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The Chemical Composition and Conservation of Late 19th and Early 20th Century Sequins

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Sequins from the late 19th and early 20th centuries have been made from a wide variety of natural and synthetic materials, including gelatin, varnishes, cellulose nitrate and various plastics, and metals such as gold, silver, steel and brass. Treatments using heat, water or humidity, or dry-cleaning solvents can severely damage or destroy a number of these materials. Sequins from a random selection of costumes were analyzed by infrared spectroscopy. Results from this analysis are compared to and combined with other reported analyses to obtain a list of materials identified in sequins. Conservation problems with these materials are discussed. Spot tests that can be used in the field to identify the most problematic materials are described, including the diphenylamine test for cellulose nitrate, copper (II) sulfate test for protein, chromotropic acid test for formaldehyde, and the iodine/potassium iodide test for starch.

Les paillettes datant de la fin des années 1900 et du début du 20^e siècle sont composées d'une variété de matériaux naturels ou synthétiques, tels que de la gélatine, des résines, du nitrate de cellulose, divers plastiques et métaux comme l'or, l'argent, l'acier et le laiton. Les traitements qui requièrent de la chaleur, de l'eau ou de l'humidité, ou bien des solvants tels que ceux utilisés commercialement pour le nettoyage à sec, peuvent sérieusement endommager ou même détruire plusieurs de ces matériaux. Des paillettes provenant d'un groupe aléatoire de costumes ont été analysées par spectroscopie infrarouge. Les résultats d'analyse ont été comparés et ajoutés à d'autres résultats publiés, afin de créer une liste de matériaux dont sont composées les paillettes. Les problèmes de conservation-restauration reliés à ces matériaux sont discutés. Des tests ponctuels que l'on peut faire aisément dans les musées pour identifier les matériaux les plus problématiques sont décrits, dont le test de diphenylamine pour identifier le nitrate de cellulose, le test du sulfate de cuivre (II) pour identifier la présence de protéines, le test à l'acide chromotropique pour identifier le formaldéhyde et le test d'iode et d'iodure de potassium pour identifier l'amidon.

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Introduction

Decorative sequins and other trim items on artifacts such as those in **Figure 1** from the late 19th or early 20th century should be examined carefully to determine their composition. The authors have previously reported that buttons, beads, and sequins on Schiaparelli costumes contained cellulose nitrate, gelatin, and shellac.¹ In the literature, sequins are described as having been made from a wide array of materials: gelatin, varnishes, cellulose nitrate and various plastics, horn, tortoiseshell, cardboard, and metals such as gold, silver, steel and brass.² Heat, water or humidity, or dry-cleaning solvents can severely damage or destroy a number of these materials and conservators should be alert to these potential issues.

Problems with moisture-sensitive costume decorations first came to the attention of one of the authors (C.P.) while visiting a small museum in the 1980s for the mobile lab service of the Canadian Conservation Institute (CCI). The curator requested advice concerning a number of garments in the collection. Once the costume boxes were opened, it was apparent that the layers of sequined gowns had become adhered to each other. The previous summer had been particularly hot and humid with extreme fluctuations in the environment of the storeroom. The curator was convinced that the sequins and beads decorating these gowns were composed of gelatin.



Figure 1. Examples of Elsa Schiaparelli (Italian, 1890-1973) designs using sequins. Detail of (Left) *Evening Ensemble*, 1937-1938, The Metropolitan Museum of Art, gift of Julia B. Henry, 1978 (1978.288.19a-c); and (Right) *Evening Jacket*, 1938, rayon crepe embroidered with metallic thread and polychrome sequins, The Metropolitan Museum of Art, gift of Mrs. J.R. Keagy, 1974 (1974.338.2). Images © The Metropolitan Museum of Art.

To determine if this was an isolated event or a bigger problem impacting other collections, sequins were analyzed by infrared spectroscopy to identify the range of materials and their distribution in collections. Samples from a range of late 19th and early 20th century objects were obtained from various collections in Canada and the United States (**Table I**). Because instrumental analysis is not always available to conservators, another goal of the investigation was to develop simple spot tests that could be carried out in the field to identify the materials that cause the most problems. The overall aims of this paper are to alert readers to the presence of moisture-sensitive sequins in collections, and to record information which could help develop expertise in recognizing material composition and potential problems.

Sequins

During the course of the investigation, the authors observed that sequins were widely used to ornament clothing and accessories such as fans, hats, shoes, and purses. Notably, they were an especially popular trim for black garments, and possibly provided a way to relieve the sombreness of mourning garb. Based on the objects that were examined, sequins were extensively used as decorative trimmings in the late 19th century and continuing into the 1930s. During this period, research and development were focused on the production of new and inexpensive materials as substitutes for the dwindling supplies of jet, ivory, tortoiseshell and mother of pearl. Advances in science and technology during the industrial revolution created great changes in the social and economic structure of society. Fashionable costume became more available to the middle and lower classes in a less expensive form that mimicked more luxurious materials.

Metallic Sequins (Spangles, Paillettes, Paillons)

Until the late 19th century, most sequins were made from metals such as gold, silver, steel and brass. Sequins sometimes were referred to as spangles, paillettes or paillons. Details of their production were outlined in *The Art of the Embroiderer*, a publication from 1770. According to this author, paillettes are “small rings of gold flattened with a planishing hammer. There is a small hole in the middle of the disk through which a threaded needle can pass to sew on the paillette. Paillettes come in various sizes [and] shapes in: oval, heart, almond, lozenge, square, clover, rosette, star or round [...]. Black steel paillettes were available for mourning dress [...]. Paillons are coloured spangles cut from thin sheets of silver and varnished in different colours, larger than paillettes, they are sometimes coloured by workers after being sewn in place.”³

Gelatin Sequins

Although there seemed to be considerable information about metallic sequins, little was available on their gelatin counterparts. Gelatin, a virtually colourless and tasteless water-soluble protein prepared from collagen, was commonly used in food preparation and photographic processing and for glue. This material was derived from the parings of animal hides, skins or bones.

Isinglass, another collagenous material, was obtained from fish bladders. Gelatin was cheap and versatile. It was often laminated with cellulose nitrate.

To make sequins, gelatin must be prepared in sheet form. Dick's *Encyclopedia of Practical Receipts and Processes of 1872* gives a description for producing gelatin wafers:

“Dissolve fine glue or isinglass in water, so that the solution when cold may be consistent. Pour it hot on a plate of glass (previously warmed with steam and slightly greased) fitted in a metallic frame whose edges are just as high as the wafers should be thick. Lay on the surface a second glass plate, also hot and greased, so as to touch every point of the gelatine while resting on the edges of the frame. By its pressure the thin cake is rendered uniform. When the glass plates have cooled, the gelatine will be solid, and may be removed. It is cut into discs of different sizes by means of proper punches.”⁴

The gelatin sheet could be coloured by adding aniline dyes or other suitable colouring material.

Gelatin sequins are described in a draper's manual edited by William Murphy⁵ in 1914:

“Sequins are little round disks made of celluloid or gelatin, but mostly of the latter substance, on account of its non-inflammable nature. In the centre is a little hole through which the thread may be passed in order to affix them for ornamental purposes. They are made in black and all colours, in sizes ranging from 1/8 in. to 1/2 in. [0.3 to 1.3 cm] in diameter.

