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Similar experiences have been encountered with quartz separates from pottery sherds. In one such case, quartz separates from Texas pottery were found to yield poor plateaus (measurements by D. Zimmerman). CL examination showed the presence of a few very bright zircon grains. Heavy liquid extraction of the zircon was required in this instance and good plateaus for the "pure" separate resulted.

Of course, this procedure prohibits subsequent use of the electron-excited grains for TL determination of archaeologic dose. In the case that sample size is limited, the CL examination can be performed on "glew" material to verify separate purity.

5 SPURIOUS TIDBITS

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(1) Ordinary aluminium sample discs show some spurious TL themselves and this is a serious problem with samples too thin to obscure the disc. We have eliminated this problem by cleaning the discs in hydrofluoric acid before each use as follows. After removal of grease with an organic solvent such as trichloroethylene the discs are placed in a glass beaker (~100 ml) with about 30 ml of water. About 3 ml of concentrated HF is added and stirred in; almost immediately there is considerable fizzing and the mixture becomes cloudy. After a few seconds the mixture is poured down the drain along with a large flow of water, and repeatedly rinsed with more water until the acid is all removed. We perform all these operations in a fume hood with a built-in water tap and drain.

(2) When some spurious TL is evident, but not dominant, it is tempting to subtract the reheat from the first glow to obtain the true TL. This is not the correct thing to do. The error of this method is immediately apparent if the reheat curve is higher than the first glow, as occurs on occasion. Another method of detecting this error is to stop the first glow at a lower temperature and then reheat. It is obivous that the first heating enhances the spurious signal although the mechanism for this is not known.

(3) We have for over a year now been using a model 8301 "Hydrox Purifier", sold by Matheson (P.O. Box 89, Whitby, Ontario, L1N 5R9, Canada) to remove oxygen from our high purity argon (< 5 ppm O_2). This has enabled us to obtain TL measurements on a number of samples with very low light levels, one as low as 20 photon counts/s, for which the TL was almost completely obscured by spurious when the purifier was not used. An example of the reduction of the spurious emission is shown in Figure 1.

Advantages of this system are its evident success and the manufacturer's statement that the purifier will easily clean the oxygen from fifty 7 m^3 cylinders of argon with 10 ppm O_2. Its theoretical capacity is 26g of oxygen or water.

Disadvantages are its high cost (~$800), it is not rechargable, it takes 30 minutes to warm up and 2 hours to cool, and argon must be kept flowing while it is warm. It cannot be used with nitrogen. Special valves and regulator are recommended for use with the purifier in order to avoid introduction of oxygen to the gas line from the air. Potential purchasers should study both the engineering report and the instruction manual before ordering in order to set up the system properly.

Another commercial purifier, model GP-100 from R.D. Mathis Co. (2840 Gundry Ave., P.O. Box 6187, California 90806 U.S.A) is available at a similar price and has the advantage of a replaceable Ti getter element and adjustable temperature; its maximum flow rate is only 1/4 ℓ/min however compared to 5 ℓ/min for the Matheson.

(4) Even with the purifier we found recently that our spurious level had been rising. We reduced it substantially by a thorough cleaning of the glow oven and using a heating tape to bake the inaccessible copper vacuum line. The problem was found after we had measured a number of samples containing organic material. It appears that such material contaminates the glow oven atmosphere and that this contamination is not removed by keeping the chamber pumped while not in use. An alternative to the cleaning that appears to work is spraying the glow oven with a silicone spray (Sil-Spray from Duxe Products, P.O. Box 1192, Cincinatti, Ohio 45201 U.S.A.) thus sealing the contaminant in place although this is only of temporary benefit.

(5) At this point two clearly separate sources of gas responsible for the spurious TL were recognized, the chamber outgassing and the argon. In order to find an optimum we tried flowing the argon through the glow oven and vacuum pump in such a way that the flow rate was 1/2 ℓ/min and pressure in the glow oven was 300 μm Hg. In this way the chamber would be continously purged and the amount of argon in the chamber would be less than 0.05% of the amount present when the chamber is at atmospheric pressure. The decrease in the spurious level was dramatic, amounting to an order of magnitude or more. An example is shown in Figure 2.

Some of these observations and developments arose as a result of severe and frustrating spurious troubles with a variety of samples. The latter included Roman pottery, Loyalty Is sherds, calcite, ocean sediments and volcanic ash samples. Despite the improvements noted we have occasionally found samples which exhibit a high degree of irreproducibility at the high temperature end of the glow curve. It is tempting to attribute this to a perhaps different form of spurious TL. We would be grateful to anyone knowing the cause or a cure if they would publicize these.

