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Environmental Testing of Tritium-Phosphor Glass Vials for Use in Long-Life Radioisotope Power Conversion Units

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Introduction and Background

Power generation in extreme environments, such as the outer solar system, the night side of planets, or other low-illumination environments, currently presents a technology gap that challenges NASA's ambitious scientific goals [1]. For missions to these destinations lasting several years or more, power generation technologies must be low-mass, have long lifetimes, and be radiation hard and durable. The availability of technologies meeting these requirements is currently limited to radioisotope thermoelectric generators (RTGs), which have masses of at least several kg, with recent examples for discovery-class missions weighing > 50 kg. This leaves a gap for power generation in small platforms like SmallSats and CubeSats, where both mass and volume are significantly constrained.

Our team, comprising a collaboration between personnel at RIT, JPL, and MSFC, is developing an integrated power supply (IPS) that addresses the low-power/long-lifetime need of deep-space SmallSats and CubeSats based upon radioisotope power converters (RPCs). These devices are based on the observation that radioactive energy can be converted to current either thermoelectrically (as in an RTG) or through direct or indirect energy conversion [2]. A direct-conversion RPC makes use of materials that emit either α or β particles at high energy, which are then coupled directly to a semiconductor diode to convert the energy to current and voltage. Because the energy of the emitted particles (10s – 100s of keV) is significantly greater than typical visible photon energies (1 – 2 eV), this method can create multiple electron-hole pairs in a standard semiconductor PN junction, and therefore has very high total system efficiency. As well, typical radioisotopes offer very high energy densities, on the order of 10 – 100 of W/cm^3 , as well as potential for long lifetimes [3]. There are currently a number of companies and research groups investigating the use of direct conversion of β particles (typically either ^{63}Ni or tritium) using wide bandgap materials such as SiC [4], [5].

However, the power outputs of these devices are still quite small, typically fractions of a mW. This is because the radioisotopes emit particles over a wide range of energies, which will produce electron-hole pairs far from the PN junction, reducing the efficiency of collection and therefore reducing total power produced. Additionally, more energetic and/or massive particles that would stop closer to the PN junction cannot be used because they cause very rapid radiation damage in the semiconductors, on the order of a few minutes for α particles and within a month for high energy and fluence β particles [6], [7].

To avoid these drawbacks, our approach to the RPC makes use of a radio-luminescent phosphor as an *indirect* conversion mechanism. Indirect RPCs down-convert the wide spectrum of energies from the radioisotope to a narrow range and place the photovoltaic cell away from any radiation damage (see Fig. 1). The light generated by this phosphor is then converted to electric current and power in much the same way as in a solar cell. The second benefit of this method is that the photovoltaic need only be optimized to

