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Forensic Science Applications of Diffuse Reflectance Infrared Fourier Transform Spectroscopy (DRIFTS): V. Direct Analysis of Metallic Paints—Screening of Panels

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ABSTRACT: Direct sampling using diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS) is particularly useful for rapidly screening metallic reference panels in some hit-and-run cases. For panels with finishes having high densities of metallic flakes, spectra identical to those obtained using conventional transmittance techniques may be obtained with DRIFTS and relatively minor spectral features may be used to distinguish between different topcoats. For panels with finishes having low densities of metallic flakes, information about the minor constituents of the topcoat and the presence of specific pigments in the undercoats is also obtained in certain cases. These data, likewise, may serve to differentiate between different finish systems.

KEYWORDS: criminalistics, spectroscopic analysis, reflectance, Fourier transform, diffuse reflectance, DRIFTS, infrared spectroscopy, forensic science applications, metallic paints, paints, automotive paints, panels, reference panels

In the previous paper [1] in this study, some of the sampling considerations involved in the direct analysis of metallic paints using diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS) were examined. In the present work, the applications of this technique for some forensic science analyses are discussed.

While DRIFTS can be used in lieu of transmittance methods for the examination and comparison of metallic paints, the requirements of either a high-density metallic finish or a fairly large sample limits this technique for most forensic science examinations. As such, the most useful application of this method may be to screen Reference Collection of Automotive Paint panels rapidly. The need for this may occur in some hit-and-run paint examinations where, given a recovered specimen chip, the make, model, and year of the automobile involved is desired. Provided that a 1974 or later U.S.-manufactured vehicle is responsible, this information, or at least a limited list of possibilities, can usually be determined using the Reference Collection.

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Assuming an original finish, characteristics of the specimen chip, including color, whether the finish is metallic or nonmetallic, sizes and concentrations of the metallic flakes (for the former), and so forth, are compared to those of the various Reference Collection panels. As a result of factors such as limited sample sizes, weathering, and similarities in color for some of the manufactured finishes, a unique match often cannot be established and several panels may be indicated as possibilities. Further analyses may then be performed, but if a large number of panels are involved, the examination of each of these individually can be quite time-consuming. Even for cases in which a single match is obtained, there are usually several different suppliers or types of paint for a given designated color, and distinguishing between these may be useful in further characterizing the type of vehicle sought.

If the specimen chip has a metallic finish, the selected reference panels can, in most cases, be rapidly screened using DRIFTS. The types of information provided by this method for finishes having various densities of metallic flakes are presented in this paper.

Experimental Procedure

The Fourier transform infrared (FTIR) instrument and the diffuse reflectance and DAC accessories used in this work have been described previously [2]. Most of the procedures used in this work have also already been described [1]. Unless otherwise indicated, DRIFTS spectra were obtained using an uncollimated sampling beam with the reflectance accessory mirrors tilted by 20°.

Most of the samples examined consisted of panels from the Reference Collection of Automotive Paints (Collaborative Testing Services, Inc.). These samples, which were not base-coat/clearcoat finishes, will be referred to by their coded designations.

The classification of a particular finish as having a high, medium, or low density of metallic flakes is based upon the DAC throughputs of such finishes, their relative DRIFTS reflectances, and their microscopic appearances [1]. This classification is somewhat arbitrary, however, since the various finishes in the Reference Collection appear to exhibit a continuum of sizes and concentrations of metallic flakes.

Results and Discussion

High- and Medium-Density Metallic Finishes

For panels having high densities of metallic flakes, spectra essentially identical to those obtained using the DAC are obtained with DRIFTS. For medium-density finishes, slight specular reflectance attenuations of some of the stronger peaks may occur, but these do not preclude differentiation of such panels based upon relatively minor differences in spectral features. This is illustrated by Fig. 1, which depicts DRIFTS spectra of five Reference Collection panels having very similar colors. The corresponding DAC spectra are shown in Fig. 2. These panels consist of three distinct colors (color designations 0196, 0272, and 0552), and for one of these, three different paint suppliers or paint types (PA76B0272, DC76B0272, and DB77B0272).

While the spectra of the two acrylic lacquers (Fig. 1*a* and *b*) are similar, the three acrylic melamine enamel spectra (Fig. 1*c* to *e*) exhibit differences, especially in the 1400- to 900-cm⁻¹ region. This region has previously been shown [3] to be useful in distinguishing between some acrylic melamine enamels because of differences in the types of acrylic monomers used in these. DC76B0272 (Fig. 1*c*) also has little or no styrene component [3] present. This may be seen from the absence of aromatic absorptions between 3200 and 3000 cm⁻¹ and the diminished intensities of the peaks between 800 and 700 cm⁻¹ (compare Fig. 1*c* to 1*d* and 1*e*). Note also the weak overtone/combination bands (2000 to 1800 cm⁻¹) characteristic of monosubstituted benzene compounds [4] in Fig. 1*d* and *e*, but not in Fig. 1*c*. These

