

01-4993114c.1
CPUB



Energy, Mines and Resources Canada
Énergie, Mines et Ressources Canada

CANMET

Canada Centre for Mineral and Energy Technology
Centre canadien de la technologie des minéraux et de l'énergie

MRL 87-138 (TR) e.1
MRL 87-138 (TR)

DUST EXPLOSIBILITY TESTS: HARTMANN APPARATUS

K.J. Mintz

CANADIAN EXPLOSIVE ATMOSPHERES LABORATORY

November 1987

BIBLIOTHÈQUE
CANMET
LIBRARY
MAY 18 1988
555 rue BOOTH ST.
OTTAWA, CANADA K1A 0G1

This document is an unedited
draft report prepared primarily
for internal and external
discussion purposes. It does not
represent the official expression of
the opinion of the Canada Centre
for Mineral and Energy Technology
(CANMET)

Ce document est un rapport
provisoire non-révisé et rédigé
principalement pour fin de
discussion et de documentation
interne. Il ne représente nullement
l'expression définitive de
l'opinion du Centre canadien de
la technologie des minéraux et de
l'énergie (CANMET)

40 pp

MINING RESEARCH LABORATORIES

DIVISION REPORT MRL 87-138 (TR)

e.1
CPUB

MICROMEDIA

MRL 87-138 (TR) e.1

7993114

01-7993114 c.1
CPUB

i

DUST EXPLOSIBILITY TESTS: HARTMANN APPARATUS

by

K.J. Mintz*

ABSTRACT

The Hartmann apparatus is used for measuring the maximum pressure and maximum rate of explosion pressure rise of dust-air explosions on a small-scale. The differences between the version used at CEAL and that described in ASTM Standard E789-81 are discussed. The operating procedure now in effect at CEAL for this equipment is described in detail.

* Research Scientist, Canadian Explosive Atmospheres Laboratory,
Mining Research Laboratories, CANMET, Energy, Mines and Resources
Canada, Ottawa

KEYWORDS: dust, explosibility, Hartmann apparatus



c.1
CPUB

ESSAIS POUR MESURER L'EXPLOSIVITÉ DE LA POUSSIÈRE : APPAREIL HARTMANN

par

K.J. Mintz*

L'appareil Hartmann est utilisé pour mesurer la pression maximale et l'énergie minimale d'inflammation des atmosphères explosives sur une petite échelle. Les différences entre la version utilisée par LCAE et celle décrite dans la norme E 789-81 de l'ASTM sont étudiées. Le mode de fonctionnement de l'appareillage, tel qu'observé présentement par LCAE, est décrit dans le détail.

*Chercheur scientifique, Laboratoire de recherche sur les atmosphères explosives, Laboratoires de recherche minière, CANMET, Énergie, Mines et Ressources Canada, Ottawa.

Mots-clé : Poussière, explosivité, Appareil Hartmann

CONTENTS

	<u>Page</u>
ABSTRACT.....	i
RÉSUMÉ.....	ii
INTRODUCTION.....	1
APPARATUS.....	2
CALIBRATION OF CEC 1000 STRAIN-GAUGE PRESSURE TRANSDUCER...	7
Apparatus required.....	8
Assembly of equipment.....	8
Digital oscilloscope/strain-gauge control unit set-up...	9
Calibration.....	11
CALIBRATION CHAMBER TESTS.....	13
Equipment.....	13
Assembly of equipment.....	13
Digital oscilloscope/strain-gauge control unit set-up...	14
Calibration test.....	16
DUST EXPLOSION TESTS.....	20
Equipment.....	20
Sample preparation.....	21
Explosion test.....	22
Analysis of the data.....	24
GENERATING A HARD COPY OF THE PRESSURE TRACE.....	27
Equipment.....	27
Assembly of apparatus.....	27
Operation.....	27
REFERENCES.....	29

FIGURES

<u>No.</u>	<u>Page</u>
1. Pressure trace for lycopodium: strain-gauge vs. piezoelectric pressure transducers.....	30
2. Pressure and derivative curves of lycopodium explosion..	31
3. Pressure and derivative curves of lycopodium explosion: expanded scales.....	32
4. Pressure and derivative curves for calibration chamber test.....	33
5. Pressure and derivative curves for calibration chamber test: expanded scales.....	34

APPENDIX

A. Data for calibration of CEC 1000 pressure transducer....	35
-------------------------------------------------------------	----

INTRODUCTION

Dust explosions occur when a cloud of combustible particles are ignited in a confined or partly confined space so that a relatively rapid pressure rise is generated. A laboratory apparatus to study or quantify dust explosions must, therefore, contain a means of dispersing dust, a source of ignition, and a pressure-measuring device. Such an apparatus was devised at the U.S. Bureau of Mines about 50 years ago and is commonly called the Hartmann apparatus, after its originator(1). Although it is now in some disfavour among researchers in dust explosions, primarily because of its small size, it is still in widespread use throughout the world and forms the basis for a recognized standard(2).

The Hartmann apparatus consists of a 50 mL air chamber, which is pressurized to 690 kPa. The air in this chamber is released quickly by a solenoid valve through a check valve to a mushroom-shaped air deflector, located at the base of a 70 mm internal diameter, 300 mm long steel tube. The air blast from this "mushroom" acts to disperse the dust which had been spread around the bottom of the tube. A continuous electrical discharge from electrodes located 114 mm from the bottom act as the igniting source. The pressure is measured continuously during the experiment by a pressure transducer located at the top of the tube.

The Mining Research Laboratories built a copy of the Hartmann apparatus several years ago, which has been used for

several studies(3,4). Recently, this apparatus has been rebuilt to conform to the ASTM Standard, and an improved electronic control and measuring system has been built(5).

The purpose of this report is to provide the operating procedures for the Hartmann apparatus and associated equipment for measuring the maximum pressure and maximum rate of pressure rise of dust-air explosions. Background information and justification of these procedures are also given.

APPARATUS

The apparatus is described completely in the ASTM Standard, with the only modification being the addition of a valve connected to the air supply in place of one of the viewing windows. The new tube, built by CANMET Technical Services Division, is sufficiently airtight that the pressure inside drops from 6.9 to 5.1 psi in 1.5 s, which is probably the maximum period of interest.

The pressure transducers used previously with the MRL Hartmann apparatus were piezoelectric types; the factory calibrations for converting voltage to pressures were used. No calibrations of these transducers had been carried out. Because dust-air explosions are deflagrations rather than detonations, it is unnecessary to use piezoelectric transducers, which have very fast response times but are not very stable. In addition, it is not possible to calibrate them statically. Instead, it was decided to use a strain-gauge transducer. The particular

transducer chosen, CEC 1000, is more accurate and stable than the ones recommended in the ASTM Standard. About the time that this transducer began to be used in the Hartmann apparatus, a dead-weight tester was purchased, which allowed absolute calibrations to be made of pressure-measuring devices. The procedure for use of this equipment is given below.

The piezoelectric transducers generated unusual pressure traces. A second peak, often larger and always broader than the first, usually was present(4). To resolve the question as to the validity of this anomalous peak, which has not been reported elsewhere in the literature, tests were carried out using both the piezoelectric transducer and the strain-gauge transducer attached to the top of the Hartmann apparatus; the pressure traces were recorded simultaneously. Fig.1 shows these traces for an explosion test on lycopodium powder, and proves that the piezoelectric transducers yielded inaccurate pressure traces.

