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Technical Report

ATTENUATED TOTAL REFLECTANCE SPECTROSCOPY OF PAINT VEHICLES

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ATTENUATED TOTAL REFLECTANCE SPECTROSCOPY OF PAINT VEHICLES

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by

R. J. McGowan

ABSTRACT

Certain problems are encountered in using infrared transmission spectroscopy for the quantitative evaluation of paint vehicles; therefore, an investigation of attenuated total reflectance spectroscopy was conducted.

In thin-film analysis, it is very difficult, if not impossible, to know the sample thickness, knowledge of which is essential in quantitative infrared transmission spectroscopy. The attenuated total reflectance technique eliminates this problem. Therefore, it was concluded that this method is superior to transmission measurements for quantitative evaluation of paint vehicles.

INTRODUCTION

A serious problem encountered in infrared transmission studies of paint vehicles is that of controlling sample size and thickness in order to produce infrared spectra of quantitative value. The intensity of the measured spectrum is related to the thickness of the sample and the concentration of the functional groups. Quantitative analysis of the spectrum is dependent on the intensity of the variations in absorption peaks caused by the concentration of the functional groups.

A very new technique, Attenuated Total Reflectance (ATR), developed by Dr. J. Fahrenfort,[1] offers a solution to this problem. It differs from infrared transmission spectroscopy in that the infrared beam is reflected from the sample into the slit of the spectrophotometer (Figure 1) instead of passing straight through the sample as in the case of infrared transmission (Figure 2). Consequently, the sample thickness will not affect the spectrum if the sample is at least 5 microns thick, because reflection is from the first 5-micron layer of the sample surface.

The principle here is that when a beam of radiation .. totally internally reflected as within a prism, there occurs some penetration of this beam into the medium in contact with the reflecting face of the prism. The degree to which this beam penetrates the interface depends upon the difference between the refractive index of the prism and of the medium in contact with it. When a sample of some organic material is placed on the reflecting face of the prism, penetration increases substantially at the wavelengths where the sample absorbs, because of the increase in the refractive index (the index of refraction of a substance is a function of wavelength). This energy is selectively absorbed, and the resultant beam passes through the opposite side of the prism, thus producing a spectrum similar to infrared transmission (Figure 3).

EXPERIMENTATION

Infrared Transmission and ATR Studies

In a field study to determine the quantitative value of infrared transmission, samples of paint vehicles were prepared and spectrograms were made at NCEL and four other laboratories. Three laboratories used Beckman IR-5's and two used IR-4's. The data were evaluated at NCEL. The details of the study and the analysis of data are given in Appendix A.

To compare ATR with infrared transmission, the field study was repeated by the three laboratories using the IR-5. The details and data analysis are given in Appendix B.

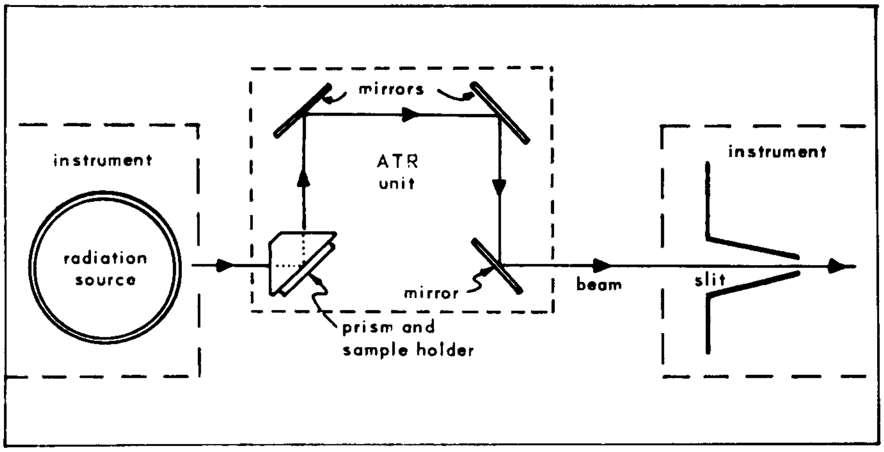

Figure 1. Attenuated total reflectance spectroscopy.

