Twentieth Century paper quality
A survey carried out at the National Archives of Australia

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ABSTRACT

The holdings of the National Archives of Australia are largely paper-based. On a very general estimate it currently holds 35 million paper-based items which roughly equates with one billion sheets of paper. This holding will continue to grow well into the future as agencies transfer their older paper-based records into custody. Even as agencies move towards digital recordkeeping the National Archives will continue to be responsible for preserving a very large and deteriorating asset.

Because of the size of the Archives' paper-based collection it would be useful to be able to break it down into categories of quality, which may be based on elements such as the date of the paper, the use the paper was produced for (e.g. typing paper, writing paper, coloured paper, telex paper), or its provenance (e.g. which Commonwealth department produced the document). This would help inform decisions about which records to target for storage improvements and other preservation actions.

To explore these issues, a survey was undertaken in 2013 to observe how paper quality varied over the period covered by the bulk of the Archives' collection, which largely comprises material from Federation onwards. The survey looked at quality-based factors such as pH level and the presence of lignin and alum. Other additives were also recorded, including gelatine, clay and optical brighteners. Additionally, a note was made of any watermarks found, as an aid to better understanding patterns of importation and local manufacture.

Results were highly revealing and indicated a range of trends, such as a rapid rise in pH levels from around 1980 onwards and the concomitant drop in alum usage and increase in the use of carbonate filler. Trends in other additives were also indicated.

Keywords Paper quality, conservation, analytical testing, paper manufacture

INTRODUCTION

The holdings of the National Archives of Australia are largely paper-based; as a very general estimate, it currently holds 35 million paper-based items, which roughly equates to one billion sheets of paper. This holding will continue to grow well into the future as agencies transfer their older paper-based records into custody. Even as agencies move towards digital recordkeeping the National Archives will continue to be responsible for preserving a very large and deteriorating asset.

Because of the size of the Archives' paper-based collection, it would be useful to be able to break it down into categories of quality. This would help inform decisions about which records to target for storage improvements and other preservation actions.

In the course of their work, Preservation staff at the Archives have noticed that paper quality through the twentieth century seems quite variable. For example, they observed that paper produced at times of economic hardship such as the war periods and the Great Depression was of poorer quality and therefore more prone to degradation. Likewise, there were periods when it appeared that one or other additive or process began to be used, or fell out of favour, leading to a change in quality.

To explore some questions raised by these observations it was decided to carry out a survey to look at a range of quality issues relating to paper and see how these varied over the period covered by the Archives' collection. It was anticipated that information could be produced to assist in making preservation decisions. It was also hoped to provide a research resource for both the Archives and other cultural institutions with paper-based collections.

HISTORICAL BACKGROUND

Introductory research before the survey aimed at getting some background on the history of fine writing and printing paper in Australia, its manufacture and importation. Although the work was only preliminary, some useful information was obtained.

In the early years of the twentieth century, there was some paper manufacture in Australia using fibre sources such as rags, straw, old sugar bags, rope and waste paper (Rawson 1953, p. 23). However, this paper was not of a suitable quality for office and printing use, and up until World War II most fine writing and printing paper was imported (Rawson 1953, p. 57). Results of this survey indicate that the chief source of paper was the UK, particularly mills in southern England, such as the Original Turkey Mill in Kent that had been producing paper since the seventeenth century. Papers also came from Scotland, for example Alexander Pirie and Sons who operated in Aberdeen. Apart from the UK, papers also came from Scandinavia, the USA and Canada.

Around 1911 importation of chemical pulp began, although this never became common due to high tariffs placed on pulp compared to finished paper. This stymied the local industry and meant that the bulk of paper continued to be imported (Commonwealth Tariff Board 1943).