The piezoelectric transducers also were sensitive to heat from the flame front of the explosion. Teflon tape or other material was attached to the diaphragm of the transducers to absorb heat. The strain-gauge transducer has a built-in temperature compensator, so that such corrective measures are not required.

The ASTM Standard recommends a recording oscillograph(such as a Visicorder) for recording the pressure trace from the transducer. The MRL system used a digital oscilloscope which offers several advantages: potentially higher precision, more convenient storage (on diskettes), the

possibility of analysing the data automatically (such as generating the derivative curve), and a more reliable triggering system. The output from the piezoelectric transducer/charge-amp system was sufficiently large that it could be input into the digital oscilloscope directly. Since the strain-gauge transducer output is too small to be used in this way, the strain-gauge module was removed from the Visicorder, modified so that it could operate independently of the Visicorder and act as input into the digital oscilloscope. An unexpected benefit from this modification was a substantial decrease in the background electrical noise from the transducer. The calibration procedure for the transducer uses exactly the same system as the explosion tests, thus, all errors of the system are included. The results, shown in Appendix A, demonstrates the excellent linearity of the transducer/ amplification system. Although it is recommended that the pressure transducer be calibrated monthly, the stability of this transducer over the past 5 years indicates that this is strictly a precautionary move.

A calibration chamber was built by the CANMET Technical Services Division to the ASTM specifications. Its purpose is to verify that the air delivery system is working satisfactorily, by using it in place of the Hartmann tube. When it was tried with the original MRL system, it was found that the maximum pressure was 214 kPa, much higher than that specified (172 ± 14 kPa). The maximum rate of pressure rise deviated even more, but in the opposite direction: 3.4 Mpa/s vs. 6.72 ± 0.34 Mpa/s. The reason was primarily the use of a relatively large amount of thin

tubing in the air system. This adds additional volume to the air chamber and thus produces a final higher pressure in the calibration chamber. The thin tubes do not allow the air to flow out sufficiently quickly, thus producing too low a rate of pressure rise. The air delivery system was rebuilt to minimize the excess volume. The total volume was measured to be 68.2 mL (by filling with water). The final pressure in the calibration chamber tests is 178 kPa, well within the tolerances of the Standard. It would appear that the actual volume of the air delivery system of the standard Hartmann is also well above 50 mL.

The use of the correct solenoid valve is important, so that the air is released from the dust dispersion system in a smooth, reproducible manner. The original solenoid valve in the MRL Hartmann apparatus was an obsolete model. Using this solenoid valve with the rebuilt air-delivery system yielded a shoulder on the pressure trace. This shoulder was eliminated by using the current model of solenoid valve. The maximum rate of pressure rise is now within the tolerances: A set of 6 trials yielded 6.82 ± 0.18 MPa/s.

The tolerance limits specified in the ASTM Standard for the calibration chamber tests are 8% for the maximum pressure and only 5% for the maximum rate of pressure rise, despite the latter measurement being intrinsically more imprecise than the former.

The Standard specifies the maximum rate of pressure rise is to be measured by drawing the tangent to the pressure curve. This is imprecise and is subject to human bias. It is doubtful

whether an accuracy of better than 10% can be achieved by this method. The method used in this report is numeric, and is thus capable of higher accuracy. A program that came with the oscilloscope calculates the slope at each point, by a least squares fit, using the number of data points specified by the user (the "window" size). Some human bias comes in through the choice of window size. A large window size decreases the calculated maximum rate of pressure rise; a small one may produce too noisy a curve. The optimum window size is probably the smallest that produces a smooth, non-noisy curve. The procedure given in this report specifies that the window size for the calibration chamber test be 20. A window size of 10 produces a curve that is too noisy and has a mean maximum value 8% higher. The selection of appropriate window size of explosion tests is specific to the test. The only generalization is that very fast explosions, such as lycopodium, should use a window size of 10; very slow explosions may have to use a window size of 45 (the maximum possible).

An Omega strip chart recorder is connected to the Nicolet oscilloscope to enable hard copies of the traces on the oscilloscope screen to be obtained. These are for illustrative purposes only and should not be used for calculation of the actual experimental results. The procedure for the use of this system is given below. Although possible, it is inconvenient to put multiple traces on the same graph using this recorder. It is possible that, in the future, this chart recorder will be replaced by either an XY recorder or a digital plotter to enable

multiple traces and publication quality plots to be obtained.

Figure 2 is the hard copy of the pressure trace of an explosion test on lycopodium powder at a concentration of 742 mg/L (which is the concentration that yields the maximum explosion pressure), along with its derivative curve. By turning the horizontal expansion to 16X, the most important part of the trace can be seen in more detail on the oscilloscope screen and reproduced on the Omega(Fig.3). The time at which the air solenoid is turned on can now be seen clearly. The spikes that go in the positive direction are due to the high voltage discharge. It can be seen that, for this particular explosion test, the maximum rate of pressure rise occurs at about half of the maximum pressure.

Figure 4 shows the hard copy of the pressure trace of a calibration chamber test. The vertical expansion for the pressure is 4X, for the derivative curve, 16X. The ripple in the baseline of the latter is due to the intrinsic noise of the transducer/amplification system. Figure 5 shows the same curves at a horizontal expansion of 16X and a vertical expansion of 16X. With this expansion, the pressure trace shows some oscillations halfway up the curve, but the derivative program is able to smooth out the data sufficiently.

CALIBRATION OF CEC 1000 STRAIN-GAUGE PRESSURE TRANSDUCER

This calibration should be carried out monthly or if it suspected that the transducer may have been damaged. The entire procedure takes less than two hours.

Apparatus Required

Ashcroft Model 1305 Dead-weight Tester

Strain-gauge control unit

Nicolet Digital Oscilloscope Model 4094 mainframe with 4562 module

Assembly of Equipment

1. Remove dead-weight tester from box and place it on the laboratory bench.
2. Install low-pressure range piston assembly in one fitting.
3. Place a bubble level on top of the piston assembly and shim the tester until it is level.
4. Connect one end of the copper pipe to the other fitting in the tester and the other end to the adaptor, which is then connected to the fitting for the transducer.

NOTE: The transducer should be at about the same height as the top of the piston assembly, otherwise a small error in the pressure due to the head of liquid will occur.

5. Turn the "release" knob on the tester counterclockwise so that the pressure in the system is at atmospheric.
6. Attach a fully shielded cable from the pressure transducer to the strain-gauge control unit (connector is at the back of the unit).
7. Connect a cable from the output of the control unit (located on the front) to the positive terminal of channel A on the 4562 module.

NOTE: This cable is normally left connected.

Digital Oscilloscope/ Strain-gauge Control Unit Set-up

1. Turn 'scope and control unit on, switch "trace" to "zero" and allow to warm up for 10 minutes.
2. Check the following switches on the 4562 module:
 - (a) the channel A switch should be "on"
 - (b) the channel B switch should be "off"
 - (c) the channel A "+" switch should be at "DC"
 - (d) the channel A "-" switch should be at "gnd"
 - (e) the volts full scale switch should be at ± 4 volts
 - (f) the "coupling" switch should be at "DC"
 - (g) the "average", "point average", "save ref", "filter", and "view" switches should all be at "off"
 - (h) the "slope" switch should be at "+"
 - (i) the "source" switch should be at "A" .
3. Check the following switches on the 4094 'scope:
 - (a) "autocenter" should be on
 - (b) the two "expansion" switches should be at zero
 - (c) the "function" switch should be at "reset num"
 - (d) "Y/T" should be selected.
 - (e) the "memory" should be at "all"

NOTE: The operations indicated by the function switch occur only when the "execute" button is pressed. "Reset num" zeros the time and voltage displays on the bottom of the screen.

