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Fourier transform infrared (FTIR) authentication and batch-to-batch consistency for different types of paints using benchtop and handheld FTIR spectrophotometers for oil and gas industry

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Abstract

Standardization of Fourier transform infrared (FTIR) fingerprint region for paints and assessment on the reproducibility using different spectrophotometers were investigated. While selective fingerprint regions may be confusing for technicians/analysts who are non-chemists, we attempt to generalize these regions (e.g., 1300–1000 cm^{-1} for Epoxy part A and 1400–1000 cm^{-1} for Epoxy part B) by choosing a universal region (2000–900 cm^{-1}) that works for different paints. Comparison result using a paired student *t*-test shows that the degree of similarity (r) values from the studied regions are not statistically different. The paint fails the screening analysis occasionally on-site when analyzed using handheld FTIR due to the higher level of noise that gives low r values ($r < 0.900 \pm 0.002$). The same samples were analyzed using a benchtop FTIR and the r values are above 0.900 ± 0.002. While the screening may lead to a false rejection of the sample on-site, there could be occurrence of false acceptance. The on-site screening of EPZ part A with different formulations, for instance, shows that the r values over the entire IR spectrum are above 0.900 ± 0.002 when analyzed using handheld FTIR. After the samples were analyzed using the benchtop, the r values fall below 0.900 ± 0.002.

KEYWORDS

coatings, FTIR analysis, FTIR fingerprint region, FTIR spectrophotometer, paints

1 | INTRODUCTION

According to NACE International, the cost of global corrosion control in 2013 exceeds US$ 2.5 trillion, which was estimated at roughly 3.4% of a country's growth domestic product.[1] Due to the high cost of corrosion maintenance and frequent occurrence of premature coating failure at offshore facilities, there was an urgent call within the same year for the provision of *Coating Fingerprint Certificate* (an equivalent to mill certificate for metal) in Malaysia using Fourier transform infrared (FTIR) authentication analysis as an additional tool for existing quality assurance/quality control (QA/QC) procedures. Procedures concerning the estimation of *degree of similarity* (r) for batch-to-batch wet paints were successfully documented in the Institute of Materials, Malaysia (IMM) standard FP01:2019.[2] This standard was adapted and improved from the existing international standards that are related to FTIR fingerprinting of wet paints in the paint mixing tanks such as ISO 12944-9:2018,[3] ASTM D2621-87(2016),[4] and ASTM D7588-11(2018).[5] This is due to insufficient details and guidelines about the interpretation of FTIR spectra by means of the estimation of *r* values for the mentioned standards.

According to IMM FP01:2019 when the offshore structures that require painting exceed 5000 m^2 surface area, the paint manufacturer is required to submit FTIR Reference spectra of wet paints to the facility owner during the process of bidding or tender qualification. A Reference spectrum can be generated from an average of nine FTIR spectra of the said paint system consisting of three replicates from three different locations in the mixing tank of paint production (Top, Middle, and Bottom). One prime requirement is that the *r* values obtained by comparing the sample spectra to Reference spectrum shall be above 0.900 ± 0.002 for wet paints. This acceptance criterion is applicable to the complete region of FTIR spectra from 4000–700 cm^{-1} and the specific fingerprint regions depending on the nature of functional groups, that present for a particular paint (e.g., 1300–1000 cm^{-1} and 900–700 cm^{-1} for Epoxy part A, 1400–1000 cm^{-1} for Epoxy part B, etc.). Chan et al. proposed this procedure for the detection of homogeneity at different locations of the mixing tank for different paints[6] and for the distinction between normal-grade and premier-grade raw materials used for epoxy paints.[7] Different FTIR manufacturers and software used for the estimation of *r* values by *normal* and *high sensitivity compare* function were also investigated.[8] The sensitivity differs slightly according to different numerical approaches which are pre-installed in the software. Li et al. (2007) showed different numerical methods used to compare the FTIR spectra of fresh and aged petroleum oil. They demonstrated that the *compare* function within the entire IR region and specific fingerprint region can be used to do fresh and aged sample matching.[9] The use of *compare* function was demonstrated also by Jiang et al. (2011). The batch-to-batch detection for any structural changes that occur in protein therapeutics was confirmed using FTIR *compare* function.[10] It was determined the protein secondary structure to be reproducible if the spectral similarity is greater than 90%. This strategy provides good guidance and information for spectroscopists, who work not only in the fields of corrosion, but also polymeric products, food, pharmaceutical, forensics, and other QA/QC-related industries by employing a practical protocol for the industry practice, in this case, through estimation of degree of similarity between sample and "qualified" Reference spectra using mathematical *compare* algorithm.[11–15] Oulahal et al. (2009) revealed that high *r* values between the FTIR spectra of unused and cleaned by brushing wooden shelves as compared with that of non-cleaned wooden surface[16] but they did not elaborate further on the *compare* algorithm used. Some web-based articles in AZoMaterials featured the application of FTIR for batch-to-batch testing in the food and drink industry,[17] in particular of alcohol, olive oil, milk powder, and meat products.[18] Other research utilizing FTIR for the detection of counterfeit paintings,[19] pathogens,[20] and drugs[21,22] have also been reported elsewhere.

For polymeric paints, *high sensitivity compare* numerical method with respect to variations of *x*-vectors and *y*-vectors (wavenumber and absorbance, respectively) is of the interest in estimation of degree of similarity. This *compare* function is quite sensitive to detect minute changes and differentiate if the supplied paint is the same as promised or whether it suffers from paint deterioration or compromised by different paint formulations. In general, these software-incorporated *compare* algorithms possess a basic concept that utilized the numerical methods to compute the degree of similarity between sample and Reference spectra by employing all of the spectral information, for example, band positions, absorbance height, width, and shape of the absorbance bands into the calculation.[23] Software A applies the squared derivative algorithm,[24,25] Software C uses correlation coefficient algorithm,[23,26–28] and Software D uses Euclidean distance algorithm.[29–31] Software A/C/D are referred in "Materials and Method." The reproducibility and reliability of different *compare* algorithms from different FTIR software was conducted in the previous studies and were analytically proven by using statistical analysis if the good and standard practices of spectral collection are being followed.[9,32,33] Statistics show the degree of similarities estimated by these *compare* algorithms are not statistically different between one another. However, two issues may arise during the implementation of *Coating Fingerprint Certificate* in local oil and gas industry. The former refers to the accuracy of measurement in which the certified paint quality controllers should report the *r* values with appropriate number of significant figures. The latter issue may arise from the chosen fingerprint regions that are specific to selective paints. During pilot project of the *Coating Fingerprint* certification at job site as well as certification training for certified personnel, we realized that the coating inspectors or the analysts tend to make mistake while reporting the FTIR results for the fingerprint regions, especially if those inspectors have no chemistry-related or science-related background. When a painting work contract is awarded to a paint manufacturer/contractor, the paint will be supplied to the job site and further verified by the contractor/owner before paint application on substrate. The contractor/owner (who appoints coating inspectors) may be using a mobile or handheld FTIR for quick

