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Ancient TL

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"I also brought it to some kind of glimmering light, by taking it into bed with me, and holding it a good while upon a warm part of my naked body."

Sir Robert Boyle, in "Observations made this 27th of October, 1663, about Mr. Clayton's Diamond".

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THE TL LABORATORY SURVEY

Each subscriber to Ancient TL has received a questionnaire with their copy of the last issue. The purpose of the questionnaire is to compile a listing of TL laboratories around the world and to obtain some indication of the types of research and applications that are under investigation at each institution. At the present time, about 35 completed forms have been received and responses are continuing to arrive. Consequently, it seems advisable to postpone publication of the data until the next issue (number 16). This delay will allow a more complete listing to be assembled. So, if you have not yet sent in your form there is still time. Many thanks to those who have responded so far. Those that have misplaced their form may simply drop me a short note giving the laboratory address, director' name and a brief description of the TL studies in progress.

S. R. Sutton, Editor

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AN INEXPENSIVE METHOD FOR SEPARATING QUARTZ FROM CLAY

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Abstract

A magnetic method is described using conventional laboratory apparatus and non-toxic liquids, which permits the separation of quartz from crushed pottery. The efficiency of the described method of separation is in excess of 90% which compares favourably in performance with commercial magnetic separators.

Introduction

Commercial magnetic methods have been developed by metallurgical engineers for separating magnetic and non-magnetic mineral components in powders and slurries. Flotation methods, although often having the disadvantage of requiring the use of toxic liquids, have also been developed. A description of these methods as applied to mineral processing have been described in a variety of text books - one particularly recent being that by B. A. Wills, "Mineral Processing Technology", Pergamon Press, Oxford, 1979. A method of particular importance in separating quartz grains is the magnetohydrostatic method described by U. Andres (Minerals Science Engng. 7, (1975)).

The above commercial techniques have been used to separate quartz from pottery samples and have been described by S. Fleming in his recent book on Thermoluminescence Techniques in Archaeology, Clarendon Press, Oxford, 1979. A more recent development is that of the application of the magnetohydrostatic method of Andres by J. H. James and H. Junger which is to appear in the publication by PACT of the Oxford T.L. seminar (1980). We wish to describe in the present note an efficient magnetic method of separating quartz from pottery which requires conventional laboratory apparatus thereby requiring a modest capital outlay for a separator of comparable usefulness as those commercially available.

Method

A schematic plan of the apparatus is shown in figure 1(a) with the various components identified in the figure caption. The method of operation consists of:

- (i) crushing the pottery in a v-shaped metal trough placed between the jaws of a machinist's vice.
- (ii) passing the crushed pottery through a series of sieves, the finest being 45 μm .
- (iii) when the sieved particles are poured into the top of the apparatus the magnetic field gradient holds back the strongly paramagnetic material in the upper chamber and the diamagnetic material drops directly through to the bottom chamber. (In order not to have the liquid surface tension hold the particles, a wetting agent, such as commercial detergent, is added to the water column.) The dividing valve is then closed off, the bottom chamber removed, and the particles and liquid separated by filtering through a Gouch crucible. The particles are then dried in a small laboratory oven.

- (iv) If a greater degree of separation is needed, then the above process can be repeated before drying the particles. It was found that after three passes there was little increase in the percentage of quartz content.

Experimental Requirement of the Method

The major piece of equipment involves the permanent magnet. In order to determine what minimum magnetic field is required a series of separations as a function of field were carried out. This was accomplished by using an electro-magnet with a variable pole gap. In order to "estimate" the minimum field necessary for the method to function effectively, consider the forces acting on a paramagnetic particle in a viscous liquid. When the falling particle has reached a terminal velocity the various forces can be represented as shown in Fig. 1(b). Substituting values for the vectors we have:

$$\frac{4}{3} \pi r^3 (k_q - k_w) H \frac{\partial H}{\partial y} = \frac{4}{3} \pi r^3 g (\rho_q - \rho_w) - 6 \pi \eta r V_t$$

where the subscript q and w refer to quartz and water respectively. In the present case the particles were found to reach terminal velocity after travelling 2 cms of the tube length.

