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# A DIFFERENTIAL THERMAL PROBE FOR ANTICIPATION OF DEFLUIDIZATION OF CAKING COALS

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# A DIFFERENTIAL THERMAL PROBE FOR ANTICIPATION OF DEFLUIDIZATION OF CAKING COALS

by

I.T. Lau* and B.J.P. Whalley**

ABSTRACT

In batch fluidized beds of caking coals, differential thermocouple (DT) probes were shown to detect incipient defluidization appreciably sooner than pressure probes and with more definite response.

Operating factors affecting the temperature of incipient defluidization and the time between initial and total defluidization (the degree of anticipation), are discussed. The resulting relationships can be qualitatively explained by fluidization and heat transfer theories.

Fluidized beds incorporating DT probes would appear to have application to other materials and to characterization and control of fluidized bed operation.

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# INTRODUCTION

It is well known that aggregative (gas-solid) fluidization is characterized by such features as high heat transfer rates and homogeneous bed temperatures [1,2]. These features are usually very important in industrial gas/solid reactors. However conditions that lead to stickiness of the surface of fluidized particles, if uncontrolled, will cause particles to stick together and to the walls and internals of the reactor and eventually lead to defluidization. This is usually the result of the formation of a semi-rigid structure channelled by escaping fluidizing gases. Loss of fluidization results in a marked drop in heat transfer rates and large temperature gradients and attendant dangers including bed solidification.

Defluidization problems were encountered in the early development of fluidized bed systems including hydrogen reduction of iron ores [3-5]. Many modern technologies that have been developed to process coal use fluidized bed techniques [6,7]. But agglomeration of caking coals, that is, coals that become plastic upon heating (generally between 350-450°C) [8]) can be especially troublesome during fluidized bed carbonization or gasification [9]. In coal gasification processes, defluidization can occur with nominally non-caking coals, especially when they are subjected to substantial concentrations of hydrogen under pressure at elevated temperatures [10].

Many different techniques are used in attempting to prevent or control defluidization. These include increasing the fluidizing gas velocity, adding larger particles or rheologically inert materials, lowering the bed temperature or a combination of such remedies [11-14]. Probably depending upon the heating system used and the type of bed material involved, defluidization can occur relatively suddently, as shown by Gluckman et al [15] for heating of copper shot, glass beads, and other plastic materials, or over several minutes as reported by others for iron ore reduction [5]. Instruments used for investigating these systems were pressure and force transducers and were intended to indicate defluidization but were not designed to anticipate it.

The objective of this investigation was to develop instruments to anticipate defluidization and to determine the operating conditions that favoured the extent of anticipation.

Because heat transfer in fluidized beds is dependent upon the intensity of particle circulation, any property of the system that interferes with it (e.g. insufficient gas velocity) will cause temperature differences in the bed. These may be quite small (e.g. 0.1°C) but the use of differential temperature (DT) thermocouple pairs is an effective method of detecting them and of impaired particle circulation.

In adapting this technique to a small fluidized bed of caking coal subjected to a rising temperature regime, Whalley and Dykstra [16] observed that a small but increasing temperature difference evidently anticipated the total defluidization that occurred several minutes later and investigated the possibilities of this observation.

## SIGNIFICANCE OF DIFFERENTIAL TEMPERATURE SIGNAL

Even though the heat transfer mechanism in a fluidized bed is not completely understood, the concept that the moving solid particles play a dominant part in the mechanism is generally accepted and much can be explained by different heat transfer models [17-20]. In general in a wall-heated fluidized bed reactor, the mechanism is described as involving frequently repeated acts of transient heat transfer between the heated wall and the solid particles, which transfer heat to the bulk of the fluidized bed. An unsteady state temperature gradient would be expected in the vicinity of the heated wall as shown in Fig. 1.

The solid curve in this figure represents the bed temperature dropping from its value ($T_W$) at the wall to the bulk bed value ($T_B$) at the thermal boundary surface A of an extensively fluidized bed. If the bed materials become sticky, the kinetic energy of the particles will be opposed by strong particle cohesive and adhesive forces and bed circulation will slow down. The extended particles residence time in the transient heat transfer zone will tend to raise the temperature profile as represented by the dotted line in Fig. 1 with its thermal boundary moving towards the bed centre (surface B). As illustrated by Fig. 1, the temperature at surface A increases by an amount $\Delta T$ over the bulk bed value.

If a pair of differential thermocouples is set up in the direction of heat transfer with one of the thermocouple tips located in the main body of the bed and the other located as close as possible to the heat transfer boundary surface A (Fig. 1) an essentially steady baseline signal indicating zero temperature difference should result as long as the bed is fluidizing. As the bed is heated up and the bed materials softening point is approached, an appreciable temperature difference between the two thermocouples would be expected as the temperature at surface A (Fig. 1) will rise to a value significantly above the main body of the bed and be detected as a DT signal of differing from that of the baseline.

At the same time in the bulk of the bed, particles may still be fully fluidized. However, a point will finally be reached when the particle cohesive forces are so strong that channelling will occur and the bed will become defluidized. The time difference between defluidization, ($t_D$) and a significant temperature difference indicated by the differential thermocouple pair ($t_s$) is called the anticipation time ($\Delta t_A$). It can be shown that factors which affect heat transfer within the bed, and the onset of defluidization will also have an effect on the anticipation time.

## EXPERIMENTAL

### 1. Materials

For the bed, a sample of Lingan-Devco mixed (high volatile) coal was used of -250μm size unless otherwise stated. Table 1 gives the size distribution and plasticities of the coal as determined by a sonic sifter and a Gieseler plastometer respectively.

To provide similar coal samples but of different plasticities, samples of another coal, Chisholm, were oxidized for different times and at different temperatures in a fluidized bed reactor using fluidizing gas of different concentrations of oxygen in nitrogen. Table 2 shows the oxidation conditions together with the corresponding dilatations and plasticities of the coal samples.

### 2. Apparatus and procedure

The fluidized bed reactor used for these experiments was a 10-cm diam electrically wall-heated unit of conventional design fitted with a fritted stainless steel distributor (avg pore size 4 μm) A schematic representation is shown in Fig. 2.

Thermocouple entries were made through glands set in the reactor wall at different levels. One thermocouple inserted in the approximate centre of the bed was used to measure the bulk bed temperature. The many entries provided a variety of possible DT pair configurations.

A pressure probe was used to continuously measure the pressure drop of the whole bed and another to measure that of a top fraction of the bed; it was inserted as shown in Fig. 2. The probes were attached to pressure transducers (model 504-24, Viatran Corp., Grand Island, N.Y., U.S.A.) which were connected to a 2-channel recorder.

