FOURIER TRANSFORM INFRARED SPECTRAL ANALYSIS OF NATURAL RESINS USED IN FURNITURE FINISHES

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This paper establishes an infrared spectral identification key for five natural resins (shellac, sandarac, mastic, copal and rosin) based on their absorbance band positions. These resins may be identified when used in mixtures, such as furniture finishes, at concentrations as low as 1%. Computer methods of deconvolution and spectral subtraction are used to augment this process. The method was validated with reference resins, then applied to finishes sampled from 18th century furniture.

INTRODUCTION

Infrared spectroscopy is a valuable method for the detection and identification of organic coating materials. Infrared spectra provide information on chemical functional groups of the sample which allow for identification of specific compounds and for general characterization of the material. Pure synthetic resins produce vibrational bands which are readily identifiable. Complex natural materials produce overlapping spectral bands resulting in a blended yet representative plot which may be used reliably to identify a material. Mixtures of natural materials, as are often found in furniture finishes, can stretch infrared spectroscopy to its limits as an analytical method. This paper outlines the capabilities and limitations of infrared spectroscopy for the identification of natural resins used in historic furniture finishes.

Fourier Transform Infrared (FTIR) spectroscopy requires minimal sample amounts to provide reliable characterization of the major components. With the aid of computer techniques such as spectral subtraction (Koenig, 1975) and deconvolution (Kaupinnen et.al., 1981), the intrinsic information from the spectra can be used to identify even minor components of a mixture. FTIR has significant advantages over less accurate dispersive spectrophotometers (Low and Baer, 1977). The Fourier Transform instrument produces a high resolution spectrum in which the total energy beam is examined at one instant with the wavelengths initially separated by distance then recombined, and transformed, by a mathematical technique, into wavelength separation. The rapid scan time allows for the integration of many scans, yielding very high signal-to-noise ratios and thus low detection limits, i.e. nanogram sample sizes (Cournoyer, et.al., 1977). Photometric accuracy is achieved in the FTIR instrument with internal laser calibration.

The usual procedure for identifying an unknown by its infrared spectrum is to visually match band positions and their relative intensities with the spectrum of a known material. This can easily be done for a pure sample using a computer search program to rapidly compare an unknown to a library of several thousand known pure compounds. However, because unknown samples are often impure, erroneous spectral matches can result. A mixture of materials contains the vibrational bands for each component, therefore a method for separating the spectra must be used to identify the individual compounds. This can be done either by solvent extraction (Mills, 1972), or spectrally, first by identifying the general class of materials, then by matching specific band positions of the unknown with similar reference bands (Smith, 1979).

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This investigation used the method of spectral identification of compounds in a mixture by band position to study natural resins typically used in historic furniture finishes. Brachert (1978) gave detailed recipes for historical furniture finishes. The five most often used resins (shellac, sandarac, mastic, copal and rosin) were selected from Brachert's compilation of recipes (see TABLE 2 for frequency of use). A variety of infrared analyses was done on these five resins.

First, sets of known resins were used to check the band position variability due to sample preparation, sample size, resin supplier, varnish preparation (solvent extraction and filtration) and resin aging (oxidation and deterioration). Specific, stable bands for each resin were then selected for an identification key. Secondly, mixtures of these five resins were prepared in formulations listed by Brachert and in test mixtures with individual concentrations as low as 1%. In these known mixtures, each component could be identified by using the infrared band position identification key along with computer methods of deconvolution and spectral subtraction. Finally, this resin identification method was applied to the analysis of finish samples from 18th century furniture to characterize their resin content.

EXPERIMENTAL

Instrumentation

Spectral analyses were made on a Digilab l5-E Fourier Transform Infrared (FTIR) spectrometer equipped with a Motorola 3200 computer. The spectrometer was purged with dry clean, CO_2 free air from a Balston 75-60 filtration unit. A Mercury-Cadmium-Telluride (MCT) detector, cryogenically cooled, was used to examine the region from 4000-500 cm^{-1} (2.5-20 um). Each spectrum consisted of two hundred co-added scans at a resolution of 4 cm^{-1}.

