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An Open Source, 3D-printed TrapGuard to Protect Oil-Sealed Vacuum Pumps from Cold Trap Warming

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Abstract

Cold traps condense volatile vapors and prevent solvent contamination of vacuum pumps. Expendable cryogens such as dry ice or liquid nitrogen are typically used to refrigerate traps. Upon loss of the cryogen, the trapped solvents can warm, aspirate into the pump, and foul the pump's oil. The TrapGuard described here protects oil-sealed vacuum pumps by monitoring the temperature of the cold trap Dewar and isolating the pump via a solenoid valve upon detection of warming. This simple device can be 3D-printed and assembled from inexpensive components. This article describes plans for microcontroller programming, temperature sensing circuit, solenoid valve circuit and 3D printing. By preventing contamination of vacuum pump oil, TrapGuard increases pump lifetimes and thus decreases costs – financial and environmental.

Graphical Abstract

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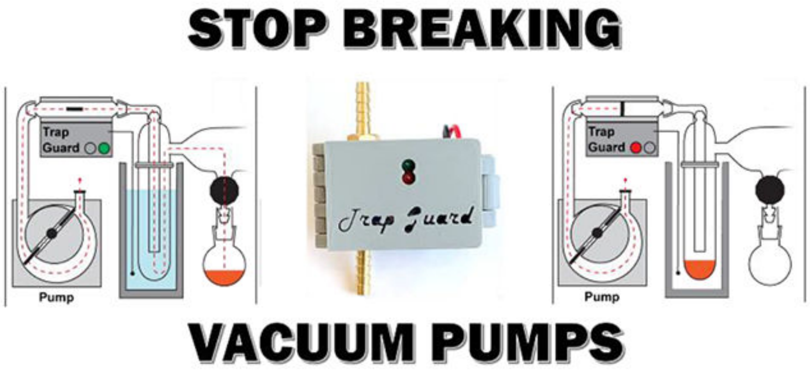
Conflict of interest
The authors declare no conflict of interest.

Supporting Information

- Original CAD models for 3D printing
- AutoDesk Eagle files for custom PCB ordering
- Arduino code for microcontroller
- Additional experimental information, text, figures and tables.



Vacuum pumps are ubiquitous and essential to modern laboratory science. For example, medium vacuum (1 mbar – 10^{-3} mbar) pumps are used for solvent removal, degassing solutions, lyophilization, distillation, and more. Many mechanisms have been invented to generate vacuum pressure including diaphragms, pistons, rotating vanes, turbines, and venturis. Each mechanism has advantages and disadvantages with respect to achievable vacuum, flow rate, manufacturing cost, and maintenance schedule. Though sometimes not the most advantageous option, the rotary vane pump is widely utilized in synthetic chemistry laboratories.[1]

Rotary vane pumps consist of a rotor mounted eccentrically inside a cylindrical stator. Rectangular channels are cut into the rotor which accommodate flat vanes. Under centrifugal acceleration, these vanes slide from the rotor to contact the inside surface of the stator.[2] A small amount of oil is drawn between the vane and the stator during this rotation. The thin film of oil minimizes friction to reduce the vane's wear and also ensures a gas-tight seal between the vane and stator. The maximum achievable vacuum is governed by the vapor pressure of this thin film. If the lubricant begins to boil due to excess heat or the presence of a volatile contaminant (e.g., organic solvent), the gas-tight seal may be compromised, decreasing the maximum achievable vacuum pressure.[3,4]

Solvent contamination of the pump oil can also dramatically reduce the longevity of the pump. Organic solvents can cause swelling of the vanes which are often made from reinforced plastic. Furthermore, contamination reduces oil lubricity, which increases friction between the vanes and stator walls and ultimately leads to pump failure. Typically, routine oil changes remove solvent contamination. Additionally, oil contamination is mitigated through use of a cold trap to condense solvent vapors and prevent them from reaching the pump.[5]

Unfortunately, cold trap maintenance is subject to operator error. Traps must be frequently monitored, emptied of trapped solvents and replenished with cryogen. If the cold trap warms during operation of the pump, the trapped solvents can become volatile and aspirate into the pump – completely negating the cold trap's function. If accidental warming events are also accompanied by infrequent oil changes, the rotary vane pump will inevitably fail. Thus, organic chemistry laboratories typically replace their vacuum pumps on an all-too-regular basis.