The fluidizing gas was technical grade nitrogen containing less than 3 ppm $O_2$. Its flow rate was controlled by a precision needle valve and measured by a Rotameter. The reactor wall was heated by external resistance heaters wrapped around the reactor wall. The wall temperature was controlled by a linear temperature programmer (model TP-2000 Theall Engineering Co., Oxford, Pa., U.S.A.). The wind box and freeboard part of the vessel were also electrically heated.

The fluid bed reactor was first loaded with a weighed amount of caking coal, then fluidized and heated at the designated rate of increasing temperature. The gas flow was adjusted manually to maintain an approximately constant superficial velocity as the temperature was raised. The bed temperature, DT signal, the fractional and total pressures were continuously recorded until the bed became obviously defluidized.

3. Location of DT thermocouples

According to our development concept and early experiments, the DT thermocouples in a wall-heated reactor should be separated radially with one inserted more closely to the heated wall than the other. The near-wall thermocouple's position is most important as it determines the DT signal performance. If this thermocouple was inserted too far from the wall, a less sensitive (or even no) signal would occur when particles started to agglomerate. On the other hand, if it were located too close to the wall, the DT trace drifted away from its zero base line even with an extensively fluidized bed. The best near wall thermocouple location was found in our reactor to be about 8 mm away from the vessel wall. At this position the DT trace of a fluidized bed stayed on the base line as the bed temperature was raised to the softening point of the coal particles.

## RESULTS AND DISCUSSION

### 1. DT Signals

A typical trace by a DT thermocouple pair in this type of configuration is shown in Fig. 3. The oscillations of the DT trace can be explained by the contacting of relatively hot particles from the boundary layer with the near wall thermocouple; the particles being propelled intermittently by rising bubbles of fluidizing gas. At a certain temperature level, depending upon the plastic property of the coal and operating conditions, the DT trace curved away in a prominent manner from its zero base line. On occasion the DT trace would momentarily return to the zero line before a persistent signal occurred. This type of behaviour was probably the result of a bubble sweeping out the agglomerating particles around the near-wall thermocouple and replacing them with particles from the main body of the bed. The bed temperature curve also recorded on the chart (Fig. 3) shows that the occurrence of the DT signal was within the coal plastic temperature range (350-450°C).

The heating rate of the bulk of the bed typically levelled off at about the same time as the DT signal. This can be explained by a slowed circulation of the sticky particles, the ultimate formation of a layer of coal on the heated wall and the resulting loss of heat transfer to the bulk of the bed.

### 2. Pressure drop signals and anticipation time

Measurement of pressure drops through the total depth of a fluidized bed ($\Delta P_t$) and through the top fraction of a bed ($\Delta P_f$) are also given in Fig. 3. As would be expected from channelling, both pressure drop curves fell off simultaneously when a bed defluidized because of its stickiness. The defluidization signal in general appeared later than the DT signal and the time difference depended on operating conditions and bed material properties.

It is of interest that the two pressure measurements differed in one aspect. A significant increase in the pressure drop across the top portion of the bed occurred before defluidization. This evidently represented the amount of bed expansion that resulted from the release of volatile matter change in physical properties from the coal. A corresponding change in $\Delta P_t$ was not seen because $\Delta P_t$ is practically equivalent to the head of the bed and relatively independant of changes in the fluidized bed height caused by bed expansion for a given charge. However, expansion of the bed results in a increase in head above the upper probe and a corresponding $\Delta P_f$ increase.

The significance of anticipation time ($\Delta t_A$) was demonstrated by a few experiments to investigate the possibility of returning the bed from a sluggish to a fully fluidized state. In these, gas flow rates were increased after the DT signal had appeared. It was found that DT traces could be returned to their original base line as long as the pressure drop signals had not fallen off significantly. Defluidization could be prevented provided that the gas flow rate had been increased sufficiently and the heat supply terminated. However, if the pressure drop signals had fallen off, the bed could not be refluidized no matter how much the gas flow was increased. This meant that there was time ($\Delta t_A$), between the DT signal and the occurrence of defluidization, in which to change reaction conditions to prevent defluidization. In this intermediate condition it is likely that impaired mixing of the bed caused by stickiness of the particles was involved rather than actual bonding of the coal particles in a semi-rigid structure. Response to changes in reaction conditions such as lowering the temperature, adding inert coal materials or coarser coal, increasing the gas velocity is consistent with the concept of impaired mixing of the bed.

### 3. Effects of operating variables

A study of operating variables upon the DT signal temperature and the extent of defluidization made at this stage were related to a wall-heated batch fluidized bed system, but they should also provide information for a proposed study using a continuously fed fluidized bed reactor. Because of the complexity of fluidization systems, especially because the solids flow patterns depend on so many factors, our experimental results are limited to qualitative analysis of the effects.

(a) Effect of superficial gas velocity

In a fluidized bed of potentially sticky material, strong bubble action and fast solid particle motion from high gas velocities would be expected to overcome surface adhesive and cohesive forces, that cause agglomeration. Therefore at higher gas flows a higher defluidization temperature should result. Results indicate in Fig. 4A that the defluidization temperature increased linearly with increasing gas flow rate which is in agreement with the finding of Gluckman et al [15] for other materials.

Fig. 4A also shows that the DT signal temperature, $T_s$, increased with increasing gas velocity. This result would also be expected since the formation of a hindered surface-bed heat transfer condition would tend to occur at higher temperatures as the result of higher kinetic energy in the bed and since a relatively short residence time of bed material in the boundary layer would work against a temperature difference between the near-wall thermocouple and that of the bulk of the bed.

A longer anticipation time also resulted from a higher gas velocity as shown in Fig. 4B. This may infer that the effect of bubble action was less at the reactor wall than in the main part of the bed. It is also interesting to note that as the gas flow increased the anticipation time fell off sharply as the gas velocity reached a value of about 7 cm/sec. (about 3 times minimum fluidization velocity). It should be pointed out that the actual gas flow through the bed were expected higher due to the coal devolatilization.

A slugging regime, was possibly approached at this point. The difference in fluidization behaviour between the two regimes (bubbling and slugging) may have been responsible for the abrupt change in anticipation time. Or that the highly expanded bed expansion accompanied by phase inversion caused a breakdown of the general surface-bed heat transfer mechanism.

(b) Effect of heating rate

Figure 5A indicates as might have been expected that the heating rate in the range studied had no effect on the defluidization temperature since particle agglomeration depends only on their stickiness and on the external forces acting on them.

In contrast the DT signal temperature decreased with increasing heating rate as shown in the same figure. This might be explained by a greater temperature gradient at the wall with increasing heating rate contributing to reduction of particle mobility in the boundary layer and to a lower DT signal temperature. As can be seen from Fig. 1, a higher temperature profile at the wall would raise the temperature at the wall thermocouple more readily.