TABLE 1 Chemical formulation and mixing ratio of three-coat maintenance paints for offshore steel structures

Paint manufacturer	Coating layer	Part (A:B) vol. ratio	2-pack	Chemical compounds
Paint Manufacturer 2	Primer epoxy zinc (EPZ)	3:1	Epoxy resin (Part A)	• Zinc powder (50–75 wt%) • Epoxy resin (M_{w} 700–1200) (10 wt%) • Xylene (10 wt%) • 1-methoxy-2-propanol (3 wt%) • Ethylbenzene (3 wt%) • Zinc oxide (3 wt%)
			Amine (Part B)	• Xylene (25–41 wt%) • 1-methoxy-2-propanol (10–25 wt%) • Ethylbenzene (10 wt%) • Solvent naphtha (petroleum), light aroma (3 wt%) • Fatty acids, C-18-unsaturated, trimers, compounds with oleylamine (3 wt%) • Amines, polyethylenepoly-, triethylenetetramine fraction (1 wt%) • 2,2′-iminodiethylamine (1 wt%)
	Middle coat epoxy	4:1	Epoxy resin (Part A)	• Xylene (10 wt%) • Bisphenol-A-(epichlorohydrin); high M_{w} epoxy resin (M_{w} 700–1200) (5 wt%) • Bisphenol-A-(epichlorohydrin); low M_{w} epoxy resin ($M_{\mathrm{w}} \leq 700$) (10–25 w%) • Benzyl alcohol (3 wt%) • 1-methoxy-2-propanol (3 wt%) • Hydrocarbons, C9-unsaturated, polymerized (3 wt%)
			Amine (Part B)	• Xylene (10–25 wt%) • Butan-1-ol (10 wt%) • Ethylbenzene (10 wt%) • 2,4,6-tris(dimethylaminomethyl)phenol (3 wt%)
	Top coat polyurethane (PU)	10:1	Polyacrylate (Part A)	• Xylene (10–25 wt%) • *n*-butyl acetate (10 wt%) • Solvent naphtha (petroleum), light aroma (5 wt%) • Ethylbenzene (5 wt%) • 2-propenoic acid, 2-methyl-, 2-(dimethylamino)ethyl ester, polymer with butyl 2-propenoate, comps. With polyethylene glycol hydrogen maleate (3 wt%) • *n*-butyl methacrylate (1 wt%) • Bis(1,2,2,6,6,-pentamethyl-4-piperidyl) sebacate (<0.3 wt%)
			Isocyanate (Part B)	• Hexamethylene diisocyanate, oligomers (75–90 wt%) • *n*-butyl acetate (10 wt%) • Solvent naphtha (petroleum), light aroma (10 wt%) • Hexamethylene diisocyanate (0.3 wt%)
Paint Manufacturer 4	Primer epoxy zinc (EPZ)	4:1	Epoxy resin (Part A)	• Zinc powder, stabilized (30–60 wt%) • Talc, not containing asbestiform fibers (10–16 wt%) • Poly(bisphenol A-*co*-epichlorohydrin) (10–30 wt%) • Xylene (3–5 wt%), mixture of isomers • Solvent naphtha (petroleum), light aromatic (1–3.3 wt%) • 1-methoxy-2-propanol (1–3 wt%) • Zinc oxide fume, dust (1–3 wt%)

(Continues)

TABLE 1 (Continued)

Paint manufacturer	Coating layer	Part (A:B) vol. ratio	2-pack	Chemical compounds
			Amine (Part B)	• Fatty acids, C18-unsatd, dimers, oligomeric reaction products with tall-oil fatty acids and triethylenetetramine (10–30 wt%) • Xylene (10–14 wt%) • Isobutyl alcohol (10–20 wt%) • Benzyl alcohol (10–14 wt%) • 2,4,6-tris(dimethylaminomethyl)phenol (1–4.6 wt%) • Ethyl benzene (1–3 wt%) • Amines, polyethylenepoly-, triethylenetetramine fraction (<1.8 wt%)
	Middle coat epoxy	4:1	Epoxy resin (Part A)	• Xylene (5–9.4 wt%) • Bisphenol-A-(epichlorohydrin); high M_w epoxy resin (M_w 700–1200) (5–10 wt%) • Bisphenol-A-(epichlorohydrin); low M_w epoxy resin ($M_w \leq 700$) (5–10 wt%) • Benzyl alcohol (3–5 wt%) • 4-nonylphenol, branched (0.1–2.5 wt%)
			Amine (Part B)	• Fatty acids, C18-unsatd., dimers, reaction products with polyethylenepolyamines (30–60 wt%) • Nonylphenol (10–30 wt%) • Ethyl benzene (10–18 wt%) • Isobutyl alcohol (10–20 wt%) • Xylene (5–10 wt%) • 2,4,6-tris(dimethylaminomethyl)phenol (5–10 wt%) • 3,6-diazaoctanethylenediamin (1–3 wt%) • *p*-nonylphenol (<1 wt%)
	Top coat polyurethane (PU)	88:12	Polyacrylate (Part A)	• Xylene (10–30 wt%) • *n*-butyl acetate (5–10 wt%) • Ethyl benzene (3–5 wt%) • Talc, not containing asbestiform fibers (3–5 wt%) • Bis(1,2,2,6,6,-pentamethyl-4-piperidyl) sebacate (<1 wt%) • Toluene (<1 wt%)
			Isocyanate (Part B)	• Poly(hexamethylene diisocyanate) (60–78 wt%) • Xylene (5.0–7.1 wt%) • Ethyl benzene (5–7 wt%) • Hexamethylene diisocyanate (<1 wt%)

Abbreviation: M_w, mass average molar mass.

"screening" and subsequently perform *compare* function of the FTIR software with the existing Reference spectrum that had been stored earlier during bidding or tender qualification. Shall the results of the comparison fail, a smaller sample container (retained sample) will be sent to third-party laboratory for "verification" analysis. The third-party laboratories shall use benchtop FTIR. As such, this study is aimed to address the discrepancy of significant figures of *r* values for acceptance or rejection of paint samples (between three and four significant figures) and to judge whether a universal fingerprint region can be used for different types of paints by using statistical test. At the end of this study, the spectra of fresh paint from different FTIR spectrophotometers (handheld) are compared against a standard laboratory-grade benchtop FTIR spectrophotometer. The comparison is made between the sample spectrum and the Reference spectrum that is generated from benchtop FTIR. We hope to answer the questions of "should the handheld analytical FTIR spectrophotometer be relied solely on verification of the batch-to-batch consistency of the paints at the job sites?"