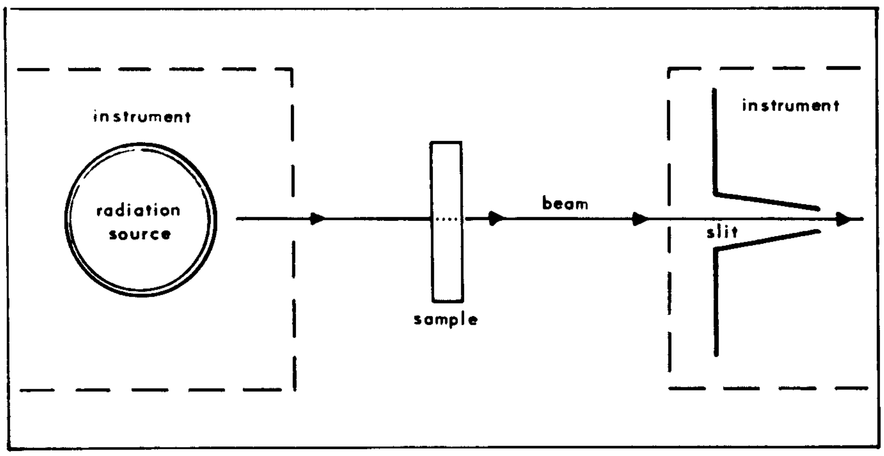

Figure 2. Infrared transmission spectroscopy.

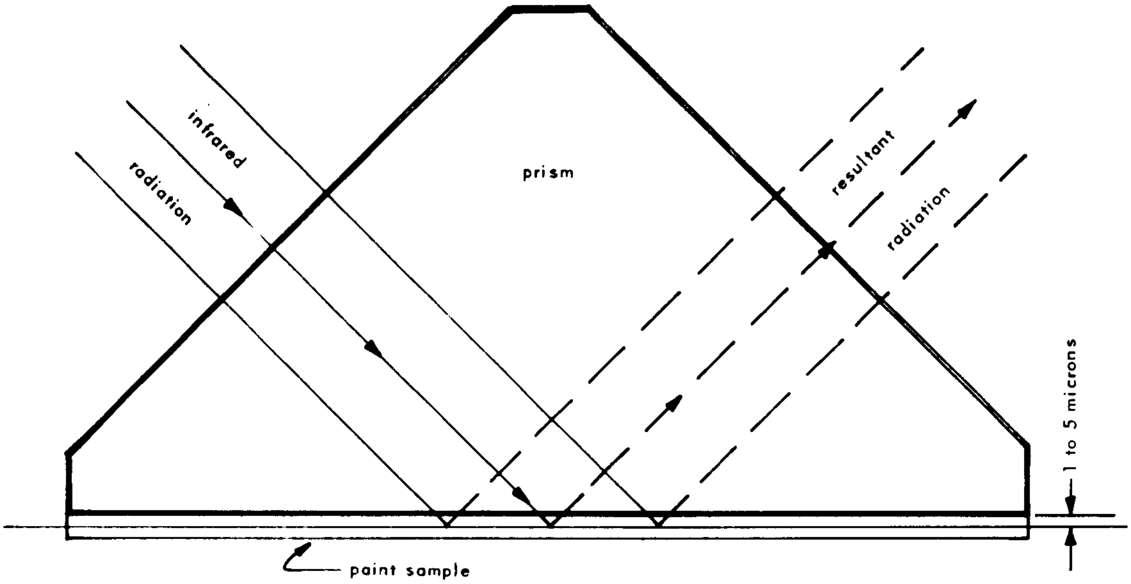

Figure 3. KRS-5 double salt prism (48% TlBr and 52% TlI).

Sample Preparation

The samples evaluated by infrared transmission were prepared as follows:

1. The pigment and vehicle were separated, using a Federal test method[2] with extraction mixture "A."

2. Aliquots of 0.2 milliters were placed on a salt window (NaCl) and dried for 15 minutes at 50 C.

3. The salt window was placed (with holder) in the spectrophotometer's sample holder.

For the evaluation of the ATR technique the samples were prepared as follows:

1. The pigment and vehicle were separated, using the same Federal test method with extraction mixture "A."

2. Aliquots of 0.2 milliters were placed on the prism of the ATR unit (Figures 4 and 5) and dried for 15 minutes at 85 C.

3. The prism was then placed in the ATR unit (Figure 6), which was put in the spectrophotometer's sample holder (Figure 7).

