# *Insight* — Application Note 3.26

## Dielectric Measurements in Batch Reactors

### The batch reaction process

The monitoring of dielectric properties is useful for controlling polymerization in a batch reactor. Previous work has shown that DEA could observe the free-radical polymerization of methyl methacrylate,[1] where the change of log(*ion viscosity*) correlated with fractional monomer conversion and physical viscosity.

Dielectric cure monitoring has the advantage of real time, in-process measurement of polymerization, unlike standard laboratory gravimetric methods, which must be performed off-line.

Resins processed in batch reactors typically start with a high concentration of monomers, as shown in Figure 26-1. At this stage mobile ions can move very easily through the material, resulting in very low ion viscosities (low resistivities).

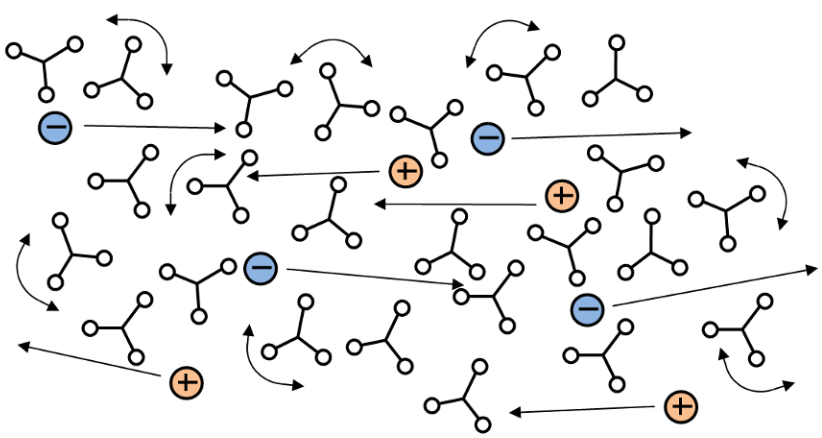

**Figure 26-1**
**Unreacted monomers**

When heated, these monomers react and bond to form polymer chains, represented in Figure 26-2. During this period the number of molecules decreases while their molecular weight increases. Mechanical viscosity also increases, as does resistance to the flow of mobile ions in an electric field. Dielectric cure monitoring measures this electrical resistance and is unique in enabling direct observation of material state in the reaction vessel.

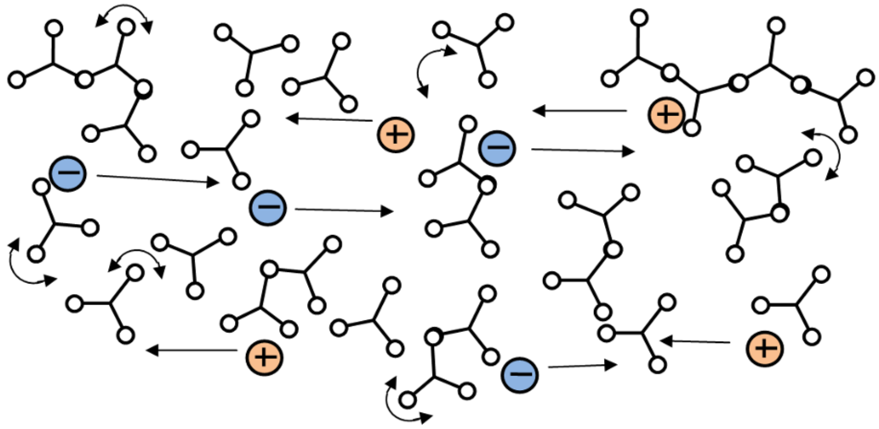

**Figure 26-2**
**Early stage polymerization**

As more and more monomers are polymerized, log(*ion viscosity*) increases in a fashion similar to the plot of Figure 26-3. *Note that only the change in log(ion viscosity) correlates with fractional monomer conversion*—the actual relationship and scaling factors must always be determined experimentally.

