Table II. Contamination Test for Hg, Cd, and Pb[a]

tube	Hg, found			Pb, found			Cd, found		
	n	$\bar{x}$, ng	s	n	$\bar{x}$, ng	s	n	$\bar{x}$, ng	s
Ti-PTFE	7	0.80	0.17	7	0.87	0.57	7	0.16	0.20
quartz	8	0.84	0.22	6	1.00	0.65	6	0.19	0.11
1 mL of HNO₃, blank runs	3	0.91	0.02	3	0.98	0.12	3	0.08	0.00

[a] 1 mL of HNO_3 (p.a.) was heated at 150 °C for 1 h. n = number of independent measurements, $\bar{x}$ = mean value, s = standard deviation.

Table III. Recovery Test for Hg[a]

tube	amt of Hg found, ng
Ti-PTFE	103.0
	100.0
	98.6
quartz	102.8
	100.0
	99.0
mean	100.6, s = 1.9

[a] To 1 mL of HNO_3 containing 0.9 ng of Hg (cf. Table II) was added 100 ng of Hg as $HgCl_2$ and the mixture heated at 150 °C for 1 h.

tubes has not been determined for the same reason by probably they are even stronger than the thick-walled quartz tubes.

By use of these fragile inner parts and a much stronger shield of stainless steel the safety of the bomb increases—if the inner part bursts the pressure will slip out through the thread.

The limiting pressure, causing the inner tube to burst, has not been determined. Nevertheless, an attempt was made with a tube (outer diameter 19.5 mm, wall 1.75 mm) which was half filled with water. The temperature was gradually increased to a maximum temperature of 280 °C (implying a pressure of 66 kp/cm²), but this was not enough to cause it to burst. The important thing is to know the amount of sample and acid that can be used at a given temperature.

Recommended cooling procedure is as follows: Place the bomb in water to half its height. This will cool the sample solution without causing large temperature changes in the PTFE lid which might lead to gas leakage. The bomb can then be opened after about 10 min.

The contamination was measured in a series of analyses (blank runs) for Hg, Cd, and Pb, see Table II. The losses was determined by recovery tests for Hg; see Table III. The PTFE material will have a rather high Hg contamination (5–10 ng per run and tube) unless it is pretreated before the tubes are manufactured. The material was therefore prepared by heating at 250–270 °C for several days.

As a further test of this bomb technique the NBS standard 1577 bovine liver was analyzed for Pb and Cd, giving 335 μg/kg

of Pb (range 60), 280 μg/kg of Cd (range 40), and 16.4 μg/kg of Hg (range 3.8), as mean of three analyses. The certified values are 340 ± 80, 270 ± 40, and 16 ± 2, respectively. The precision of the technique at low concentrations is shown by analysis of sample of freeze-dried cod which gave 65 μg/kg of Pb (n = 8, s = 15) and 6.2 μg/kg of Cd (n = 10, s = 1.2).

CONCLUSIONS

There are three advantages of this bomb construction compared to others described in the literature. First, and most important, the seal is not achieved by pressing PTFE against PTFE, but the rim of a quartz tube against a PTFE lid, which makes it possible to apply a higher pressure on the seal without causing deformation. Further, the contamination is low because of the space between the seal and the outer steel tube, and, finally, it is easy to evaporate surplus acid by placing the quartz tube on a hot plate.

The Ti-PTFE inner tube should only be regarded as a substitute for the quartz tube (e.g., if HF is utilized); in the long run the sealing of the quartz tube is superior to that of the Ti-PTFE tube.

The results of the bomb tests show that this new bomb technique is a good method of achieving fast and safe digestion, low blanks, good precision and accuracy (on Bovine Liver 1577 standard reference material).

ACKNOWLEDGMENT

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Temperature Monitor with Dual-Channel Platinum Resistance Elements

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Often seemingly outdated laboratory equipment may be easily modified to serve a new purpose at little expense. Such is the case with a laboratory oven we have refurbished by the addition of a digital temperature monitor to house our ion