Samples

Reference resins were obtained from A.F. Suter & Co and the conservation laboratories at the J. Paul Getty Museum. Homogeneous samples were obtained by crushing and thoroughly mixing a 2-3 gram portion, which was then stored in glass vials prior to analysis.

Naturally aged resin samples were obtained from the Harvard Art Museums' collection of Gettens and Stout materials. The resins were films on glass plates. The dates on these plates were from 1933-1937. It is assumed that the plates, which were labeled as controls, were stored in a drawer during the last 5 decades. A 50 mm^2 section of each resin was removed from the plate with a scalpel and placed in a clean glass vial with a teflon-lined cap.

The unknown sample set was composed of finishes on furniture pieces from the J. Paul Getty Museum. Each piece was of 18th century European manufacture and was typically of veneer on oak. Samples were arbitrarily chosen from obscure areas on the furniture, near or under mounts, in the rear or on the leg. A scalpel was used to scrape barely visible amounts of sample (<100 microgram) from clean, surface layers of the finish. This minimized the chance of contamination from dirt and wood particles which can obscure the resin spectra. In a multilayered finish, this sampling method selectively removes the more recent coatings. In these cases, more than one sample may be scraped from different depths.

Sample Preparation

Samples were prepared as KBr pellets in either the standard 13 mm size or 1.5 mm micropellet size. Approximately 1 mg of a crushed bulk resin sample was used for the 13 mm KBr pellets. These standard sized pellets were used in the tests on variability due to sample preparation, resin supplier and resin refinement. Additionally, the 13 mm pellets were used for the resin mixtures; each mixture had a total weight of 1 mg with the individual components weighing from 0.01 mg to 1.20 mg. The 1.5 mm pellets were prepared from approximately 30 micrograms of sample in a glove bag under a dry nitrogen purge and analyzed using a Harrick Scientific 4x beam condenser. Micropellets were prepared for the natural and artificially aged samples, the solvent dependency study and the furniture finish samples.

For the solvent dependency study, 5% solutions were prepared with sandarac, mastic and rosin in ethanol, turpentine and mineral spirit solvents and with shellac and copal in ethanol. After 48 hours, the solutions were filtered through one layer of cheesecloth, then 20-30 ml of each solution was cast onto clean thin sheets of aluminum using 6”x6” teflon molds (Hansen and Taketomo, 1988). The films were air- dried for approximately one week, then analyzed.

For the accelerated deterioration studies, a thin layer of four resins in ethanol (mastic-20%, rosin-20%, sandarac-33% and shellac-33%) were painted with an ethanol-cleaned paint brush onto clean, polished surface aluminum plates (2”x2”x1/16”) resulting in film thicknesses of 7 to 49 urn with the mastic films being the thinnest. These samples were allowed to dry overnight in a fume hood, then placed in a Weather-Ometer with controlled light, heat and humidity conditions. The samples were irradiated with a xenon lamp set at 0.30 W/m^2 (340 nm) with inner and outer borosilicate filters simulating outdoor sunlight (Brennan, 1987). The black panel temperature was 53˚C and the relative humidity was 50%. The total irradiance for the samples ranged from 0 to 108 kJ. The controls were kept in a dark for an equivalent time period.

Computer Methods

A Fourier Transform spectrometer requires a computer for an online conversion of the interferogram to a spectrum. This computer may subsequently be used for - spectral processing in a variety of ways, such as spectral storage, signal averaging, library searching, scale expansion, absorbance subtraction and addition, deconvolution and replotting of spectra. These spectral manipulations may be used to elucidate information and quantify spectral variations.

For each sample, the computer printed out band position values for peak maxima. In regions where peaks are overlapped, a deconvolution program was used to elucidate the individual features. The program operates on 2 or more overlapping bands to reduce the line width of the individual components and therefore improve the resolution of each line.

Spectral subtraction methods were used to separate components of a mixture. Intense bands were used to identify major components whose pure spectra were then multiplied by a scaling factor and subtracted out. After spectrally removing the bands corresponding to the major component, a scale expansion was used to enhance the remaining bands and identify the minor components. Following the same general scheme, computer addition was used to simulate spectra of mixtures, such as varnish recipes, from single component spectra which may then be stored in a search library.

