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Title: Laboratory Scale Slow Cook-Off Testing of Rocket Propellants: The Combustion Rate Analysis of a Slowly Heated Propellant (CRASH-P) Test

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Author Questionnaire

1. **Microscopy**: Does your protocol require the use of a dissecting or stereomicroscope for performing a complex dissection, microinjection technique, or something similar? **No**

2. **Software:** Does the part of your protocol being filmed include step-by-step descriptions of software usage? **Yes**

If **Yes**, we will need you to record using screen recording software to capture the steps. If you use a Mac, QuickTime X also has the ability to record the steps. Please upload all screen captured video files to your project page as soon as possible.

Authors: It was not clear which of the provide screen capture videos corresponded to the steps for which screen capture videos have been requested. Please indicate the file name to be used for each step, as well as a time stamp indicating which section of each video can be used to demonstrate the accompanying voiceover narrative. Please note that the video should match the text as closely as possible, so that viewers will understand how to perform the described actions. Please see the text for steps 4.2. and 4.4. and the shot descriptions for shots 4.2.2. and 4.4.1. as examples.

3. **Interview statements:** Considering the COVID-19-imposed mask-wearing and social distancing recommendations, which interview statement filming option is the most appropriate for your group?

☒ Interviewees self-record interview statements. JoVE can provide support for this option.

4. **Filming location:** Will the filming need to take place in multiple locations? **No**

Introduction

1. **Introductory Interview Statements**

REQUIRED:

NOTE: Interview statements should have been provided by authors to Devon

1.1. **Jonathan Essel**: The CRASH-P test can be used to quantitatively evaluate the slow cook-off reaction violence of rocket propellants on a much smaller scale than conventional testing methods **[1]**.

1.1.1. LAB MEDIA: To be provided by Authors: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

1.2. **Jonathan Essel**: The test is a less expensive and faster method for screening rocket propellants for their slow cook-off violence than current full-scale tests **[1]**.

1.2.1. LAB MEDIA: To be provided by Authors: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

1.3. **Andrew Nelson**: It is difficult to conduct full scale slow cook-off tests in the early stages of rocket propellant development. The CRASH-P test can provide much of this critical information early in the formulation process **[1]**.

1.3.1. LAB MEDIA: To be provided by Authors: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

1.4. **Andrew Nelson**: The test is fast and inexpensive enough that many of the underlying questions involving chemical kinetics of propellant ingredients during slow cook-off can be elucidated in greater detail than ever before **[1]**.

1.4.1. LAB MEDIA: To be provided by Authors: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

1.5. **Christopher Gray**: The CRASH-P test requires previous experience in thermal fluid instrumentation, as well as setting up high speed data acquisition for temperature and pressure measurements **[1]**.

1.5.1. LAB MEDIA: To be provided by Authors: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

1.6. **Christopher Gray**: A visual demonstration of this method can be helpful, as setting up the diagnostics and positioning the sample can be difficult from written instruction alone **[1]**.

1.6.1. LAB MEDIA: To be provided by Authors: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

Introduction of Demonstrator on Camera

1.7. **Jonathan Essel**: Demonstrating the procedure with Christopher Gray will be Scott Sumner, a research engineer from our laboratory **[1]**.

1.7.1. LAB MEDIA: To be provided by Authors: Author saying the above while named demonstrator looks up from workbench or desk or microscope and acknowledges the camera.

2. Propellant Sample Installation

2.1. For installation of the propellant sample, bolt the sample holder cover to the CRASH-P (crash-P) sample holder to seal the sample. Then bolt the sealed sample to the steel plank attached to the chamber cap of the CRASH-P test to keep the sample in the middle of the chamber **[1]**.

2.1.1. WIDE: Talent attaching CRASH-P sample to the steel plank OR Talent checking that plank is secure to chamber cap. *Videographer: This step is important*

2.2. Place one of the thermocouples from the electrical feedthroughs inside the propellant sample holder to capture any exothermic reactions **[1]**. Place another thermocouple on the steel plank pointed up to record the air temperature inside the CRASH-P chamber **[2]**. Place a small amount of RTV where the thermocouple enters the propellant sample holder to seal the orifice **[3]**.