They are used both for millinery trimmings and evening dresses, are lightweight and extremely effective in appearance. Sequins are made in Offenbach, Belgium, Beyrouth [*sic*], Luneville, Ulnice [*sic*], Paris and London.

Offenbach and the district around it may be considered the German centre for millinery trimmings of all descriptions, such as sequin net – imitation wings and mounts, of which beads and sequins form the component parts. Luneville in France, is also a centre from which large quantities of millinery trimmings of a similar character emanate. In the village of Buckinghamshire the beaded and sequined net trade is still carried on to some extent though foreign competition in this as in many other trades, has considerably diminished the output.”

Cellulose Nitrate Sequins

In the 1840s, European scientists discovered that by reacting cellulosic materials such as wood dust, linen or paper with nitric acid, they could isolate a substance that could be molded. In 1855, an Englishman, Alexander Parkes, succeeded in developing a form of cellulose nitrate called Parkesine, using plasticizers such as camphor or castor oil. He manufactured buttons, buckles and other ornaments from this material. Celluloid was the American trade name for cellulose nitrate. The history of cellulose nitrate is well documented in the literature.⁶

Cellulose nitrate was not necessarily an inexpensive material, but it was cheaper than the materials that it imitated. Its advantage over natural materials was the lack of restriction on size, shape, design and colour. It was flexible, durable and not subject to shrinkage or cracking like wood, horn or ivory. Cellulose nitrate was also versatile and could be made to imitate more expensive materials, such as horn, tortoiseshell or mother-of-pearl. Hillman⁷ discusses a method developed in 1884 to give an iridescent appearance to sequins by utilizing translucent cellulose nitrate mixed with finely ground mica. The iridescence was enhanced with the addition of fluorescent dyes or phosphorescent pigments. Later, lead phosphate was used for the same purpose. It was evident that these shimmering iridescent sequins enjoyed great popularity, judging from their prevalent use on gowns. Two major disadvantages of cellulose nitrate are that it is extremely flammable, particularly in the form used for motion picture film, and that it degrades to produce acids that can damage adjacent material.⁸

Other Sequin Materials

Although listed as sequin materials by Kerr,² cardboard, horn and tortoiseshell were not found in the sequins analyzed in this study. Casein-formaldehyde was used extensively to make buttons, but because it cannot be made in sheets thin enough, it was not used for sequin making. As plastic sheet materials like cellulose acetate, polyester and polystyrene, as well as methods for decorating these, came onto the market, they were used to make sequins, especially in modern times. Methods of coating plastics, such as electroplating and vacuum coating, eventually enabled substitution of metal sequins with sequins made of metallized plastics, especially metallized polyester. Modern plastics have the advantage of being less sensitive to moisture, but many are more sensitive to organic solvents such as those used in dry-cleaning.

Methods of Analysis

Samples were analyzed by Fourier Transform Infrared (FTIR) spectroscopy using a diamond anvil microsample cell and by wet chemical spot tests.⁹ Analysis of metal components was not performed.

If instrumental methods of analysis, especially FTIR, are not available, then the following chemical spot tests, described in detail in the **Appendix**, may be used to identify some of the materials in decorative elements.

- When diphenylamine test reagent is applied to a sample of cellulose nitrate, a blue stain is produced on the sample surface or in the solution surrounding the sample.
- When sodium hydroxide is applied to a sample of protein that has reacted with blue copper sulfate test solution for a few minutes, a violet stain is produced on the surface or in the solution surrounding the sample.
- When a colourless, strongly acidic chromotropic acid test solution reacts with formaldehyde in the sample or produced by hydrolysis of the sample, a violet-blue coloured solution

is produced under the special conditions of the test.

- When iodine/potassium iodide test solution is applied to starch, a bluish-purple stain is produced on the surface of the sample or in the solution surrounding the sample.

Opaque cross-sections of sequins mounted in Bioplastic polyester resin were prepared using standard petrographic grinding and polishing techniques under dry conditions, and transparent thin sections were microtomed from Spurr's epoxy embedments. These were examined using incident light microscopy.

Results of Analysis

A description of the samples and the detailed results of their analysis is given in **Table I**. For this study, the sequins are called historic if they are pre-1930 and modern if from 1930 or later. Some sequins have dates of provenance associated with them; these are listed in the table. Those without dates were classified as historic or modern based on connoisseurial appraisal of the appearance of the sequins or the garment. Samples analyzed were chosen from various sources to represent either a distinct colour or shape of decoration on the same object or from different objects. Thirty historic sequins and seven modern sequins were sampled and analyzed to determine their composition. The full analytical report is available from CCI.⁹

Historic (Pre-1930) Sequins

All thirty historic sequins analyzed were made predominantly of gelatin, with nine having thin cellulose nitrate coatings. No sequins were found to be made of casein plastic (casein-formaldehyde).

Nineteen of the thirty historic gelatin sequins were opaque black as seen on the garment, but appeared blue or violet when viewed in thin section. None contained carbon black pigments, but rather their colour was due to having been deeply dyed with blue or violet water-soluble dyes. None of the black sequins contained cellulose nitrate and none was laminated.

Six of the eleven non-black historic gelatin sequins were colourless or whitish and showed iridescence. All had cellulose nitrate surface layers covering an iridescent pigment layer on the gelatin core. Three of the remaining non-black historic gelatin sequins were greyish brown. These had a brown pigmented layer on the gelatin core covered by a cellulose nitrate coating that sometimes was iridescent. One historic gelatin sequin was a laminate of two thin transparent blue-dyed cellulose nitrate layers on each side of a colourless gelatin core.

Modern (1930 and Later) Sequins

Of the seven modern sequins, one was gelatin, one was a laminate of gelatin and metal foil, one was metal with a pink-coloured resin coating (possibly shellac), and four were cellulose acetate (or diacetate) plasticized with triaryl phosphates (or






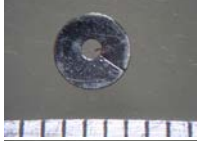



(Continued on page 52)

Table I: Description and Composition of Sequins on Various Types of Objects.

Note: “Unattached” indicates that sequins were associated with a particular costume or collection of costumes but not attached to fabric (“loose in the box”).







* *Contributing institutions are as follows: AEAC: Agnes Etherington Art Centre, Kingston, ON, Canada; GVMA: Greater Vernon Museum and Archives, Vernon, BC, Canada; Brome: Brome County Museum, Knowlton, QC, Canada; Parks: donation from Parks Canada Conservation Lab, Ottawa, ON, Canada; Paulocik: private collection of Chris Paulocik.*

** *Analytical Methods: FTIR: Fourier Transform Infrared Spectroscopy; DPA: spot test for cellulose nitrate using diphenylamine (see Appendix); CTA: spot test for formaldehyde using the disodium salt of chromotropic acid (see Appendix); Biuret: spot test for protein using copper (II) sulfate (see Appendix).*

