Radiocarbon dating of the Shroud of Turin

Supplementary Information for: *Nature* 337, 611 – 615

This document details further information available for the research done at the Oxford Radiocarbon Accelerator Unit on the dating of the Shroud of Turin, published in *Nature* **337**, 611 – 615.

Photographic archive

The photographs taken during the research are available at:

https://archdams.arch.ox.ac.uk/?c=1203&k=1bcdc90a8b

Details of chemical cleaning of the Turin samples

This is given in the following pages of this document.

The chemical treatment given to the sub-samples was as follows:

1M HCl at 80°C for 2 hours

Rinse free of acid with H_2O

1M NaOH at 80°C for 2 hours

Rinse free of alkali with H_2O

1M HCl at 80°C for 1 hour

Rinse free of acid with H_2O

5% NaO_2Cl at pH 3.0 and 75°C for 45 minutes

Rinse free of bleach with H_2O

This procedure was carried out manually in 4 cm^3 screw-top "Wheaton" vials.

Removal and addition of solution was done using pasteur pipettes and most of the sample loss on P2574 and P2576 was caused by this. The losses were considered to be unacceptable, so it was decided that the samples themselves should have a slightly milder chemical treatment, and that this should be carried out using the 4.5 cm^3 volume continuous-flow cells.

It was proposed that each sample should be subdivided into three parts: 20% being acid, alkali, acid treated (and therefore similar to Arizona and Zurich samples), while the remaining 80% should be split in half, with both halves having a bleaching step added after the acid, alkali, acid treatment. The sub-division of the samples is recorded below (Table 2):

Sample & Q No.		
2574.0	11.4 mg	Acid-alkali-acid
2574.1	19.4 mg	" " " + bleach
2574.2	18.0 mg	" " " "
2575.0	9.5 mg	Acid-alkali-acid
2575.1	19.5 mg	" " " + bleach
2575.2	17.8 mg	" " " "
2576.0	8.5 mg	Acid-alkali-acid
2576.1	16.5 mg	" " " + bleach
2576.2	16.7 mg	" " " "
2589.0	21.4 mg	Acid-alkali-acid
2589.1	21.6 mg	" " " + bleach
2589.2	20.4 mg	" " " "

Table 2

The use of continuous-flow cells ensured that any threads which became detached from the sample during treatment would be retained within the cell by the 20μm porosity sintered disc. To ensure that dust was excluded from the reservoir solutions, these were made from analar grade chemicals in distilled, deionised water and were either filtered before use (GF-F filter) or in-line, using a stainless steel HPLC filter. The cells were placed in a heating block which was maintained at 75 C throughout the sample processing.

The chemistry cycle programmed into the continuous-flow apparatus was as follows:

1M HCl, 2 cell volumes, allowed to equilibrate at 75°C for 1¼ hrs

H_2O, 10 cell volumes, at 0.5 cm^3 min^{-1}

0.5M NaOH, 10 cell volumes, at 0.5cm^3 min^{-1}, equilibrate at 75 C for 1 hour.

H_2O, 12 cell volumes at 0.5 cm^3 min^{-1}

1M HCl, 10 cell volumes at 0.5 cm^3 min^{-1}, equilibrate at 75°C for 1 hr

H_2O, 12 cell volumes at 0.5 cm^3 min^{-1}

The flow-chemistry was completed successfully overnight. The cells were emptied as usual by using filtered compressed air, and samples 2574.0, 2575.0, 2576.0 and 2589.0 were removed from the cells and placed into pre-weighed combustion tubes for drying at ≈60°-80°C. The yields for the unbleached samples are recorded below (Table 3).

Sample	Initial wt (mg)	Final wt (mg)	% recovery (yield)
2574.0	11.4	9.8	≈ 85%
2575.0	9.5	7.1	≈ 75%
2576.0	8.5	5.4	≈ 65%
2589.0	21.4	15.0	≈ 70%

Table 3

To the other samples was added 2 cm^3 of 2.5% W:vol NaOCl(pH 3.0) by pasteur pipette (through the inlet port of the flow-cells), and these were placed in a hot block at 80°C for the following time periods: (Table 4).

2574.1	30 minutes	2576.1	30 minutes
2574.2	30 minutes	2576.2	30 minutes
2575.1	45 minutes	2589.1	45 minutes
2575.2	45 minutes	2589.2	45 minutes

Table 4

Time of bleaching was determined by the physical appearance of the samples; the more robust ones being bleached for the longest times. The bleach was removed and replaced with 2 cm^3 of distilled water. After a short period of agitation, this was also removed, and a fresh aliquot of distilled water was added to each cell. The cells were then replaced in the continuous flow rig, and flushed with distilled water at 0.3 cm^3 minute (10 cell volumes of water per sample).

After removal of the final wash water the caps were loosened, and the whole assembly was placed in an oven at ≈60°C to dry overnight. The samples were then placed in pre-weighed combustion tubes for final drying and yield measurements, which are noted in Table 5.

Sample	Initial wt	Final wt	%recovery (yield)	Approx.* Mg C.
2574.1	19.4	15.4	79%	5.1
2574.2	18.0	14.0	78%	4.7
2575.1	19.5	13.8	71%	4.6
2575.2	17.8	13.0	73%	4.3
2576.1	16.5	9.2	56%	3.0
2576.2	16.7	10.2	61%	3.3
2589.1	21.6	14.9	69%	5.0
2589.2	20.4	14.5	71%	4.8

Table 5

* assumes that samples ≈33% C

Conclusions

Yields are very good and higher than is usually found for similar samples processed by the manual method. Disturbance of the sample

during solution exchange is minimal in the continuous-flow cells, and the physical breakdown of samples is also reduced. The milder chemical treatment (0.5M cf 1.0M NaOH, and 2.5% W:vol cf. 5.0% W:vol NaO_2Cl) has also helped to produce better yields while still producing final samples which appear to be "pure". It is recommended that this mild procedure is retained for textile samples, and that the more vigorous treatment is reserved for tree-ring and other wood samples.

TURIN SAMPLES 13/7/88.

Sample.	Wt.(mg)	Final Wt.	%recovery	CO2(mb)	Mg C.	Rig	FE.NOS.
2574.0	11.4	9.8	85	236	4.5	3&12	1175.0,1175.1
2574.1	19.4	15.4	79	308	6	1&2	1165.0,1165.1
2574.2	18.0	14.0	78	244	5	10&8	1168.0,1168.1
2575.0	9.5	7.1	75	127	2.5	12	1170.0
2575.1	19.5	13.8	71	256	5	11&9	1174.0,1174.1
2575.2	17.8	13.0	73	257	5	1&6	1171.0,1171.1
2576.0	8.5	5.4	65	103	2	5	1169.0
2576.1	16.5	9.2	56	176	3.5	7&10	1172.0,1172.1
2576.2	16.7	10.2	61	154	3	1	1176.0
2589.0	21.4	15.0	70	283	5.5	2&8	1173.0,1173.1
2589.1	21.6	14.9	69	266	5.5	7&6	1167.0,1167.1
2589.2	20.4	14.5	71	277	5.5	3&4	1166.0,1166.1

Burn samples in the usual way.

100mb = one Iron rig
150 = one Iron rig
200 = two Iron rigs of std size.
250 = two Iron rigs of std size.

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