In summary, it has become apparent to us that spurious TL can be eliminated or reduced to managable proportions with straight-forward techniques. It is also clear that a vacuum system which is very "tight" and clean and which has a good pump will prevent the occurance of many of these annoyances.

The Spencer Gulf, South Australia, sample was prepared by J.R. Prescott during a visit and kindly left here for us.

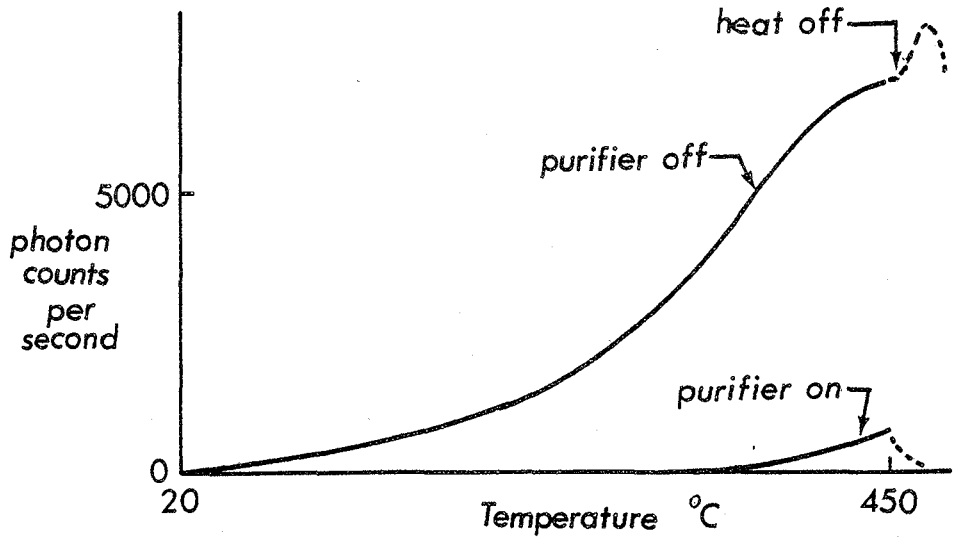

Figure 1. Demonstration of the reduction in the spurious intensity produced by the use of the Matheson purifier. The sample is Spencer Gulf sediment and has previously been glowed. The rapid increase in spurious intensity when the heat is switched off, shown in the upper curve, is curious, reproducible and unexplained.

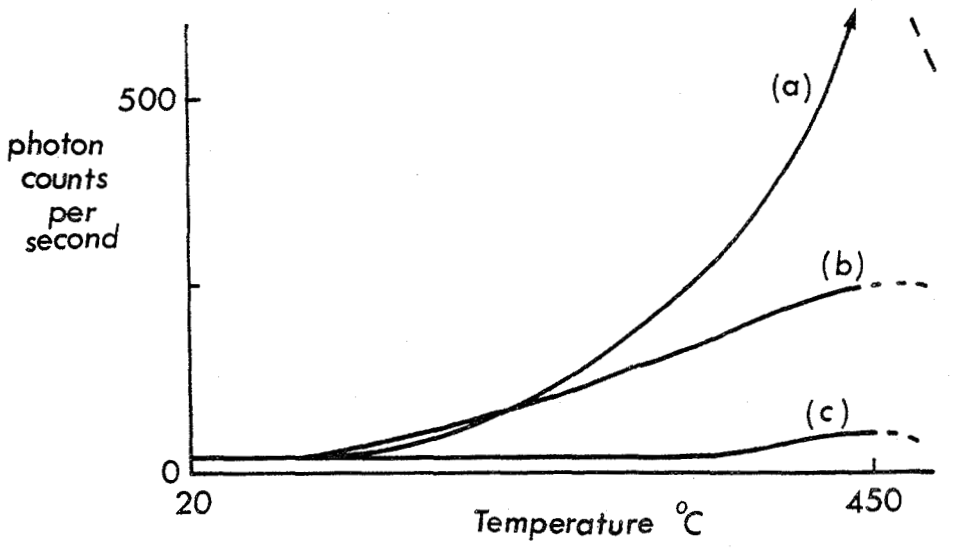

Figure 2. The same sample disc heated
a) under vacuum and being pumped
b) Ar flow 1 l/min at atmospheric pressure with the purifier on.
c) Ar flow ½ l/min at a pressure of about 300 μm Hg (not critical) with the purifier on.

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