Historic Sequins (pre-1930)			
Type of Object; Date (if known); Institution*	Sequin Description (colour and shape)	Composition (method of analysis**)	Photograph (scale units are 1 mm)
Unattached; AEAC	Black (violet-grey when thin), 6-facetted circle	gelatin (FTIR) <i>no cellulose nitrate (DPA)</i>	
Unattached; AEAC	Black (blue-grey when thin), unfacetted circle	gelatin (FTIR) <i>no cellulose nitrate (DPA)</i>	
Unattached; AEAC	Black (blue-violet when thin), large circle	gelatin (FTIR) <i>no cellulose nitrate (DPA)</i>	
Black bonnet with horse hair; ca. 1880; Paulocik	Black (blue when thin), circle	gelatin (FTIR) <i>no formaldehyde (CTA)</i> <i>no cellulose nitrate (DPA)</i>	
Unattached; Brome	Black (violet when thin), 6-facetted circle	gelatin (FTIR) <i>no cellulose nitrate (DPA)</i>	
Evening Dress; GVMA 961.18.1	Black, circle	gelatin (FTIR) <i>no cellulose nitrate (DPA)</i>	
Evening Dress; GVMA 961.18.2	Black (blue when thin), circle	gelatin (FTIR) <i>no formaldehyde (CTA)</i> <i>no cellulose nitrate (DPA)</i>	
Dress (English); GVMA 976.125.14	Black (blue when thin), curled 2-holed oval	gelatin (FTIR)	
Evening Gown with Train; GVMA 976.125.15	Clear colourless with bluish iridescence, circle	gelatin (FTIR) cellulose nitrate skins (DPA) <i>no formaldehyde (CTA)</i>	

Historic Sequins (pre-1930)			
Type of Object; Date (if known); Institution*	Sequin Description (colour and shape)	Composition (method of analysis**)	Photograph (scale units are 1 mm)
Belt; GVMA 986.38.29	Black (violet when thin), oval	gelatin (FTIR) <i>no formaldehyde (CTA)</i> <i>no cellulose nitrate (DPA)</i>	
Jet Scarf; <i>ca.</i> 1890; Paulocik	Black (blue when thin), large 2-facetted circle	gelatin (FTIR) <i>no formaldehyde (CTA)</i> <i>no cellulose nitrate (DPA)</i>	
Jet Scarf; <i>ca.</i> 1890; Paulocik	Black (violet when thin), large 4-holed oval lozenge	gelatin (FTIR) <i>no formaldehyde (CTA)</i> <i>no cellulose nitrate (DPA)</i>	
Jet Collar; <i>ca.</i> 1890; Paulocik	Black (violet when thin), 2-holed square	gelatin (FTIR) <i>no formaldehyde (CTA)</i> <i>no cellulose nitrate (DPA)</i>	
Dress; <i>ca.</i> 1900; GVMA	Translucent greyish brown, almond shaped ring	gelatin (FTIR) <i>no formaldehyde (CTA)</i> <i>no cellulose nitrate (DPA)</i>	
Dress; <i>ca.</i> 1900; GVMA	Translucent greyish brown, circle	gelatin (FTIR) protein (Biuret) <i>no formaldehyde (CTA)</i> <i>no cellulose nitrate (DPA)</i>	
Dress; <i>ca.</i> 1900; GVMA	Translucent greyish brown, 7-pointed star	gelatin (FTIR) cellulose nitrate (DPA) trace <i>no formaldehyde (CTA)</i>	
Dress; <i>ca.</i> 1900; GVMA	Hazy colourless, circle	gelatin (FTIR) cellulose nitrate skins (DPA)	
Unattached; Parks	Small black (blue when thin), circle	gelatin (FTIR) <i>no cellulose nitrate (DPA)</i>	
Unattached; Parks	Medium black (blue when thin), circle	gelatin (FTIR) <i>no cellulose nitrate (DPA)</i>	
Unattached; Parks	Small black (blue when thin), circle	gelatin (FTIR) <i>no cellulose nitrate (DPA)</i>	

Historic Sequins (pre-1930)			
Type of Object; Date (if known); Institution*	Sequin Description (colour and shape)	Composition (method of analysis**)	Photograph (scale units are 1 mm)
Unattached; Parks	Blue with clear colourless core and two thin blue surface layers, circle	gelatin core (FTIR) cellulose nitrate skins (FTIR and DPA)	
AEAC C53.488.3	Black (blue when thin) circle	gelatin (FTIR) <i>no cellulose nitrate (DPA)</i>	
AEAC C53.488.3	Black (blue when thin) with whitish bloom, circle	gelatin (FTIR) calcium sulfate bloom (FTIR) <i>no cellulose nitrate (DPA)</i>	
AEAC C54.501.6	Iridescent white, circle	gelatin (FTIR) cellulose nitrate skins (DPA)	
AEAC C54.501.6	Black (grey when thin), curled 6-facetted circle	gelatin (FTIR) <i>no cellulose nitrate (DPA)</i>	
AEAC C66.576.1	Black (blue when thin), circle	gelatin (FTIR) <i>no cellulose nitrate (DPA)</i>	
Trim (Edwardian); <i>ca.</i> 1900; Paulocik	Iridescent off-white, small 6-facetted circle	gelatin core (FTIR) cellulose nitrate skins (FTIR and DPA) <i>no formaldehyde (CTA)</i>	
Trim (Edwardian); <i>ca.</i> 1900; Paulocik	Iridescent off-white, tiny 6-facetted circle	gelatin core (FTIR) cellulose nitrate skins (FTIR and DPA) <i>no formaldehyde (CTA)</i>	
Trim; <i>ca.</i> 1930; Paulocik	Iridescent off-white, large facetted circle	gelatin core (FTIR) cellulose nitrate skins (FTIR and DPA) <i>no formaldehyde (CTA)</i>	
Trim; <i>ca.</i> 1930; Paulocik	Iridescent off-white, small 6-facetted circle	gelatin core (FTIR) cellulose nitrate skins (FTIR and DPA) <i>no formaldehyde (CTA)</i>	
			Left: un-wetted; Right: wetted
			
			Left: un-wetted; Right: wetted

Modern (1930 and later) Sequins			
Type of Object; Date (if known); Institution*	Sequin Description (colour and shape)	Composition (method of analysis**)	Photographs (scale units are 1 mm)
Hat Feather; Paulocik	Blue opaque, un laminated, circle?	cellulose acetate (FTIR) <i>no cellulose nitrate (DPA)</i>	
Hat Feather; Paulocik	Green transparent, with green layers on clear core, circle	cellulose acetate (FTIR) N. B. Skins swell in DPA reagent <i>no cellulose nitrate (DPA)</i>	
Hat Feather; 1930s; Paulocik	Pink opaque, un laminated, circle	cellulose acetate (FTIR) <i>no cellulose nitrate (DPA)</i>	
Unattached; Parks	Pink with clear colourless core and one pink and iridescent surface layer, circle	gelatin core (FTIR) metal foil <i>no cellulose nitrate (DPA)</i>	<i>Photograph not available</i>
Unattached; Paulocik	Blue, circle	gelatin (FTIR) <i>no cellulose nitrate (DPA)</i>	
Unattached; Paulocik	Bluish pink, circle	cellulose acetate plasticized with triaryl phosphates (FTIR) <i>no cellulose nitrate (DPA)</i>	
Unattached; Paulocik	Metal with pinkish coating, circle	shellac coating (FTIR) N. B. Some orange fluorescence in ultraviolet light	

tricresyl phosphate). No cellulose nitrate was detected in any of the modern sequins.

Discussion

The results of the analysis of this suite of sequins can be compared to the composition of similar material in the literature.