RESULTS AND DISCUSSION

General Classification

Infrared spectroscopy produces a characteristic spectrum for any compound dependent on the functional groups that are present. For identification purposes, natural products may be grouped into the following classes that contain similar IR active functional groups: resins (tree), resins (insect), oils, gums and waxes. TABLE 1 shows these together with specific peak positions that occur within each material class. Figure 1 gives exemplary spectra of each category. The spectra of each of these classes are readily identifiable by their characteristic functional group vibrations. Typically OR stretches occur as broad bands near 3400 cm^{-1}, methyl and methylene stretches occur in the region of 2800-3200 cm^{-1} and carbonyl bands are seen at 1690-1750 cm^{-1}. The bands in the fingerprint region (below 1800 cm^{-1}) are due to C-R, C-O and C-C wags and bends. The exact location (wavenumber) for each band depends on its molecular environment. More information on vibrational group assignments for these natural materials may be found elsewhere (Bellamy, 1968 and Omecinsky and Carriveau, 1986).

Resin Identification Key

TABLE 2 gives an IR absorbance band identification key for five commonly used natural resins in furniture finishes along with their family name and major components. Additional information is included on the use of these resins in historic finishes. Figure 2 shows an infrared absorbance spectrum for each resin examined. Once a natural material has been classified as a resin using the information in TABLE 1, then the type of resin may be distinguished using the additional information provided in TABLE 2.

This key provides a simplified structure for the easy identification of the presence of a resin in a spectrum by a list of its absorbance band positions. The strongest bands in the resin spectra are generally the carbonyl and hydrocarbon stretching frequencies. These bands, listed at the top of the key can provide a distinction for four of the resins. Sandarac and copal are chemically and spectrally very similar and may be distinguished only by some smaller bands in the fingerprint region of the spectra. The presence of at least half of these peaks in an unknown sample spectrum is a strong indication that a particular resin is present. An identification made on the basis of band positions should be confirmed by visual comparison to a reference spectra.

Reproducibility

The premise of the identification of resins by band position alone is based on the assumption that the frequency of the absorption bands is reproducible. Several factors were examined individually to assess band position variability. They were 1) instrument stability; 2) sample preparation methods; 3) source or degree of refinement of the resin; 4) sample size; 5) preparation of a varnish; and 6) the extent of deterioration or oxidation of the resin. A summary of standard deviations for the band position reproducibility is presented in TABLE 3. Deviations in TABLE 3 are the average for the given number of samples for all the wavenumbers listed for that resin in TABLE 2. The maximum band deviation, omitting the degraded resins, was 1.2 cm^{-1}. From this value a precision of 2.4 cm^{-1} (i.e. 2 sigma) was placed on the bands listed in the identification key. Inclusion of the variations seen in the photochemical degradation samples would give a precision of 4.2 cm^{-1}. In either case, this is a significant improvement to the variations of 20-50 cm^{-1} that were found by Beck et. al. (1965) in their analysis of amber using a dispersive infrared spectrophotometer.

TABLE 1. Characteristic absorption bands for several classes of natural materials.

NATURAL MATERIAL	RESINS SOURCE: TREE	RESINS SOURCE: INSECT	OILS	WAXES	GUMS
CHEMICAL CLASSIFICATION	TERPENOIDS	POLYHYDROXY ACIDS	TRIGLYCERIDES	LONG CHAIN HYDROCARBONS	POLYSACCHARIDES
REPRESENTATIVE SAMPLE SHOWN IN FIGURE 1	Sandarac	Shellac	Linseed Oil	Beeswax	Gum Arabic
STRONG BANDS (in order of size)	2930-2958	2920-2934	2926-2928	2918-2920	1068-1078
	1695-1715	1716-1722	1740-1747	2849-2851	
	2865-2875	1730-1738	2855-2857	2955-2957	
MEDIUM BANDS (variable size)	1448-1467	1466	1462-1464	1472-1475	2926-2936
	1382-1387	1414-1416	1238-1244	1464-1466	1616-1645
	1178-1184	1377-1381	1159-1167	729-731	1423-1440
	1078-1092	1251-1254	1097-1099	719-721	1385
	1028-1038	1161-1176	721-727	(1736-1742)*	1220-1240
	887-897	1112-1113		(1171-1184)*	
		945			
		928-932			