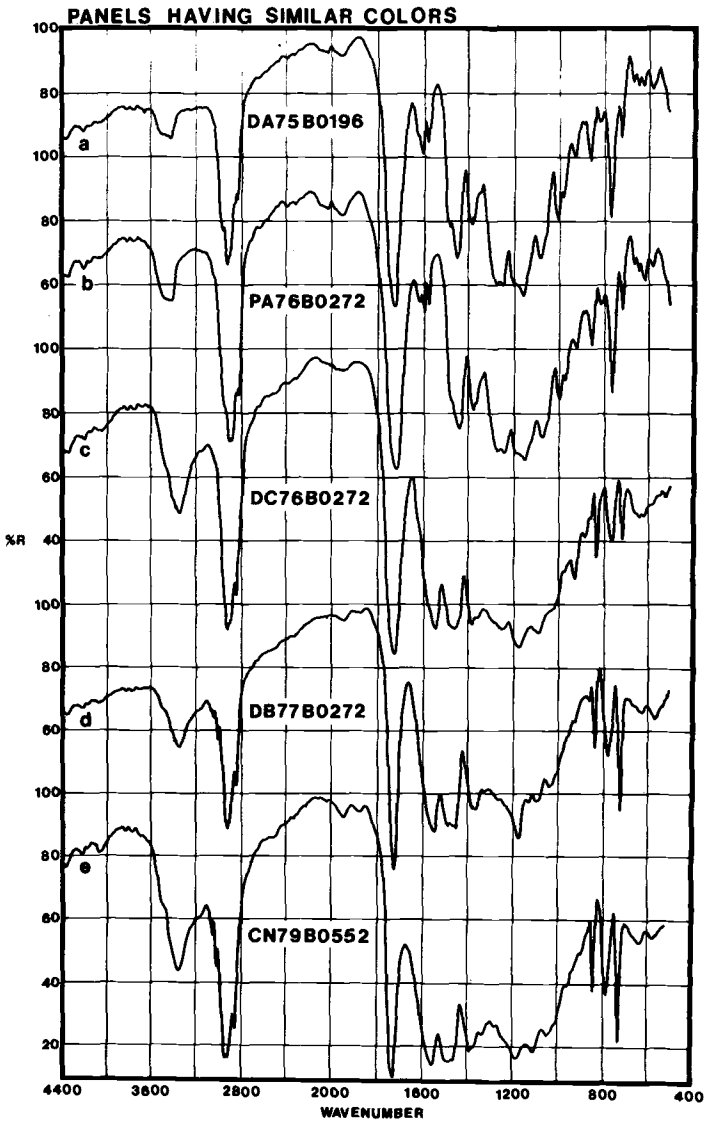


FIG. 1—Reflectance spectra of five different panels having similar colors sampled directly: (a) DA75B0196; (b) PA76B0272; (c) DC76B0272; (d) DB77B0272; and (e) CN79B0552; a gain of 16R was used for all of these and 510 scans were collected.

bands are not clearly observed in DAC spectra (Fig. 2*d* and *e*) since they occur in a region where the diamond anvils absorb.

FTIR instruments having "on-the-fly" Fourier transform capabilities (such as the Analect FX-6200 used in this work) are particularly well suited for screening panels, since DRIFTS spectra of these may be observed a few seconds after the panels have been placed in position. This feature can be used to eliminate quickly a number of panels, for example, based upon whether or not their finishes consist of acrylic or acrylic melamine enamel-type composi-

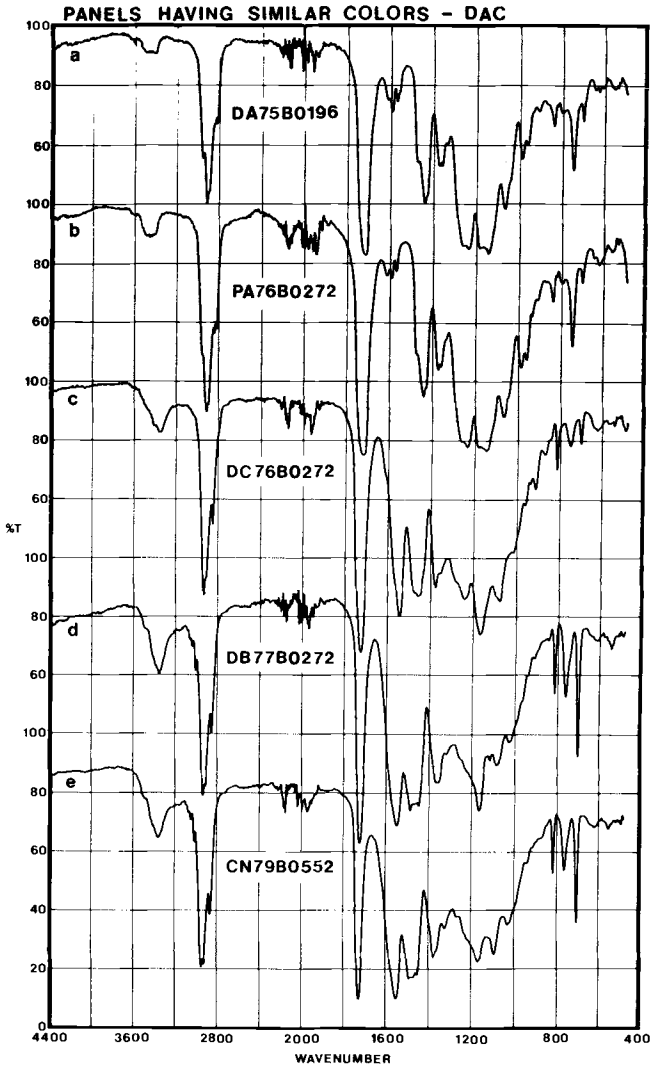


FIG. 2—DAC spectra of the finish layers of five panels whose DRIFTS spectra were depicted in Fig. 1: (a) DA75B0196, gain 32, 510 scans; (b) PA76B0272, gain 16R; (c) DC76B0272, gain 16; (d) DB77B0272, gain 32, 510 scans; and (e) CN79B0552, gain 16.

tions. Panels observed in this manner to have spectra similar to the specimen can then be examined in more detail, either by further DRIFTS analyses or by other methods.

Low-Density Metallic Finishes

For finishes having low densities of metallic flakes, the specular reflectance and other spectral distortions vary somewhat depending upon the particular finish. Relatively minor distortions are observed for FN84 1050 (Fig. 3a and b, for example), whereas spectra of the two panels of color 0694 (Fig. 4b and c) exhibit considerable distortion. Even for the latter