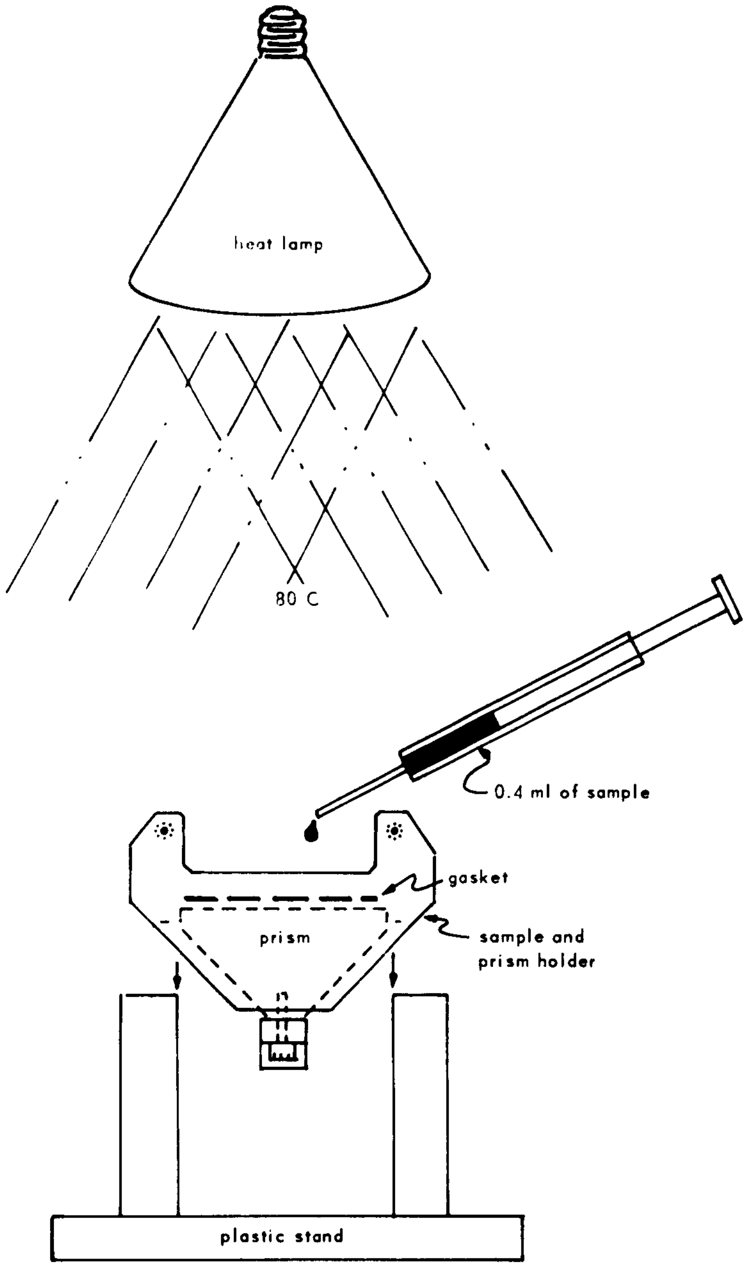

Figure 4. Position while applying sample.

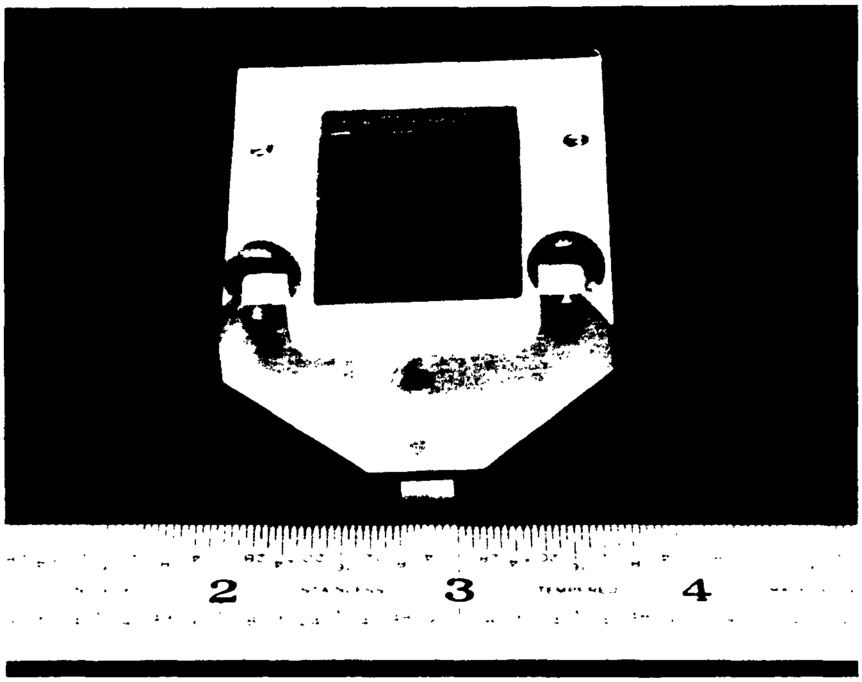

Figure 5. KRS-5 double salt prism in holder.