Kerr² stated that sequins could be stamped from any material which could be formed or cast into a sheet, including:

- metals, uncoated or coloured by coating a metal sheet with a coloured film, often cellulose acetate, or a natural varnish
- horn and tortoiseshell
- cardboard, covered with a foil of silver or copper (glued-on spangles)
- cellulose nitrate and gelatin, used from the late 1880s until probably well into the 20th century
- cellulose acetate and cellulose butyrate, used from around 1920 to the 1960s cellophane (regenerated cellulose)
- polystyrene
- aluminum film or foil, laminated between two layers of

- plastic such as celluloid or cellulose acetate/butyrate
- polyester, with a coloured laminating adhesive if coloured sequins were desired
- metallized polyester or polyamide films, introduced in the late 1950s, used for sequins punched from a single layer of film on which metallic effects were produced by vacuum-coating a thin layer of metal on the reverse side
- a three-layer sandwich of aluminized film which could be coloured with synthetic resin before lamination between two sheets of clear film.

Mackinnon¹⁰ reported the IR spectroscopic analysis of forty sequins. The materials identified in her samples are shown with those found in the current study in **Table II**, sorted by date of costume (including those which were not dated, in order to allow inclusion of all data).

Although this is a relatively small sample of sequins, the combined results from the two studies indicate that:

- metal foil or coated metal foil sequins were more common on historic costumes than on modern costumes

Table II. Abundance of Materials in Sequins Sorted by Date of Costume.

MATERIAL	NUMBER OF SEQUINS				
	Paulocik and Williams (this work) - Total: 37		Mackinnon (2003) - Total: 40		
	Historic (Pre-1930) Total: 30	Modern (1930 or later) Total: 7	Historic (Pre-1930) Total: 13	Modern (1930 or later) Total: 9	Undated Total: 18
Metal (sheet or foil substrate)		1 (n.d.) metal foil /gelatin laminate 1 (n.d.) resin-coated	4 (2 pre-1914) 1 resin-coated	1 cellulose nitrate- coated	2 1 gelatin/metal/ resin laminate
Gelatin	6 (pre-1914) 15 (n.d.)	1	6 (1 pre-1914)	1	6
Gelatin plus cellulose nitrate (CN)	1 (pre-1914) 1 (pre-1914, trace CN) 2 (pre-1914, iridescent) 2 (ca. 1930, iridescent) 1 (n.d.) laminated 2 (n.d.) iridescent			3 iridescent 1 metallized	1 iridescent 1 metallized
Gelatin plus polyester					1 metallized
Gelatin plus unknown			2		1
Polyester				1 laminated	1 1 metallized 1 iridescent
Polyester plus cellulose nitrate				1 metallized	2 iridescent†
Cellulose acetate		1 1 (n.d.) laminated 2 (n.d.)		1†	

† After reinterpretation by Williams of spectra published in Mackinnon's thesis, composition of some Mackinnon samples were reassigned as follows: #25 changed from cellulose nitrate to cellulose nitrate plus polyester, #36 changed from polyester to cellulose acetate, and #39 changed from polyester to cellulose nitrate plus polyester. The changes are reflected in the table.

- most non-metallic sequins on historic costumes were made of gelatin
- no sequins analyzed were made from cellulose nitrate sheet
- cellulose nitrate was present only as a coating to create colours on metal sequins and iridescent effects on gelatin and plastic sequins
- sequins with thin iridescent cellulose nitrate coatings were found on historic and modern costumes
- non-metallic sequins with metallization were found only on modern costumes
- sequins made of cellulose acetate and of polyester (*i.e.*, poly(ethylene terephthalate)) appeared only on modern costumes
- non-metallic sequins were made from a relatively small number of materials, namely gelatin, cellulose acetate, and polyester
- complex forms like laminates and coated plastics were common.

The relatively widespread use of gelatin for the production of sequins became the focus of this survey, due to the potential problems related to the water-sensitive nature of the material. Analysis revealed variations which often utilized gelatin as a core material and cellulose nitrate as an exterior laminate or coating.

The numerous examples found suggest that this modification was a popular method for producing iridescent surfaces that imitated mother-of-pearl.

The analysis confirmed that sequins of a wide variety of compositions can be found in typical collections. Conservators should therefore characterize their materials before undertaking any treatment. Some sequins are made of metals coated with coloured lacquers based on resins such as shellac or cellulose nitrate. If the sequin or other decorative embellishment is not metallic, it is most likely one of the following (and any of these could be pigmented or dyed):

- gelatin
- a cellulose nitrate/gelatin laminate
- other plastic material.

Problems with Sequins on Costume

Heat

Heat, especially in combination with high relative humidity, will noticeably soften gelatin and make it susceptible to permanent damage. Sequins may curl and distort with heat and will readily imbibe dust and grime.

Heat accelerates all deterioration reactions, including oxidation and hydrolysis. Cellulose nitrate, and to a lesser extent, cellulose acetate, can break down, resulting in loss of the dramatic surface effects such as iridescence, and disintegration. In addition, the acidic and oxidizing gases produced during degradation corrode metals, and discolour and disintegrate fabrics, contributing to their deterioration. Evidence of many of these symptoms was observed while examining objects during the course of the investigation.

Sequins made of the more modern plastics are not so susceptible to damage by heat and moisture but may be damaged by direct contact with hot irons.

Solubility in Water

Gelatin sequins will swell and become permanently distorted if immersed in water for even a short time (**Figure 2a** and **2b**). If the sequin is secured to the substrate by threads, or composed of layers that swell at different rates, then it may be forced to distort when it swells (**Figure 3**). Prolonged immersion in water can cause eventual dissolution (**Figure 4**). Even if a sequined artifact is removed from water after only a few minutes, permanent deformation will occur. Any suspicious decorative trim should be tested using the spot tests described in the **Appendix**.

In particular, sequins containing cellulose nitrate in combination with gelatin must be identified carefully. A fellow conservator recounted to the authors his experiences with this material after a spot test on the outer portion of a sequin indicated cellulose nitrate. He proceeded to wash the artifact, only to see with horror the sequins beginning to buckle. Apparently the core was composed of gelatin, which swelled; this can cause the cellulose nitrate to pull away (**Figure 5**), crack in an alligator pattern (**Figure 6**), or lose iridescence (**Figure 7**).

Moisture Sensitivity

High humidity will cause gelatin to soften and develop a sticky surface. The authors have seen examples of gowns where the sequins had lost their shiny exterior and developed a dull bloom in their appearance. This probably resulted from episodes of high humidity, which caused a sticky surface to develop on the sequins to which dust and dirt could adhere. Other examples that were examined exhibited a whitish coating. During the study, analysis revealed that the whitish powder was actually gypsum, which may have come from plaster, drywall, or cement dust in the storage area. Another possible explanation for hazy surfaces on sequins is disruption of the gelatin surface or changes in the cellulose nitrate coating. Additionally, gelatin provides a good substrate for mould growth and this may account for some whitish surfaces. Damage to sequins can also occur in underarm areas of garments, caused by moisture and perspiration residues. Storage in humid environments can cause adhesion between sequins and adjacent materials. It is therefore important to ensure that storage facilities and exhibition spaces are kept clean, and to maintain a stable relative humidity (RH) and temperature, preferably between 30 and 50 percent RH and below 18°C.

Treatments involving steaming of garments with gelatin sequins should be strictly avoided.

Plastics other than gelatin are much less susceptible to distortion by exposure to high humidity, but can undergo hydrolysis reactions. This is particularly noticeable for thin cellulose nitrate coatings, and leads to loss of gloss and iridescent effects. Colouring of sequins was often achieved with a surface coating of varnish. In these cases, moisture absorption can cause swelling and mechanical stresses leading to delamination, or chemical reactions of hydrolytic decomposition products can occur, leading to colour changes or corrosion.

