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THREE CHEMICAL TESTS FOR COMPARING THE AGE OF PAPER

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The specialist practising forensic chemistry sometimes has to report on the genuineness or otherwise of old documents like land title deeds. When examination of water marks, chemical analysis of ink, and chemical examination of any discoloration or stains present or special search for evidence of any deliberate alteration or erasures do not avail, the chemical and microscopic examination of paper material becomes of utmost importance. Cases of forged certificates and anonymous correspondence also often hinge on the quality and composition of papers used. This led the authors to investigate whether the natural process of aging in case of paper produces any significant effect which might be discerned by a quantitative chemical examination of the paper material. To eliminate the easier cases which can be distinguished by the microscopic examination of the paper fiber after it is suitably prepared, four samples of old papers representing similar raw materials or mode of manufacture were tested with various reagents first qualitatively and then quantitatively.

As a result of this investigation it has been found that the acidity of paper and its capacity of absorbing bromine in carbon tetrachloride solution in the cold increases but its resistance to the action of nitric acid decreases during the natural aging of paper.

EXPERIMENTAL

The four paper materials examined in this investigation consisted of blank strips of paper excluding some marginal area cut out from books published in the years 1909, 1920, 1935, and 1946.

Examination of paper fibres. Each sample of the paper when freed of sizing and loading material by alkali treatment, and stained with iodine showed yellow medullary rays or cross bindings as shown by Clapperton and Henderson[1], indicating that the papers were all made of mechanical wood pulp.

Examination of mineral constituents. Each paper was ashed, and the ash qualitatively analysed by performing spot tests as described in the *B.D.H. Spot Tests Outfit Handbook.* Each sample was found to contain calcium, magnesium, and aluminum, but was free from tin, iron, and antimony. Thus the above tests could not discern any material difference in the samples.

The comparative action of a reagent on each sample of paper was studied quantitatively under identical conditions in the following way.

Acidity of paper. 0.5 g. of each paper, finely powdered, was heated with 20 ml. rectified spirit over a boiling water bath for 20 minutes. The mixture, after allowing to cool to room temperature was titrated against standard NaOH solution (0.01785N) using phenolphthalein as indicator. Since 20 ml. alcohol required 0.73 ml. alkali in a blank, this was subtracted from each titre, and the weight of NaOH equivalent to 100 g. of each paper was calculated. (See column 3 of table.)

Nitric acid-resistance of paper. To 0.5 g. of each sample was added a mixture of 5 ml. water + 15 ml. conc. nitric acid (sp. gr., 1.3788 at 34°), and the mixture immersed in boiling water for 20 minutes, after which 50 ml. water was added to arrest the reaction and the mixture allowed to cool. It was filtered through a tared sintered glass crucible, washed with 15 ml. water, and dried in

[1] MODERN PAPER MAKING, Basil Blackwell, Oxford, 1947.

Age of paper in years	Alkali required for paper ml.	NaOH reqd. for 100g. paper g.	HNO_3-insol. residue g.	Residue per 100g. paper g.	Bromine solution absorbed ml.	Bromine absorbed per 100g. paper g.
15	1.11	0.1585	0.3390	67.80	0.07	0.1130
26	2.05	0.2927	0.3132	62.64	0.10	0.1612
41	2.935	0.4191	0.3109	62.18	0.15	0.2433
52	4.16	0.594	0.2491	49.82	0.25	0.4093

an air oven to constant weight. The weight of residue from 100 g. of each paper was calculated. (See column 5 of table.)

Bromine absorption by paper. 0.5 g. of each sample was heated with 20 ml. carbon tetrachloride over a boiling water bath for 20 minutes using a water cooled reflux condenser. The mixture, after allowing to cool, was titrated against standard bromine-in-carbon tetrachloride solution (0.8096% w/v) using starch-iodide paper as an external indicator. The bromine absorbed by 100 g. of each paper was calculated. (See column 7 of table.) Carbon tetrachloride absorbed no bromine in a blank experiment.

Conclusion

The observations show that the above quantitative technique is useful in the verification of approximate age claimed for a document, possibly by comparison with paper of similar age and in distinguishing between papers of different ages.

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