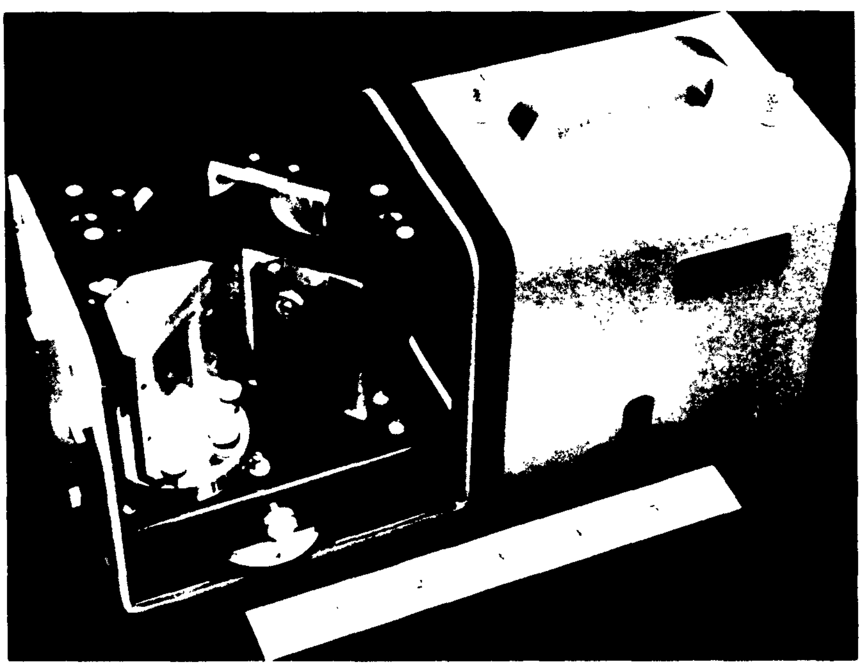

Figure 6. ATR unit with cover removed.

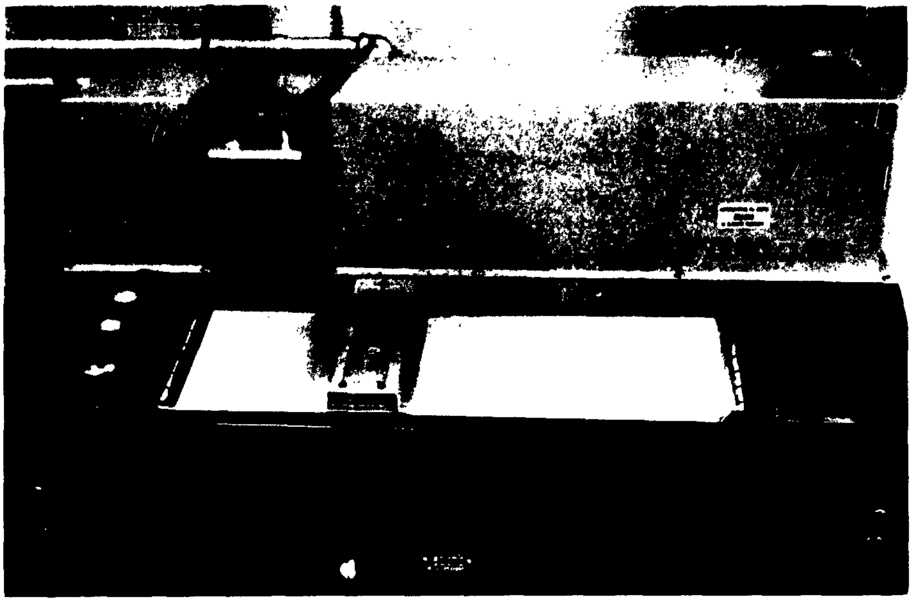

Figure 7. ATR unit in the IR-5 spectrophotometer.

RESULTS

The two techniques are compared in the following tabulations for the three laboratories using the IR-5. The results of the infrared transmission study detailed in Appendix A are summarized in Table I. The results of the ATR study detailed in Appendix B are summarized in Table II. Four random spectrographs were used to obtain the average peak values for each laboratory.

Ranges were calculated as follows:

X_i = average peak values arranged in ascending order (i = 1, 2, 3)

Range = X(max) - X(min)

Example: (first set of values at 3.4 microns)

Average peak values: 3.25, 3.23, 1.91

X_1 = 1.91, X_2 = 3.23, X_3 = 3.25

X(max) = 3.25, X(min) = 1.91

Range = 1.34

Table I. Summary of IR-5 Infrared Transmission Results

Peak Location (microns)	Average Peak Values			Overall Average	Range
	Bremerton	Cal-Colonial	NCEL		
3.4	3.25	3.23	1.91	2.80	1.34
3.5	1.90	2.18	1.46	1.85	0.72
5.8	67.80	6.71	3.13	25.88	64.67
7.9	23.00	5.26	2.76	10.34	20.24
8.9	4.66	2.92	1.81	3.13	2.86
9.4	3.66	2.63	1.71	2.67	1.95
13.5	1.91	1.73	1.23	1.69	0.69