The years of World War I proved difficult for paper consumers, as space on shipping was at a premium with the result that paper became scarce and prices doubled (Rawson 1953, p. 59). Because of this difficulty, it became apparent that a local papermaking industry utilising locally available pulp was required. To this end, in the inter-war years research began into the use of eucalypts for the production of pulp. Initial results from the research were not promising as eucalypts had some properties that were unsuitable for papermaking. These included high levels of tannins that tended to corrode papermaking machinery, and a particularly dark-coloured pulp, which required bleaching (Williams 1948, p.311). Papermakers persevered, however, and in 1938 the first commercial fine paper from eucalyptus pulp was produced in Tasmania, at the Burnie Mill (Rawson 1953, p. 82). This paper bore the watermark U-Clypt. During World War II the Burnie mill would be kept very busy serving local needs, when it became increasingly difficult to import paper or pulp (Rawson 1953, p. 100).

The Australian paper industry continued to grow in the post war years, with mills being established at Boyer (Tasmania), Shoalhaven (NSW), Albury (NSW), Tumut (NSW), Maryvale (Victoria) and Morwell (Victoria) (Rawson 1953). Paper continued to be imported from around the world, and in more recent years importation patterns have shifted, with much of our fine paper now coming from mills in Korea, China and Indonesia (Department of Agriculture 2013).

THE SURVEY

There was only limited time available to complete the survey, so tests were chosen to produce maximum results with minimum effort. A combination of high and low tech approaches was chosen, involving a series of analytical devices and organic spot tests. The analytical devices used were:

- UV light source (output 366nm)
- FTIR spectrophotometer (Bruker Optik Alpha-P Fourier Transform Infra-Red Spectrophotometer with ATR stage)
- Portable x-ray fluorescence (pXRF) (Innov-x Delta)

The FTIR was particularly useful because, in a single test, taking only about a minute, it could reveal the presence of a range of materials.

Where spot testing was done, reagents were applied using a fine glass writing nib which allowed the placement of a tiny spot of solution, less than a millimetre across. To further minimise the effects, spots were applied in an inconspicuous place; verso, top right corner, near the file pinhole.

ALUM

Alum was introduced as a sizing ingredient as early as the fifteenth century in the form of aluminium potassium sulphate. This was replaced by aluminium sulphate in the nineteenth century. Alum was used predominantly in rosin-alum sizing but was also used in a range of other papermaking processes. In the presence of moisture the aluminium sulphate generates sulphuric acid. The ions of sodium sulphate and sulphuric acid combine to create the acidic product responsible for paper breakdown (El-Saied et al. 1998, p. 155).

Alum was tested using an aluminon solution prepared as described by Browning (1977, p. 318). Any colour change from light pink to darker pink was recorded as a positive. In some cases the aluminon solution can go a dark plum colour which apparently could indicate the presence of iron in the paper (Browning 1977, p. 318). Results were checked occasionally using pXRF.

LIGNIN

Lignin, an organic polymer, is the binding component of wood that keeps cellulose fibres together. It is highly unstable and causes yellowing of the paper sheet due to its limited photostability. It also releases acids as it oxidises, leading to deterioration and embrittlement of the sheet (McCrady 1991, p. 33).

Two methods were used to test for lignin: firstly, the phloroglucinol spot test prepared as described by Browning (1977, p. 73); and secondly, by FTIR, by carrying out an integration of the paper spectrum in the 'fingerprint' area for lignin around 1508 cm-1.

PH

The principal agent affecting paper permanence and durability is acidity (Trafela et al 2007, p. 6321). During the mid-twentieth century there was great alarm amongst archivists and librarians regarding the rapid deterioration and embrittlement of documents and books consisting of machine made paper (Williams 1981, p. 203). Due to alum-rosin size, introduced around 1850, papers produced up until around 1990 frequently have a pH below 6. In addition to this, research carried out by the Library of Congress demonstrates that cellulose, considered one of the most stable components of paper, also generates acids (formic, acetic, lactic and oxalic) as it ages (Shahani & Harrison 2002).

pH was tested using three indicator solutions: bromocresol green, phenol red and methyl orange which allowed for a good estimate of pH to half a unit in the range 3–8 .