4. Check the following switches on the control box:
 - (a) The "cal/supp" switches should be at "x10" and "0".
 - (b) The pot should be at 8.44 mV.

NOTE: The original pot in the SGC module was replaced by one with

a higher resolution; therefore, the actual mV is one-fifth of the reading.

5. On the 4562 module, press the "live" button (which should light up), turn the "time per point" to 20μ , and turn trigger to "auto".

NOTE: The "trigger" light should flash about once per second, indicating a new sweep.

6. The trace should be near the bottom of the screen, just above the time and voltage display. If not, adjust the position of the trace using the "position" knob on the 4562 module.
7. Press "execute" on the 4094: the voltage should oscillate between -2 and +2 mV.
8. Turn "trace" on control unit to "norm" and adjust "balance" until the voltage on the 'scope is within the range of -10 and +10 mV. The voltage should oscillate over a range of 4 mV. Turn "trace" to "rev": the voltage should be the same but with the sign reversed.

NOTE 1: The balance adjustment is very sensitive: turn it very slowly. The resolution of the balance potentiometer is insufficient to bring the balance exactly to zero.

NOTE 2: If the range of oscillation on "norm" is the same as on "zero", and the voltage reading remains at zero, then there is probably a problem with the transducer cable.

9. Turn "trace" to "norm" and press "execute" on 4094.
10. Turn bottom toggle switch of "cal/supp" on control unit to "-". The trace on the screen should be near the top and

the voltage should read $5.98 \pm .02$ V. If not, repeat all previous steps.

NOTE: If the problem persists, then it indicates that the sensitivity pot on the control unit may have been adjusted. Do not adjust the sensitivity pot until the pressure calibrations below have been carried out.

Calibration

1. Turn "cal/supp" switch to "0".
2. Turn "release" knob on tester fully clockwise.
3. Place the weight marked L10 weight on piston assembly.

NOTE 1: The L10 marking means that that weight exerts a pressure of 10 psi in the tester.

NOTE 2: The piston assembly exerts a pressure of 5 psi, so the actual pressure in the tester is 15 psi.

4. Pump tester slowly until the piston assembly rises about 2 cm.

NOTE: If the assembly rises too much, lower it by opening the "release" knob slightly.

5. Rotate the piston assembly and record the mean voltage displayed on the 'scope while the piston assembly is turning freely.
6. Increase the load on the piston assembly by 5 psi increments until the total is 120 psi, each time repeating steps 4 and 5.
7. Calculate the regression (least squares) line, requiring it to pass through the point (0,0), for voltage vs. pressure.

The results of the calibration carried out on Nov. 2, 1987 are shown in Appendix A. The maximum deviation from the line was 4 mV, which is approximately the noise level in the system. The scatter of errors in the positive and negative directions indicates that the transducer is very linear over this pressure range. The results should be similar to these, both in the actual values and in the scatter.

The reciprocal of the slope is the calibration factor and should be $20.06 \pm .06$ psi/volt (13.83 kPa/volt).

NOTE: The calibration factor is expressed in volts displayed on the 'scope. This is not the calibration factor of the transducer because the strain gauge module has amplified the transducer signal by a factor of 354.3. The factory calibration in 1982 was 0.1428 mV/psi, which is 1.5% higher than the current value of 0.1407 mV/psi.

8. If the calibration factor is incorrect and the voltage in step 10 of the previous section was incorrect, then the sensitivity of the strain-gauge module must be readjusted:
 - (a) Turn the "trace" to "zero" and press "execute" to zero numbers on the screen.
 - (b) Switch cal/supp to "-" and adjust "sens" so that voltage on screen reads 5.98 V.
 - (c) Switch cal/supp to "0" and adjust "pos" so that voltage on screen reads 0 V.
 - (d) Repeat (b) and (c) until both zero and span are

correct.

9. If the calibration factor is incorrect and the voltage in step 10 of the previous section was correct, then the transducer is suspect. If the linearity of the transducer is satisfactory, then repeat the calibration procedure for several days to ensure stability of the transducer. If the linearity of the transducer is unsatisfactory, replace the transducer.
10. THOROUGHLY CLEAN THE OIL FROM THE EXPOSED PARTS OF THE TRANSDUCER.

CALIBRATION CHAMBER TESTS

This test should be carried out before each series of dust explosion tests to confirm that the apparatus is functioning correctly. It takes about half an hour.

Equipment

Base of Hartmann apparatus with attached 50 cc air chamber
 Air cylinder (grade extra dry)
 Calibration chamber
 Explosion control panel
 Strain-gauge control unit
 Nicolet 4094 digital oscilloscope with 4562 module and XF-44
 disk drive CEC 1000 pressure transducer

Assembly of Equipment

1. Place Hartmann base, which has attached to it a Circle Seal model 2232 check valve, an Asco model 8210D2 solenoid valve and a 50 mL air chamber, in the fume hood.

NOTE: The results obtained in this section are critically dependent on the specified equipment being used.

2. Connect the hose from a cylinder of air to the valve leading to the air chamber.
3. Turn the mushroom four full turns counterclockwise from the closed position.
4. Ensure that the O-ring is in place on the base.
5. Place the calibration chamber on the base, and clamp down finger-tight.
6. Attach the transducer to the top of the calibration chamber.
7. Connect a fully-shielded cable from the transducer to the strain-gauge control unit.
8. Connect a cable from the output of the control unit to the positive terminal of channel A on the 4562 module.

NOTE: This cable is normally left connected.

Digital Oscilloscope/ Strain-gauge Control Unit Set-up

1. Turn 'scope and control unit on, switch "trace" to "zero" and allow to warm up for 10 minutes.
2. Check the following switches on the 4562 module:
 - (a) the channel A switch should be "on"
 - (b) the channel B switch should be "off"
 - (c) the channel A "+" switch should be at "DC"
 - (d) the channel A "-" switch should be at "gnd"
 - (e) the volts full scale switch should be at ± 4 volts
 - (f) the "coupling" switch should be at "DC"
 - (g) the "average", "point average", "save ref", "filter",

and "view" switches should all be at "off"

- (h) the "slope" switch should be at "+"
- (i) the "source" switch should be at "A" .
- (j) the trigger "level" and "sens" knobs should be fully counterclockwise.

3. Check the following switches on the 4094 'scope:

- (a) "autocenter" should be on
- (b) the two "expansion" switches should be at zero
- (c) the "function" switch should be at "reset num"
- (d) "Y/T" should be selected.
- (e) the "memory" should be at "all"

NOTE: The operations indicated by the function switch occur only when the "execute" button is pressed. "Reset num" zeros the time and voltage displays on the bottom of the screen.

4. Check the following switches on the control box:

- (a) The "cal/supp" switches should be at "x10" and "0".
- (b) The pot should be at 8.44 mV.

NOTE: The original pot in the SGC module was replaced by one with a higher resolution; therefore, the actual mv is one-fifth of the reading.

5. On the 4562 module, press the "live" button (which should light up), turn the "time per point" to 20μ , and turn trigger to "auto".

NOTE: The "trigger" light should flash about once per second, indicating a new sweep.

