Electrical heating with polyimide-insulated magnet wire

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Polyimide-insulated copper magnet wire is capable of continuous service above 200 °C and can be used intermittently above 300 °C. For the construction of heaters where fast heating and cooling or novel geometries are required it offers a versatile alternative to resistance wire.

Heaters for laboratory instruments or experiments usually employ a resistance wire such as Nichrome. Commonly, wire with a mass for a few g is coupled thermally to some load with a mass of a few hundred g. Often, in order to achieve a satisfactory heating rate, the wire is driven far from equilibrium. The resulting high temperature requires that the wire be supported by a refractory electrical insulator. A great variety of reliable heaters based on this approach are available commercially. Where very rapid heating rates, novel geometries, or miniaturization are required, the availability of polyimide-insulated copper magnet wire[1] capable of continuous service above 200 °C permits a different approach. Such wire can be close wound on a spool, packed in a bed, or wound directly on an object to be heated, as long as the voltage drop between segments of wire that come in contact is limited to a few V. Three examples are given below where such wire was used to construct fast heating devices for a microchemical process. A number of additional experiments are described which demonstrate the scope of this approach.

An apparatus for the remote chemical processing of ^{11}C–CO_2 (20-min half-life) provides three examples of the advantageous use of copper wire for rapid heating. The process is described here in abbreviated detail only (Fig. 1), in order to indicate the scale of the reactions and temperature control situations encountered. In the overall process, ^{11}C–CO_2 (10^{-7} mol total CO_2 with an activity of 1 Ci) is produced by bombarding N_2 with protons. The CO_2 is separated from the bulk of the N_2 by a cold trap immersed in LN_2, then purged into 100 μl of a solution of $LiAlH_4$ in tetrahydrofuran. The solvent is removed by heating at 120 °C in a stream of N_2. After cooling, 50 μl of aqueous phosphoric acid is added, and the ^{11}C–methanol liberated is distilled at 80 °C into a stream of N_2. The methanol passes through a column of aqueous HI at reflux, and the resulting ^{11}C–iodomethane is trapped in a Teflon loop (− 50 °C) containing a substrate to be methylated. The loop is closed under pressure and heated at 60 °C to effect the reaction.

The cold trap (Fig. 2) for concentrating the CO_2 from the N_2 stream consists of a continuous length (54 m, 30 Ω) of 32 AWG polyimide-insulated copper magnet wire packed into a Pyrex U-shaped tube. The wire provides both an inert matrix on which the CO_2 is trapped and also a means for rapid heating during the purge step. The trap is cooled by immersion in LN_2. During the trapping step, 12 V is applied across the wire to maintain its temperature at approximately 90 K to prevent condensation of liquid nitrogen in the trap. To purge the CO_2 from the trap, the Dewar of LN_2 is lowered while 120 V is applied across the wire allowing rapid, uniform heating.

The temperature sequence required for reduction of the CO_2, and subsequent distillation of methanol, is achieved by a novel heat gun (Fig. 3). A continuous length of polyimide-insulated 32 AWG magnet wire (37 m, 20 Ω) is packed into one end of a Pyrex tube. A thermistor, connected to a phase-proportional controller,[2] is imbedded in the wire packing near the top. A flow of air is directed through the bed at the reaction vessel. The intermediate cooling step is accomplished by momentarily reversing the air flow.

For the conversion of ^{11}C–CH_3OH to CH_3I the aqueous HI is maintained at reflux by a positive temperature coefficient heater (doped barium titanate) removed from a commercially available glue gun.[3]

Temperature control for the trapping and subsequent reaction of the ^{11}C–iodomethane is accomplished by the device shown in Fig. 4. In this case 28 AWG Teflon-insulated copper wire[4] (40 Ω) is wound in a spool around a core consisting of an aluminum tube and also the perforated segment of a heavy-walled Teflon tube connected to a liquid CO_2 supply. A thermistor is embedded in the winding. Cooling to − 50 °C is accomplished by admitting several brief pulses of liquid CO_2 to the Teflon tube. The wire winding, which acts as a matrix for the controlled expansion of the CO_2 and as a medium for heat exchange, rapidly becomes impregnated

