

**Institute of Paper Science and Technology  
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**STUDIES ON THE FACTORS GOVERNING  
RETENTION AND EFFECTIVENESS OF STARCH  
XANTHATES AND XANTHIDES BY WOOD  
PULP IN PAPERMAKING**

**Project 2580**

**Report Twelve**

**A Final Report**

**to**

**AGRICULTURAL RESEARCH SERVICE  
UNITED STATES DEPARTMENT OF AGRICULTURE**

**March 28, 1968**

THE INSTITUTE OF PAPER CHEMISTRY

Appleton, Wisconsin

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OF STARCH XANTHATES AND XANTHIDES BY  
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SUMMARY

The findings of Project 2580 are summarized in this report and are correlated with factors affecting the potential uses of starch xanthide in paper.

Starch xanthide is best formed when the xanthate groups are the strongest nucleophiles in the system. The dispersing effects of electrokinetic charges on the starch xanthide are to be maximized during cross-linking and storage of the starch xanthide and minimized during sorption onto the wood fibers. This may be accomplished by cross-linking at low concentrations and by adding alum to the fiber suspension before introducing the starch xanthide.

Starch xanthide improves the drainage of the stock suspension on the paper machine and, consequently, may be used at levels greatly exceeding those normally accepted for conventional internal adhesives and which usually would be achieved only by separate, additional processing. The effect of starch xanthide is to increase both the dry and wet physical strength properties of paper. This is an unusual combination of effects. Therefore, specific uses can be visualized only in a limited way since the greatest potential probably lies in the development of paper grades not yet in existence. The properties imparted by starch xanthide have not been available in this particular combination.

It is recommended that alternatives to alum be found for bringing about sorption by wood fibers for the widest use of this product in paper. Further,

that ways be found to eliminate the yellow color cast caused by what is thought to be polysulfides and free sulfur formed from xanthation by-products reacting with the cross-linking reagent. This would be a problem with high brightness pulps and paper grades. It is further recommended that systems concepts be developed so that the production of starch xanthide by the papermaker will require the least transfer of technological information. The cooperating agency has developed the basic processes for such a system.

The cross-linking reaction itself requires further study since analysis of the chemical aspects has opened alternatives to the xanthide disulfide bond as an explanation of the differences in the behavior of starch xanthate and starch xanthide.

## INTRODUCTION

This is the final report for Project 2580 which has been carried out in cooperation with the Agricultural Research Service of the United States Department of Agriculture. It is but one phase of a much larger program dealing with potential industrial uses of cereal xanthates being carried out by the Northern Utilization Research and Development Division. Research Contract 12-14-100-8308(71), approved Sept. 29, 1965 and amended Oct. 4, 1967, has been the basis for this project. Its purpose is to expand the use of cereal products in papermaking by determining factors governing sorption of starch xanthate and starch xanthide by wood fibers in dilute aqueous suspensions.

This report will concentrate on the correlation of the findings covered in detail in the preceding nine quarterly reports and two phase reports, and upon recommendations for further work or courses of action.

The focus of the discussion will be upon the use of starch xanthide formed separately from the fiber (ex situ) rather than in the presence of the wood fiber (in situ).

## STARCH XANTHATE

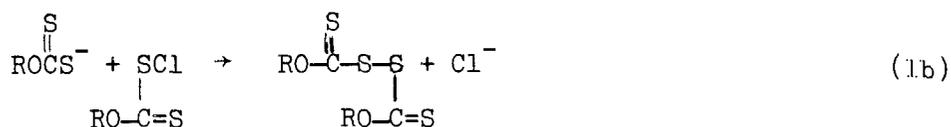
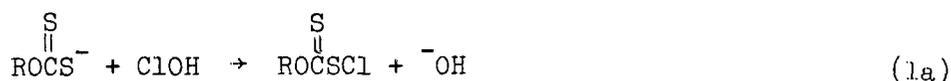
Starch xanthate is a rather unstable starch derivative obtained by reacting starch in concentrated sodium hydroxide with carbon disulfide. As prepared for this project by the cooperating agency, the xanthation product was reduced to about 10% starch by weight, shipped to The Institute of Paper Chemistry where it was stored in a refrigerator until needed.