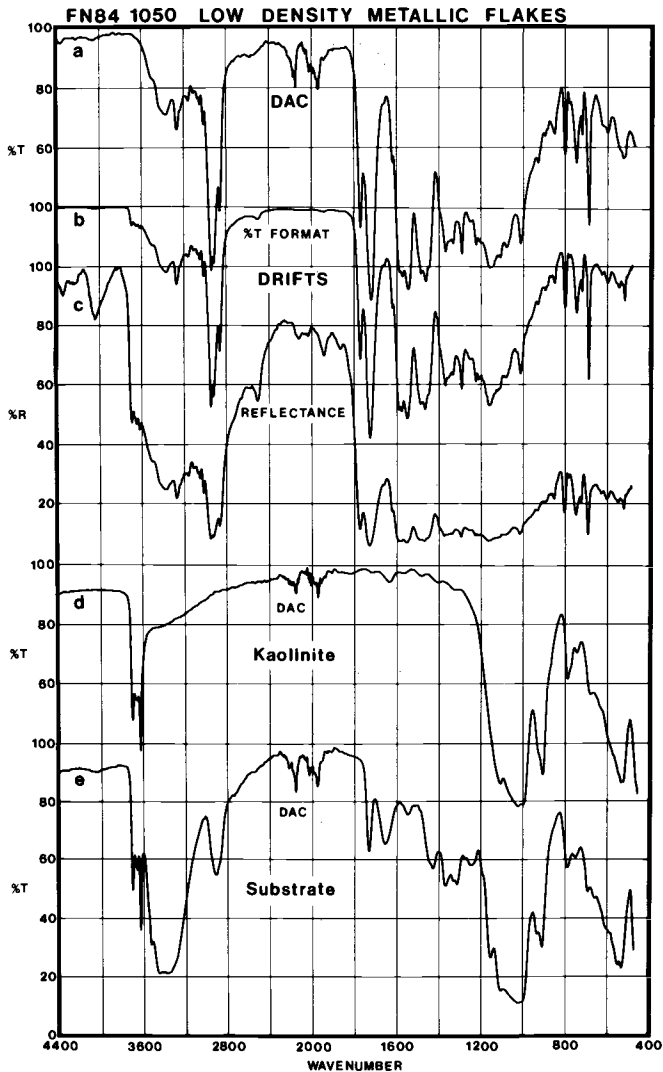


FIG. 3—Spectra of FN84 1050: (a) DAC spectrum of the finish layer; (b) %T format spectrum, sampled directly, gain 32, 510 scans; (c) reflectance spectrum (which was converted to the % format spectrum depicted above); (d) DAC spectrum of kaolinite; and (e) DAC spectrum of a piece of the cardboard substrate used for panel FN84 1050.

(which might be considered a “worst-case” example), one can distinguish between the acrylic (Fig. 4b) and acrylic melamine enamel (Fig. 4c).

The differences between the spectra of FN84 1050 and those of NA80F0694 and NW80F0694 cannot be attributed solely to differences in metallic flake densities since these do not vary significantly. Microscopic examinations of these panels indicate, however, a clear difference in topcoat turbidity. The finishes of color 0694 are much clearer than the finish of FN84 1050, which may account, in part, for the apparently greater sample beam penetration and the interference fringes observed for the former panels (both of which have primers beneath their topcoats). The turbidity of FN84 1050, in contrast, would be expected

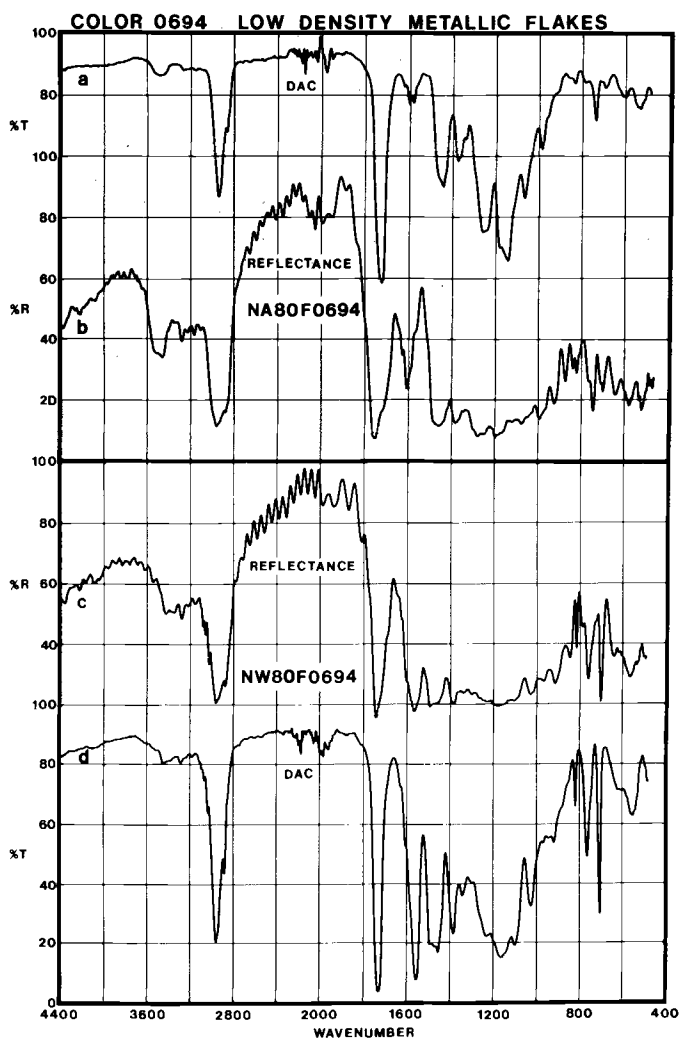


FIG. 4—Spectra of NA80F0694: (a) DAC spectrum of the finish layer; (b) reflectance spectrum, sampled directly, gain 32R, 510 scans. Spectra of NW80F0694: (c) reflectance spectrum, sampled directly, gain 32, 510 scans; and (d) DAC spectrum of the finish layer.

to increase scattering with the topcoat and thus the amount of Kubelka-Munk sampling that can occur in this finish. Note the good agreement between the %T format of the DRIFTS results (Fig. 3b) and the DAC spectrum (Fig. 3a) for FN84 1050.

Another factor that could explain these differences is the microscopic surface texture of a particular finish. This texture obviously has a strong bearing on the effectiveness of rotation of the accessory mirrors on minimizing specular reflectance. This factor, together with differences in topcoat turbidity, may explain the reason mirror rotation appears to be more effective for some low-density finishes than for others.

Most of the panels in the Reference Collection have a metal backing with the majority of these having primers. Some panels (mostly produced by Ford), however, have a cardboard

backing without a primer. DRIFTS spectra of low-density finishes on the latter exhibit four absorptions (which may appear to be a triplet as a result of the close spacing between the middle two weaker peaks) at 3695, 3654, 3667, and 3622 cm^{-1} . These may be seen in the reflectance spectrum of FN84 1050 (Fig. 3c).