- k - volume magnetic susceptibility
- ρ - density
- V_t - terminal velocity of falling particle
- η - viscosity of water
- r - average radius of the particle
- $H \frac{\partial H}{\partial y}$ - value of constant magnetic field intensity and field gradient in the vertical direction

Rearranging terms we have as an estimate for the product of field strength and gradient

$$H \frac{\partial H}{\partial y} = \left(\frac{1}{k_q - k_w} \right) g (\rho_q - \rho_w) \frac{-9\eta V_t}{2r^2} \sim \frac{10^2}{k_q - k_w} \left(\frac{\text{Gauss}^2}{\text{cm}} \right)$$

If one uses the value of the susceptibility of Fe_2O_3 as the major magnetic component in the clay then a minimum value of $4 \times 10 \text{ Gauss}^2/\text{cm}$ is estimated, i. e. a field of 4 kilogauss and a gradient of 10 Gauss/cm. These values can be obtained from large yoked permanent magnets, the gradient being produced either by tapering the pole caps of the magnet or by using steel wool in the pass tube. Magnetron

permanent magnets are ideally suited. One note of caution if the latter method is used: the density of steel wool should be sufficiently low that it does not act as a sieve in filtering the particles. In fact, the density of packing should be adjusted until tapping the tube in the presence of the field does not yield further increase in the mass of collected particles in the lower tube. Some experimentation is required in this regard but we found best results when a mass of 0.6 gms of number 422 stainless steel wool was teased to a length of 10 cm in a tube of 2.6 cm cross section.

Results

Ten 1.5 samples of crushed and sieved pottery from the same source were run through the apparatus at three different magnetic field strengths. After each run the samples were etched 20 minutes in an aqua regia solution and then for 10 minutes in a 10 N hydrofluoric solution in order to remove feldspars and calcites. After the particles were dried, a microscopic count was made of the percentage of quartz particles in the calibrated grid. The results are summarized in Table 1 where it can be seen that smaller fields are nearly as effective as larger ones provided the number of passes is increased. In order to judge the efficiency of this method of separation, a comparison with a commercial apparatus (Franz separator) indicated an efficiency of 90% for crushed and sieved pottery from the same source.

<u>Maximum field</u>	<u>Original weight of sample(gr.)</u>	<u>No. of Runs through system</u>	<u>Final weight of sample(gr.)</u>	<u>Particles Counted</u>	<u>Percent Quartz</u>
5K gauss	1.40225	1	0.78370	1220	66 %
	1.67535	2	0.8281	869	62
	1.57617	3	0.36712	956	80
10K gauss	1.40623	1	0.79033	944	70 %
	1.42163	2	0.45087	686	81
	1.47751	3	0.30045	1047	85
15K gauss	1.7276	1	0.99753	563	80 %
	1.488195	2	0.42946	869	88
	1.55681	3	0.2408	438	95

Table 1 — Weight and percentage of quartz as a function of uniform magnetic field. The percentage of quartz as counted in the untreated sample used in this experiment was 30%.

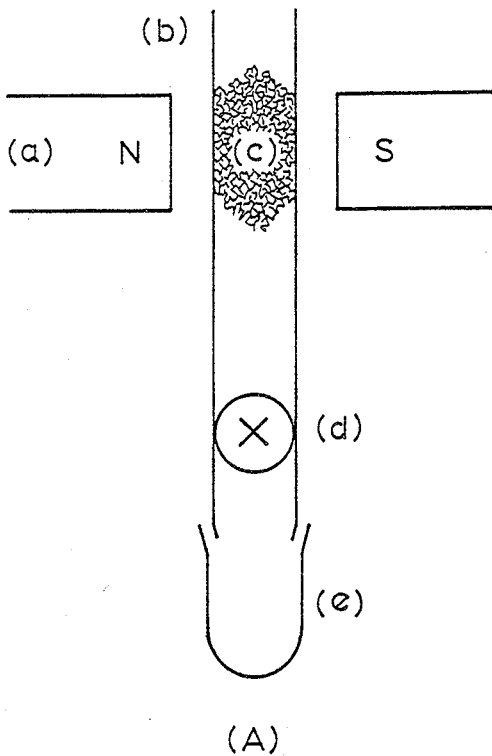


Fig.1(a) Schematic figure of apparatus

- (a) Pole pieces of magnet
- (b) Copper tube 1" diameter 12" long
(also possible to use glass if preferred)
- (c) Steel wool
- (d) 1" Gate valve - brass
- (e) Removable chamber