6. The trace should be near the bottom of the screen, just above the time and voltage display. If not, adjust the

position of the trace using the "position" knob on the 4562 module.

7. Press "execute" on the 4094: the voltage should oscillate between -2 and +2 mV.
8. Turn "trace" on control unit to "norm" and adjust "balance" until the voltage on the 'scope is within the range of -10 and +10 mV. The voltage should oscillate over a range of 4 mV. Turn "trace" to "rev": the voltage should be the same but with the sign reversed.

NOTE 1: The balance adjustment is very sensitive: turn it very slowly. The resolution of the balance potentiometer is insufficient to bring the balance exactly to zero.

NOTE 2: If the range of oscillation on "norm" is the same as on "zero", and the voltage reading remains at zero, then there is probably a problem with the transducer cable.

9. Turn "trace" to "norm" and press "execute" on 4094.
10. Turn bottom toggle switch of "cal/supp" on control unit to "-". The trace on the screen should be near the top and the voltage should read $5.98 \pm .02$ V. If not, repeat all previous steps.

NOTE: If the problem persists, then it indicates that the sensitivity pot on the control unit may have been adjusted. Do not adjust the sensitivity pot. Carry out the calibration procedure of the pressure transducer given above.

Calibration test

1. Turn the "memory" switch to H1.

NOTE: H1 means that the data points will be stored in alternate

locations in the memory. The other half of the memory will be used for the derivative curve.

2. On the 4562 module, turn the time per point to 200μ and "auto" to "norm".
3. Set trigger position:
 - (a) Press "hold last" and "hold next" buttons simultaneously. When they are released, all three lights should go on, and the screen should show 1AS on the bottom line. The cursor should be at the left edge of the screen and the time should be at 0 s.
 - (b) Turn "function" to "data move".
 - (c) Press trigger position A to right (crosshair will move to right) until the time on the screen reads 100 ms.

NOTE: The "pretrigger" is now set up so that the 'scope will store and display 100 ms of pressure trace prior to the triggering caused by the pressure rise. The delay light indicates that the pretrigger option is in use.
4. Press "hold last" to exit the set-up mode.
5. Turn "function" to "reset num".
6. Insert 4094 program diskette into the disk drive. "P01" (= program no.1) will be displayed.
7. Press "up" until display shows "P10".
8. Press "recall", which transfers this program into the mainframe. The screen will display "derivative".
9. Turn on control panel and set "delay" to 30 ms, "air on time" to 150 ms, and "delay" to "air".
10. Unplug the transformer from the control unit (so that arc

does not operate) and ensure that air plug is in.

11. Open main valve of air cylinder and adjust outlet pressure to about 760 kPa(110 psi).
12. Fill air chamber to exactly 690 kPa (100 psi). Close inlet valve of air chamber.
13. Press "live" and "hold next" on the 4562 module.
14. While pressing "enable" button, move toggle switch to "run". The pressure in the air chamber should drop to about 240 kPa(35 psi), the "hold last" button should light, the other two button lights should go off and a pressure trace should appear on the screen.
15. Turn the horizontal expansion to X8, move the cursor to the beginning of the spike, which is located a few ms before the pressure rise.

NOTE: In order to see this spike (which is due to the opening of the relay that controls the solenoid valve), it may be necessary to turn the vertical expansion on.

16. Press "execute" to zero the time and voltage on the screen.
17. Move the cursor to the right and find the maximum voltage. Multiply it by the calibration factor of the pressure transducer to determine the maximum pressure.

NOTE: The maximum pressure should be 178 ± 0.4 kPa (25.8 ± 0.4 psi). A higher value may indicate that the pressure transducer is out of calibration. A lower value may indicate that the calibration chamber is not fastened down tightly, the solenoid or check valve is faulty, or the pressure transducer is out of

calibration.

18. Move the cursor to the second spike, which should be readily observable even with no expansion. The time at the beginning of this spike should be 150 ± 2 ms.

NOTE: If it is not, then the control panel must be checked by an electronics technician.

19. Measure the maximum rate of pressure rise:

- (a) Turn "function" to "prgm".
- (b) Press "execute" to start program and follow instructions on screen.
- (c) Set the window size to 20, by using the cursor buttons: the "up" button increments the numbers, the "left" button moves the cursor to the tens position.

NOTE: The window size refers to the number of points used for the calculation of the derivative at each point. The larger the number, the lower the calculated maximum rate, because points away from the maximum slope are included. Too small a window causes a high noise level. A window size of 20 was chosen on the basis of experiments.

- (d) For speed, set the start position just before the pressure starts to rise, and set the stop at the beginning of the plateau.
- (e) After the 'scope has generated the derivative curve, turn the vertical and horizontal expansions to x8 and move the cursor to the maximum of the peak. Multiply the voltage reading at the maximum by the transducer calibration factor to obtain the maximum rate of pressure rise.

NOTE 1: The maximum rate of pressure rise should be 6.72 ± 0.34 MPa/s (975 ± 50 psi/s). A value outside this range may indicate a faulty solenoid or check valve.

NOTE 2: The original trace can be seen by turning "memory" to H2; turning "memory" to "all" will superimpose both traces.

20. The pressure and derivative traces may be compared to those shown in Fig. 4 and 5. The former shows the entire trace, i.e., no horizontal expansion; the latter shows only the area of interest (horizontal expansion set to X16). Although the shape of the pressure trace should be virtually identical to that shown, the shape of the derivative curve cannot be expected to be reproducible.

DUST EXPLOSION TESTS

SAFETY GLASSES AND A LAB COAT MUST BE WORN.

It is assumed that the calibration chamber test (previous section) has already been carried out, and the equipment has been assembled and tested out.

Equipment

Base of Hartmann chamber with attached 50 cc air chamber

Air cylinder (grade extra dry)

Explosion control panel

Strain-gauge control unit

Nicolet 4094 digital oscilloscope with 4562 module and XF-44

disk drive CEC 1000 strain-gauge pressure transducer

Hartmann chamber (ASTM version)

Luminous tube transformer, 12kV, 30mA (Allanson model N453B)

Balance, accurate to 0.1 mg

Oven

Desiccator

Brass spacer, 6.2 mm

Wire brush

Ro-Tap sieve shaker

Sample preparation

NOTE: The method of preparation of the sample may differ from the procedure given here. Check with the supervisor before carrying this work out.

1. Sieve the sample:

- (a) Ensure that the stainless steel collector pan, lid and 200 mesh sieve (75 μ opening) are all clean and dry.
- (b) Place the screen and collector pan on the support for the shaker.
- (c) Pour the sample onto the screen and place the lid firmly on the sieve.
- (d) Place the top of the shaker on the lid .
- (e) Turn the timer on the Ro-Tap for 10 minutes.
- (f) If after that time, insufficient material has passed into the collector pan, turn on the shaker for a longer time.

2. Place the -200 mesh fraction in a glass or polypropylene beaker and heat in a oven at 80°C for 24 hours.

3. Remove from the oven and place in a desiccator.

NOTE: The desiccator must contain indicating silica gel, which is bright blue. If it is not that colour, heat the silica gel in an oven at 100°C until it turns bright blue.

Explosion Test

1. Plug the transformer into the control unit. The time delay should be set at 30 ms, the air time at 150 ms and the arc time should be at 500 ms.

NOTE: The arc time can be adjusted downwards if the reaction is fast and the spikes due to the arc are interfering with the analysis of the pressure trace. Similarly, the air on time can be lowered to 100 ms if the spike associated with the solenoid closing happens to coincide with an interesting part of the pressure trace.

