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CHARACTERIZATION OF SOLID WASTES FROM
CIRCULATING FLUIDIZED BED COMBUSTION

E.J. Anthony, G.G. Ross, E.E. Berry,
R.T. Hemings, R.K. Kissel and C.C. Doiron

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FROM CIRCULATING FLUIDIZED BED COMBUSTION

by

E.J. Anthony¹, G.G. Ross², E.E. Berry³
R.T. Hemings⁴, R.K. Kissel⁵, C.C. Doiron⁶

ABSTRACT

The characterization of solid wastes from full-scale circulating fluidized bed combustors (CFBC) is necessary to ensure that disposal procedures or utilization strategies for the waste solids are successful. plants are extremely useful in providing hydrodynamic heat and mass transfer data, that can be used to design and predict the performance of larger units. Combustion studies indicate that data from pilot scale units can be used to approximate the behaviour of a full-scale plant for different fuels and operating conditions even when the pilot plant is not designed to properly scale the commercial unit. However, the same does not seem to be true for the determination of reduced sulphur species and geotechnical or physical properties of the solid wastes generated from pilot plants.

The results of analyses of samples generated from two units, are discussed. One is a 150 by 150 mm square, 7.3 m high pilot-scale CFBC located at the University of British Columbia and a 22 MW_e CFBC located at Chatham, New Brunswick. This unit is operated by the New Brunswick Electric Power Commission (NBEPC). Both used the same New Brunswick coal containing 7% sulphur. The data presented indicate that the pilot-scale unit can significantly over-predict the formation of sulphides and compared with the full-scale unit produces residues with much less promise for either disposal or utilization in low strength concretes. The results strongly suggest that further work is necessary to understand better the phenomena that produce sulphides and affect the geotechnical properties of wastes.

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INTRODUCTION

This paper presents results from combustion trials conducted in a 150 by 150 mm square, 7.3 m high pilot plant operated under contract for Energy, Mines and Resources Canada (EMR) by the University of British Columbia (UBC) and a 22 MW_e CFBC boiler, designed by Lurgi, being located at Chatham, New Brunswick and operated by the New Brunswick Electric Power Commission (NBEPC). Both units employed Minto coal, a high sulphur bituminous coal (7% S) and a high calcium limestone, Elmtree, both from New Brunswick.

The UBC pilot plant was not designed to simulate the Chatham unit. Rather it was designed as a general purpose well instrumented experimental facility carrying out a wide range of combustion, heat transfer and hydrodynamic tests. A pilot plant designed to simulate the Chatham unit should give residues which would be similar. However, in most cases, it is not practical to design a pilot plant to simulate a commercial unit. Often the necessary design information is either proprietary or unknown. Economic considerations are also likely to prohibit the design of a pilot plant to simulate a specific unit. In such circumstances one will be forced to use pilot plant designed for general combustion studies. The purpose of this paper is to compare the results obtained with a pilot plant designed to study the combustion of a wide range of fuels, but with the identical fuel and sorbent combinations and operating conditions as nearly equivalent to the full-scale unit as possible.

Data were obtained for the various residues generated in terms of permeability, exothermic behaviour, compressive strength and geotechnical behaviour. Residues for one set of tests were generated from the UBC pilot plant operated without the baghouse. Another trial was carried out in which nitrogen quenched samples were taken from various positions in the UBC pilot plant, in order to understand the process of sulphide formation. Baghouse samples were also obtained from this run to compare with the earlier samples obtained without a baghouse. Data were also obtained from residues generated by the 22 MW_e Chatham CFBC boiler during two different demonstration combustion runs.

THE FUEL AND SORBENTS

Minto coal was used at UBC and the Chatham 22 MW_e unit. Its composition is given in Table 1. The limestone used to determine sulphide concentration as a function of bed geometry was a mixture of two high calcium limestones, i.e. Texlime and Elmtree in a 76/24% mixture respectively by weight. For all other trials, including those with the Chatham unit, Elmtree limestone was used.

Table 1 - Composition of Minto Coal on a dry basis (wt %)

Carbon	64.8
Hydrogen	4.2
Nitrogen	0.7
Sulphur	7.2
Oxygen (by diff.)	4.1
Ash	19.0
Higher heating value (MJ/kg)	34.3

Chemical compositions and physical properties of the two limestones are given in Tables 2 and 3 respectively. The tables indicate that both composition and fluidization characteristics of the two limestones are very similar and should make no substantial difference to the results obtained from the UBC unit.

