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PROCEDURE FOR DUST EXPLOSION TESTS USING THE 20L VESSEL

K.J. MINTZ

CANADIAN EXPLOSIVE ATMOSPHERES LABORATORY

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by

K.J. Mintz*

ABSTRACT

The 20 L vessel is becoming recognized as the most practicable apparatus for studying dust explosions in the laboratory. One has been installed and commissioned in the Canadian Explosive Atmospheres Laboratory. A detailed procedure for using it and associated equipment is provided in this report.

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

KEYWORDS: Explosions, Dust, 20 L vessel

MARCHE A SUIVRE POUR LES ESSAIS SUR LES EXPLOSIONS DE POUSSIERE A L'AIDE DE LA CHAMBRE DE 20 L

par

K.J. Mintz*

RESUME

La chambre de 20 L est de plus en plus reconnue comme l'appareil le plus pratique pour étudier les explosions de poussière en laboratoire. Un modèle a été installé et mis en service au laboratoire canadien des atmosphères explosives. Une marche à suivre détaillée concernant son installation et celle du matériel connexe est décrit dans le présent rapport.

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*Chercheur, Laboratoire canadien des atmosphères explosives, Laboratoire de recherche minière, CANMET, Energie, Mines et Ressources Canada, Ottawa

MOTS CLES: Explosions poussiére, chambre de 20 L

CONTENTS

INTRODUCTION

The 20 L vessel has been installed in the dust explosion laboratory and is now being used as the "standard" apparatus for dust explosion studies at CEAL. In order to ensure that this apparatus is used safely and that the results obtained are meaningful, this report provides detailed instructions. It is strongly recommended that a user of the apparatus study the following section and give himself the self-test provided in the Appendix.

SAFETY CONSIDERATIONS

The dust explosion tests involve use of compressed gas cylinders, a high pressure chamber, various electronic equipment, high voltage, pyrotechnic ignitors and most important, handling explosible dusts and creating an explosion. There is little hazard when a technically competent person follows the instructions herein. Deviation from the instructions or use of the apparatus by an untrained person may damage the equipment or cause serious injury. The operator should not be disturbed or allow himself to be distracted during the test. Permission to use the apparatus for a specified set of experiments must always be obtained from the author of this report.

Wearing safety glasses with side shields is compulsory when conducting these tests. A laboratory coat is highly recommended. Safety shoes are recommended when moving gas cylinders, the 20 L vessel or any other heavy equipment. Open-toe shoes are not allowed in the laboratory. Ear protection is recommended during sieving.

Any testing of self-explosive dusts is currently carried out in the Hartmann apparatus at CERL; no self-explosive dust is permitted in the CEAL

dust laboratory (with the single exception of the chemical ignitor), because CEAL does not have a licence from the Explosives Branch to store or use explosives.

Generally, only dusts with a relatively low toxicity will be tested. Nevertheless, eating is not permitted in the laboratory and thorough washing of hands is recommended before eating. Disposable vinyl gloves are kept in the laboratory and can be used to prevent any skin irritation. Dust should be vacuumed up regularly. If the dust is explosible, the storage compartment of the vacuum cleaner should be checked to ensure that it is moist.

Often the gaseous products of combustion are toxic. The operation of the fume hood should be checked before testing. The 20 L vessel is pumped out after each test; the exhaust of the pump is connected to the duct of the fume hood. Hence, the fume hood fan should be on and the fume hood doors closed during this procedure.

The chemical ignitors require special mention, because they fall under the Explosives Act. They are manufactured by Fr. Sobbe, West Germany, and purchased from Cesana Corp., Verona, N.Y. Before purchasing them, a special permit must be obtained from the Explosives Branch. The conditions of the permit are that they must be kept inside Building 9, in a locked wooden box inside a cabinet. Only trained laboratory personnel are to have access to them. Upon receipt, the ignitors must be wrapped individually in aluminum foil and the exposed leads twisted together, to prevent any possibility of static discharge accidentally igniting them. They should be held with the plastic cap pointing away from anyone.