The starch xanthate can be stored under these conditions for as long as one month without seriously affecting the performance of the starch xanthide subsequently formed from the aged D.S. 0.12 product. Further dilution is necessary for the cross-linking reaction which produces starch xanthide. It was found that solutions of starch xanthate reduced to concentrations of 0.165% may be held as long as 4 hours at 20°C. when dealing with the D.S. 0.12 product.

Only trace amounts of starch xanthate were sorbed by the wood fibers used in this project (fines-free bleached western softwood kraft). Therefore, sorption of starch xanthate prior to cross-linking does not play a significant role in the retention of starch xanthide in paper.

### STARCH XANTHIDE

Starch xanthide is formed from starch xanthate by consecutive  $S_N2$  displacement reactions. Hydroxyl groups are displaced from hypochlorous acid by ionized dithiocarbonate (xanthate) groups on the starch to form a sulfenyl chloride derivative. The chlorine atom is then displaced from the sulfenyl chloride by a second xanthate group to form a disulfide cross-link as shown in Equation (1)



This reaction is analogous to the formation of sodium tetrathionate from sodium thiosulfate by oxidation with iodine. In general, reagents containing polarizable bonds, such as those containing a positive halogen atom, will produce disulfide linkages. The course of the reaction depends upon the relative strength of the nucleophile and the stability of the leaving group.

When dealing with starch xanthate cross-linked by sodium hypochlorite, reaction conditions must be selected which assure that the xanthate groups are the strongest nucleophiles in the system. This means ionization of the hypochlorite ion must be suppressed so that the positive halogen of hypochlorous acid can serve as the electrophilic half of the first  $S_N2$  reaction. This may be done by adjusting the pH of the sodium hypochlorite at least 1.5 to 2.0 units below 7.2, the  $pK_a$  of hypochlorous acid. It appears the best combination is to have both the xanthate solution and the hypochlorous acid solution at pH 5.0 to 5.5. This recommendation is followed in the procedure for making laboratory quantities of starch xanthide

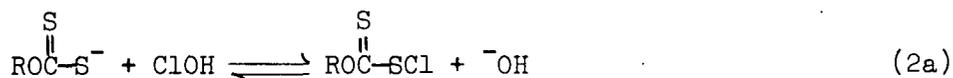
formed separately from the fiber (ex situ). When considering continuous cross-linking systems, it appears some compromises may have to be accepted. If both the starch xanthate and sodium hypochlorite streams cannot be brought to about pH 5 before being combined, then it is felt that the acid and sodium hypochlorite should be blended first in ratios sufficient to bring the starch xanthide reaction mixture to the desired pH level--presumably in the range of pH 5 to 7.

The consequences of adding hypochlorite ions to starch xanthate can be complete dexanthation and conversion of the xanthate groups to free carbonate and sulfate ions. Less extreme effects of the hypochlorite ion are dexanthation and conversion of the sulfur atoms to lower oxidation levels than the sulfate ion. The hypochlorite ion also appears to be capable of destroying the xanthide bond.

A small amount of potassium iodide has been added to the xanthide reaction to serve as a source of iodine in the presence of a small excess of active chlorine. The iodine complexes with the starch portion of the starch xanthide to produce a blue color which indicates the termination point for the addition of the cross-linking reagent. The amount of potassium iodide present affects the amount of sodium hypochlorite needed to produce the blue color at the end point. It is suggested that the iodide ion reduces the difference between relative reactivities of the two nucleophiles, that is, of the hypochlorite ion and the ionized xanthate group. The hypochlorite ion could form iodine or iodochlorine which are energetically more favorable to the cross-linking reaction.

In spite of the fact that the blue end-point reaction is related only to the first step of the consecutive reactions leading to starch xanthide, it does indicate the quantity of cross-linking reagent needed for obtaining the practical benefits of the material called starch xanthide. Unless the addition of the

cross-linking reagent is carried out swiftly with excellent blending, the reagent consumption is near 2 to 3 times that predicted by Equation (1). It is probable that more xanthate groups are converted to the sulfenyl chloride derivative than are needed for the second step of the reaction which involves the actual formation of the disulfide bonds. If the sulfenyl chloride derivative can react directly with hydroxyl groups on other portions of the starch xanthate (1) then it is possible cross-linking can occur with only one group rather than two xanthate groups.



This reaction relieves the system of some of the steric requirements for the formation of intermolecular cross-links between starch polymers.

