

Lab 11, Bug 4: Putting it all together

March 31, 2025

1 Goals

- Data acquisition: Implement a temperature sweep.
- Measure $C(T)$.
- Data analysis: Estimate ferroelectric critical temperature, T_c .

1.1 References

- Read pages 86–89 of the “Introduction to LabVIEW in 6 hours” (on Canvas) to understand the concept of a “local variable.”
- M. Kahn, [Multilayer ceramic capacitors – materials and manufacture](#), technical report from AVX Corp., Myrtle Beach, SC (USA). How these materials are made into capacitors and other devices.
- M. Trainer, [Ferroelectrics and the Curie-Weiss law](#), *Eur. J. Phys.* **21**, 459 (2000). Fancier version of our experiment that can explore temperature below ambient.
- A. Amir, Y. Oreg, and Y. Imry, [On relaxations and aging of various glasses](#), *Proc. Natl. Acad. USA*, **109**, 1850–1855 (2012). Discusses the origin of $\log-t$ dependence of material parameters, such as capacitance.
- M. Zhang et al., [Ultrahigh energy storage in high-entropy ceramic capacitors with polymorphic relaxor phase](#), *Science* **384** 185–189 (2024). Example of current research interest in these materials.

2 Prelab questions

Question 1. Plot Eq. 1 using values of $\kappa = 0.05$, $\gamma = 1.5$, $C_{\max} = 10$ nF and $T_c = 50^\circ\text{C}$, for temperatures you can measure above T_c .

Question 2. Review the LabVIEW VI illustrated Fig. 1 and the general description of it below. Construct a **flow chart** that summarizes the flow of the logical steps in the proposed measurement VI. It need not use official LabVIEW symbols but should indicate clearly the logical decisions that will be needed in the VI.

3 Experiments

Finally, we are ready to tackle our problem of measuring $C(T)$ for the Bug. We start from the working LabVIEW program you finished last week to control the temperature T and measure the capacitance C , by doing a curve fit to data acquired from the triggered signal acquisition of the decay of a series RC circuit. Your task this week will be to sweep the temperature from just above room temperature to 100°C , recording the time, temperature, and capacitance. Of course, you can already do this by hand by typing in a value of the setpoint, waiting for the capacitance to stabilize, going to the next set point, waiting, etc. Our goal today is to get the computer to do it all automatically.

1. **Data acquisition (LabVIEW):** We begin by considering the overall structure of our program. Figure 1 shows a suggested VI configuration. Actual details may differ according to individual choices (there are VIs that do the same thing, and you may want more indicators to monitor/debug things). Note that it has three frames in the main sequence: an initialization section, the main **While** loop, and a final section. We consider each frame in turn.

- (a) **Initialization.** The first step in your program is to initialize variables, e.g., the starting temperature T_{start} , final temperature T_{stop} , and temperature increment dT . We want to take more than one measurement at each temperature to get the benefit of signal averaging. The quantity N_{sample} will determine the number of measurements we do at each temperature, after waiting for the temperature to stabilize. After each measurement we decrease an index variable N_{current} until it reaches zero as explained in the following section.
- (b) **Main loop.** The main code that you need to add this week is the control logic to implement the temperature sweep. The overall idea is to set the temperature to some starting value, then wait until the temperature has stabilized according to some criterion. In this lab, we use an approach similar to the “measure of spread” idea that we used in the second bug lab. Here, we calculate the average value of