Table 2 - Chemical analysis of limestones (wt %)

	Elmtree	Texlime
CaO	54.32	54.57
MgO	0.60	0.34
Fe ₂ O ₃	0.56	0.45
Al ₂ O ₃	1.43	0.62
SO ₃	-	1.17
Na ₂ O	0.05	0.05
K ₂ O	-	0.16
BaO	0.01	0.03
SrO	0.03	0.10
MnO ₂	0.08	0.01
TiO ₂	0.11	0.00
NiO	-	0.01

Table 3 - Physical properties of limestones

	Elmtree	Texlime
Particle density (kg/m ³)	2400	2500
Mean particle diameter (m)	210	214
Voidage at minimum fluidization	0.44	0.42
Minimum fluidization velocity at room temperature (m/s)	0.041	0.044

OPERATING CONDITIONS

For the UBC pilot plant combustion tests, the bed was charged with a 150 kg of an olivine sand. Limestone was introduced as soon as the bed had been brought to thermal steady state. In the case of a high sulphur coal like Minto, with correspondingly high limestone feed rates, calculations indicate that the system ought to reach chemical steady state after 24-h.

Operating conditions were as similar to those of the Chatham plant as possible, i.e. a mean operating temperature of 850°C, and a fluidizing velocity of 7.0 m/s. Samples were taken during the latter part of a 43-h run when, operating data showed that steady state had been achieved. Table 4 gives comparative operating data for the two units.

Table 4 - Comparative operating conditions for the UBC rig and Chatham 22 MW_e units

	UBC (Pilot Rig)	Chatham (Field Tests)
MW _e	-	21.6
Fuel sulphur	7.09	6.59
Ca/S molar ratio	2.2	2.7
Bed temperature °C	880	831
O ₂ vol %	2.7	8.3 ¹
CO ₂ vol %	16.3	14.9
NO _x ppm	74	12
SO ₂ ppm	725	1884
CO ppm	75	-
HC* vol %	0.014	-
Carbon Comb. Effic.	91.5	98.8
Sulphur Capt. Effic.	87	-

1. Measured at baghouse

* Hydrocarbons

TEST PROCEDURE FOR OBTAINING SOLID SAMPLES FROM PLANT

A major objective was to characterize solids composition as a function of the pilot plant geometry in order to determine where the sulphides are principally formed. Also, since sulphides are known to react with oxygen at temperatures above 600°C (Taylor et al, 1980), it was decided to collect samples under dry nitrogen in order to prevent any possible reaction with the atmosphere during the sampling process. An

exception was the seventh sampling point, the baghouse. These samples are quenched by a heat exchanger before entering the baghouse and should therefore not be subject to any oxidation by air. Figure 1 shows a schematic of the UBC pilot plant, and indicates sampling point locations.

Sampling positions 1, 3, 4, 5, and 6 (Figure 1) were associated with a packed bed or dense fluidized regime where solids were expected to flow easily under gravity, while position 2 was expected to be associated with solids in a dilute phase. Consequently two different sampling systems were developed at UBC (Grace, 1987), to obtain the required samples (Figures 2 and 3). Solids were withdrawn and allowed to cool under nitrogen then placed in plastic containers flushed with dry nitrogen and transported for analysis. The samples were analyzed for major elements; total carbon, total sulphur and sulphide. They were also subjected to X-ray diffraction (XRD) and Thermogravimetric analysis (TGA).

TEST RESULTS

The results are given in Table 5. The carbon analyses do not differentiate between elemental carbon and carbonate forms. However, the elemental form must predominate, since very little carbonate is found in the baghouse stream. Microscopic analysis of materials showed four particle types: olivine sand, spent limestone, shale and carbon. The only identified crystalline phase containing sulphide appeared to be CaS.

Table 5 - Analysis of solids, taken under nitrogen, from the UBC unit

Position*	C	Total S wt %	CaS
1	0.83	6.15	0.86
2	1.13	6.19	0.90
3	0.71	6.41	1.08
4	1.64	6.38	0.94
5	0.64	5.49	0.85
6	9.23	8.27	4.68
7	10.0	8.82	4.14

* Figure 1 identifies sampling location

Table 5 shows that high concentrations of CaS in the solid samples occur with high concentrations of unburnt carbon. The same phenomenon was observed in previous work (Anthony et al, 1987). This suggests that the mechanism for production of sulphides is not due to the direct reaction of CaO with H₂S or other reduced species in substoichiometric portions of the combustor, e.g. prior to the introduction of the secondary air as has been suggested previously (Salib et al., 1987).