CALCIUM CARBONATE

Calcium carbonate has been found in papers as early as 1400 (Barrett 1989, p. 20) but was first used industrially as a paper additive in 1925 (Collings & Milner 1990, p. 59). The presence of calcium salts provides significant stability to paper because the calcium cations neutralise acidic materials, either generated within the sheet, or from external sources (Williams 1981, p. 207).

Carbonate was tested for by FTIR; its presence is indicated by a series of distinct peaks at 720, 870, 1420 and 1793 cm-1.

GELATINE

Gelatine was a common paper size from the earliest time but was on the wane towards the end of the nineteenth century according to the graph of gelatin use in paper on the *Paper* *through time* website (Barrett et al 2012). Studies have been carried out that suggest gelatine may be beneficial to permanence (Baker 1997, Barrett & Mosier 1995, Wilson & Parks 1983, Courts 1980). Although the mechanisms are not fully understood, its effects appear to relate to aiding in the control of the pH of the paper sheet and buffering moisture content.

Gelatine was tested for by FTIR; its presence is indicated by two distinct peaks at 1524 and 1626 cm-1.

OPTICAL BRIGHTENER

Optical brighteners were introduced to papermaking to make the paper sheet appear whiter and brighter. The presence of an optical brightener in a paper sheet is of potential concern for permanence. Rachel Mustalish, who wrote a paper on the history and properties of this class of chemical (Mustalish 2000), was asked by the author in 2000 if she thought optical brighteners were of concern in relation to paper permanence. Mustalish stated that since they absorb and radiate different wavelengths of light optical brghteners may well accelerate light-based deterioration. Henry Wilhem on his website dedicated to the assessment of modern photographic printing products (www.wilhelm-research.com), believes there is also the possibility that the additives may yellow over time. .

The presence of optical brighteners was determined by a simple visual test. The paper was held under a UV source emitting light at wavelength 366 nm and if it exhibited any fluorescence then a ‘yes’ was recorded for the presence of optical brighteners.

CLAY

Historically kaolin clay was a common paper filler, used to improve smoothness, gloss, optical properties and printability of the paper sheet (Cross & Bevan 1916, p. 248). These improvements do however come at the expense of mechanical properties. The use of filler results in a sheet which contains fewer fibres per unit grammage and blocks fibre to fibre bonding. Although bonding between cellulose and filler is relatively good, filler to filler bonding is poor (Eklund & Lindström 1991, p. 235).

Clay was tested for by FTIR; its presence is indicated by three distinct peaks at 3618, 3650 and 3690 cm-1.

In addition to the testing, the following were also recorded for each sample:

- Date
- Description (e.g. typing paper, cablegram etc.)
- Watermark (whether present and description of wording)
- Provenance (e.g. Commonwealth department, commercial business, private individual, etc.)
- Image (e.g. typed, letterhead, electrostatic, colour, etc.)
- Size (e.g. foolscap, A4, 174 x 208 mm, etc.)
- Condition based on a scale of 1–3 (1 being very good to 3 being bad condition)

SAMPLING

The sample set included 1130 samples: ten samples of paper from each year from 1900 up to and including 2013. Every attempt was made to ensure randomness although it was not possible to be perfectly random as papers from some eras were scarce and thus had to be actively sought out. It is recognised that 10 samples per year is a very small sample population and that the survey could thus only be considered preliminary. Nevertheless, it proved sufficient to indicate major trends in papermaking over the period.

Samples were collected in a variety of ways:

a. As files were prepared and treated in the Archives’ Conservation Laboratory in readiness to go to the reading room, any loose fragments from a file were placed into zip lock bags labelled with the year and details of the originating file. These fragments were blank (ie. no information was lost from the file);

b. Pages taken from temporary archival files marked for destruction;

c. Personal paper from colleagues’ collections that could be accurately dated;

d. For more recent years, stocks of general use copy papers;

e. Where none of these options was possible, testing was carried out on items from the collection.