The most common solutions to this problem are (1) to use an active chiller for the cold trap and (2) to replace the pump with an oil-free pump. Both solutions work well, but can be cost prohibitive to implement or pose other limitations (e.g., in maximum achievable vacuum and flow rate). Furthermore, chillers still require their traps to be emptied and therefore also create opportunities for operator error. Other solutions include automation of gas ballasts,[6] refilling[7] and flushing[8] of Dewars, and purging of cold trap ice.[9] Despite these advances, innovation has overlooked the need to block volatiles collected in cold traps from reaching their vacuum pumps.

Here, we address this problem with the TrapGuard — a simple, inexpensive device that actively protects oil-sealed pumps from cold trap warming. The TrapGuard monitors the temperature of the cold trap Dewar and controls a solenoid valve installed between the trap and the pump. Cold trap warming to a specific temperature closes the solenoid valve. Thus, the TrapGuard prevents the pump from aspirating volatile solvents.

In addition to protecting oil-sealed pumps from solvent contamination, the TrapGuard also facilitates daily maintenance of the cold trap. With the system in place, the chemist can start each day with the trap's Dewar empty of liquid nitrogen without fear of contaminated vacuum oil. Since it's already at room temperature, the trap can be conveniently disassembled, cleaned, and reinstalled within minutes. No thawing is required, and no valves are manually operated. To reconnect the vacuum pump to the manifold, one simply replenishes the cold trap Dewar with cryogen. Upon detection of the lower temperature, the TrapGuard automatically opens the solenoid valve, Figure 1.

We sought to quantify the efficiency of the TrapGuard to retain common laboratory solvents. Each evening, 25 mL of pure solvent was evaporated from a round bottom flask and condensed into the cold trap. The Dewar was allowed to gradually become depleted of liquid nitrogen until the trap reached room temperature. Each morning the trap was weighed to quantify the retained solvent. The amount of solvent aspirated into the pump oil was estimated as the difference between the initial mass and the recovered mass of solvent. With the exception of high boiling dimethyl formamide, the tested solvents were completely aspirated into the oil, Figure 2. The TrapGuard was then installed and programmed to close at −66 °C before repeating the experiments. Greater than 85% of solvent was recovered in every instance. As currently described, the TrapGuard is unoptimized and could be improved by decreasing the temperature threshold and reducing solenoid chatter near the threshold.

The TrapGuard was intentionally built with simple and inexpensive components that are readily available in laboratories. A proof-of-concept was assembled on a prototyping breadboard, Figure 3. This prototype worked as expected but suffered from sporadic electrical disconnections, as is often the case with breadboard circuits. To prevent disconnections, a permanent solution was devised by fabricating a custom circuit board and 3D-printing an enclosure to keep the electronics and solenoid valve in place, Figure 4. With the printed circuit board (PCB) in hand, only 13 components require soldering to the board. The PCB then fits into a 3D-printed chassis.

As described in the experimental section, the TrapGuard was programmed to close the solenoid valve at temperatures several degrees warmer than a dry ice / iPrOH bath. The threshold for closing the solenoid can be readily adjusted in the microcontroller code. For our experiments, an analog-to-digital value of 468 was used and corresponds to −66 ± 1 °C. This temperature was experimentally measured with a K-type thermocouple and can be calculated using a combination of Ohm's Law, Kirchoff's law, and the β parameter equation.[10] Equations 5 and 6 in the Supporting Information relate the temperature to the expected analog-to-digital value. In a separate set of experiments, we sought to determine the lowest temperature that this implementation of hardware could detect. To do this, we removed resistor **R2** from the circuit board and set the threshold value in the code to 1020. These modifications triggered the solenoid to close at −135 ± 1 °C (referenced with a K-type thermocouple).

The TrapGuard offers a robust and inexpensive solution to daily maintenance of cold traps. The required hardware to build a TrapGuard cost $33.17 (in 2022 USD) and thus provides an economical method to decrease the excessive time and costs associated with pump maintenance, repair, and replacement. At the time of manuscript submission, our laboratory has maintained three TrapGuards in continuous operation for 18 months without failures. These include TrapGuards used to monitor Dewars for a Schlenk manifold, a high-vacuum manifold, and a lyophilizer. Recently, five TrapGuards have been installed in neighboring laboratories for a total of eight TrapGuards in operation without issues.