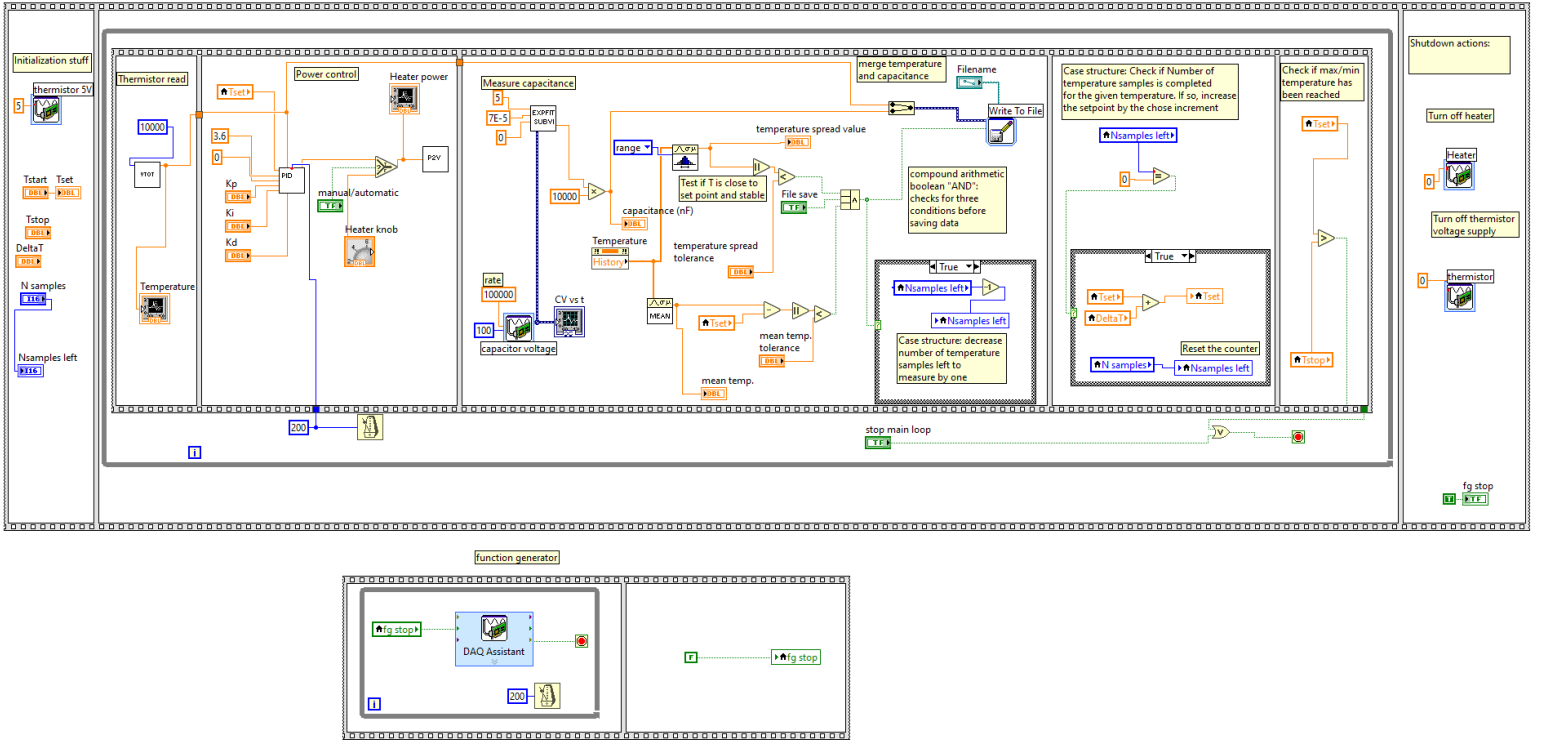


Figure 1: Block diagram of the starting VI, showing the overall structure.

of our temperature chart over some time interval (30 s or 150 points). Once the average value of the temperature data is close to the setpoint (e.g. within 0.05°C), then we save N_{max} capacitance values. Then we increment the temperature and repeat until we reach the maximum temperature. We use the concept of **local variables** to pass the values of quantities like starting temperature and temperature increment without having to use wires. To create a local variable, right click a particular control (e.g., T_{set}) and **create / local variable**. Note that local variables must be selected as either **read** or **write**, again by right clicking.