These absorptions are due to kaolinite (also known as kaolin or china clay) used in the cardboard substrate. The DAC spectrum of a portion of a panel cardboard, taken from the surface just beneath the finish, is shown in Fig. 3e. Absorptions of kaolinite (Fig. 3d), which is a very common filler used in paper [5], are clearly seen along with those of cellulose. Previously, it was shown that DRIFTS sampling extends to the bottom of the finish layer for low-density metallic finishes and that these samples thus are not "infinitely thick" as are those having higher densities of metallic flakes [1]. The observation of substrate kaolinite peaks indicates that some sampling of subcoats may occur in these cases—at least for some frequencies.

Finishes considered to have low densities of metallic flakes are usually quickly identified from a stereomicroscopic examination: a relatively large fraction of the topcoat surface area is devoid of metallic flakes (including flakes at all depths). From the examination of a large number of Reference Collection panels, it was observed that such finishes almost always occur in darker colors. This undoubtedly reflects the need to have a higher hiding power in these finishes which are deficient in opaque flakes. These finishes are thus "infinitely thick" when viewed by visible radiation, but are not so for DRIFTS sampling.

Talc and Kaolinite in Undercoats

While the observation of cardboard panel kaolinite peaks in the DRIFTS spectra of low-density finishes provides little useful information, the ability to make this observation can also be used to screen certain panels to determine if kaolinite and talc are present in the undercoats. Talc, in particular, is commonly used in automotive paint primers along with other inorganic pigments, including titanium dioxide, barium sulfate, calcium carbonate, silica, kaolinite, and iron oxide [3,6]. These pigments and extenders may appear alone or in combination, and their presence serves to distinguish between different primers [3].

Talc, like kaolinite, has a very characteristic hydroxyl stretching absorption at 3678 cm^{-1} (Fig. 5c). This absorption may be seen in the reflectance spectrum (Fig. 5b) of KN78F0247, which has a low density of metallic flakes. The DAC spectrum of the primer used on this panel is shown in Fig. 5d, and as may be seen (compare to Fig. 5c), this primer contains talc.

Reflectance spectra of two other panels (KN81F0247 and PN81F0247) having the same color as KN78F0247 are shown in Fig. 6b and e to illustrate how this information might be useful in distinguishing between different automotive paint systems having similar topcoats. The 3678- cm^{-1} talc absorption is not observed in these spectra; the corresponding primers, whose DAC spectra are shown in Fig. 6c and d, respectively, do not contain talc. Note that the DAC spectra of the topcoats of KN78F0247 (Fig. 5a) and KN81F0247 (Fig. 6a), in particular, are quite similar (which may not be too surprising since both are Cook nonaqueous dispersion enamels), whereas their primer spectra (Figs. 5d and 6c) are easily distinguished.

The range in the intensity of the 3678- cm^{-1} talc peak observed in the reflectance spectra of various panels is depicted in Fig. 7. The majority of low-density finishes which have talc in the primer exhibit a 3678- cm^{-1} peak having an intensity intermediate between that observed in Figs. 5b and 7b. A few, notably those having a black-colored primer, exhibit weaker talc peaks such as depicted in Fig. 7e; talc peaks for some of these may not be discernible, especially if water vapor absorptions, which occur in this same region, are present. For finishes having higher densities of metallic flakes, sampling of the subcoats decreases and this talc

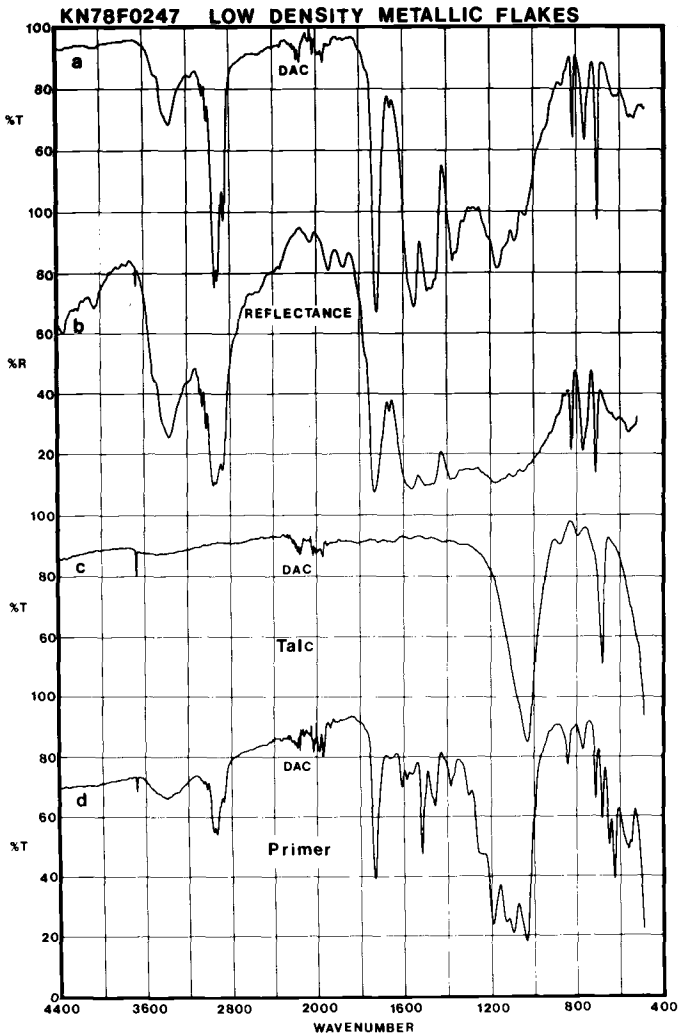


FIG. 5—Spectra of KN78F0247: (a) DAC spectrum of the finish layer; (b) reflectance spectrum, sampled directly, gain 32, 510 scans; (c) DAC spectrum of talc; and (d) DAC spectrum of the primer layer, gain 16.

absorption is usually not observed (for the medium-density finish whose spectrum is shown in Fig. 5d of the previous paper [1] in this study, this peak is barely perceptible).

Somewhat surprisingly, relatively strong talc—and especially kaolinite—absorptions are observed for some panels in which these substances occur in a second undercoat layer, but not in the undercoat layer directly below the topcoat. An example of this is BN85 1108, which has two undercoat layers: a brown layer on the metal substrate which contains kaolinite (Fig. 8d) and a light-gray layer adjacent to the topcoat which does not (Fig. 8c). Note the kaolinite absorptions in the reflectance spectrum (Fig. 8b) of this panel. It is thus clear that this type of DRIFTS sampling can occur through *two* diverse layers (one of which contains an appreciable concentration of suspended particulates) in certain cases.