Conclusion

We demonstrate how a common problem of vacuum pump deterioration can be minimized by using readily available components to build a device that protects oil-sealed vacuum pumps. The device described here can be assembled on a prototyping breadboard in minutes. A more robust solution is offered using a printed circuit board and 3D-printed enclosure (files provided in Supporting Information). This device fills a gap for maintaining an essential item in the laboratory. Since using the TrapGuard can reduce wear and replacement frequency for both vacuum pumps and their oil, this solution increases sustainability and green metrics for chemistry laboratories.

Experimental Section

Vacuum manifold assembly

A rotary vane vacuum pump (Pittsburg, 22.5 micron, 3 $ft^3 \cdot min^{-1}$) was connected to the TrapGuard's outlet barb and secured with a screw clamp to vacuum tubing (Fisherbrand). The TrapGuard's inlet was connected to the outlet of a digital pressure gauge (J-KEM Scientific Model 200) with vacuum tubing secured by a screw clamp on each end. The inlet of the pressure gauge was connected to the outlet (continuous with the down tube) of the borosilicate cold trap (eq. to Chemglass CG-4510-05) with vacuum tubing and screw clamps at each end. The inlet of the cold trap was connected to a borosilicate vacuum manifold (eq. to Chemglass CG-4434-02) with vacuum tubing. No hose clamp was used on this joint for convenience. Glass joints and hose barbs were greased with high-vacuum grease (Dow Corning) and secured with hose clamps. A stainless-steel vacuum-insulated Dewar

(Themo-Scientific 2122) filled ¾ full with liquid nitrogen was placed on a lab-jack (eq. to Troemner 02-214-203) and raised until the bottom of the cold trap was 15 mm above the inner surface of the Dewar. The TrapGuard’s thermistor was placed 20 mm above the bottom of the cold trap and secured in place with one rubber band 30 mm above the bottom of the cold trap and another rubber band 75 mm below the top of the cold trap.

Circuit Board Assembly

All circuitry was prototyped on breadboard for proof of concept. A semi-permanent prototype was soldered on perfboard before committing to a final design. A printed circuit board was designed using AutoCAD Eagle v9.4.2 and custom manufactured by JLCPCB (Shenzhen, China) by uploading the provided Eagle board files to their website. The electronic components were hand-soldered to the board. More details and pictures can be found in the Supporting Information.

3D-Printed Case & Assembly

3D models were designed with SketchUp Make v17.2.2555 and exported as stereolithographic files using the STL Import & Export v2.2.0 from the extension warehouse. Ultimaker Cura v4.7.1 was configured for the Creality Ender-3 printer and used to slice models. The stereolithography files were sliced using the dynamic quality settings for generic PLA with the following adjustments: 0.3 mm layer height, generate support, support overhang angle 86°, build plate adhesion type brim, brim width 5 mm, printing temperature 220 °C, build plate temperature 60 °C, retraction speed of 50 $\text{mm}\cdot\text{s}^{-1}$, and retraction distance of 6.5 mm. 3D-printing was accomplished on a Creality Ender-3 with a 0.4 mm brass nozzle using Inland 1.75 mm PLA filament and a glass print bed.

Next, the brass hose barbs were screwed into the solenoid valve using Teflon tape on the threads. The valve was fastened to the back of the 3D-printed case using M3 x 7 mm button head screws and the 3D-printed bracket. After attaching the valve, the assembled circuit board was placed into the case. The solenoid wires were trimmed to approximately 5 cm, the ends were stripped of insulation, tinned, and inserted through the holes of the case nearest the screw terminal block labeled ‘solenoid’ and screwed in tightly. Lastly, each leg of a negative temperature coefficient thermistor (10 kΩ, β = 3020) was soldered to a 1 m section of 20 gauge insulated wire and the solder joints were protected with heat shrink tubing. The available ends of the wire were stripped of insulation, tinned, and inserted through the holes of the case nearest the screw terminal labeled ‘thermistor’ before being screwed in tightly.

Material compatibility note: A complete teardown and list of materials used in the solenoid valve is available in the Supporting Information. The solenoids reach 55 °C during normal operating conditions at a room temperature of ≈23 °C. The experimentalist must assess compatibility of these materials and conditions for their application.