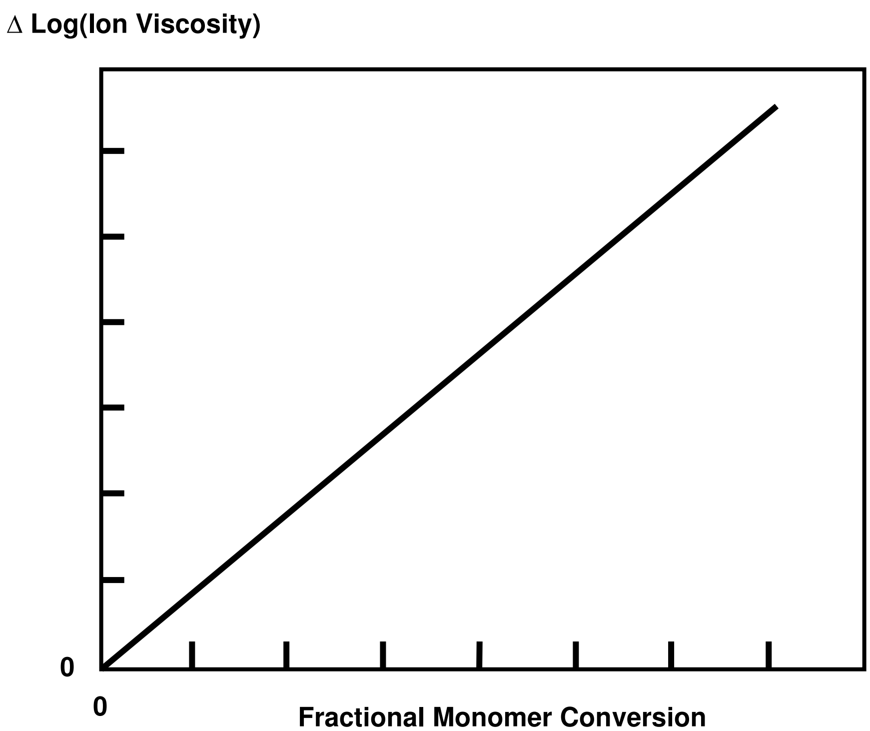


**Figure 26-3**
**Change in log(*IV*) vs. Fractional Monomer Conversion**

 

The value of log(*ion viscosity*) depends on the level of ions and impurities, and may not be the same from batch to batch. As a result, log(Ion Viscosity) alone is not a reliable measure of resin polymerization—only the *change* is useful.

## Standard instrument configuration

A computer with data acquisition software connects to a dielectric cure monitor through an RS-232 or USB serial port. The instrument connects to a dielectric sensor and a thermocouple, which are immersed in the batch reactor. This standard configuration is shown in Figure 26-4.