To summarize the overall main loop of the VI:¹

- i. Frame 1: measure the temperature
- ii. Frame 2: use PID to control the heater
- iii. Frame 3: if the average value of the temperature is within 0.05°C of the setpoint, then measure C and save the data. Then decrease N_{current} by 1 (this is done by the **case** structure, which is like an **if** statement in Python).
- iv. Frame 4: when N_{current} reaches zero, increase the temperature setpoint by dT and reset N_{current} to N_{sample} .
- v. Frame 5: if the (new) set point exceeds T_{max} (set by a control), or if the user

¹The programming method here is a simple example of a **state machine**.

presses the “Stop” button, then stop. Otherwise, execute the next `while` loop iteration.

One caution is that the capacitance (i.e., the decay constant) can take a VERY long time to come to its equilibrium value (days may not be enough!). You need to find some practical compromise when deciding how long to wait before going to the next set point. (You can use your `Bug3.vi` program to *quickly* get a feel for this. You will measure it more thoroughly later.)

- (c) **Finishing up.** A very important last step is to make sure the heater is OFF, either when the user aborts or when the sweep is finished. Make sure to output a 0 V signal to the heater before stopping. Do the same for the thermistor supply voltage, to be safe.

2. Running the experiment:

These measurements take a long time. Plan accordingly.

Now that you have your LabVIEW program working, you are ready to take data. Start with a short trial run, to make sure everything works (including the data analysis). Make sure that you have three columns of data: time, temperature, and capacitance. When you are ready, do a real run, going from close to room temperature (try 30 °C) to 100 °C, with an adjustable temperature increment. (Choosing 1 °C would be nice, but use a bigger increment if you are short on time.)

Next, do a run starting from 100 °C and going down to 30 °C. (You will have to make some obvious modifications to your VI.) Do you need to take as many points coming down as up? (Think about why we take data in both directions.)

With luck (and skill!), you will have seen a big peak in capacitance. The physics behind the peak is still not fully understood (see the discussion below), but a “phenomenological” law² that fits the data well *in thermodynamic equilibrium*, for $T \gtrsim T_{\text{peak}}$, is

$$C(T) = \frac{C_{\text{max}}}{1 + K(T - T_c)^\gamma}, \quad (1)$$

with T_c the transition temperature, C_{max} the capacitance at the peak, K an amplitude, and γ a power that is typically in the range of one to two. Make a quick plot of your C vs. T data in Python to confirm that you have a good set of data for increasing and decreasing T . Do you see a difference between the up and down curves? Do they agree in some range?

²“Phenomenological” is a fancy word one uses when one has a formula that fits the data without understanding why it works. Although the goal of curve fitting is to compare a model to data, sometimes, one does not have a good model. In that case, finding some function that fits the data can be a first step. Phenomenological fits are not great, but they can help predict new experiments, by interpolating or extrapolating, and they are better than nothing.

Finally, investigate how the capacitance continues to change even after the temperature stabilizes. Heating from around room temperature, acquire a capacitance time series for a setpoint of 40 °C. Acquire $N_{\text{sample}} = 10\,000$ points, if you have time. (How long should this measurement take?)

Repeat this measurement for a setpoint temperature significantly above the Curie temperature, for example at 80 °C. Compare this time series behaviour with the one at 40 °C.

4 Data analysis

[grading]

Fit Eq. (1) to your C vs. T data. Since the system is supposed to be in equilibrium at a temperature T , the fit will be meaningful in the temperature range where up and down curves agree. Why? You can also try fits to the entire range of temperatures past the peak. Compare the parameters you find in the three fits (to the range where the curves agree, the decreasing part of the “up” data and the decreasing part of the “down” data. Which set or sets of parameter values would have the most meaning? Is γ in the expected range?

For the C vs. time measurements: first, plot the form of T vs. t for 40 °C, and 80 °C. Determine the range over which T is basically stable for each one.