The paper samples were limited to a particular set of characteristics in order to keep results from becoming skewed by too many variants in paper types. The samples were all plain coloured, fine writing, typing and copying papers. No coloured papers, card or file folder paper etc. were included in the sample set.

RESULTS

Considering the small size of the survey sample (ten papers per year) the following results must be considered as preliminary and conclusions tentative.

LIGNIN

The results of both the phloroglucinol test and the FTIR analysis (peak area ratio number) were recorded (Figure 1). The two methods did not always give corresponding or predictable results. A positive with FTIR did not always produce a positive colour change when phloroglucinol was applied. Conversely, a colour change with phloroglucinol did not always show a positive reading with FTIR, although this conflict was rare. As referred to earlier, due to lignin’s complexity, phloroglucinol does not give a positive for all types of lignin. In total only 5.2% of the total sample set showed a positive with this method. Also, FTIR was clearly able to detect the presence of minute amounts of lignin whereas phloroglucinol would only detect more significant amounts.

Some tentative observations can be made from the graph, for example the continued higher levels of lignin during the Great Depression years (1929-1939) and an apparent leap in lignin content late in World War II. The latter is possibly as a result of the high use of poor quality recycled pulp for papermaking due to the lack of availability of imported pulp (Rawson 1953, p. 100). The other large peaks around 1902, 1961 and 1987 are harder to explain.

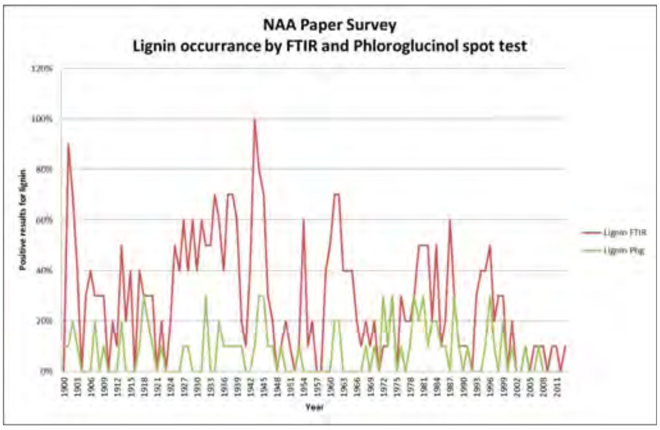

Figure 1. Lignin occurrence by FTIR and phloroglucinol spot test

PH

The pH results are very easy to read and interpret (Figure 2). There is a gentle rise in paper pH up until the early 1970s where there is a series of small hiccups until around 1994 when the pH becomes alkaline. The early gentle rise could be a result of the lowering of alum content whilst the major jump to alkaline would almost certainly be a result of the introduction of alkaline papermaking methods, specifically the introduction of calcium carbonate as a paper filler.

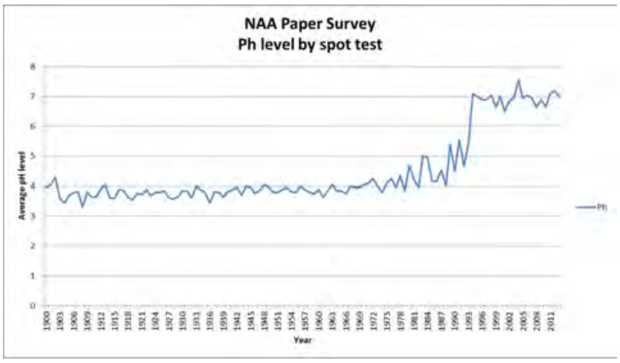

Figure 2. pH level by spot test

CALCIUM CARBONATE

As predicted in early research, carbonate begins to be used around 1990 and was virtually universal by 1999 (Figure 3). As expected, due to the buffering effect of calcium carbonate, papers which tested positive for calcium carbonate were higher in pH (see Discussion and Conclusions below).