TrapGuard Code and Calibration

After assembling the electronics, the code provided in the Supporting Information was uploaded to the Arduino Nano microcontroller using the Arduino integrated development environment (IDE) version 1.8.13. With the microcontroller connected to the computer, the serial monitor was opened (Tools > Serial Monitor) and the serial communication streaming from the Arduino was visualized on the computer screen while the thermistor was submerged in a dry ice/isopropanol bath. The highest numerical value was noted for dry ice/isopropanol (485). The thermistor was removed and submerged in liquid nitrogen. The highest numerical value was noted for liquid nitrogen (500). Line 5 of the TrapGuard.ino code was altered to reflect the highest number noted for dry ice/isopropanol minus an arbitrary amount for buffering purposes, as shown below:

```
#define THRESHOLD 468
```

The code was re-uploaded to the Arduino Nano after editing line 5. Next, the TrapGuard was powered on by the 12 V wall adapter, the thermistor was immersed in the dry ice/isopropanol bath, and the solenoid was verified to click open.

Solvent Trapping Experiments

At the beginning of each day, the cold trap was removed from the Dewar, patted dry of any external condensation, and weighed on a semi-analytical balance (Sartorius Entris). The round bottom flask was disconnected from the manifold and weighed. The vacuum pump was then powered off and serviced with an oil change (Inland-19, 250 mL). The round bottom flask and cold trap were thoroughly cleaned and dried before re-installing on the vacuum manifold. A vacuum check was then performed by turning the pump on and filling the Dewar with liquid nitrogen to open the TrapGuard solenoid. The manifold pressure was observed with a digital pressure gauge. If the gauge was not <100 torr in under 10 min, the system was checked for leaks. **Cryogen safety note**: If using liquid nitrogen, ensure that the vacuum system is airtight. An explosion can result if atmospheric oxygen is allowed to condense in the cold trap with organic material. After the vacuum check was complete, the round bottom was removed from the manifold, tared on an analytical balance, and partially filled with approximately 25 mL of solvent. The partially filled flask was weighed and immediately reinstalled on the manifold where it was subject to overnight vacuum. The next morning the procedure was repeated. Each solvent was tested three times with the TrapGuard installed. For comparison, the TrapGuard was removed from the vacuum system and the experiments were repeated with the slight modification that every morning the vacuum pump would be manually turned off before removing the cold trap.

Supplementary Material

Refer to Web version on PubMed Central for supplementary material.

Acknowledgements

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References

(1). Chambers A; Fitch RK; Halliday BS Basic Vacuum Technology., 2nd Ed.; IOP: London, 1998.

(2). Ramprasad BS; Radha TS On Some Design Aspects of Rotary Vane Pumps. Vacuum 1973, 23, 245–249.

(3). Lachenmann R Oil Reservoir Evacuation for Pumping Condensable Vapours through Oil Sealed Rotary Vane Pumps. Vacuum 1988, 38, 659–663.

(4). O’Hanlon JF Vacuum Pump Fluids. J. Vac. Sci. Technol. A Vacuum, Surfaces, Film. 1984, 2, 174–181.

(5). Cole M A New Type of Vacuum Condensable Gases. Vacuum 1988, 38, 647–649.

(6). Abraham J-L Vacuum Pump And Method For Controlling A Gas Ballast Supply To A Vacuum Pump. European Patent 2191137B1, 2014.

(7). McCorkle EJ; Vogel H; Marcel M; Ferranti RT Apparatus for Controlling Level of Cryogenic Liquid. European Patent 0777078A1, 1997.

(8). Neeser TA Cryogenic Dewar Level Sensor And Flushing System. United States Patent 5275007, 1994.

(9). 오이카와겐. Cold Trap and Recycling Method. Korean Patent 101054683B1, 2011.

(10). Sze SM; Ng KK Physics of Semiconductor Devices.; 2006.

