ASTM Apparatus at EDL[1]

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An ASTM apparatus, following the ASTM E659 protocol [ASTM, 2005], has been constructed by Conor Martin [Martin, 2020, 2023]. The injection system and the temperature acquisition system have been recently improved. This report describes the material needed to reproduce the ASTM apparatus currently used at the Explosion Dynamics Laboratory. The first section lists the main equipment pieces required for the apparatus. The second section describes the setup and gives selected pictures of the ASTM apparatus. The third section contains the experimental protocol. The fourth section includes the lessons learned and recommendations to ensure a good quality of measurements and improve the apparatus. The last section contains details of the setup, with three photographs and a list of components.

I- List of the main pieces of equipment

Furnace:

- Mellen CV12 crucible furnace – 13.3 cm diameter, 20 cm deep cylindrical volume
- 500 ml borosilicate round bottom flask
- 5 cm diameter flat mirror
- 3 x 34-gauges thermocouple, mineral insulated metal sheath
- 1 x 36-gauge thermocouple inside a ceramic sheath
- NI 9213 Spring, 16-ch TC, 24-bit, 75 S/s acquisition module from National Instruments
- cDAQ-9171 CompactDAQ USB Chassis from National Instruments

Injection system

- 6.5“x 4.5“ turntable
- 4” air cylinder
- 8” air cylinder
- 2 electro-pneumatic valves connected to 80-psi pressurized airline
- Velmex slide NEMA 17, 4.5” travel
- Vexta Type 17,1.8º/step 2 Phase, Single Shaft Stepper Motor
- VXM-1 motor controller
- Laptop with 2 USB ports (+ 1 ethernet if a camera is used)

[1] This work was supported by The Boeing Company through a Strategic Research and Development Relationship Agreement CT-BA-GTA-1.

Flush system

- Heat gun
- Aluminum cylinder

II- Description of the apparatus

a) Furnace

Figure 1 is a photograph and a cross-section drawing of the ASTM apparatus as originally used by Conor Martin [Martin, 2020, 2023], constructed based on the ASTM E659 specifications [ASTM, 2005].

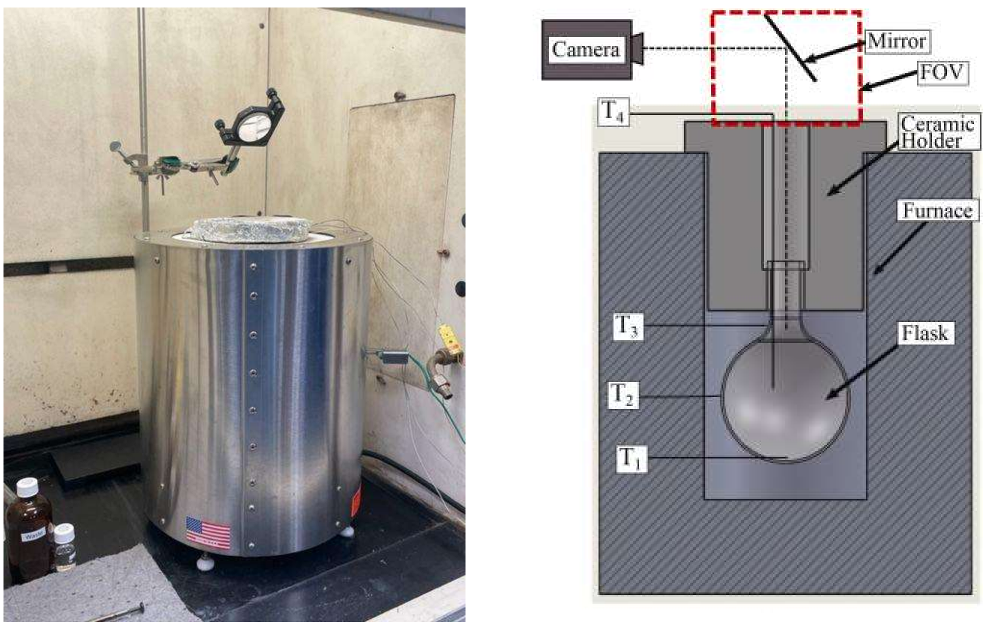

Figure 1: ASTM Apparatus [Martin, 2023]

The apparatus is composed of a Mellen CV12 crucible furnace with a 13.3 cm diameter and 20 cm deep cylindrical volume, which heats up at a controlled temperature a 500 mL round bottom borosilicate flask up to 1250°C with a PID controller (Love Controls series 16B) system accurate to 1°C (a furnace that reaches temperatures up to 600°C can also be used, as this is the maximum temperature the flask can handle without deforming). The flask is suspended in the furnace by a ceramic holder molded in the laboratory from silica based Cotronics Rescor 750. More details on the molding process can be found in [Martin, 2020, 2023]. A 5 cm diameter mirror is positioned above the apparatus's opening at a 45° angle to allow the user to visualize the flames inside the flask. When desired, a high-speed camera can be used to record images of the flame produced by the fuel combustion. In that case, a Phantom VR3746 high-speed camera is used at 800 Hz.