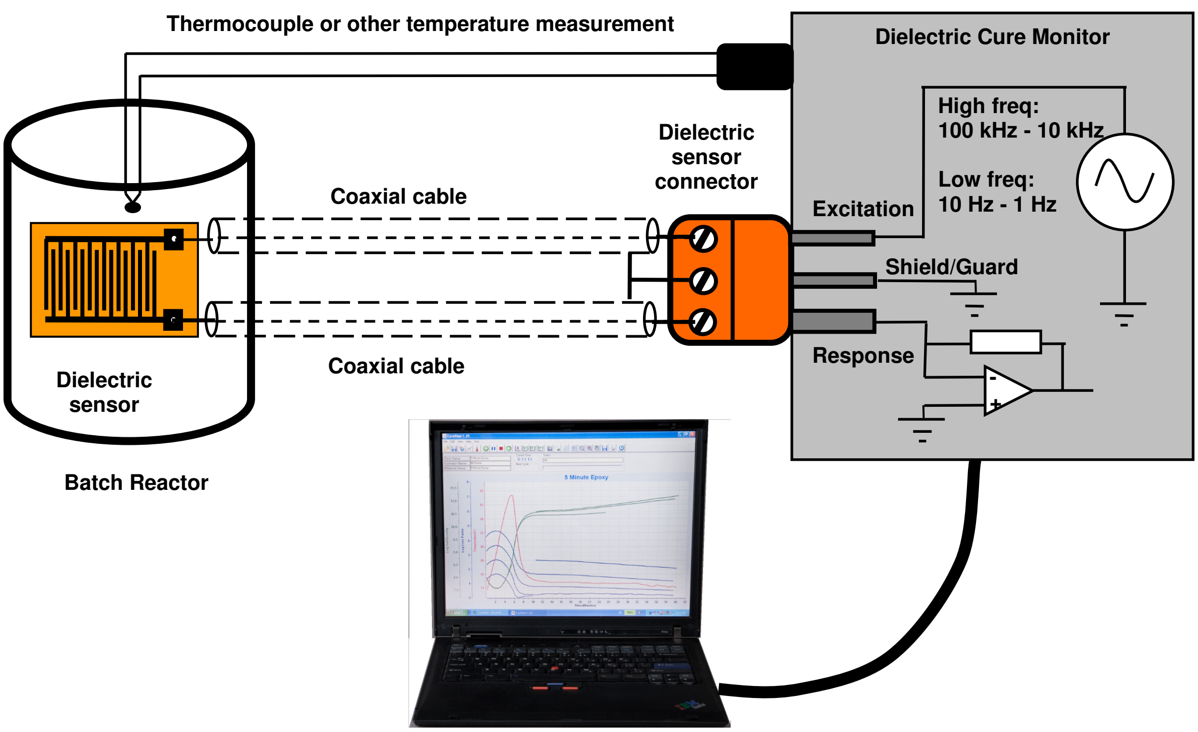


**Figure 26-4**
**Dielectric cure monitoring system for a batch reactor**

A dielectric sensor usually consists of a pair of interdigitated electrodes on a polyimide or ceramic substrate. A thermocouple measures the process temperature, which is important because dielectric properties vary with both the material's degree of conversion *and* temperature.

 

### Simplified sensor for batch reactors

In many cases the resin in a batch reactor has low ion viscosity during the entire process. Consequently, it is often not necessary to use a sensor with interdigitated electrodes. Because the electrodes occupy a large area and are close together, this type of sensor is very sensitive.

Thermosets at the end of cure have very high ion viscosities, so studying materials at this stage requires a sensor with correspondingly high sensitivity. However, when ion viscosity is low, the large signal from interdigitated electrodes can exceed the range of the dielectric cure monitor, resulting in bad measurements.

For batch reactors it may be better to use a simple electrode design with an output level more suitable to the instrument. Figure 26-5 shows how a pair of wires with exposed ends becomes a low sensitivity dielectric sensor.

