FLUO-SOLIDS COMBUSTION PROCESS FOR REGENERATION OF SPENT ZINC CHLORIDE CATALYSTS

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INTRODUCTION

The application of molten zinc chloride catalysts to the hydrocracking of polynuclear hydrocarbons, coal, and coal extract has been discussed previously.[1,2,3] During the hydrocracking process, the catalyst partially reacts with nitrogen and sulfur in the feed according to the reactions,

$$ZnCl_2 + H_2S = ZnS + 2\ HCl \quad (1)$$

$$ZnCl_2 + xNH_3 = ZnCl_2 \cdot xNH_3 \quad (2)$$

$$ZnCl_2 \cdot yNH_3 + yHCl = ZnCl_2 \cdot yNH_4Cl \quad (3)$$

Addition of zinc oxide to the hydrocracking catalyst eliminates reaction (3) and introduces a new reaction

$$ZnO + 2\ HCl = ZnCl_2 + H_2O \quad (4)$$

The melt leaving the hydrocracker thus, in general, will contain in addition to $ZnCl_2$, the following compounds: ZnS, $ZnCl_2 \cdot xNH_3$, $ZnCl_2 \cdot yNH_4Cl$. The ratio of the NH_4Cl to NH_3 adducts is dependent on the ratio of nitrogen to sulfur in the feed and the amount of ZnO added to the feed melt. In addition to inorganic impurities, the melt contains organic residue that cannot be distilled out of the melt. These impurities are quite uniformly distributed in the melt and normally do not settle out of the bulk melt phase.

A commercial process using zinc chloride must provide a viable scheme for regeneration of the catalyst. Regeneration of the melt comprises removal of the bulk of the nitrogen, sulfur and carbon impurities and return of the melt as relatively pure zinc chloride.

The purpose of this paper is to present data on regeneration of the melt using a fluo-solids combustor. The combustion process removes carbon, nitrogen and sulfur from the melt, simultaneously vaporizing the zinc chloride which is subsequently condensed downstream from the combustor.

EXPERIMENTAL

Equipment

A diagram of the equipment is shown in Figure 1. The melt feed is dropped from a drip tip on the top flange of the combustor into the fluo-solids bed contained in the mullite reactor liner. The fluidizing air is supplied via a tube that enters the combustor at the upper flange and extends to within 3/4" of the tip of the liner cone where it discharges into the fluo-solids bed. The vapors from the combustor are cooled to 650-700°F in the condenser where the zinc chloride condenses and HCl and ZnO, formed by hydrolysis in the oxidation zone, interact to re-form $ZnCl_2$ and H_2O. Residual $ZnCl_2$ fog from the condenser is removed from the gas stream by electrostatic precipitators operated at 650°F, i.e., above the melting point of $ZnCl_2$.

The effluent gas from the precipitators (essentially $ZnCl_2$-free) is passed through a cooler where water and some HCl are removed. A small side-stream of the "dry" gas is passed through an Ascarite trap to remove acid gases and then to a Beckman Model E-2

oxygen analyzer. The main stream of dry gas is passed through tandem scrubbers containing aqueous hydrogen peroxide which removes SO_2 plus HCl and aqueous sodium hydroxide which removes CO_2 from the product gas. A fraction of the offgas is diverted to a gas holder and the remainder is metered and vented.

Procedure and Product Workup

To charge the fluo-solids bed and start a run, the thermowell of the combustor is replaced with a tube surmounted by a closed hopper containing fluosolids. The fluidizing air and argon purge flows are then established, and the fluosolids are charged to the reactor held at 1200-1400°F. After replacing the thermowell, the desired pressure is established, and the combustor is heated to about 50°F below the desired run temperature. The feed is then started with the vapors going to the line-out train. When all temperatures are lined out and the oxygen content of the effluent gas is constant, the vapors are diverted through the balance train to start the balance period. The weight of melt fed during the balance varied from 900 to 6700 grams, depending on the feed rate.

All products, including the fluo-solids, are collected and analyzed. Determinations are made of chlorine on the product water, chlorine and sulfur on the hydrogen peroxide scrubber effluent, and chlorine and CO_2 on the sodium hydroxide scrubber effluent to obtain the amounts of HCl, SO_2 and CO_2 collected in these materials. The scrubbed gas collected in the gas holder is analyzed for H, CO, CO_2, SO_2, N_2, A and O_2 by two-stage gas chromatography.[4] The results from the water, scrubbers, and gas holder are consolidated to obtain the effluent gas composition. Material balances and elemental balances are made. The amount of ammonia decomposition is determined by the difference between inorganic nitrogens in the feed and effluent melts. The methods for determining the amount of ZnS and NH_3 in the melts have been described previously.[2]

Range of Variables Explored

Temperature, °F	1800 and 1900
Pressure, psia	15-46
Superficial air velocity, fps	0.16-2.67
Melt feed rate, lb/(ft^2-hr)	7.1-347
Superficial vapor residence time, sec.	0.4-6.3
% of Stoichiometric air	44-115
Fluo-solids bed depth, inches	12-16
Types of fluo-solids	Silica sand, ZnO sinter, mullite

Superficial air velocity is defined as the velocity of the air feed at process conditions based on the empty reactor. Superficial residence time is based on the superficial velocity and the fluo-solids bed depth. Stoichiometric air is defined as the amount to completely burn the NH_3, ZnS and organic residue contents of the feed melt according to the following reactions,

$$NH_3 + 3/4\ O_2 = 1/2\ N_2 + 3/2\ H_2O \quad (5)$$

$$ZnS + 3/2\ O_2 = ZnO + SO_2 \quad (6)$$

$$CH_x + (1 + \frac{x}{4})\ O_2 = CO_2 + \frac{x}{2}\ H_2O \quad (7)$$

The size consists of the fluo-solids were chosen to match the fluidizing velocities used. The criteria are that the bed be well fluidized (bed expansion over the fixed bed is typically 50-150%) and the terminal velocity of the smallest particle is such that it is not elutriated out of the bed. The size consists used varied from 100 x 150 mesh at a fluidizing velocity of 0.16 fps to 20 x 28 mesh at a fluidizing velocity of 2.67 fps.