It can be shown that because the defluidization temperature was independant of the heating rate, a corresponding decrease in DT signal temperature by a higher heating rate represented an increase in anticipation time. In Fig. 5B this effect is shown as an appreciable time anticipation at heating rates in excess of 7.5°C/min. At lower heating rates, even hindered solid circulation could provide a sufficient rate of heat transfer to prevent a significant temperature gradient in the surface boundary layer and preclude a DT signal.

(c) Effect of bed height

The effect of bed height upon the DT signal temperature (Fig 6A) can be explained if a net solid downward flow along the reactor wall is assumed to occur consistent with a common observation of other fluidized bed systems [21,22]. Because a deeper bed would make a longer path, particles in the boundary layer would acquire more heat before coming in contact with the wall thermocouple. This would result in an earlier DT signal (i.e. lower $T_s$).

The defluidization temperature was found to drop only slightly with increasing bed height as shown in Fig 6A. A similar effect of bed depth was found by others [15] using glass spheres. Changes in pressure in the bed with varying bed depth were small in these experiments and do not seem to account for this effect. Possibly the coalition of bubbles in a deep fluidized bed could create channels more easily and deprive some parts of the bed of bubble action to break down agglomerates.

Because of the earlier DT signal a longer anticipation time resulted from a deeper bed. This effect is shown in Fig 6B. The bed heights given in this figure are those measured at fixed bed conditions. From the results of present studies, only beds with L/D value larger than 1 could provide significant anticipation time in our fluidized bed system. However, as solid circulation depends on many factors such as vessel geometry, and the performances of the gas distributor it is possible that different results would be obtained on fluidized beds of different solids flow pattern.

(d) Effect of coal plasticity

In these experiments samples of the high volatile coal were oxidized to different levels and their dilatations and plasticities determined. The results are shown in Table 2. As predicted, reduction in stickiness of the bed material by oxidation raised both defluidization and DT signal temperatures as given on Fig 7A. The effect of plasticity was consistent with the results of experiments in which inerts were added in varying amounts to a caking coal [23]; the effect was qualitatively similar to reducing the plasticity by oxidization.

Despite nearly identical plasticity effects on $T_D$ and $T_S$, the effect of decreasing plasticity upon $\Delta t_A$ was to reduce $\Delta t_A$ to a low limiting value at zero plasticity or dilatation. The apparently long anticipation time obtained from high plasticity coal samples is considered to be caused by formation of a heat transfer resistance layer on the wall. That is, reduction in heat transfer resulted in a delay in defluidization. It is interesting that for coals with plastic properties nearly completely destroyed by oxidization, clear DT signals were still obtainable in the fluidized bed even though there were difficulties in obtaining plasticities by the Gieseler plastometer. The DT signal method and apparatus may correlate well enough with coal plastic properties to be potentially useful in supplementing existing equipment and techniques in the determination of rheological properties of caking coal and other plastic materials.

(e) Effect of particle size

As deduced from preliminary experiments [23], the determination of the effect of particle size can be erroneous if the coal is significantly oxidized. The error can occur if there is a preferential accumulation of oxidized particles in the finer sizes in which case they would act as rheological inerts. This would tend to raise the $T_S$ and $T_D$ and decrease the $\Delta t_A$ according to the plasticity effect shown in the previous section.

Therefore, efforts were made to prepare samples of different size distribution but approximately the same plasticity by mixing coal samples of different plasticities (different levels of oxidation) and different sizes. Rheologically active fine particles are known to be prone to defluidization. This can be explained by their low inertia and relatively large specific surface area. Such fines will easily adhere to the heated wall and to each other. The effect of decreasing particle size upon the defluidization temperature as shown in Fig 8A, was expected and was in agreement with other studies with other materials [15]. The same reason can be used to explain the similar effect on DT signal temperature also shown in Fig 8A that is, that a sharper decrease in $T_S$ resulted from a reduction in particle size. In addition, coarse particles have a less effective conductivity in the heat transfer boundary layer and consequently delay the near-wall thermocouple reaching a temperature above the bulk of the bed.

Fig. 8A shows the difference in effect of particle size on $T_S$ and $T_D$ and Fig. 8B shows the corresponding anticipation time ($\Delta t_A$). The sharp rise in $\Delta t_A$ for the very small particle sizes was evidently caused by the greater tendency of these fines to adhere to the heated wall and thus to resist heat transfer to the bed.

It can be shown that many of the effects described above are also found in continuously fed wall heated beds.

## CONCLUSION

In a batch fluidized bed of caking coal, wall-heated at a rising temperature, a suitably located DT probe can anticipate defluidization considerably in advance of the commonly measured pressure-drop change. Such a warning should permit remedial measures to prevent defluidization.

Within the experimental range of conditions chosen, the defluidization anticipation time interval will increase with increasing fluidizing gas velocity, increasing heating rate, increasing bed depth, increasing bed materials' plasticity, and decreasing particle size. At a very low heating rate the DT probe may fail to give an advanced signal anticipating defluidization. At a high heating rate using fine sized highly caking coals, total defluidization of the bed may not occur because of coating of the heat transfer surface by agglomerating material.

The results obtained can be explained qualitatively by general fluidization and heat transfer theories. It is evident from our development concept that the use of the DT probe with thermocouple tips arranged in the direction of heat transfer is not restricted to wall-heated batch units but should have much more general applicability.

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TABLE 1. Plasticities and Size Distribution in wt % of Coals

| Coal Sample | Gieseler Plasticity | | Size Range (Mesh U.S.) | | | | | | $dp^{(1)}$ |
|---|---|---|---|---|---|---|---|---|---|
| | Start ($^oC$) | Max Fluidity (ddm) | +60 | 60-100 | 100-140 | 140-200 | 200-325 | -325 | (μm) |
| *L-D 1 | 402 | 715 | 1.4 | 41.0 | 15.4 | 11.8 | 12.2 | 18.2 | 90.7 |
| L-D 2 | 398 | 370 | 0.3 | 2.0 | 22.1 | 41.3 | 23.7 | 10.7 | 76.1 |
| L-D 3 | 402 | 180 | 0.9 | 45.4 | 15.8 | 11.9 | 11.6 | 14.5 | 96.9 |
| L-D 4 | 394 | 710 | 2.0 | 77.7 | 19.0 | 1.2 | 0.1 | 0.1 | 172.8 |
| L-D 5 | 405 | 29 | 0.1 | 3.0 | 5.1 | 8.7 | 59.5 | 23.7 | 57.6 |
| L-D 6 | 403 | 180 | 0.4 | 12.2 | 7.6 | 9.5 | 48.1 | 4.4 | 65.6 |
| L-D 7 | 402 | 180 | 0.2 | 2.6 | 12.6 | 23.1 | 43.7 | 17.9 | 65.8 |
| **C | 395 | 4833 | 6.2 | 45.5 | 16.9 | 10.7 | 10.5 | 10.3 | 107.2 |