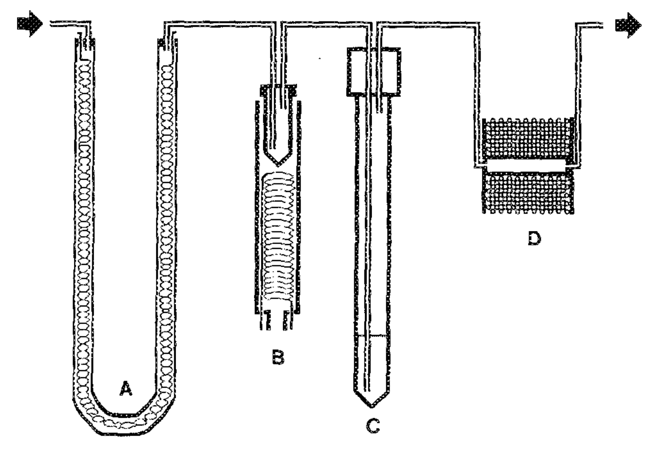

FIG. 1. Simplified diagram of an apparatus for processing ^{11}C–CO_2. (A) Cold trap (LN_2 Dewar not shown), (B) heat gun for reduction with $LiAlH_4$, (C) refluxing HI, (D) reactor for methylation with ^{11}C–iodomethane.

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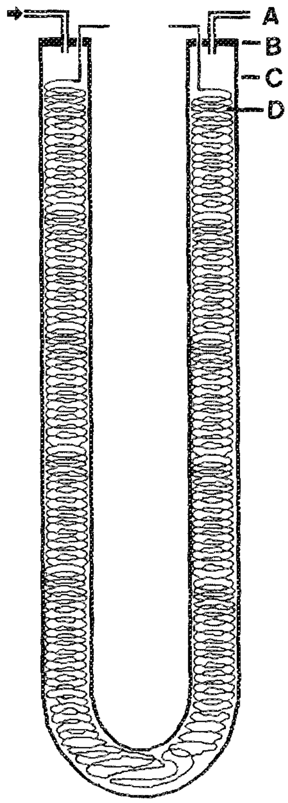

FIG. 2. Cold trap for concentrating [11]C–CO_2. (A) Teflon transfer line, (B) epoxy seal, (C) Pyrex U-tube, 7-mm i.d. × 500 mm, (D) wire packing.

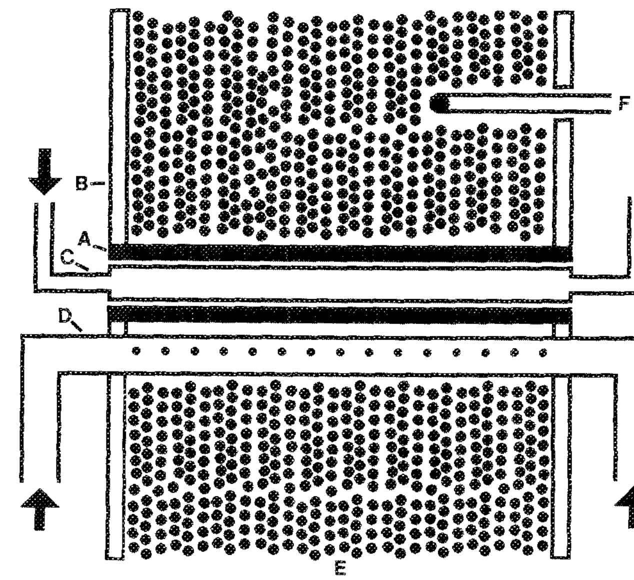

FIG. 4. Reactor for methylation with [11]C–iodomethane. (A) Aluminum tube, 6.4-mm o.d. × 50 mm, (B) Teflon end piece, (C) Teflon reaction loop, (D) liquid CO_2 supply line: heavy-walled Teflon, 3.2-mm o.d., perforated with 28-gauge needle, (E) wire winding, (F) thermistor.

with solid CO_2. During the subsequent heating step, 120 V is applied across the copper winding by a phase-proportional temperature controller to raise the temperature to 60 °C.