In metallic sequins, oxidation of the metal or the development of corrosion products becomes more likely as humidity increases, and this is exacerbated if hygroscopic materials like perspiration residues are present.

Solvent Sensitivity

The sensitivity of sequins to water is not surprising, since most of the ones analyzed are composed of gelatin, a very water-sensitive material. The gelatin does not appear to be hardened or cross-linked by the addition of formaldehyde. No formaldehyde was detected in any sample by the very sensitive chromotropic acid test (see **Appendix**) on acid-hydrolyzed sequins.

The laminar structure of some sequins will severely complicate treatments. Non-aqueous treatments such as dry-cleaning may dissolve some of the lamina and cause drastic colour shifts. For example, gelatin is relatively unaffected by ethanol and acetone. However, the pink-coloured shellac on some metal sequins will dissolve, resulting in a change of colour of the sequin and probably staining of the garment. In a similar way, cellulose nitrate surface lamina will dissolve in acetone (and sometimes ethanol), causing loss of iridescent pigments and coloured layers (see **Figure 7**). Dissolution of coloured lamina will change the colour of the decorations on the textile. Bleeding colours may stain the underlying textile.

Examination of Teas partial solubility parameter charts, such as those given by Horie¹¹ will give some idea of the susceptibility of the various sequin components to common solvents. Gelatin should be relatively unaffected by organic solvents. Hydrocarbon solvents like Stoddard's solvent and mineral spirits should not affect cellulose nitrate, cellulose acetate or shellac. Natural resin varnishes (such as dammar) are soluble in hydrocarbons. Information on the effect of dry-cleaning solvents like perchloroethylene on shellac or cellulose nitrate is ambiguous. The *Handbook of Common Polymers*¹² indicates that cellulose nitrate is swollen by ethylene dichloride. Horie¹¹ reports that dichloromethane, chloroform, carbon tetrachloride, trichloroethylene, and trichloroethane are non-solvents for lacquer and adhesive grades of cellulose nitrate. Shellac seems to be less soluble than cellulose nitrate.

When used in conjunction with knowledge of chemical composition, these charts can provide some guidelines for



Figure 2a. Gelatin/cellulose nitrate sequins on gown before immersion in water. Each sequin is flat and has a smooth circular perimeter. The lighter sequins have an iridescent appearance created by optical interference of reflections from a very thin layer of colourless cellulose nitrate on a thick colourless gelatin base.

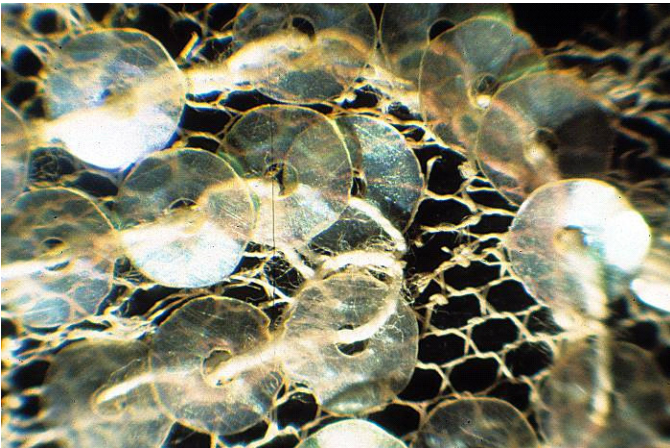


Figure 2b. After immersion, gelatin/cellulose nitrate sequins are warped and have wavy perimeters. (See also **Figure 5**, showing the cellulose nitrate layer delaminated from the gelatin base, and **Figure 8**, a diphenylamine-stained cross-section). Sequins diameter: 4 to 5 mm.



Figure 3. A gelatin sequin restrained by stitches to a substrate has swelled and buckled upwards as a result of exposure to water. It is possible that the dyes used to colour the sequin will bleed. This sequin is about 12 mm in diameter.

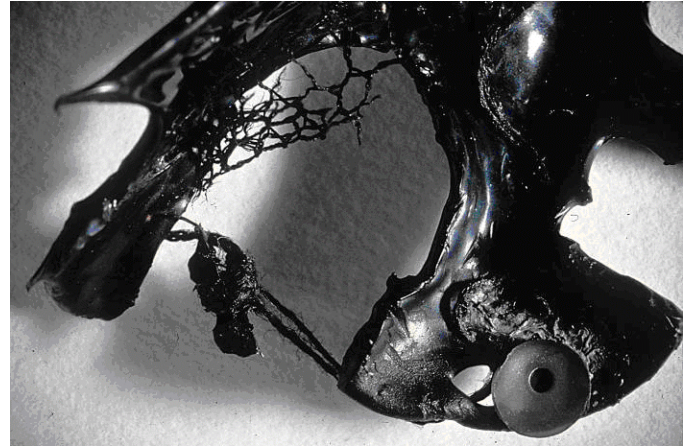


Figure 4. After immersion in water for 20 minutes, a gelatin sequin has started to dissolve. The seed bead at lower right is about 1 mm in diameter.

treatment proposals. It must be remembered that sequins or decorations of several different compositions can be found on one garment because different effects are produced by different materials and processes. These will have different solvent resistance properties. The suitability of a treatment can only be ascertained by testing each solvent on each sequin type.

Mechanical Damage

Often used to lavishly decorate garments, metal sequins can prove quite weighty, sometimes creating mechanical damage to their more lightweight substrates and resulting in fracturing and losses to fabrics.

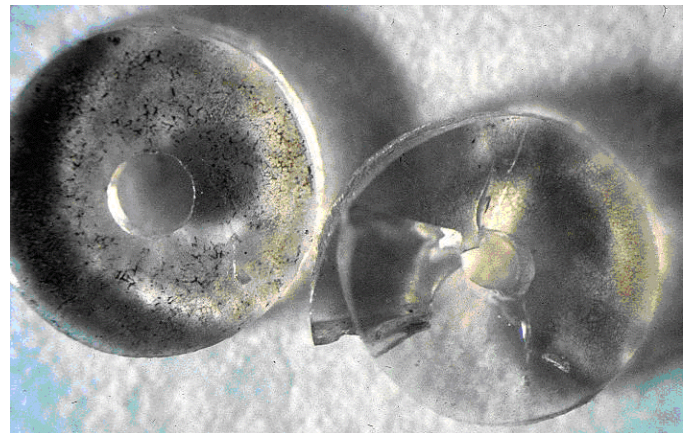


Figure 5. A gelatin/cellulose nitrate sequin after 10 minutes in water, starting to dissolve (left: not immersed; right: after immersion). In extreme cases, the thin cellulose nitrate surface layer can separate from the gelatin base. Separation can also be caused by exposure to high relative humidity, which can plasticize the gelatin, weaken the bond between gelatin and cellulose nitrate, and lead to delamination of the unswollen cellulose nitrate layer from the swelled and softened gelatin layer. This figure shows the thinness of the cellulose nitrate layer compared to the gelatin base (see also **Figure 8** in the Appendix). The sequins are about 5 mm in diameter.