If the reaction illustrated in Equation (2) can be demonstrated to actually occur, then steps should be taken to emphasize this cross-link over the disulfide bond since it makes more effective use of the xanthate derivative.

The concentration of the starch xanthate entering into the cross-linking reaction has an important effect upon the performance of the starch xanthide in paper. If the starch xanthate is too concentrated, starch xanthide coagulates before the end-point color change occurs. It was shown that microscopically detectable xanthide particles are ineffective for producing physically stronger paper. For starch xanthate preparations which have not been deionized, the critical concentration for avoiding coagulation is below 0.5% and seems to vary with the xanthate preparation.

The laboratory procedure for preparing ex situ starch xanthide was established with 0.33% starch xanthate. However, this concentration was dropped to 0.165% when xanthide coagulation occurred when using batches of D.S. 0.12 xanthate provided later on in the program. Coagulation occurred at the 0.165% concentration only when the solution had been neutralized with alum. It appears that the starch xanthide suspension is stabilized by mutual repulsion of like electrostatic charges. Further, that the ionic strength of the aqueous phase plays an important role in this stability. About 0.006 mole of monovalent salts per gram of xanthate starch is introduced from the xanthation reaction and the use of sodium hypochlorite as the cross-linking reagent. This estimate does not include bicarbonate and hydrosulfide ion from xanthation by-products. The ionic strength in the aqueous phase increases with the xanthate concentration, thereby limiting the maximum concentration at which stable xanthide suspensions may be prepared.

The laboratory ex situ xanthide preparations have a useful life of about one hour. Beyond that limit, coagulation begins so that the use of the starch xanthide in handsheets produces paper having lower wet and dry tensile strength in spite of increased retention efficiency.

Coagulation improves xanthide retention efficiency by entrapment of relatively large particles in the fiber mat during formation of the paper. However, starch xanthide is most effective in paper when it is adsorbed as a gel-like layer on the fiber surface. It is not identifiable by light microscopy except by the fact that it obscures normally visible fiber surface features. For judging starch xanthide distribution in paper it may be generalized that if the xanthide may be seen, it is not functional.

### SORPTION MECHANISM

The results of the sorption rate studies at 15 to 35°C. in  $2 \times 10^{-5}$  to  $2 \times 10^{-3}$  molar alum are consistent with the conclusion that starch xanthide is adsorbed by wood fibers by a physical process inhibited by the mutual repulsion of like electrokinetic charges. The sorption rate is relatively independent of the sorption temperature but is strongly affected by the alum concentration. Changes in the handsheet dry tensile strength with the time the fiber has been exposed to the xanthide suspension show similar activation energies and alum concentration dependence as the process rates based on the starch xanthide content of the handsheets. However, the sorption rates based on the wet tensile strength increments are much more temperature dependent at low alum concentration. For starch xanthide from D.S. 0.12 xanthate the wet tensile activation energy ranges from 2.8 to 10.6 to 20.5 kcal./mole in, respectively,  $2 \times 10^{-3}$ ,  $2 \times 10^{-4}$ , and  $2 \times 10^{-5}$  molar alum. This covers a range consistent with physical sorption to activation energies found for chemical reactions. By comparison, the activation energies based on starch xanthide sorption rates range from -3.8 to +1.1 kcal./mole and from -1.8 to +6.8 kcal./mole for the dry tensile data. It is suggested that the wet tensile data reflect a second process; lateral diffusion to fiber cross-over points and that wet tensile strength is more affected by the location of the xanthide deposit on the fiber than dry tensile strength.

The possibility of xanthide migration over the fiber surface seems to be borne out by the greater wet tensile strength of handsheets dried on a steam drum compared with air-dried handsheets. However, heating of the drum-dried paper in an oven further increases in wet tensile strength but at the sacrifice of some dry tensile strength.

If the sulfenyl chloride derivative [Equation (1a)] can exist in water for the length of time involved in preparing the ex situ starch xanthide and the handsheets, then chemical bonding between xanthide and fiber could occur. However, it is felt that short-range bonding, such as hydrogen bonding, is increased by driving of water molecules from the area of fiber bonding and is a more likely explanation of the heat-curing effect upon the wet tensile strength.