b) Temperature measurement

The temperature of the flask surface is measured with 3 thermocouples type K, gauge 34, inside a mineral-insulated metal sheath, set at position T1, T2, and T3 (see Figure 1 right). According to the ASTM standards [ASTM, 2005], the surface temperature measurements can be made with thermocouples with a gauge up to 20. Extra attention must be paid when using thermocouples with metal sheath insulation. The thermocouple wires should never touch the metal sheath to avoid incorrect measurements. Thermocouples should not touch each other either to prevent electrical disturbances. Kapton tape is used to insulate the cables electrically and secure them around the flask. Kapton tape usually burns during the flask cleaning (600°C), so thermocouples should be positioned correctly, far from each other, to avoid any possible issues. The air temperature is measured with a thermocouple type K, gauge 36, inside a ceramic sheath set at T4, in the middle of the flask. Pictures of the thermocouple setup are proposed in Figure 2. A thermocouple mounting hardware made of flame-retardant high-temperature Garolite has been created to ease the acquisition and ensure that the cables are immobile and not damaged with time. When needed or possible, the thermocouple cables are protected from heat using fiberglass high-temperature sleeving.

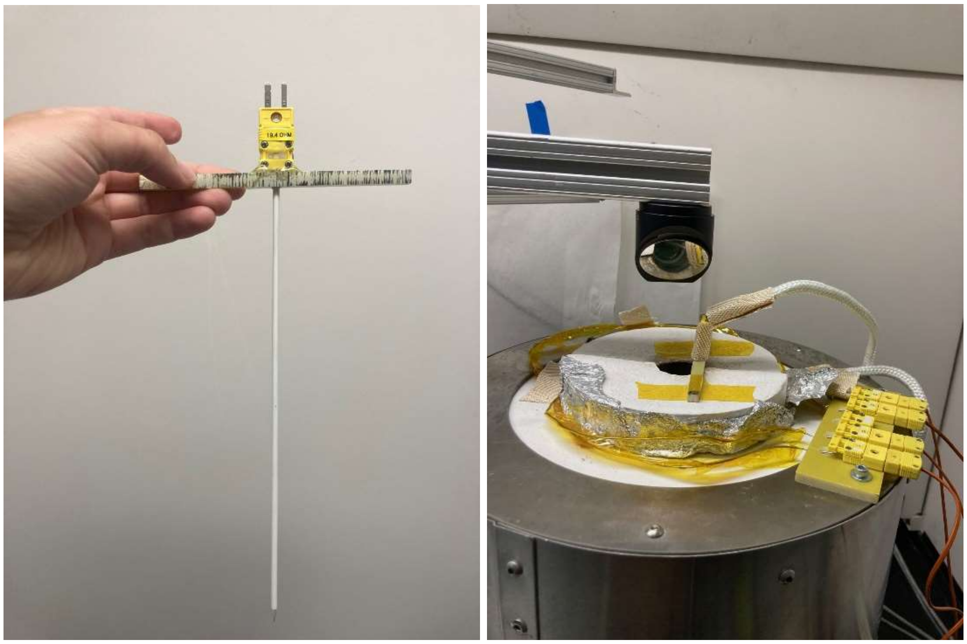

Figure 2: Thermocouple systems. Left: Air thermocouple inside its ceramic sheath, Right: Thermocouples set up in the ASTM apparatus.

The temperature variation is recorded with a NI 9213 module (spring connector), 16-ch TC, 24-bit, 75 S/s acquisition module from National Instruments, connected to a cDAQ-9171 CompactDAQ Chassis. The acquisition is controlled with a custom LabVIEW code on a laptop. Type K extension wires and miniature thermocouple connectors from Omega are used to connect the thermocouples to the acquisition system.

c) <u>Injection system</u>

Efforts have been made to improve the injection repeatability and control key parameters, such as the injection velocity, height, and duration. Pictures of the setup are proposed in Figure 3. More details about the construction can be found in [Pagano, 2023]. The system is composed of a central vertical quad T-Slotted framing rail (2” x 2” x 35“) connected to a turntable (6.5“x 4.5”) and a 4” horizontal air cylinder, which allows a 45° spin of the system around the profile axis. A vertical sliding rail is attached to the vertical

T-Slotted rail and another 8” air cylinder, allowing a vertical displacement. The two air cylinders are controlled by electro-pneumatic valves, connected to an 80-psi pressurized airline. A Velmex slide (NEMA 17, 4.5” travel) is mounted on an aluminum plate to the vertical rail. The slide is controlled by a Vexta Type 17, 1.8º/step, 2-Phase, Single-Shaft Stepper Motor, and a VXM-1 motor controller from VELMEX, connected to a laptop. A horizontal T-slotted framing rail is mounted on the Velmex slide to be used as the fuel injector arm. The syringe is held by a horizontal T-slotted rail connected to the aluminum plate and a detachable piece, which pictures are given in Figure 4. Two small screws with soft ends are added to secure the syringe in the support. Extra layers of material (here, rubber) are added to optimize the position of the syringe so that the needle end is near the bottom of the ASTM flask during the injection.