Sulphides are probably produced by the direct reduction of sulphates. Another explanation (Keairns, 1988), is that they are formed by the decay of a sulphite intermediate and their subsequent oxidation is prevented where reducing conditions occur, such as regions where high concentrations of carbon are found. Further work will be necessary to differentiate between these two mechanisms.

The higher concentrations of CaS would be cause for concern if they were typical of the technology. However, CaS concentrations from the Chatham unit are, for the most part, significantly lower; almost an order of magnitude lower compared to samples taken from position 6 and 7, with values ranging from 0.45 to 0.60%. There were some significant differences in the configuration, operating conditions and sampling procedure between the two trials. For example, the Chatham unit has an external heat exchanger, and none of its solid samples were withdrawn under a nitrogen blanket. Nevertheless, it seems unlikely that the very large differences in sulphide concentrations are due primarily to the observed differences in geometry, operating conditions or sampling procedures.

More recent results from the Chatham unit, obtained using Devco another high sulphur (5% S) bituminous coal, indicate CaS concentrations of 0.40. This tends to confirm that the UBC results are significantly higher than those from the full-scale unit.

It is interesting to compare these results (Table 5) with earlier ones (Table 6) obtained from the UBC unit using Minto coal and Elmtree limestone. The unit was operated at conditions similar to those for the work described above, i.e., overall superficial velocities 6.8 m/s, to 7.9 m/s, temperatures 840°C to 880 °C and oxygen in the flue gas about 3.5%. Table 6 shows that for positions 1 and 5, the results are fairly similar, but for position 6, the sulphide concentrations are

considerably lower. This suggests that, particularly where finer elutriated particles are concerned, care must be taken in sampling them for analysis for reduced sulphur species, unless they are already quenched to less than 200 C, as is the case with baghouse material.

Table 6 - CaS concentrations in solids obtained from UBC unit without quenching with nitrogen.

Ca/S molar ratio	1.63	2.1	2.72
Position 1 (bed drain)	0.50	0.47	0.52
Position 5 (L-valve drain)	0.43	0.61	0.38
Position 6 (2 nd cyclone drain)	0.88	0.54	0.47

Thus this study also confirmed our earlier work which showed that pilot-scale units tend to over-predict the amount of sulphides found in larger units, and that sulphide formation was associated with reducing conditions either in the bed itself or the return leg. Our previous work also showed that fuels associated with lower inherent sulphur had lower sulphide concentrations. In contrast, the work discussed here deals with a very high sulphur fuel representing extreme conditions for sulphide formation. Withdrawal under inert conditions would also tend to maximise the measured sulphide levels. Although it is possible that high levels of sulphite might be generated from a full-scale unit which was badly designed, there are no reported data suggesting that this has been a problem.

OTHER SULPHUR STATES

Wastes from circulating and bubbling FBCs can contain sulphur in reduced forms, namely sulphides, as has already been demonstrated (Anthony et al., 1987, Environment Canada, 1988). However, no other reduced sulphur forms, such as sulphite or elemental sulphur have been found with residues generated from pilot-scale circulating and bubbling FBCs (de Iribarne, 1987; 1988). These results also failed to show the presence of any other kinds of reduced sulphur species.

COMPARISON OF CHEMICAL AND GEOTECHNICAL PROPERTIES OF RESIDUES FROM THE
UBC AND CHATHAM UNITS

Prior to the commissioning of the Chatham CFBC unit it was decided to use residues generated from the UBC in order to carry out scoping trials for circulating residues. At that time there appeared to be no satisfactory characterization of such wastes, with exception of the Canadian Electrical Association study (Kissel, 1986). The rig was operated at conditions which, it was hoped, would approximate those of Chatham unit, using the same feedstocks, i.e. Minto Coal and Elmtree limestone. Table 7 gives the operating conditions employed for these runs and those of Chatham unit in a subsequent run designed to provide samples for laboratory evaluation.

Although Table 7 shows differences in the operating conditions between the UBC pilot plant and the Chatham unit, the operating conditions are generally in the same range. The two primary differences are that the sulphur capture efficiencies associated with the Chatham unit are significantly lower than those of the UBC pilot plant and the oxygen concentrations in the baghouse are significantly higher.