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*present only in the esterified waxes, such as beeswax

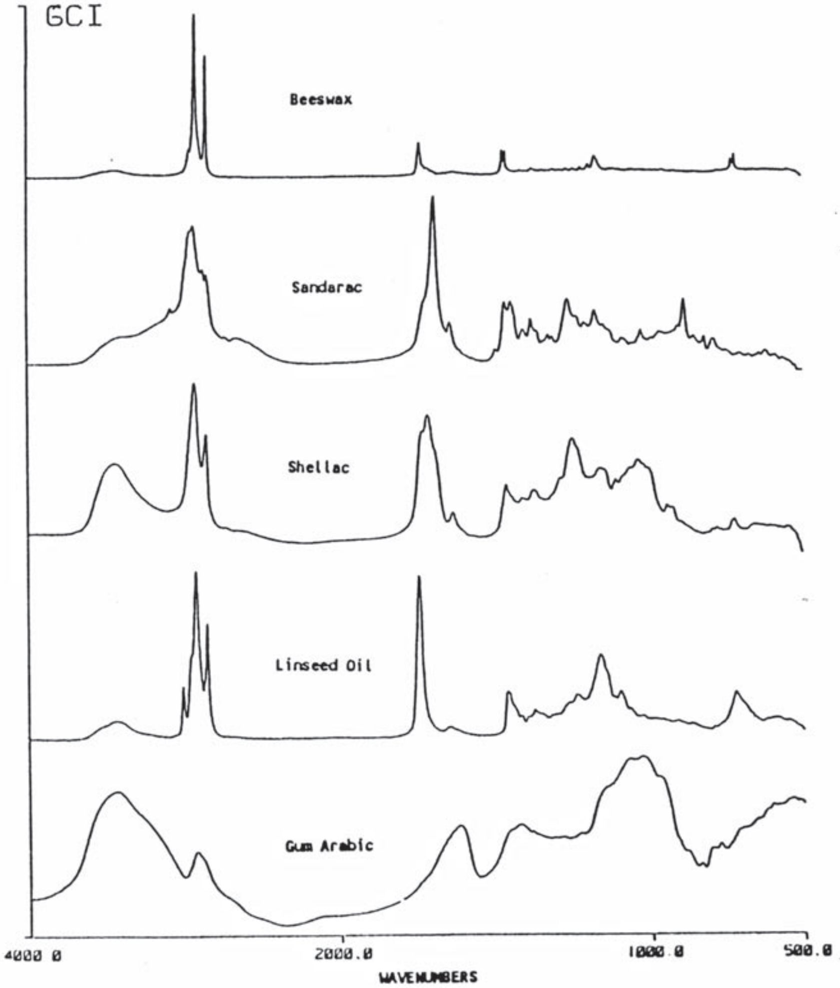

Figure 1. Exemplary spectra for materials listed in TABLE 1.

TABLE 2. IDENTIFICATION KEY FOR INFRARED SPECTRA OF NATURAL RESINS
Absorption bands represented as wavenumbers ($\pm$ 2.4cm^{-1})

	SOURCE = TREE				SOURCE=INSECT
Carbonyl bands	1690-1700			1700-1715	1715-1722 1730-1738
C-H stretches	2936 2870 2646-2654	2933 2873 2849		2949 2874	2930 2857
Vinyl stretches and bends	1697	3079 1694 1643		1650	1636
Characteristic fingerprint region bands (in addition to bands in TABLE 1.)	1612 1496 1365 1275 1239 1151 1130 1107 980 910 823 707 652	1466 1449 1329 1315 1259-1263		1458-1460 1245 1161 1115 1046 1008 837 580	1466 1412 1377 1291 1250 1165-1176 1112 1064 1041 945 930 722
		1497 1236 1213 1153 972 909 856 823 789-792	1228 1149 889 850 795		
RESIN	ROSIN	SANDARAC	COPAL	MASTIC	SHELLAC
FAMILY^{A,B}	Pinacae	Cupressaceae	Araucariaceae	Anacardiaceae	secreted by Laccifer lacca
COMPONENTS^{A,B}	Acids	Sandaracopimaric Acid Communic Acids	Acid Communic Acids	of triterpenes	Shelloic Acids
USE IN BRACHERT'S RECIPES					
--FREQUENCY (%)	22	55	24	44	29
--CONCENTRATION RANGE (%)	4-100	6-100	7-100	6-100	10-100

[A] Mills, J., 1977.