Feedstocks Used

Analyses of the melt feedstocks are given in Table I. The natural melts were produced in a continuous $ZnCl_2$ hydrocracker with coal extract feed as described previously.

The low carbon and high carbon melts contained, respectively, 7.2 and 11.6% of the carbon in the feed extract. A synthetic melt was used before natural melt became available. It was prepared from lampblack (Columbian Eagle Brand), $ZnCl_2$ (Fisher Scientific Co., certified reagent, 98.3% pure), ZnS (Fisher Scientific Co., "purified" grade, 97% pure, 0.3 μ particle size) and anhydrous NH_3 (Matheson Co., 99.99% pure).

RESULTS

Results With Low-Carbon Feedstocks and Near-Stoichiometric Air

Some basic features of fluo-solids combustion with the amount of stoichiometric air varying between 77 and 116% are illustrated in Runs 1 through 4 of Table II. In these runs, the air input was maintained constant to give a linear velocity of 0.4 fps and the percent of stoichiometric air was varied by changing the melt feed rate. Nearly complete burn-out of the organic, sulfur, and nitrogen impurities is obtained at air inputs from 115 to 90% of stoichiometric. Sulfur is burned to SO_2, carbon to CO_2 plus CO, and ammonia to nitrogen plus water. With excess air, there is a substantial amount of oxygen in the off-gas since the impurities are insufficient to utilize all of the oxygen fed. However, with a slight deficiency of air a substantially oxygen-free offgas is generated which contains sufficient CO to provide for reduction of the SO_2 to elementary sulfur by the reaction

$$2\ CO + SO_2 = 1/2\ S_2 + 2\ CO_2 \qquad (8)$$

As the percent of stoichiometric air is lowered further from 90 to 77%, the CO content of the gas further increases and a substantial amount of hydrogen appears. There is also a very marked decrease in the burnout of inorganic sulfur, but only a moderate decrease in carbon burnout. Thus, in this case, sulfur rejection to gas is poor and the amount of CO in the gas is in large excess of that required for reduction of the SO_2 to elementary sulfur. The rejection of NH_3 by combustion is also poorer at low air rates.

The heating value of the gas increases with decreasing amounts of stoichiometric air due to the increasing amounts of CO and H_2 produced, but at 77% of stoichiometric the heating value is still too low for the gas to be of value.

The above data show that the impurities are selectively oxidized from the melt in preference to zinc chloride.

Zinc chloride is essentially inert to direct oxidation according to the reaction

$$ZnCl_2 + 1/2\ O_2 = ZnO + Cl_2 \qquad (9)$$

This is shown by the fact that no Cl_2 was found in the effluent gas at any condition tested. However, significant quantities of HCl appear in the effluent gas as the data of Table II show. It is produced by hydrolysis of $ZnCl_2$ vapor with the steam generated in the combustion process. This is discussed more fully below.

Operability of the fluo-solids combustor was demonstrated over a wide range of spent melt feed rates, i.e., 7.1-347 lb/(ft^2-hr). The latter value closely approaches commercially attractive rates. Run 5 of Table II illustrates results at the high feed rate of 347 lb/(ft^2-hr) under 3 atmospheres pressure. Allowing for the lower residence time, it is seen that the results are similar to those obtained at 1 atmosphere in the lower capacity operation of Run 4. The same high feed rate was demonstrated at 1.2 atmospheres pressure using a linear velocity of 2.67 fps at 65% of stoichiometric air. The results, not presented here, were quite similar to those of Run 5. No difficulties due to agglomeration of the bed solids were encountered in any of the work.

Effects of Variables

Figures 2, 3 and 4 show the effect of the variables on the distribution of carbon and sulfur in the product gas, melt, and fluo-solids. The reason that the distribution in the fluo-solids has to be taken into account is that the fluo-solids tend to trap and retain non-volatile solids (C, ZnS, and ZnO), and that a steady-state condition in the fluo-solids bed was not reached with respect to this retention. The carbon or sulfur retained in the bed, as obtained by difference on the plots, includes losses. However, analyses of the used fluo-solids confirm that the bulk of the sulfur and carbon not appearing in either the gas or melt was retained in the fluo-solids bed. The tendency of the fluo-solids bed to trap non-volatile solids provides for retention and better utilization of the combustibles. In addition, the bed acts as a thermal flywheel for maintaining ignition.

Figure 2 shows the effect of the percent of stoichiometric air on the distribution of carbon in the products, using the natural melt feed and the similar synthetic melt feed. With the natural melt feed there is a steady decrease of carbon burnout as the percent of stoichiometric air decreases, but burn-out still is quite good even at 54% of stoichiometric air. As the carbon burnout decreases, the carbon retained in the bed substantially increases. Carbon burnout does not decrease appreciably with reduction of percent stoichiometric air in the case of the synthetic melt feed. This may be due to a higher reactivity of the carbon black but more likely it is due to superior fluidization properties of the silica sand used as compared with the ZnO sinter used for the natural melt runs.