Figure 3: Pictures of the injection system

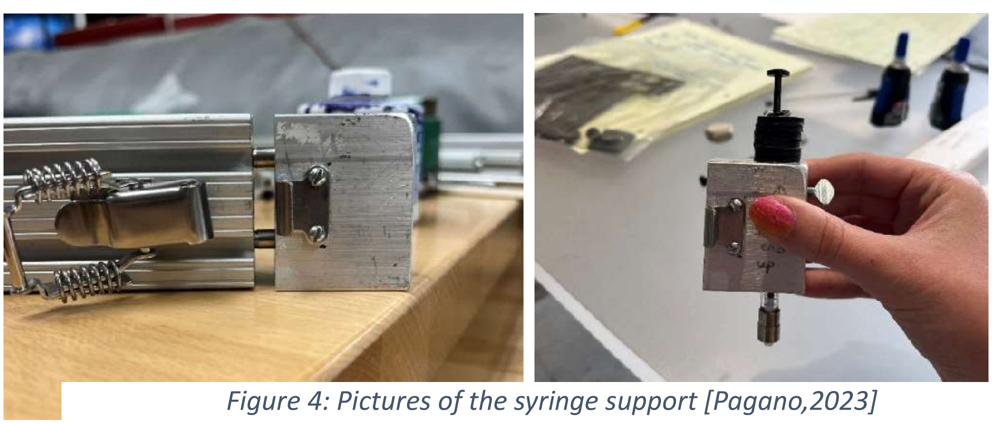

Figure 4: Pictures of the syringe support [Pagano,2023]

III- Experimental protocol

Before testing, the furnace needs to reach a stable temperature. This process usually takes between 1:30 hours and 2 hours. The temperature is considered stable when the variation is less than 0.4 C/min*.

The experimental protocol is the following and can be adjusted based on users' preferences. The steps highlighted by * are discussed in the next section.

- Verify that the temperature is constant
- Prepare the syringe with the desired quantity of fuel
- Set the syringe inside the syringe support
- Acquire for 30 seconds the air temperature inside the flask while setting the syringe support on the T-slotted profile and closing the latches*
- Lower the syringe and the Velmex arm by activating the vertical air cylinder (calibration must be done before so that the needle reaches the wanted height – near the bottom of the flask)
- Start the LabVIEW code, which triggers at the same time the temperature acquisition (10 minutes)* and the Velmex arm, leading to the fuel injection
- Rise the syringe by activating the vertical air cylinder
- Spin the arm by 45° by activating the horizontal air cylinder to set the mirror over the ASTM opening
- Observe if a flame occurs in the first 10 minutes

After the test, the burned gases are flushed using hot air from a heat gun injected inside the flask via an aluminum cylinder for 30 seconds.

The user must wait for the temperature to stabilize before conducting another test. This process usually takes 20 to 30 minutes.

The flask should be cleaned when the surface is visibly dirty*.

IV- Lessons learned and recommendations

Actions can be taken to improve the reliability of the test results. This paragraph gathers some observations and recommendations.

- A repeatability analysis must be conducted before testing a fuel. This leads to a better understanding of the sensitivity of the results regarding different parameters, such as the air temperature, the air thermocouple position, or the injection characteristics. An example of repeatability tests conducted with Hexane is given in Figure 5. In this example, tests have been conducted in conditions as constant as possible.
- The thermocouples should not be moved between tests. It has been observed that the maximum value and the shape of the air temperature signal measured by T4 highly depend on the sensor position, as illustrated in Figure 6, where the temperature of one ignition test has been measured

by 3 thermocouples of the same gauge, at different positions (2, 5 and 7 cm away from the flask bottom).