TABLE 2. Oxidized Chisholm Coal Properties

| Chisholm Coal sample | $O_2$ Conc. x $10^{-3}$ (ppm) | Oxidation Temp. ($^oC$) | Oxidation Time (Min) | Dilatation (%) | Max Fluidity (ddm) |
|---|---|---|---|---|---|
| C-0 | - | - | - | 104 | 4833 |
| C-1 | 6.6 | 300 | 60 | 11 | 551 |
| C-2 | 6.6 | 300 | 30 | 42 | 1507 |
| C-3 | 6.6 | 300 | 90 | - | - |
| C-4 | 6.6 | 300 | 180 | - | - |
| C-5 | 6.6 | 300 | 15 | 78 | 2464 |
| C-6 | 24.7 | 340 | 60 | 8 | 110 |
| C-7 | 24.7 | 340 | 180 | - | - |
| C-8 | 24.7 | 340 | 90 | - | - |
| C-9 | 24.7 | 340 | 20 | 40 | 841 |
| C-10 | 6.6 | 300 | 45 | 53 | 1377 |

*L-D: Lingan-Devco Mixed Coal
**C: Chisholm Coal
(1): Reciprocal mean particle diameter

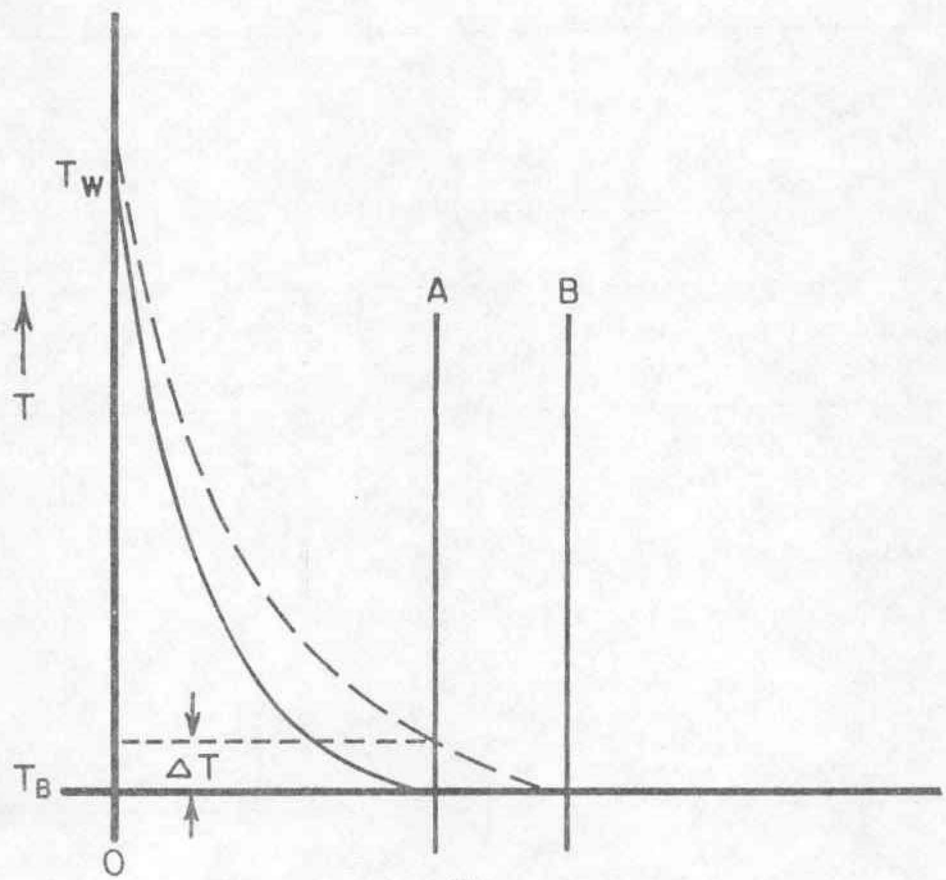


Figure 1. TEMPERATURE PROFILE CHANGE CAUSED BY STICKY BED MATERIAL.