Figure 3 shows how, with a deficiency of air, the carbon burnout efficiency increases with increasing residence time. At constant residence time, increasing the pressure fro 1.0 to 3.1 atmospheres appears to increase somewhat the efficiency of carbon burnout.

Figure 4 illustrates how sulfur burnout drops abruptly from almost complete burnout at 90% or more of stoichiometric air to where only small amounts of sulfur are gasified at air inputs below about 80% of stoichiometric. Although some of the unburned ZnS is carried over into the melt, the majority is retained in the bed. With the synthetic melt feed, the sulfur burnout is less complete, whereas carbon burnout was more complete Possible reasons for this were given above. The results do indicate that, at some conditions, it is possible to produce a low-sulfur off-gas with as high as 80% of stoichiometric air.

Figure 5 shows that ammonia decomposition decreases quite rapidly and almost linearly as the percent of stoichiometric air is reduced below 100% at 2.4 seconds residence time. Ammonia can be destroyed either by oxidation according to reaction (4) or by thermal decomposition according to the following reaction:

$$ZnCl_2 \cdot NH_3 = ZnCl_2 + 1/2\ N_2 + 3/2\ H_2 \qquad (10)$$

When operating with approximately stoichiometric air, the oxidation reaction likely predominates, and decomposition is quite complete in 2.4 seconds residence time. With a deficiency of air, thermal decomposition by Reaction (10) probably predominates. Since thermal decomposition of ammonia is relatively slow, longer residence times and/or high temperatures are required to get efficient ammonia decomposition when operating with a deficiency of air. It is important that the ammonia content of the regenerated melt be low because ammonia inhibits the hydrocracking activity of the melt.

Results With the High-Carbon Feedstock

The results with the low-carbon feedstocks indicated that, by operating with a deficiency of air, a low-sulfur, low-Btu gas might be produced during the melt regeneration. Table III gives experimental examples of results with the high-carbon melt feed using a large deficiency of air, i.e., 44 to 59% of stoichiometric.

These results show that a low-sulfur fuel gas can be obtained by operating with 56% or less of the stoichiometric air at 1800°F and 6.25 seconds contact time. The Btu content of the gas goes up as the air i put is reduced, as expected. The highest heating value achieved in this work was 81 Btu/ft^3 at 44% of stoichiometric air input.

Two runs, 6 and 7, are shown in Table III wherein the residence time was decreased to 2.4 seconds with approximately the same air input as Run 8. A substantial increase in sulfur dioxide content of the gas and a decrease in carbon burnout efficiency was observed. Comparison of runs 6 and 7 show that increase of temperature from 1800 to 1900°F strongly increases the extent of NH_3 decomposition and also increases the Btu level of the gas. As with the lower-carbon melt, HCl appears in the effluent gas in an amount equivalent to 2 to 3 wt. % of the feed melt.

Effects of the Variables

Figures 6 and 7 show the distribution of carbon and sulfur, respectively, in the products as a function of percent stoichiometric air at residence times of 6.25 and 2.4 seconds. These distributions generally follow a similar pattern to that obtained with the low-carbon melts. Figure 8 shows the heating value of the offgas as a function of percent stoichiometric air at the residence times of 6.25 and 2.4 seconds. The main point brought out by Figures 6, 7, and 8 is that a minimum gas residence time in excess of 2.4 seconds is required to get efficient carbon burnout at 1800°F and to produce a substantially sulfur-free gas of relatively high heating value. The residence time required to achieve similar results is lower at a higher temperature as comparison of results in Table III with those in Figures 6, 7, and 8 show.

DISCUSSION

HCl in the Product Gas and Recovery

Steam generated in the combustion process hydrolyzes $ZnCl_2$ vapors to produce HCl by the reaction

$$ZnCl_2 + H_2O = ZnO + 2\ HCl \qquad (11)$$

The HCl formed is largely recovered by reversal of the hydrolysis reaction at the lower temperature of the $ZnCl_2$ condenser. There is sufficient total hydrogen in the low-carbon feed melt, for example, to potentially hydrolyze 74% of the $ZnCl_2$ in the feed if all of the hydrogen were oxidized to water. Reversal of the reaction in these studies was incomplete, and hence, significant amounts of HCl appeared in the product gas as shown in Tables II and III. The reasons for this are: (1) Relatively inefficient contact between the ZnO dust and HCl in the condenser, and (2) ZnO starvation in the condenser. The ZnO starvation was caused by retention of some ZnO in the fluo-solids bed.

Separate studies, not reported here,[5] have shown that with an efficient scrubber operated at 650-750°F much better HCl recovery can be obtained than was demonstrated in this work. The equilibrium constant for the hydrolysis reaction at 650 and 750°F was bracketed experimentally by studying the reaction from the H_2O side as well as the HCl side. The values obtained are:

	650°F	750°F
$K = \frac{(P_{HCl})^2}{P_{H_2O}} =$	0.59×10^{-4} to 1.62×10^{-4}	0.32×10^{-3} to 0.87 to 10^{-3}

This work indicates that if equilibrium had been reached at 650°F in the condenser with the gas generated in Run 2 of Table II, HCl in the effluent gas would be 0.63 wt % or lower (on melt), instead of the 2.8% actually observed.