- Tests must be conducted as consistently as possible to allow a correct comparison. This comes with a strict protocol with a constant delay between each step and a good understanding of the apparatus behaviors (such as the time needed for the temperature to be constant again after a test, the effect of the injection on the temperature, or the variation of the flask temperature with time).
- The temperature must be as stable as possible before testing. However, it takes around an hour for the flask temperature to be stable (i.e., no average temperature variation over the test duration). A compromise must be made between the stability of the initial temperature condition and the number of tests conducted in a day. During our tests, an increase of 0.4 C/min leads to a proper analysis while allowing a test every 30 minutes (for a fixed targeted temperature).
- The fuel should be loaded into the syringe at the last moment to reduce vaporization as much as possible. Similarly, the needle should stay as little time as possible inside the hot flask.
- Ignition highly depends on the initial air temperature. A variation of 0.1°C significantly impacts the combustion delay time. During our tests, the air temperature is measured just before the test for at least 30 seconds. If recorded consistently across the tests, this allows a reference temperature to support the combustion analysis. It is best to start the acquisition while attaching the syringe to the T-Slotted rail. This allows the user to reduce the time between the reference measurement and the test and make sure that no fuel was accidentally dropped inside the flask, which would result in a temperature increase on the reference temperature signal.
- The acquisition time is 10 minutes by default but can be reduced once the user understands the behavior of the fuel. For SAF, the chemical reaction usually happens before the first 5 minutes.
- A difference of around 20 °C is observed between the surface thermocouples T1, T2, and T3, with the highest temperature measured at T2. This is due to the fact that the furnace heating elements are closer to the side of the flask, where T2 is set.
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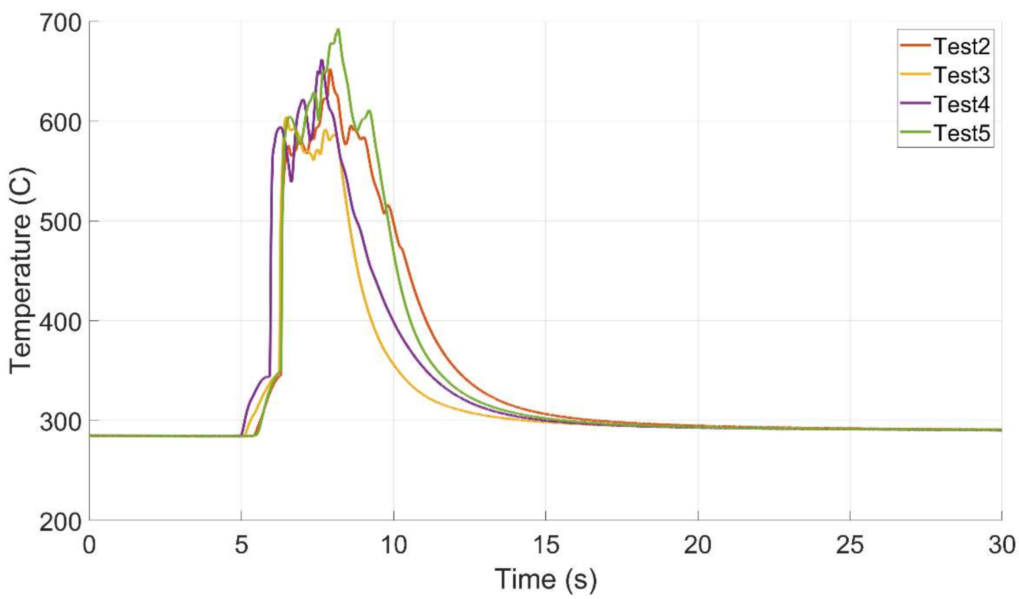