Following some optimization of operating procedures at the Chatham unit, recent trials with Havelock limestone as the sulphur sorbent, have indicated 90 % capture with a Ca/S molar ratio of about 2 (Razbin, 1989). The high oxygen concentrations in the baghouse seem to be due to some air dilution which occurs between the cyclone and the baghouse. It could be argued that this extra air is the reason that the CaS concentrations are lower for the Chatham unit. However previous data (Anthony et al., 1987) showed the same effect, i.e. smaller units were associated with higher sulphide concentration than larger ones, which suggests that residence times are a significant factor. In any case the impossibility of controlling such phenomena in a full-scale unit and the existence of such large differences argue against depending on pilot-scale data to predict the concentration of sulphides in a full-scale unit.

Table 7 - Operating conditions for the UBC and Chatham during sampling.

Parameter	UBC Test Run			Chatham
	1.0	2.0	3.0	
Power output MW _e	-	-	-	19.9
Fuel sulphur %	7.2	7.2	7.2	7.4
Bed temperature °C	860	840	870	841
Ca/S molar ratio	1.6	2.2	2.7	2.2
Sup. velocity m/s	7.1	6.8	7.9	-
Sulphur capture %	74.0	80.0	88.0	-
NO _x ppm vol	113.0	134.0	149.0	74.0
SO ₂ ppm vol	1576.0	1205.0	770.0	1275.0
CO ₂ % vol	18.3	18.8	18.4	13.2
O ₂ % vol	3.4	3.4	3.4	5.9 ¹
Calcium utilization %	49.0	40.0	35.0	-

1. Baghouse oxygen

Chemical Properties

The major chemical components of the UBC and Chatham residues are shown in Table 8. The composite residues from test 2, which had a Ca/S molar ratio of 2.2 should to correspond most closely to the Chatham unit residue. In fact, the residues from the Chatham unit have lower CaSO₄ and higher CaO contents, which is expected given its poorer sulphur capture. However the differences are not enormous and the concentrations of species like CaCO₃, Fe₂O₃ and silicates are similar. For test 2 even the CaS concentrations of the UBC sample are quite similar to that of the Chatham residues. This, however, is an artifact due to the way in which the composite samples were made. As discussed earlier, however, Table 8 shows that the UBC baghouse samples have considerably higher CaS concentrations than those from the Chatham unit, for the test work quoted here.

The heat released by the samples was measured by a modification of the test procedure described in ASTM C110-76. This method requires a liquid to solid ratio of 5:1, and that the temperature of the well stirred mixture be monitored (as a function of time) at least 30 minutes or until

a change of less than 0.5°C is observed between three consecutive temperature readings. The heat released during hydration was 71.3, 83.4, and 95.3 kJ/kg for samples from tests 1 to 3 respectively. These values are about one third which might be expected on the basis of the chemical and crystalline composition of the residue. They are very similar to those obtained from the baghouse sample, subsequently provided by UBC, and those obtained from the Chatham residues. All of the residues were associated with exotherms in the range of 100°C when hydrated.

Table 8 - Major chemical components of composite residues UBC and the Chatham CFBC Units

Component	Concentration (wt %)				
	Test 1#	Test 2#	Test 3#	Baghouse*	Chatham
CaSO ₄	26.1	31.6	21.7	27.7	26.1
CaS	0.8	0.6	0.7	5.2	0.45
Free CaO	24.8	28.3	24.7	15.7	23.5
CaCO ₃	3.4	3.2	5.2	6.8	4.6
Fe ₂ O ₃	10.7	9.5	9.1	10.6	15.9
Other, mainly					
SiO ₂ and C	27.3	20.8	33.8	19.5	24.2
LOI (corrected)	11.4	5.4	5.1	8.0	4.3
Sum	104.5	99.4	100.3		99.0

Composite UBC Residues

* Calculated from TGA and other analyses

Laboratory leachate tests indicated that both the UBC and Chatham residues produced a leachate with a pH of about 12, while total dissolved solids ranged from 4660 to 4770 mg/L for the leachate from the composite UBC samples and were about 4700 mg/L for the leachate from the Chatham sample. The principal constituents of the leachate from the composite UBC residues were sulphate (1700-1750 mg/L), calcium (1700-1749 mg/L) and strontium (2.2-2.7 mg/L). This compares very closely with the

analyses of the leachate from the Chatham samples which gave the principal constituents as sulphate (1700 mg/L), calcium (1700 mg/L) and strontium (3 mg/L).

Subsequent analyses of other samples generated from Chatham unit have indicated that composition, exothermicity and leachate data presented here are typical. In short, there seems to be no difficulty obtaining chemical data equivalent to those from the Chatham unit using residues generated in the UBC pilot plant. Concentrations of sulphides would appear to be the only exception, in terms of chemical data.