In addition to the above, the following demonstrations were done to indicate the scope of applications and to demonstrate the remarkable durability of the polyimide insulation:

Conforming heating block (Fig. 5). It is often difficult to machine a heating block to conform to a complex shape such as a conical glass vessel. Though there are other solutions to the problem solved by this example, it serves to demonstrate the ability of polyimide insulation to maintain its electrical

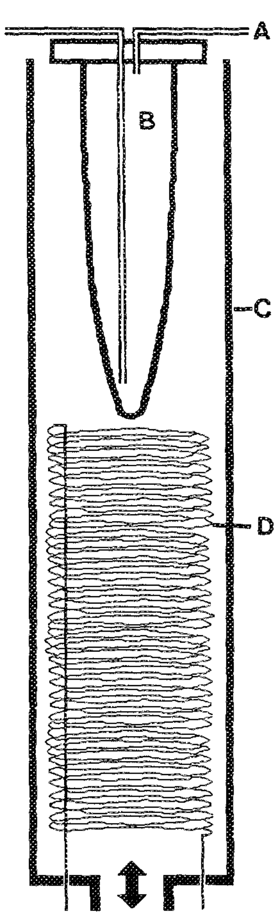

FIG. 3. Heat gun with reaction vessel. (A) Teflon transfer line, (B) Pyrex vessel, (C) Pyrex tube, 11-mm i.d. × 190 mm, (D) wire packing. Arrow indicates reversible air flow.

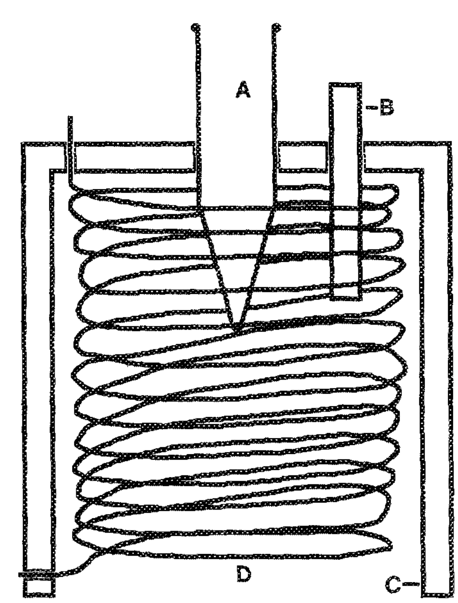

FIG. 5. Conforming heating block. (A) Conical Pyrex tube, (B) Teflon rod for thermocouple well, (C) Teflon mold, 28-mm i.d. × 32 mm, (D) wire packing.

integrity under prolonged exposure to high temperatures. A continuous length of 32 AWG polyimide-insulated magnet wire (40 m, 21 Ω) was packed into a Teflon mold around the base of a conical glass vessel. A Teflon rod was inserted into the mold to provide a thermocouple well. The assembly was heated in an oven to 300 °C, and molten lead was poured in and agitated to fill the interstices in the wire bed. Application of 24 V across the wire maintained the block at an equilibrium temperature of 280 °C, and this temperature was maintained for 6 days with no apparent damage. Increasing the voltage to 26 V gave an equilibrium temperature of 310 °C. The wire failed suddenly, however, after a few hours at that temperature.

Free-standing heated column. It is frequently necessary to heat a segment of a manifold or distillation apparatus. This is often accomplished by wrapping a heating tape around the segment. The following experiment shows that a layer of copper magnet wire can be used to obtain relatively high temperatures. A Pyrex tube (7 mm × 100 cm) was close wound over its entire length with a single layer of 32 AWG polyimide-insulated magnet wire (70 Ω). The ends of the wire were cemented in place with epoxy resin. This assembly was inserted into a length of 9-mm-o.d. Pyrex tubing to provide support, thermal insulation, and a controlled atmosphere. At 120 V in air the column reached an equilibrium temperature of 230 °C in 8 min. No degradation of the insulation was detected after 4 days of continuous heating. An atmosphere of N_2 was then applied to the outer tube and the voltage was raised gradually over 6 days to 237 V. A gradual darkening of the insulation occurred. At 370 °C the darkening progressed rapidly over the course of a few hours. At 237 V the equilibrium temperature was 430 °C, and the wire became totally black within 1 day, while some resinous material condensed on the inner surface of the outer tube. No further change in the appearance of the wire, equilibrium temperature, or room-temperature resistance was observed after continuous heating for 8 days. The carbonized insulation was shiny, and no cracks or discontinuities could be detected. The column was cooled and the N_2 atmosphere replaced by air. The carbonized insulation appeared to be stable in air up to about 250 °C, but began to decompose over the course of several hours at 320 °C.