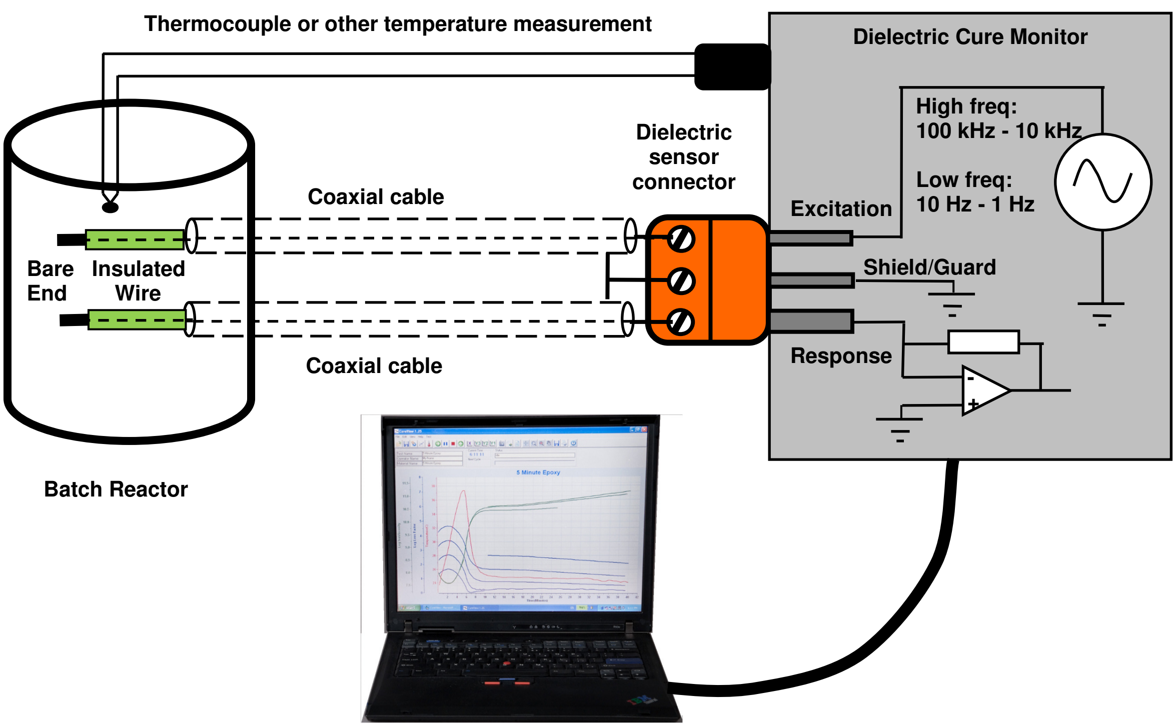


**Figure 26-5**
**Simplified sensor for use with a batch reactor process**

The optimum configuration must be determined by trial and error, but a reasonable design could start with 5 mm of exposed wire and 5 mm separation between the bare ends, as in Figure 26-6. Use solid wire 24 AWG or thicker, so it may easily formed and remains rigid after shaping. The insulation should be Teflon for ruggedness and chemical resistance, and the ends should be secured so their separation cannot change.

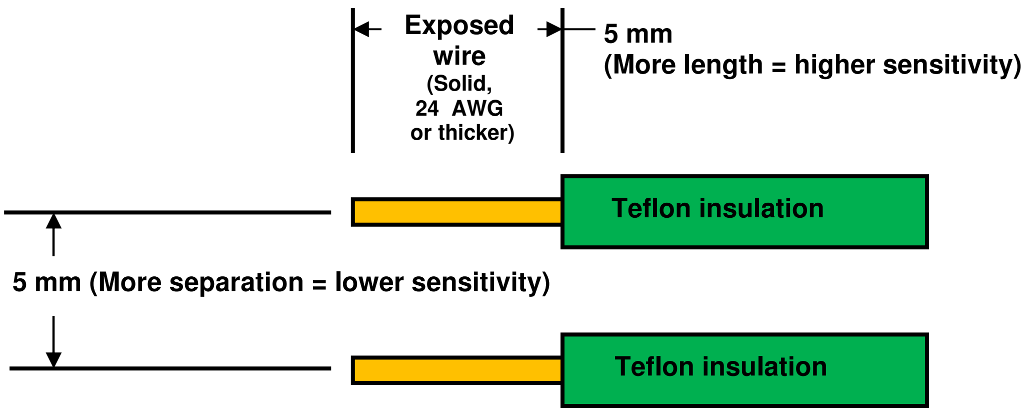


**Figure 26-6**
**Simplified dielectric sensor for low ion viscosity, example configuration**

Test the sensor by making a measurement in the material under test at the process temperature. Adjust the configuration of the sensor as necessary using the following guidelines:

- If the signal level is too low, decrease the separation or increase the length of the exposed wire
- If the signal level is too high, increase the separation or decrease the length of exposed wire

### Hazardous environments around batch reactors

Batch reaction processes use large volumes of resin and may produce volatile, flammable gases. The National Fire Protection Association (NFPA) Publication 70, the NEC and CEC categorize hazardous environments by *Class*, *Division* and *Group* as follows:

- **Class**—Type of flammable substance
    - **Class I**—Locations where flammable vapors or gases may be present
    - **Class II**—Locations where combustible dust may be present
    - **Class III**—Locations where easily ignitable fibers or flyings may be present
- **Division**—Area classification
    - **Division 1**—Ignitable substances exist under normal operating condition and/or caused by frequent maintenance or repair work or frequent equipment failure
    - **Division 2**—Ignitable substances are handled or used but normally in closed containers or systems, with escape only under abnormal operating conditions such as rupture of the container or breakdown of the system
- **Group**—Gas group, or flammability
    - **Group A**—*Example*: Acetylene
    - **Group B**—*Examples*: Hydrogen, butadiene, ethylene oxide, propylene oxide
    - **Group C**—*Examples*: Ethylene, cyclopropane, ethyl ether
    - **Group D**—*Examples*: Propane, acetone, ammonia, benzene, butane, ethanol, gasoline, methanol, natural gas

### Preventing ignition or explosion in hazardous environments

In hazardous locations, electrical equipment such as a dielectric cure monitor often requires *intrinsically safe barriers* in-line with sensors to prevent the ignition or explosion of flammable gases. Intrinsically safe barriers (I.S. barriers) are protective circuits designed to limit voltage and current to electrical devices. The limitations prevent dangerous energy discharge and depend on the classification of the hazardous environment and the requirements of the device.

