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The minimum number of measurements for colour, sparkle, and graininess characterisation in gonio-apparent panels

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Society of Dyers and Colourists

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Materials with new visual appearances have emerged over the last few years. In the automotive industry in particular there is a growing interest in materials with new effect finishes, such as metallic, pearlescent, sparkle, and graininess effects. Typically, for solid colours the mean of three measurements with repetitions is sufficient to obtain a representative measurement for colour characterisation. However, gonio-apparent panels have non-homogeneous colours, and there are no studies that recommend the minimum number of repetitions for colour, sparkle, and graininess characterisation of this type of panel. We assume that colour panels incorporating special-effect pigments in their colour recipes will require a higher minimum number of measurements than solid colour panels. Therefore, the purpose of this study is to verify this assumption by using a multiangle BYK-mac spectrophotometer, given that it is currently the only commercial device that can measure colour, sparkle, and graininess values simultaneously. In addition, a possible methodology is given for establishing the minimum number of measurements when characterising gonio-apparent materials using a specific instrument, able to be implemented in future instruments when determining multiple appearance attributes (colour, gloss, sparkle, etc.) for many coloration technologies. Thus, we studied the minimum number of measurements needed to characterise the colour, sparkle, and graininess of three types of sample with solid, metallic, and pearlescent coatings respectively. Twenty measurements were made at twenty random positions (different target areas) of 90 samples. The minimum number of measurements for all these variables was determined on the basis of the point at which the cumulative mean value became constant. Thus, applying new statistical tools, it is clearly shown that metallic and pearlescent panels require more colour measurements than solid panels, in particular when geometries are being measured in a specular direction. As regards texture (sparkle and graininess), more measurements are needed for graininess than for sparkle, and more for metallic panels than for pearlescent panels.

Introduction

Gonio-apparent panels refer to materials that present notable colour changes under different viewing angles and lighting conditions. Panels with metallic finishes usually show a change in lightness, while those with an interference finish effect also exhibit hue and chroma changes. On account of this, to characterise this type of panel it is important to measure the colour at different measurement geometries [1–3]. Nowadays, there are several colour-measuring instruments, known as multiangle spectrophotometers or gonio-spectrophotometers, which have been designed to measure and characterise homogeneous colours in these types of panel. The measurement geometries of these devices usually agree with the requirements established in the ASTM, DIN, and ISO standards [4–6], which refer to the characterisation of gonio-colour appearance. However, there are other types of non-industrial device or scientific prototype providing many extra measurement geometries [2].

Materials with new visual appearances have emerged over the last few years. In the automotive industry in particular, and also in cosmetics and plastics for consumer electronics, there is a growing interest in materials with new effect finishes. Their popularity is due to the fascinating interplay of colours and the effects produced by the various materials used in their layered structures [7–9]. Refractions and reflections of light at and within these layers cause light interferences that yield certain colours [10]. Thus, depending on the pigments, the panels can have an interference finish or sparkle and graininess effects when special-effect pigments are used. The sparkle effect (also known as sparkling effect, microbrilliance, glint, or glitter) is generated by the reflectivity of the individual flakes, which act like tiny mirrors, and is observed under intense directional illumination. In contrast, and for the same panel, the viewing angle is not relevant when studying the graininess effect (also described as coarseness or salt and pepper appearance), which is observed under diffuse illumination. Both contribute in different ways to the visual texture over the panel. It should be pointed out that, when we say visual texture, this refers to the textural appearance due to the sparkle and graininess of the sample. In addition, because of the variable position of these pigments, the measured colour and visual texture are not constant over the entire panel.

Panels with solid colours can be characterised with conventional colour devices, such as those based on integrating spheres. Panels with a metallic or interference finish effect must be characterised with multiangle spectrophotometers, which are capable of characterising homogeneous colours at different measurement geometries. However, these colour devices calculate the colour values by averaging the spectral reflection over the entire illuminated spot, which means that they are unable to extract information about the reflection of the metallic or pearlescent flakes. In spite of the fact that these types of device can measure colour at several geometries, they cannot measure the grade of sparkle or graininess of the panels with this procedure.