Mechanism of Formation of Low-Sulfur Fuel Gas

The results above have shown that by operating with a relatively large deficiency of air, a low-sulfur fuel gas is produced providing sufficient residence times are employed. Large amounts of CO and H_2 are produced by incomplete combustion and also by secondary gasification of unburned carbon by CO_2 and H_2O. Undoubtedly, SO_2 is generated in the lower part of the fluo-solids bed (near the air inlet) by roasting of ZnS (Reaction 6). However, if sufficiently long vapor residence times are used, the following reactions tend to proceed to completion:

$$3\ H_2 + SO_2 = H_2S + 2\ H_2O \qquad K_{12} = (H_2S)(H_2O)^2/(H_2)^3(SO_2) = 6.3 \times 10^4 \text{ at } 1800°F \quad (12)$$

$$H_2S + ZnO = ZnS + H_2O \qquad K_{13} = (H_2O)/(H_2S) = 6.8 \times 10^2 \text{ at } 1800°F \quad (13)$$

$$3\ H_2 + SO_2 + ZnO = ZnS + 3\ H_2O \qquad K_{14} = (H_2O)^3/(H_2)^3(SO_2) = 4.3 \times 10^7$$

The equilibrium constant K_{12} was calculated from spectroscopic data of Gordon.[6] The value of K_{13} was obtained from the product of the equilibrium constants for Reactions (1) and (4). The equilibrium constant for Reaction (1) was obtained from experimental data of Kapustinskii[7] and that for Reaction (4) was calculated from available thermodynamic data on zinc compounds.[8] Extrapolation to 1800°F on a plot of log K vs 1/T was required to obtain K_{13}.

The ZnO trapped in the fluo-solids bed thus acts as a sulfur acceptor, and if equilibrium in Reaction (13) is established, the gas produced is almost completely free of sulfur. Thus, for example, if $P_{H_2O} = 5 \times 10^{-2}$ atm and $P_{H_2} = 5 \times 10^{-2}$ atm, then at equilibrium $P_{SO_2} = 2.3 \times 10^{-8}$ atm and $P_{H_2S} = 7.3 \times 10^{-5}$ atm.

Application of Results to a Commercial Process

In order to conduct the regeneration process adiabatically, it is necessary that the heats of combustion of the organic, NH_3, and ZnS impurities in the spent melt be sufficiently great to vaporize the melt and achieve the desired bed temperature. The operating temperature range would generally lie within the limits of about 1600-2100°F. The maximum temperature is dictated by the point at which defluidization occurs due to incipient sintering of the fluo-solids. The minimum temperature is dictated by the point at which adequate reaction kinetics prevail for carbon combustion and nitrogen and sulfur removal. In general, the preferred operating temperature is probably in the range of 1750-1950°F.

Figure 9 gives the adiabatic combustor temperature for several feed melts as a function of the percent of stoichiometric air and the melt carbon gasified. The input melt and air temperatures were assumed to be 800°F and 440°F, respectively, while product distributions obtained in actual runs were used in calculation of the heat and material balances. Extrapolation of the results of Figure 9 shows that at 100% stoichiometric air the feed melt must contain between 3.5 to 4.7% in order to remain within the preferred temperature limits. Somewhat higher carbon melts, i.e., up to about 6%, can be processed by lowering the preheat temperatures or by making a slight reduction in the amount of air used. Still higher carbon feeds require further lowering the percent of stoichiometric air or cooling of the combustor. Operation within the intermediate range of stoichiometric air (between 70 and 90%) has two drawbacks. First, the Btu content of the offgas is too low, and its sulfur content too high, for it to be useful as a fuel gas. Second, rejection of sulfur in a single-stage oxidation is incomplete.

Therefore, two types of operating regimes are of chief interest. The first is single-stage combustion with near-stoichiometric air using a feed containing 3.5-6.0% carbon. In this case substantially all of the impurities are burned out of the melt. By fine adjustment of the air input at about 90% of stoichiometric, a substantially oxygen-free gas containing sufficient CO can be generated to permit subsequent reduction of the SO_2 in the off-gas to elementary sulfur.

The second operating regime is a two-stage process that handles a relatively high-carbon melt and uses a deficiency of air, i.e., about 35-60% of stoichiometric, in order to stay within the operating temperature limits. In this type of process, a low-sulfur fuel gas is produced in the first stage and the sulfur is largely trapped as ZnS in the fluo-solids bed. This sulfur is rejected by a separate combustion operation where the trapped sulfur is removed by Reaction (6). The sulfur is liberated as a separate, concentrated SO_2 stream, from which it is relatively easy to recover elementary sulfur.

Acknowledgment

The sponsorship by the U.S. Office of Coal Research of this investigation is gratefully acknowledged.