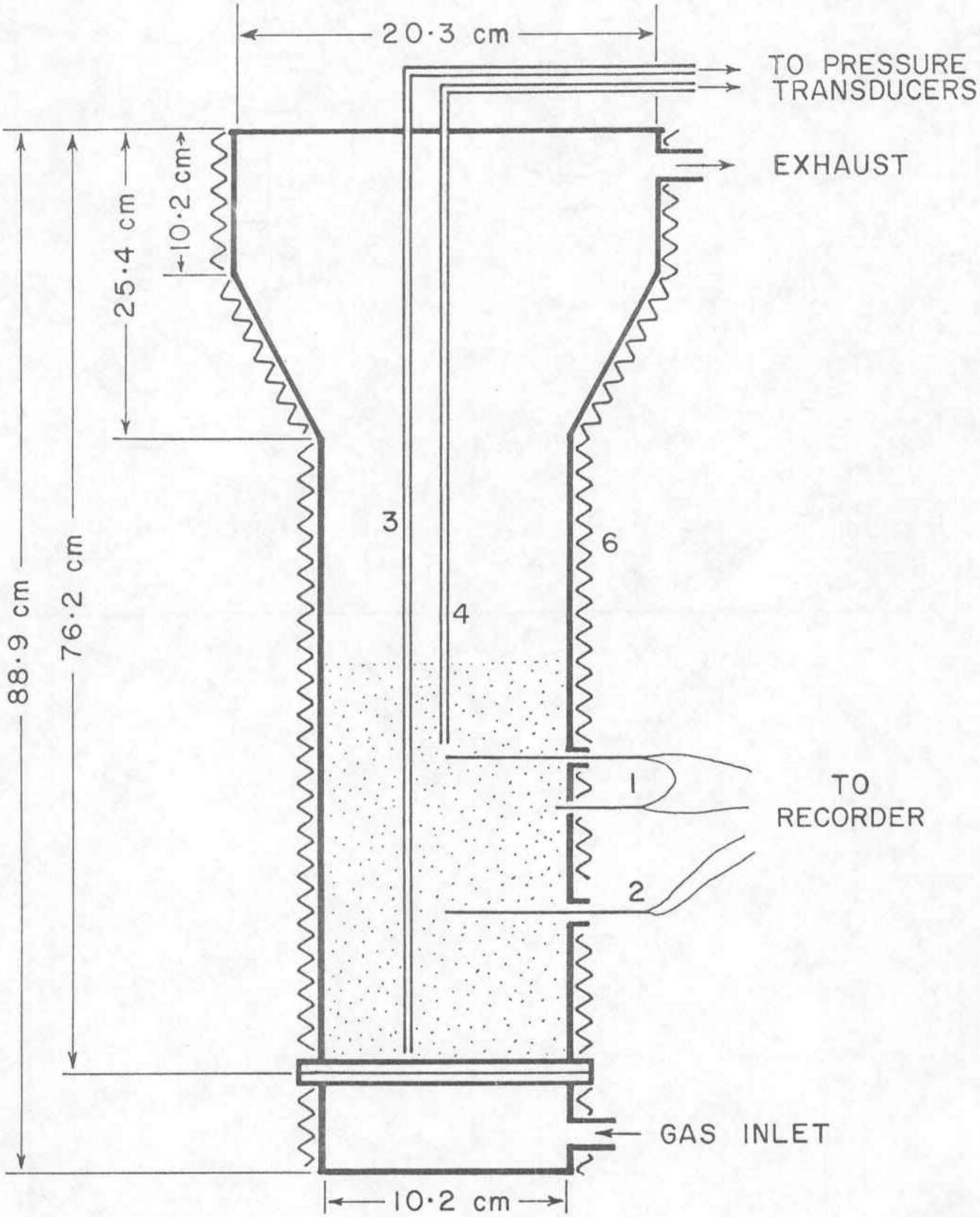


FIGURE 2. SCHEMATIC OF FLUIDIZED BED COLUMN

1. DIFFERENTIAL THERMOCOUPLE PROBE
2. BED TEMPERATURE INDICATING THERMOCOUPLE
3. TOTAL BED PRESSURE PROBE
4. FRACTIONAL BED PRESSURE PROBE
5. GAS DISTRIBUTOR
6. HEATERS