Intrinsically safe barriers consist of a resistor, a fuse and one or two zener diodes, and must be connected to a ground point. Intrinsically safe barriers may handle positive, negative or AC signals, with configurations shown in Figure 26-7.

 

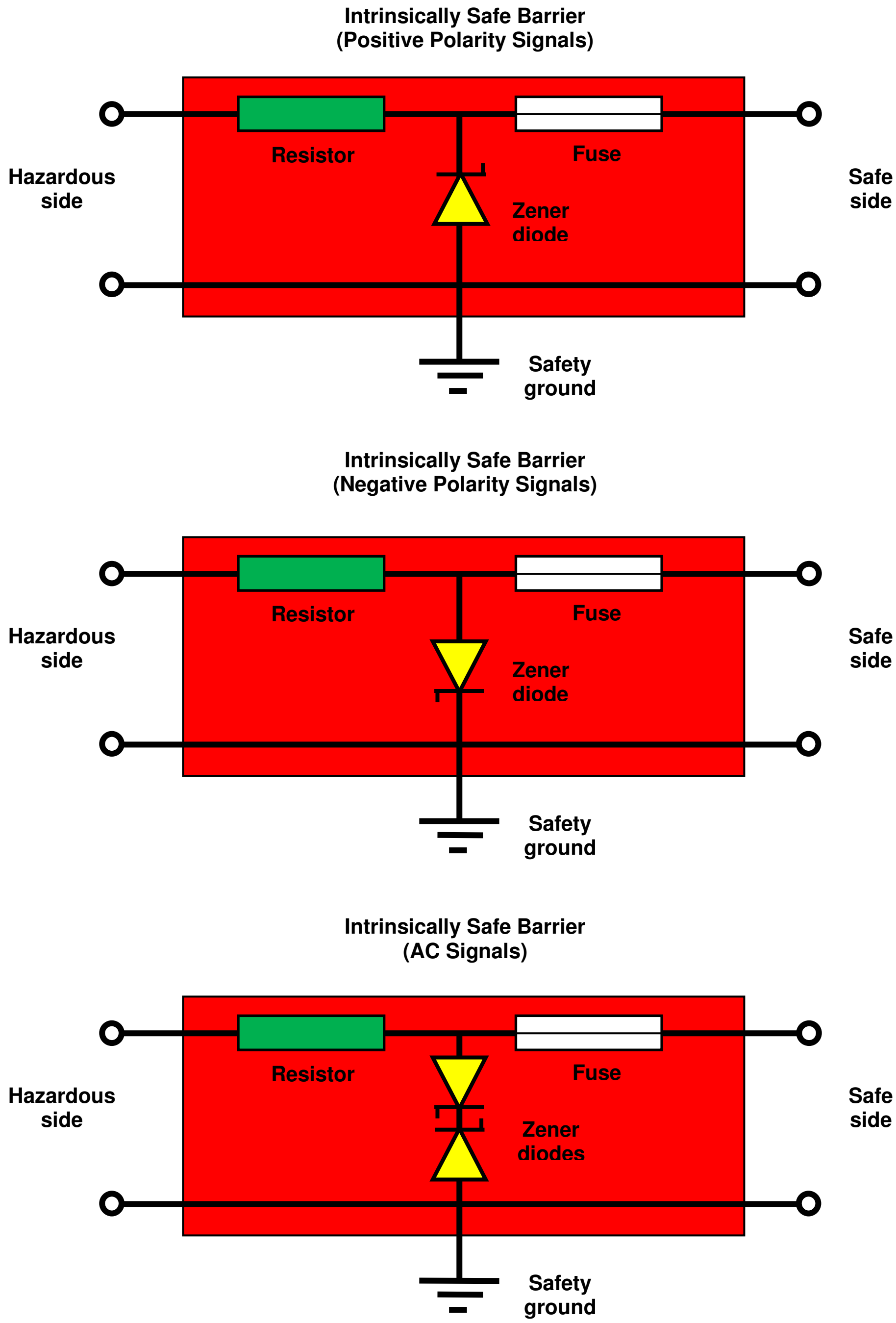


**Figure 26-7**
**Intrinsically safe barrier configurations**

 

