A portable near infrared-based turquoise analyser

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Background

An exposure of fraudulent turquoise sales (presented on a New Mexico TV station) included a laboratory test in a strongly heated crucible. The undesirability of destructive testing is obvious. Fake turquoise is made of coloured plastic and since plastic materials are good candidates for analysis by NIR, this approach seemed self-evident. After contacting several jewelry stores (where they were horrified that I would ask for samples of *fake* stones!) and wholesale jewellers' supply houses, proof of principle was established using a commercial instrument from FOSS/NIRSystems, Model 6500 (Silver Springs, Maryland, USA) with remote reflectance attachment. Untreated turquoise is not much to look at and it is almost invariably treated in some fashion. Figure 1 shows an initial Log 1/*R* tracing, a derivative treatment and cluster diagrams involving natural and stabilised turquoise and plastic fakes.

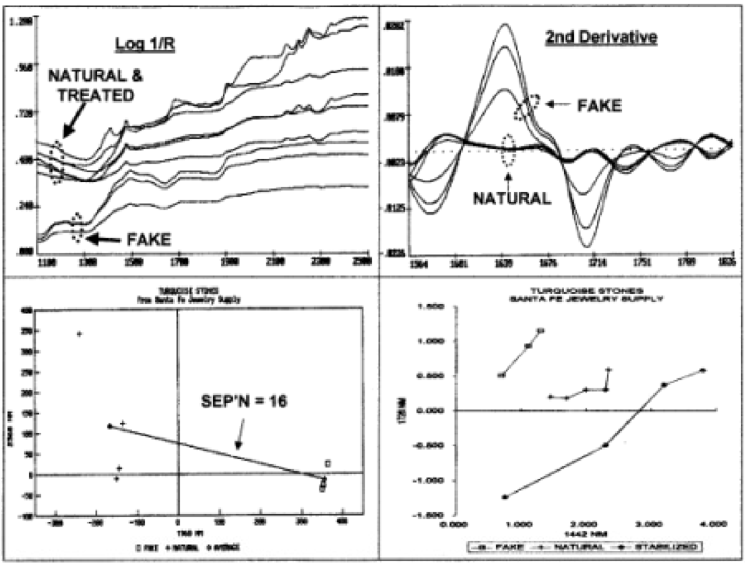

Figure 1. Top left, Log 1/*R* tracing (absorbance v. wavelength) for ten samples; top right, 2nd derivative treatment, distinguishes natural and fake turquoise; bottom left, cluster diagram showing Mahalanobis separation of natural and fake turquoise; bottom right, cluster diagram distinguishing among natural, stabilised and fake turquoise.

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Experimentation

Light from a 28-watt tungsten/halogen lamp with integral reflector (Bulb Direct, type FLS-WI) was directed through a lens into a monochromator with 600 μm entrance and exit slits (Optometrics, Manual Mini-Chrom, 750–1700 nm). The emerging NIR light was passed through an order-sorting filter (Oriel, 51330, 665 nm cut-on), reflected upward by a gold-coated front surface mirror and focused on a sapphire window. Lenses, mirror and window were purchased from Edmund Scientific. Figure 2 shows the entire optical path in the top half and a close-up showing the position of the detectors in the bottom half. The monochromator arrived with a 10-turn digital control. Its knob was removed and the shaft connected via flexible rubber tubing to the shaft of a stepper motor (Oriental Motor USA, PX243M-03AA).

Since temperature control was deemed important, a heat sink was fabricated from a block of aluminium (Figure 3, top left). The pair of PbS detectors were mounted on its sloping surfaces and a hole was drilled in the block to accommodate a thermistor probe. The detectors

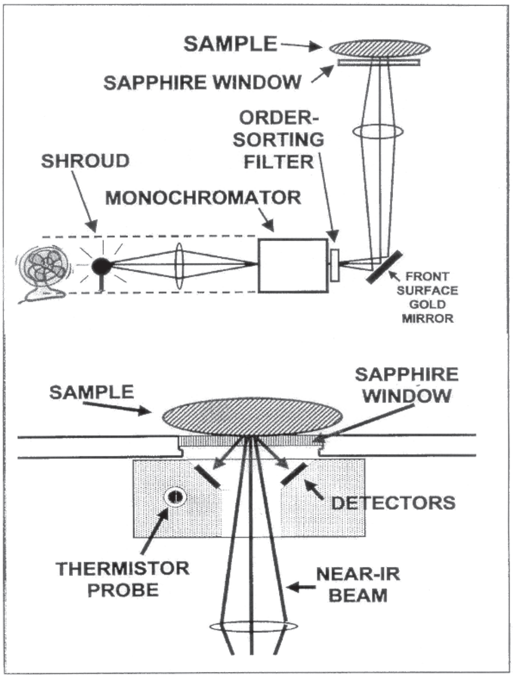

Figure 2. top, optical path from source to sample; bottom, close-up depicting reflection from sample to detectors.

