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AQUA REGIA WASH FOR THE LARGE GRAIN DATING METHOD

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M. Miyamoto¹ 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 :

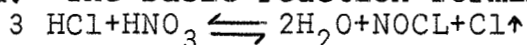
1. When integrated into the grain-sizing step it assures that the desired size range (.074mm < d < .125mm) is obtained. By sizing, washing, and resizing, the artificial grain sizes (i.e. those grains made up from smaller quartz grains cemented together by the clay matrix) are removed.

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 uniformly 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 HNO₃ in a molar ratio of 3 to 1. Properly mixed the acids produce a solution 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:



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.

REFERENCES

¹Massahiro Miyamoto, Estudo e Aplicacao da Dosemetria Termoluminescente na Datacao, MSc Thesis, Instituto de Fisica da Universidade de Sao Paulo, pp. 78-80. (1973)

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

³Aqua 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.