2. Ensure that the Hartmann tube and base are clean, the electrodes are clean, and the O-ring is in place on the base.
3. With the Hartmann tube upside down, insert the electrodes into their holders, and adjust the spacing between them using the 6.2 mm brass spacer.
4. Weigh out the required amount of sample.

NOTE: The purpose of these tests is usually to determine the maximum explosion pressure. To do that, the optimum concentration must be determined. The concentrations normally used are: 100, 200, 500, 1000 and 2000 g/m³, which correspond to these quantities to be weighed out: 0.123, 0.246, 0.615, 1.23 and 2.46

g. Once the approximate concentration is found, at least 4 tests are carried out at that concentration.

5. Spread out the dust in a uniform layer on the base of the Hartmann.
6. Place the Hartmann tube on the base and clamp down finger-tight.
7. Screw the pressure transducer into the hole of the lid of the Hartmann and attach the cable.
8. Ensure that the O-ring is on the lid and screw the lid locking ring onto the Hartmann tube, fingertight.
9. (Optional) Turn on the valve from the air cylinder to allow a low flowrate of clean, dry air to flush out the tube for 5-10 minutes. The locking ring is left slightly loose during this procedure so as to allow the air to exit. At the end of this conditioning time, close the air valve and tighten the locking ring simultaneously.

NOTE: Moisture decreases the severity of explosions. Hence, dry air will be the worst case condition. The above procedure is strongly recommended when the relative humidity in the laboratory is above 30%. The procedure above is also carried out when tests are required to be carried out in pure oxygen.

10. Flush out the air chamber at least three times with dry air, then adjust the pressure inside to 690 kPa (100 psi).
11. Close the doors of the fume hood and turn on the fan.
12. Press "live" and "hold next" on the 4562 module.
13. While pressing "enable" button, move toggle switch to "run". The pressure in the air chamber will drop to about

120 kPa (18 psi), the "hold last" button will light, the other two button lights will go off and a pressure trace will appear on the screen.

NOTE: Additional spikes will appear on the pressure trace; these are due to pick-up from the high-voltage discharge.

14. Open the locking ring of the Hartmann lid slowly to release the pressure.
15. Remove the locking ring, together with the lid and transducer.
16. Remove the electrodes and use emery cloth to clean them.
17. Remove the Hartmann tube from the base and clean all components with the vacuum cleaner, compressed air and the wire brush.

Analysis of the data

It is convenient to carry out the analysis while the next sample is being conditioned in the Hartmann chamber.

1. Insert a formatted diskette into the right hand disk drive.
2. Make a title for the pressure trace.
 - (a) Press "up" on disk drive until P29 is displayed.
 - (b) Press "recall", which transfers this program to the mainframe. The screen will display "title".
 - (c) Turn "function" to "prgm", and press "execute".
 - (d) Use the cursor buttons to write the sample name and/or number and the date. The left/right buttons move the cursor along the title line; the up and down buttons

scroll through the alphanumeric characters.

- (e) When completed, turn "memory" to "all", press "execute" turn "function" to "reset num", and turn "memory" back to H1.

NOTE: The title will remain in the mainframe (though sometimes invisible), thus it is unnecessary to rewrite the title until a change is required.

3. Press the "down" button on the disk drive until P10 is displayed.
4. Press the "recall" button to bring the derivative program to the mainframe.
5. Select a blank track by pressing the "up" button until the "protect" light goes off.
6. Press "store" on the disk drive; the light on the right disk drive will go on briefly, indicating that the data is being written on the diskette. The track number will increment; press "down" to display the original track number. The "protect" light will go on.
7. Turn "function" to "prgm"; press "execute" to start the "derivative" program. The pressure trace will be transferred to H2, and will be replaced in H1 by the derivative curve.

NOTE: The selection of the appropriate window size is a matter of trial and error. For very fast reactions, such as lycopodium at 1 g/L, the appropriate window size is 10; for very slow reactions, the maximum window size of 45 must be used. If there is a lot of noise on the derivative trace, then a larger window size should be

used (if possible). The objective is to have the smallest window size that generates a reasonably smooth derivative curve.

8. Move the cursor to the maximum of the derivative curve. (Turn vertical and horizontal expansion switches as necessary to locate the maximum). Multiply the voltage displayed by the calibration factor to obtain $(dP/dt)_m$, the maximum rate of pressure rise.
9. Turn "function" to "reset num", and turn "memory" to H2.
10. Move the cursor to the beginning of the pressure rise. Use vertical and horizontal expansions as required.
11. Press "execute" to zero the time and voltage on the screen.
12. Move the cursor to the beginning of the steeper pressure rise. Multiply the voltage display by the calibration factor to obtain P_i , the air pressure at the time of ignition. The time display is t_i , the time between air entering the chamber and ignition of the dust cloud.

NOTE: The location of the ignition point is often very difficult to decide, particularly for very fast reactions. An example of one such case is shown in Fig.3. The value of P_i must always be less than 48 kPa (6.9 psi), the pressure reached in the chamber when no explosible dust is present.

13. Press "execute" to zero the voltage and time.
14. Move the cursor to the maximum of the curve. (Expand vertical and horizontal scales as necessary.) Multiply the voltage by the calibration factor to obtain P_m , the explosion pressure. The time is t_m , the rise time of

the explosion.

15. Switch "memory" to H1, which is the derivative curve.
16. Move the cursor to the maximum, press "execute", then turn back to H2. The time display is t_r , the time from the ignition to the maximum rate of pressure rise.

GENERATING A HARD COPY OF THE PRESSURE TRACE

Apparatus Required

Nicolet 4094 Digital Oscilloscope

Omega 555 strip chart recorder

Assembly of Apparatus

1. Pull the "rate" knob on the back of the Nicolet out and turn it fully counterclockwise.

NOTE: This selects the output for an x-t chart recorder and sets the speed at the slowest possible (about 3 minutes for the entire trace).

2. Connect the cable from the "vert" BNC connector on the back of the Nicolet to the + and - inputs of the Omega.

NOTE: The recorder should be left connected in this way.

Operation

1. Turn on the recorder.
2. Ensure that the "input" is at 5 V, "atten" is fully counterclockwise and the toggle switch is at "record".
3. Select chart speed of 12 if the graph is desired to go vertically on standard size paper or 20 if horizontal.
4. Use the "zero" knob on the Omega to set the pen at the