The high-voltage system used to create a discharge for ignition of the dust-air mixture can also be dangerous. It should be checked on a daily basis to ensure that all connections are sound and that there are no frayed

wires.

The last operation to be done with the lid of the vessel open is the connection of the ignition source, to prevent any possibility of the ignition of a dust cloud.

The control panel was designed to require the use of both hands on it during the ignition to reduce the chance of exposure to high voltage. No attempt should be made to defeat this safety feature.

EQUIPMENT

20 L vessel

Explosion control panel

Nicolet 4094 digital oscilloscope with 4562 module and XF-44 dual disk drive

Strain-gauge control unit

CEC 1000 or 5000 strain-gauge pressure transducer

16 L air pressure chamber

Air cylinder (extra dry grade, containing 20.9% oxygen)

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

Balance, accurate to 0.1 mg

Oven

Desiccator

Ro-Tap sieve shaker

Beckmann oxygen analyzer (modified) with transfer pump

Infrared spectrometer (optional)

Vacuum pump

Tungsten electrodes or Sobbe chemical ignitors

SAMPLE PREPARATION

Note: The method of preparation of a particular sample may differ from the general 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 screen are all completely 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 Ro-Tap timer on for 10 minutes. The hammer of the Ro-Tap should be tapping while the sieves are being shaken.
   (f) If, after that time, insufficient material has passed into the collector pan, turn the shaker on for a longer time.
2. Place the -200 mesh fraction in a glass beaker and heat in an oven for 24 hours at 80°C.
3. Clean the sieves thoroughly by washing in hot soapy water, rinsing and drying.
4. Remove the sample from the oven and place it in the desiccator.

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

20 L VESSEL SET-UP

1. If not already there, the 20 L vessel should be placed in the fume hood.
2. Using the pulley, raise the lid to the top of the fume hood. Lock the pulley rope.
3. Clean the inside of the vessel if necessary.
4. Check the pressure transducer to ensure that it is clean, and screw it into its fitting on the vessel. Attach its cable.

   Note: Dust often accumulates inside the fitting. If this dust is not removed, the measured rate of pressure rise will be significantly lower than it should be.

5A. If electrodes are to be used, grind the ends to be needle-like, but do not install them in the vessel yet.

6. Weigh out the required sample on a polystyrene weighing dish and return the sample container to the desiccator.
7. Pour the sample carefully into the sample holder on the bottom of the 20 L vessel.
8. Screw the nozzle into place.

9A. If electrodes are used, install them and adjust their spacing using the 6.4 mm brass spacer.

9B. If the Sobbe chemical ignitor is used, place it in its holder facing downwards and attach both wires to the leads from the control panel, making sure that there is good electrical contact.

10. Lower the lid gently onto the main part of the 20 L vessel and tighten all the bolts with the wrench.
11. Evacuate the vessel by turning on the vacuum pump and opening the

valves with the blue and red handles.

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 mainframe:
   (a) "AUTOCENTER" toggle switch should be up;
   (b) the two "EXPANSION" switches should be at "OFF";
   (c) the "FUNCTION" switch should be at "RESET NUM";
   (d) "Y/T" should be selected;
   (e) the "MEMORY" should be at "H1".

   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 unit:

   (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 10μS and turn "AUTO/NORM" toggle switch to "AUTO".

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

6. The trace should be at 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 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 ± .02 V. Turn the toggle switch back to the centre position. If the voltage reading is incorrect, repeat steps 4 - 7. If problem persists, adjust the "SENSITIVITY" carefully.

9. On the 4562 module, turn the "TIME PER POINT" to 500μS and the "AUTO/NORM" toggle switch to "NORM".

10. 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.

(b) Turn "FUNCTION" switch to "DATA MOVE";

(c) Press "TRIGGER POSITION A" to right (crosshair will move to right) until the screen reads -600 ms.

Note: The "pretrigger" is now set up so that the 'scope will store and display 600 ms of pressure trace prior to the triggering caused by the pressure rise. The delay light indicates that the pretrigger is armed.