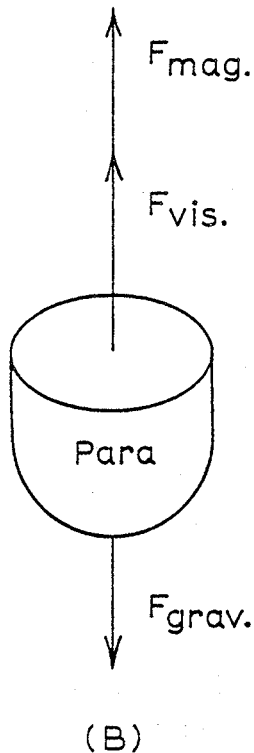


Fig.1(b) Forces exerted on a paramagnetic particle

$$F_{\text{grav.}} = mg = \frac{4}{3} \pi r^3 (\rho_{\text{quartz}} - \rho_{\text{water}}) g$$

where r is the radius of the particle, ρ the density and g the acceleration due to gravity.

$$F_{\text{vis.}} = 6 \pi \eta r v_t \text{ (Stoke's law)}$$

where η is the viscosity of the fluid and v_t is the terminal velocity of the particle.

$$F_{\text{mag.}} = (k_{\text{quartz}} - k_{\text{water}}) v H \frac{\partial H}{\partial y}$$

where k is the volume susceptibility, v the volume of the particle, H the magnetic field strength and $\frac{\partial H}{\partial y}$ the magnetic field gradient in the vertical axis.

PUBLICATION OF THERMOLUMINESCENCE DATE LISTS

In this issue, Ancient TL will begin the publication of thermoluminescence date lists. The purpose of including date listings is to provide for the publication of the details of dating procedures and results which may then be referred to in journal articles. The adopted format is essentially that proposed by David Zimmerman at the 1978 TL Specialist Seminar at Oxford and is similar to the format used by Radiocarbon for the publication of radiocarbon dates. Immediately following this note is a listing from the Physical Research Laboratory (Ahmedabad, India) which can also serve as a format reference for future contributors.

The TL date report consists of an introductory paragraph giving information on technique, apparatus and pertinent references followed by a listing of the dates. In the first date list from each laboratory, the introductory paragraph should be reasonably detailed. Subsequent reports from that laboratory may then simply refer to this first report for details of technique. The date listing itself is divided according to site. Each TL date is given a three entry identification code which consists of a laboratory identification acronym, 'TL' to distinguish the date from a radiocarbon date and a laboratory sample/context number. The actual dates and error assessment values are listed following the format suggested by Aitken and Allred (1972) and Aitken (1976). It is suggested that a base year of 1980 be adopted when quoting ages in years before present. Directly under this information is given the type of material dated and context information. Finally, a comments section appears containing detailed information on the measurements and calculations. This section is subdivided into three subsections- the natural dose, the annual dose and general comments. The actual information given in these subsections will depend on the material and dating technique used.

The format suggested here should not be considered rigid. Readers are encouraged to suggest modifications and/or additions to this format as well as alternative methods of listing. It is also hoped that some discussion of TL date reporting will take place at the upcoming specialists' seminar.