Geotechnical Testing

Initially it was not certain what the particle size distribution from Chatham would be. Consequently a synthetic composite waste was generated at UBC, made up of primary cyclone capture, secondary cyclone capture, and bed drain, blended in 55:45:5 proportions. It was hoped that this would produce a composite waste for characterization, which would be similar to the residues from the Chatham unit. In addition it was thought this would demonstrate what, if any effect the absence of fines would have on the geotechnical properties of such residues.

Although residues from the UBC pilot plant and the Chatham unit were shown to be quite similar with respect to chemical properties (with the exception of CaS concentrations), their geotechnical properties were significantly different. It was initially thought that the differences were due to the absence of fines in composite UBC samples first used for geotechnical testing. These samples with a mean size, D_{50} , of about 0.2 mm, were much coarser than those from the Chatham unit, which had a D_{50} of about 0.04 mm. To check, samples were taken from the UBC baghouse during the run measure sulphide concentrations (see Table 5 for the operating conditions of the UBC unit). This produced a residue with a much finer size distribution and a D_{50} of 0.04 mm, which was fortuitously the same as that of the Chatham samples.

The geotechnical properties of these new samples from the UBC unit subsequently were also shown to be quite unlike those from the Chatham unit and more closely resembled the earlier composite UBC samples. The results are discussed below, and indicate that the

geotechnical behaviour of wastes from full-scale units is to predicted with caution from pilot-scale results. They also indicate the need for further study of the phenomena which are responsible for such properties, in order to use pilot plant data with more confidence.

Table 9 gives some of the geotechnical test results. In order to determine water content/density relationships, samples were wetted and compacted using the standard Proctor procedure (ASTM D6978-78) following a two-staged water addition. Samples were also subjected to freeze/thaw cycles and their hydraulic conductivity (ASTM D2434-68) and unconfined compressive strength (AD2166-66) were determined.

The curing times for tests to determine the unconfined compressive strength and the number of freeze/thaw cycles are unfortunately not identical for the different samples (composite UBC samples, without a baghouse; the samples from the UBC rig; and the Chatham unit samples). However, it is clear that unconfined compressive strength and freeze/thaw behaviour are different for the samples, taken as a group, than those from Chatham. In fact the composite UBC samples and the UBC baghouse samples are very similar, which indicates that the differences between the UBC samples and those from Chatham are not primarily due to particle size.

Similarly, when hydraulic conductivities were determined, all the samples had values which were initially in the 1×10^{-4} cm/s range. Those of the UBC composite samples fell to 1×10^{-5} cm/s after 11 days curing, while those of the UBC baghouse samples were typically around 4×10^{-5} cm/s after a similar curing time. Residues from the Chatham unit had hydraulic conductivities around 2 to 5×10^{-6} cm/s after 7 days curing and in the case of the moist cured samples, these fell to 2.5×10^{-7} cm/s. Thus it appears that the permeabilities of the samples from the UBC rig, were as a group, quite similar but different from the residues produced by the Chatham unit. Similar observations can be made for the unconfined compressive strength and freeze/thaw data. This indicates that as far as geotechnical testing is concerned the use of samples from a pilot-scale rig, not specifically designed to simulate the full-scale boiler in question, in order to predict the behaviour of residues is fraught with considerable risks.

Table 9 - Geotechnical properties of the UBC composite and baghouse samples from UBC and Chatham Residues

	Composite UBC residues	UBC Baghouse residues	Chatham
Specific gravity	2.83 - 3.07	2.58	2.95
Mean size, D ₅₀	0.2	0.04	0.04
Optimum water content %*	14.5 - 17.5	32	26.5 - 30.5

Unconfined Compressive Strength, kPa#

Curing period, days

0	230 - 360	-	-
3	-	-	2470
7	150 - 290		4120
8	-	309	-
10	260 - 425	385	-
12	-	461	
28	-	-	4660

Freeze/thaw cycles

4 cycles	-	201	-
6 cycles	540 - 1020	sample destroyed	-
7 cycles	-		3200
15 cycles	-		880

* As determined by Standard Proctor Test.

Samples were cured at 100% relative humidity at 23 ± 2°C for periods shown.