(d) Press "HOLD LAST" button to exit the set-up mode.

(e) Turn "FUNCTION" to "RESET NUM".

11. Insert the 4094 program diskette into the left disk drive. "P01" (which means program no. 1) will be displayed.

12. Press "UP" until display shows "P29".

13. Press "RECALL", which transfers this program into the mainframe. The screen will display "TITLE".

14. Write the title (name of sample, concentration and date):

(a) Turn the "FUNCTION" switch to "PROGRAM";

(b) Press "EXECUTE";

(c) Use the vertical arrow keys to scroll through the characters and the horizontal arrow keys to move along the title line;

(d) To erase a character, press the "AUTOCENTER" switch downwards;

(e) Press "EXECUTE" to leave the program;

(f) Turn the "FUNCTION" switch back to "RESET NUM".

Note: Steps 12 - 14 can be used to edit a title that is already on the screen, which is much easier than starting from a blank.

15. Insert a formatted diskette into the right disk drive.

EXPLOSION TEST

1. Turn on the control panel. Ensure that the solenoid lead is plugged into the proper outlet on the left side of the control panel. Set "DELAY" to "ARC".

   Note: This means that the ignition will occur after the air has dispersed the dust.

2. Set "AIR ON TIMER" to 310 ms (= t_s).

   Note: This is the time that the solenoid valve is open, and is intended to pressurize the vessel to exactly one atmosphere absolute. For high concentrations of dust or for heavy dusts, a longer time may be required.

3. Set "DELAY" to 410 ms (= t_d).

   Note: This time is the total elapsed time from the start to the ignition. Therefore it represents a delay of 100 ms (410 - 310) from the closing of the "air" solenoid. If the "AIR ON TIMER" is increased (see note above), the "DELAY" time should also be increased to maintain the 100 ms difference. The only exception is for tests to measure the effect of turbulence.

4A. For tests carried out using an electric discharge, ensure that the transformer lead is plugged into the " TRANSFORMER" outlet on the left side of the control panel.

4B. For tests carried out with the chemical ignitor, ensure that the its lead is plugged into the "CAP.DISCHARGE/IGNITOR" outlet, and that the toggle switch on the left side of the control panel is set to

"IGN".

5. Check that the pressure gauge on the manifold from the vacuum pump is reading below 100 kPa. Close the red plug valve to vacuum. Turn "TRACE" on control unit to "NORM" and adjust "BALANCE" until the voltage on the 'scope is between -10 and +10 mv. The voltage should oscillate over a range of 4 mv.

   Note: The balance adjustment is very sensitive; turn it slowly.

6. Press "EXECUTE" on 4094 to zero the voltage at zero pressure absolute.
7. Fill the air chamber with dry compressed air to a pressure of 1100 kPa. Close inlet valve to the air chamber.
8. Press "LIVE" and "HOLD NEXT" on the 4562 module.
9. While pressing "ENABLE" button, move toggle switch to "RUN". The pressure in the air chamber should drop to 960 kPa, the "HOLD LAST" button should light, the other two buttons should go off, and a pressure trace should appear on the screen. A bright flash will appear through the windows in the 20 L vessel if either the chemical ignitor is used or an explosion occurs.
10. Press "STORE" on the disk drive.
11. After waiting for a few minutes for the vessel to cool, measure the post-explosion oxygen concentration:
    (a) Check the zero and span on the analyzer using pure nitrogen and the 50% oxygen/nitrogen mixture;

    Note: Normally, this need be done only 1 - 2 times per day.

    (b) Measure and record the pressure in the vessel, P_p;
    (c) Turn on the transfer pump;
    (d) Wait until the flowmeter beside the oxygen analyzer indicates that the flow rate has dropped below 20 on the scale;

(e) Turn on the fume hood fan;

(f) Slowly open the Whitey valve on the 20 L vessel and adjust the flow rate through the flowmeter to 100;

(g) The digital display on the analyzer will not show any change for a couple of minutes and will take several minutes before it reaches a stable value;

(h) Close the Whitey valve.