It was shown that the specific scattering coefficients of dry handsheets decrease linearly with increasing starch xanthide content and increasing wet and dry tensile strength. This is evidence that starch xanthide improves paper by increasing the bonded area. However, it does not offer insight into the higher temperature dependency of wet tensile strength development rates with the lower alum concentrations. A plausible explanation is not easily proposed. One possibility is that starch xanthide strengthens the fiber-to-fiber bonds within the paper structure but not the interfibril bonds within the fiber structure. Further, that the wet tensile strength is due to the strength of xanthide sheaths enveloping the fibers in the regions of the fiber-to-fiber bonds and that wet strength of the sheaths and the bonds between the sheaths exceed the cross-sectional tensile strength of the wet fiber.

The maximum rate of starch xanthide sorption obtained with fines-free kraft fiber is not changed as the degree of refining is increased. However, the minimum alum concentration necessary for the achievement of the maximum sorption rate increases with the degree of fiber beating or refining. The increased alum demand is not due just to the effects of increased specific surface area of the fibers. Fiber length, flexibility, number per gram, and specific gravity are factors also affected by the beating process and should have an influence upon the collision frequency of dispersed xanthide particles and fiber.

Starch xanthides prepared from starch xanthates having degrees of substitution of 0.08 and 0.12 appear to follow the same sorption mechanism since quite similar activation energies are obtained under the same conditions. However, as prepared by the laboratory procedure used for the sorption rate study, the D.S. 0.12 xanthide is preferable from the standpoint of retention efficiency and the development of wet tensile strength.

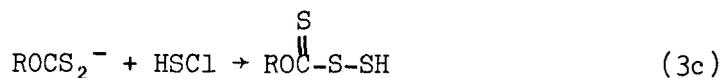
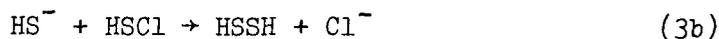
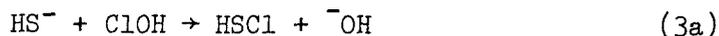
The optimum conditions derived from these studies call for titrating 0.165% solutions of D.S. 0.12 starch xanthate at pH 5.0-5.5 and 20°C. with 1% sodium hypochlorite (pH 5.0-5.5) as rapidly as possible to the appearance of the blue-colored end point occurring in the presence of a small amount of potassium iodide. Rapid titration is possible with excellent blending such as is obtained with an Eppenbach homogenizer. The starch xanthide is added to a fiber suspension of 0.04 to 2.0% consistency which contains  $2 \times 10^{-4}$  to  $2 \times 10^{-3}$  molar alum in the aqueous phase. The choice of fiber consistency is a matter of convenience, but higher consistencies are favored since less alum per unit weight of fiber is required to attain the specified aqueous concentration. Rosin may be used effectively if added first, followed by the alum and then by the starch xanthide. The suspension should be blended 15 minutes before forming the handsheets.

The dispersing effects of electrokinetic charges are to be maintained or maximized during the cross-linking step to form starch xanthide and to be minimized during sorption by wood fibers.

Removal of xanthation by-products before cross-linking reduces reagent consumption and improves the performance of the handsheet made with the reaction product of hypochlorous acid added relatively slowly to the starch xanthate. In contrast with excellent blending and rapid addition of the cross-linking reagent

the best handsheet physical test results are obtained when the xanthation by-products are not removed. It would appear that, with the combination of rapid addition and excellent blending of the reagent and starch xanthate, removal of xanthation by-products is detrimental to optimizing the use of starch xanthide. However, the performance of the xanthide-treated paper is better with high-speed mixing than low-speed mixing (rapid reagent addition vs. less rapid addition) even with the purified starch xanthate.

Removal of xanthation by-products removes hydrosulfide ions which are expected to react with hypochlorous acid by the mechanism shown for the starch xanthate in Equation (1). Thus:



The hydrosulfide ion is shown to interfere with starch xanthide bond formation and to lead to dihydrogen disulfide. The latter compound will ionize, react with hypochlorous acid and hydrosulfide ions to produce longer polysulfide chains and free sulfur. The polysulfides and free sulfur and possibly polysulfide cross-links through steps repeating those shown in Equation (3) probably are responsible for the yellow color of paper made with large amounts of starch xanthide. The compounds could also account for some of the brightness loss seen in paper made with bleached pulps. Therefore, purification of the starch xanthate before cross-linking is desirable when high levels of starch xanthide addition are needed and in those grades of paper where free sulfur must not be present.