2 | MATERIALS AND METHOD

Three-coat maintenance paints for offshore steel structures consisting of epoxy zinc rich paint (coded as EPZ), epoxy paint (coded as Epoxy), and polyurethane paint (coded as PU) were kindly provided by two paint manufacturers, Jotun Paints (Malaysia) Sdn Bhd and PPG Performance Coatings (Malaysia) Sdn Bhd. The samples were coded as follow: SampleP_x_A _BzCY ["Sample" refers to either EPZ, Epoxy or PU; P refers to paint manufacturer (coded as "2" or "4"; the assigned coding to individual paint manufacturer was made randomly); subscript x refers to the software of FTIR being used (coded as "A" or "C" or "D"); A represents part "A" or part "B" of 2-pack paints; Bz represents batch number "z" of paints; C represents location of the mixing tank where the sample is collected into the container (T for Top, M for Middle, and B for Bottom); and finally Y represents the replicate number during FTIR analysis ("1" or "2" or "3")]. The chemical formulation and mixing ratio of these paints to form dried coatings are summarized in Table 1. Chemical components in Table 1 were mixed in at least 1000-L mixing tank (depending on the required quantity) at room temperature for at least 2 hours. The term "at least 2 hours" was used because different paint formulations had different mixing time. Prior to FTIR scanning, individual paint sample was stirred homogeneously. The stirring time varied depending on the viscosity of paint sample. Higher viscosity requires longer stirring time, that is, within 1–5 min. For each analysis, the sample in the paint container was immediately closed after sample withdrawal from the paint container in order to minimize atmospheric reactions.

Three replicates of homogeneous paint sample after stirring were analyzed using four FTIR spectrophotometers: in-house laboratory used Nicolet iS10 (Thermo Scientific, Madison, Wisconsin), on-site screening used 4300 Handheld (Agilent Technologies Inc., Santa Clara, California) and third-party laboratory used Nicolet iS5 (Thermo Scientific, Madison, Wisconsin) and Spectrum Two (Perkin Elmer, Waltham, Massachusetts). The software used for respective FTIRs is labeled as Software A, Software D, and Software C. All spectrophotometers were equipped with ATR diamond crystal. The analysis ranged from 4000 to 700 cm^{-1} at a resolution of 4 cm^{-1} by averaging 32 scans.

Nine FTIR spectra (three each from Top, Middle, and Bottom of the mixing tank of paint production) of fresh paint sample were averaged and used as a Reference spectrum. The r value was obtained by comparing the spectra of retained sample to that of Reference spectrum following the *high sensitivity compare* algorithm for different FTIR software. The expressions of *high sensitivity compare* algorithm for different FTIR software are described as follow; (a) Software A uses squared derivative algorithm that applies an exponential scaling function on both x-(wavenumber) and y-(absorbance) vectors of the sample and Reference spectra,[24,25] (b) Software C uses correlation coefficient algorithm that applies a specific weighting factor of exponential function on both x-(wavenumber) and y-(absorbance) vectors of the sample and Reference spectra,[23,26–28] and (c) Software D uses Euclidean distance algorithm that compute a normalized least squares of x-(wavenumber) and y-(absorbance) vectors of the Reference to the sample spectra.[29–31] In general, the above-mentioned algorithms described when r value is close to 1, it signifies a complete spectrum matching. It had been initially proposed that the r value of at least 0.900 ± 0.002 and above is the criterion in which the supplied paint to job on-site is accepted. The acceptance criterion of $r \geq 0.900 \pm 0.002$ was defined based on the considerations of the sample size, types of sample, and sample uncertainties.[34–36]

Paired student t-test was used to compare the difference between (a) three and four significant figures of the r values, as well as (b) the choice of fingerprint region for different paints.[37–39] In this test, two null hypotheses were created. The former null hypothesis ($H_{0[\mathrm{i}]}$) assumes that the average difference of r values between three significant figures and four significant figures is zero ($H_{0[\mathrm{i}]} = 0$). The latter null hypothesis ($H_{0[\mathrm{ii}]}$) assumes that the average difference of r values between selectively chosen fingerprint region and universal fingerprint region is zero ($H_{0[\mathrm{ii}]} = 0$). The alternate hypothesis is the value other than zero $H_1 \neq 0$. The significance level used in this assumption test is $p = 0.05$. The t-statistic of a given set of data is calculated according to the following equation[37–39]:

$$t = \frac{\overline{D} - \mu_{\mathrm{D}}}{s_{\overline{\mathrm{D}}}/\sqrt{n}} \tag{1}$$

where, $s_{\overline{\mathrm{D}}}/\sqrt{n} = \mathrm{SE}(\overline{D})$ is the standard error of average differences ($\overline{D}$), μ_{D} is the population mean of different values, n is sample size, and $s_{\overline{\mathrm{D}}}$ is the standard deviation of the differences. If the t-statistic value is greater than t-critical (t-critical can be found in the statistic table using the degree of freedom and pre-selected level of significance) and p-value that is greater than 0.05, we reject the null hypothesis.

3 | RESULTS AND DISCUSSION

3.1 | Monitoring paint consistency as per IMM FP01:2019

Figure 1 shows the spectra comparison between EPZ2, Epoxy2, and PU2, all in (A) part A and (B) part

B. Obvious absorption bands of EPZ2 part A can be assigned to the stretching vibration of C–O–C (1100–1300 cm^{-1}), =C– of benzene (765 cm^{-1}) and –OH (3200–3650 cm^{-1}) while the absorption bands of part B paint can be assigned to C≡N (2268 and 1039 cm^{-1}), –NH (1245 cm^{-1}), and =C– (765 cm^{-1}). The functional groups of EPZ2 along with other paint system, for example, Epoxy2 and PU2 correspond well to the nature of chemicals used in paint formulation. For instance, C–O–C is of bisphenol A, =C– is of xylene, and C–N is of 2,4,6-tris(dimethylaminomethyl)phenol. Further analyses about the assignment bands are not elaborated further, however it should be noted that any changes made in the paint formulation (i.e., chemical type, compositions, and purity) will result in different absorption spectra. The differences between two paints with different formulations, if any, have been successfully demonstrated by determining the r values using *high sensitivity compare* function that is available in most FTIR software.