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TABLE I

Analysis of Melts Used

Composition	"Low" Carbon Natural Spent Melt	"High" Carbon Natural Spent Melt	Synthetic Melt
Wt %			
NH_3	1.56	1.35	1.37
C	5.42	8.76	5.78
org. H	0.25	0.44	--
org. N	0.17	0.16	0.13
org. S	0.05	0.07	0.08
$ZnCl_2$	84.27	80.55	84.77
ZnS	4.32	3.62	4.59
ZnO	0.46	1.37	0.70
H_2O	3.50	3.68	2.58

TABLE II

Results With Low-Carbon Natural Melt at Higher Air/Melt Ratios

Temperature = 1800°F

Run No.	1	2	3	4	5
% of Stoichiometric Air	115	108	90	77	74
Superficial Air Velocity, fps	0.41	0.38	0.42	0.42	1.05
Superficial Residence Time, sec	2.44	2.63	2.39	2.38	0.95
Pressure, psia	15.6	16.9	15.0	15.2	46
Melt Feed Rate, lb/(hr)(ft^2)	28.6	31.0	34.0	54.5	347
Results					
% NH_3 Burned to N_2 + H_2O	98	89	78	67	~ 50
% C Burned to CO + CO_2	95	91	89	86	87
% ZnS Burned to SO_2 + ZnO	100	100	91	28	29
HCl Produced, Wt % Feed Melt	3.3	2.0	2.8	3.5	2.4
Dry Exit Gas, Vol %					
SO_2	1.44	1.43	1.49	0.52	0.55
CO	2.00	2.62	3.57	4.77	5.76
O_2	2.57	2.57	0.23	0.05	0.00
H_2	0.05	0.08	0.06	0.55	1.84
Exit Gas - Heating Value Gross Btu/ft^3	6.6	8.7	11.7	17.0	24.5

TABLE III

Results With High-Carbon Natural Spent Melt

Run No.	6	7	8	9
% of Stoichiometric Air	58	59	56	44
Superficial Velocity, fps	0.41	0.42	0.16	0.16
Superficial Residence Time, sec	2.44	2.38	6.25	6.25
Temperature, °F	1800	1900	1800	1800
Pressure, psia	15.3	15.2	15.0	14.8
Melt Feed Rate, lb/(hr)(ft^2)	37.2	36.9	14.1	17.6
Fluo-Solids Used	← Mullite →			
	← 65x100 M →		← 100x150 M →	
Results				
% NH_3 Decomposed	28	70	52	63
% C Burned to CO + CO_2	72	75	84	78
% Inorganic S Burned to SO_2	28	24	0	1.5
HCl Produced, Wt % Feed Melt	2.6	2.9	3.2	2.0
Dry Exit Gas, Vol %				
CO_2	13.2	12.7	9.9	9.2
CO	6.2	6.9	10.8	13.4
H_2	4.8	7.8	7.8	11.7
SO_2	0.38	0.33	0.00	0.03
N_2	72.7	69.4	68.5	63.5
O_2	0.0	0.0	0.0	0.0
HCl	2.7	2.9	3.0	2.2
Dry Exit Gas Gross Heating Value, Btu/ft^3	35	47	60	81