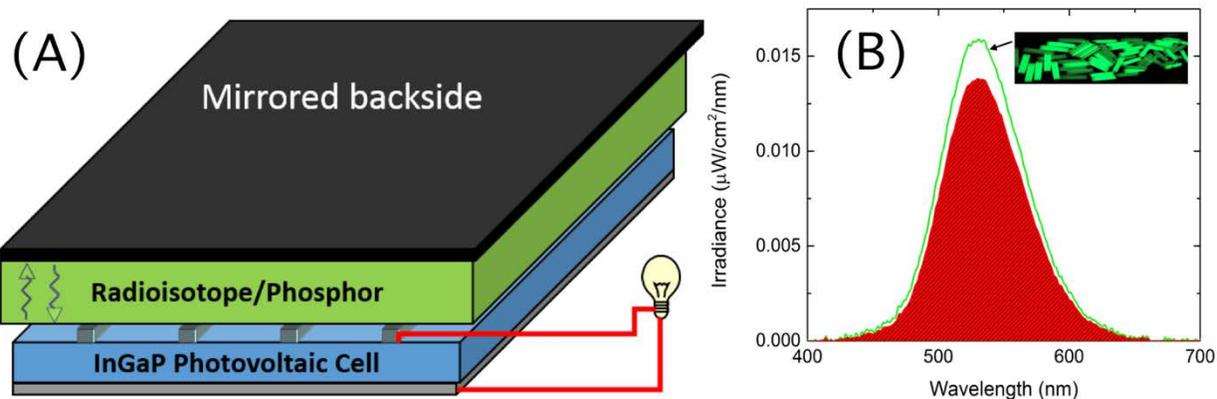


Figure 1: Panel (A) shows a schematic representation of our proposed RPC using a tritium/phosphor illuminator coupled to an InGaP photovoltaic cell. Panel (B) shows the measured spectral irradiance from one side of a rectangular vial of commercially available tritium gas coupled to a ZnS based phosphor (a *trigalight*® laser sealed gaseous tritium light source) obtained from tests performed at RIT. The red area shows the fraction of energy that can be converted by a high-efficiency InGaP photovoltaic).

collect the narrow band of wavelengths produced by the intermediate phosphor, leading to very high efficiencies in excess of 30%.

Our proposed indirect RPC system utilizes commercially available tritium (^3H , half-life of 12.3 years and 5.7 keV peak energy; [8]) light sources and standard 1.85 eV InGaP₂ photovoltaic cells to convert the β particle energy. Though custom glass vials could, in principle, be coated with a phosphorescent paint and filled with tritium gas, commercial products exist in formats that could easily be adapted for use in our proposed RPC.

Objectives

Though using a commercial product reduces the cost and complexity of the required system development and fabrication, questions about the strength and robustness of the glass vials are apt. In the test program described here, we perform environmental tests on commercially available borosilicate glass vials internally coated with a ZnS luminescent phosphor that are designed to contain gaseous tritium in our proposed power source. Such testing is necessary to ensure that the glass containing the radioactive tritium is capable of withstanding the extreme environments of launch and space for extended periods of time. Should the glass vials fail, the tritium gas would escape and render our RPC inoperable.

The standard environmental tests required for CubeSats, which are the most applicable test suites to our proposed application, are summarized in Table 1. There are two major tests that must be passed, namely the “thermal vacuum bake out” test (with specifications given in [9], summarized in Table 1) and a launch-level vibration test (with specifications given in [10], summarized in Table 2).

Table 1: Thermal vacuum test requirements.

Minimum Temperature	70°C
Number of Cycles	1
Dwell time	Minimum 3 hours after thermal stabilization
Transition rate	< 5°C/minute
Vacuum	< 1×10^{-4} Torr
Test reference	MIL-STD 1540 B, GSFC-STD-7000

Table 2: Vibration test requirements.

	Qualification	Acceptance	Preflight
Directions	X, Y, Z	X, Y, Z	X, Y, Z
Sweep Rate	2 oct/min	4 oct/min	4 oct/min
Amplitude (g), 5 – 100 Hz	2.5	2	2.5
Amplitude (g), 100 – 125 Hz	1.25	1	1.25

Testing Procedure

Test Articles

Commercial tritium light sources are available in a variety of sizes, shapes and colors. Different phosphor chemistries result in different colors of these lights (as can be seen in emergency exit signs, gun sights or military style watches). The brightest and most efficient phosphor is a mixture of ZnS and is typically green with a peak wavelength of 528 nm and intensity from one face of the vial near $1.2 \mu\text{W}/\text{cm}^2$ (as shown in Fig. 1). We have identified trigalight® laser sealed gaseous tritium light sources (GTLS) cells as a commercially available candidate phosphorescent illumination source [11]. These cells are $2.0 \times 4.0 \times 50.0 \text{ mm}$ and are made with borosilicate glass. Each can be filled with approximately 23 GBq of gas.

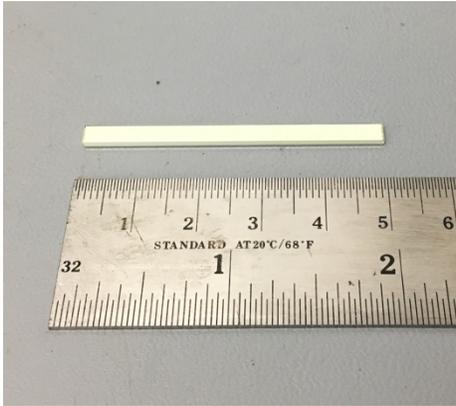


Figure 2: A $2.0 \times 4.0 \times 50.0 \text{ mm}$ trigalight® vial filled with inert gas used in our tests.

