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"For holding betwixt my fingers a steel bodkin, near the lower part of it, I pressed the point hard against the surface of the diamond, and much more if I struck the point against it, the coruscation would be extremely sudden, and very vivid, though very vanishing too: and this way, which commonly much surprized and pleased the spectators, seemed far more proper than the other, to shew, that pressure alone, if forcible enough, though it were so sudden and short, that it could not well be supposed to give the stone any thing near a sensible degree of warmth, as may be suspected of rubbing, yet it is sufficient to generate a very vivid light." Sir Robert Boyle, in "Observations made this 27th of October, 1663 about Mr. Clayton's Diamond".

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THE EFFECT OF SAMPLE REFLECTANCE IN ALPHA COUNTING

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It has been known for some time that the procedure for determining the correct discriminator setting for an alpha counter yields a setting which depends on which standard material is used. Aitken found that the average pulse height for a white sample could be up to twice as large as that for a black one, and this was attributed to different sample reflectivities (Bowman, 1976). The pulse height analyses given by Huntley (1977) show variations attributed to the same effect. Here I have attempted a more quantitative experiment to see how accurately the effect can be predicted and corrected for.

We have measured the reflectances of six counting standards using a Pye Unicam SP-8000 Spectrophotometer, and determined the discriminator setting for each (82% threshold uranium, 85% threshold thorium); the results are shown in the figure. (The samples and sources are described in Huntley, 1977 except for NBL-106 and 108 which are 1.00 and 0.052% Th obtained from ERDA, and DH-1 which is a Canadian Certified Reference Material containing 0.177% U and 0.104% Th.

A simple model for the reflectance effect is to consider that the size of the light pulse is proportional to (1 + R), where R is the reflectance; complete reflection thus doubles the light intensity. The straight line drawn in the figure is a fit of this relation to the data. Although the scatter in the data is a little larger than is desirable the relation appears to be a reasonable fit. It is also worthwhile noting that the two thorium standards, with reflectances of about 0.9, are white, while the remaining samples are brown or gray.

It is apparent from this that if one sets the discriminator using a highly reflecting thorium standard and then uses the alpha counter to measure a gray sample, the resulting count rate will be about 20% too low. Errors of the order of 5% or more are thus probably quite common.

A possible reason for the scatter of the data in the figure is that the sample reflectance will depend on the fraction of it that is in optical contact with the ZnS; this in turn will depend on whether the powder was spread out and pressed with a spatula or simply poured on. The surface roughness of both the sample and the ZnS may also affect the reflectance. It is difficult to see how to measure or calculate these effects in a precise way, however a simple reflectance measurement will clearly be better than none at all and a simple device for measuring sample reflectivities would be most useful.

I wish to thank Mr. Falko Tilgner for making the reflectivity measurements and J.C. Irwin for clarification of the optical effects.

Bowman, S.G.E., D. Phil. Thesis, Oxford, p. 28, 1976.
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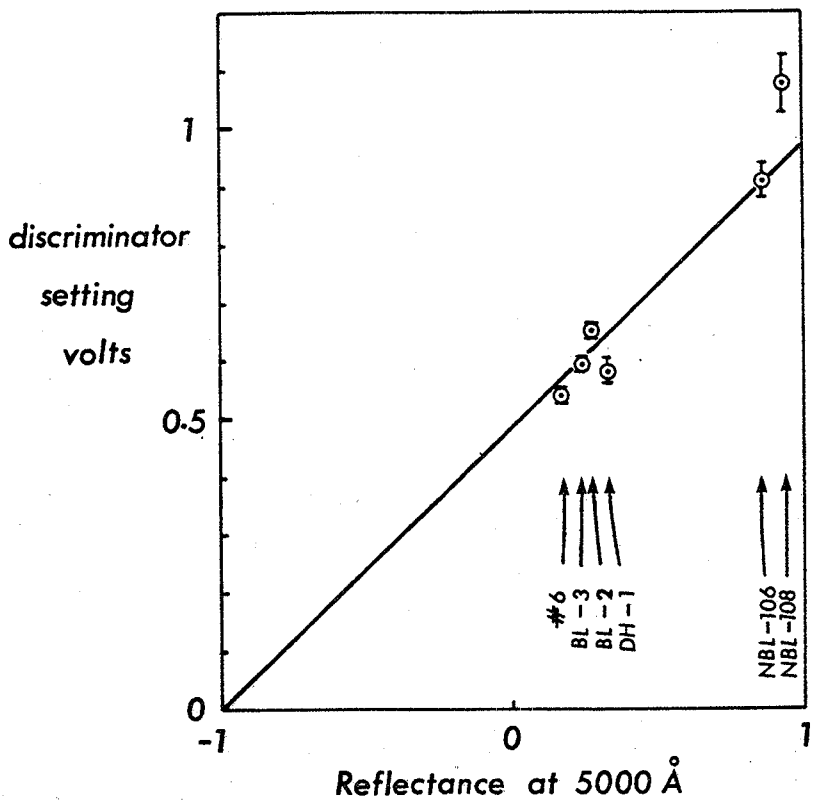

