfur loads on plant boilers and therefore dimished loads for SO_2 stack gas cleaning equipment. Such a conclusion is necessarily tentative since time series analyses on the sulfur content of char were not conducted in this experimental program. If char sulfur contents decline with time as tars have been shown to do (see Page 39), the overall sulfur distribution would shift accordingly.

Effect of Coal Injection Geometry on Total Product and Methane Gas Yields

Summary statistics on gasifier temperature profiles, total product gas, equivalent methane yields, and effective product gas residence times for each of the three coal injection geometries investigated in the current program are presented in Table XI. Similar statistics are included for 13 North Dakota lignite tests conducted previously and cited earlier (Tables I and II). Within the accuracy of the data available, equivalent methane yields are clearly unaffected by changes in coal injection geometry in both current and past lignite tests. Similarly, within the degree of accuracy of the available data, total product gas yields are essentially unaffected by changes in coal injection geometry.

Possible Mechanisms to Explain Observed Results

It is startling to find that production rates of essentially all undesirable "byproducts" of coal gasification (except sulfur) can be dramatically reduced by minor modifications in gasifier design without any measurable reduction in desirable products, viz., equivalent methane, total product gas and ammonia. Such a result clearly suggests that distinctly different mechanisms must be at work in the production and/or destruction of various gasifier constituents. In varying the coal injection geometry from free fall to deep bed-injection, several operating conditions change in the gasifier. Among these are: (1) the heatup rate of the fresh coal is greatly increased; (2) the mean reaction temperature is increased; (3) gas-solid mixing is enhanced; (4) product gas residence time is increased; and (5) contact between fresh product gas and potentially catalytic char particles is increased. Depending upon the specific coal injection geometry involved, changes in various subsets of these operating conditions tend to dominate.

In shifting from free fall to shallow bed-injections of coal, the major reactor conditions undergoing change are fresh coal heatup rate and mean reaction gas temperature. Although a study is needed to verify it, a major portion of the observed improvement in effluent production might well be attributed to a reduced rate of formation under the more severe conditions of shallow bed-injection operation rather than to decomposition of formed material. However, even if such a supposition is shown to be correct,

Table XI. Summary Statistics, Gasification of North Dakota Lignite: Free Fall, Shallow, and Deep Bed-Injection of the Coal.

Parameter	Coal Feed Configuration[a]		
	Free Fall Injection	Shallow Bed-Injection	Deep Bed-Injection
PAST NORTH DAKOTA LIGNITE TRIALS (See Table I.)			
Fluid Bed Temp Profile, °C			
Bottom	878 ± 29.00 (13)[a]	879 ± 4.00 (3)	-- --
Average	842 ± 20.00 (12)	844 ± 2.00 (3)	-- --
Top	665 ± 63.00 (13)	716 ± 19.00 (3)	-- --
Total Product Gas SCF/lb C Gasified[b,c]	28.63 ± 5.56 (13)	33.24 ± 2.58 (3)	-- --
Equiv. CH_4 Production SCF/lb C Gasified[b,d]	6.24 ± 1.19 (13)	7.32 ± 0.53 (3)	-- --
Effective Gas Residence Time, sec[e]	-0-	2.58 ± .03 (3)	-- --
CURRENT NORTH DAKOTA LIGNITE TRIALS (See Table III.)			
Fluid Bed Temp Profile, °C			
Bottom	866 ± 9.00 (5)	843 ± 26.00 (8)	803 ± 20.00 (6)
Average	828 ± 5.00 (5)	789 ± 8.00 (8)	773 ± 12.00 (6)
Top	768 ± 36.00 (5)	723 ± 13.00 (8)	723 ± 10.00 (6)
Total Product Gas SCF/lb C Gasified[b,c]	33.26 ± 2.10 (4)[f]	29.76 ± 3.10 (8)	35.50 ± 2.30 (5)[g]
Equiv. CH_4 Production SCF/lb C Gasified[b,d]	5.70 ± .47 (4)[f]	5.92 ± 1.13 (8)	5.62 ± 0.47 (5)[g]
Effective Gas Residence Time, sec[e]	-0-	2.82 ± .11 (8)	6.62 ± .11 (6)

Footnotes:

(a) Data in parentheses throughout this table indicate the number of experiments considered in reported average results.

(b) Gas production figures are stated relative to lbs of carbon gasified to correct for wide variations in the extent of gasification achieved. See text for explanation.

(c) Total gas production includes CH_4, C_2H_6, H_2, and CO.

(d) Equivalent CH_4 is the sum of SCF of CH_4 and twice the SCF of C_2H_6.

(e) Estimated effective residence time of freshly volatilized coal hydrocarbons in the high temperature, fluidized bed portion of the gasification reactor (see Figure 3). Calculations are based on an assumed plug flow of gases through the reactor. The effective residence time for all free fall runs is assumed to be essentially zero.