Table 2 summarizes the r values that were obtained using *high sensitivity compare* function within the entire wavenumber region (4000–700 cm^{-1}) and specific fingerprint regions following IMM FP01:2019. The fingerprint region was selectively chosen based on the active functional groups that are present mainly in the paint formulation. For instance, a region of 1300–1000 cm^{-1} in EPZ2 part A was selected due to the presence of carbonyl in the bisphenol A. In EPZ2 part B however, a region of 1800–1000 cm^{-1} was selected due to the presence of amine-based components such as tris(dimethylaminomethyl) phenol and triethylenetetramine. Estimation of the r values from different locations of the mixing tanks shows that all paints have the values above the acceptance criterion ($r \geq 0.900 \pm 0.002$ for qualification of epoxy paints supplied to the oil and gas industry[6]). This result suggests homogeneity of the paints in the mixing tanks.

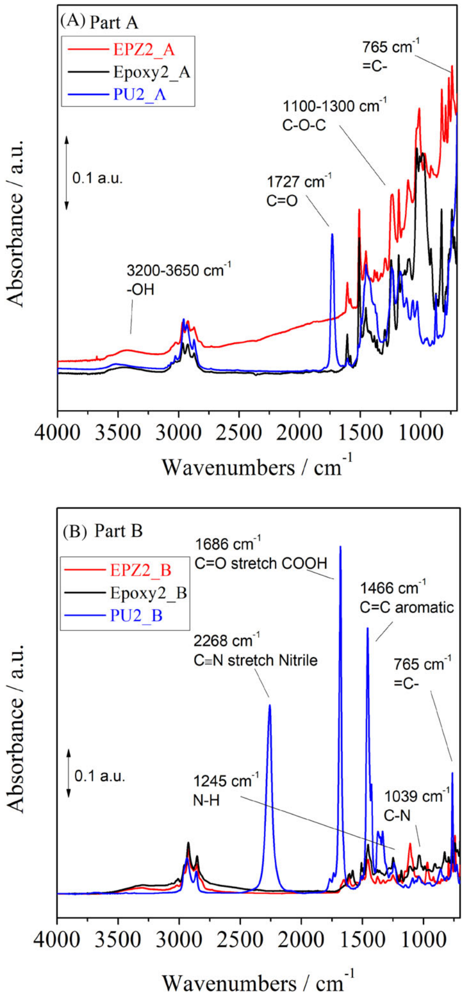

FIGURE 1 FTIR spectra of freshly formulated EPZ2, Epoxy2, and PU2 paints: (A) Part A and (B) Part B. The spectra were generated using benchtop FTIR, where in-house laboratory is referred in subsequent sections

3.2 | Significant figures

In the past, different significant figures of r values had been reported. They include two significant figures,[40] three significant figures,[6,41–44] and four significant figures.[6–8,45] The accuracy measurement of r values is important and therefore is validated by comparing the r values between three and four significant figures using the paired student t-test. As shown in Tables 3 and 4, the results from FTIR *high sensitivity compare* function of EPZ part A shows (a) larger values of t-statistic as compared with that of t-critical and (b) p-values that are above 0.05. These indicate that the r values with four significant figures are not statistically different than those with three significant figures after round up. For practicality, three significant figures for r values are strongly encouraged for the certified coating fingerprint quality controllers to assess the batch-to-batch paint consistency and for authentication of paints. If the average r value of $\overline{D}$, $S_{\overline{D}}$, or SE($\overline{D}$) were 0.000, there will be an error in calculation. The significant figure has to be increased until a nonzero number is obtained. Such example can be found on the significant figures used in $\overline{D}$ and SE($\overline{D}$) values shown in Tables 4–6. Other paint samples (EPZ part B; Epoxy part A and part B; and PU part A and part B) follow similar observation (no significant difference between three and four significant figures) to that of EPZ part A. Discussion about the number significant figures of the r values henceforth is limited to three significant figures.

TABLE 2 r values of fresh maintenance paint analyzed using *high sensitivity compare* function for the entire region (4000–700 cm^{-a}) and specific fingerprint regions

	Entire region	**Specific fingerprint regions**[a]					
Sample code	***r* (4000–700 cm^{-1})**	***r* (1300–1000 cm^{-1})**	***r* (900-700 cm^{-1})**	***r* (1800–1600 cm^{-1})**	***r* (1400–1000 cm^{-1})**	***r* (2500–2000 cm^{-1})**	**Reference spectrum**[b]
EPZ2$_{A}$_A_B1T1	0.9801	0.9839	0.9795				EPZ2$_{A}$_A
EPZ2$_{A}$_A_B1T2	0.9839	0.9818	0.9894				
EPZ2$_{A}$_A_B1T3	0.9839	0.9823	0.9895				
EPZ2$_{A}$_A_B1M1	0.9892	0.9842	0.9960				
EPZ2$_{A}$_A_B1M2	0.9885	0.9813	0.9969				
EPZ2$_{A}$_A_B1M3	0.9881	0.9932	0.9912				
EPZ2$_{A}$_A_B1B1	0.9944	0.9982	0.9961				
EPZ2$_{A}$_A_B1B2	0.9942	0.9985	0.9962				
EPZ2$_{A}$_A_B1B3	0.9919	0.9973	0.9972				
EPZ2$_{A}$_B_B1T1	0.9890				0.9953		EPZ2$_{A}$_B
EPZ2$_{A}$_B_B1T2	0.9891				0.9955		
EPZ2$_{A}$_B_B1T3	0.9890				0.9948		
EPZ2$_{A}$_B_B1M1	0.9904				0.9939		
EPZ2$_{A}$_B_B1M2	0.9899				0.9954		
EPZ2$_{A}$_B_B1M3	0.9904				0.9943		
EPZ2$_{A}$_B_B1B1	0.9903				0.9949		
EPZ2$_{A}$_B_B1B2	0.9915				0.9956		
EPZ2$_{A}$_B_B1B3	0.9883				0.9917		
Epoxy2$_{A}$_A_B1T1	0.9862	0.9883	0.9871				Epoxy2$_{A}$_A
Epoxy2$_{A}$_A_B1T2	0.9915	0.9921	0.9921				
Epoxy2$_{A}$_A_B1T3	0.9904	0.9934	0.9940				
Epoxy2$_{A}$_A_B1M1	0.9961	0.9970	0.9967				
Epoxy2$_{A}$_A_B1M2	0.9950	0.9964	0.9966				
Epoxy2$_{A}$_A_B1M3	0.9930	0.9958	0.9949				
Epoxy2$_{A}$_A_B1B1	0.9968	0.9992	0.9986				
Epoxy2$_{A}$_A_B1B2	0.9965	0.9983	0.9981				
Epoxy2$_{A}$_A_B1B3	0.9964	0.9978	0.9973				
Epoxy2$_{A}$_B_B1T1	0.9911				0.9955		Epoxy2$_{A}$_B
Epoxy2$_{A}$_B_B1T2	0.9876				0.9942		
Epoxy2$_{A}$_B_B1T3	0.9871				0.9947		
Epoxy2$_{A}$_B_B1M1	0.9715				0.9727		
Epoxy2$_{A}$_B_B1M2	0.9791				0.9918		
Epoxy2$_{A}$_B_B1M3	0.9820				0.9944		
Epoxy2$_{A}$_B_B1B1	0.9800				0.9907		
Epoxy2$_{A}$_B_B1B2	0.9828				0.9921		
Epoxy2$_{A}$_B_B1B3	0.9621				0.9853		
PU2$_{A}$_A_B1T1	0.9798			0.9990			PU2$_{A}$_A
PU2$_{A}$_A_B1T2	0.9732			0.9990			
PU2$_{A}$_A_B1T3	0.9670			0.9930			
PU2$_{A}$_A_B1M1	0.9925			0.9989			