2.2.1. Talent placing thermocouple inside the sample holder. *Videographer: This step is important!*

2.2.2. Talent placing thermocouple on the steel plank. *Videographer: This step is difficult and important!*

2.2.3. Talent sealing thermocouple to sample holder with RTV.

2.3. Once the sample has been secured, slide the chamber cap into the body of the chamber **[1-TXT]**.

2.3.1. Talent sliding chamber cap into chamber body. **TEXT: Mark but do not rotate chamber cap**

2.4. Use a cylindrical rod to completely thread and tighten the retaining head onto the chamber **[1]** and use a pitch of 9 threads per inch set screws hex bolt to install the 7/8 inch-diameter screws into the chamber head **[2]**.

2.4.1. Talent threading the retaining head onto the chamber.

2.5. Tighten the bolts in a to ensure that the chamber is evenly secured and use a torque wrench to ensure a uniform sealing **[1]**. Install the chamber retainer clamps, holding clamps in place with dowel pins **[2-TXT]**.

2.5.1. Talent using torque wrench to tighten screws on chamber.

2.5.2. Talent installing retainer clamps and/or dowel pins **TEXT: Use rubber mallet to ensure snug clamp fit as necessary**

2.6. To install the chamber end plate, bolt the plate to the testing table to prevent axial

movement of the CRASH-P test during an ignition event **[1]**.
2.6.1. Talent bolting plate.

2.7. Then plug the dynamic pressure sensor co-axial cables into the dynamic pressure sensors **[1]** before plugging the electrical band heaters into the outlet sockets that connect to the temperature controllers **[2]**.
2.7.1. Talent plugging in cables
2.7.2. Talent plugging in electrical band heaters.

3. Test Instrumentation Setup

3.1. Turn on temperature control panel and temperature controller. Cycle through the menus to make sure everything is in its default setting **[1]**.
3.1.1. Talent cycling through the temperature controller menu.

3.2. Make sure the inlet and outlet wires are plugged into the dynamic pressure signal conditioner **[1]** and turn on the conditioner **[2]**.
3.2.1. Shot of inlet and outlet wires plugged into condition
3.2.2. Talent turning on the dynamic pressure signal conditioner.

3.3. Set the temperature values on the temperature controller as needed for the 16 time intervals **[1]**, using the first three intervals to set up a ramp and soak period at 50 degrees Celsius for 2 hours **[2]**.
3.3.1. Talent setting values, with monitor visible in frame
3.3.2. SCREEN: To be provided by Authors: Ramp and soak period being set NOTE: Footage not uploaded to AWS yet

3.4. Enter the remaining intervals to supply the data points for the test to obtain a linear heating profile that does not change slope during the test and set the final temperature to 350 degree Celsius **[1]**.
3.4.1. SCREEN: To be provided by Authors: Intervals being entered NOTE: Footage not uploaded to AWS yet

3.5. Using three K-type thermocouples, confirm that the amplifier is turned on and turn on the monitoring camera for the test to record the CRASH-P test by video **[1]**.
3.5.1. SCREEN: To be provided by Authors: Amplifier being turned on and CRASH-P test being recorded. NOTE: Footage not uploaded to AWS yet

3.6. Turn on the electric power to the heaters on the control console **[1]** and turn on the temperature controller to run the test remotely **[2]**.
3.6.1. Talent turning on the heaters.
3.6.2. Talent turning on temperature controller.

3.7. From the **Control** page of the temperature controller set the **RSEN** *(pronounce 'R-E-S-N')* On. Then press the **auxillary** button to change the test condition from **standby** to **run** to start heating the chamber **[1]**.

3.7.1. SCREEN: To be provided by Authors: Temperature for the test condition being set. NOTE: Footage not uploaded to AWS yet

4. Data Acquisition and Test Cleanup

4.1. For data acquisition, in the data acquisition system software **[1]** set one test data collection region to allow measurement of the pressure by the main board that measures dynamic pressure and another to allow measurement of the temperatures by the thermocouple amplifier **[2]**.

4.1.1. WIDE: Talent at computer, opening data acquisition software, with monitor visible in frame

4.1.2. SCREEN: To be provided by Authors: Data collection regions being set. NOTE: Footage not uploaded to AWS yet

4.2. Set the system to run on a triggered sweep mechanism so that, after a threshold voltage is reached, the pressure sampling rate changes from one sample/second to 50,000 samples/second **[1]**.