There is currently only one commercial device able to measure colour and visual texture (sparkle and graininess) under different viewing angles and illumination conditions simultaneously – the BYK-mac multiangle spectrophotometer (Byk, Germany) [11]. This device measures the sparkleand graininess effects using a high-resolution monochrome CCD camera which characterises effect changes perceived under direct and diffuse lighting conditions [8,12]. The model used in the present work was the BYK-mac 23 mm.

Given that the orientation of special pigments is variable [13], for panels with a sparkle effect it is expected that different sparkle and graininess values, and even different colour values, will be obtained at different measurement positions. There are studies based on standard institutions (CIE, ASTM, etc.) recommending a minimum number of measurements at different positions to characterise solid colour samples, even with low graininess or sparkle. However, they do not provide recommendations on the minimum number of measurements needed to characterise colours with special-effect pigments, such as panels with sparkle and/or graininess effects.

Our expectation is that colour panels incorporating special-effect pigments [9,14] in their colour recipes will require a higher minimum number of measurements than solid colour panels for colour, sparkle, and graininess characterisation, because colour solid samples are homogeneous over the entire surface and special effects are variable. Consequently, the purpose of this study is to confirm this by using the multiangle spectrophotometer BYK-mac. In addition, the study also focuses on analysing whether there are differences in the minimum number of measurements needed to characterise three types of sample typically used in the automotive sector: solid, metallic, and pearlescent. Colour and visual texture at different measurement geometries were examined for the characterisation. The parameters studied were the colorimetric, sparkle, and graininess values at six, three, and one measurement geometry respectively.

Materials and Methods

Measurement device

The BYK-mac multiangle spectrophotometer is capable of measuring spectral reflectance at six geometries for colour characterisation, three geometries for sparkle, and one geometry for graininess characterisation simultaneously. This device characterises colour by measuring the sample at different aspecular angles: -15, 15, 25, 45, 75, and 110°. Following CIE standards, these geometries are represented as 45°*x*:-60°, 45°*x*:-30°, 45°*x*:-20°, 45°*x*:0°, 45°*x*:30°, and 45°*x*:65° respectively (Fig. 1).

Similarly, this device characterises sparkle, S_g, by measuring in a perpendicular direction to the sample and at an illumination angle of 15, 45, and 75°. Following CIE standards, these geometries are represented as 15°*x*:0°, 45°*x*:0°, and 75°*x*:0° respectively (Fig. 2). Graininess, G, is evaluated by measuring in a perpendicular direction to the sample and under diffused illumination (Fig. 2).

The sparkle and graininess measurements are determined by taking a picture with a monochrome CCD camera under direct and diffuse lighting conditions respectively. The pictures are analysed by means of image-analysis algorithms using the histogram of intensity levels as the basis for calculating sparkle and graininess parameters [11]. Although the algorithms used are not public, it is known that this device calculates the sparkle, S_g, by combining two intermediated values of sparkle: area and intensity, S_a and S_i. The sparkle of area, S_a, is a measure indirectly related to the flake size, but it does not mean that sparkle of area measures the size of the individual effect pigments. The sparkle of intensity, S_i, is designed to take into account the intensity of the reflection. Accordingly, we used these three parameters S_g, S_i, and S_a in the present study, taking into account the relative intensity (in digital counts) of the reflection. The graininess value is obtained directly from the histogram of intensity levels of the picture recorded under diffuse lighting conditions.

Sample measurement

Thirty samples were chosen for each type of pigment or colour recipe: 30 solid colour, 30 metallic, and 30 pearlescent samples. The colours were selected to cover a large proportion of the colour gamut, as shown in Fig. 3, where all measurement geometries are plotted together.

For solid colour samples (blue dots), some clusters can be observed. This can simply be explained by the fact that solid colour samples do not show a colour shift when the measurement geometry changes. On the other hand, for metallic and pearlescent samples, no clusters are shown because they exhibit changes in colour with measurement geometry.

Twenty measurements at random positions were made for each sample, from which the cumulative mean value for colorimetric values at six measurement geometries ($L^*a^*b^*$ at -15, 15, 25, 45, 75, and 110°), sparkle values at three measurement geometries (S_g, S_i, and S_a at 15, 45, and 75°), and graininess values at one measurement geometry (G) were calculated for all measurement geometries and plotted. As an example, Fig. 4 provides three of the 28 cumulative mean values (L^* at -15°, S_i at 45°, and G) for a solid, a metallic, and a pearlescent sample.