mobility spectrometer (1). Unlike the majority of gas chromatography detectors which may be conveniently heated by cartridge heaters, the ion mobility spectrometer requires a heated and insulated enclosure due to high voltages present
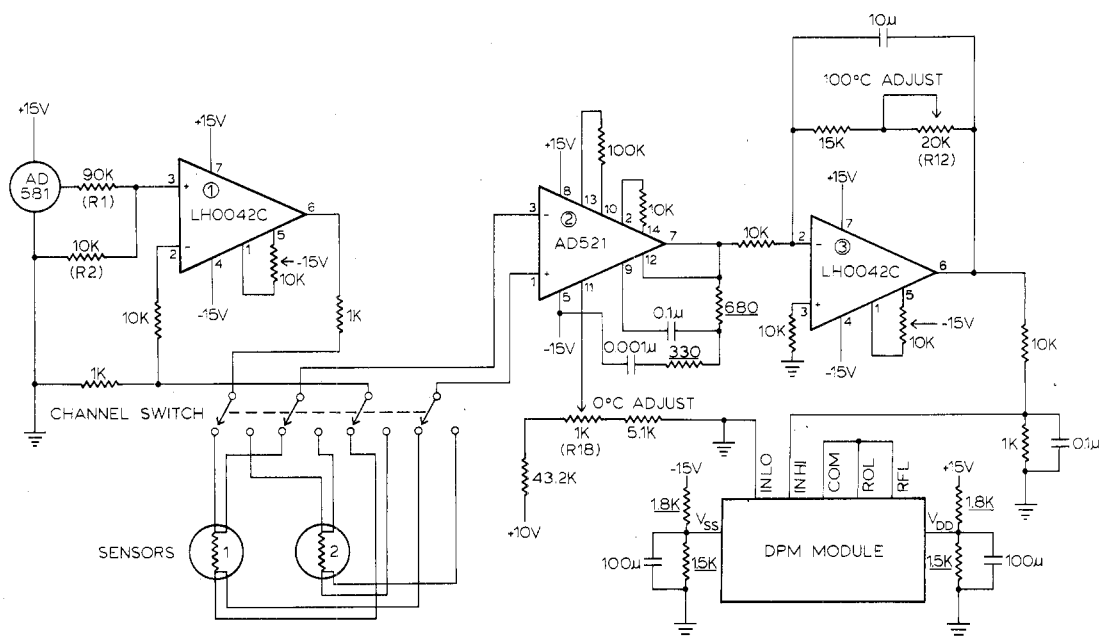
Figure 1. Circuit diagram of temperature monitor. A ±15 V power supply, not shown, is required. Underlined resistor values are ±5%; all others are ±1%.

on the drift tube. Because ion mobilities on which the instrument's tunable selective capabilities are based are temperature dependent, accurate monitoring of detector temperature is imperative.

An inexpensive dual channel temperature monitoring circuit has been designed to measure the temperature of the ion mobility spectrometer and a heated transfer line connecting the instrument to a gas chromatograph. The circuit employs platinum resistance temperature elements (0.062 in. (0.16 cm) diameter × 1 in. (2.5 cm) long) and is capable of resolving 0.02 °C. Due to the small size of the elements and accuracy of the electronics, a wide variety of applications is possible.

EXPERIMENTAL SECTION

Two matched Model 78-0012-0120 platinum resistance temperature elements (Rosemount, Inc., Minneapolis, MN) are used as temperature sensors. A four-pole double throw toggle switch selects which sensor is in use. The sensors are supplied by a constant current source. Changes in temperature which cause linear variations in resistance result in linear variations in voltage across the sensor. This voltage is scaled and offset so that the temperature can be directly read from the display of a low cost digital panel meter (PCIM 176 ± 200 mV DPM Module, Printed Circuits International, Inc., Sunnyvale, CA, or equivalent). A circuit diagram is provided in Figure 1.

CIRCUIT DISCUSSION

The monitor's simplicity is due to the temperature characteristics of the platinum resistance elements. Changes in resistance are directly proportional to temperature over a wide range (-50 to +250 °C). An integrated circuit (IC-1) and its

associated components maintain a stable 1 mA current through the selected sensor. The AD581 10-V reference and the voltage divider constructed from R1 and R2 provide a precise 1.000 V reference for IC-1. IC-2, IC-3, and their components scale and offset the voltage across the sensor for use by the DPM, such that the temperature can be directly read on the display. IC-2, a medium grade instrumentation amplifier configured for a gain of -20, has a high common mode rejection ratio which is used to cancel out any 60-Hz ac noise induced on the long sensor wires. Trim pots R18 and R12 are adjusted to set 0 and 100 °C, respectively. The final result is sent to the DPM with a full scale of 200 mV.

It should be noted that the sensor detection wires are separate from the current supply leads. This avoids errors due to the variations in voltages on the supply leads which change resistance with temperature. When the monitor is used below 0 °C, the current source should be adjusted below 1 mA to lower current heating in the sensor. Multiple sensors can be used as long as they have matched temperature-resistance characteristics. Any good, low-cost FET input OP-AMP can be substituted for the LH0042C's (such as LF411). The circuit can resolve 0.02 °C although it is currently limited to 0.1 °C by the DPM. Construction cost, approximately $95 for parts, could be lowered if less precise performance is required.

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Modified Soxhlet Extractor for Room-Temperature Extraction

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Most continuous extraction procedures for solids utilize the Soxhlet extractor, an efficient and widely used laboratory tool. For characterization of environmental particles such as coal fly ash, spent oil shale, atmospheric and urban particulates,

etc. by leaching procedure it is important to obtain quantitative information about the nature and amounts of potentially toxic elements leached from these materials. As a consequence, it is intended to establish a laboratory-based system