Ignition testing work instructions

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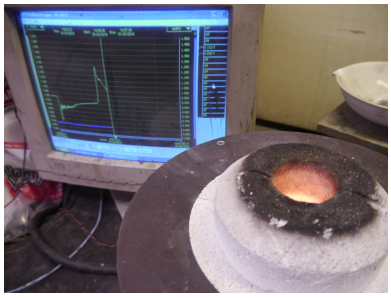
Introduction

Ignition testing provides a quick and easy way to compare exothermic formulations. Specifically, we use this test to compare various Exactcast sleeve formulations to one another. The test involves taking a small piece of the sleeve material, igniting in a high temperature furnace, and monitoring temperature over time. This allows calculations of various key points of interest, namely: time to ignition, maximum temperature increase, and exotherm duration.



In a quality control setting where quick testing is desired, a technician needs only: a high temperature furnace, infrared thermometer, and stopwatch. A stopwatch would be started upon sample insertion and then stopped when the exothermic reaction is noticed. Maximum temperature would then be recorded to ensure specifications are met. In scenarios where, for example, formulary comparisons are taking place, it is beneficial to use additional analytical tools such as a data logger and analytical software to further interpret and present data.

The following procedure outlines sample preparation, data logging, data interpretation.

Safety equipment required

- **Standard laboratory safety attire** *e.g.* eye/ear protection, steel toes, etc.
- **Insulating gloves** or heat resistant gloves may be beneficial when handling samples.
- **Welding glasses** shade 5 or greater may provide relief from the incandescence of the sample and oven crucible.
- **Fume hood** must be used with high temperature furnace.

Testing equipment required

1. **Drill press** for creating sample 'plug' to be inserted into furnace[1].
   1.1 **Diamond hole saw** 1-inch diameter.
2. **Straight crucible tongs**[2] for sample insertion into narrow crucible, ~10" length.
3. **High temperature furnace** Harrop, model CF-14 crucible furnace.

[1] Plugs can also be created using a suitable mold

[2] Tongs often have an indentation along the length to aid holding vials, etc. In our case, the indents may prevent sample insertion. Purchase new or modify with a hammer.

3.1 **Heating element**[3] Orton Ceramics, I6-2401.

3.2 **Thermocouple** Orton Ceramics, I6-TC-CF1S.

3.3 **Alumina crucible**[4] Coors Ceramics Co., part #65539: 100mL, 31mm OD x 91mm tall, 110g-120g.

3.4 **Alumina bubbles, 14mesh** Washington Mills Electro Minerals Corporation.

3.5 **Alumina disc** Coors Ceramics Co., part #65640, 32mm x 2mm.

4. **Temperature control unit** Harrop, model UTC-240 with Honeywell controller and 20 amp meter.

5. **Infrared thermometer** Cole Parmer, model #39800-47, operating range 200-1800C.

   5.1 **Ring stand with clamp attachment** or other suitable suitable stand to support the thermometer above the high temperature furnace.

6. **VirtualBench data logging software**[5] National Instruments, part #777197-01.

   6.1 **PC-LPM-16/PnP multifunction I/O board**, allows thermometer to communicate with software.

7. **Cooling rack**, capable of holding materials ~1400C, refractory sheet or large crucible is generally suitable.

8. **Spoonula** Fisherbrand Spoonulet lab spoon or similar, used to scrape sides of crucible and remove ash from within.

9. **R and RStudio** are used to calculate key points from collected data[6].

   9.1 **R** can be downloaded here: `https://cran.r-project.org/`

   9.2 **RStudio** can be downloaded here: `https://www.rstudio.com/`

[3] Heating element must be wrapped in insulation, specify when ordering.

[4] The alumina crucible acts as the sample receptable within the furnace. A small amount of alumina bubbles cover the bottom of the crucible and support the alumina disc, roughly 1/2 inch from the bottom of the crucible.

[5] Likely out of distribution, though any data logging software should suffice. May need to consult with NI or similar for equivalent software.

[6] Optional software. Although other programs like Excel can be used, using R and RStudio dramatically speeds up the process.

Furnace preparation

The temperature controller is preprogrammed to ramp temperature from 0 to 1000C over approximately 2 hours. If the controller requires reprogramming, see appropriate manual[7].

[7] Manual is stored by the equipment.

To begin the temperature ramping sequence press the following buttons on the temperature controller:

1. Power on, let controller run through test.
2. Press `Lower display` until `SP` is displayed.
3. Lower to `0` by using the `Up` and `Down` arrow buttons.

4. Press `Run/Hold`.
5. Press `Manual/Auto`.
6. Press `STBY/Run`.
7. After ramping sequence, ensure ignition furnace is at target temperature of 1000C.
8. Remove insulating crucible lid.
9. Ensure IR thermometer is plugged in, trigger is locked on, and aiming at the center of furnace crucible.

Sample preparation

See drill press manual for safe operating procedures

Tensile testing almost always takes place alongside ignition testing. For this reason we utilize the left over dogbone sample to prepare the ignition testing plug.

1. With the 1" hole-saw, drill a hole into the dogbone sample.
2. Collect the cylindrical sample from the hole saw[8], label the sample.

[8] Aggressive tapping may be required, use a metal rod or similar.