Table II. Summary of IR-5 ATR Results

Peak Location (microns)	Average Peak Values			Overall Average	Range
	Bremerton	Cal-Colonial	NCEL		
3.4	1.28	1.26	1.45	1.33	0.19
3.5	1.25	1.20	1.25	1.23	0.05
5.8	2.12	2.34	2.25	2.24	0.21
7.9	2.03	1.09	1.87	1.96	0.16
8.9	1.82	1.94	1.78	1.85	0.17
9.4	1.59	1.69	1.60	1.62	0.10
13.5	1.43	1.38	1.42	1.41	0.05

DISCUSSION

Because of the sensitivity of infrared transmission to sample size and thickness, its application to quantitative evaluation in thin-film studies is limited. In quantitative evaluation, sample size must remain constant, because the intensity of functional groups is directly proportional to concentration and sample size. If the thickness is known, it can be compensated for. In the case of thin films, such as paint vehicles, it becomes a problem to determine the exact thickness. In some cases, variations in thickness can be compensated for mathematically by the absorbance-ratio method.[3] A practicable technique for infrared evaluation of paint vehicles should be free of problems related to sample thickness.

The results of this study show that sample measurement methods are inadequate for controlling sample size; i.e., it is not possible because of the nature of the sample to reproduce the same thickness. These results show that infrared transmission has a high variability (3 to 62 percent) and range (0.7 to 64.6), as shown in Appendix A and Table I respectively. The ATR technique shows a much lower variability (4 to 16 percent) and range (0.05 to 0.21), as shown in Appendix B and Table II respectively. Thus ATR offers a solution to the problem of sample size and makes quantitative evaluation of paint vehicles practicable.

ATR also offers the possibility of eliminating the need to separate the pigment from the vehicle in order to obtain a spectrum.

The cost of the ATR attachment for an infrared spectrometer would be offset by fewer prism replacements and a reduction in the time required to prepare a sample for analysis. The increased precision alone of a spectrometer with the ATR attachment would warrant the small additional cost.

FINDING

The variability of the different absorption peaks in ATR spectra is less than the variability of the same peaks in infrared transmission.

CONCLUSIONS

1. Attenuated total reflectance spectroscopy (ATR) can be used for the quantitative evaluation of paint vehicles.

2. ATR makes it unnecessary to know the sample size and thickness, and thus simplifies preparation.

3. ATR is superior to transmission methods because the results are independent of sample size and thickness.

RECOMMENDATIONS

1. Because it eliminates problems encountered in quantitative transmission spectroscopy, ATR should be adopted for all future paint studies.

2. Interlaboratory quantitative evaluation tests should be conducted with ATR.

ACKNOWLEDGMENT

The author expresses appreciation to Mr. George E. Hayo, NCEL statistician, for his assistance in designing the experiment and analyzing the data.

REFERENCES

1. Fahrenfort, J. "Attenuated Total Reflection," Spectrochimica Acta, Vol. 17 (1961), pp. 698-709.

2. General Services Administration, No. 141 Federal Test Method Standard, 15 May 1958, Method 4021.

3. Chicago Society for Paint Technology. Infrared Spectroscopy — Its Use as an Analytical Tool in the Field of Paints and Coatings, by the Infrared Spectroscopy Committee. Chicago, Illinois, 31 October 1960.

Appendix A

STATISTICAL ANALYSIS OF INFRARED TRANSMISSION DATA

by G. E. Hayo

SUMMARY

Experiments were conducted at five laboratories to evaluate the infrared transmission method of paint analysis. Two laboratories used the IR-4 and the remaining three used the IR-5 (Beckman Instruments). Paint consisted of two batches from one manufacturer, distributed to each of the laboratories.

Laboratory differences, batch differences, laboratory-batch differences, and sample-preparation error (including instrument error) are evaluated in this appendix.

It was found that the greatest error could be attributed to the method of sample preparation.

DATA ANALYSIS

The data, presented at the end of the appendix, consisted of measurements of amplitude for seven peaks. Peaks 3.4, 3.5, 5.8, 8.9, 9.4, and 13.5 were compared to peak 7.9 in the equation

$$R(i) = \frac{\ln(P_i)}{\ln(P_{7.9})}$$

where i = 3.4, 3.5, 5.8, 8.9, 9.4, 13.5 (microns)

P = absorbance ratio at a particular peak

$R(i)$ = absorbance of a logarithmic peak ratio

Analysis-of-variance procedures were used to analyze the calculated values of $R(i)$.