Next, plot the data for C vs. t for the two different temperature sets, 40 °C, and 80 °C. Restrict the time range of the C vs. t plot so that you only display points for which T is approximately constant (fully stabilized). Do you see a qualitative difference between the 40 °C, and 80 °C data? At low temperatures, you should find that the capacitance relaxes slowly, a consequence of the slow motion of ferroelectric domains (see below under **Requiem**). Plotting the two sets of data at the same scale can be clearer. (Hint: use offsets or normal and twin axes for the plot; align the time axes similarly).

Explore plotting the 40 °C data on a linear/log t scale. Try to fit the data to a function of the form $C(t)|_T = \text{const} \times \log(t_w/t)$, where the constant and t_w are fit parameters.

The slow change of the capacitance at constant T below T_c is an example of *creep* and occurs in a wide variety of physical systems ranging from crumpling of paper, conductivity of granular metals, and dielectric properties of ferroelectrics (our case). Details on the general theory can be found in the 2012 paper by Amir et al.

5 Requiem for a Bug

The big peak in the $C(T)$ plot comes because the material in the capacitor is *ferroelectric* and we have crossed a special temperature for ferroelectrics known as the *Curie temperature*, T_c . A ferroelectric material is the electrical equivalent of a magnet (the “ferro” in ferroelectric has nothing to do with iron but is coined as an analogy to ferromagnet). The molecules that make up the material all have permanent electric dipoles attached to them that can change their orientation. The microscopic state of the material is summarized in Fig. 2. In (a), at high temperatures, disorder (entropy) dominates and the dipoles have random direction from point to point. In (c), at low temperatures, energy considerations dominate, and the dipoles align. However, because the material is a polycrystal, there will be small domains, with the orientation in each domain varying from domain to domain. In (b), at a special temperature known as the Curie temperature, T_c , there is an intermediate situation between order and disorder. Here, the molecules like to align but they still have some freedom to reorient. The result is large groups of molecules that temporarily align in one direction, and then another, and another, ... , following a kind of collective motion. The Curie temperature T_c is known as a *phase-transition* temperature, between the disordered *paraelectric* phase of high temperature, with no dipole alignment, and the ordered “ferroelectric” phase at low temperature, with local electric dipole ordering.

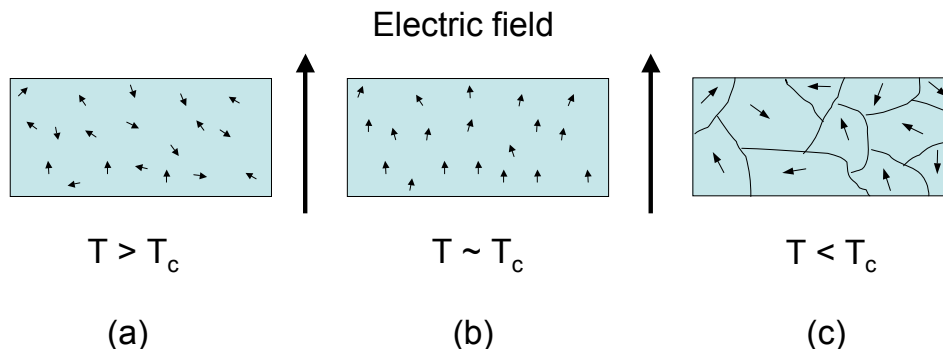


Figure 2: Ferroelectric material in a vertical applied electric field. (a) At high temperatures ($> T_c$), the dipoles fluctuate freely, with little interaction with their neighbors and show little aligning effect from the field. (b) Near the Curie temperature T_c , the dipoles are on the verge of ordering and are very susceptible to being aligned by the field. (c) At low temperatures ($< T_c$), in the ferroelectric phase, the domains with aligned dipoles block each other from reorienting.

What does all of this have to do with capacitance? Well, recall that the charge Q on a capacitor is $Q = CV$, so that we can think of C as being the amount of charge induced per voltage (or electric field) applied. In Fig. 2(a), an applied E -field will have little effect because thermal disorder will fight any ordering effects of the field. In Fig. 2(c), the ordering of dipoles will also not change much because each crystal grain is locked in place by all the others. However, in Fig. 2(b), at the special Curie temperature, a small field can lead to a significant reordering. We are at the “tipping point” where the molecules are just about

to order, and thus it takes only a small perturbation to decide what that ordering direction will be. Since a large amount of ordering implies a large charge to the surface, we conclude that at (or near) T_c , we expect the capacitance to become large.