To avoid complications associated with the release of radioactive gas in the laboratory, for these initial tests we acquired test cells containing inert gas. A photograph of such a vial is shown in Figure 2. In addition to the standard NASA tests, the vial manufacturer performs their own tests on their product [12], which include a temperature test requiring the vials withstand -190°C for 10 minutes and 480°C for 5 minutes. They also use a submersion test to limit the gas leak rate of the vials to $< 1850 \text{ Bq}$ in 24 hours. Both of these tests are conducted randomly on samples during the manufacturing process.

Thermal Vacuum Validation

As a preliminary check, we first subjected a vial to low vacuum to test whether it could withstand a pressure drop to $< 1 \text{ Torr}$ without damage. With a simple rotary vane pump we evacuated a bell jar to about 0.2 Torr, at which pressure it remained for 3 hours. Following the test, the glass was removed from the bell jar and inspected under a microscope for damage. None was observed.

We then performed a simple thermal test on the vials at atmospheric pressure. The vial was bonded to a small heating and thermometry unit with GE 7031 varnish, as shown in Fig. 3. The heating unit consisted of an $R = 100 \Omega$ power resistor and a LakeShore DT-670 diode thermometer, which was monitored by a LakeShore model 218 temperature monitor. The heating unit was thermally isolated from the laboratory bench using a $1/16''$ -thick slab of glass-mica ceramic. The heater was wired to a standard DC power supply. For this test, the heater was set to a constant 15 V, during which time the temperature of the heating unit increased at a rate of $< 5^\circ\text{C}$ per minute until it reached 50°C , at which point the voltage was increased to 20 V in order to reach 70°C . Once the cell had reached the test minimum 70°C , the voltage was reduced to 19.5 V, which maintained the temperature at a constant 71.5°C for 3 hours. After 3

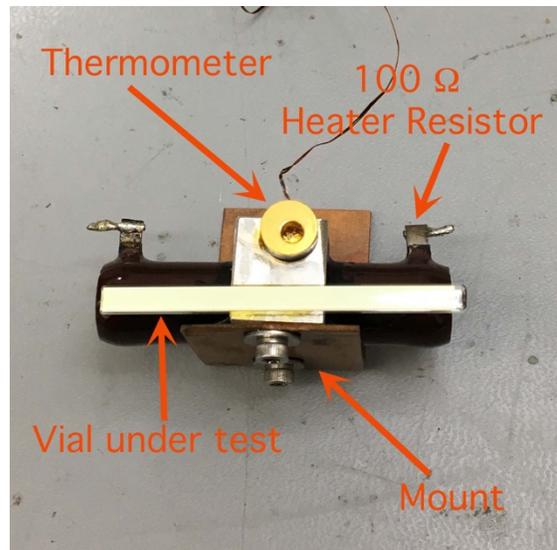


Figure 3: Thermal-vacuum test rig, which includes a heater resistor and thermometer that can be used in vacuum.

hours, the glass was removed from the setup, allowed to cool, and once more inspected for damage under a microscope. None was observed.