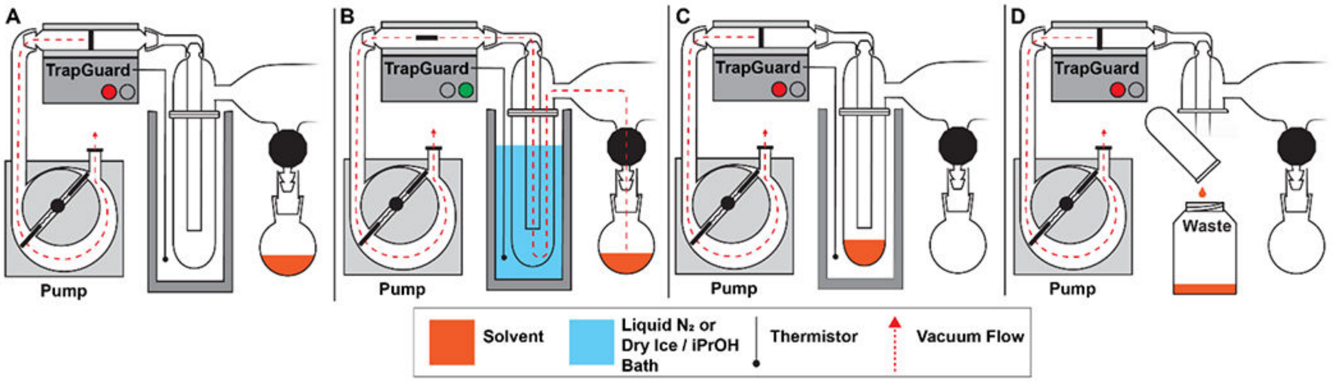

Figure 1.

A) A sample with liquid solvent is placed on the vacuum manifold. B) To initiate vacuum, cryogen (e.g., liquid nitrogen) is added to the Dewar. The TrapGuard detects the cold temperature in the Dewar and opens the solenoid valve, exposing the sample to vacuum. C) When the cryogen in the Dewar dissipates, the TrapGuard senses the change in temperature and closes the solenoid valve, which prevents the condensed solvent from reaching the vacuum. D) The cold trap can be readily disassembled and cleaned without shutting off the vacuum pump.