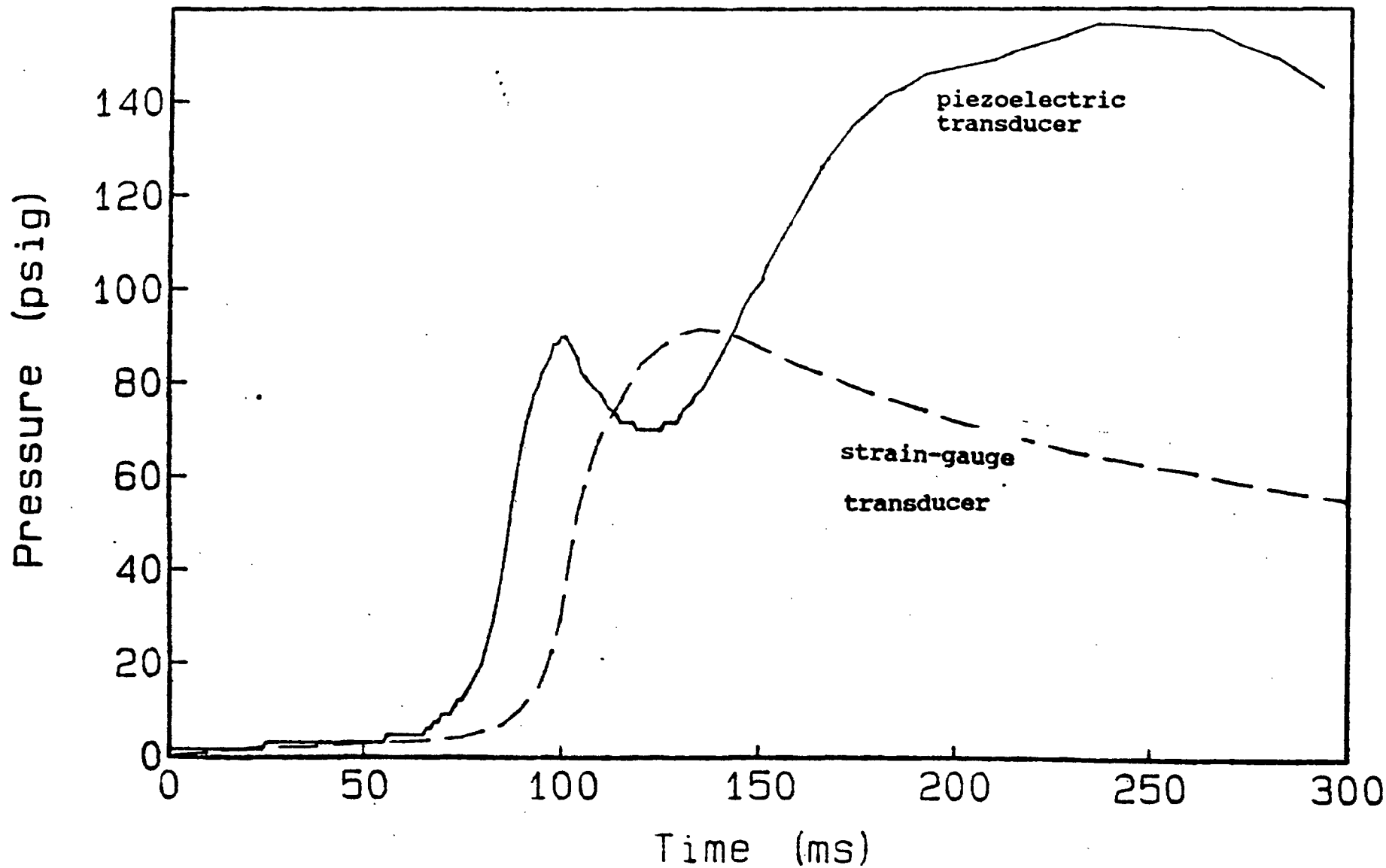
baseline. If pen is not writing, replace pen.

5. Select the area of interest and vertical and horizontal expansion desired.
6. Turn the "function" switch to "pen".
7. Turn the switch on the Omega to "cm/min". Paper will start to move.
8. Press "execute" to start the transfer of data. The screen will go blank except for a bright dot which traces out the curve.
9. After the trace has been completed, remove the hard copy by lifting the lever on the left-hand side of the recorder and pulling the paper through.

REFERENCES

1. Dorsett, H.G. et al "Laboratory equipment and test procedures for evaluating explosibility of dusts", U.S. Bureau of Mines RI5624; 1960
2. ASTM Standard E789-81 "Pressure and rate of pressure rise for dust explosions in a closed vessel", American Society for Testing and Materials; 1981
3. Feng, K.K. "Hazardous characteristics of Canadian coal dusts", Division Report ERP/MRL 82-132(OPJ); CANMET, Energy, Mines and Resources; 1982
4. Cheng, K.C. and Cox, D. "Explosibility tests on ferrosilicon dust", Division Report MRL 86-151(TR); CANMET, Energy, Mines and Resources; 1986.
5. Judge, K.J. "Instrumentation for Hartmann and 20-L dust explosibility tests at Canadian Explosive Atmospheres Laboratory", Division Report MRL 87-124(TR); CANMET, Energy, Mines and Resources; 1987.

Fig.1. Pressure Trace: Lycopodium 86/12/10



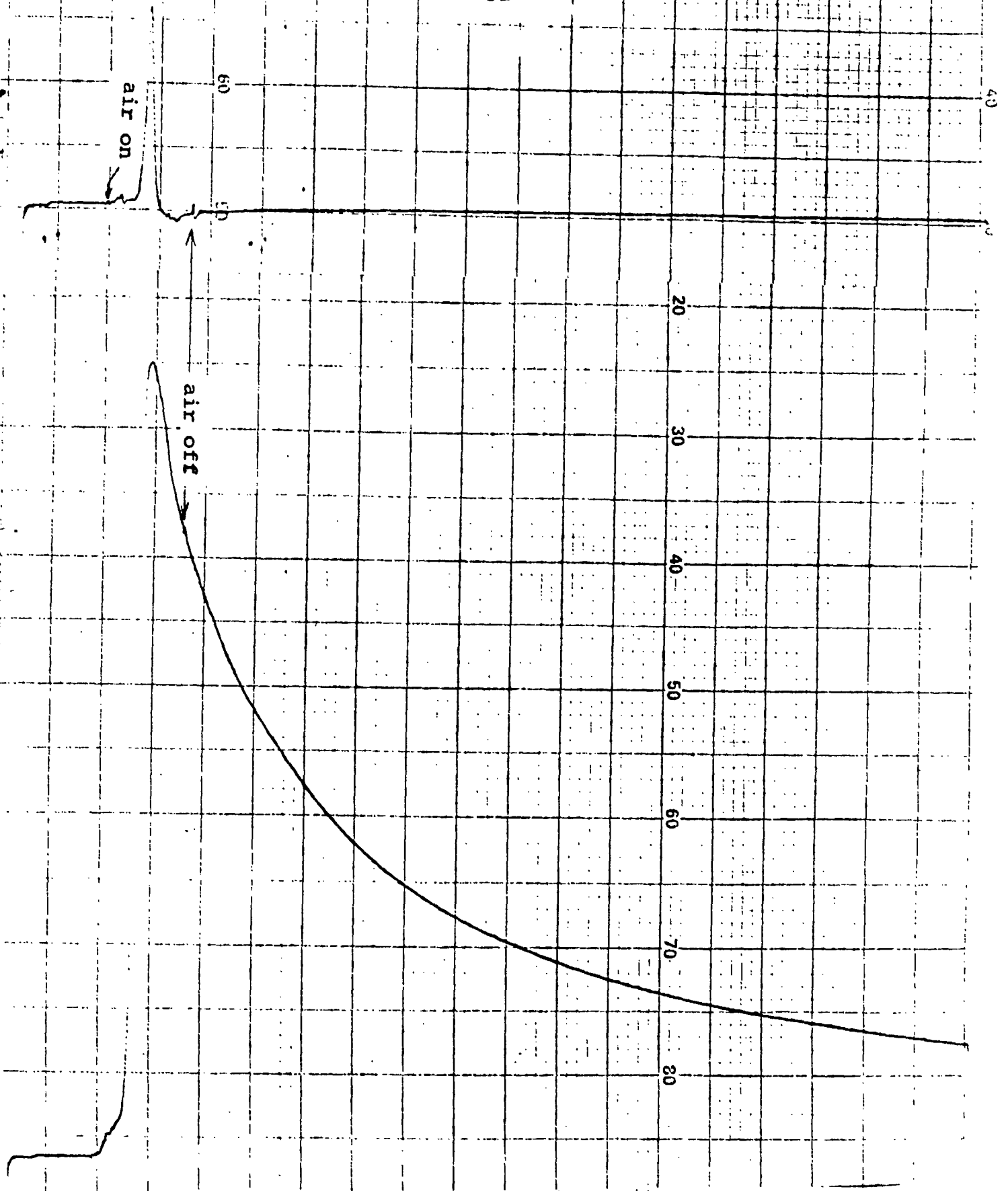


Fig.2. Pressure trace (lower plot) and derivative curve (upper plot) of an explosion test on lycopodium powder at a concentration of 742 mg/L, in the Hartmann apparatus