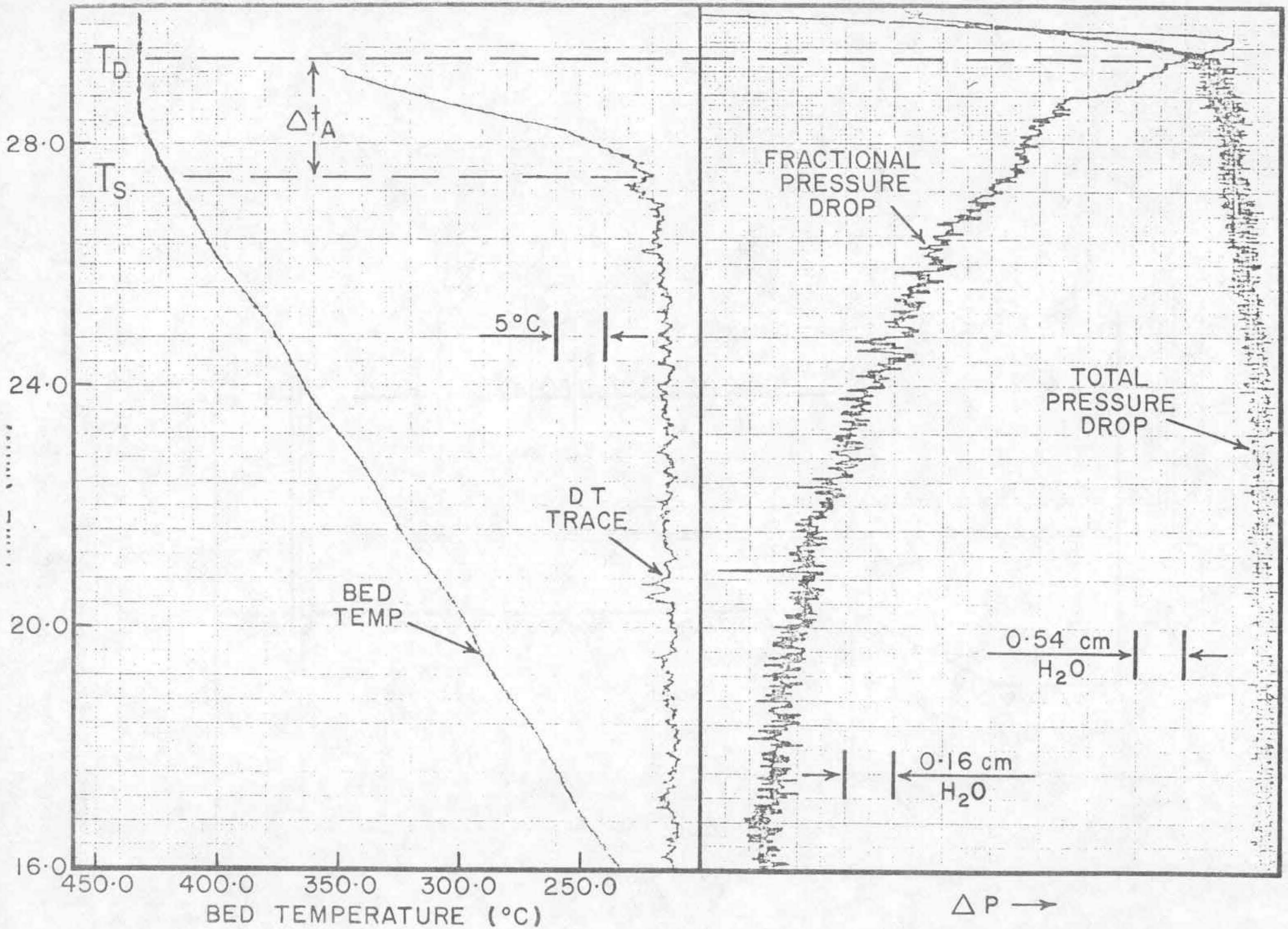


FIGURE 3. TYPICAL DIFFERENTIAL TEMPERATURE AND PRESSURE DROP TRACES

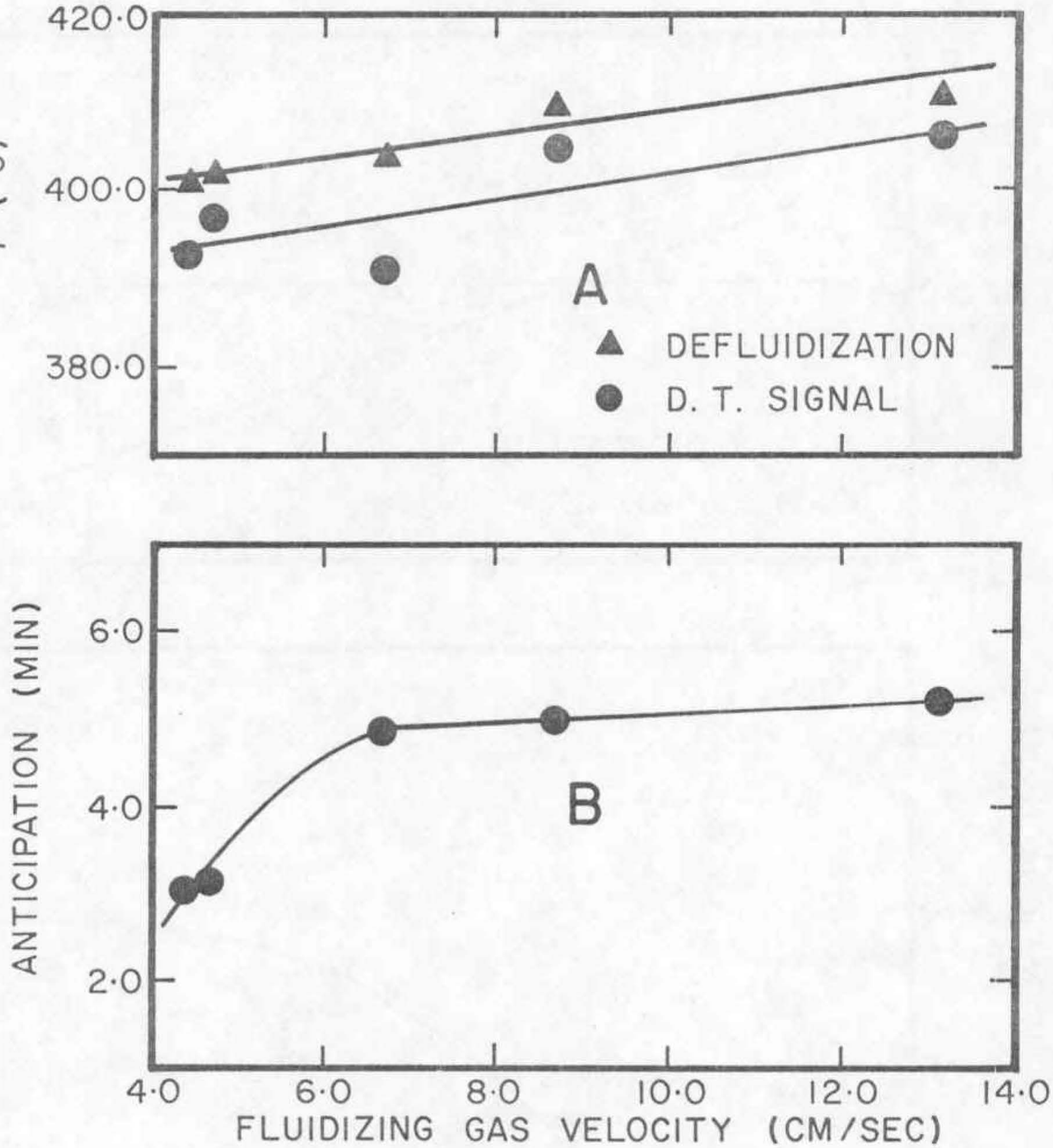


FIGURE 4. EFFECT OF FLUIDIZING GAS VELOCITY.

AVG PARTICLE SIZE: 90.7 μM
HEATING RATE: $10^{0}$C/MIN

FIXED BED HGT: 15 CM
MAX FLUIDITY: 715 DDM

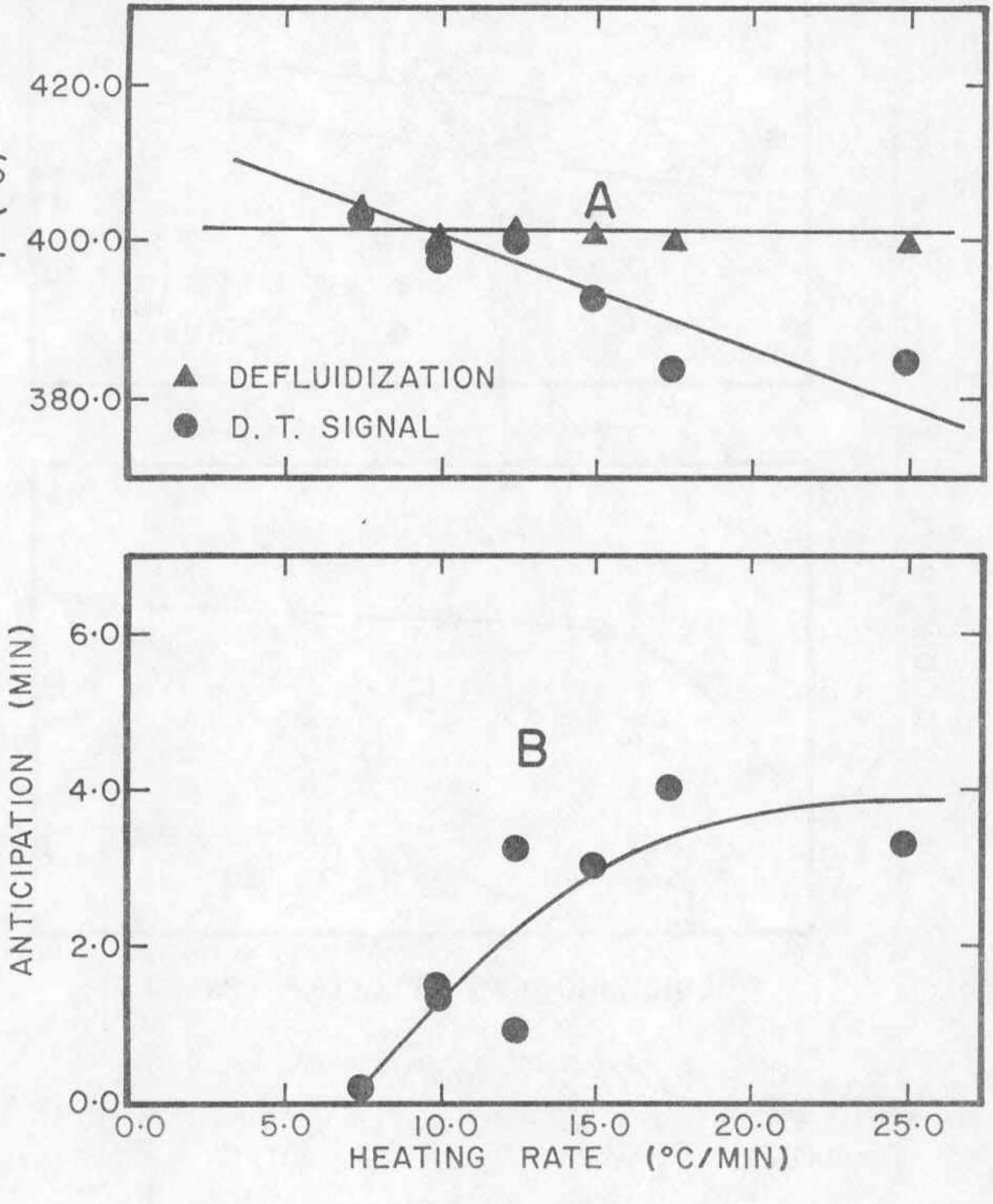


FIGURE 5. EFFECT OF HEATING RATE.