## Explosion-Proof Enclosures

By ensuring that the available energy is insufficient to ignite the hazardous substance that may be present, intrinsically safe barriers remove the need for special *explosion-proof* enclosures around measurement circuitry. Article 100 of the NEC defines Explosion-Proof Apparatus as follows:

***Apparatus is enclosed in a case that is capable of withstanding an explosion of a specified gas or vapor that may occur within it and of preventing the ignition of a specified gas or vapor surrounding the enclosure by sparks, flashes, or explosion of the gas or vapor within, and which operates at such an external temperature that a surrounding flammable atmosphere will not be ignited thereby.***

NEMA Type 7 enclosures are designed to meet explosion-proof requirements in indoor Class I, Group A, B, C or D locations. They can withstand the pressures from an internal explosion of specified gases, and are able to contain the explosion and prevent ignition of a surrounding explosive atmosphere.

While intrinsically safe barriers can eliminate use of explosion-proof enclosures, *the user should always consult with the local or company safety authority to confirm equipment meets safety requirements*.

## Implementing Intrinsically Safe Barriers into a Batch Reaction Process

Identifying the category of hazardous environment is the first step to implementing dielectric cure monitoring in a batch reaction process. Then select appropriate intrinsically safe barriers for use with thermocouples and dielectric sensors. Install these barriers in an enclosure to support connectors to the sensors and connectors for cabling to the instrumentation.

For example, a batch reactor is in a Class I, Division 2, Group D hazardous environment. The company R. Stahl[2] is a manufacturer of intrinsically safe barriers and the following components may be used to assemble an Intrinsically Safe Interface:

- For thermocouples: R. Stahl 9001/01-050-150-101 I.S. barrier
  (Ω = 42 – 49, V = 1 – 3 VDC, $I_{MAX}$ = 20 – 61 mA)
- For dielectric sensors: R. Stahl 9001/02-093-390-101 I.S. barrier
  (Ω = 31 – 36, V = ±6 VAC, $I_{MAX}$ = 110 mA)
- Enclosure: R. Stahl 8150/5-0200-0300-150-3321 stainless steel enclosure

 

- **Note that the enclosure is not explosion-proof** but is safe and suitable when used with intrinsically safe barriers for Class I, Division 2, Group D environments

This enclosure with the intrinsically safe barriers is the *Intrinsically Safe Interface* and is shown schematically with the dielectric cure monitoring system in Figure 26-8. Because of the added circuitry in the I.S. barriers, dielectric measurements may have frequency or accuracy limitations compared to the base instrument.

***IMPORTANT***: For safe operation, the Intrinsically Safe Interface and the Dielectric Cure Monitor must both be connected to the same electrical ground.

