

THE ANALYSIS OF DEGRADATION PRODUCTS

EXTRACTED FROM SELECTED 19th CENTURY PAPERS

Abstract

One approach to understanding paper degradation is to study the chemical products of paper degradation. If we can identify the products and know the reactants, then we might begin to understand the mechanisms occurring. Preliminary results obtained using this approach are reported here. A comparison of the thin-layer chromatographic (TLC) patterns obtained from extracts of a number of 19th century papers indicated the presence of many of the same chemical degradation products. Application of this same technique to foxed spots suggests that many of the same products also occur in foxed spots. The TLC separation patterns of foxed spots and their related papers show strong similarities. Some of the TLC bands obtained have been isolated and identified by IR and FTIR spectrophotometry.

TLC of Paper Extracts

Extracts of a number of 19th century papers were applied to silica gel TLC plates and separation patterns compared. The accompanying slides show the patterns obtained by TLC for about twenty different papers. The remarkable similarity of the twelve 19th century papers in the slides of TLC plate 51C indicates that most papers from the same period have the same degradation products. This relationship holds even though some papers appear much more degraded than others. Results evident indicated that the suggested approach should be pursued.

TLC of Foxed Spot Extracts

When foxed spots from a sheet of paper were excised, extracted, and the products separated by TLC, comparison of the separation patterns for the foxed spots vs the associated paper indicated little difference.

The accompanying slides show the patterns obtained. Of course some similarities are to be expected since the excised foxed spots are on their associated paper. In the examples of slides from plates JJ3, 5, 6 and NC6 care was used in choosing paper sheets with maximum foxing and care was used in excision to include as little paper as possible. Results indicate little or no differences in the patterns of the papers and their foxed spots. These results suggest that foxing and aging have the same degradation products and, therefore, possibly the same degradation mechanisms. The difference between overall degradation and foxing may be a matter of degree, and foxing could be considered localized accelerated aging catalyzed by some agent at the site.

Separation of Major Degradation Products

The isolation of some of the major degradation products for identification by IR and FTIR was accomplished by applying extract across the TLC plate, separation, and removal of the bands containing the various degradation products as shown in the slide of plate 8.

The slide sequence of plates 52S through 56S show the major bands present after separation by TLC (plate 52S) and four of the major bands after removal from plate 52S, extraction, and further purification on another TLC plate. Note in the case of bands 1, 3 and 4 that there are either closely associated impurities or that the compound present is degrading during the separation process.

Identification of Major Degradation Products

The purified bands were extracted from the silica gel absorbant to that IR and FTIR spectra could be taken. These were studied to

determine the chemical nature of the products and the spectra obtained were compared with standard spectra of compounds having similar chemical characteristics. Some of the compounds are shown in slides of; (1) Band 7 and a standard, palmitic acid, (2) N-1-B-9 and a standard, dibutyl tin dilaurate, (3) NC-2-B-1 and a standard, cis-9-octadecene-1,12-diol diacetate.

Sometimes it was helpful to convert the product into a derivative and to match that derivative with standard spectra as shown in the slides of; (1) the methyl ester of Band 6 and a standard, methyl tridecanoate, and (2) the salt of Band 9 and a standard, potassium salt of stearic acid. The identification of degradation products is a continuing project: great care must always be used to ensure that the compound indentified is not some artifact of the method or solvent used. It is interesting that the compounds identified so far all seem to be related to naturally occurring fatty acids. The significance of this is not yet clear and it may be that the pattern will not persist. It should be mentioned that in the later, rosin sized papers there occur abetic acid type compounds as expected.

It is hoped that more definitive results will be available before long. The results reported included the findings of hard-working student researchers:

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References

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