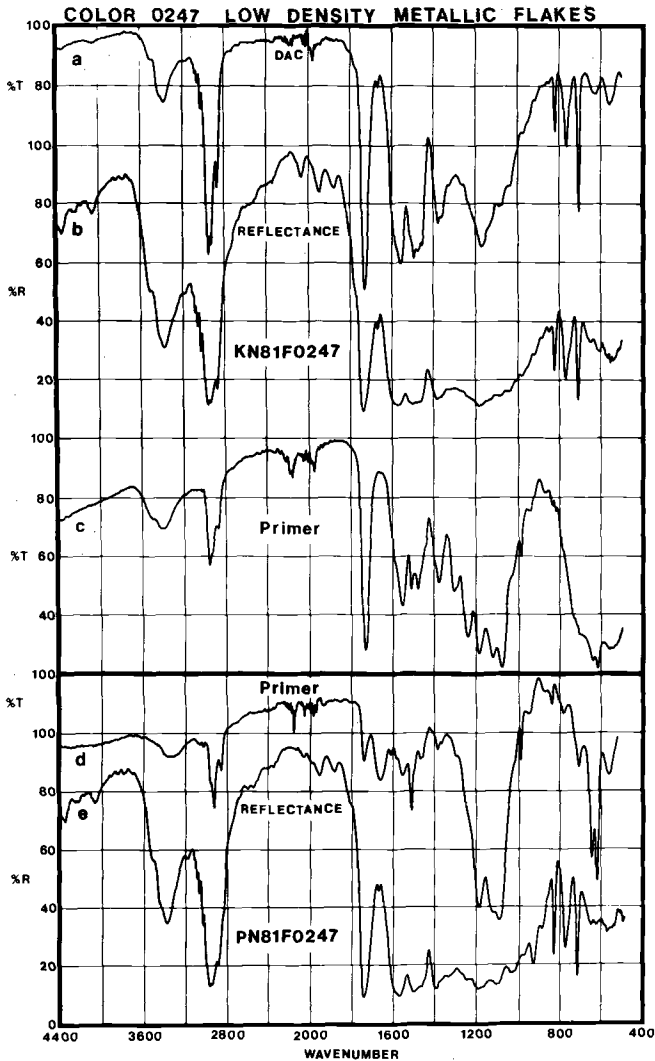


FIG. 6—Spectra of KN81F0247: (a) DAC spectrum of the finish layer; (b) reflectance spectrum, sampled directly, gain 32, 510 scans; (c) DAC spectrum of the primer layer. Spectra of PN81F0247: (d) DAC spectrum of the primer layer; and (e) reflectance spectrum, sampled directly, gain 32, 510 scans.

Although this DRIFTS feature of sampling subcoats can provide useful screening information, note that its use with the Reference Collection is quite limited. While some of the primers used on Reference Collection panels are those actually used with the specified topcoat, this is not true in general as this collection is concerned only with topcoats. This method could be useful, however, for laboratories (such as the FBI and Royal Canadian Mounted Police [RCMP] laboratories) which maintain reference panels representing the entire plant system as actually produced in a particular manufacturing plant. The RCMP laboratories, for example, rely heavily upon information about undercoats [3, 6-10] in their automotive paint examinations and can, in some cases, determine the particular assembly plant from which a paint originated based primarily upon undercoat compositions.

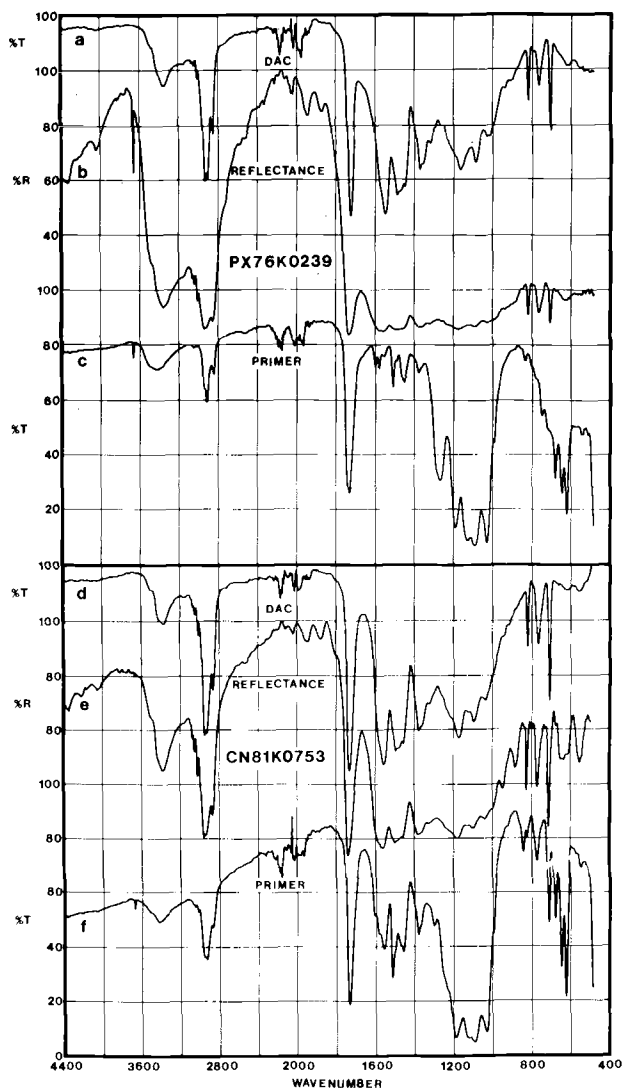


FIG. 7.—Spectra of PX76K0239: (a) DAC spectrum of the finish layer; (b) reflectance spectrum, sampled directly, gain 16R, 510 scans; (c) DAC spectrum of the primer layer. Spectra of CN81K0753: (d) DAC spectrum of the finish layer; (e) reflectance spectrum, sampled directly, gain 32, 510 scans; and (f) DAC spectrum of the primer layer, gain 16.

Since talc and kaolinite hydroxyl peaks (which are actually relatively weak absorptions, see Figs. 5c and 3d) appear prominently in many reflectance spectra, the question may arise as to why the other stronger absorptions of these substances are not likewise observed. In particular, because DRIFTS sampling generally occurs to greater depths for the lower frequencies [11], one might reasonably expect to observe the lower-frequency absorptions of these substances (or other primer constituents) rather than the hydroxyl stretching peaks. This unexpected behavior arises from differences in the reflective properties of the undercoat and the absorption and scattering properties of both the topcoat and undercoat.