Ignition procedure

1. With the VirtualBench software open, give the sample a filename:

   1.1 Navigate to *File -> Logger settings*, or press *Ctrl+E*.

   1.2 Press the *File Config…* button at the top right.

   1.3 In the top left text box enter the file name using the following convention: *e.g.*, *2017-12-11,93A-1*, starts with the date, then lab notebook page of the experiment, sample letter, then a dash followed by the replicate number. The sacrificial plug is generally given a filename ending in '0', *i.e. 2017-12-11-A-0*.

   A sacrificial plug is burned before actual test samples so ambient furnace temperatures are comparable from one test sample to the next.

2. Press start
3. Allow approximately 10-seconds to pass so a temperature baseline is established.
4. Insert sample into furnace crucible.
5. After measurement is complete, remove sample from furnace, set on cooling rack to be discarded later.
6. Allow approximately 60-seconds to pass for furnace to return to baseline temperature once again[9].
7. Repeat steps 1-6 with each sample to be tested. Randomizing samples will help minimize potential error in the test method.
8. After all testing is completed, residue must be scraped from the crucible walls and then scooped from the bottom and discarded. This may also need to be done after burning several plugs, depending on the total sample size[10].

[9] Temperature equilibration time *must* remain consistent between samples to allow for a comparable *duration of exotherm* measurement. The time itself is less important than the consistency between samples. This is the reason for starting measurements with a sacrificial sample.

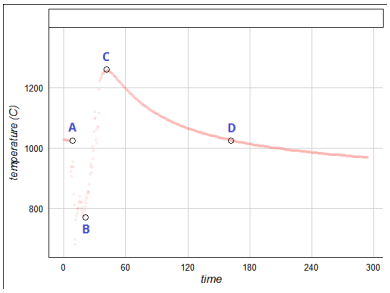
[10] Remember to use a sacrificial plug to equilibrate temperatures if scraping the crucible between samples.

Data analysis

As mentioned above, R and RStudio are optional but recommended. We can use the program to quickly and conveniently calculate our values of interest from the produced temperature vs time graph. In the event that R/RStudio are not used, the below points can be calculated quite simply using Excel or even pen and paper.

Time to ignition

Is the time required for the sample plug to ignite after being inserted into the furnace. On the graph: *time B - time A*.



Delta temperature

Is the maximum temperature subtracted from the baseline furnace temperature. On the graph: *temperature C - temperature A*.

Exotherm duration

Is the time, after ignition, that the recorded temperature is above the baseline temperature. On the graph: *time D - time B*.

Using R and RStudio

1. Install R.
2. Install RStudio.
3. Copy the *ignition_newshit*[11] folder from the `X:\ASUS\Dublin\cas-j\0 - Yu\01 R Scripts` directory to your *My Documents* folder.
4. Open the `launcher.R` script from the folder.
5. Change the `work.dir` variable on line 17 to match the location your placed your folder, above. Make sure to follow the double forward slash convention.
6. All measured data are placed in a subfolder of the `Logfiles` directory, named based on date of measurement and lab notebook number. For example, The folder `2017-03-15,91` contains the logfiles: `2017-02-14,87A-1.log`, `2017-02-14,87A-2.log`, `2017-02-14,87A-3.log`, etc.[12]
7. With all logfiles in the proper subfolder, copy and paste the folder name to the `prefix` variable, located on line 22.
8. Run the code by pressing `Ctrl + Shift + R`. The code will now run calculations on all data in the designated folder.
9. A summary graphic will be generated and exported to the *!Output* folder of your working directory. It will be named automatically based on the subfolder name.

[11] Pronounced *"news-hit"*

[12] Maintaining these naming conventions is important, the script depends on it. See the example folders and .log files for proper naming convention.

Appendix

Heating element replacement

The element fits within the furnace and connects to two leads on the outside of the furnace. Power needs disconnected, top plate removed, wires disconnected from terminal blocks, and old heating element removed.

Testing equipment required: 3.1

1. Unplug from power source.
2. Remove the top plate.
3. Remove wires connecting element to terminal.
4. Remove old heating element if replacing.
5. Insert new heating element.
6. Reconnect wires to terminal.
7. Reattach top plate to furnace.
8. Reconnect power.

Thermocouple replacement

Testing equipment required: 3.2

Will need to remove the top plate and crucible to ensure thermocouple is inserted at correct depth.

1. Disconnect power.
2. Remove top plate, crucible, and crucible resting pad.
3. Lay furnace on side to access thermocouple.
4. Disconnect thermocouple wire from thermocouple.
5. Unscrew and remove thermocouple from furnace, take note of thermocouple depth inside furnace.
6. Replace with new thermocouple at same height, should be at same height as the alumina disc.
7. Reassemble furnace, reconnect power.

Crucible replacement

The crucible sometimes cracks due to high fluctuations in temperature. To replace, disconnect power, remove top plate, pull out crucible. Replace with new crucible, which has been filled with alumina beads and alumina disc, mindful of the bottom resting pad.

Testing equipment required: 3.3, 3.4, 3.5.

1. Disconnect power
2. Remove protective top plate by removing the 4 screws on top
3. Remove old crucible, discard

4. Prepare new crucible by padding bottom with approximately 2.0g alumina bubbles and placing alumina disc on top
5. Place new crucible package in furnace, reattach top lid