Figure 5: Repeatability tests, Hexane

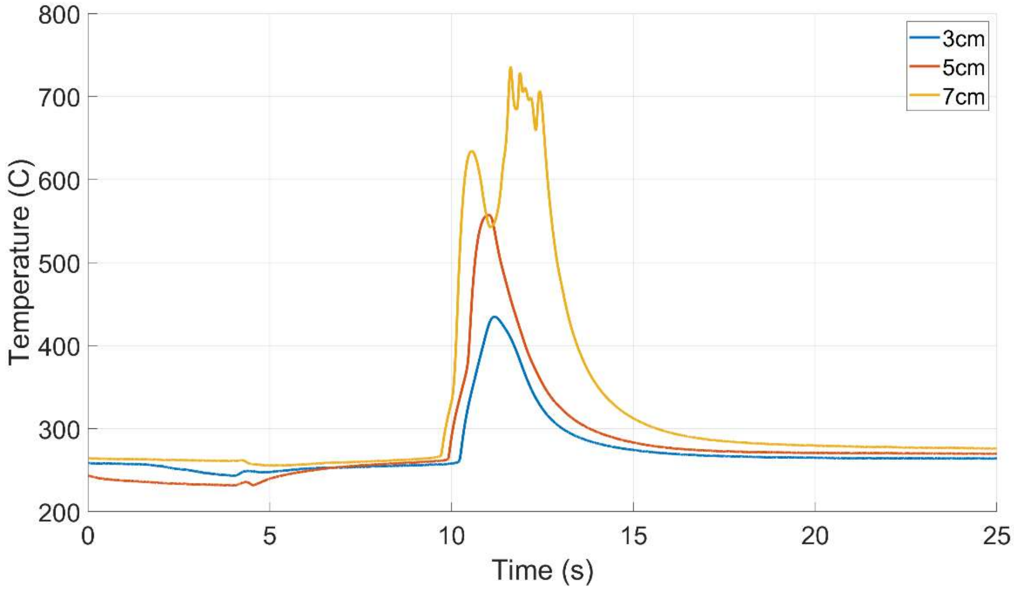


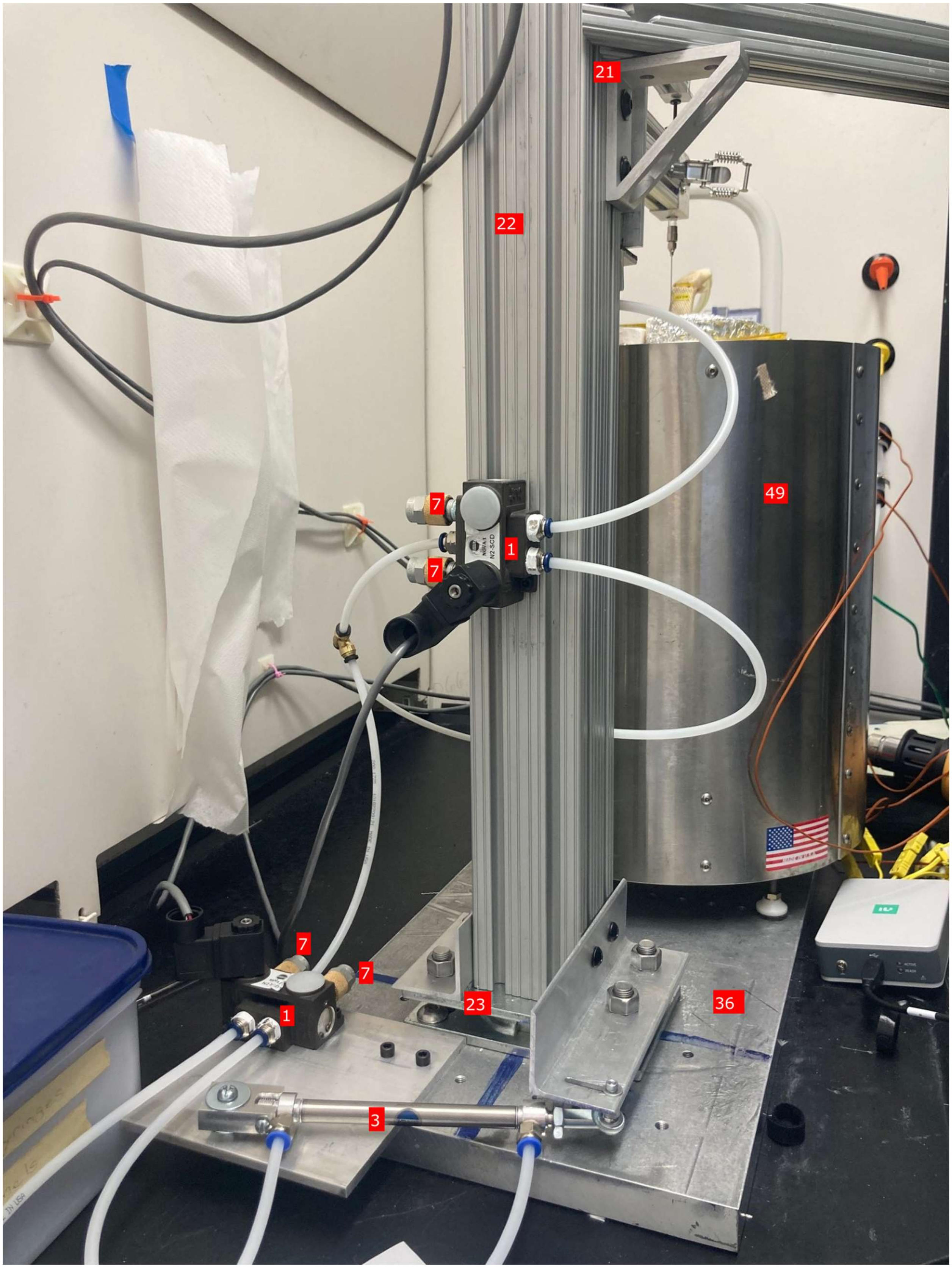
Figure 6: Effect of T4 position on the temperature signal

The apparatus is still a prototype, and improvements can be made to make it sturdier, more user-friendly, more compact, and optimal for fuel characterization. This paragraph contains some suggested modifications.