[B] Mattiello, J., 1941.

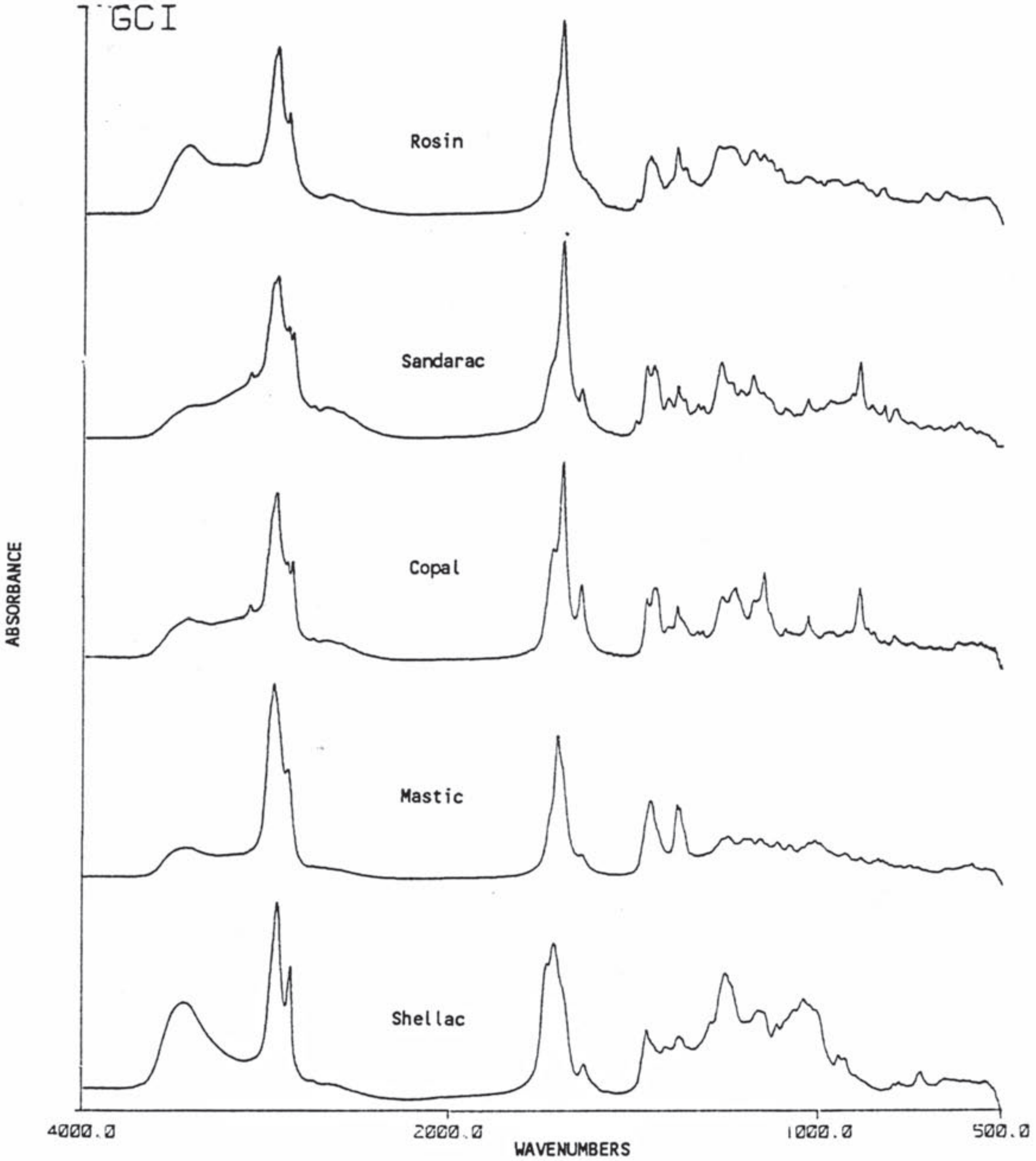

Figure 2. Sample spectra for resins listed in TABLE 2.