(Continues)

TABLE 2 (Continued)

	Entire region	Specific fingerprint regions[a]					
Sample code	***r* (4000–700 cm^{-1})**	***r* (1300–1000 cm^{-1})**	***r* (900–700 cm^{-1})**	***r* (1800–1600 cm^{-1})**	***r* (1400–1000 cm^{-1})**	***r* (2500–2000 cm^{-1})**	**Reference spectrum[b]**
PU2$_A$_A_B1M2	0.9935			0.9991			
PU2$_A$_A_B1M3	0.9940			0.9990			
PU2$_A$_A_B1B1	0.9960			0.9974			
PU2$_A$_A_B1B2	0.9740			0.9972			
PU2$_A$_A_B1B3	0.9577			0.9992			
PU2$_A$_B_B1T1	0.9992					0.9975	PU2$_A$_B
PU2$_A$_B_B1T2	0.9932					0.9964	
PU2$_A$_B_B1T3	0.9987					0.9974	
PU2$_A$_B_B1M1	0.9991					0.9974	
PU2$_A$_B_B1M2	0.9977					0.9976	
PU2$_A$_B_B1M3	0.9993					0.9970	
PU2$_A$_B_B1B1	0.9975					0.9981	
PU2$_A$_B_B1B2	0.9989					0.9974	
PU2$_A$_B_B1B3	0.9987					0.9979	

[a] Specific FTIR fingerprint regions based on the functional groups of individual paint.

[b] Reference spectrum was generated by averaging all the nine spectra (three replicates each from Top, Middle, and Bottom of mixing tank) of individual paint system.

3.3 | Selective vs. universal FTIR fingerprint regions

Table 2 describes the quality control of two-pack three-layer maintenance paints according to the method described in IMM FP01:2019.[2] For simplification to the previously used FTIR regions that are comprised of the entire range of FTIR spectra (4000–700 cm^{-1}) and "selective" or "specific" fingerprint regions (that depends on the type of functional groups in the paint), Table 7 describes how a "universal" choice of fingerprint region regardless of functional groups in the paint can be used to replace the predecessor approach. Results show that all the *r* values of part A: EPZ2, Epoxy2, and PU2 from the universal fingerprint region (2000–900 cm^{-1}) are well above 0.900 ± 0.002. These values are similar to that obtained from the specific fingerprint regions (1650–900 cm^{-1} for EPZ2 and Epoxy2; and 1800–1300 cm^{-1} for PU2). The specific fingerprint regions chosen for comparison (1650–900 cm^{-1} for EPZ2 and Epoxy2; 1800–1300 cm^{-1} for PU2) are slightly different than those (1300–1000 cm^{-1} and 900–700 cm^{-1} for EPZ2/Epoxy2; and 1800–1600 cm^{-1} for PU2 according to IMM FP01:2019[2]) in Table 2. The difference is due to the selection of specific fingerprint regions (Table 2) that ought to overlap with the universal fingerprint region (Table 7) in order to avoid any biases (a bias that involves the data outside selective population and/or FTIR fingerprint regions) during paired student *t*-test analysis.

Based on the average difference ($\overline{D}$) between the *r* values from specific and universal fingerprint regions shown in Table 8, the calculated *t*-statistic values of individual paint system are below *t*-critical [e.g., $1.000 < 2.306$ (*t*-critical) in the case of two-pack EPZ2 and PU2, and

TABLE 3 Hypothesis test of the average difference ($\overline{D}$) between the *r* values of three and four significant figures in EPZ2 Part A sample

EPZ2$_A$_A		
r_{4sf} (900–1650 cm^{-1})	r_{3sf} (900–1650 cm^{-1})	D (r_{3sf}–r_{4sf})
0.9870	0.987	0.0000
0.9887	0.989	0.0003
0.9896	0.990	0.0004
0.9954	0.995	−0.0004
0.9902	0.990	−0.0002
0.9883	0.988	−0.0003
0.9941	0.994	−0.0001
0.9982	0.998	−0.0002
0.9978	0.998	0.0002

Abbreviations: 4sf, four significant figures; 3sf, three significant figures.

TABLE 4 *t*-statistic of EPZ sample from Paint Manufacturers 2 and 4

	EPZ2$_{A}$_A	**EPZ2$_{A}$_B**	**EPZ4$_{A}$_A**	**EPZ4$_{A}$_B**
$\overline{D}\ (r_{3sf}-r_{4sf})$	−0.00003	0.00003	0.00001	−0.00004
$S_{\overline{D}}$	0.00028	0.00031	0.00028	0.00024
$SE(\overline{D}) = s_{\overline{D}}/\sqrt{n}$	0.00009	0.00010	0.00009	0.00008
n	9	9	9	9
$f = n - 1$	8	8	8	8
t-statistic	−0.359	0.324	0.121	−0.567
|*t*-statistic|	0.359	0.324	0.121	0.567
t-critical	2.306	2.306	2.306	2.306
p-value	0.73	0.75	0.91	0.59

Note: The n is the size of $\overline{D}$; f is the degree of freedom [defined by $(n - 1)$ as it only comprises of the mean of one sample]; p represents the level of significance and was defined based on the confidence level from cross-analysis of the r values across different FTIR spectrophotometers and different Software.