CONCLUSIONS

Samples from a 150 by 150 mm CFBC pilot plant and a 22MW_e CFBC unit, both burning a high sulphur coal and using high calcium limestone, have been compared in terms of their chemical and geotechnical properties. The solid residues have been shown to be very similar in terms of their bulk chemical properties with the single exception of sulphide content. The pilot plant residues have been shown to have higher sulphide concentrations, particularly for solid streams from the baghouse. Sulphides have been shown to be associated with high concentrations of carbon, which suggests that they be formed by reduction of sulphates.

However, in terms of geotechnical properties residues from the two sources have been shown to be significantly different in terms of unconfined compressive strength, freeze/thaw behaviour and hydraulic conductivity. This leads to the conclusion that properties other than particle size and bulk chemical composition, perhaps particle shape, surface texture or surface chemistry, govern the geotechnical properties of the residues.

In general, the residues from the pilot plant exhibited less favorable disposal properties than those from the full-scale unit. For these reasons it is concluded that residues from pilot plants should be used with caution to predict the geotechnical behaviour of residues from full-scale.

The use of residues, from full-scale units, for chemical and geotechnical assessment is not always possible. Often residues obtained from pilot plants must be used. Ideally one ought to design the pilot plant unit to simulate the critical design and operating parameters of the commercial unit being studied. This should ensure that the results from testing residues from a pilot plant successfully duplicate those obtained from the full-scale unit in question. Where this is not practical for economic or other reasons, considerable care will have to be used in interpreting the results obtained from geotechnical testing. Nevertheless pilot plants represents the most convenient and economic method of producing residues for the study of the problems associated with waste disposal and utilization. Therefore it is necessary to better understand the phenomena which lead to the production of sulphides and those which determine the geotechnical properties of residues from CFBC units.

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Figure 1. Schematic of the UBC Pilot Scale CFBC and Solid Sampling Locations.