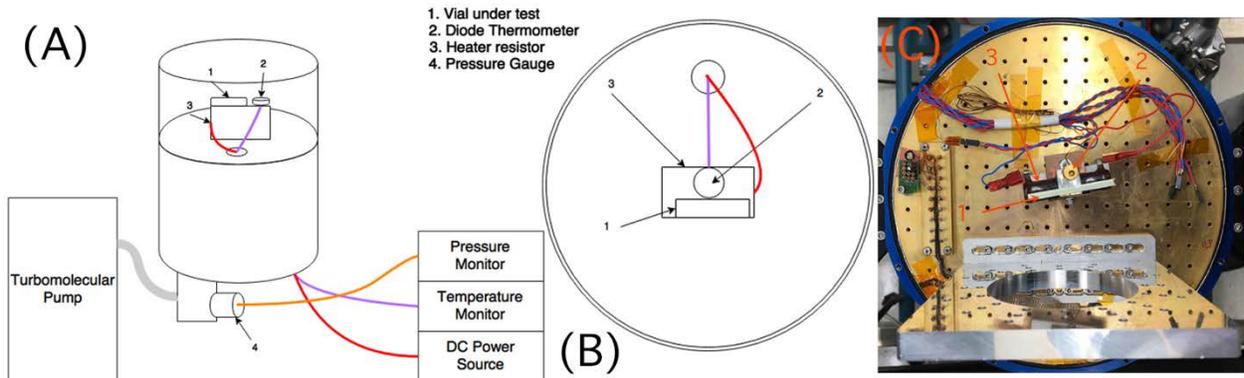


Figure 4: Schematic and photograph of the setup for the thermal vacuum test. Panel (A) shows a schematic representation of the vacuum chamber and the relative position of the test article and various readout systems. Panel (B) shows a schematic view of the heater unit, including bonded thermometer and test article, on the vacuum chamber work surface. Panel (C) shows a photograph of the as-built system, with leaders highlighting the various components of the test unit. The large metal structure visible at bottom is not related to this test.