The material we are using is known as barium titanate (BaTiO_3), doped with 10% tin (Sn). In pure BaTiO_3 , the expected $C(T)$ resembles the form sketched in Fig. 3(a). The Curie temperature is 120°C , which is a bit high to explore in our system. In our impure, “doped” material, the impurities are incorporated into the solid’s crystal lattice. The effect is to have T_c temperatures that are locally shifted by the presence of the nearest impurity molecules. Thus, one can think of the overall response of the capacitor as the sum of many different response curves, each shifted by a small amount up or down from the “pure” state. The situation is roughly as depicted in Fig. 3(b). Summing all those contributions up gives the observed response curve Fig. 3(c). The overall transition temperature is lowered, and the peak is broadened.

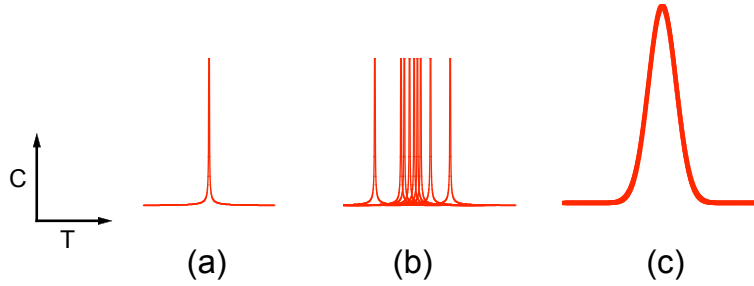


Figure 3: Capacitance of a ferroelectric material vs. temperature. (a) Response of a single-crystal. Peak is at the Curie temperature T_c . (b) Individual contributions from different crystal grains to the capacitance of a polycrystalline material. (c) Overall measured capacitance of a polycrystalline ferroelectric material. The individual contributions broaden the curve and make its shape more like a Gaussian.

Barium titanate mixtures are an important material industrially. They are used in many types of ceramic capacitors, the most common type. In addition, ferroelectric materials have interesting, nonlinear optical properties that are a key element in communications systems. And, in recent years, they have been studied as candidates for new ways to store energy. (Since $E = \frac{1}{2}CV^2$, a big C means a lot of energy.) See the [2024 paper in *Science*](#) by Zhang et al. for a taste of research in that field.

Our explorations here have just touched on many of the fascinating properties of BaTiO_3 . If we had more time, it would be interesting to explore how the capacitance relaxes to equilibrium values at different temperatures. It turns out that the crystal lattice of BaTiO_3 is cubic above the Curie temperature and tetragonal below. The change in lattice is accompanied by a change in volume. Thus, as one crosses the Curie temperature, all sorts of elastic stresses are generated because the Curie temperature is different at different locations in the material. Since stress itself changes the transition temperature, very complex effects can occur. These are behind the slow relaxations mentioned. Note also the shape of the $C(T)$ curve below T_c has the slight “shoulder.” This actually corresponds to another phase

transition, from the tetragonal phase discussed above to an “orthorhombic” phase.

In a more detailed experiment, one could also look at things like non-ideal impedance contributions to the capacitance (there is a small effective resistance, too, whose value depends on frequency). Measuring the charge rather than the voltage across the capacitor also turns out to be an interesting thing to do. We hope that you will want to go on to explore such issues in a future course.

As we mentioned at the start of these labs four weeks ago, the Bug experiment has all the features of a modern, computer-controlled experiment, including measurement and control of the crucial dependent variable (T), signal averaging, automated sweep, recording to disk, automated data analysis, etc. These are found both in research laboratories and in industrial testing. If you have made it this far, you are in good shape for working in either university or industry labs.