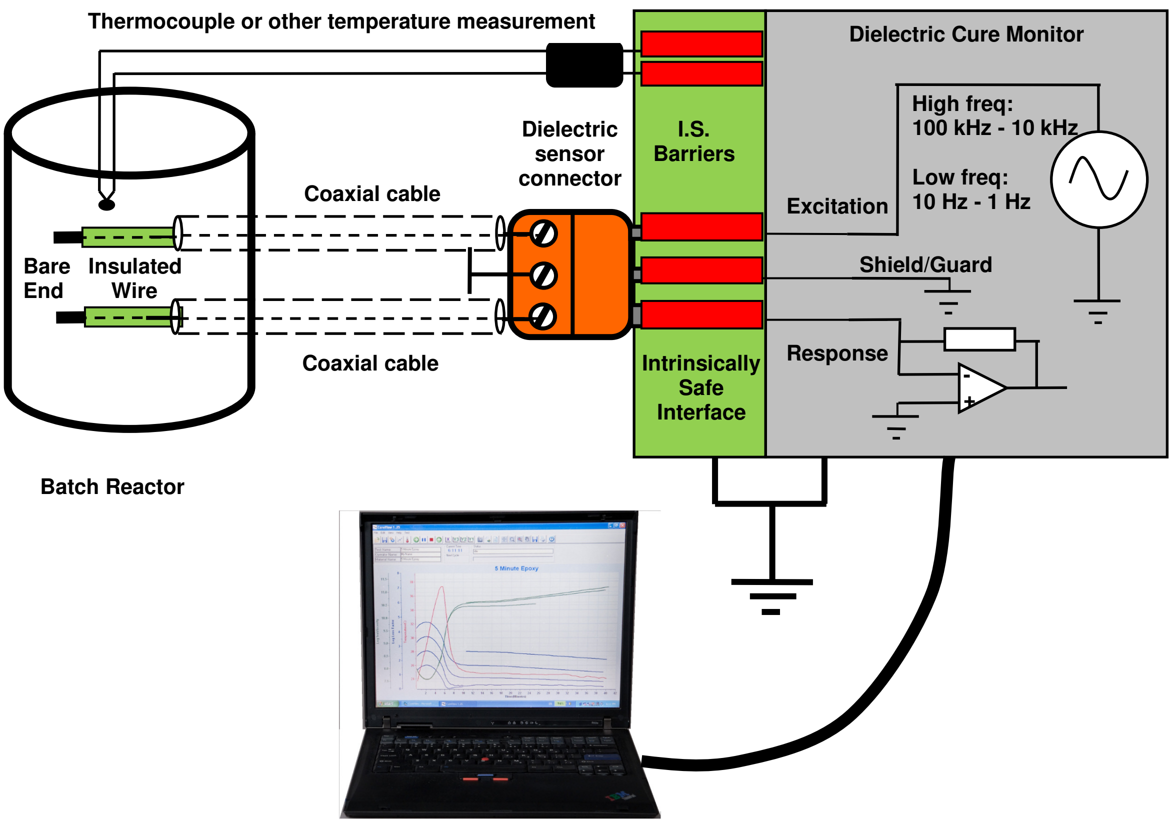


**Figure 26-8**
**Apparatus for single channel of intrinsically safe process monitoring in a batch reactor**

 

Figure 26-9 schematically shows a full, four-channel system with cabling, using an LT-451C Dielectric Cure Monitor.[3]