(f) Run CHPFL-133 has been omitted from this average.

(g) Run CHPFL-143 has been omitted from this average.

it cannot account for all of the observed reduction in effluent production rates since deep bed-injections of lignite produce additional reductions in effluent production. Further, since reaction temperatures in the deep bed-injection tests were actually lower than those in the shallow bed-injection tests, temperature cannot explain the observed reductions. The only significant changes in operating conditions appear to have been increased product gas residence time and additional gas-solid contacting. It would appear that at least these two significant mechanisms influence the observed reductions in effluent production with shifts from the shallow to the deep bed coal injection geometry. No attempt was made in the present experimental program to sort out the dominant mechanisms involved. However, bench-scale work is now in progress to determine the relative importance of various reactor operating parameters in effluent production.

Applicability of Current Results to Other Coals and/or Different Gasifiers

Lignite coal was selected for testing here specifically because it is a non-caking coal. Although caking coals (e.g., Illinois No. 6 and Pittsburgh Seam) appear to produce most effluents in yields similar to those of lignite (refer to Table II), they differ in one important respect; tar production is substantially greater for caking than for non-caking coals. In addition, due to their agglomerating properties during heatup, caking coals are difficult to feed to a gasifier in any geometry other than free fall. PERC efforts to date to inject pretreated caking coal directly into the fluidized bed portion of the Synthane gasifier have been unsuccessful. If a satisfactory means of injection is eventually found, results of the present study on lignite indicate that more severe processing conditions will be required to treat caking coal to reach the reduced levels of effluent production achieved with lignite.

Without some quantitative understanding of the mechanisms responsible for the reductions in effluent production observed here, projections of possible effects in other gasifiers are speculative at best. A qualitative interpretation of the data here suggests that observed effluent reductions are not specific to the Synthane gasifier; this should be achievable in other reactors.

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INFLUENCE OF SYNTHANE GASIFIER CONDITIONS ON EFFLUENT AND PRODUCT GAS PRODUCTION

by

David V. Nakles, Michael J. Massey, Albert J. Forney
and W. P. Haynes

ERRATA

Page 45, Table X should read:

Table X. Distribution of Coal Sulfur Among Tar, Char, and Product Gases: Free Fall, Shallow, and Deep Bed-Injections of North Dakota Lignite

Trial No.	$\left(\frac{\text{lbs S in Char and Tar}}{\text{Total lbs S in Feed Coal}}\right)$	
Free Fall Injections		
CHPFL-131	.1017	Average --- .1065 ± .051
CHPFL-133	.0580	
CHPFL-135	.1598	
Shallow Bed-Injection		
138	.0800	
Deep Bed-Injection		
147	.2256	

Page 44, the text should read:

Effect of Coal Injection Geometry on the Distribution of Sulfur in Gasifier Effluents

It has been noted that all the chars produced in both the free fall and the shallow bed-injection tests met the New Source Performance Standard (NSPS) for SO_2 emissions during combustion; deep bed-injection chars consistently were out of compliance. In contrast, tar sulfur contents exceeded the NSPS regardless of coal injection geometry. However, with increasing depth of injection, total tar production diminishes rapidly. This raises a significant question: is the partitioning of feed coal sulfur in the gasifier between eventual boiler fuels (tar and char) and raw product gas influenced by coal injection geometry? Due to limited tar sulfur analyses, this partitioning factor (pounds of sulfur in char and tar per pound of sulfur in the feed coal) was determined for only 5 trials: 3 free fall, 1 shallow, and 1 deep bed-injection. These factors are presented in Table X. As can be seen from these calculated ratios, there appears to be no

significant effect of coal injection geometry until the deep bed-injection trial. The increase observed during this trial reflects the production of a large quantity of high sulfur char, since little or no tar was produced. As discussed earlier (pg. 36), it is believed that this high sulfur char was the result of the feed coal exiting the gasifier prematurely through the char extractor after undergoing only partial conversion. In a properly modified reactor, this may not be the case. Although only limited data are available, it is apparent that modified gasifier design does not in itself offer significant prospects for decreased sulfur loads on plant boilers and therefore diminished loads for SO_2 stack gas cleaning equipment. Such a conclusion is necessarily tentative since time series analyses on the sulfur content of char were not conducted in this experimental program. If char sulfur contents decline with time as tars have been shown to do (see pg. 39), the overall sulfur distribution would shift accordingly.

Pg. 9, Table II, in the column titled "Trial"

CHPFI-74 should read CHPFI-74[d]

CHPFI-75 should read CHPFI-75[d]

CHPFI-77 should read CHPFI-77[d]

Pg., 12 Figure 1a and 1b. The following trial numbers represent shallow bed injections:

CHPL-5
CHPL-6
CHPL-7

All other trial numbers represent free fall injections.

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