In a comparison of whole and fines-free pulp, the whole pulp handsheets have better wet and dry tensile strength and better xanthide retention efficiency. This shows that the fiber fragments usually present in papermaking suspensions are not detrimental to the effective use of starch xanthide. This frequently is not true when other materials are used as internal adhesives.

Whole pulp suspensions prepared from bleached and unbleached softwood kraft fibers, bleached hardwood kraft fibers, and unbleached softwood groundwood all produce improved paper when treated with starch xanthate. The improvements are consistent with the properties of the fibers. The groundwood of course is the weakest pulp and shows the least improvement. An unexpected interaction is found between starch xanthide and the unbleached softwood kraft fibers but not with the unbleached hardwood kraft fibers. A high level of sizing is found which resembles rosin sizing but without having added any resin. It is believed to be an interaction between the softwood fines, perhaps by improved fines retention. The bleached softwood kraft fibers do show a small sizing effect with the whole pulp but none with the fines removed. The production of sized paper without adding a sizing agent is, of course, in itself a valuable property of the starch xanthide treatment. However, there may be ramifications that should be explored. For example, can ex situ starch xanthide be used as a pitch-control agent with southern softwood kraft pulps?

A rather remarkable improvement of folding endurance is seen with the bleached hardwood kraft whole pulp.

The effect of the scale of production of xanthide-treated paper was examined by comparing handsheets with paper produced on The Institute of Paper Chemistry's Continuous Web Former and on the cooperating agency's Pilot Machine. The scale

of production ranges from 30-gram batches of pulp, to 4 pounds, to many pounds of pulp. On the basis of percent improvement in sheet properties over the control, the handsheets and the pilot machine papers were quite similar. However, xanthide retention efficiency was about half that of the handsheet system. The papermaking scale does have an effect upon the performance of paper made with starch xanthide but there is good agreement with the largest and smallest scales compared.

The paper made on the Continuous Web Former compared two ways of adding starch xanthide: as prepared in a continuous cross-linking device and after collection and aging 15 minutes before pouring the dispersion into the 2% fiber suspension. The aged xanthide produced paper having a small advantage in physical properties while the unaged xanthide produced a more freely draining stock on the machine wire.

## DISCUSSION AND RECOMMENDATIONS

The use of starch xanthide in papermaking is a sophisticated concept of an internal adhesive. Starch xanthide is formed by the oxidative cross-linking starch xanthate which appears to be the result of consecutive  $S_N2$  displacements. Therefore, reaction conditions should be adjusted so that the xanthate groups are the strongest nucleophiles in the system. This means that, if sodium hypochlorite is to be used as the oxidizing agent, ionization of the hypochlorite ion must be suppressed to the point that hypochlorous acid is the predominant form of active chlorine. Adjustment of both the starch xanthate and the sodium hypochlorite solutions to pH 5.0 to 5.5 appears to be sufficient to achieve the desired relative reactivities. If a compromise must be made in a continuous cross-linking system, it is suggested that the neutralizing acid be blended with the sodium hypochlorite solution before it is mixed with the starch xanthate.

Starch xanthide sorption by wood fibers is a physical sorption process inhibited by mutual repulsion of like electrostatic charges whose effects may be overcome by the addition of alum. Alum concentrations in the range of  $2 \times 10^{-4}$  to  $2 \times 10^{-3}$  molar are adequate for all but the more highly refined fibers. The maximum sorption rate is independent of fiber refining and of the alum concentration above the minimum required to achieve the maximum rate. However, the minimum alum concentration requirement increases with increased refining. This effect appears to be due to more than just the increase in specific fiber surface area.

Starch xanthide sorbed by the fibers as what appears to be a gel-like coating, is effective in producing higher tensile strength, burst, and folding resistance in paper and in improving the wet tensile strength of paper soaked in

water for several hours. Coagulated xanthide particles retained by filtration mechanisms are relatively inert in regard to strength improvements.

The effects of electrokinetic charge repulsion should be maximized during cross-linking and minimized after the xanthide has been added to the fiber.

These electrokinetic charge effects upon xanthide sorption make the use of recirculated and reclaimed water desirable for dilution of the stock to operating consistencies. Dissolved salts in these low-quality water sources should improve xanthide retention and, therefore, its effectiveness. However, fresh water should be used to dilute the starch xanthate before cross-linking.