(i) Apply the correction factor to the displayed reading and record that value as the $\%O_2$.The fraction of oxygen consumed is $1 - P_p/P_a * \%O_2/20.9$. (P_a is determined below.)

12. (optional) Analyze other gases by the infrared spectrometer:

(a) Open the green plug valve to thoroughly evacuate the line to the spectrometer and the cell itself;

(b) Close the blue plug valve (to the pump), open the red plug valve (to the 20 L vessel) which will allow expansion of the contents of the 20 L vessel to the connecting line and the spectrometer.

(c) Operate the spectrometer according to its directions.

13. Open the blue plug valve to evacuate the 20 L vessel completely (several minutes), then close the red plug valve.

14. Open the black plug valve to allow the vessel to reach atmospheric pressure.

15. Remove the bolts and use the pulley to raise the lid. Lock the pulley rope. After a couple of minutes, turn off the fume hood fan.

16A. If electrodes were used, unscrew the holders together with the electrodes.

16B. If a chemical ignitor was used, detach the lead wires and discard.

17. Clean the inside of the vessel starting with the lid, using first the vacuum cleaner then some Kimtowels.
18. Remove the nozzle and use the vacuum cleaner to clean the dust chamber.
19. Turn the strain-gauge control unit off, detach the cable from the transducer, remove the pressure transducer and clean gently but thoroughly with a small brush.

ANALYSIS OF THE DATA

1. Move the cursor to approximately the beginning of the first pressure rise; turn the horizontal and vertical expansions to X8, then set the cursor exactly at the beginning of the rise, and press "execute" to zero the time and voltage .
2. Move the cursor to the right until it corresponds to the end of the first pressure rise. Record the time display, t_a, which should be about 310 - 340 ms. Multiply the voltage by the calibration factor of the pressure transducer (which was 136.6 kPa/volt for the CEC 1000 (ser. no. 24176) and 131.1 kPa/volt for the CEC 5000 (ser no. 5848) at the time of writing), and record that value, P_a, the pressure due to the air used for dispersing the dust. (The subscript "a" refers to air.)

   Note: P_a should be about 100 kPa. If it is less than 95 kPa, the dust being dispersed is probably restricting the flow of air too much and therefore the time that the solenoid is open should be increased.
3. Press "EXECUTE" to zero the time and voltage. Move the cursor to the beginning of the second rise. Record the time, t_i, which should

be about 100 ms, and the pressure, P_i, which should be 0 - 2 kPa less than that determined in step 1. (The subscript "i" refers to ignition.)

4. Press "EXECUTE" to zero the time and voltage. Move the cursor to the maximum. Record the time, t_m, and pressure, P_m. (The subscript "m" refer to the maximum.)

5. If the chemical ignitor was used, it is necessary to carry out the following steps to subtract off that portion of the pressure trace due to the ignitor.

   (a) Turn the "MEMORY" switch to H2.

   (b) Insert the diskette containing the pressure trace due to the ignitor alone. ("CAL8" - track 5, H1)

   (c) Press "UP" on the disk drive until the display reads "05".

   (d) Press "UP" and "DOWN" simultaneously; the display will read H1.

   (e) Press "RECALL" to transfer the pressure trace of the ignitor into the H2 part of the mainframe memory. The title on the screen should read "CHEM IGNITOR 18-5-88".

   (f) Press "UP" and "DOWN" simultaneously, then press "UP" until "P14" appears. Press "RECALL" to transfer the "horizontal shift" program into the mainframe.

   (g) Turn the "FUNCTION" switch to "PRGM" and press "EXECUTE".

   (h) Move the cursor to the beginning of the second peak; turn horizontal expansion to X8 to locate cursor exactly; press "EXECUTE".

   (i) Turn the "MEMORY" switch to H1 (the screen will display the pressure trace just obtained); move the cursor to the beginning of the second peak; press "EXECUTE". The pressure trace will be

transferred slightly. Turn "MEMORY" to all to ensure that the two curves displayed start the second peak at the same time.