S. R. Sutton, Editor

PHYSICAL RESEARCH LABORATORY TL DATES - 1981 (II)

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Physical Research Laboratory, Ahmedabad 380009, India

The following TL dates on pottery from a protohistoric site, Sanghol (District Ludhiana) in Punjab and one date from Bagor (Rajasthan) were assayed. Fine grain technique (Zimmerman, 1971) was used. The alpha activity of the sample was measured using thick source ZnS(Ag) counting method and NaI(TL) gamma spectrometry was used for potassium determination. For dose-rate calculations, equal alpha activity of uranium and thorium series was assumed and the decay series were taken to be in equilibrium. Both

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unsealed (α_0) and sealed (α_1) counts were measured. In cases where α_1/α_0 was greater than 1.05, an average of α_1 and α_0 was used for the dose-rate estimation. For other cases, α_0 was considered appropriate. Saturation water content was used both for soil and the sherd. Beta irradiations were performed using a 40 mCi, $^{90}\text{Sr}/^{90}\text{Y}$ beta plaque. Alpha irradiations were carried out under vacuum using six-seater alpha irradiation facility (Singhvi and Aitken, 1978). The details of the experimental procedures and the apparatus have been described elsewhere (Agrawal et al., 1981). In view of the fact that in general a glow curve is a composite of many glow peaks, the natural dose (AD) and the alpha efficiency (a) were estimated at intervals of 10°C on the glow curve. These values were then used to compute the age at various points on the glow curve. The quoted ages are the mean of these ages, averaged over the age plateau. The natural dose and alpha efficiency and thus the dose-rate reported are only typical values corresponding to a particular glow curve temperature. In all the cases anomalous fading was estimated as the loss in the TL signal integrated over the plateau region. The error estimates were made using procedures suggested earlier (Aitken, 1976). No allowance was, however, made for σ_0 as saturation water content was assumed. The samples were collected by A. K. Singhvi (PRL) and G. B. Sharma (Department of Archaeology, Punjab). All the dates are in years B. P. and use 1980 as the base year. Errors are given within parenthesis.

Acknowledgements

We thank Professors D. Lal and M. J. Aitken for their kind help and encouragement and the Ford Foundation (India) for a generous financial assistance. Y. P. Sharma is thankful to the Department of Science and Technology, India, for a fellowship.

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A. SANGHOL (Dist. Ludhiana, $30^\circ 20' \text{N}$, $75^\circ 15' \text{E}$), India

PRL-TL-1 Late Harappan Bara Ware 3120 (\pm ---; ± 270) 1140 B.C.

Pottery: Loc. SGL-II, Tr. EX-1, kiln sealed by Layer 40.

COMMENTS- Natural dose: 2440 rads ($I=360$), $\delta D = 5\%$, plateau $\approx 60^\circ\text{C}$, anomalous fading $< 5\%$ (14 days). Annual dose: 0.79 rads/yr., $\alpha_1/\alpha_0 = 1.05$, sherd wt. sat/dry = 1.13, $a = 0.12$, $\delta a = 5\%$.

^{14}C date on associated charcoal is 3640 ± 150 (PRL-513).

PRL-TL-6 Grey Ware 2100 (\pm ---; ± 180) 120 B.C.

Pottery: Loc. SGL-II, Tr. EX-1, Layer 31.

COMMENTS- Natural dose: 1640 rads ($I=270$), $\delta Q = 5\%$, plateau $\approx 70^\circ\text{C}$, anomalous fading $< 5\%$ (18 days). Annual dose: 0.80 rads/yr., $\alpha_1/\alpha_0 = 1.0$, sherd wt. sat/dry = 1.13, $a = 0.13$, $\delta a = 5\%$.

PRL-TL-7 Late Harappan Bara Ware 3220 (\pm ---; \pm 450) 1240 B.C.

Pottery: Loc. SGL-II, Tr. EX-1, Layer 45.

COMMENTS- Natural dose: 2350 rads ($I=170$), $\delta Q = 10\%$, plateau $\approx 70^\circ\text{C}$, anomalous fading $< 5\%$ (25 days). Annual dose: 0.72 rads/yr., $\alpha_1/\alpha_0 = 1.03$, sherd wt. sat/dry = 1.12, $a = 0.11$, $\delta a = 10\%$.

PRL-TL-9 Upper level of Late Harappan Bara Ware 3110 (\pm ---; \pm 270) 1130 B.C.