AVG PARTICLE SIZE: 90.7 μM  FIXED BED HGT: 15 CM
MAX FLUIDITY: 715 DDM  FLUIDIZING GAS VELOCITY: 4.4 CM/SEC

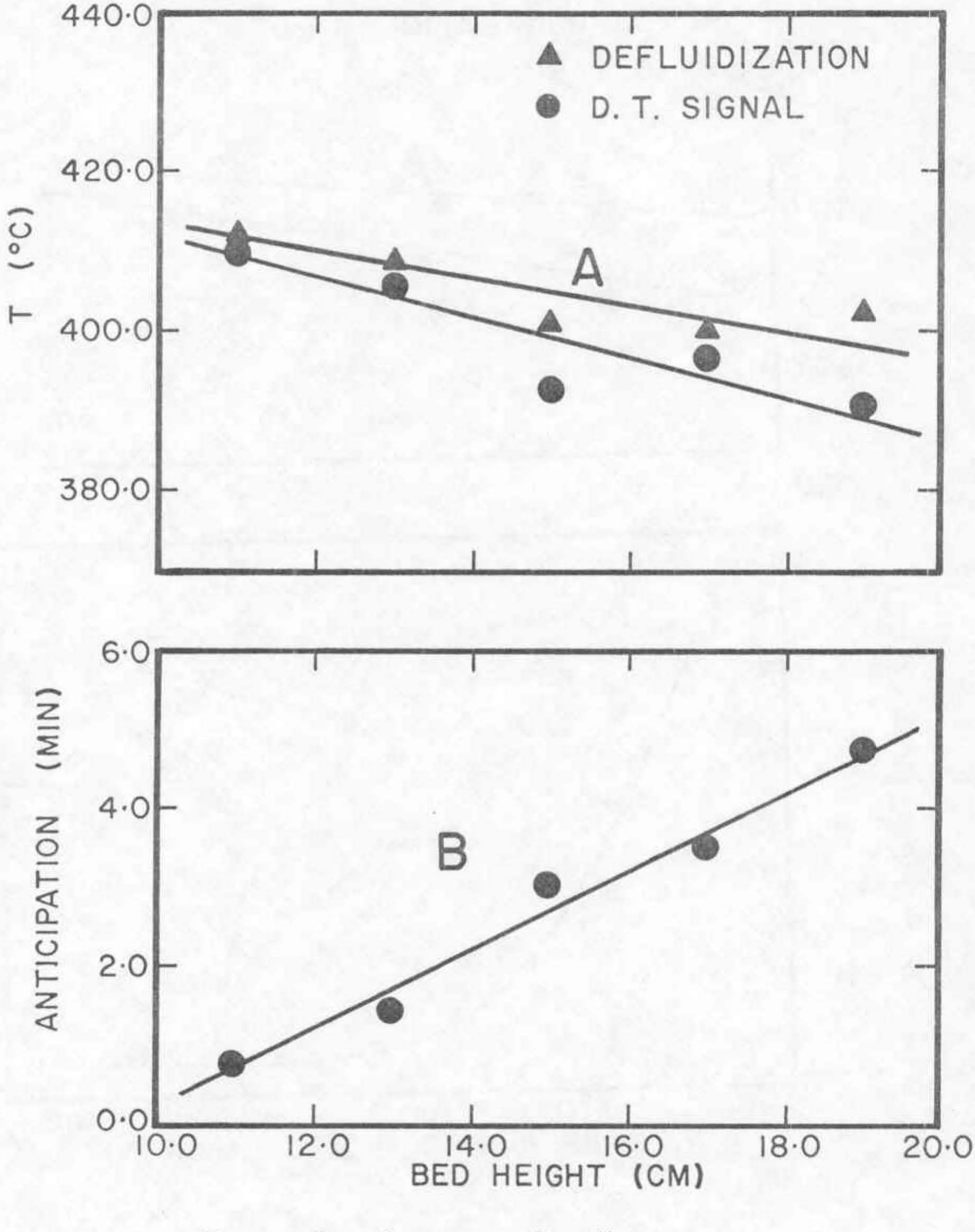


FIGURE 6. EFFECT OF BED HEIGHT.