The minimum number of measurements was selected as that obtained when the corresponding cumulative mean value reached the horizontal asymptote, i.e. became constant. The following statistical formula [15] was used to establish objectively a method for determining when the minimum number of measurements became constant:

$$n = \frac{N\sigma^2 Z^2}{(N-1)e^2 + \sigma^2 Z^2} \quad (1)$$

where N is the population size, σ is the standard deviation, Z is a value linked to the required confidence level (equivalent to 1.96 for a confidence interval of 95%), and

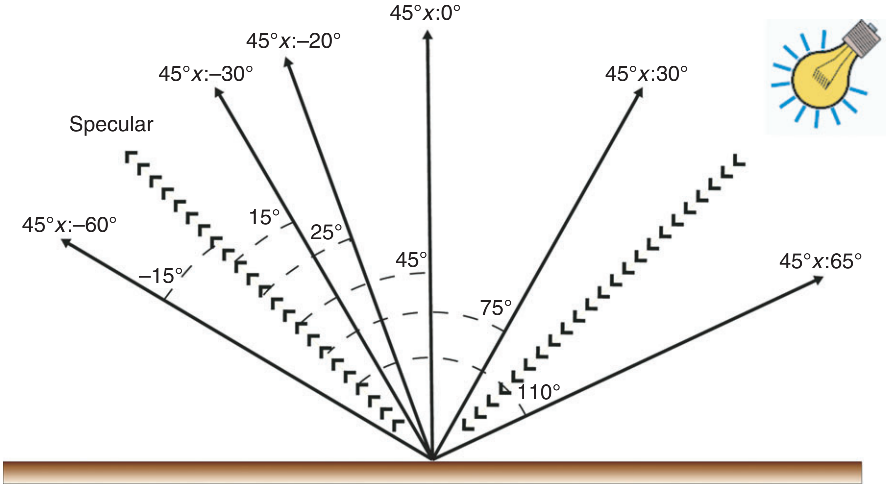

Figure 1 Schematic representation of the illumination/measurement geometries used by the BYK-mac device for colour characterisation [Colour figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

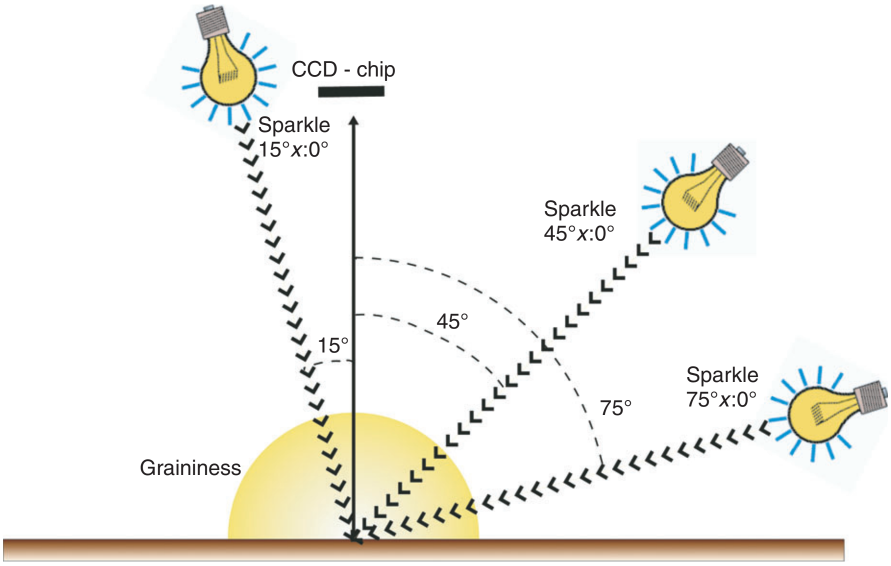

Figure 2 Schematic representation of the illumination/measurement geometries used with the BYK-mac device for sparkle and graininess characterisation [Colour figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