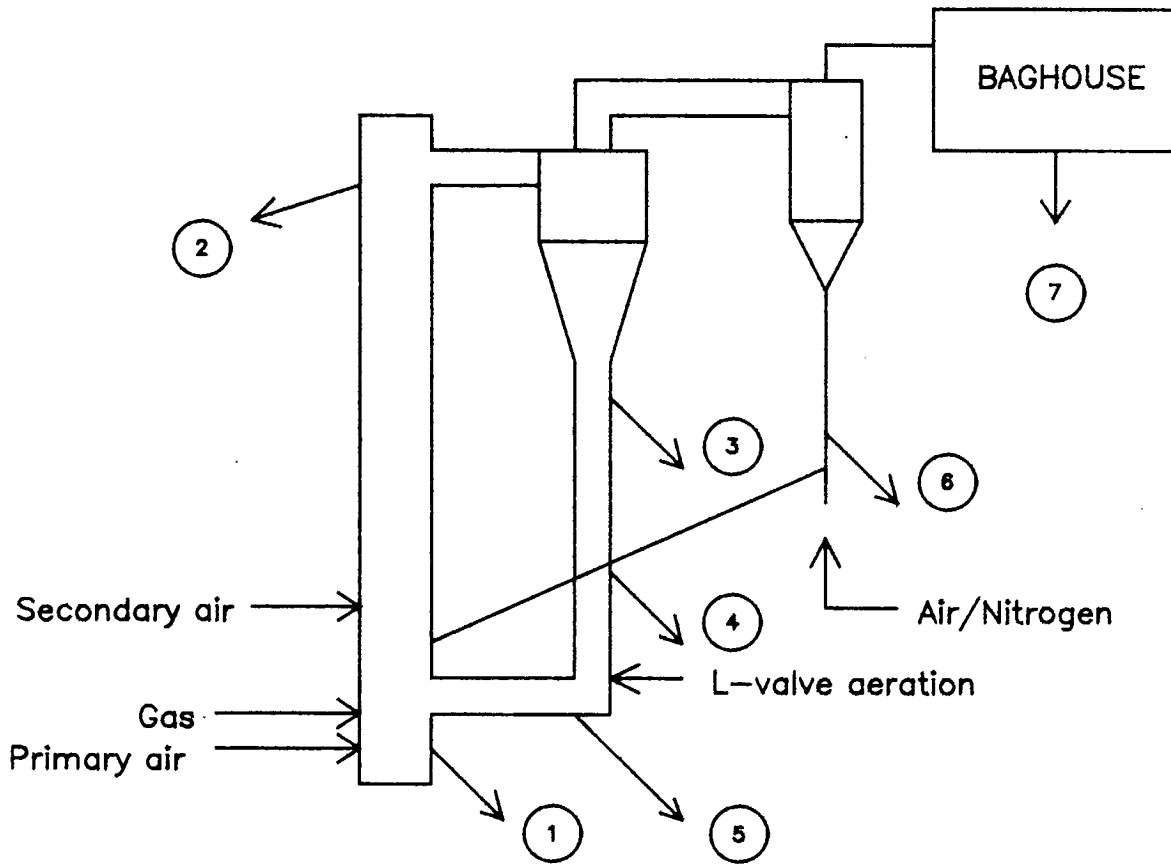
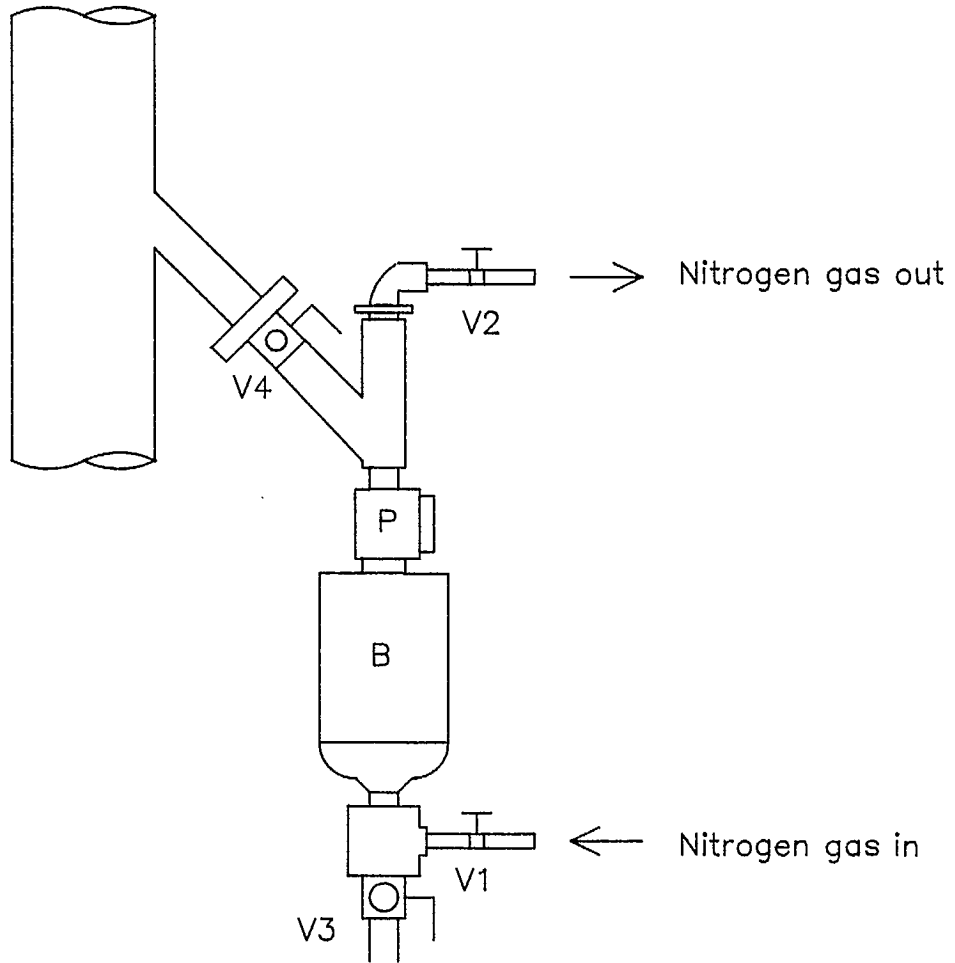


Figure 2. Solid Sampling Apparatus for Locations 1, 3, 4, 5 and 6.