AVG PARTICLE SIZE: 90.7 μM FLUIDIZING GAS VELOCITY: 4.4 CM/SEC
HEATING RATE: 10°C/MIN MAX FLUIDITY: 715 DDM

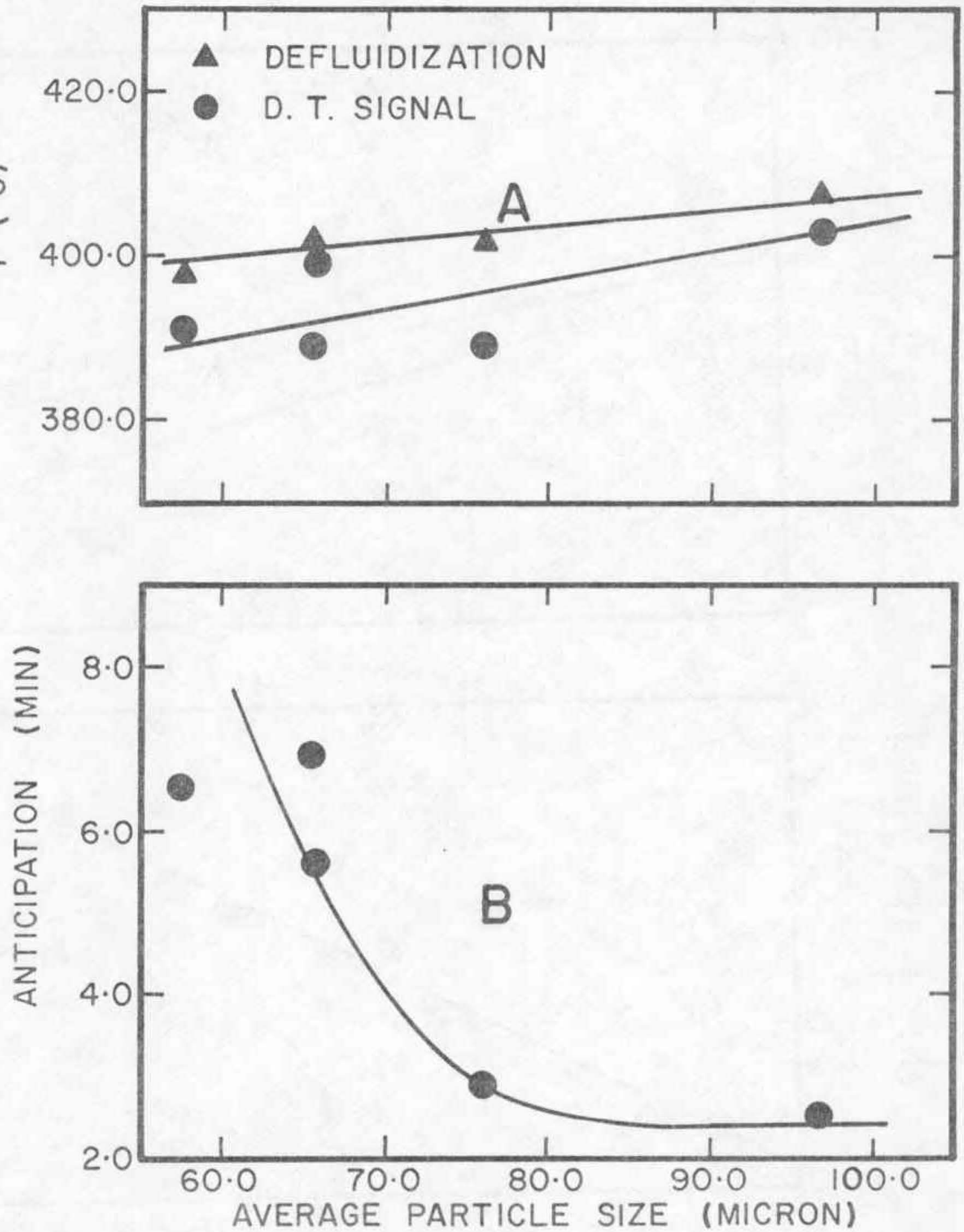


FIGURE 7. EFFECT OF PARTICLE SIZE.

FIXED BED HGT: 15 CM

HEATING RATE: $10^{O}$C/MIN

FLUIDIZING GAS VELOCITY: 4.4 CM/SEC

MAX FLUIDITY: 29-710 DDM

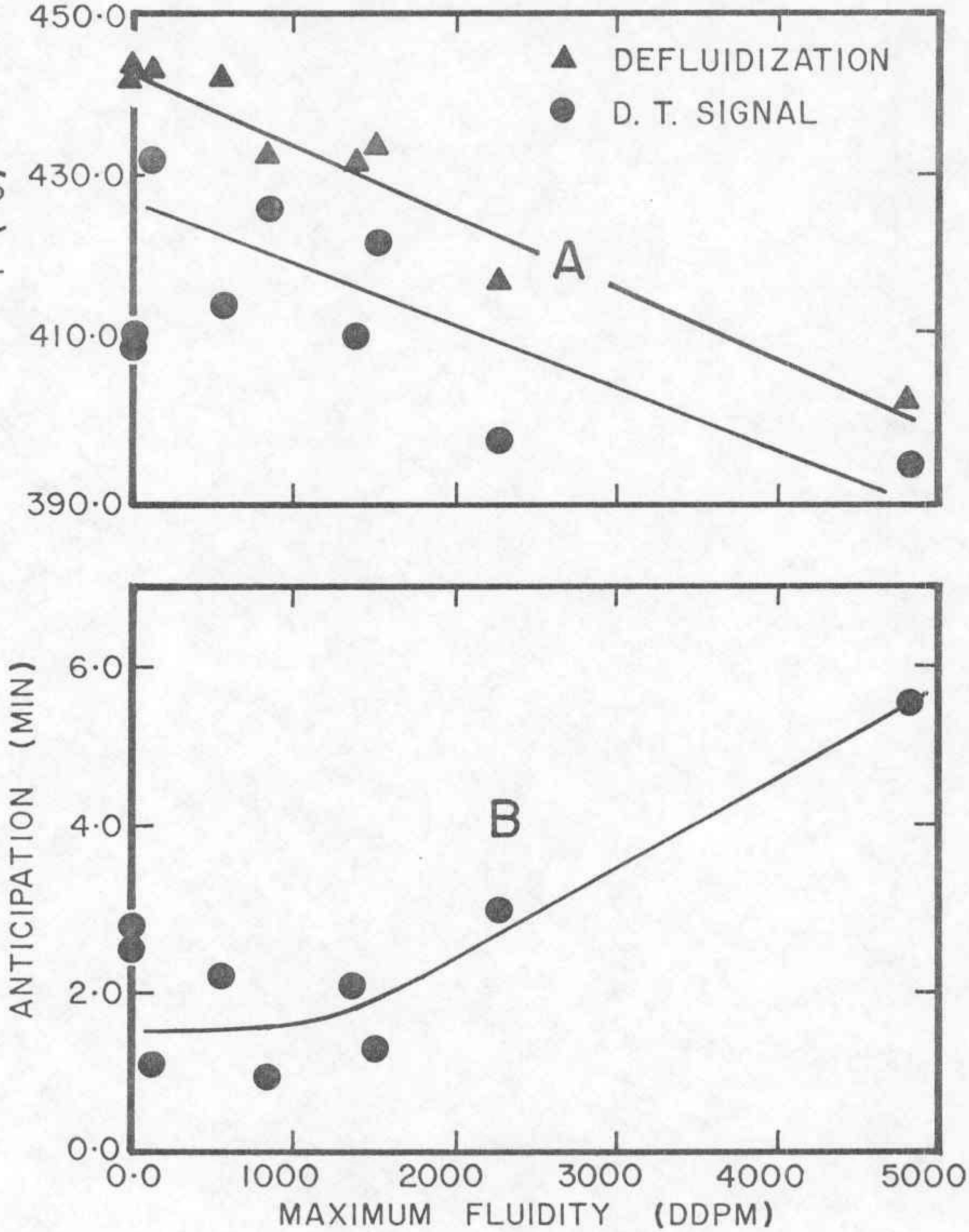


FIGURE 8. EFFECT OF COAL PLASTICITY.

FIXED BED HGT: 15 CM — FLUIDIZING GAS VELOCITY: 4.4 CM/SEC

HEATING RATE: 10°C/MIN — AVG PARTICLE SIZE: 107.1 μM