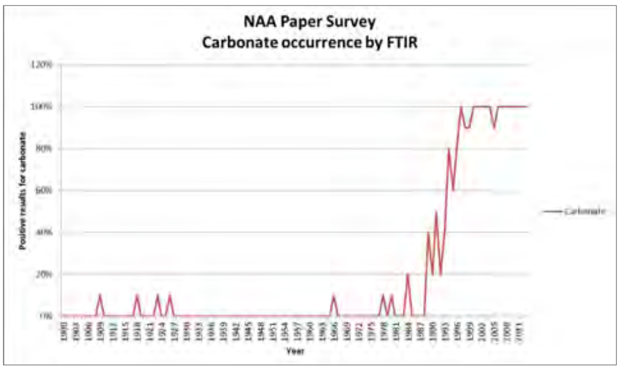

Figure 3. Carbonate occurrence by FTIR

ALUM

Alum was present in almost every paper up until 1993 at which point it tapers off dramatically and appears to vanish in 2011 (Figure 4). Although it was not specifically recorded, there was a trend in the results relating to the strength of the colour change. As a general rule, the strength of colour of the spot test dropped off over the period under examination, most likely indicating that the quantity of alum in papers also dropped off over the period of the survey. This could conceivably mean that more recent papers would have better permanence than older papers, at least where permanence was a result of alum content. In a small number of cases the aluminon solution went a dark plum colour which apparently could indicate the presence of iron in the paper (Browning 1977, p. 318). It is hoped that further research will better explore the question of alum concentration and its effects on permanence, as well as examining the prevalence of iron.

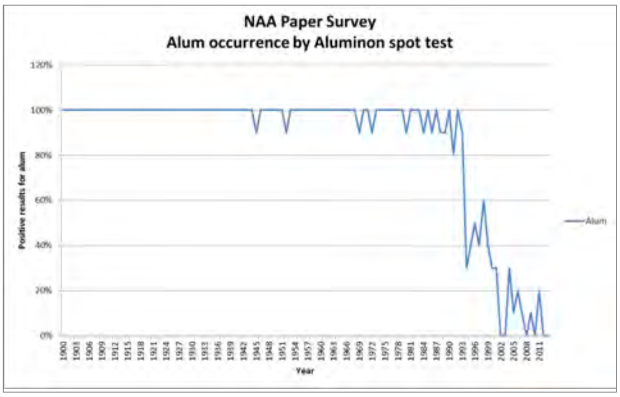

Figure 4. Alum occurrence by Aluminon spot test

CLAY

The occurrence of clay in paper is highly erratic across the testing period (Figure 5). There is a slight increase beginning around 1945 which is difficult to explain. The one clear observation to be made is the sudden drop in usage around 1996 and total absence after 2008. This can easily be explained by the increased use of carbonate, which would serve the same purpose as clay.

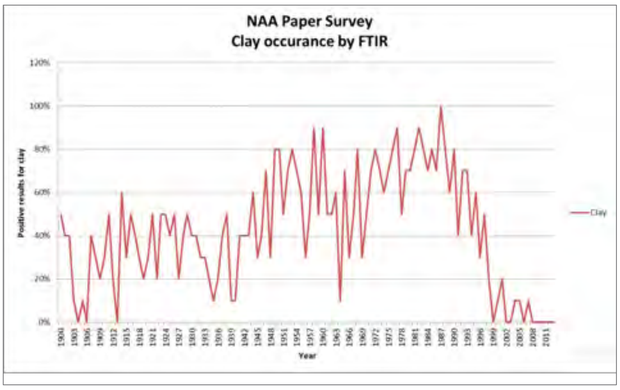

Figure 5. Clay occurrence by FTIR

GELATINE

From a peak early in the century, a spill over from the strong popularity of gelatine as a size in the nineteenth century, gelatine usage quickly drops off and becomes minimal by 1943 (Figure 6). By 1969 it has virtually vanished apart from a single example in 1977. Clearly other sizing systems must have taken over from gelatine in the second half of the century.