(j) Turn "MEMORY" to H1; turn "AUTOCENTER" off; turn "MEMORY" to H2; turn "FUNCTION" to "DATA MOVE"; turn horizontal expansion off; press "EXECUTE". The pressure trace will move. Turn "MEMORY" to "ALL" to ensure that the two traces are lined up.

(k) Turn "MEMORY" to H2; turn "FUNCTION" to "SUB"; press "EXECUTE". The trace that appears to the screen is H1 - H2, and is the one to be used for the analysis.

(l) Remove diskette, insert the original diskette, press "UP" until the "RECORD" shows the track used for storing the uncorrected pressure trace. Press "STORE".

(m) Turn the "FUNCTION" switch to "RESET NUM"; press "EXECUTE".

(n) Turn horizontal and vertical expansion to X8; move the cursor to the peak maximum and record time, t_{mc}, and pressure, P_{mc}. ("mc" means maximum, corrected)

6. Determine the maximum rate of pressure rise:

(a) Press "UP" on the disk drive until "P10" appears. Press "RECALL" to transfer the derivative program to mainframe.

(b) Turn "FUNCTION" to "PRGM"; press "EXECUTE" to start program; follow instructions on screen.

(c) Set the "window" size by using the cursor buttons on the mainframe: 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. It is recommended that a window size of 20 be used for most tests.

(d) For speed, set the start position just before the pressure starts to rise, and set the stop just after the maximum.

(e) After the 'scope has generated the derivative curve, turn the vertical and horizontal expansions as required and move the cursor to the maximum of the peak. Multiply by the transducer calibration factor to obtain $(dP/dt)_m$, the maximum rate of pressure rise.

(f) Turn the "MEMORY" switch to H2 (H1 if the chemical ignitor was used); press "EXECUTE" to zero the time; move the cursor to the beginning of the peak. The time from ignition to the maximum rate of pressure rise, t_r, is the absolute value of the time display.

7. (Optional) Make a hard copy using the HP Color Pro Plotter.

(a) Display the desired curve, or portion thereof, on the screen. (Use horizontal and vertical expansions and move the cursor as desired.)

(b) Turn the plotter on (upper left white key); place the plotter paper on the platen so that it touches the bottom of the white strip and press the load key (the white key just below the on/off key).

(c) Press the pen selector key (bottom row, second from left) to load the pen. Hold down that key to change pens.

(d) Turn the "FUNCTION" switch to "PEN"; press "EXECUTE". The title and axes will be drawn.

(e) Press "EXECUTE". The parameters of the coordinate system will be written first:

V/D - volts/division (Y-axis)

V_Y - value of Y at the graph origin

T/D - time/division (in seconds)

T_L - time at the graph origin

NOTE: If the EXPANSION is off, V/D will be 1 V (equivalent to 136.6 kPa for the CEC 1000 pressure transducer), T/D will be 0.5 s, T_L will be -0.6 s.

(f) If it is necessary for a part of the curve to be omitted, press the pen up key (upper right) to raise the pen. (The pen will continue to follow the graph, but will not be in contact with the paper.)

(g) Press load button to put the pen away; the paper can then be removed.

NOTE: If more than one graph is desired on the same paper, first place all graphs (up to four) in the memory. After completing the first graph, turn the "MEMORY" switch to the other graph and press "EXECUTE".

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The writer would like to thank Mr. Ken Judge for his assistance.

APPENDIX: SELF-TEST

1. What safety equipment is required to be worn when conducting dust explosion tests?
2. Can self-explosive dusts, such as TNT, be tested in the 20 L vessel in CEAL?
3. Where do you get permission to use the 20 L vessel for dust explosions?
4. Why is the 20 L vessel used inside the fume hood?
5. What piece of equipment in the dust lab falls under the Explosive Act?
6. Why should the fume hood fan be on when the vacuum pump is evacuating the vessel after a test?
7. Why do you connect the ignition source just before closing the lid of the 20 l vessel?
8. Why is the control panel designed to require the use of both hands?

Answers are in the text.