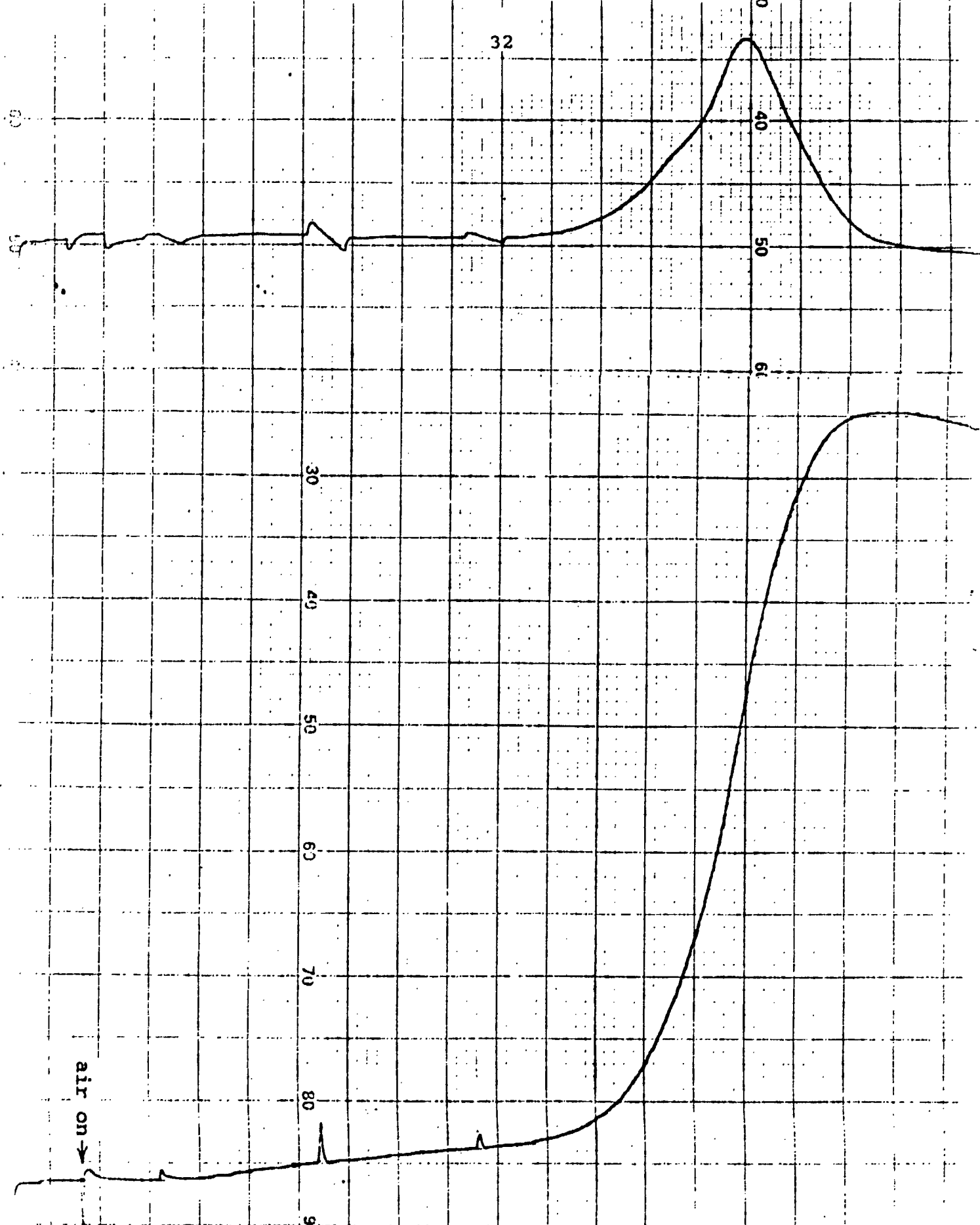


Fig.3. Pressure trace (lower curve) and derivative curve (upper plot) of an explosion test on lycopodium powder, with the horizontal expansion at 16X