TABLE 3. BAND POSITION PRECISION FOR SEVERAL PARAMETERS

PARAMETER	RESIN/ DESCRIPTION	# ANALYSES	AVERAGE STANDARD DEVIATION FOR THE BAND POSITIONS IN TABLE 2 (± CM^{-1})
Instrument stability	Polystyrene	7	0.1
Batch reproducibility	Mastic	5	1.1
Sample size	Sandarac 1.8mg to 8 ug	5	0.3
Supplier variability	Mastic from different suppliers	8	1.4
Supplier and degree of resin refinement	Shellac from different suppliers and with different degrees of refinement	14	1.2
Solvation	Sandarac, mastic and rosin cast from ethanol, turpentine and mineral spirits; 2 types of shellac and copal cast from ethanol	13	0.8
Photochemical degradation from xenon lamp exposure	Shellac, sandarac mastic and rosin (0 to 108 kJ)	20	2.1

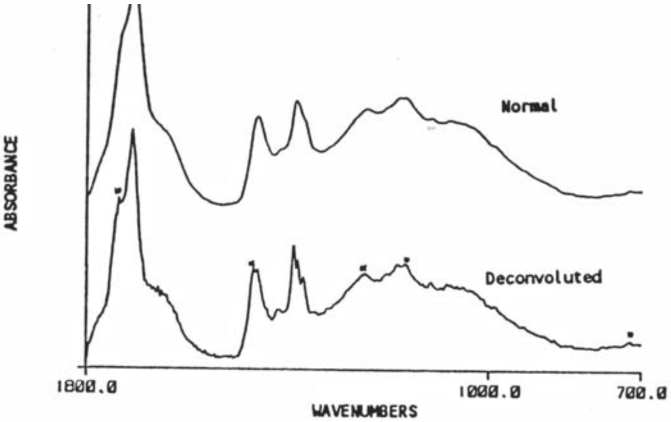

Figure 3. Deconvolution spectra of a sample of 5% oil in mastic.

* Bands used to identify oil

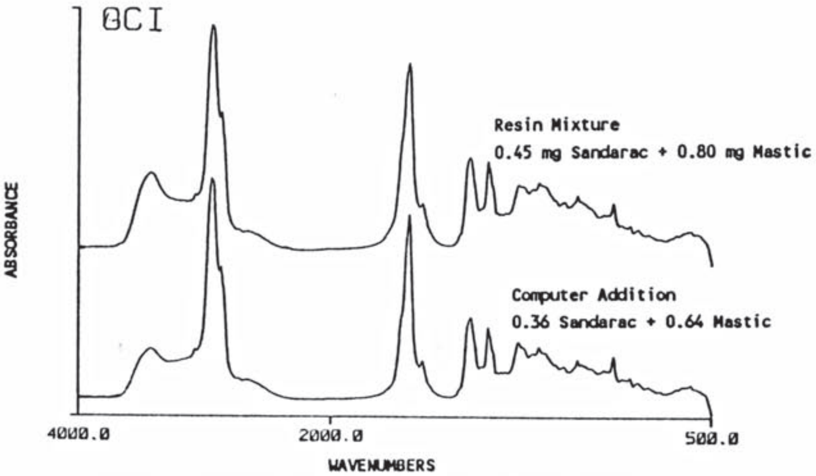

Figure 4. Spectrum produced by computer addition of pure sandarac and mastic spectra compared to a spectrum from a resin mixture of the same proportion

Instrument stability was determined using the reference polystyrene film which had a precision of 0.1 cm^{-1} over a period of three years. Spectra were generated from a standard film using direct transmission which required no sample preparation. Reproducibility in sample preparation was then checked by preparing several pellets from the same batch of mastic resin which gave variations of 1.1 cm^{-1}. Additionally, variations in wavenumber due to sample size were examined. Sandarac bands, using sample sizes ranging from 8 micrograms to 1.8 mg, were found to vary only 0.3 cm^{-1}. This higher precision may be attributed to the sharper bands in the sandarac spectra or to the possibility that the sandarac bulk sample was more homogeneous.