- Concerning the hardware, the turn table used (from McMaster-Carr) is not sturdy enough to keep the vertical rail from wobbling during rotation. Another option might be preferred. A new design can also be done to make the setup lighter and more compact.
- The furnace used here can reach temperatures up to 1250°C. Temperatures over 600°C led to a deformation of the flask due to the viscoelastic behavior of the borosilicate glass under high temperatures. If higher temperatures are needed, another material should be used for the flask.
- Efforts are currently underway to automate the injection process.
- From a scientific point of view, more work is currently being conducted to investigate the measurement tools needed for a deeper analysis of the combustion process and, therefore, a better characterization of the fuel thermal ignition. For example, a photodiode is being considered to analyze the flame light intensity. The ignition modes I and III must also be further investigated to correctly assess the auto-ignition temperature.

V- <u>List of the components</u>

The following pages contain photographs of the setup where the main components have been numbered and a listing of all components. An effort has been made to supply sources and, when possible, part numbers for all components. The numbers in orange cells correspond to the numbers in the photographs.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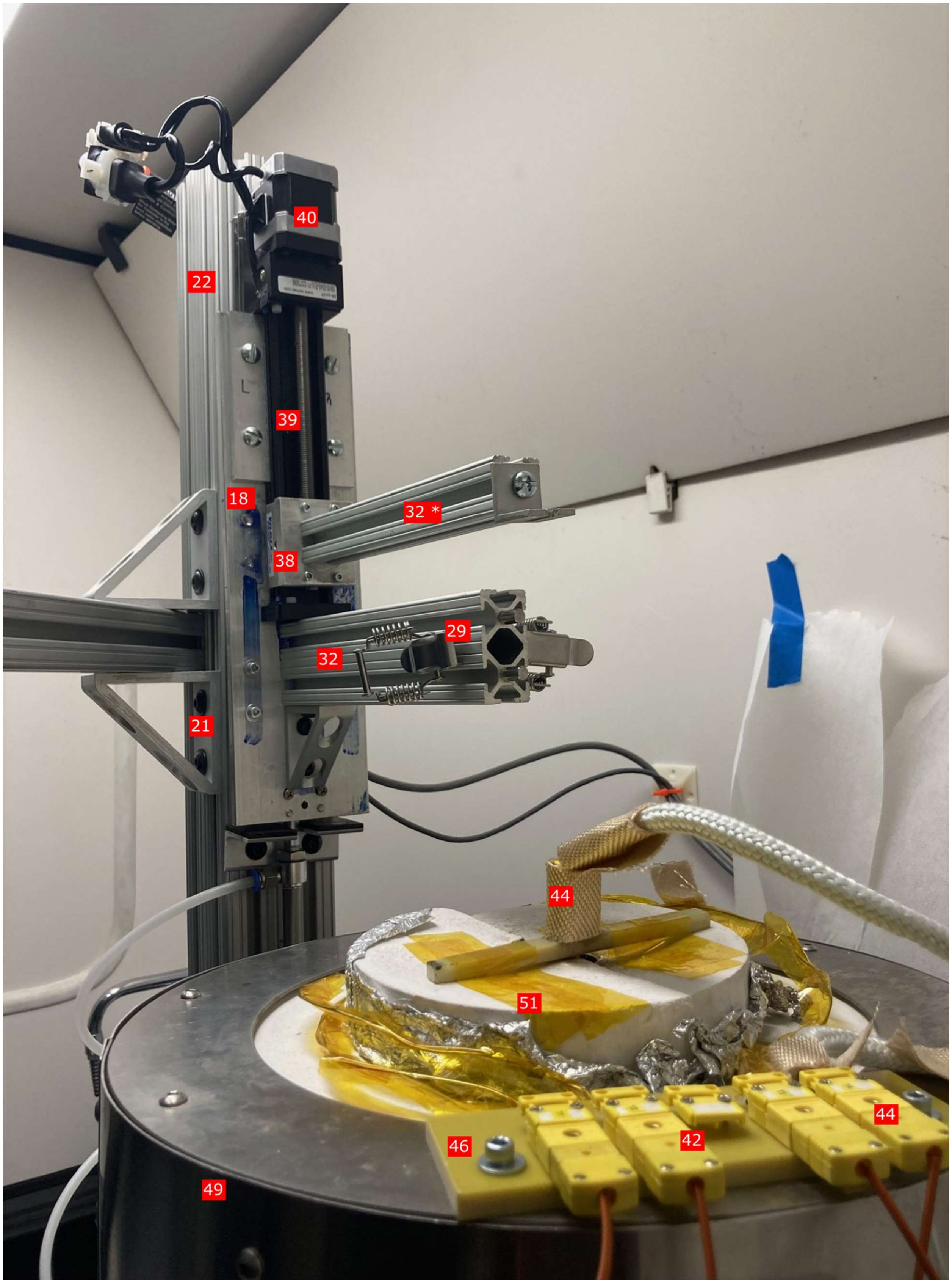
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Item	Qty	Vendor Information	Explanation	Notes
1	2	https://www.mcmaster.com/catalog/6196K232	Electro-pneumatic valves	Important to use directional valves and adjustable exhaust valves to control the speed of operation
2	1	https://www.mcmaster.com/catalog/6498K5	8-in stroke air cylinder	For the vertical motion of Velmex
3	1	https://www.mcmaster.com/catalog/6498K606	4-in stroke air cylinder	For rotation of vertical post
4	2	https://www.mcmaster.com/catalog/60645K31	Ball joints	Mechanical connection to the air cylinder
5	4	https://www.mcmaster.com/catalog/6498K71	Bracket	Mechanical connection to the air cylinder
6	4	https://www.mcmaster.com/catalog/93890A727	Clevis pin	Mechanical connection to the air cylinder
7	4	https://www.mcmaster.com/catalog/129/1237/9164K12	Adjustable valves	Installed on Item 1 exhaust ports of electro-pneumatic valves
8	6	https://www.mcmaster.com/catalog/7397N19	Connectors	For airline and valves
9	1	https://www.mcmaster.com/catalog/5779K229	Tee connector	Airline and pressure gauge
10	1	https://www.mcmaster.com/catalog/1979T1	Airline	Any ¼ line for air service will work
11	1	https://www.mcmaster.com/catalog/8844K11	Compressed air regulator	May not be necessary but used to control pressure on cylinders
12	1	https://www.mcmaster.com/catalog/3847K71	Pressure gauge	Used in connection with pressure regulator
13	2	https://www.mcmaster.com/catalog/8674K157	Bushing blocks (cut out of stock)	Need to put these under the turntable to prevent excessive wobbling
14	1	https://www.mcmaster.com/7593K29/	Enclosure for valve switches	Manual control of valves
15	2	https://www.mcmaster.com/catalog/7343K763	Toggle switch for valve power	Manual control of valves
16	1	https://www.meanwell-web.com/en-gb/ac-dc-single-output-enclosed-power-supply-lrs--50--12	Power for valves	Any 12 DC power supply with >= 2 A capacity will work
17	1		Electrical connection: 2-conductor 16 ga wire to connect valves, switches, and power supply	Low voltage wire will work
18	1	https://www.mcmaster.com/catalog/9246K423	Plate for mounting Velmex to carriage	Machine to size and holes for mounting Velmex and carriages
19	4	https://www.mcmaster.com/catalog/4952K114	Bracket and pin	Used to connect air cylinders to other components
20	1	https://www.mcmaster.com/catalog/8982K14	Aluminum angle	Machine to size and add holes to connect air cylinders to other components