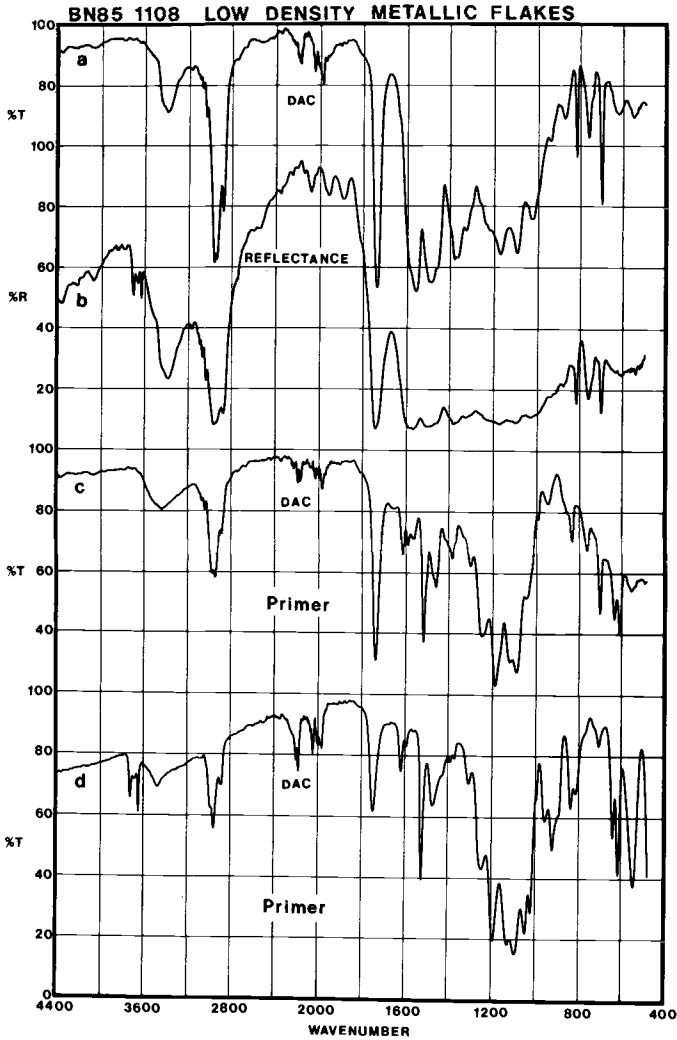


FIG. 8—Spectra of BN85 1108: (a) DAC spectrum of the finish layer; (b) reflectance spectrum, sampled directly, gain 32R, 510 scans; (c) DAC spectrum of the first primer layer (a gray-white layer just beneath the finish layer); and (d) DAC spectrum of the second primer layer (a brown layer beneath the gray-white primer).

For observable DRIFTS sampling of an undercoat to occur, it is clear that an appreciable fraction of the sampling radiation must reach and penetrate the undercoat, then return to the topcoat surface. This limits such sampling to those spectral regions where the topcoat does not absorb strongly. Since the strongest talc and kaolinite absorptions (the silicon-oxygen stretches at 1100 to 1000 cm^{-1} , see Figs. 3d and 5c) occur in regions where the topcoats also absorb strongly, this could explain, in part, their absence in the DRIFTS spectra.

A more important factor, however, appears to be specular reflectance from the surface of the undercoat (that is, the topcoat/undercoat interface). Direct DRIFTS spectra of primers containing talc or kaolinite, for example, display "normal" diffuse reflectance peaks for the hydroxyl stretching peaks, but inverse specular reflectance peaks for the other talc or kaolin-

ite absorptions (Fig. 9). Even with the reflectance accessory mirrors tilted by 20°, the spectrum of the talc-containing primer (Fig. 9*b*) displays strong specular reflectance peaks for the talc absorptions below 1200 cm⁻¹. While somewhat less specular reflectance may occur for sampling radiation incident upon such a primer surface from an adjacent topcoat (due to less of a difference² in index of refraction between primer and topcoat versus primer and air), it is likely that this effect still prevents much of the strongly absorbed primer frequencies from being sampled. The difference in reflective behavior between the talc hydroxyl absorption and other undercoat peaks might also be due, in part, to the λ² factor in the Fresnel equation.³

The relative weakness of the talc absorptions observed for panels having black primers (Fig. 7*e*) appears to be due to the strong scattering and absorption of these primers (presumably caused by the carbon pigments used in them). This strong scattering is evident from the prominent slopes of the baselines (above 1800 cm⁻¹) in the DAC spectra of such primers (see, for example, Fig. 7*f*; compare to Figs. 5*d* and 7*c*). Since scattering and absorption both decrease the penetration depth of the sampling beam [11, 14] in DRIFTS (and hence the amount of absorption that can occur), this could explain the weakness of the talc absorptions observed for these primers.

Talc in Topcoats

Certain Reference Collection panels having low densities of metallic flakes were observed to have very weak peaks at 3678 cm⁻¹ in their reflectance spectra, but no talc was found in the undercoats of any of these. These peaks are typically⁴ much weaker than those observed for most panels having talc in an undercoat. An example of such a panel is DB85 1195 (Fig. 10*a*), for which this 3678-cm⁻¹ peak is relatively strong; a portion of this same spectrum is shown expanded in Fig. 11*a*. As may be seen from Fig. 10*d*, the primer layer of DB85 1195 does not contain talc. A portion of the spectrum of NA75F0201, which also has the weak 3678⁻¹-cm⁻¹ peak, is shown in Fig. 11*d* (the entire spectrum of this panel may be seen in Fig. 4*d* in the previous paper [1] in this study; see also Fig. 1*d* in this same paper).