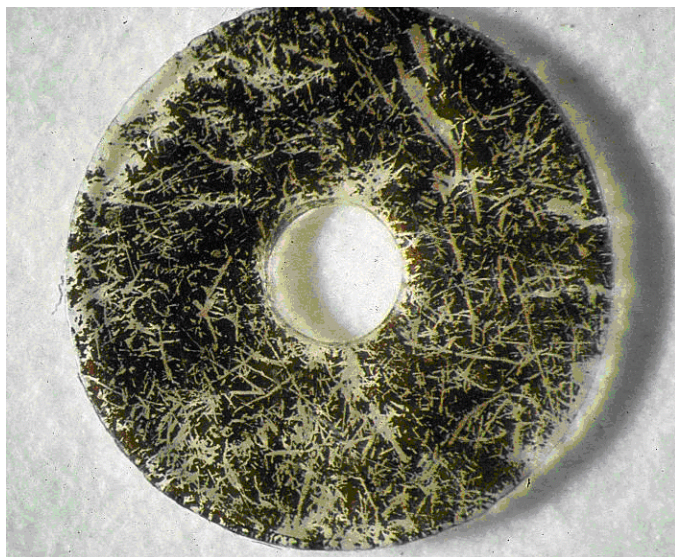


Figure 6. Sequin composed of a gelatin core and cellulose nitrate laminate, after immersion in water. The gelatin core has swelled, causing the outer layers of cellulose nitrate to develop an “alligator” pattern in the surface. The colour of some sequins is due to a thin coloured cellulose nitrate layer on thick gelatin base. The thin layer of cellulose nitrate is easily abraded, resulting in loss of colour, and a faded appearance. In this case, the fading is more by mechanical loss of coating than by fading of the colour in the coating. The sequin is about 5 mm in diameter.

Conclusions

If an artifact is from the late 19th or early 20th century and ornamented with sequins, it should be treated with caution. Sequins from this period were made of metal, gelatin, cellulose nitrate, varnishes or various plastics, sometimes in combination. Heat, water and solvents, including dry-cleaning solvents, may therefore cause severe damage, if not completely destroy them. If doubt exists, the following procedures are recommended:

- a) Identify the materials. If instrumental methods are not available, then use the spot tests outlined in the **Appendix**.
- b) If gelatin is found:
 - Do not use water.
 - Do not steam or humidify.
 - Avoid heat (*e.g.*, hot lights in display cases).
 - Ensure that storage areas are clean and dry and maintain a stable environment between 30 and 50% RH and below 18°C. Avoiding fluctuating temperature and relative humidity is important since gelatin is one of the most moisture-sensitive materials found on textile artifacts.
 - Provide protective coverings where necessary. Simple covers can be made from Tyvek or GORE-TEX to provide protection for costumes from dust and dirt.
- c) If cellulose nitrate is found:
 - Rates of degradation increase with material thickness.
 - Corrosion to adjacent metal component parts can occur.
 - Artifacts with cellulose nitrate components should be regularly monitored for degradation. Cresol red



Figure 7. After immersion in water, a sequin of cellulose nitrate with a gelatin core has lost its iridescence (left: before immersion; right: after immersion). High ambient humidity, or treatments involving steaming or water washing swells the gelatin core which then warps after drying. This swelling distorts the cellulose nitrate layer, and therefore disrupts the optical interference of reflections from it, altering and even destroying the iridescence of the sequin. The sequins are about 5 mm in diameter.

indicator strips as described by Fenn¹³ can be used to determine if objects are actively degrading.

- When necessary, segregate and ventilate.
- Further information can be found in:
 - CCI Note 15/3 Display and Storage of Museum Objects Containing Cellulose Nitrate⁸ and
 - CCI Note 15/1 Care of Objects Made from Rubber and Plastic.¹⁴

Sequins produced in the 20th century and later are problematic, due to the various plastic formulations and coatings used in their manufacture. Dry-cleaning of modern apparel such as wedding dresses and evening clothes is often recommended in the public domain, and this is a serious concern as solvents may dissolve some of the lamina and cause bleeding of colours, resulting in permanent staining of a garment. Sequins may be attached to a garment with adhesives that soften or dissolve with certain solvents. Each trim component on a garment should be tested with the cleaning solution in order to determine its suitability.¹⁵ Costumes decorated with trimmings should be treated with caution and the decorations carefully examined in order to determine their composition. Simple spot tests exist that can help determine materials' composition and aid in treatment options. Conservators beware!

Acknowledgements

The authors wish to thank Sheilah Mackinnon for allowing them to reproduce data from her thesis.

Materials List

All chemicals, reagents, and labware can be obtained from major scientific, chemical and equipment suppliers. Small amounts of chemicals in the range of 10 g to 25 g can be obtained from suppliers such as Chem Service, Inc. and Sigma-Aldrich Canada Ltd. Some test solutions are available as pre-made solutions, or as stock solutions that can be diluted with water, from specialty

suppliers such as Caledon Laboratories Ltd. or LabChem Inc. Material Safety Data Sheets (MSDS) are available from all suppliers.

Suppliers

Caledon Laboratories Ltd.,
40 Armstrong Avenue, Georgetown (Halton Hills), Ontario,
Canada, L7G 4R9
Phone: (905) 877-0101
<http://www.caledonlabs.com>

Chem Service, Inc.,
PO Box 599, West Chester, PA, USA, 19381-0599
Phone: (610) 692-3026
Fax: (610) 692-8729
<http://web1.chemservice.com/csiwebsite.nsf/index?openpage>

CR Scientific,
Pennsylvania, USA
Phone: 1-877-260-6039
<http://www.crscientific.com>

Fisher Scientific Company,
112 Colonnade Road, Ottawa, Ontario, Canada, K2E 7L6
Phone: 1-800-234-7437
<http://www.fisherscientific.ca>

LabChem Inc.,
200 William Pitt Way, Pittsburgh, PA, USA, 15238
Phone: (412) 826-5230
<http://www.labchem.net>

Sigma-Aldrich Canada Ltd.,
2149 Winston Park Dr., Oakville, Ontario, Canada, L6H 6J8
Phone: 1-800-565-1400
Fax: 1-800-265-3858
<http://www.sigmaaldrich.com/canada-english.html>

Ted Pella, Inc.,
P.O. Box 492477, Redding, CA, USA, 96049-2477
Phone: (530) 243-2200, 1-800-237-3526
Fax: (530) 243-3761
<http://www.tedpella.com>

Ward's Natural Science,
P.O. Box 92912, Rochester, NY, USA, 14692-9012
Phone: 1-800-962-2660
<http://www.wardsci.com>

Materials

Bioplastic
Ward's Natural Science Liquid Bioplastic™ and catalyst, Item
No. 35 V 1720, 1 quart, US\$35.50

Chromotropic acid disodium salt dihydrate
(1,8-dihydroxynaphthalene-3,6-disulfonic acid disodium salt;

4,5-dihydroxynaphthalene-2,7-disulfonic acid disodium salt)
Chem Service, Inc., Cat. No. 3925B, 5g, US\$31.50
Sigma-Aldrich Canada Ltd., Cat. No. 213276-10G, 10 g,
C\$52.10

Copper (II) sulfate pentahydrate (cupric sulfate pentahydrate)
Chem Service, Inc., Cat. No. I-50, 10 g, US\$18.50

Copper (II) sulfate solution
2% (w/v), Caledon Laboratories Ltd., Cat. No. LC13420-1, 500
mL, C\$17.46
4% (w/v) prepared from copper (II) sulfate pentahydrate,
Sigma-Aldrich Canada Ltd., C2284-25 mL, C\$29.40

Diphenylamine (DPA)
Chem Service, Inc., Cat. No. O-344, 5 g, US\$31.70; 10 g,
US\$39.30
Sigma-Aldrich Canada Ltd., Cat. No. 242586-5G, 5 g, C\$38.30

Diphenylamine, 1% in sulfuric acid
Caledon Laboratories Ltd., Cat. No. LC13650-1, 500 mL,
C\$54.81
Caledon Laboratories Ltd., Cat. No. LC13650-7, 125 mL,
C\$30.64

Iodine, 0.01 N, 0.13% w/v, in aqueous potassium iodide solution
Caledon Laboratories Ltd., Cat No. LC15600-1, 500 mL,
C\$20.02

Solutions with higher concentration (up to 1 N) are available, and
can be used as stock solutions that need to be diluted
appropriately before use as test solutions.