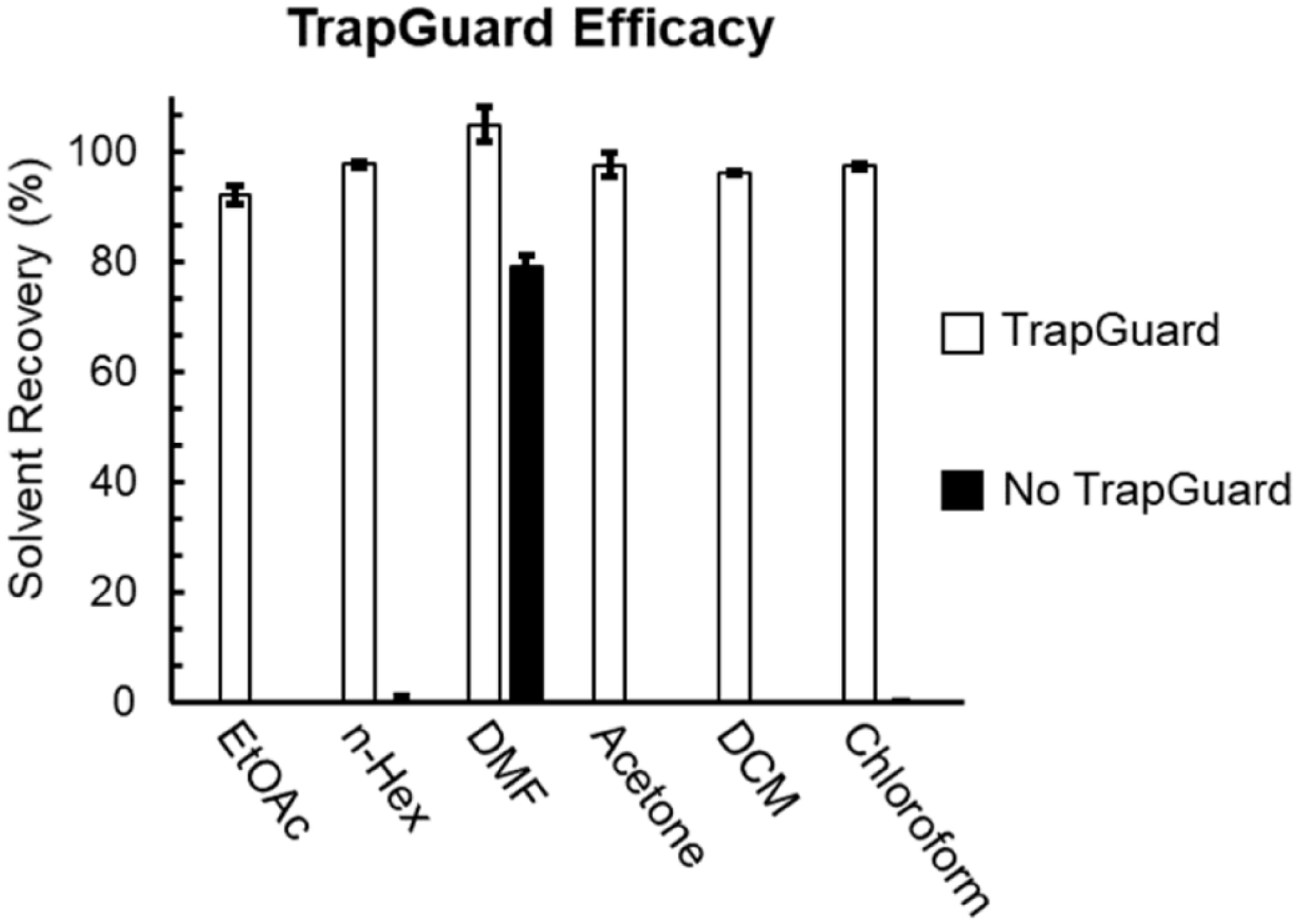

Figure 2.

Solvent recovered from the cold trap after overnight evaporation of solvent (≈25 mL). Error bars represent the standard deviation of n=3 independent replicates.

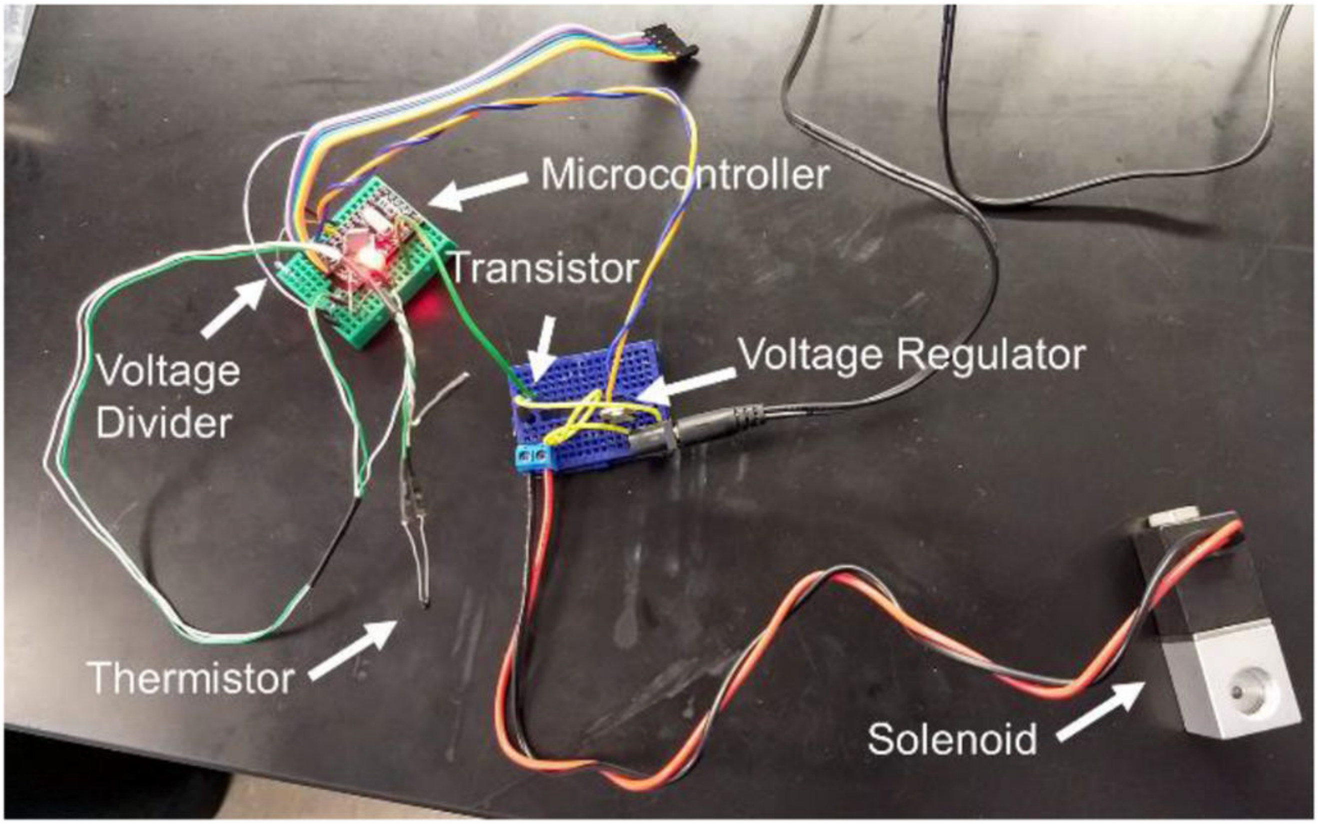

Figure 3.
The first prototype of the TrapGuard assembled on an electronic breadboard.