IR-4 Data

This data, obtained from Philadelphia Naval Air Station and Philadelphia Naval Shipyard, consisted of two samples for each peak for each batch for each of the two laboratories. Laboratory and batch differences were not significant.* The variability of sample preparation ranged from 2 to 14 percent. Table III lists the effects of different factors and the coefficient of variation** for the i values.

Table III. PHILAIR vs PHILNAVSHIPYD (Infrared Transmission, IR-4)

i Value of R(i)	F-Ratio Values of R(i)			Coeff. of Variation (%)
	Lab	Batch	Lab-Batch	
3.4	0	0	0	10
3.5	0	0	0	14
5.8	3.0	0.1	0.2	5
8.9	7.6	0.9	1.7	2
9.4	6.4	0	1.3	3
13.5	5.0	0.7	0.3	5

F-ratio ≥ 7.7 (Significant at 0.05 level)

IR-5 Data

The IR-5 data was obtained from Cal-Colonial at Orange, California, Bremerton Naval Shipyard, and NCEL. The variability of sample preparation ranged from 17 to 62 percent at Bremerton Naval Shipyard. The corresponding variability at NCEL ranged from 3 to 24 percent. The Cal-Colonial variability of sample preparation was not calculated. Table IV lists the coefficient of variation for Bremerton Naval Shipyard and NCEL for the i values.

* Not statistically significant at the 0.05 level, using the F-test.

** Coefficient of variation: a measure of dispersion about the sample mean (sometimes referred to as "relative standard deviation").

Table IV. BREMNAVSHIPYD and NCEL
(Infrared Transmission, IR-5)

i Value of R(i)	Coeff. of Variation (%)	
	NCEL	BREMNAVSHIPYD
3.4	24	27
3.5	9	34
5.8	19	23
8.9	7	17
9.4	6	19
13.5	3	62

It was decided to compare the IR-5 data at Cal-Colonial with that at NCEL and the IR-5 data at Bremerton Naval Shipyard with that at NCEL.

Cal-Colonial Data. In the Cal-Colonial vs NCEL data, the initial sample of NCEL data and its replicate constituted Test 1. NCEL samples 3 and 4 were Test 2. NCEL Test 1 and Test 2 were compared with the Cal-Colonial data.

In the Test 1 comparison with Cal-Colonial, batch differences were significant for R(3.4). In the Test 2 comparison with Cal-Colonial, laboratory differences were significant for R(13.5). The variability of sample preparation for the two laboratories together ranged from 3 to 25 percent. Table V lists the effects of different factors and the coefficient of variation for the i values within the two test samples.

Bremerton Naval Shipyard. In the Bremerton vs NCEL data, laboratory differences were significant for R(3.4), R(3.5), and R(5.8). Batch differences were significant for R(8.9). Laboratory-batch differences were significant for R(3.4). The variability of sample preparation for the two laboratories together ranged from 13 to 32 percent. Table VI lists the effects of different factors and the coefficient of variation for the i values.

Table V. CAL-COLONIAL vs NCEL (Infrared Transmission, IR-5)

Test No.	i Value of R(i)	F-Ratio Values of R(i)			Coeff. of Variation (%)
		Lab	Batch	Lab-Batch	
1	3.4	7.4	11.7*	2.3	6
	3.5	2.1	3.8	0.6	17
	5.8	0.8	0	0	10
	8.9	6.7	6.1	3.2	4
	9.4	2.6	2.4	0	5
	13.5	4.1	1.0	0.3	25
2	3.4	0.8	3.0	3.2	8
	3.5	5.3	2.4	0.4	23
	5.8	0.2	0.5	0.4	10
	8.9	1.5	1.6	0.1	3
	9.4	3.9	0.9	0.1	5
	13.5	11.6*	0.4	0	13

* F-ratio ≥ 7.7 (Significant at 0.05 level)

Table VI. BREMNAVSHIPYD vs NCEL (Infrared Transmission, IR-5)

i Value of R(i)	F-Ratio Values of R(i)			Coeff. of Variation (%)
	Lab	Batch	Lab-Batch	
3.4	5.1*	2.8	4.7*	18
3.5	6.2*	0.1	1.2	26
5.8	5.1*	0	0.4	18
8.9	0.5	6.1*	1.1	12
9.4	1.1	2.8	1.1	13
13.5	0	1.3	0	32

* F-ratio ≥ 4.7 (Significant at 0.05 level)

DISCUSSION

Laboratory differences, batch differences, and laboratory-batch differences were not significant for the IR-4 data. Variability due to sample preparation was consistently less for the IR-4 users.

The IR-5 data appears to be rather erratic. The sample preparation variability of NCEL and Cal-Colonial data ranged from 3 to 25 percent. The sample preparation variability of Bremerton Naval Shipyard's data ranged up to 62 percent in the data it provided NCEL.