FLUOSOLIDS COMBUSTION UNIT

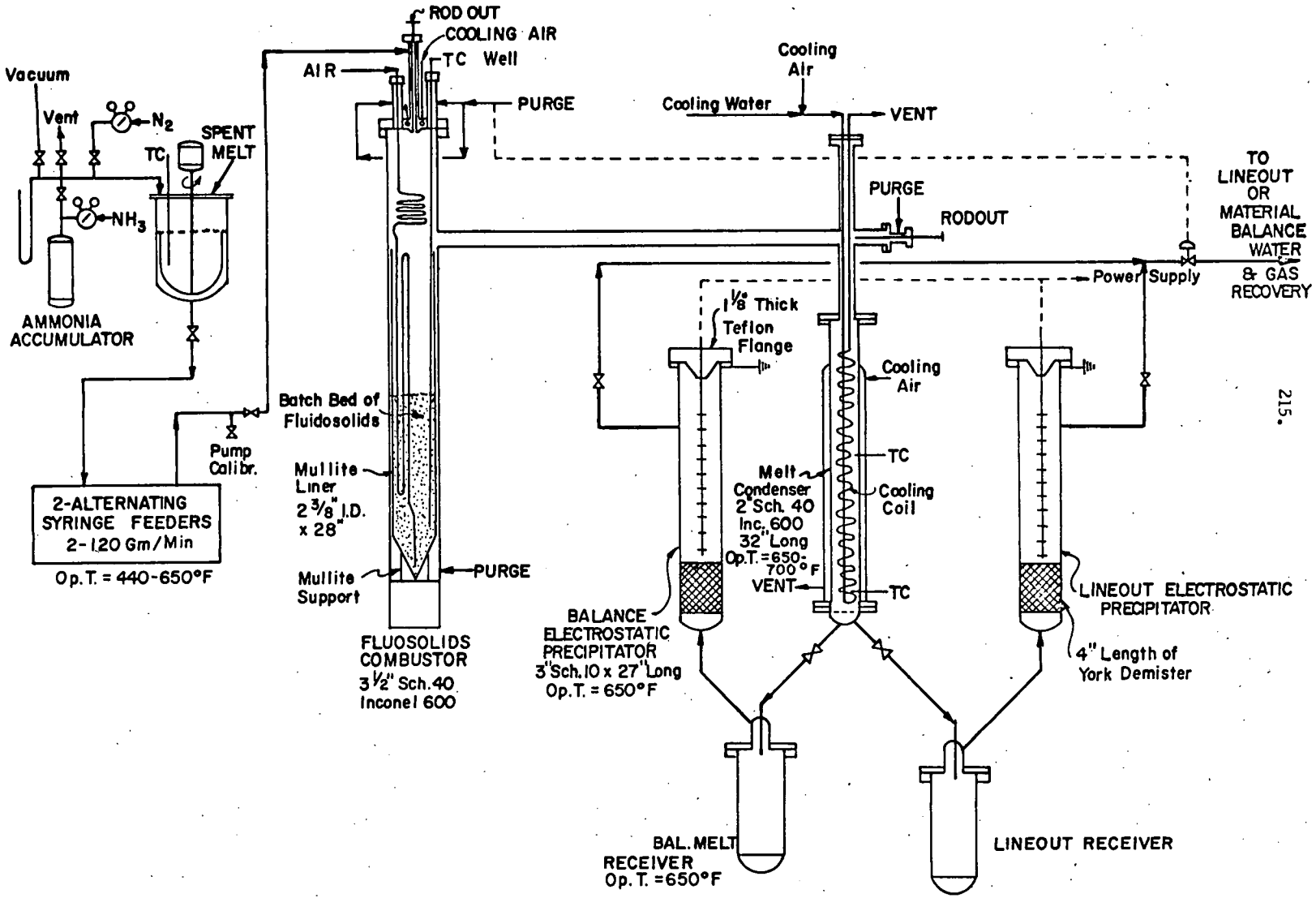


Figure 2

DISTRIBUTION OF CARBON FROM LOW-CARBON MELTS vs PERCENT OF STOICHIOMETRIC AIR

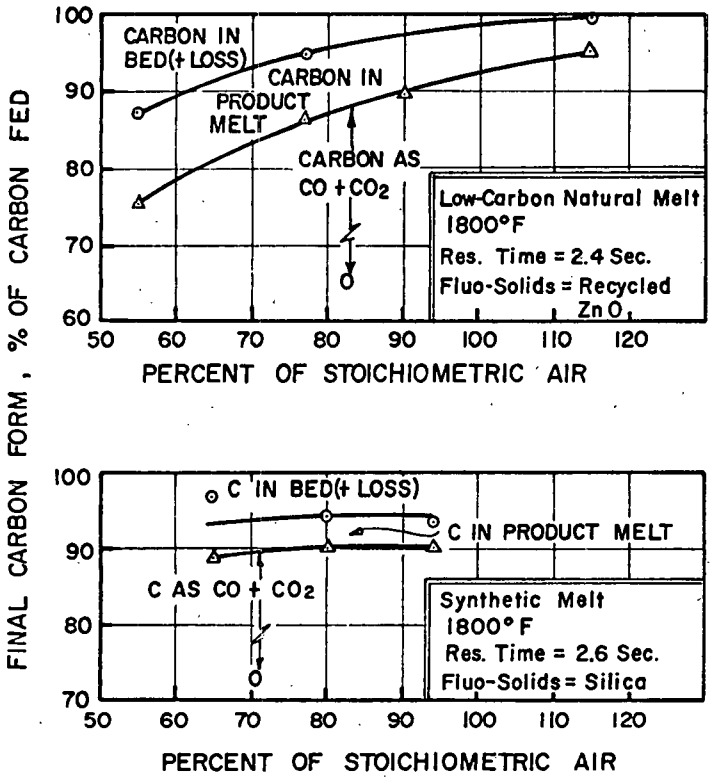


Figure 3

DISTRIBUTION OF CARBON FROM LOW-CARBON NATURAL MELTS vs RESIDENCE TIME

	Bed Height Inches	Press. Atm.	Fluidizing Velocity ft./sec.	% of Stoich. Air	Bed Solids
⊙	12	1.2	2.67	65	Mullite
⊕	12	3.1	1.05	76	
⊖	12	1.1	1.01	76	
⊡	16	1.1	1.01	73	
△	12	1.0	0.42	77	ZnO