Differences due to the resin's source or supplier were checked by analyzing 8 mastic samples obtained from different suppliers. Additionally, 14 samples of shellac were analyzed, not only from different suppliers, but also with varying degrees of processing or refinement ranging from seedlac to dewaxed, decolored shellac. The band position reproducibility was 1.2 cm^{-1} for both sets of samples. While the frequencies are relatively stable between the resins from various sources, there are small differences that occur in band intensity and band shape which may be used to further identify genera and source of the resin (Gianno, et.al., 1987).

Varnish preparation involves dissolution of the resins in a solvent, then filtration or sedimentation to remove insoluble fractions. Cast films of the resins prepared from different solvents gave an average variability of 0.8 cm^{-1}. While some residual solvent was seen in the spectra of the films cast from turpentine, the spectra of the resins were still easily recognizable by their band positions. In general, spectra of the resins after solvation and filtration contained slightly sharper and better resolved bands than those prepared from the ground resins.

Since finishes found on pieces of furniture in a museum may be of considerable age, the stability of the band positions in the IR spectrum of a resin as it deteriorates was considered. Naturally aged resin films obtained from the Gettens and Stout collection were analyzed, in addition to new resin films for which the photochemical deterioration process was accelerated by exposure to a xenon lamp. Overall, this set of resins showed a deviation of 2.1 cm^{-1}. A comparison of the spectra of the naturally aged samples versus the light exposed samples will be covered in another paper (Derrick and Hansen, 1988). The most important factor for this paper is that the band positions are stable and may still be used to identify each of the resins after they have been degraded.

Resin Mixtures

Up to this point, reproducibility of the band positions has been demonstrated as a function of several variables. The resin identification method was then applied to resins in mixtures. Combinations of two or more resins are given in 80 recipes in Brachert's paper. In this study, resin mixtures were prepared in the proportions specified by 6 recipes. In addition, 44 mixtures of sandarac, copal, mastic, rosin and shellac were prepared in combinations of two to four resins ranging in proportion from 0.8% to 99.2%. The purpose of this section was to determine whether the spectrum of anyone particular resin may be obscured by the presence of another resin. Additionally, the minimum concentration of resin present for detection was to be determined.

After the infrared analysis of each mixture, a list of band positions was generated. The spectrum was then deconvoluted and a new band position list was generated. Only the deconvolution band positions were used in this resin mixture section. It was found that when a resin accounts for at least 50% of the total mixture a deconvolution step was unnecessary, i.e. all the bands required for the identification of the resin were present whether or not the spectrum had been deconvoluted. However, as the concentration of a resin decreased within the mixture, the deconvoluted spectrum consistently provided a higher proportion of the peaks necessary for an accurate identification of the resin. The 5 resins examined in the mixtures could be detected at concentrations as low as 2%. At these low levels, approximately half of the bands for a resin were present. At resin concentrations above 6%, most (>90%) of its bands could be found in the deconvoluted spectra. Figure 3 shows a spectrum of mastic/oil film (Gettens and Stout collection, dated 1934, 5cc oil in 100cc ethanol with 33g mastic) before and after deconvolution. The bands specific for oil, visible only after deconvolution, are marked.

Copal was the one exception to the identification of a resin in a mixture by its band positions. The peaks used for the identification of copal and sandarac overlap to a large extent due to the presence of sandaracopimaric acid and communic acids in both resins (Mills and White, 1977). In sandarac, 3 characteristic bands (908, 1180, and 1498 cm^{-1}) appear consistently, even at small concentrations. However, Y copal, at low concentrations, loses its identifying characteristics. Thus, when a resin was identified as either copal or sandarac, the presence or absence of the 3 sandarac bands was used to specify which it was.