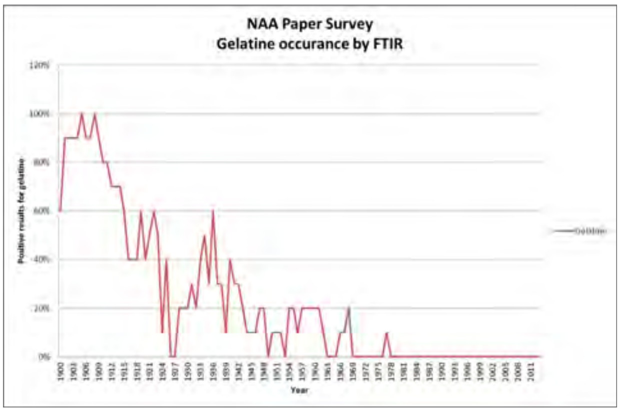

Figure 6. Gelatine occurrence by FTIR

OPTICAL BRIGHTENERS

Fluorescent additives first appeared in papers around 1960 and within 20 years virtually all papers included such materials. These results should be approached with a little caution as it is possible that there are paper additives that fluoresce but which were not specifically added as optical brighteners.

OTHER OBSERVATIONS

Watermarks

Watermarks in the papers tested were very much the norm in the early part of the century, with up to 80% of papers displaying them in some years. Over the century their occurrence drops off and by 2006 they appear to have vanished completely. The slow loss of watermarks over the century is an indication of a change in attitude and a change in manufacturing process. The introduction of high speed paper machines made the application of a watermark more problematic; at the same time the public became less interested in paper quality, as paper usage increased.

Paper dimensions

Paper dimensions were recorded for each sheet tested. From this it was possible to see trends in paper sizes across the set of paper samples tested.

- Foolscap was a standard size from 1900 but becomes uncommon after 1968 and was last found in 1978.
- Quarto came in a number of variations, the short dimension varying between 192mm to 207mm and the long dimension varying between 242mm and 262mm. It was a standard size from 1900. It becomes uncommon after 1976 and was last used in 2007. Between 1976 and 2007 most occurrences come from North America where the size remained a standard.
- A4 first appears in 1965 but does not become common until 1970, after which it is almost the only paper size.
- A5 and B5 were used infrequently after 1971.
- Junior Legal (approx. 8 x 5 inches, 203.2 x 127 mm) was used infrequently between 1901 and 1956.
- Kings (approx. 8 x 6.5 inches, 203.2 x 165.1 mm) was used infrequently between 1909 and 1995.
- Letter (approx. 8.5 x 11 inches, 215.9 x 279.4 mm) was used infrequently between 1921 and 2007.

DISCUSSION

A very clear idea of the changes in papermaking over the twentieth century can be gained from the following graph (Figure 7) plotting paper pH against the occurrence of calcium carbonate and alum as found in the paper samples tested.

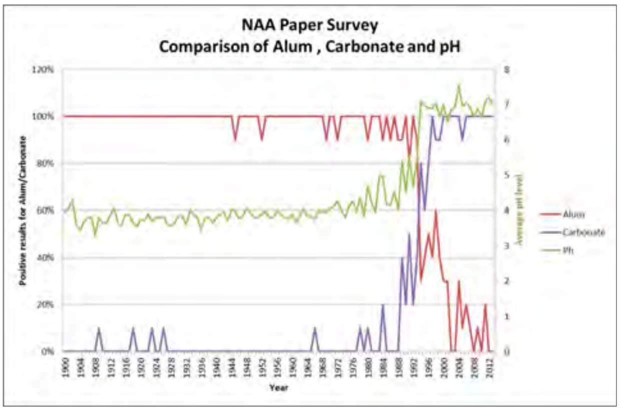

Figure 7. Comparison of Alum, Carbonate and pH

From this graph it seems clear that towards the end of the twentieth century two major changes occurred in paper manufacture, the first is that alum is no longer present as a paper additive and the second is that carbonate becomes common. It is not unreasonable to assume these two factors lead to the increase in paper pH observed at the same time. It can be further assumed that these changes will have a major impact on paper permanence in the future.