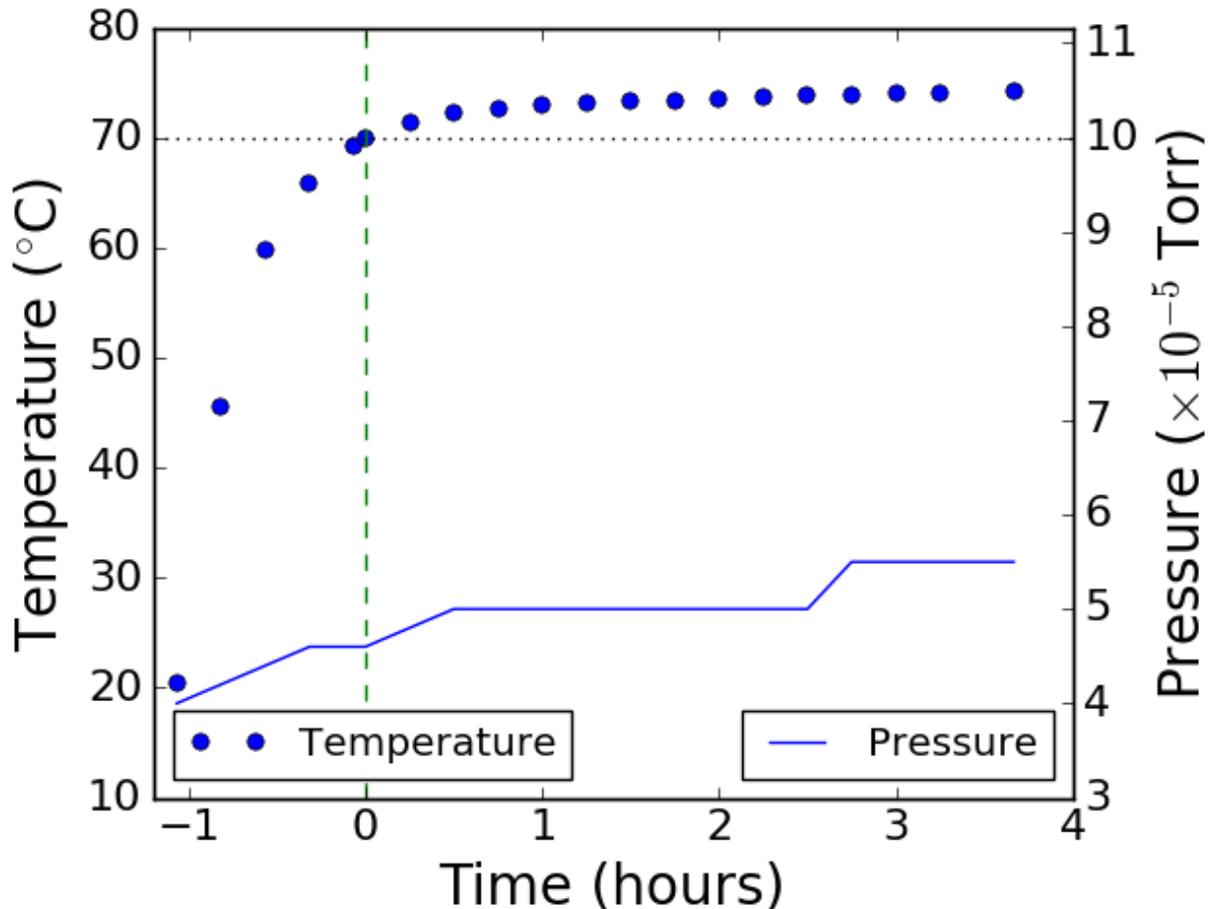


Figure 5: Plot of the pressure and temperature of the test article during the thermal-vacuum test. The requirement for the temperature is to remain $> 70^{\circ}\text{C}$ for 3 hours, and for the pressure to remain $< 10^{-4}$ Torr during the duration of the test, both indicated by the black dotted line. In this plot we also show the warmup period, where we maintained a heating rate $< 5^{\circ}\text{C}/\text{min}$ to the beginning of the test (green dotted line).

The final part of this experiment consisted of exposing the vial to a vacuum $< 1 \times 10^{-4}$ Torr and a temperature of greater than 70°C simultaneously for 3 hours. The vial and heating unit were placed in a sealable vacuum chamber, shown in Fig. 4. To achieve high vacuum, we used an Oerlikon 350-L turbomolecular pump. A pressure gauge was instrumented to the inlet of the vacuum chamber to monitor the pressure. Wires to operate the thermometer and heater were passed through hermetic electrical connections integrated on the vacuum chamber bulkhead. For this thermal-vacuum test, the test vessel was evacuated for several days until a steady pressure of 5×10^{-5} Torr was reached. The power source was set to 10 V, which increased the temperature of the heating unit from 20°C to 70°C in ~ 1.5 hours, with a derivative always less than $5^{\circ}\text{C}/\text{minute}$. We then maintained the temperature of the test article at 71°C for 3 hours. The temperature and pressure observed during the measurement are shown in Fig. 5. Following the test, we removed the test article from the chamber and inspected it under a microscope. No damage was observed. As a further test, we submerged the test article in 100% isopropanol to check for micro-fractures. No liquid penetrated the vial.

Sinusoidal Vibration Validation

We conducted three different sinusoidal vibration tests in all of the X, Y, and Z axes on the test vial. Our vibration test system consists of an air-cooled LDS Shaker-V555 vibration simulator, a Hafler Pro5000 amplifier, and a B&W Engineering DSC 8 Digital Sine Controller. We mount a $6'' \times 6''$ aluminum breadboard to the shaker head and manufactured a custom jig to hold the vial in each of the three orientations. The test setup for the vial in the Y-axis configuration is shown in Fig. 6. For the test, we monitor the acceleration of the test head using an Endevco 2271A accelerometer, which is used to servo-control the vibration head through an input on the DSC controller. Following each test, we inspected the vial for damage. None was observed.