TABLE 5 *t*-statistic of epoxy sample from Paint Manufacturers 2 and 4

	Epoxy2$_{A}$_A	**Epoxy2$_{A}$_B**	**Epoxy4$_{A}$_A**	**Epoxy4$_{A}$_B**
$\overline{D}\ (r_{3sf}-r_{4sf})$	0.00017	0.00013	0.00004	0.00007
$S_{\overline{D}}$	0.00029	0.00041	0.00034	0.00040
$SE(\overline{D}) = s_{\overline{D}}/\sqrt{n}$	0.00010	0.00014	0.00011	0.00013
n	9	9	9	9
$f = n - 1$	8	8	8	8
t-statistic	1.741	0.970	0.389	0.504
|*t*-statistic|	1.741	0.970	0.389	0.504
t-critical	2.306	2.306	2.306	2.306
p-value	0.12	0.36	0.71	0.63

TABLE 6 *t*-statistic of PU sample from Paint Manufacturers 2 and 4

	PU2$_{A}$_A	**PU2$_{A}$_B**	**PU4$_{A}$_A**	**PU4$_{A}$_B**
$\overline{D}\ (r_{3sf}-r_{4sf})$	−0.00008	−0.00004	−0.00013	−0.00019
$S_{\overline{D}}$	0.00022	0.00033	0.00032	0.00028
$SE(\overline{D}) = s_{\overline{D}}/\sqrt{n}$	0.00007	0.00011	0.00011	0.00009
n	9	9	9	9
$f = n - 1$	8	8	8	8
t-statistic	−1.077	−0.406	−1.265	−2.021
|*t*-statistic|	1.077	0.406	1.265	2.021
t-critical	2.306	2.306	2.306	2.306
p-value	0.31	0.70	0.24	0.08

$2.000 < 2.306$ (*t*-critical) for two-pack Epoxy2]. In addition, all the *p*-values from paired student *t*-test are above 0.05. Both fulfilled criteria (*t*-statistic < *t*-critical and $p > 0.05$) mean that the null hypothesis H_0 shall be accepted, that there is no significant difference between the r values extracted from "selective" and "universal" fingerprint regions. Thus a replacement of selective fingerprint regions with universal fingerprint region seems reasonable and practical for the analysts. Paired student *t*-test using the entire data ($n = 108$) involving the r values from two-pack EPZ2, Epoxy2, and PU2 also confirms that we could accept the null hypothesis H_0.

TABLE 7 Comparison between "specific" FTIR fingerprint regions as per IMM FP01:2019 and "universal" fingerprint regions in Part A paint samples

	Fingerprint region according to functional group of individual paint		**Proposed universal region**	
Sample code	r **(1650–900 cm^{-1})**	r **(1800–1300 cm^{-1})**	r **(2000–900 cm^{-1})**	D **($r_{\text{universal}}-r_{\text{specific}}$)**
EPZ2$_{A}$_A_B1T1	0.987		0.985	−0.002
EPZ2$_{A}$_A_B1T2	0.989		0.987	−0.002
EPZ2$_{A}$_A_B1T3	0.990		0.988	−0.002
EPZ2$_{A}$_A_B1M1	0.995		0.989	−0.006
EPZ2$_{A}$_A_B1M2	0.990		0.987	−0.003
EPZ2$_{A}$_A_B1M3	0.988		0.993	0.005
EPZ2$_{A}$_A_B1B1	0.994		0.997	0.003
EPZ2$_{A}$_A_B1B2	0.998		0.997	−0.001
EPZ2$_{A}$_A_B1B3	0.998		0.993	−0.005
Epoxy2$_{A}$_A_B1T1	0.990		0.989	−0.001
Epoxy2$_{A}$_A_B1T2	0.994		0.993	−0.001
Epoxy2$_{A}$_A_B1T3	0.994		0.993	−0.001
Epoxy2$_{A}$_A_B1M1	0.998		0.997	−0.001
Epoxy2$_{A}$_A_B1M2	0.997		0.996	−0.001
Epoxy2$_{A}$_A_B1M3	0.996		0.995	−0.001
Epoxy2$_{A}$_A_B1B1	0.999		0.988	−0.011
Epoxy2$_{A}$_A_B1B2	0.998		0.999	0.001
Epoxy2$_{A}$_A_B1B3	0.998		0.998	0.000
PU2$_{A}$_A_B1T1		0.991	0.992	0.001
PU2$_{A}$_A_B1T2		0.988	0.990	0.002
PU2$_{A}$_A_B1T3		0.986	0.988	0.002
PU2$_{A}$_A_B1M1		0.997	0.997	0.000
PU2$_{A}$_A_B1M2		0.997	0.997	0.000
PU2$_{A}$_A_B1M3		0.998	0.997	−0.001
PU2$_{A}$_A_B1B1		0.993	0.993	0.000
PU2$_{A}$_A_B1B2		0.993	0.994	0.001
PU2$_{A}$_A_B1B3		0.967	0.974	0.007

3.4 | Quality control and assurance of on-site three-layer maintenance paint based on the chosen universal fingerprint region and three significant figures for *r* values

After accepting the null hypothesis related to the use of significant figures as well as the choice of universal fingerprint region, the on-site r values of the receiving EPZ, Epoxy and PU paints were screened at the job site using handheld FTIR and were estimated by *high sensitivity compare* function over the entire (4000–700 cm^{-1}) and universal fingerprint region (2000–900 cm^{-1}). Figure 2 shows example FTIR spectra of some paint samples analyzed by the in-house laboratory (which serves as a reference spectrum), on-site screening (analyzed using handheld FTIR) and third-party laboratory verification (for sample which failed on-site screening and further analyzed using benchtop FTIR). Here, there are three possible scenarios based on the evaluation of r values (shown later) that are obtained by spectra comparison: (a) true acceptance of the paint on-site, (b) false rejection (or false negative) of the sample on-site, or (c) false acceptance (or false positive) of the sample on-site. For on-site screening of the paints, the contractor may be using handheld FTIR for quick analysis. A quick visual inspection reveals that the spectra generated by handheld FTIR contains lower signal-to-noise ratio (example shown at

TABLE 8 An example of paired student *t*-test for individual paint system as well as collective data between the accepted and proposed fingerprint regions

	$EPZ2_A\_A$	$Epoxy2_A\_A$	$PU2_A\_A$	Entire sample population[a]
$\overline{D}_{\text{universal-specific}}$	−0.001	−0.002	0.001	−0.002
S_D	0.004	0.004	0.002	0.007
$SE(\overline{D})$	0.001	0.001	0.001	0.001
n	9	9	9	108
f	8	8	8	107
t-statistic	−1.000	−2.000	1.000	−2.000
|*t*-statistic|	1.000	2.000	1.000	2.000
t-critical	2.306	2.306	2.306	1.990
p-value	0.35	0.08	0.35	0.05

[a] Samples include EPZ (Part A, Part B), Epoxy (Part A, Part B), and PU (Part A, Part B) from Paint Manufacturer 2 and 4.

2600–1800 cm^{-1} which is possibly due to the presence of carbon dioxide from atmospheric weather) as compared with that of benchtop FTIR. Miller et al. (2012) used portable FTIR spectrophotometers to quantify the silica content in coal dust and commented that the portable FTIR is less sensitive than benchtop FTIR.[46] Portable FTIR with lower sensitivity as compared with benchtop FTIR may be due to the noise observed in the FTIR spectrum as shown in Figure 2(C).