Pottery: Loc. SGL-II, Tr. EX-1, Layer 35.

COMMENTS- Natural dose: 2360 rads ($I=420$), $\delta Q = 5\%$, plateau $\approx 70^\circ\text{C}$, anomalous fading $< 5\%$ (40 days). Annual dose: 0.76 rads/yr., $\alpha_1/\alpha_0 = 1.0$, sherd wt. sat/dry = 1.14, $a = 0.11$, $\delta a = 5\%$.

PRL-TL-10 Lower levels of Late Harappan period 4030 (\pm ---; \pm 330) 2050 B.C.

Pottery: Loc. SGL-II, Tr. EX-1, Layer 46.

COMMENTS- Natural dose: 3190 rads ($I=380$), $\delta Q = 5\%$, plateau $\approx 70^\circ\text{C}$, anomalous fading $< 5\%$ (20 days). Annual dose: 0.80 rads/yr. (using $(\alpha_1+\alpha_0)/2$), $\alpha_1/\alpha_0 = 1.11$, sherd wt. sat/dry = 1.13, $a = 0.12$, $\delta a = 5\%$.

PRL-TL-14 Painted Grey Ware (?) 1760 (\pm ---; \pm 210) A.D. 220

Pottery: Loc. SGL-II, Tr. EX-1, Later 33.

COMMENTS- Natural dose: 1350 rads ($I=390$), $\delta Q = 8\%$, plateau $\approx 70^\circ\text{C}$, anomalous fading $< 5\%$ (30 days). Annual dose: 0.76 rads/yr., $\alpha_1/\alpha_0 = 1.0$, sherd wt. sat/dry = 1.14, $a = 0.11$, $\delta a = 8\%$.
The date is anomalously younger than expected.

B. BAGOR (25 21'N, 74 23'E), India

PRL-TL-42 Bagor Ware 2060 (\pm ---; \pm 210) 80 B.C.

Pottery: Loc. sample from depth 0.41-0.55m from a sand dune which also yielded microliths.

COMMENTS- Natural dose: 2220 rads ($I=60$), $\delta Q = 7\%$, plateau $\approx 80^\circ\text{C}$, anomalous fading $< 5\%$ (90 days). Annual dose: 1.09 rads/yr. (using $(\alpha_1/\alpha_0)/2$), $\alpha_1/\alpha_0 = 1.07$, sherd wt. sat/dry = 1.13, $a = 0.18$, $\delta a = 5\%$.

SOME RECENT BIBLIOGRAPHY

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THE 1982 SYMPOSIUM ON ARCHAEOOMETRY

The 22nd Symposium on Archaeometry will be held March 30 - April 3, 1982 at the University of Bradford, Bradford, U. K. The session format of the previous symposium will be followed. Following is a list of the planned sessions and convenors:

Reconstruction of Exchange Systems:

A. Aspinall and S. Warren, Postgraduate School of Studies in Physics, University of Bradford, W. Yorkshire BD7 1DP, U.K.

Provenance Studies:

G. Harbottle and E. Sayre, Dept. of Chemistry, Building 555, Brookhaven National Laboratory, Upton, New York 11973, U.S.A.

Ancient Metals and Metallurgy:

R. Maddin, University of Pennsylvania, Philadelphia, PA 19104, U.S.A.

Ancient Technology: non-metals:

M. S. Tite, The British Museum, Research Laboratory, London EC1B 3DG, U.K.

Prospection:

R. Lington, Fondazione Lerici, Via Vittorio, Veneto 108, I 00187 Rome, Italy

Dating of Organic Materials (e.g. radiocarbon and other cosmogenic nuclides, dendrochronology, amino acid dating):

E. T. Hall, Research Laboratory for Archaeology, 6 Keble Road, Oxford OX1 3QJ, U.K.

Dating of Inorganic materials (e.g. thermoluminescence, ESR, fission tracks, uranium-series, archaeomagnetism):

L. Langouet, Université de Rennes, Campus du Beaulieu, Avenue de General Leclerc, 35031 Rennes Cedex, B. P. 25A, France

Mathematical methods and date management:

I. Scollar, Rheinisches Landesmuseum, Colmansstrasse 14, 5300 Bonn 1, West Germany

Two copies of each abstract (minimum 200 words, maximum 1 page) must be submitted no later than January 15, 1982.