These weak 3678⁻¹-cm⁻¹ peaks are due to small amounts of talc present in the topcoats themselves. Although they are not observed in normal DAC spectra (Fig. 10*b*), these talc peaks may be seen in DAC spectra of very thick samples of such finishes (Fig. 10*c*). Their

²The specular reflectance from the undercoat surface is qualitatively described by the Fresnel equations, which for radiation perpendicularly incident from a transparent phase onto the surface of an absorbing medium is given [12, 13] by:

$$R_s = \frac{(n_2 - n_1)^2 + n_2^2(\lambda^2/16\pi^2)\alpha^2}{(n_2 + n_1)^2 + n_2^2(\lambda^2/16\pi^2)\alpha^2}$$

where

- R_s = reflectivity of an incident ray,
- n_2 = index of refraction of the absorbing medium (the undercoat),
- n_1 = index of refraction of the transparent medium (the topcoat for nonabsorbing spectral regions),
- λ = wavelength of the incident radiation, and
- α = absorption coefficient of the absorbing medium for the wavelength λ as defined in the Beer-Lambert equation $T = e^{-\alpha t}$ where
- T = the transmittance of the absorbing medium having a thickness t .

Note that even if the topcoat and undercoat have the same index of refraction, ($n_1 = n_2$), specular reflectance still occurs if α is nonzero. Since inorganic compounds have relatively high absorption coefficients and indices of refraction, the second terms of this equation are large, resulting in greater reflectances.

³See footnote 2.

⁴DRIFTS spectra of a few panels from 1974 and 1975 were observed to have relatively strong 3678-cm⁻¹ peaks with intensities comparable to those observed for many panels having talc in an undercoat.

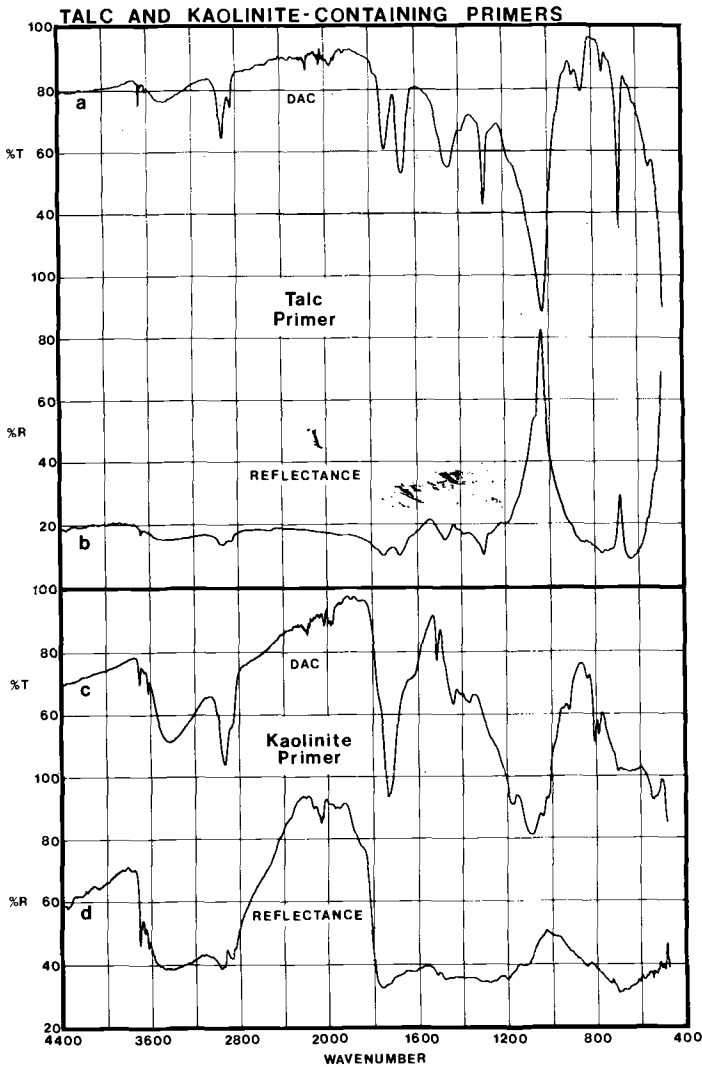


FIG. 9—Spectra of the red-brown primer layer from an unknown vehicle: (a) DAC spectrum; (b) reflectance spectrum, sampled directly, gain 16R, 510 scans. Spectra of the gray primer layer from a 1977 Volkswagon Sirocco: (c) DAC spectrum; and (d) reflectance spectrum, sampled directly, gain 32, 510 scans.

observation in reflectance spectra, like the styrene overtone/combination bands previously discussed, arises from the enhancement effects [1,2,11] of Kubelka-Munk sampling (note also the increased intensities of the other weak absorptions in Fig. 10a; compare with Fig. 10b and c).

While a considerable amount of sample (and effort) was required to obtain the DAC spectrum depicted in Fig. 10c, it is possible to observe these very weak talc peaks for relatively small samples using DRIFTS. This is illustrated by Fig. 11b, which shows a portion of the reflectance spectrum obtained for a 1- by 1-mm square chip of DB85 1195. A collimated beam was used for this sampling with the reflectance accessory mirrors untilted. Note that