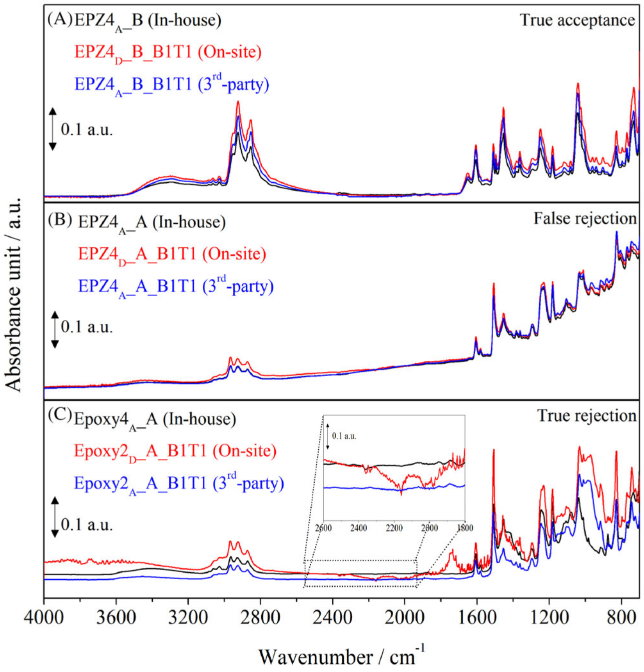

FIGURE 2 Overlay FTIR spectra of some paint samples (EPZ4 part A and part B, Epoxy4 and Epoxy2) generated during qualification of paint in the in-house laboratory (by benchtop FTIR), on-site screening (by handheld FTIR), and third-party verification (by benchtop FTIR) under different circumstances, (A) true acceptance, (B) false rejection, and (C) true rejection

The column chart illustrated in Figure 3 shows the on-site *r* values of all paint systems that were analyzed using handheld FTIR and in reference to in-house generated spectra. For EPZ4 part A, the *r* values generated for the entire region (4000–700 cm^{-1}) are higher than the threshold limit ($r \geq 0.900 \pm 0.002$) while the *r* values from universal fingerprint region (2000–900 cm^{-1}) is below the threshold. For EPZ4 part B sample, the *r* values generated for the entire region as well as universal fingerprint region are higher than the threshold limit. Similar observations can be seen in Epoxy4 samples. All the samples, including those with the *r* values that are below the threshold limit, were verified further by third-party laboratory.

The result of those that failed [approximately 20% rejection rate from the entire (4000–700 cm^{-1}) and universal fingerprint regions (2000–900 cm^{-1}) of three replicate analysis in each location (Top, Middle, and Bottom)] as well as passed the screening by handheld FTIR and then analyzed again with benchtop FTIR is summarized in Table 9. The *r* values of EPZ and Epoxy samples from paint manufacturer 4 that had previously failed in the universal fingerprint region (c.f., red-color bracket text in Table 9) during the screening test using handheld FTIR are now above 0.900 ± 0.002. This result indicates that the arriving samples on-site are similar to that received during bidding qualification, and that the *r* values generated from specific handheld FTIR may lead to a false rejection of the paint samples on-site. The false rejection due to lower *r* values than the threshold limit can be avoided when the screened sample is sent to the third-party laboratory for further verification. The limitation of handheld equipment may be seen in other tools for positive material identification (PMI) such as Raman spectroscopy, optical emission spectroscopy, and X-ray fluorescence spectroscopy.[47–49] When a non-conformance or incorrect use of a PMI tool to estimate the metal compositions is detected on-site, the metal sample (e.g., carbon steel, stainless steel) is sent to independent third-party laboratory and analyzed using benchtop PMI. In our case since the handheld FTIR fails occasionally to pass the on-site paints for the FTIR screening analysis. The handheld FTIR seems to have its limitations as verification tool but it serves well as a screening tool on-site. The benchtop unit on another hand is preferred over mobile/handheld for verification measurements. The handling of handheld FTIR (e.g., aiming at the same spot at the same distance for longer duration, increasing background scan) is important to have accurate measurements during screening of paint samples on-site. It is also worth noting that false rejection was observed in the paints with high solid contents (e.g., EPZ and Epoxy). A thorough stirring prior to analysis may help to reduce this rejection.

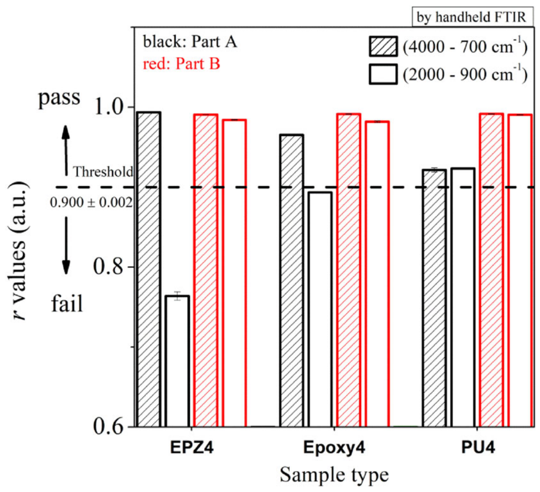

FIGURE 3 Column chart showing the *r* values of three-coat maintenance paints (from Paint Manufacturer 4) in reference to an in-house generated spectrum. The spectra were obtained using handheld FTIR and analyzed using Software D during on-site screening

Table 10 shows a clear-cut example for the rejection of three-coat maintenance paints with different formulations (or different paint manufacturers) after they were first analyzed on-site, failed the tests, and later on sent to another third-party laboratory for further verification. The paint samples (e.g., EPZ, Epoxy, and PU) analyzed on-site using handheld FTIR has the *r* values (indicated by the red numerical values in the bracket) that are generally higher than what would have been expected for the paint with different formulations. The highest *r* values can be observed in EPZ2 part A sample. These values are so close to the threshold limit ($r \geq 0.900$

TABLE 9 *r* values of three-coat maintenance paints (Paint Manufacturer 4) that had initially failed in "some" selected samples during the screening test on-site by handheld FTIR and then delivered to off-site facility of third-party laboratory for further verification