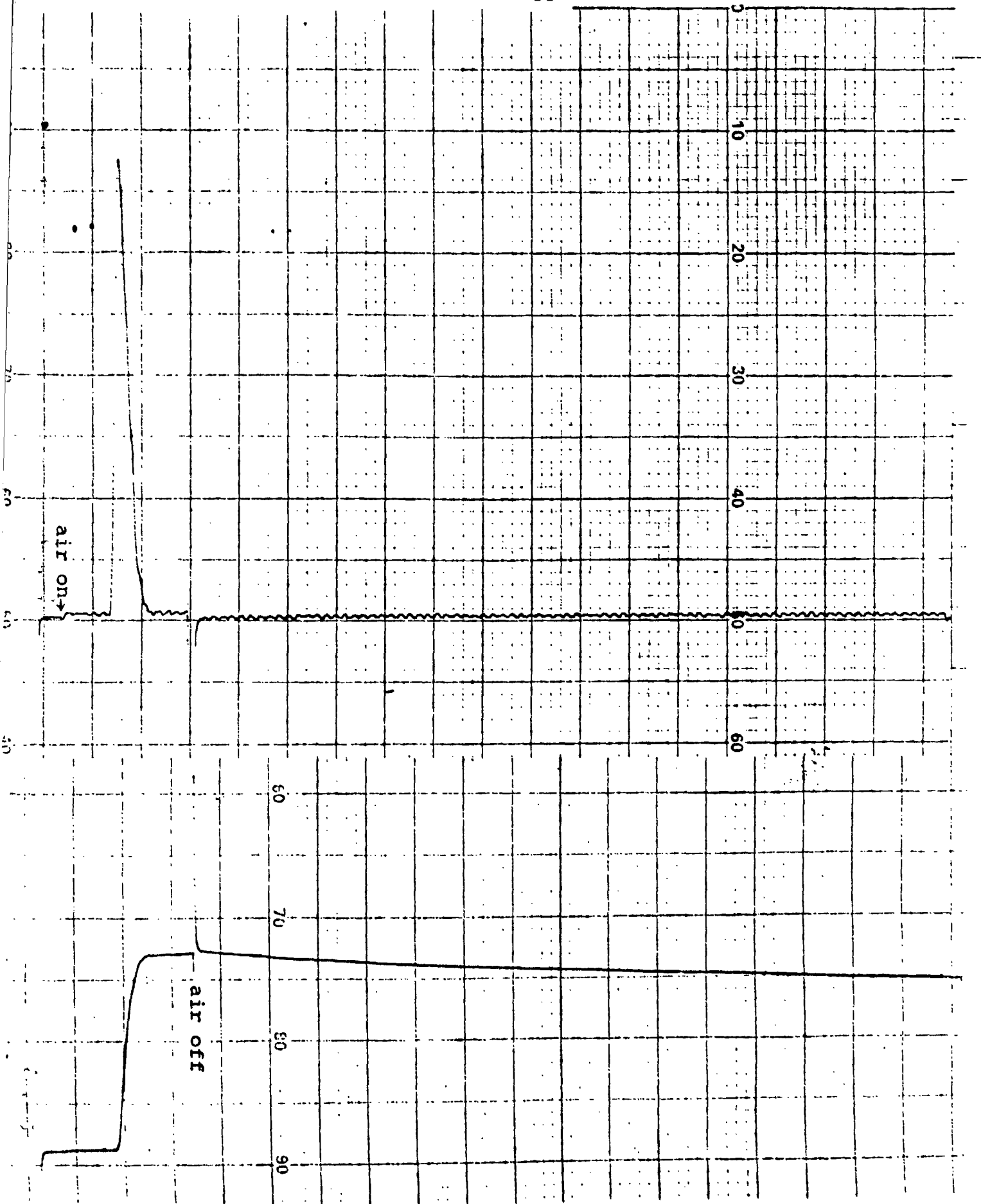


Fig.4. Pressure trace (lower plot) and derivative curve (upper plot) for a calibration chamber test (Vertical expansion 4X for the pressure, 16X for the derivative)

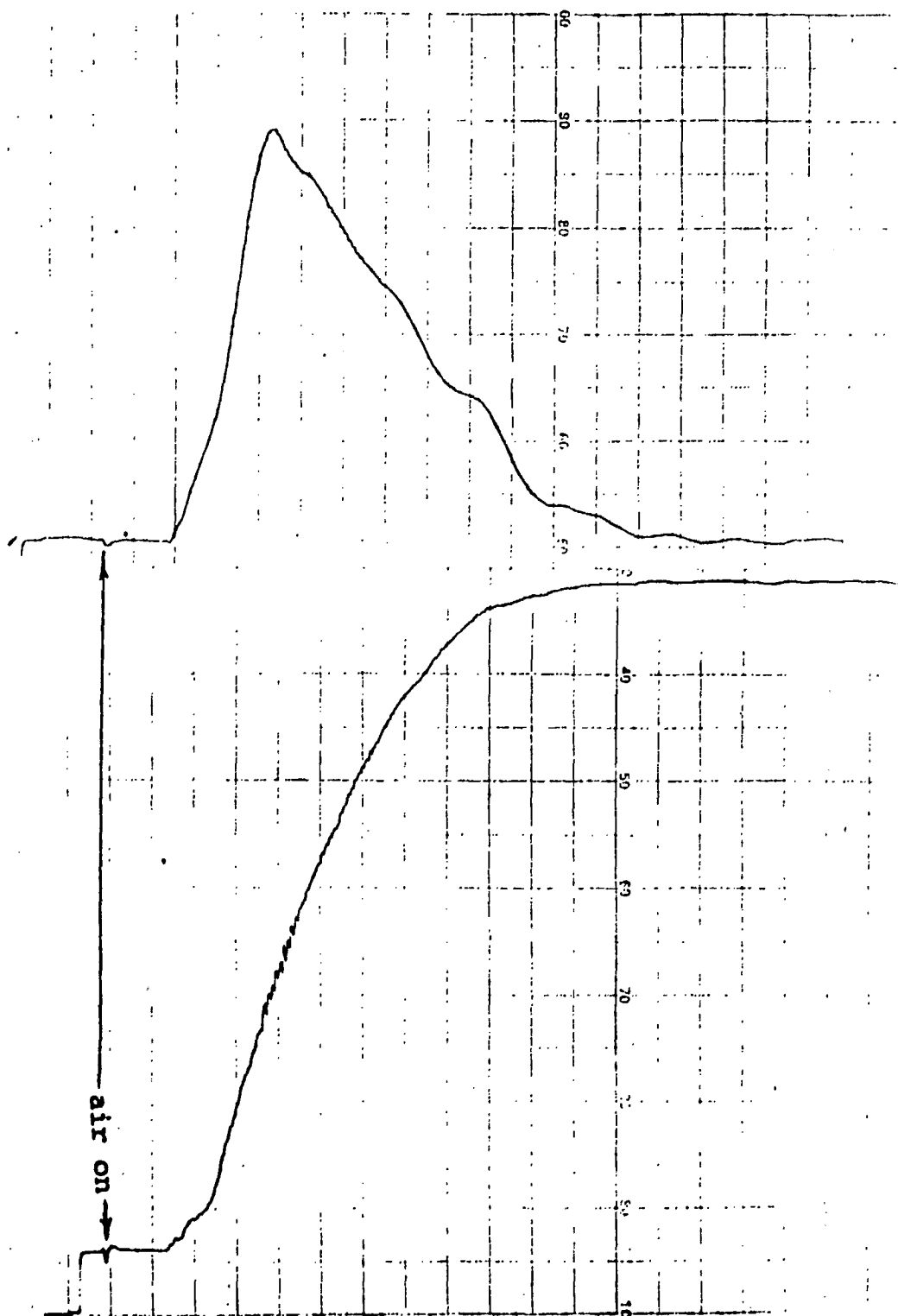


Fig. 3. Pressure trace (lower plot) and derivative curve (upper plot) for a calibration chamber test, with the vertical and horizontal expansions at 16X.

APPENDIX A

LEAST-SQUARES STRAIGHT LINE FIXED AT A POINT

The (x,y) data is fitted to the line: $y = a + bx$
 and fixed at $x_0 = .000000$ and $y_0 = .000000$
 Calibration of pressure transducer Nov.2, 1987

Name of file: cal.1

number of points = 22

i	x	y	y(calc)	y(calc)-y	% error
1	15.000000	.748000	.747620	-.000380	-.051
2	20.000000	.996000	.996826	.000826	.083
3	25.000000	1.244000	1.246033	.002033	.163
4	30.000000	1.494000	1.495239	.001239	.083
5	35.000000	1.740000	1.744446	.004446	.256
6	40.000000	1.990000	1.993652	.003652	.184
7	45.000000	2.240000	2.242859	.002859	.128
8	50.000000	2.490000	2.492065	.002065	.083
9	55.000000	2.744000	2.741272	-.002728	-.099
10	60.000000	2.990000	2.990478	.000479	.016
11	65.000000	3.242000	3.239685	-.002315	-.071
12	70.000000	3.490000	3.488892	-.001108	-.032
13	75.000000	3.736000	3.738098	.002098	.056
14	80.000000	3.984000	3.987305	.003305	.083
15	85.000000	4.234000	4.236511	.002511	.059
16	90.000000	4.486000	4.485718	-.000282	-.006
17	95.000000	4.736000	4.734924	-.001076	-.023
18	100.000000	4.984000	4.984131	.000131	.003
19	105.000000	5.236000	5.233337	-.002663	-.051
20	110.000000	5.486000	5.482544	-.003456	-.063
21	115.000000	5.734000	5.731750	-.002250	-.039
22	120.000000	5.980000	5.980957	.000957	.016

y-intercept "a"	=	.000000
standard deviation of y-intercept	=	.002525
slope of line "b"	=	.049841
standard deviation of slope	=	.000007
coefficient of correlation "r"	=	.999999

100

100

100