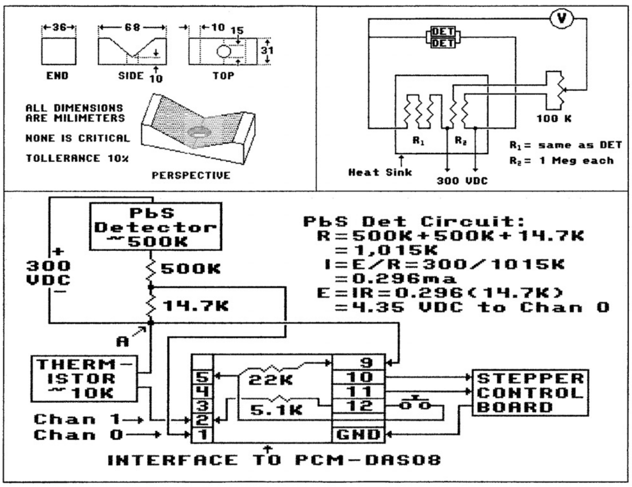

Figure 3. Top left, dimensions of aluminium heat sink; top right, detector circuit—bridge; bottom, detector circuit with series resistor.

(Cal-Sensors Inc, AF-6(ME), 6 × 6 mm) were biased with 300 VDC from a tiny power supply (Pico Electronics, 12AV300) whose input is 12 VDC. The detector circuitry was varied: the most sensitive involved a bridge (Figure 3, top right), but the least noisy merely plucked the signal from a series resistor (Figure 3, bottom). The stepper motor is driven through a control board (Gateway Electronics, kit 158) whose timing pulses are generated in a laptop computer (Winbook , model XL). Communication with the computer is through an 8-channel 12-bit A/D PCMCIA card (Computer Boards Inc, PCM-DAS08). This card's analog inputs were connected to circuits from the detectors and from a thermistor temperature probe.

The final configuration with the computer is shown in Figure 4—the wiring diagram in the top half showing the interconnections of the various modules and a photograph of the packaged unit with its laptop computer in the lower half.

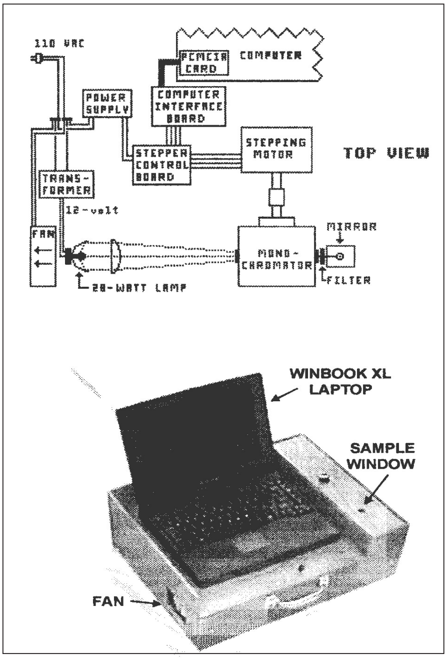

Figure 4. Top, overall wiring diagram of the modules; bottom, repackaged analyzer with its computer.

Instrumentation

A modest grant from Los Alamos National Laboratory funded a project to construct a portable instrument weighing less than 20 pounds, with dimensions 18" W × 15.5" D × 7.5" H (meeting the requirements of under-seat storage on commercial aircraft), including computer. While presently powered by 115 VAC, all components require only 12 volts for eventual battery operation.

Much of above described hardware is summarised in Table 1. Control and data handling software was written in QuickBASIC v. 4.5 for easy conversion to a stand-alone *.exe file. Admittedly, the software was written with many bells-and-whistles long before it was established that more work needed to be done on the detector circuitry to render it less noisy.

Table1. List of major parts.

	$
Monochromator	1,363
Stepper motor	78
Control board	68
Laptop computer	1,239
Interface board	50
A/D converter	370
Light source	42
Lenses/mirror/window	162
PbS detectors	185
300-v power supply	120
Assorted hardware	133
	TOTAL $3,709

Operation

In operation, the temperature of the heat sink is first measured and displayed in real time on the computer's screen. When it has stabilised (usually within 5 to 15 minutes), the dark current is measured and stored for later subtraction. Next, a reference scan is taken from a block of white Teflon. Finally, an opening dialogue appears, giving the operator such options as (1) evaluate a sample, (2) observe a stored family of turquoise tracings, (3) read animated operating instructions or (4) quit.

Results

This section will suggest to the reader that a more appropriate title might have been "Pitfalls in Trying to Save Money by Building Your Own Instrument." Although the NIR approach has clearly been established using commercial instrumentation, the noise level on the prototype generally exceeded the signal level so that no publishable tracings are yet available. Even after mounting the detectors on a metal heat sink whose temperature was monitored, replicate tracings were not adequately reproducible. Accordingly, development is continuing and will be published as Part 2 of this undertaking. It will address the relative merits of (a) a scanning monochromator, (b) a monochromator stopping at a few specified wavelengths and monitoring the signal for variable time periods (i.e., until S/N is acceptable) and (c) a small number of required interference filters.

Additional effort

Performance will improve significantly when the following items are addressed:

- Operational amplifiers v. existing noisy bridge circuitry.
- A chopper may be required; AC coupling is less noisy than DC coupling.
- Larger detectors may be in order, possibly with thermoelectric cooling.
- A more stable high-voltage power supply may be in order.
- A smaller, lighter, cheaper laptop computer, combined with more efficient packaging, could reduce the size and weight.
- Rechargeable batteries will make the unit truly portable.

Conclusion

Lessons learned: (1) Don't submit abstracts until the work is actually finished, or you're absolutely sure that it will be successful and (2) don't bite off more than you can chew. The proper design of detector circuitry is complex and tricky at the least. If you've got more time than money and want to learn-by-doing, then go for it—but have sufficient expertise readily available. And if this isn't the case, then purchase a proven instrument.