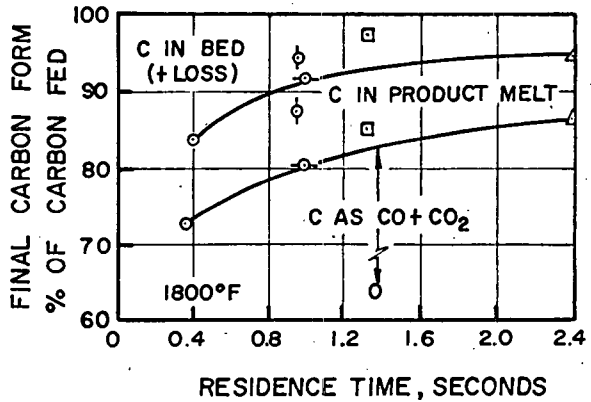


Figure 4

DISTRIBUTION OF SULFUR FROM LOW-CARBON MELTS

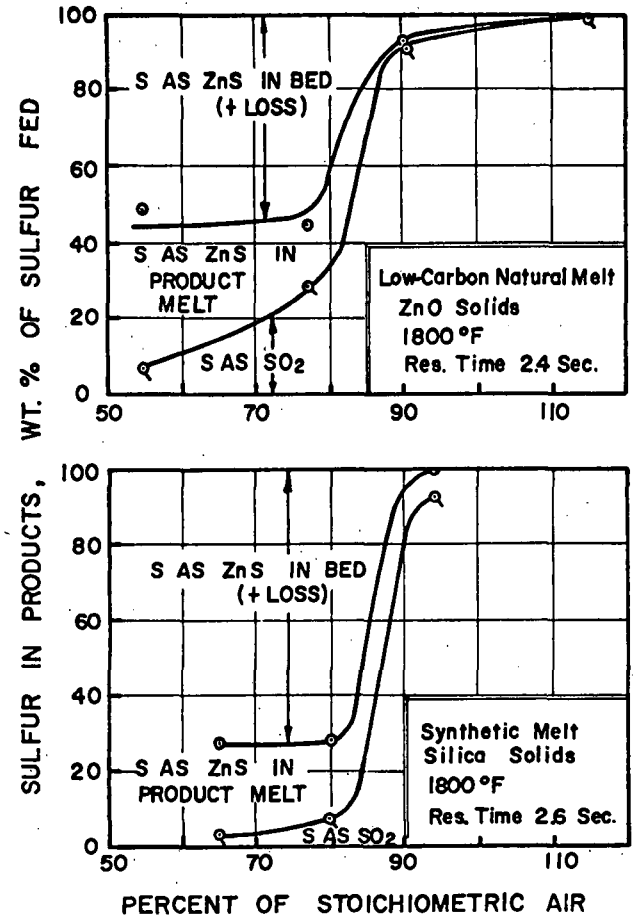


Figure 5

AMMONIA DECOMPOSITION vs PERCENT OF STOICHIOMETRIC AIR

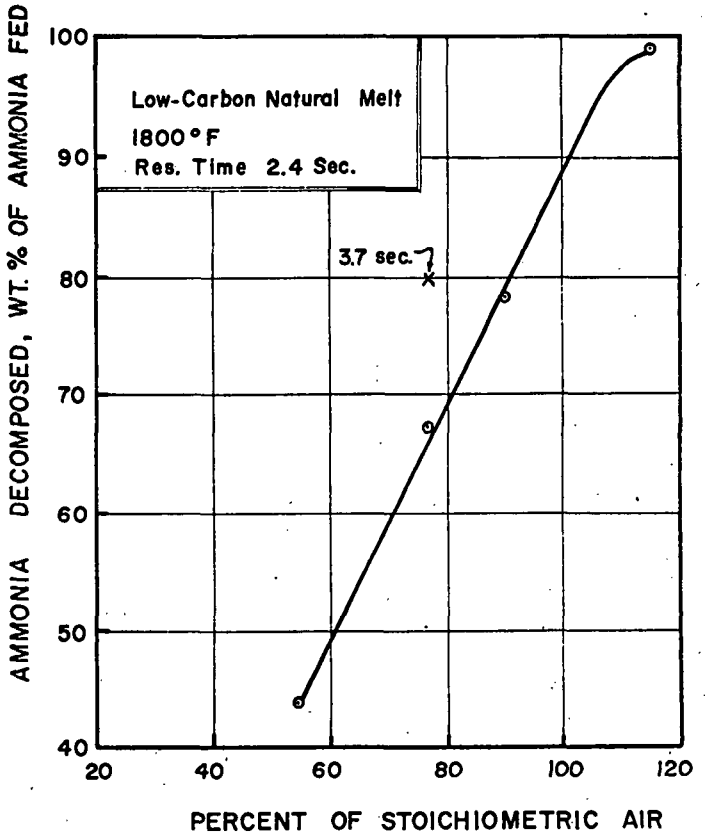


Figure 6

DISTRIBUTION OF CARBON FROM HIGH-CARBON NATURAL MELT

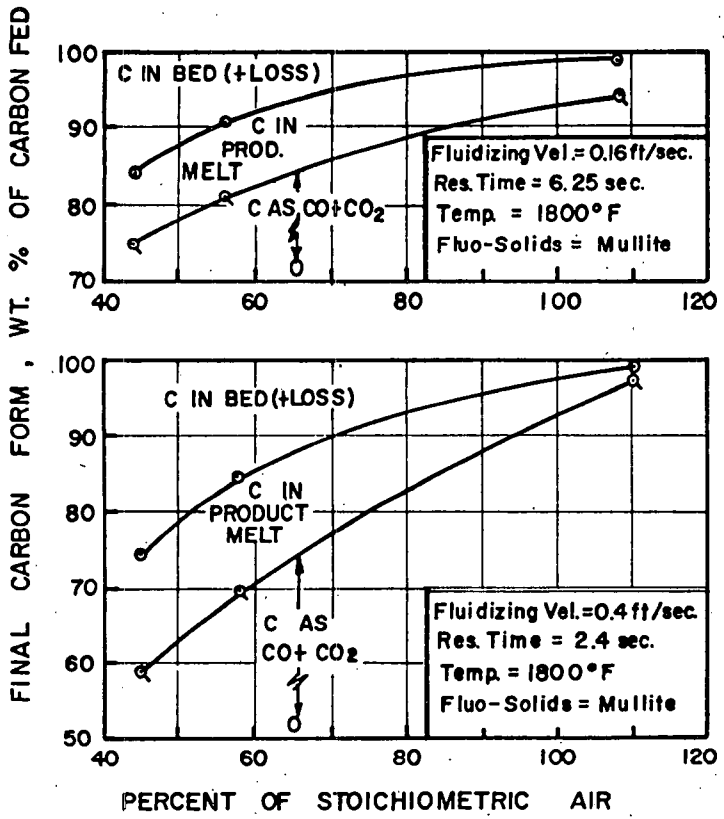