Spectral Subtraction and Addition

Computer assisted spectral subtraction techniques were employed to remove the major peaks in a mixture's spectrum revealing the presence of trace components. For one mixture containing 99.2% sandarac and 0.8% mastic, no mastic peaks were found in the deconvoluted spectra. Spectral subtraction of a known spectrum of pure sandarac was applied to the original spectrum, not on the deconvoluted spectrum, to remove 90% of the intensity of the sandarac-related bands. Afterwards the subtracted, spectra were deconvoluted and examined for the presence of mastic bands. Half of the expected mastic bands were found confirming that mastic was indeed present.

One problem can result in the subtraction spectra. Variations in line widths between the two spectra on which the subtraction is performed can result in negative baseline aberrations. In this case, a peak due to mastic may still not be present in the subtracted spectrum if it was close to a large sandarac peak. Alternatively, the peaks that do appear may be shifted outside the normally expected band range. This problem may be lessened by not subtracting the total amount of the major component. Actually, it only appears necessary to subtract enough of the major component to bring the minor component up to the equivalent of 10-20% composition.

Another use for the computer is to synthesize spectra of resin mixtures via spectral addition methods. This procedure could be used to efficiently create a large reference set of spectra for typical furniture finish mixtures. This method was tested using a computer synthesized mixture of 64% of a mastic spectrum added to 36% of a sandarac spectrum in order to emulate a previously prepared mixture of 0.80 mg mastic and 0.45 mg sandarac. The spectrum for the actual mixture and the computer synthesized mixture corresponded well (Figure 4). A computer search using the Sadtler Library programs was made of the synthesized spectra versus this laboratory's library files consisting of over 300 natural and synthetic materials including all resin mixtures used in this study. The best match for the search was the actual resin mixture which the synthesized spectra were trying to emulate. The corresponding hit index was 0.0716 on a scale where 0.0000 is a perfect match and a typical match of 0.2000 is considered a good fit. In addition, mastic was a component in each of the 10 best hits and sandarac was present in 6 of these. The search programs take the difference of the spectra in question versus the reference spectra and match according to the least differences. Computer synthesized spectra appear to be a viable, time saving alternative to manual preparation of resin mixtures. This may be especially useful in the case of some resins, such as amber, of which only small amounts are easy to obtain.

Unknown samples

Infrared analysis was then done on samples taken from 18th century European furniture, even though it was unlikely that the outer layers sampled from the finishes contained the original resins. Samples from the exterior of 2 tables (72.DA.49, 84.DA.77), a cabinet (77.DA.91), a cupboard (72.DA.39), a commode (55.DA.5) and a desk (67.DA.IO) were found to be shellac, while the finishes on a rolltop desk (72.DA.47), a double desk (70.DA.87) and a music stand (85.DA.216) were found to be mixtures of shellac and cellulose nitrate. According to Mattiello (1941), cellulose nitrate was often added to shellac in the 1930's and 40's to improve its water resistance. An additional sample, however, from the inside of a drawer on the rolltop desk contained only shellac. In a previous sample set (Derrick, Grzywacz and Preusser, 1988), a finish on a European black lacquered cupboard (78.DA.119) was determined to be pigmented sandarac using the identification key. For each sample the major components were identified, then the spectra were deconvoluted to check for minor components.

CONCLUSIONS

Fourier Transform Infrared analysis can identify five natural resins (shellac, sandarac, mastic, copal and rosin) individually and as components in a mixture based on a set of band positions presented in this paper. The precision of the band positions was demonstrated to be within 2.4 cm-l for: 1) the instrument; 2) sample preparation used in this study; 3) variations due to resin supplier or degree of refinement. Changes in band position due to varnish preparation and aging were shown not to diminish the effectiveness of the band identification scheme.

For resin mixtures, with a total sample mass of approximately 1 mg, a component with a concentration of 50% or greater may be identified by the presence of the band positions listed in TABLE 2. Minor components in a resin mixture may be identified by computerized deconvolution of the spectra to resolve any overlapping bands. With deconvolution, a component with a concentration as low as 6% may be readily recognized and components with concentrations as low as 2% may be tentatively recognized. Additionally, the computer may be used to spectrally subtract most of the major component from a mixture, thus, in effect increasing the relative proportion of the minor components. Using computer subtraction followed by deconvolution, components in concentrations as low as 1 % may be identified.

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