Internally heated transfer line. Condensation of water or other volatiles within long transfer lines is a problem often encountered and difficult to solve inexpensively. Reasonably uniform heating can be easily accomplished by placing the heating element inside the tubing rather than outside. A continuous length of 28 AWG Teflon-insulated magnet wire was threaded through an 8-m length of Teflon tubing (2-mm i.d.). A flow of air (1 l/min) was maintained, and the tube was heated by applying 23 V (7 A) across the wire. Samples of water and camphor (100 mg) evaporated at the inlet were recovered at the outlet within 1 min. The Teflon insulation developed cracks from thermal stress below 200 °C. Polyimide insulation would have been preferable, but no such wire with the desired diameter was at hand for the demonstration.

Injection of a thermal marker into a fluid stream. A 9-m length of 32 AWG copper wire (5 Ω) was packed into a 6-cm segment at one end of a 14-m length of 7-mm-i.d. Tygon tubing. A flow of water (1.3 l/min) was maintained through the tube. The wire packing was heated by applying 120-V, 200–800-ms pulses across the wire. The resulting thermal pulses, though broadened, could be readily detected by a thermistor near the outlet of the tube. The use of a thermal pulse to measure linear gas flow is the basis of a commercially available instrument.[5] The present demonstration suggests that the principle might be used, for example, to measure fluid flow in experimental physiological preparations. Copper wire with polyimide insulation is readily available as small as 44 AWG,[1] permitting small heaters to be made *in situ* in any required shape.

Miniature heat gun (Fig. 6). A Pyrex capillary (1.3-mm i.d. × 40 mm) was packed at one end with 44 AWG polyimide-insulated magnet wire (3.3 m, 20 Ω). A flow of N_2 (50 ml/min) was passed through the capillary while the voltage across the wire was slowly increased. At 17 V paper was scorched 1 mm from the tip of the capillary, and at 22 V small amounts of solder could be melted (~320 °C). At this temperature the insulation darkened rapidly and the wire failed after 30 min. The expedient of packing a bed of wire to form a heating element makes it possible to construct very small heaters simply, where scaling down conventional resistive heaters would be difficult.

Because of the thin insulation and very rapid rates of heating in the devices described, certain precautions were taken in their construction. Sensors were placed in intimate contact with the wire beds at points where the highest temperatures were expected. In packed beds, contiguous segments of wire were not permitted to bear more than a small

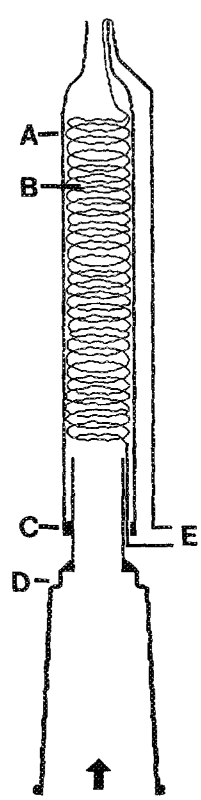

FIG. 6. Miniature heat gun. (A) Pyrex capillary, (B) wire packing, (C) epoxy seal, (D) 18-gauge hypodermic needle, (E) wire leads.

fraction of the total voltage drop (though the example of Fig. 5, where all points of the wire were in intimate contact with an electrical conductor, temporarily withstood 26 V above 300 °C). In cases where a failure may have serious consequences, a second sensor and limit switch are desirable.

In the applications where rapid temperature change and rapid thermal exchange with a fluid medium are the object, a shorter length of aluminum wire of equivalent diameter would permit devices that were significantly smaller, lighter, and faster.[6] A number of wire suppliers[7] are able to apply a polyimide coating to aluminum wire on a custom basis.

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[1]Hudson Wire Co., Trenton, GA, 30752. The insulation is a heat curable, aromatic polyimide enamel, PYRE-M.L.® marketed by DuPont, Polymer Products Dept., Industrial Films Div., Wilmington, DE 19898. It is identical chemically to Kapton® film.

[2]Model 70A, RFL Industries, Inc., Brandon, VT 05733.

[3]GTE Products Corp., Standish, ME 04084.

[4]W. L. Gore, Inc., Newark, DE. This wire was used instead of polyimide-insulated wire because it was readily available in the required diameter.

[5]Molytek, Inc., Pittsburgh, PA 15222.

[6]Specific heat × volume for aluminum: 0.115 cal/g × 2.7 g/cm^3 = 0.31 cal/cm^3. For copper: 0.061 cal/g × 8.9 g/cm^3 = 0.54 cal/cm^3.

[7]For example, MWS Wire Industries, Westlake Village, CA 91362; Rea Magnet Wire Corp., Fort Wayne, IN 46806.