4.2.1. SCREEN: CRASHP Sampling and Trigger Control: 00:33-00:48 *Video Editor: please speed up*

4.3. As data acquisition does not stop on its own, periodically check the test for temperature exotherm or triggered pressure responses **[1]**. If either response is observed, manually stop the recording **[2]**, and turn off the heater, video, and temperature controller powers **[3]**. Authors: Do you want to show this optional step or should we leave this information for the manuscript?

4.3.1. Talent checking the data acquisition for any triggered response.

4.3.2. SCREEN: To be provided by Authors: Shot of exothermic triggered reaction, then stop button being clicked. NOTE: Footage not uploaded to AWS yet

4.3.3. Talent turning off the instruments.

4.4. At the end of experiment, manually export the temperature and pressure data into individual, tab delimited text files to an appropriate computer for their downstream analysis **[1]**.

4.4.1. SCREEN: CRASPH Data Export: 00:13-00:30 *Video Editor: please speed up*

4.5. Allow the test chamber to cool for at least 12 hours before venting. The chamber is carefully disassembled to release any product gases from the exothermic reaction **[1]**.

4.5.1. Talent disassembling and venting the chamber.

4.6. Capture any sample container fragments of the sample holder before cleaning the chamber **[1]**.

4.6.1. Fragments being captured after test.

Results

5. Results: Representative Combustion Rate Analysis of a Slowly Heated Propellant Test

5.1. Here temperature traces for the inside chamber air and internal propellant temperature acquired by the data acquisition system can be observed **[1]**. Minor exothermic reactions before the ignition were measured **[2]** along with the main exothermic reaction **[3]**.

5.1.1. LAB MEDIA: Figure 5A

5.1.2. LAB MEDIA: Figure 5A *Video Editor: please emphasize black data line from 0 to just after 0 h*

5.1.3. LAB MEDIA: Figure 5A *Video Editor: please emphasize black data line from plateau to end of graph*

5.2. Dynamic pressure readings for the reaction **[1]** are typically recorded for the front **[2]**, back **[3]**, and rear dynamic pressure gauges **[4]**.

5.2.1. LAB MEDIA: Figure 5B.

5.2.2. LAB MEDIA: Figure 5B *Video Editor: please emphasize Front data line*

5.2.3. LAB MEDIA: Figure 5B *Video Editor: please emphasize Back data line*

5.2.4. LAB MEDIA: Figure 5B *Video Editor: please emphasize Read data line*

5.3. Like most laboratory cook-off events, the state of the sample container can be assessed for damage after the reaction **[1]**.

5.3.1. LAB MEDIA: Figure 5C.

5.4. Quite a large degree of measured variation in the reaction violence of different propellant samples is commonly observed **[1]**, allowing the violence to be quantified and compared for the different reactions **[2]**.

5.4.1. LAB MEDIA: Figure 5D

5.4.2. LAB MEDIA: Figure 5D *Video Editor: please emphasize data lines*

5.5. In general, faster pressurizing reactions exhibit more scatter or noise in the pressure data, which is consistent with the greater oscillation of the chamber due to a more violent response **[1]**.

5.5.1. LAB MEDIA: Figure 5D *Video Editor: please emphasize "noisy" green, red, and khaki data on left side of graph*

Conclusion

NOTE: Interview statements should have been provided by authors to Devon

6. Conclusion Interview Statements

6.1. **Scott Sumner**: Be sure to place the thermocouples in the same location each time for a smooth and consistent heating rate and also be sure to clean the pressure sensor surfaces to reduce the noise in the reaction violence measurements **[1]**.

6.1.1. LAB MEDIA: To be provided by Authors: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B-roll: 2.2.*

6.2. **Andrew Nelson**: After the analysis, inspect the propellant residue and sample container for final chemical products and check the degree of damage to the sample container from the slow cook-off reaction **[1]**.

6.2.1. LAB MEDIA: To be provided by Authors: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

6.3. **Jonathan Essel**: On a final note, remember that certain rocket propellant ingredients can react very differently than expected while being heated under confined conditions, thank you for watching **[1]**.

6.3.1. LAB MEDIA: To be provided by Authors: Named talent says the statement above in an interview-style shot, looking slightly off-camera.