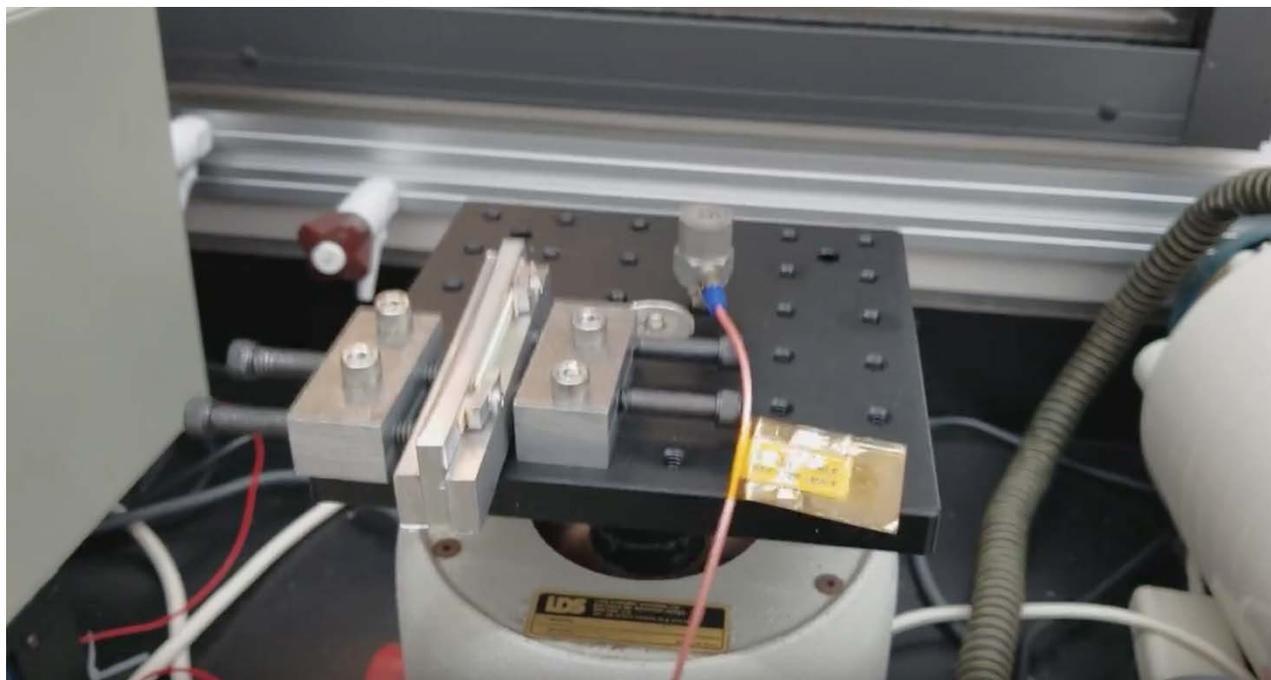


Figure 6: A photograph of our test setup for the vial vibration tests. The vial is clamped at either end using the test jig, visible at the center left of the photograph. In the jig, material is milled away both above and below the test piece, to allow it to flex naturally under vibration.

Conclusions

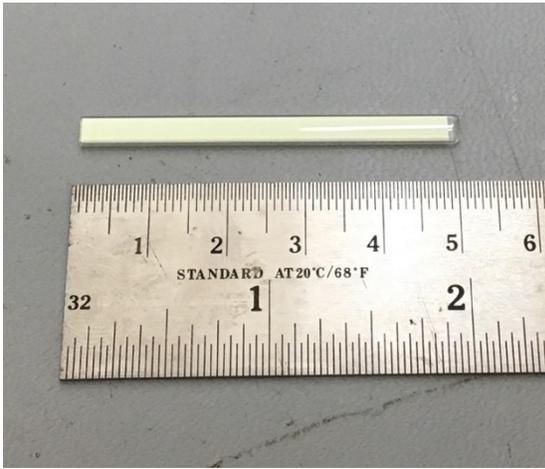


Figure 7: Photograph of the test piece following both thermal-vacuum and vibration testing. No damage to the vial is observed either under visual inspection with a microscope, or after a 24-hour submergence test in isopropanol.

In this study, we performed environmental testing of $2.0 \times 4.0 \times 50.0$ mm borosilicate glass trivalight® tritium-phosphor vials. In Fig. 7 we show a photograph of the vial following the test suite; it appears to have suffered no damage compared with the initial image shown in Fig. 2. In addition to visual inspection under a microscope we also performed a final 24-hour submergence test in 100% isopropanol and observed no liquid leaking into the gas volume of the test piece. We conclude that this model of glass vial is appropriate for use in space, and that it will serve as a good illumination source for our RPC. Of course, the final as-built system will need to undergo its own flight qualification testing, but this component test gives us confidence to proceed with the technology development.

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