Copy #1 should be sent directly to the symposium organisers:

A. Aspinall and S. E. Warren
Schools of Physics and Archaeological Sciences
University of Bradford
Richmond Road
Bradford BD7-1DP, West Yorkshire, U. K.

Copy #2 should be sent to the appropriate session convenor.

A workshop for "small computer" enthusiasts will be held immediately following the regular symposium program on April 3. Contact I. Scollar, J. Litvak (Institute for Anthropological Investigations, CIUDAD, Universitaria Mexiso 20, D. F. Mexico) or the Symposium Organisers for details.

REPORT ON THE 1981 SYMPOSIUM FOR ARCHAEOOMETRY

The 21st Symposium for Archaeometry was held May 18 through May 22, 1981, at Brookhaven National Laboratory, Long Island, New York. This year's conference was divided into seven sessions:

- Provenance studies
- Ancient metals
- Ancient technology: non-metals
- Prospection
- Dating of organic materials (eg. radiocarbon and other cosmogenic nuclides, dendrochronology, amino acid dating)
- Dating of inorganic materials (eg. thermoluminescence, ESR, fission tracks, uranium series, archaeomagnetism)
- Mathematical methods and data management

Each session was organized and conducted by a session convenor. The "parallel sessions" format, used at previous symposia, was eliminated in favor of presenting nearly half of the 100 papers in two poster sessions.

The "Dating of Inorganic Materials" session included an introductory review of archaeometric dating by Martin Aitken, the session convenor, and twelve papers pertaining to TL studies. Ten of these papers were presented in the poster sessions. Five papers concerned the results of TL dating studies - Bailiff (Durham) reported predose dates for 25 Medieval pottery sherds from England; Slusallek, Goedicke (Berlin), and Kubelik (Wurzburg) presented fine grain dating results on bricks from Venetian villas of architectural importance; Stoneham (Oxford) reported the successful dating of porcelain using the predose method; Liritzis (Patras) reported good agreement between TL and ^{14}C dates for Hellenic materials; Li Hu Hou (Beijing) described the dating of an ancient oven.

Four other papers dealt primarily with the TL characteristics of certain materials - Brito, Deza and Román (Santiago) showed glow curves of bone samples prepared by the fine-grain settling technique; Haskell, Wrenn (Utah) and Sutton (St. Louis) described predose measurements on bricks exposed to nuclear fallout radiation; Pavlish and Sheppard (Toronto) studied the heat treatment history of chert retouch flakes; Schwartzman and Levy (New York) examined the TL properties of granitic minerals.

An oral presentation by Goedicke described the etching of euhedral quartz crystals in HF acid. Their size reduction was determined microscopically and anisotropic etching was observed. Although the etching rate of the individual surfaces was variable, the average size reduction was found to be in reasonable agreement with Fleming's weight loss measurements (i.e., $\sim 6\mu\text{m}$ removed in 30 to 40 minutes).

Two papers on TL kinetics discussed second order behavior (Levy) and the quartz 325°C peak (Carranza (Lima), Román, and Langouet (Rennes)).

Immediately following the Symposium, a "round table" discussion entitled "Future Directions in Archaeometry" was convened under the sponsorship of the Smithsonian Institution. The discussion was divided into four sessions - "Teaching Archaeometry", "Interdisciplinary Collaboration", "Organizing Research Problems", and "Roles of Museum, University, Government and Industrial Laboratories". Each session consisted of five, 15 minute commentaries by invited speakers followed by a period of general discussion. Much of the discussion concerned ways in which archaeologists and archaeometrists could work together most effectively.

S. R. Sutton

DANISH RESEARCH COUNCILS' ARCHAEOOMETRY PROJECT
REPRINT LIST (1977-1981)

Risø National Laboratory
DK-4000 Roskilde, Denmark

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