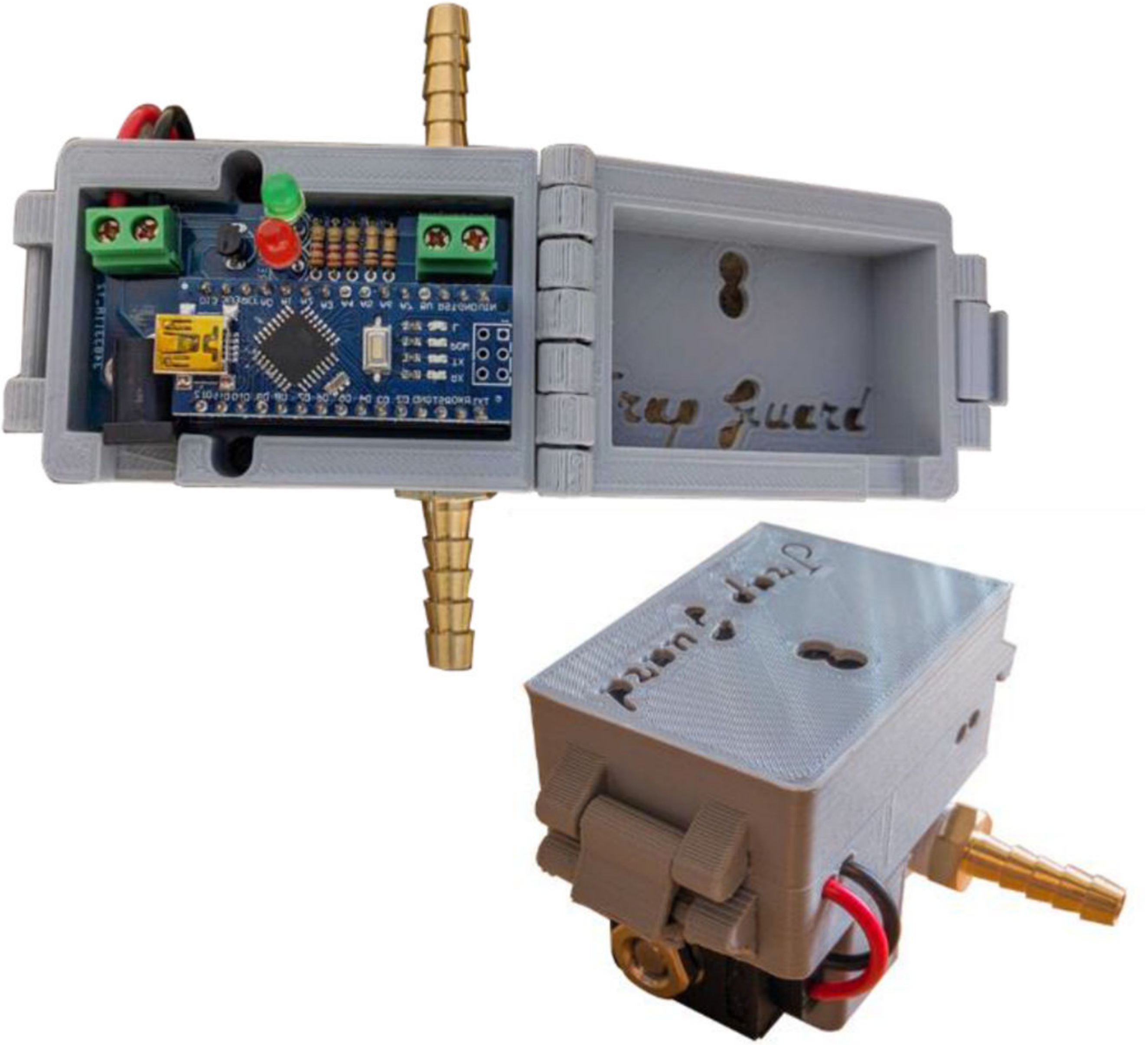

Figure 4.
The final version of the TrapGuard with printed circuit board and 3D-printed case.