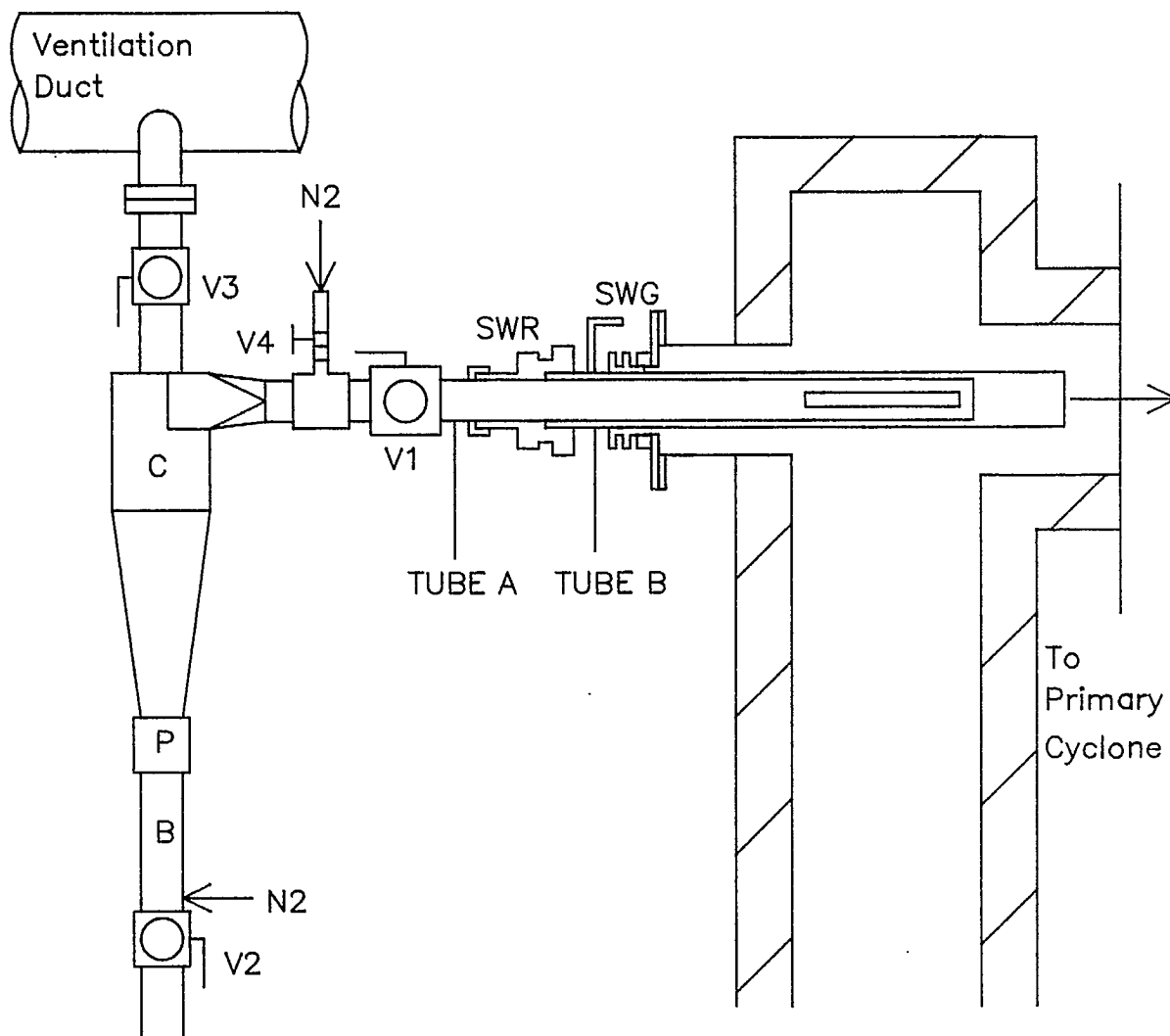


Procedure:

1. Open V3(*) and V4 to purge sampling system.
2. When hot solids come out, shut V4.
3. When system is empty, shut V3
4. Start N2 flow. Open V1 and V2.
5. Purge with N2 for two minutes.
6. Leaving N2 on, open V4 and fill bottle (B) until solids reach the viewport (P).
7. Shut V4 and leave solids to cool under N2.
8. Shut V2 and V1, place nitrogen flushed plastic bag at the outlet and open V3.
9. Seal the plastic bag.

* VX reads as "valve x".

Figure 3. Solid Sampling Apparatus for Location 2.



Procedure:

1. Insert double pipe inside reactor (as in Figure 3)
2. Open V3(*)
3. Open V4 and purge cyclone (C) and bottle (B) with N₂ for one minute.
4. Rotate tube (B) one quarter turn (to align both slot openings).
5. Open V1 slowly and fill bottle (B) until solids reach viewport (P)
6. Close V1 and leave solids to cool under N₂ (V4 open, V3 slightly open)
7. Rotate tube(B) one quarter turn and retract pipe from reactor using the last 6" long (solid bar) to seal sampling port with swagelock.
8. When solids are cool, close V3 and V4.
9. Place N₂-flushed plastic bag after V2.
10. Open V2 to collect sample.

* VX reads as "valve x".