Figure 7

DISTRIBUTION OF SULFUR FROM HIGH-CARBON NATURAL MELT

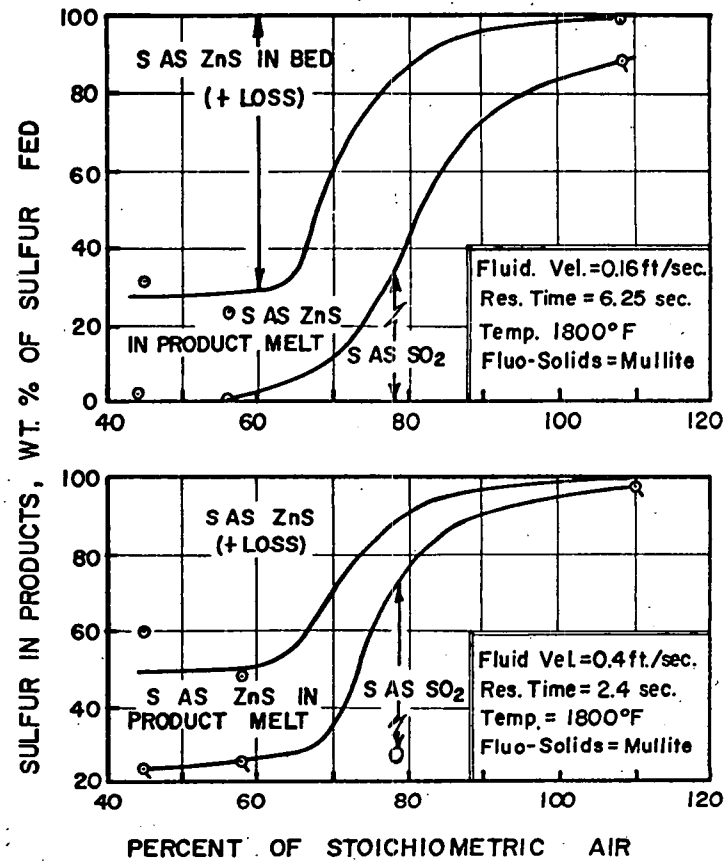


Figure 8

HEATING VALUE OF PRODUCT GAS
vs
PERCENT OF STOICHIOMETRIC AIR

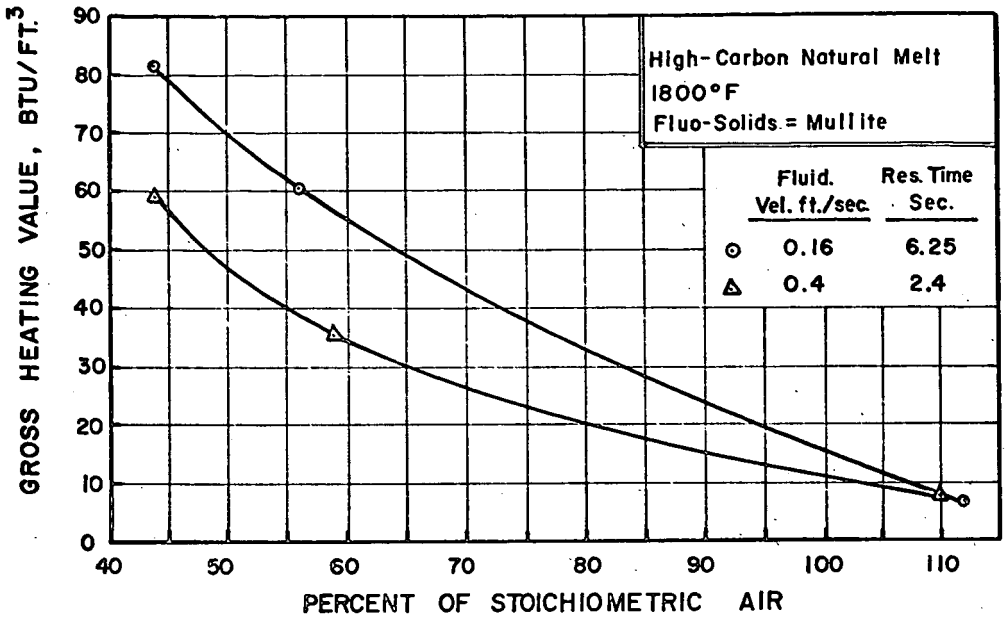
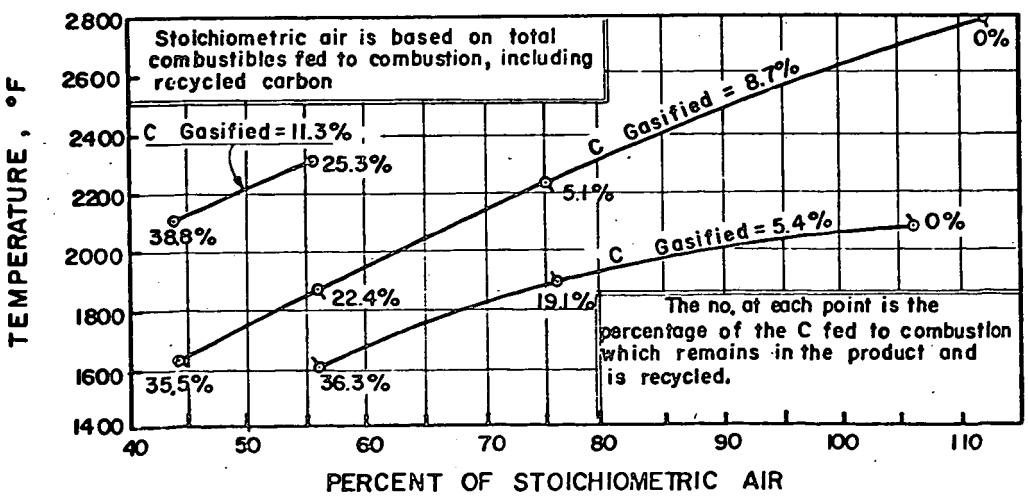


Figure 9

"ADIABATIC" COMBUSTOR TEMPERATURES FOR SEVERAL MELTS