Microscope slides with sandblasted frosted ends
Fisher Scientific Company, Cat. No. 12-550-34, 3 x 1 in. (25 x
75mm, 1mm thick), frosted on one side, one end, 144 for
US\$11.36
Fisherbrand Extra-Thick Microslides: Cat. No. 12-550-11, 3 x 1
in. (25 x 75 mm; 1.2 mm thick); sides frosted, one end, 144 for
US\$93.21

White Porcelain Spot Plates
12 cavity: Fisher Scientific Company, Cat. No. S337241,
US\$7.60 each
6 cavity: CR Scientific, Cat. No. #1876, US\$2.29 ea
<http://www.crscientific.com/spotplates.html>
(unglazed back can be used as a streak plate to collect small
samples of plastic).

Sodium hydroxide, 10% w/v aqueous solution
Caledon Laboratories Ltd., Cat. No. LC2470-1, 500 mL,
C\$12.70
Solutions with higher concentration (up to 50%) are available,
and can be used as stock solutions that need to be diluted
appropriately before use as test solutions.

Spurr Embedding Kit
Ted Pella, Inc., Product No. 18300, 950g, US\$ 91.50
Sulfuric acid, reagent, concentrated, specific gravity 1.84, 96%

(18 M, 36 N)

Caledon Laboratories Ltd., Cat. No. 8825-1-05, 500 mL, C\$20.87

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APPENDIX: Spot Tests

These spot tests have been known in various versions for decades. The test reagents and procedures presented here are for spot tests that can be done most conveniently by applying a few drops of test reagents to samples of plastic on white porcelain spot test plates or on microscope slides. The white porcelain background makes the colours which develop during diagnostic tests more visible.

The samples consist of material removed from sequins in the form of powders, grains or pieces, obtained by clipping, cutting, filing, scraping, slicing or streaking, having a volume about the size of a pin-head. A separate sample is used for each test. When collecting a sample by streaking, the frosted end of a microscope slide can be used. It is important that the frosted end is sandblasted, not etched, inked, or painted. Only sandblasted slides have the appropriate roughness to remove a sample suitable for spot tests from plastics.

It is useful to perform the spot tests while observing the samples with a stereomicroscope. This often reveals the location of the target compound on the sequin (e.g., the surface layer, or the core of a laminated structure). For all tests, a blank test without any sample present should be performed to ensure that no interfering colour reactions occur in the absence of sample.

Also, a control test, with a sample that is known to give a positive test, should be performed to ensure that reagents and procedures will produce a positive test result in the presence of the target material. Substances which can be used as control materials for each test are listed in the appropriate section below.

The diphenylamine test for cellulose nitrate, chromotropic acid test for formaldehyde, and iodine test for starch have been proven to be reliable as spot tests and the results are consistent with those using FTIR.

The copper (II) sulfate spot test (modified Biuret test) on samples in this laboratory has given no false positive results (i.e., no sample that was not protein gave a positive test), but about 10% of known protein samples gave negative tests. In most cases, these have been from samples with small amounts of protein mixed with another material, such as gelatin emulsion from photographs, or an adhesive.

Proper laboratory chemical handling procedures should be used for all reagents. Protective clothing and eye protection should be worn, and work should be done in a well-ventilated area. Most reagents are alkaline or acidic and quite corrosive.

Test for cellulose nitrate using diphenylamine (DPA)^{16,17}

Reagent

- 0.8% (w/v) diphenylamine in 80% (v/v) sulfuric acid: add 100 mL of commercially available pre-made stock solution of 1% (w/v) diphenylamine in sulfuric acid to 25 mL water with constant stirring.

Alternate Reagent

- 0.5% (w/v) diphenylamine in 90% (v/v) sulfuric acid: add 90 mL concentrated sulfuric acid (96% reagent) to 10 mL water with constant stirring, then add this, in successive small portions, to 0.5 g diphenylamine.

Control Sample

Celluloid, collodion.

Procedure

1. Place a pinhead-sized sample on a glass microscope slide or spot plate, or rub the ground glass (sandblasted) portion of another slide against the object to create a streak of material on the slide.
2. Add one drop of the reagent to the sample on the spot plate or slide.

Positive Test Result

If cellulose nitrate is present, a blue-violet stain appears on the sample (**Figure 8**), which diffuses into the test drop. Other colours are negative results.

Notes

Addition of sulfuric acid to water generates much heat and can cause spattering of the solution. Always add acid to water. Always add acid slowly while stirring. Wash all materials used during the test with water and discard carefully. The reagent is corrosive.

Within limits, the concentration of the reagent is not critical. The same colour reaction is obtained with reagents having concentrations of DPA in the range 0.2 - 5% (w/v) in sulfuric acid with concentrations in the range 70 - 90% (v/v).¹⁸ The 0.8% DPA reagent has been chosen because it is very easy to prepare from a commercially available stock solution (which cannot be used directly because the sulfuric acid concentration is too high).

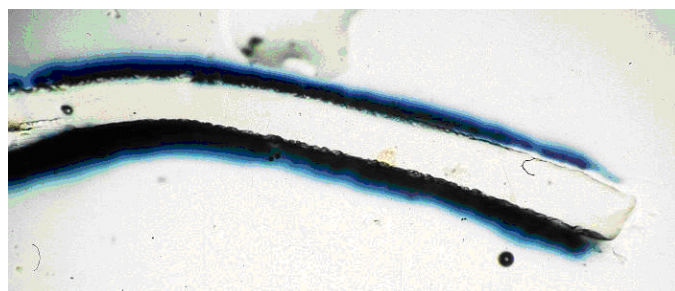


Figure 8. Cross-section of an iridescent sequin with a thin cellulose nitrate layer on a thick gelatin base. This sample was prepared by shaving a thin slice from the edge of the sequin, placing the slice on a microscope slide so that the cross-section was visible, covering with a coverslip, then placing a drop of 0.5% diphenylamine dissolved in 90% sulfuric acid at the edge of the coverslip and allowing the DPA solution to flow across the cross-section. Within seconds, the cellulose nitrate layers on the surface were stained blue. The thickness of the blue layer appears greater in the image than the true cellulose nitrate layer thickness because the cellulose nitrate was hydrolysed by the acid in the DPA solution and the colour started to bleed into the DPA solution. The cellulose nitrate layer is about 1/5 to 1/10 the thickness of the gelatin base (the clear centre layer, unstained by DPA). The cross-section is about 0.5 mm thick.