The next graph (Figure 8) looks at the occurrence of fillers and sizes, calcium carbonate, clay and gelatine. From this graph a possible set of trends in sizing and fillers can be seen. Early in the twentieth century gelatine is more common than clay whilst carbonate is almost unknown. As the presence of gelatine drops off the presence of clay becomes more common and remains so until late in the century. Although this seems clear graphically, it may not indicate a direct link as gelatine and clay perform different tasks: gelatine is a sizing agent whilst clay is a filler. The link may be that a third agent, for example rosin (which was not directly looked for in this study) may have taken over from gelatine as a size. In this scenario the use of clay may have been increased to simply bulk up the papers.

At the end of the century it seems very clear that there is a link between the drop in the use of clay and the increase in the use of carbonate. This latter link is easily understood as clay and carbonate perform the same function.

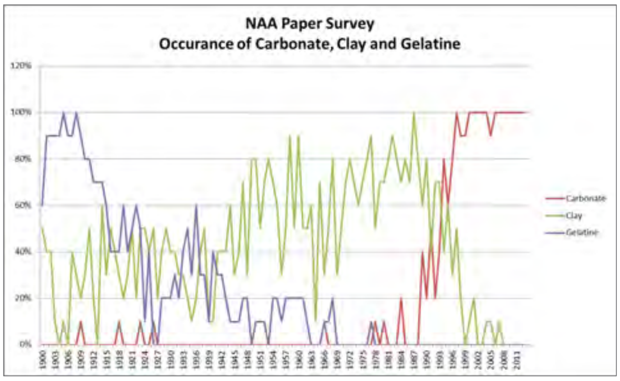

Figure 8. Comparison of Carbonate, Clay and Gelatine

CONCLUSIONS

The conclusions of the study of the paper samples tested can be summarised as follows:

- Office paper was acidic until around 1992 when it becomes alkaline.
- This alkalinity can be credited to the cessation of the use of alum and the introduction of carbonate as an additive, both of which occurred at around this time.
- There is a slight but consistent rise in alkalinity over the period 1900–1992.
- Gelatine sizing, common in the early part of the century, vanishes around mid-century.
- Clay was a fairly common filler during most of the twentieth century, although its use from year to year was quite variable. There was, however, a clear increase in its use beginning around 1940 and continuing until 1996. Clay vanishes from use around 2008.
- There may be a link (perhaps indirect) between a drop in the use of gelatine as a size and a rise in the use of clay filler, which both happen around 1940.
- A watermark appears to be no indicator of paper quality.
- Optical brighteners (fluorescing agents) first appear in 1960 and are present in all samples tested by 1984.
- Identifiably Australian made paper (bearing the U-Clypt watermark) first appears in 1942.

FURTHER WORK

This survey has provided some very useful information but raised some further questions:

- What additives, apart from calcium carbonate, have replaced clay, gelatine and alum as paper size/filler? Candidates could include Aquapel (AKD), cellulose ethers, resins etc.
- What are the effects on permanence of the new additives?
- How quickly is alkalinity from carbonate filler used up?
- Is there a link between alum concentration, pH and permanence?
- When was rosin sizing used in papermaking? (not tested in this survey)
- What can we learn about paper quality, trade and usage from watermarks?
- What more can we learn about the history of paper in Australia, generally and particularly since World War II?

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Caroline Milne is currently studying Bachelor of Cultural Heritage Conservation at the University of Canberra. Whilst studying she has worked for ACT Historic Places and in 2012 she was awarded The National Archives of Australia Jikji Scholarship and Internship. Caroline's research during the internship focussed on the analysis of paper quality over the twentieth century and the correlation of production methods and changes in stability. Other professional interests include the biodegradation of documentary heritage, its causes, treatment and prevention. Caroline works as an assistant in the National Archives' Canberra conservation lab while completing her degree.