The IR-4 and IR-5 appear to sense laboratory and/or batch differences when they occur. The variability is due to sample preparation; a better method of controlling sample preparation should be investigated.

INFRARED TRANSMISSION ABSORPTION DATA

Laboratory and Instrument	Batch No.	Peak Locations in Microns						
		3.4	3.5	5.8	7.9	8.9	9.4	13.5
NCEL	2	2.20	1.57	3.80	3.51	2.21	2.02	1.40
IR-5	2	2.06	1.55	4.43	3.70	2.33	2.11	1.42
	2	1.61	1.17	2.17	2.17	1.64	1.54	1.22
	2	1.76	1.33	2.54	2.26	1.69	1.54	1.22
	1	1.94	1.44	3.12	2.70	1.68	1.72	1.31
	1	2.17	1.72	3.72	3.52	2.11	1.93	1.13
	1	1.72	1.32	2.85	2.48	1.71	1.60	1.22
	1	1.83	1.35	2.81	2.32	1.72	1.59	1.23
CAL-COLONIAL	2	2.69	1.88	6.39	4.66	2.74	2.51	1.71
IR-5	2	2.60	1.73	5.96	4.91	2.83	2.56	1.68
	1	4.69	3.25	7.64	7.14	3.61	3.26	2.05
	1	2.95	1.87	6.83	4.33	2.51	2.18	1.49
BREMNAVSHIPYD	2	6.11	2.62	63.00	61.50	10.18	7.00	2.75
IR-5	2	3.06	1.86	27.71	13.36	4.47	3.54	1.94
	2	2.82	1.77	19.30	8.17	3.63	3.04	1.27
	2	3.28	2.00	31.66	5.67	4.39	3.58	2.00
	1	2.11	1.49	9.60	5.00	2.33	2.33	1.27
	1	3.45	2.05	46.50	33.75	5.38	4.00	2.09
	1	2.92	1.86	29.09	11.23	3.86	3.30	1.93
	1	4.48	2.20	186.00	42.00	6.88	5.00	2.37

Laboratory and Instrument	Batch No.	Peak Locations in Microns						
		3.4	3.5	5.8	7.9	8.9	9.4	13.5
PHILNAVSHIPYD	2	2.24	1.48	13.19	6.82	3.11	2.66	1.56
IR-4	2	2.47	1.66	9.40	5.46	2.75	2.42	1.54
	1	2.16	1.50	10.96	4.64	2.70	2.37	1.48
	1	2.08	1.46	7.33	4.35	2.31	2.07	1.41
PHILAIR	2	1.27	1.13	2.17	1.68	1.36	1.30	1.14
IR-4	2	1.30	1.14	2.04	1.69	1.39	1.34	1.15
	1	1.38	1.18	2.43	1.91	1.48	1.41	1.17
	1	1.38	1.18	2.66	1.94	1.51	1.45	1.20

Appendix B

STATISTICAL ANALYSIS OF ATR DATA

by G. E. Hayo

SUMMARY

Experiments were conducted at three laboratories to evaluate the Attenuated Total Reflectance (ATR) method of paint analysis. As in the infrared transmission study, the three laboratories — Cal-Colonial, Bremerton Naval Shipyard, and NCEL — all used the IR-5. Paint samples consisted of four replicates each of two batches from one manufacturer.

Laboratory differences, batch differences, laboratory-batch differences, and sample-preparation error (including instrument error) are evaluated in this appendix.

The ATR method was found to be more precise than the infrared transmission method; that is, one can expect better agreement between duplicate measurements under a given set of experimental conditions.

DATA ANALYSIS

The data, presented at the end of this appendix, were analyzed by analysis-of-variance procedures and by the same equation used in Appendix A for the same peak values.

Data comparisons are made of Cal-Colonial with NCEL and of Bremerton Naval Shipyard with NCEL to correlate with Appendix A. The three laboratories are also grouped together to obtain an indication of the overall precision with the ATR method.

Cal-Colonial. In the Cal-Colonial vs NCEL data, laboratory differences were significant for R(3.4), R(3.5), R(8.9), and R(13.5). Laboratory-batch differences were significant for R(3.4) and R(13.5). The variability of sample preparation ranged from 4 to 11 percent. Table VII lists the effects of different factors and the coefficient of variation for the i values.