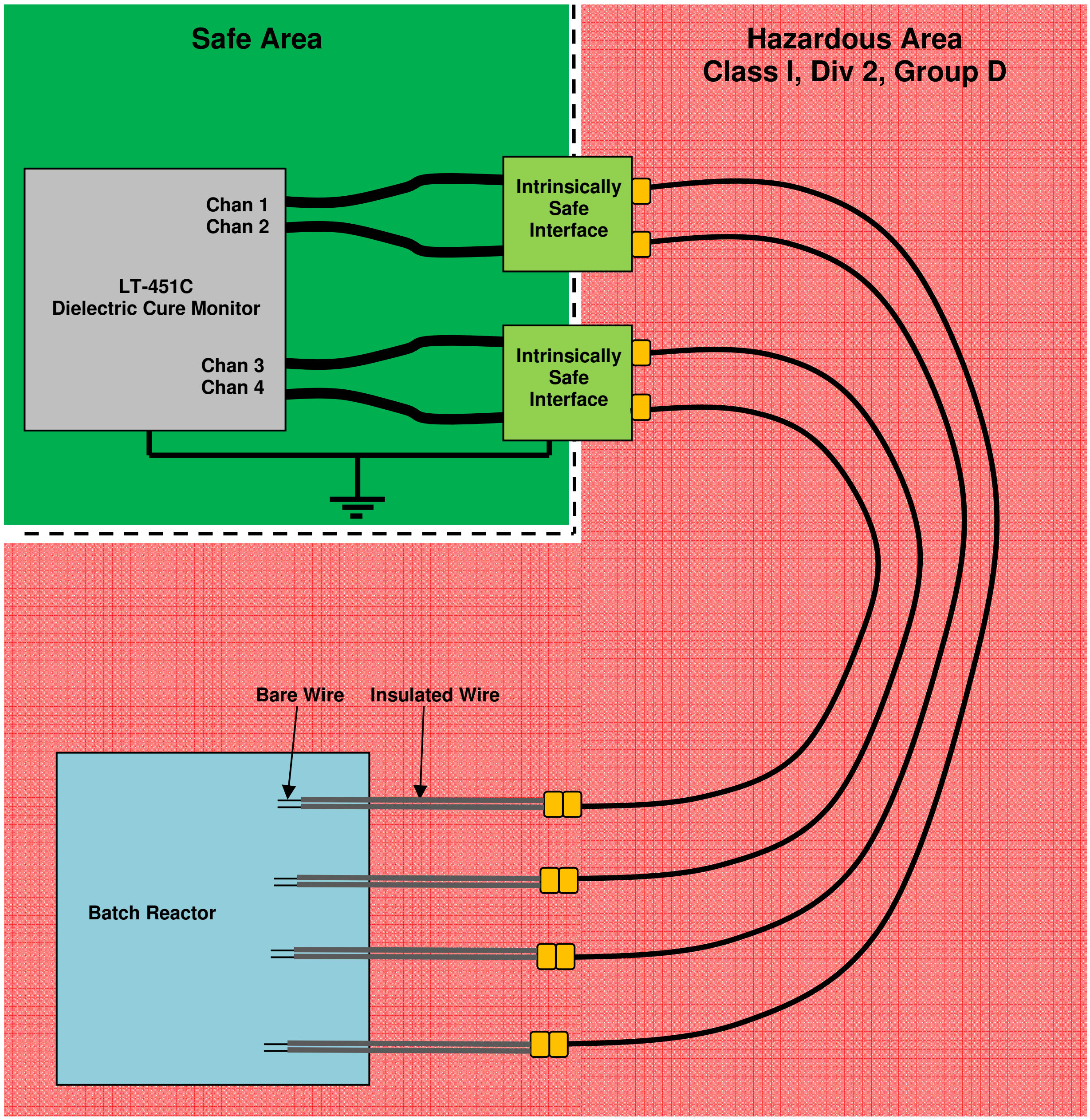


**Figure 26-9**
**Four-channel dielectric cure monitoring system with intrinsically safe barriers**
**(Computer and thermocouples not shown for clarity)**

The intrinsically safe barriers require a ground reference and the enclosure must be grounded to ensure their protective function. For good measurements, the LT-451C Dielectric Cure Monitor must have the same ground as the barriers. Figure 26-10 shows the ground connection points of the Intrinsically Safe Interface and the LT-451C. These points must be connected to the ground reference. **This ground reference must be the same as the safety ground of the AC Mains supply.**

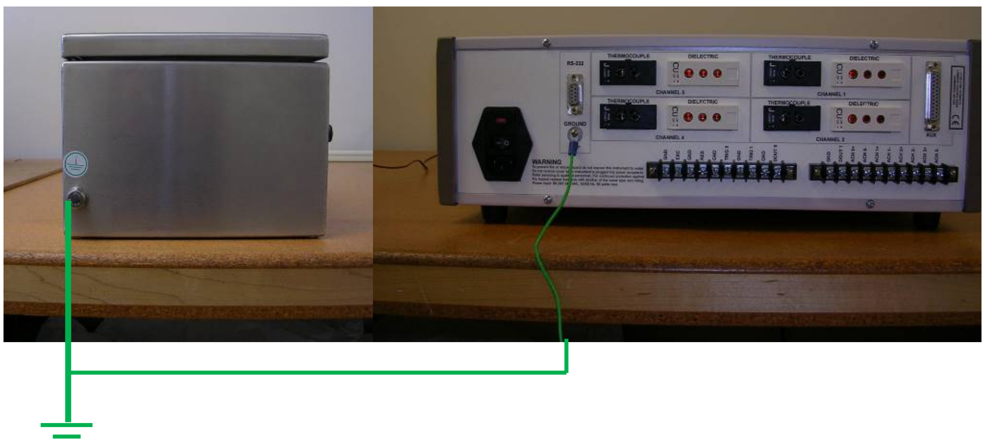

**Figure 26-10**
**Grounding points of the Intrinsically Safe Interface and LT-451C Dielectric Cure Monitor**

Dielectric cure monitoring can provide on-line, real time information about the polymerization in a batch reaction process. Batch reactors, however, often operate in hazardous environments with flammable gases. Intrinsically safe barriers can be used with thermocouples and dielectric sensors to limit energy below the ignition point of these gases and prevent explosion. A dielectric cure monitoring system with an Intrinsically Safe Interface can enable valuable process control of batch reactions in large scale resin production.

 

## References

1. Crowley, Timothy J. and Choi, Kyu Yong, In-line dielectric monitoring of monomer conversion in a batch polymerization reactor, *Journal of Applied Polymer Science*, Feb. 28, 1995, pp 1361-1365

2. R. Stahl, Inc., Stafford, TX USA. https://www.rstahl.com/

3. LT-451C Dielectric Cure Monitor, manufactured by Lambient Technologies, Cambridge, MA USA. https://lambient.com

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