Test for protein using copper (II) sulfate (modified Biuret test)¹⁸

Reagents

- 2% (w/v) aqueous copper (II) sulfate (CuSO₄) solution: add 0.5 g CuSO₄ to 25 mL distilled water, or use a commercially available pre-made solution.
- 5% (w/v) (1.2 M) aqueous sodium hydroxide (NaOH) solution: add 1.25 g NaOH pellets to 25 mL distilled water, or use commercially available pre-made solution.

Control Sample

Gelatin, casein, collagen, animal glue, bone glue, fish glue, rabbit skin glue, isinglass.

Procedure

1. Place a small piece or a few grains of sample on a spot-test plate.
2. Add a drop of copper (II) sulfate solution to the sample.
3. Wait a few minutes. The sample should absorb some of the solution and may turn slightly bluish.
4. Optional: Remove excess copper (II) sulfate solution by wicking it onto a piece of blotting paper.
5. Add a drop of the sodium hydroxide solution to the sample.

Positive Test Result

If protein is present, a violet colour will form on surface of the sample, or in the solution if the sample dissolves (**Figure 9**).

Notes

A blank test will show a blue solution and perhaps a sky blue gelatinous precipitate of copper hydroxide but no violet reaction product. A test on a control sample will show violet colouration on the solid sample and/or a violet-coloured solution surrounding the sample, which will be visible with good colour contrast against the sky blue background of copper hydroxide precipitate.

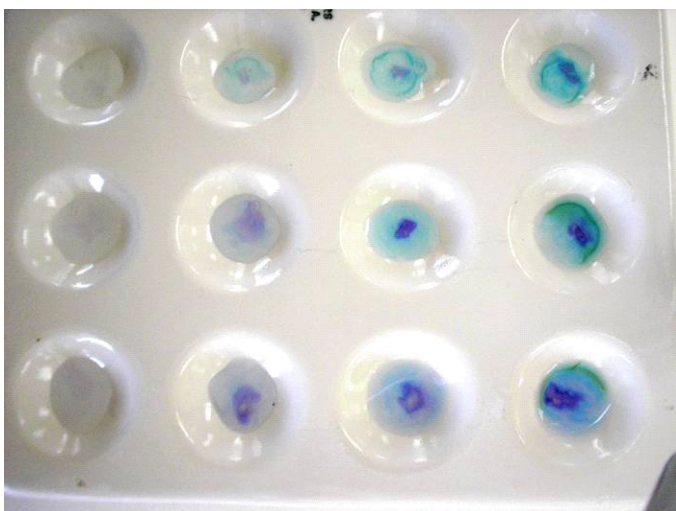


Figure 9. Results of the test for protein using various concentrations of copper sulfate and sodium hydroxide. Four wells in each row have the same concentration of sodium hydroxide: top 20%, middle 10%, bottom 1%. Three wells in each column have the same concentration of copper sulfate: first column 0.5%, second 1.0%, third 2%, fourth 5%. Best results are with 2% copper sulfate (column three) and 1 to 10% sodium hydroxide (the middle and bottom rows).

If the sample does not dissolve in the copper sulfate solution, then excess copper solution can be removed by blotting. If the sample is soluble, then blotting will remove dissolved sample which may reduce the sensitivity of the test; in this case, the blotting step should be avoided.

Test for formaldehyde using the disodium salt of chromotropic acid (CTA)¹⁹

Reagents

- 10% (v/v) sulfuric acid in water: add 10 mL concentrated sulfuric acid (96% reagent) to 90 mL water in a large vessel with constant stirring.
- 5% (w/v) chromotropic acid disodium salt in water: add 5 g chromotropic acid disodium salt to 100 mL water.
- Concentrated sulfuric acid (96% reagent).

Control Samples

Formaldehyde, formalin, paraformaldehyde, Bakelite, some Gatorfoam or Gatorboard skins.

Procedure

1. Place a pinhead-sized sample on a spot plate.
2. Add 2 drops of 10% sulfuric acid.
3. Wait a few minutes. Some sample should dissolve or disperse at this stage.
4. Add 1 drop of chromotropic acid reagent and wait a minute.
5. Add 5 drops of concentrated sulfuric acid by slowly running a stream from a pipette down the sloping wall of the spot plate well so that the sulfuric acid sinks below the water layer to form a separate layer below the water.

Alternate Procedure

1. Place a pinhead-sized sample in a 7.5 cm (3 in.) test tube.
2. Add 1 mL of 10% sulfuric acid.
3. Heat but do not boil, then cool to room temperature. Some sample should dissolve or disperse at this stage.
4. Add 5 drops of chromotropic acid reagent.
5. Tilt test tube away at an angle of 45 degrees.
6. Add 1 mL of concentrated sulfuric acid by slowly running a stream from a pipette down the sloping wall of the test tube so that the sulfuric acid sinks below the water layer to form a separate layer below the water.

Positive Test Result

If formaldehyde is present, a blue-violet ring develops at the acid-water interface within a few minutes. After 5 minutes, gently swirl the spot plate or test tube to slightly mix the solutions at the interface. Observe the colour. After another 5 minutes, gently swirl the test tube until the interface is lost and the solution is uniformly mixed. The time taken for the blue colour to develop indicates how much formaldehyde is present and how strongly it is bound into the sample. Other colours indicate negative results.

Notes

Adding concentrated sulfuric acid to water can generate sufficient heat to cause the solution to boil and spatter. Ensure that the test tube is tilted away from people.

Test for starch using iodine/potassium iodide²⁰

Reagent

- 0.01 N (0.13% w/v) iodine in potassium iodide: dissolve 0.13 g iodine in a solution of 2.6 g potassium iodide in 5 mL water then dilute to make 100 mL, or use a commercially available solution.

Control Samples

Vegetable starches, flours.

Procedure

1. Place pinhead-sized sample in a spot test plate.
2. Add one drop of water. The sample may dissolve.
3. Add one drop of iodine/potassium iodide solution.

Positive Test Result

If starch is present, a blue-violet colour is produced on the solid sample or in the solution. If the test solution is too concentrated, the stained sample will appear black.

Notes

Test solutions have been suggested for the detection of starch that have a wide variation in concentration of iodine and potassium iodide (with iodine concentration ranging from 0.114% (0.01 N) to 5.88% (0.468 N) and potassium iodide concentration ranging from 0.8% (0.048 N) to 11.8% (0.7 N). Iodine solutions of 0.01 N (0.127%) are yellow to amber in colour and stain starch to a blue colour. Iodine solutions of higher concentration have a browner colour which interferes with the observation of the stain colour produced on the starch, which also tends to be brown or black, rather than the blue which is produced at the recommended iodine concentration.

Iodine solutions, already prepared, are available in various types, including: aqueous solutions of iodine called Strong Iodine Solution USP, compound iodine solution, or Lugol's solution, containing 5% iodine and 10% potassium iodide; Iodine Topical Solution (USP) containing 2% iodine and 2.4% potassium iodide; USP Strong Iodine Solution and Aqueous Iodine Solution BP containing 130 g/L (13%) of free and combined iodine; and Tincture of Iodine USP containing 2% iodine and 2.4% sodium iodide diluted in 50% ethanol. These solutions are all brown to dark brown, and stain starch brown or black, which obscures the characteristic blue colour. To obtain the blue colour reaction, the solutions must be diluted until they are yellow- to amber-coloured.