Table VII. CAL-COLONIAL vs NCEL (ATR, IR-5)

i Value of R(i)	F-Ratio Values of R(i)			Coeff. of Variation (%)
	Lab	Batch	Lab-Batch	
3.4	320.5*	0.6	13.0*	6
3.5	43.3*	1.1	3.2	9
5.8	4.1	0.7	3.1	5
8.9	8.2*	2.2	0.4	4
9.4	1.2	2.7	0.2	5
13.5	11.5*	0	6.5*	11

* F-ratio ≥ 4.8 (Significant at 0.05 level)

Bremerton Naval Shipyard. In the Bremerton Naval Shipyard vs NCEL data, laboratory differences were significant for R(3.4), R(5.8), R(8.9), R(9.4) and R(13.5). Batch differences were significant for R(3.5), R(5.8), and R(8.9). Laboratory-batch differences were significant for R(8.9), R(9.4), and R(13.5). The variability of sample preparation ranged from 3 to 17 percent. Table VIII lists the effects of different factors and the coefficient of variation for the i values.

Table VIII. BREMNAVSHIPYD vs NCEL (ATR, IR-5)

i Value of R(i)	F-Ratio Values of R(i)			Coeff. of Variation (%)
	Lab	Batch	Lab-Batch	
3.4	38.2*	0.6	0.6	17
3.5	1.2	6.7*	0	9
5.8	49.1*	9.5*	0.6	6
8.9	22.1*	5.3*	27.4*	3
9.4	20.7*	0.7	7.9*	6
13.5	5.3*	0	9.0*	9

*F-ratio ≥ 4.8 (Significant at 0.05 level)

Cal-Colonial, Bremerton Naval Shipyard, NCEL. In the Cal-Colonial - Bremerton Naval Shipyard - NCEL grouped data, laboratory differences were significant for all R(i). Batch differences were significant for R(5.8). Laboratory-batch differences were significant for R(8.9), R(9.4), and R(13.5). The variability of sample preparation ranged from 4 to 16 percent. Table IX lists the effects of different factors and the coefficient of variation for the i values.

Table IX. CAL-COLONIAL - BREMNAVSHIPYD - NCEL (ATR, IR-5)

i Value of R(i)	F-Ratio Values of R(i)			Coeff. of Variation (%)
	Lab	Batch	Lab-Batch	
3.4	36.6*	0	1.1	16
3.5	21.4*	3.5	1.8	10
5.8	26.6*	4.8**	2.8	6
8.9	22.7*	1.2	9.6*	4
9.4	20.8*	0	5.3*	5
13.5	7.8*	1.1	6.0*	10

* F-ratio ≥ 3.6 (Significant at 0.05 level)

** F-ratio ≥ 4.5 (Significant at 0.05 level)

DISCUSSION

The ATR method reduces the variability between duplicate measurements as much as 72 percent below that obtained by the infrared transmission method. This variability (sample-preparation error) includes errors in sample size, instrument resolution, instrument operation, paint sample, and other factors the experimenter cannot control. The increased precision of the ATR method also increases its sensitivity to laboratory, batch, and laboratory-batch differences. This requires the experimenter to fix differences that are acceptable before performing the paint analysis. An example of a 10-percent difference is

Given:

Sample 1 Peak 3.4 = 1.41, Peak 7.9 = 2.00

Sample 2 Peak 3.4 = 1.46, Peak 7.9 = 2.00

Calculate:

Sample 1 $R(3.4) = \frac{\ln(1.41)}{\ln(2.00)} = 0.50$

Sample 2 $R(3.4) = \frac{\ln(1.46)}{\ln(2.00)} = 0.55$

$$\text{Difference} = 100\left(\frac{0.55 - 0.50}{0.5}\right) = 10\%$$

ATR ABSORPTION DATA

Laboratory and Instrument	Batch No.	Peak Locations in Microns						
		3.4	3.5	5.8	7.9	8.9	9.4	13.5
NCEL	1	1.45	1.23	2.28	1.96	1.86	1.65	1.47
IR-5	1	1.46	1.25	2.33	1.92	1.84	1.66	1.46
	1	1.45	1.26	2.29	1.93	1.86	1.66	1.48
	1	1.43	1.25	2.31	1.93	1.97	1.66	1.51
CAL-COLONIAL	1	1.24	1.18	2.28	1.85	1.89	1.64	1.26
IR-5	1	1.28	1.20	2.23	1.85	1.84	1.60	1.26
	1	1.27	1.18	2.22	1.94	1.89	1.67	1.39
	1	1.26	1.19	2.38	2.10	1.99	1.75	1.41
BREMNAVSHIPYD	1	1.29	1.27	2.07	2.01	1.74	1.48	1.36
IR-5	1	1.28	1.27	1.94	1.94	1.72	1.53	1.34
	1	1.21	1.18	1.76	1.94	1.73	1.52	1.36
	1	1.32	1.29	2.31	2.19	1.81	1.63	1.49

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