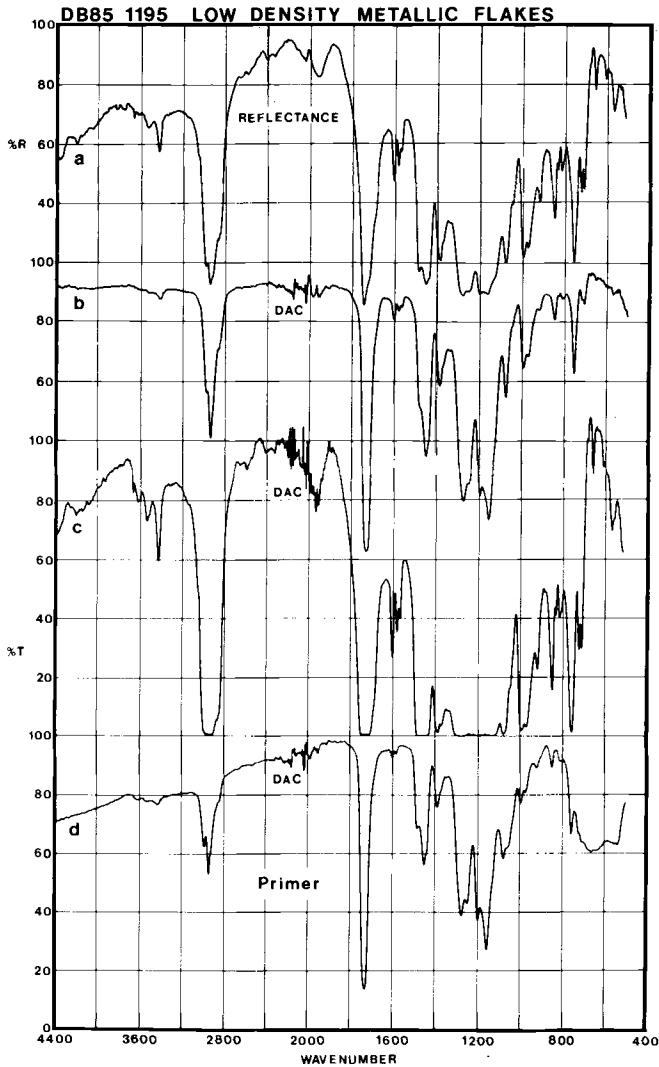


FIG. 10—Spectra of DB85 1195: (a) reflectance spectrum, sampled directly, gain 32, 510 scans; (b) DAC spectrum of the finish layer; (c) DAC spectrum of the finish layer using a very thick sample, gain 128, 715 scans; and (d) DAC spectrum of the primer layer.

the intensity of the talc absorption obtained for this spectrum is not appreciably less than that observed using the entire panel. Although most of the stronger absorptions (not shown) for this chip are distorted due to specular reflectance, the weak high-frequency talc peak is unaffected (for reasons already mentioned).

For some finishes, this talc peak is quite weak and may be difficult to discern from the background noise (or water vapor absorptions if these are present) as may be seen for DB81K0781 (Fig. 11c; the talc peak is indicated by an arrow). Talc peaks may also be observed in the spectra of some finishes having medium densities of metallic flakes, although these usually are weaker since less Kubelka-Munk sampling occurs for these. An example of such a finish is DA75B0196 (Figs. 1a and 11e). As a comparison, note the absence of this

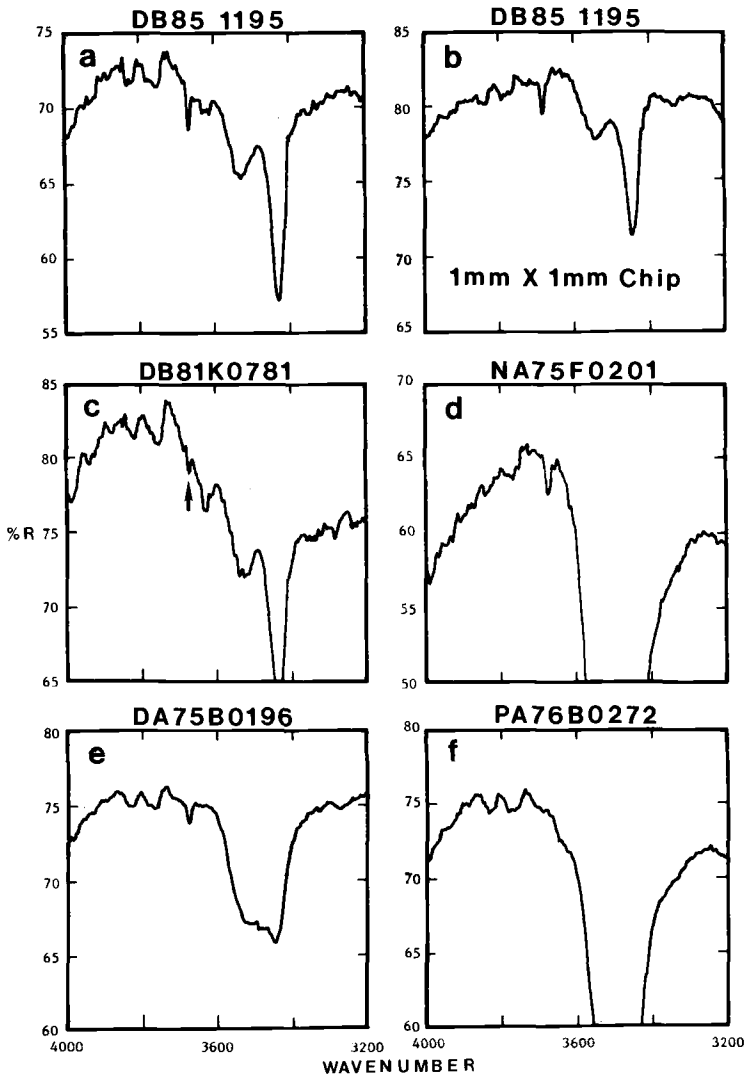


FIG. 11—Reflectance spectra of some panels or chips in the 4000- to 3200- cm^{-1} region: (a) DB85 1195 (same spectrum as depicted in Fig. 10a); (b) DB85 1195, 1- by 1-mm square chip sampled using a collimated beam with the reflectance accessory mirrors untilted, gains 8R/1R (background); (c) DB81K0781, gain 32; (d) NA75F0201 (same spectrum as depicted in Fig. 4d in the previous paper [1] in this study); (e) DA75B0196 (same spectrum as depicted in Fig. 1a); (f) PA76B0272 (same spectrum as depicted in Fig. 1b).

talc peak in a finish (PA76B0272) having a very similar color and infrared spectrum (Figs. 1b and 11f).

Although it is common in undercoats, one does not normally associate talc with a metallic topcoat. We have learned,⁵ however, that small amounts of talc are used in certain metallic automotive finishes as an anti-settling agent (not only is talc itself easily dispersed, but it also

⁵The DuPont Company, Automotive Paint Division, private communication, Feb. 1987.

tends to keep other pigments suspended [15]). Typically, talc may comprise only 3 to 5% of the total solids content of such a paint, and it is used together only with certain pigments.

Most of the Reference Collection panels examined which had talc in an undercoat were found to have acrylic melamine enamel topcoats. In contrast, almost all of the finishes observed to contain talc are acrylic lacquers. More significantly, the majority of these lacquers were identified as DuPont finishes, with the remainder produced by BASF Inmont and a few by PPG Industries. Since the acrylic lacquers produced by these manufacturers are used exclusively [16] on General Motors vehicles (since 1974 when the Reference Collection began), the presence of talc in a topcoat might be a useful supplemental feature in identifying the manufacturer or type of vehicle for certain original U.S. automotive finishes.

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