Item	Qty	Vendor Information	Explanation	Notes
21	2	https://www.mcmaster.com/catalog/47065T762	Gusset	Support for mirror mount t-strut
22	1	https://www.mcmaster.com/catalog/47065T502	3x3 T-Strut 3 ft long	Vertical post supporting Velmex and mirror T-strut
23	1	https://www.mcmaster.com/catalog/22345T3	6.5-in x 4.5-in Turntable	Mounts to vertical post and base, enables rotation
24	2	https://www.mcmaster.com/catalog/92949A751	½-20 button head screws	Attaching turntable to base
25	2	https://www.mcmaster.com/catalog/6709K33	Guide rails	Mount to vertical post for carriages
26	4	https://www.mcmaster.com/catalog/6709K12	Carriages	Slides on guide rails and mounts plate with Velmex (we wound up only using 2 of these)
27	2	https://www.mcmaster.com/catalog/5537T359	Guide rail mount to T-slot	Mounting parts for guide rails
28	10	https://www.mcmaster.com/catalog/47065T383	t-Slot spring nuts	Mounting parts for guide rails
29	2	https://www.mcmaster.com/catalog/1734A27	Latches	For attaching syringe mount to T-strut arm on Velmex
30	1	https://www.mcmaster.com/catalog/90128A212	M4 screws	For Velmex mounting
31	1	https://www.mcmaster.com/catalog/90128A332	M4 screws	For Velmex mounting
32	1	https://www.mcmaster.com/catalog/47065T107	1 x 2 T-slot 1 ft long	For mounting the syringe to Velmex
32*	1	https://www.mcmaster.com/47065T101	T-Slot and bracket	Attached to the Velmex rail to apply pressure to the syringe to inject the fluid
33	1	https://www.mcmaster.com/catalog/8975K237	1 X 2 Aluminum stock 6-in long	Machine for syringe mount
34	2	https://www.mcmaster.com/catalog/90772A383	Screw	For holding syringe
35	2	https://www.mcmaster.com/catalog/97042A157	Pins	For locating syringe mount
36	1	https://www.mcmaster.com/8975K628	Aluminum base plate, 12" x 24"	Machine for mounting turntable and air cylinder
37	1	https://www.thorlabs.com/thorproduct.cfm?partnumber=H45CN https://www.edmundoptics.com/p/508mm-diameter-4-6lambda-mirror/23583/	Visualization mirror: T-Strut and mirror mount, 5cm diameter mirror	Used for visual observation of combustion event