THE 1978 TL SEMINAR - OXFORD

The TL seminar 3-8 July at Oxford was the largest meeting ever on TL dating, both in length and number of papers, exceeding, for example, previous TL sessions at the Archaeometry Symposia by more than a factor of two. The over 80 participants from 23 countries, nearly all of whom contributed papers, reflects the rapid increase in worldwide development of TL dating. All aspects of TL dating were covered. There were theme lectures on:

- basic TL measurements and sample preparation
- fine-grain, quartz inclusion and predose techniques
- beta and gamma dosimetry
- dose-rate evaluation
- rare earth impurities
- authentication
- phototransfer
- TL kinetics
- defects in quartz
- anamolous fading

Research papers covered the above topics as well as many others, in particular:

- radon and Ra-226 disequilibrium
- TL dependence on dose-rate and temperature
- dating techniques for flint, calcite, ocean sediments, zircons, volcanic rocks, heated rocks and meteorites
- electron spin resonance
- new apparatus
- pottery dating applications
- supralinearity
- emission spectra
- alpha dosimetry

The proceedings of the seminar will be published as Volume 3 of the Council of Europe's PACT Journal, and should be available by early 1979 (contact Dr. Vagn Mejdahl, Atomic Energy Commission, Research Establishment Risø, Roskilde, Denmark, for further details).

AQUA REGIA WASH FOR THE LARGE GRAIN DATING METHOD

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Rationale:

M. Miyamoto[1] has compared the TL yields of materials cleaned with hot HCl and with aqua regia. We have experimented further with the effects of aqua regia washes as part of the material processing procedure for large grain TL studies, and have found that this step provides several advantages :

1. When integrated into the grain-sizing step it assures that the desired size range (.074mm<d<.125mm) is obtained. By sizing, washing, and resizing, the artificial grain sizes (i.e. those grains made up from smaller quartz grains cemented together by the clay matrix) are removed.

2. The clay matrix adhering to the outer surface of the large grain quartz crystals is removed without affecting the quartz crystals themselves. This cleaning permits the easy separation of the quartz grains from the rest of the gently crushed matrix, using a Frantz Isodynamic Separator. This advantage may not be of consequence when the crushed and sized materials being examined lend themselves to clean magnetic separation. Attempts made at the Toronto lab were, however, unsuccessful because much of the ceramic material being processed tended to coagulate making separation without a wash impossible.

3. The clean quartz crystals that are produced after the wash provide an environment in which the entire surface of the quartz is exposed uniformally to the HF. The differential rate of attack of aqua regia on clay vs. quartz is greater than for HF because the latter attacks both materials, while the former with its affinity for dissolving metals attacks the chief constituent of most clays which is an hydrated silicate of aluminum preferentially over quartz.

Method:

Aqua regia is a fuming, yellow, corrosive liquid produced by mixing concentrated HCl and HNO_3 in a molar ratio of 3 to 1. Properly mixed the acids produce a solution[3] of nitrosylchloride(NOCL), chlorine, and water which is a powerful oxidizer capable of dissolving metals including platinum and gold.[2] The basic reaction forming aqua regia is:

$$3\ HCl+HNO_3 \rightleftharpoons 2H_2O+NOCL+Cl\uparrow$$

The aqua regia is added to a beaker containing the sample in a ratio of approximately 10 to 1 by volume. The sample should be left in the aqua regia for one to two days. A fumehood is a requirement throughout the acid preparation and washing procedure.[3] When the aqua regia wash process is completed, distilled water is added and the dilute solution discarded. This washing is repeated until the aqua regia is removed. The final cleaning is done with methyl Alcohol, again several times, to remove the residual water. The beaker of material is then placed under the hood to dry at room temperature. The fume hood should be the kind that with modifications facilitates the maintenance of a dark environment. The materials are now ready for the magnetic separation step.

Discussion:

In addition to the advantages of the aqua regia wash noted above, the removal of the clay materials attached to the quartz crystals was found to greatly improve the optical properties of the quartz crystals with regard to their ability to luminesce. The use of the wash for longer time periods than one day did not greatly improve the increase in signal gained relative to a one day wash. The actual light output was found to increase by only 30% above that attained after a one day wash when the sample was left in the wash for seven days. While this optical quality may not be of value in the absolute dating of ceramics, the larger TL signal associated with the wash may be of direct assistance in the fingerprinting and relative dating of ceramics because the HF processing step can be eliminated.

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[2]J. W. Mellor, A Comprehensive Treatise on Inorganic and Theoretical Chemistry (London: Longmans, Green and Co., 1940), pp. 617-619.

[3]Aqua regia is very dangerous when heated to decomposition. It emits highly toxic fumes of nitrosylchloride, and can react vigorously with reducing materials (see N. Irving Sax, Dangerous Properties of Industrial Materials, Reinhold Book Corporation, 1968, p. 433).

CONTRIBUTIONS REQUESTED

There is space available in the next issue of Ancient TL. Do you have a contribution? Send it to the editor.

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