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HINTS FOR THE REDUCTION OF SPURIOUS TL

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The use of ovens that can be evacuated and then filled with "oxygen-free" inert gas for the TL heating has generally reduced spurious TL levels to negligible levels. However, some fine-grain samples from ceramics are still found to be obviously spurious (indicated by the nonexistence of a plateau and the characteristic gradual increase in TL with increasing temperature above ~ 300°C). And frequently one wonders if a given sample is emitting a few per cent spurious TL, not quite enough to upset the plateau but nevertheless introducing perhaps as much as a 10% error in the date. Recently, we made some measurements with a low noise system (less than 5 cps black body plus dark count at 350°C) and found spurious signals of 5 to 50 cps from all of the following:

1. 1 mg samples of "clean" 100 μm grains of quartz and zircon
2. Mica
3. Aluminum, brass, nichrome, and silver

We have had success in reducing these spurious levels by increasing the purity of the atmosphere in the oven. This was accomplished by means of two filters; one on the vacuum line to stop back diffusion of pump oil vapor into the TL oven, and one on the inert gas (argon) line to scavenge oxygen and water vapor from the inert gas (see Figure 1).

The coaxial trap is from Veeco Instruments, Inc., Terminal Drive, Plainview, N. Y. (with offices on all continents except Africa), Model VS-120, $138.00. The trap's metallic absorbent removes pump oil very efficiently and is claimed to work maintainance free for many years. The trap slightly increased pumping time to ~ 100 microns (because of reduced pumping speed), but below 100 microns evacuation was more rapid than without the trap, indicating that considerable oil diffusion had been occurring. After installation of the trap, we also found it possible to remove the beaker of P_2O_5 desiccant from the oven with no apparent change in evacuation rate.

The oxygen/water-vapor trap housing is a molecular sieve trap (also from Veeco, Model TR-101, $175.00). It is filled with a mixture of molecular sieve beads (supplied with the trap) which absorb water vapor, and a reagent "Ridox" from Fisher Scientific Co., U. S. A. (with branches in Germany, Switzerland, Mexico, and Puerto Rico) No. R-30, which scavenges oxygen. The trap housing contains a heater for activating the molecular sieve material at 200°C. The Ridox regent is activated simultaneously at the same temperature by flowing a regeneration gas (4-6% hydrogen in an inert gas, e. g. helium, a non-explosive mixture) through the housing. The product data sheet from Fisher gives the details for calculating the oxygen reduction to be expected and regeneration procedure. For our particular trap, holding 30cc of Ridox, a flow rate of 1 ℓ/min, and assuming the gas initially contains 5 ppm oxygen, 95% or more of the oxygen should be removed for a flow period of 80 hours. Then the unit is regenerated.

The benefit of the oxygen trap was measured to some extent by use of an oxygen meter (Teledyne, P. O. Box 70, San Gabriel, Cal., 91776, Model 311). Although in principle capable of measuring oxygen levels to ~ 0.1 ppm, the particular meter we were using was limited to a ~ 1 ppm by a small leak in the intake manifold. Without the oxygen trap, a level of 3.5 ppm was measured directly from the argon tank, and the same with the meter connected on the outlet of the TL oven (after first evacuating the oven, then refilling with argon). (Incidentially, the argon tank came with a guarantee of <0.5 ppm oxygen.) With the oxygen trap in place and activated, the output from the oven read~ 1 ppm, and as mentioned above was believed to be the limit of the meter. No improvement was seen in oxygen level by flushing the gas, as opposed to simply filling the oven after evacuation. Even heating a fine-grain disk to 450°C, with a static gas, the oxygen level still measured 1 ppm. The importance of evacuation was demonstrated by flushing the oven at 3 ℓ/min for 5 min. from room pressure without first evacuating the oven; giving an oxygen level of 3000 ppm.

The effect of the two traps on TL from a 1 mg sample of our "spurious standard", a finely ground limestone sample, is shown in Fig. 2. The traps appear to have given a sustantial reduction in the spurious level. However, they are not a cure-all; some spurious TL still remains. Similar effects were seen with fine-grain samples from a potsherd. The spurious levels from 1 mg samples of quartz and zircons were reduced from ~ 30 to < 3 cps.

As a final hint, small spurious signals (~ 30 cps) from aluminum pans were eliminated by replacing them with pans made from gold foils. Attempts to gold plate aluminum and brass pans were not successful; after heating to high temperatures the gold coating disintegrated, although a more sophisticated plating might be more successful.

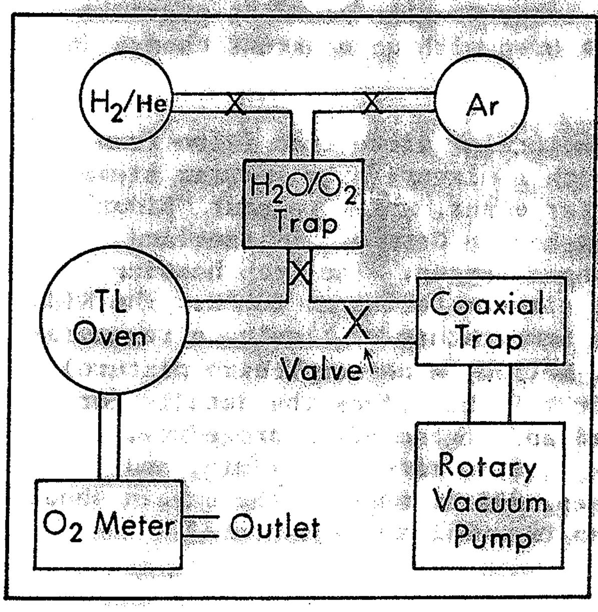

FIGURE 1

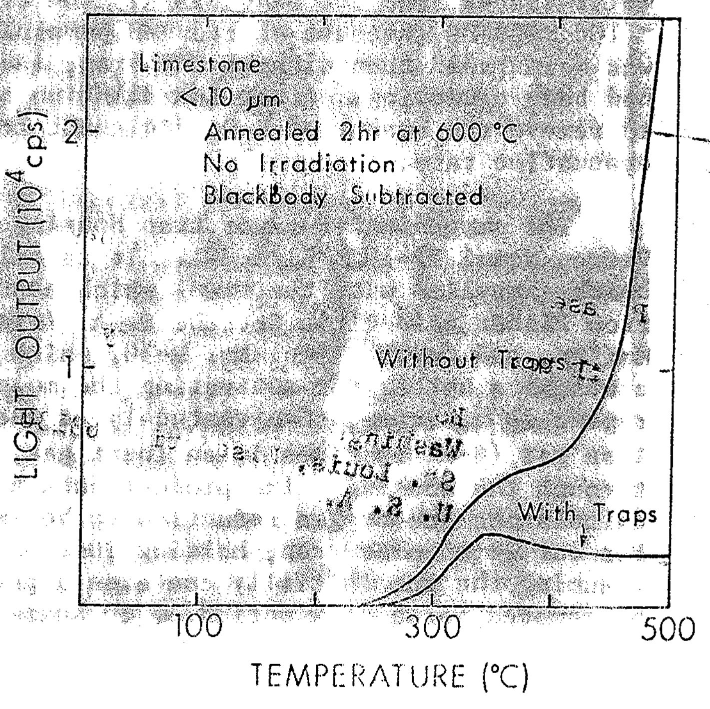

FIGURE 2