Sample code	*r* 4000–700 cm^{-1}	*r* 2000–900 cm^{-1}	Reference spectrum
EPZ4$_{\text{A}}$_A_B1T1	0.905	0.915 **(0.769)**[a]	EPZ4$_{\text{A}}$_A
EPZ4$_{\text{A}}$_A_B1T2	0.902	0.917 **(0.764)**[a]	
EPZ4$_{\text{A}}$_A_B1T3	0.913	0.940 **(0.759)**[a]	
EPZ4$_{\text{A}}$_B_B1T1	0.925	0.915	EPZ4$_{\text{A}}$_B
EPZ4$_{\text{A}}$_B_B1T2	0.932	0.919	
EPZ4$_{\text{A}}$_B_B1T3	0.936	0.917	
Epoxy4$_{\text{A}}$_A_B1T1	0.982	0.985 **(0.893)**	Epoxy4$_{\text{A}}$_A
Epoxy4$_{\text{A}}$_A_B1T2	0.987	0.989 **(0.893)**	
Epoxy4$_{\text{A}}$_A_B1T3	0.980	0.982 **(0.894)**	
Epoxy4$_{\text{A}}$_B_B1T1	0.912	0.972	Epoxy4$_{\text{A}}$_B
Epoxy4$_{\text{A}}$_B_B1T2	0.990	0.993	
Epoxy4$_{\text{A}}$_B_B1T3	0.990	0.993	
PU4$_{\text{A}}$_A_B1T1	0.966	0.982	PU4$_{\text{A}}$_A
PU4$_{\text{A}}$_A_B1T2	0.968	0.984	
PU4$_{\text{A}}$_A_B1T3	0.961	0.978	
PU4$_{\text{A}}$_B_B1T1	0.986	0.983	PU4$_{\text{A}}$_B
PU4$_{\text{A}}$_B_B1T2	0.962	0.952	
PU4$_{\text{A}}$_B_B1T3	0.962	0.952	

Note: The *r* values indicated by the bold numeric in brackets are the values that are below the threshold limit and were obtained during the screening test on-site by handheld FTIR. Analysis was performed using a benchtop iS5 spectrophotometer FTIR (Software A) operating in *high sensitivity compare* function within the entire (4000–700 cm^{-1}) and universal fingerprint regions (2000–900 cm^{-1}).

[a] False negative (false rejection) as per Figure 3.

TABLE 10 *r* values of three-coat maintenance paints with different paint formulations (Paint Manufacturer 2 against Paint Manufacturer 4)

Sample code	*r* 4000–700 cm^{-1}	*r* 2000–900 cm^{-1}	Reference spectrum
EPZ2$_{C}$_A_B1T1	0.449 (0.922[a])	0.600 **(0.897)**	EPZ4$_{A}$_A
EPZ2$_{C}$_A_B1T2	0.434 (0.923[a])	0.583 **(0.894)**	
EPZ2$_{C}$_A_B1T3	0.422 (0.923[a])	0.561 **(0.895)**	
EPZ2$_{C}$_B_B1T1	0.138 **(0.272)**	0.138 **(0.426)**	EPZ4$_{A}$_B
EPZ2$_{C}$_B_B1T2	0.137 **(0.272)**	0.137 **(0.426)**	
EPZ2$_{C}$_B_B1T3	0.137 **(0.251)**	0.137 **(0.428)**	
Epoxy2$_{C}$_A_B1T1	0.362 **(0.713)**	0.382 **(0.746)**	Epoxy4$_{A}$_A
Epoxy2$_{C}$_A_B1T2	0.359 **(0.711)**	0.379 **(0.744)**	
Epoxy2$_{C}$_A_B1T3	0.358 **(0.710)**	0.377 **(0.744)**	
Epoxy2$_{C}$_B_B1T1	0.127 **(0.593)**	0.169 **(0.814)**	Epoxy4$_{A}$_B
Epoxy2$_{C}$_B_B1T2	0.125 **(0.595)**	0.168 **(0.818)**	
Epoxy2$_{C}$_B_B1T3	0.125 **(0.596)**	0.169 **(0.817)**	
PU2$_{C}$_A_B1T1	0.469 **(0.574)**	0.519 **(0.488)**	PU4$_{A}$_A
PU2$_{C}$_A_B1T2	0.463 **(0.574)**	0.513 **(0.486)**	
PU2$_{C}$_A_B1T3	0.458 **(0.574)**	0.507 **(0.484)**	
PU2$_{C}$_B_B1T1	0.114 **(0.339)**	0.119 **(0.281)**	PU4$_{A}$_B
PU2$_{C}$_B_B1T2	0.114 **(0.340)**	0.119 **(0.283)**	
PU2$_{C}$_B_B1T3	0.114 **(0.340)**	0.119 **(0.283**)	

Note: The *r* values indicated by bold numeric in brackets are the values that are below the threshold limit and were obtained during the screening test on-site by handheld FTIR. The *r* values that had initially failed in almost all the samples during the screening test on-site by handheld FTIR were then delivered to third-party laboratory for further verification. Analysis was performed using a benchtop Spectrum two FTIR (Software C) operating in *high sensitivity compare* function within the entire (4000–700 cm^{-1}) and universal fingerprint regions (2000–900 cm^{-1}).

[a] False acceptance.

± 0.002) on the entire region of infrared spectra (4000–700 cm^{-1}) that it can be perceived, if accepted during on-site, as "false acceptance" or "false positive." It is worth noting that the occurrence of false acceptance in the studied paint samples is one fifth less than those with false rejection (only observed in EPZ2 part A Top sampling from the paint container). Further verification by the third-party laboratory shows more accurately determined *r* values. The *r* values analyzed using a benchtop FTIR are somewhat lower than those obtained using benchtop FTIR, and they are also below the threshold limit. Although handheld FTIR can be used to detect reformulation on-site, the benchtop FTIR has better accuracy. The results for paints with different formulations, although occasionally may lead to "false acceptance," was subsequently verified by third-party laboratory.

4 | CONCLUSIONS

There are three concluding remarks that can be drawn from this study. Using a paired student *t*-test, it can be observed that (a) there is no statistical difference between three and four significant figures for reporting the degree of similarity and this prompts us to choose three significant figures and (b) the choice between selective FTIR fingerprint region (that varies depending on the nature of chemical compounds in the paint formulation) and universal fingerprint region (2000–900 cm^{-1}) is of no distinction for different paints. Thus, three significant figures of degree of similarity and universal FTIR fingerprint region were used henceforth. With these methods set in place, the arriving three-coat maintenance paint on-site was screened using handheld FTIR spectrophotometers. Results show that some of the supplied paints that failed the screening on-site after analyzed with handheld FTIR were further analyzed using benchtop FTIR. The handheld FTIR gives the *r* values that are less reliable as compared with that of the *r* values generated by benchtop FTIR (which may occasionally lead to "false negative/rejection" or "false positive/acceptance"). It can be concluded that (c) the handheld FTIR is only to be used as screening tool while the benchtop FTIR is to be used for verification purposes. Based on the above-mentioned conclusions, the reference standard of IMM FP01:2019 was then updated to IMM FP01:2020 (Coating fingerprinting overall procedures for paints using FTIR and other related methods).

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