Fresh starch xanthide prepared separately from the fiber improves the drainage of the treated stock on the paper machine wire. This is an unusual property for an internal adhesive which permits the use of abnormally high rates of addition. The use of starch xanthide at the wet end of a paper machine could result in the production of paper normally produced by separate conversion processes. New grades of paper for specialized service are likely to evolve from the use of high levels of starch xanthide added at the wet end of the paper machine if this addition can be introduced into the industry.

Aside from the economic factors involved in using starch xanthide, which will control the use of this product in the more common grades of paper and which are being evaluated and reduced by the studies being conducted by the cooperating agency, two aspects are in need of further development. They are the dependency of sorption upon alum and the yellow color cast of papers made with starch xanthide.

Alum is a common papermaking ingredient. However, there are some papers for which alum is not required and some in which alum must not be present.

Consequently, for the widest application, alternatives to alum for starch xanthide retention must be found. A few simple exploratory experiments, outside the scope of this project, have demonstrated that there are ways to retain starch xanthide without alum.

The problem of the yellow color cast may be more troublesome. Very likely the yellow color is due to polysulfide and sulfur formed during the addition of hypochlorous acid to cross-link starch xanthate as suggested by Equation (3). The hydrosulfide ion forming the colored material upon reacting with hypochlorous acid can be oxidized with the hypochlorite ions to form sulfite and sulfate ions which are colorless. This suggests that treatment of the xanthide preparation with sodium hypochlorite could remove the colored material. However, as was discussed in the report on Phase II of this project, hypochlorite ions and hydroxyl ions in the alkaline reagent are expected to break starch xanthide links. Thus, oxidative removal of colored products with the reagent would tend to undo the sequence of processes leading to starch xanthide. It would perhaps be best if hydrosulfide ions or potential hydrosulfide ions (excluding the xanthate groups) were removed prior to cross-linking. If aeration is used for this purpose, then the odor and the toxic properties of hydrogen sulfide must be dealt with. Perhaps this could be done by scrubbing the gaseous effluent in a tower containing sodium hypochlorite or containing chlorine gas with liquid water present. The effluent from the chlorine--water tower may have use in neutralizing the xanthation mixture and in effecting cross-linking.

The continuous xanthation process and the continuous cross-linking units already developed by the cooperating agency are essential elements of a systems engineering concept for incorporating starch xanthide into the commercial paper-making processes. It is felt that a completed systems concept would hasten the

commercial acceptance of starch xanthide as an internal adhesive producing wet strength. The transfer of technology at the development--production interface is decreased. This is particularly important when the interface occurs between independent organizations.

On a more academic level, it is felt that the products of the cross-linking reaction should be examined. It is possible that disulfide cross-links are not the reason starch xanthide behaves differently than starch xanthate. The use of hypochlorous acid leads to the proposal of xanthate sulfenyl chloride groups for the product of the first bimolecular reaction of the consecutive reactions leading to xanthide bonds [see Equation (1)]. It may be possible for the sulfenyl chloride to react with starch hydroxyl groups to produce one cross-link per xanthate group instead of one cross-link from two xanthate groups. The point is that the existence of the xanthide cross-link has not been shown for starch xanthate oxidized with hypochlorous acid. It has been assumed on the basis of a rather extensive accumulation of knowledge of xanthate chemistry. The behavior of starch xanthate treated with sodium tetrathionate is so different from treatments with hypochlorous acid that there must be some difference in the structure of the cross-linked products. Sodium tetrathionate forms a gel whereas the hypochlorous acid forms a product that behaves like a finely divided dispersion or the result of the coagulation of such a dispersion. With the former reagent, the first intermediate would be:

$$\text{ROC}-\overset{\text{S}}{\parallel}\text{S}-\text{S}-\text{SO}_3^-$$

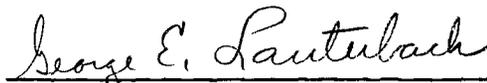
which can enter only into a second displacement reaction to form a true xanthide linkage (or be destroyed by hydrolysis).

The result of such a fundamental study should at least produce a better understanding of the xanthide cross-linking reaction thereby leading to more precise control of the reaction.

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