Item	Qty	Vendor Information	Explanation	Notes
38	1		Aluminum plate to mount T-strut to Velmex slide	Scrap in labs machined to fit.
39	1	https://www.velmex.com/Products/xSlide/xSlide-motorized.html	Velmex slide NEMA 17, 4.5"travel, # XN10-0045-E50-71	Velmex slide
40	1	https://www.velmex.com/Products/Controls/Motors.html	Vexta motor Type 17,1.8º/step 2 Phase, Single Shaft Stepper Motor, # PK245-01AA	Velmex motor
41	1	https://www.velmex.com/Products/Controls/VXM_Controller.html	Velmex controller,1 axis programmable stepping motor control, w DT60PW240P, # VXM-1	Velmex controller
42	3	https://www.omega.com/en-us/temperature-measurement/temperature-wire-and-cable/mineral-insulated-cables/super-omegaclad-xl/p/XL-K-MO-040	34-gauge thermocouple wires type K, mineral insulated metal sheath, #304-K-MO-040	Surface temperature sensor
43	1	https://www.omega.com/en-us/accessories/tools/wire-strippers/pst-clad-strip/p/PST040A	Thermocouple Wire Stripper, #PST040A	Stripping tool for 34-gauge thermocouples
44	1	https://www.omega.com/en-us/temperature-measurement/temperature-wire-sensors/irco-chal-p13r-p10r/p/CHAL-005	36-gauge bare thermocouple type K, # CHAL-005	Air temperature sensor
45	1	https://www.omega.com/en-us/insulator/orx-insulators/p/TRA-164116-18	Ceramic sheath– 1⁄8" diameter, 9" long	
	9	https://www.omega.com/en-us/temperature-measurement/temperature-connectors-panels-and-block-assemblies/temperature-connectors/smpw-cc/p/SMPW-CC-K-M	Male miniature thermocouple connector	Connections to the acquisition system and extension wires
	8	https://www.omega.com/en-us/temperature-measurement/temperature-connectors-panels-and-block-assemblies/temperature-connectors/smpw-cc/p/SMPW-CC-K-F	Female miniature thermocouple connector	Thermal protection of the thermocouple wires
	4		Thermocouple K duplet extension wires	Scrap from lab – Omega
46	1	https://www.mcmaster.com/catalog/129/4112/5076N27	Thermocouple mounting hardware	Garolite
46*	1	https://www.mcmaster.com/catalog/129/986/7324T11	Fiberglass high-temperature sleeving	Thermal protection TC wires

Item	Qty	Vendor Information	Explanation	Notes
47	1	https://www.ni.com/en-us/shop/model/cdaq-t1101-bundle.html	cDAQ-T1101 Bundle, composed of a NI-9213 C Series Temperature Input Module and a cDAQ-9171 CompactDAQ Chassis for voltage	Thermocouple data acquisition unit
48	1	Any PC will work.	Computer with 2 USB ports and LabVIEW License	Control of the temperature acquisition system
49	1	https://www.mellencompany.com/furnaces	Mellen CV12 crucible furnace	Furnace
50	1	https://us.vwr.com/store/product/16426303/vwr-round-bottom-boiling-flasks	500 ml round bottom glass flask	3.3 Expansion Borosilicate Glass
51	1	https://www.cotronics.com/vo/cotr/cm_castable.htm	Ceramic holder for flask- silica based Cotronics Rescor 750	Molded in the lab

References

[ASTM, 2005] ASTM-E659 (2005): Standard test method for autoignition temperature of liquid chemicals

[Martin, 2020] *Autoignition testing of hydrocarbon fuels using the ASTM-E659 method*. Technical Report EDL2020.001, Graduate Aeronautical Laboratories, California Institute of Technology, March 2020.

[Martin,2023] Martin, Conor Daniel (2023) *Experiments in Thermal Ignition: Influence of Natural Convection on Properties of Gaseous Explosions.* Dissertation (Ph.D.), California Institute of Technology. doi:10.7907/twcf-m219

[Pagano, 2023] Pagano, Isabella (2023) Improving repeatability in minimum autoignition temperature testing in aviation fuels. Surf internship report, California Institute of Technology.