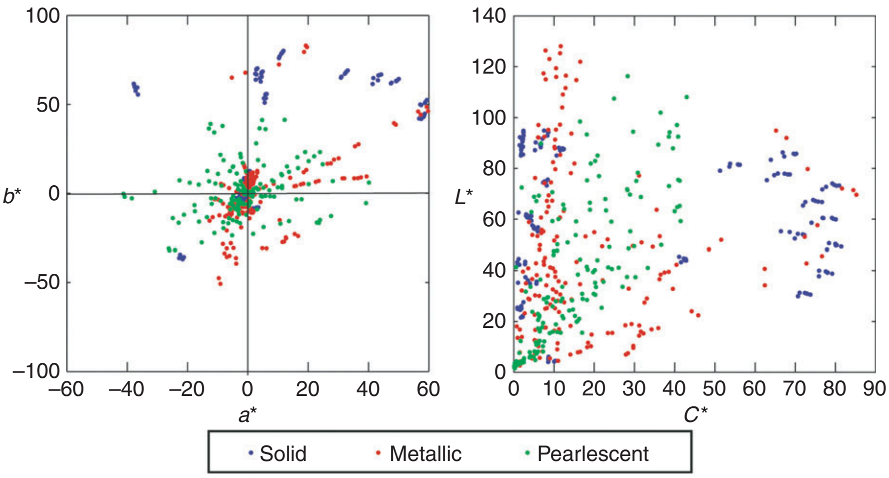

Figure 3 Sample selection represented in the diagram of CIE $L^*a^*b^*$ space. All measurement geometries are plotted together [Colour figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

e is the acceptable limit of sampling error, which we assumed to be 1 unit. The following equation was used to calculate the mean confidence interval in a small population or when sampling without replacement:

$$\bar{x} - Z\frac{\sigma}{\sqrt{n}}\sqrt{\frac{N-n}{N-1}} = \mu = \bar{x} + Z\frac{\sigma}{\sqrt{n}}\sqrt{\frac{N-n}{N-1}} \qquad (2)$$

Following this procedure, we obtained the results for each type of pigment recipe with the minimum number of colour measurements for individual samples and geometries.

The same results were computed for the minimum number of sparkle and graininess measurements. The values obtained for all samples are plotted with a vertical blue line in Fig. 4.

Equation (2) was applied for each individual sample and each geometry in order to determine the minimum number of measurements to characterise colour and visual texture. In Fig. 4, the results for some samples are plotted with a vertical blue line in the graphs.

Statistical analysis

Data analysis was performed using SPSS software (v.21; SPSS, USA) for Windows. An α-value of 0.01 was considered to be significant.

The main purpose of the present study was to assess the dependency on the minimum number of measurements to characterise the colour, sparkle, and graininess with the type of panel colour recipe and measurement geometry. With this in mind, samples were measured in all geometries, which means that they were related measurements. Therefore, multivariate analysis of variance (MANOVA) [15–17] was used to analyse the data. The SPSS statistical program was used for the computations. Some considerations have to be borne in mind to apply this analysis. As said before, the samples have been measured in all geometries, which means that they are related measurements. Thus, the geometry acts as a dependent variable, and the type of pigment as an independent one. On the other hand, for applying MANOVA analysis, it is necessary to fulfil some requirements (normal distribution and homogeneity of variance). However, in some geometries the data did not satisfy these requirements because some had a constant value. Because of this, we represented the data using a box plot, which provided us with an overview of the quantitative data. Based on the results of these box plots, we were able to decide which data could be subjected to independent statistical analysis for individual geometries and which data did not require further analysis.

In a second step, an analysis of variance (ANOVA) was used to test for significant differences in the minimum number of measurements. The null hypothesis of this test is that the minimum number of measurements for solid samples is the same as that for metallic or pearlescent samples. When the ANOVA F-test was significant, post hoc analyses were carried out. The Kolmogorov–Smirnov test (K–S test) was used to test for normality of data, and the Bartlett test was used to check for homogeneity of variance. When significant heterogeneity was found, data were transformed by $\sqrt{(x+1)}$ or $\ln(x+1)$. When transformations did not remove heterogeneity and/or the distribution did

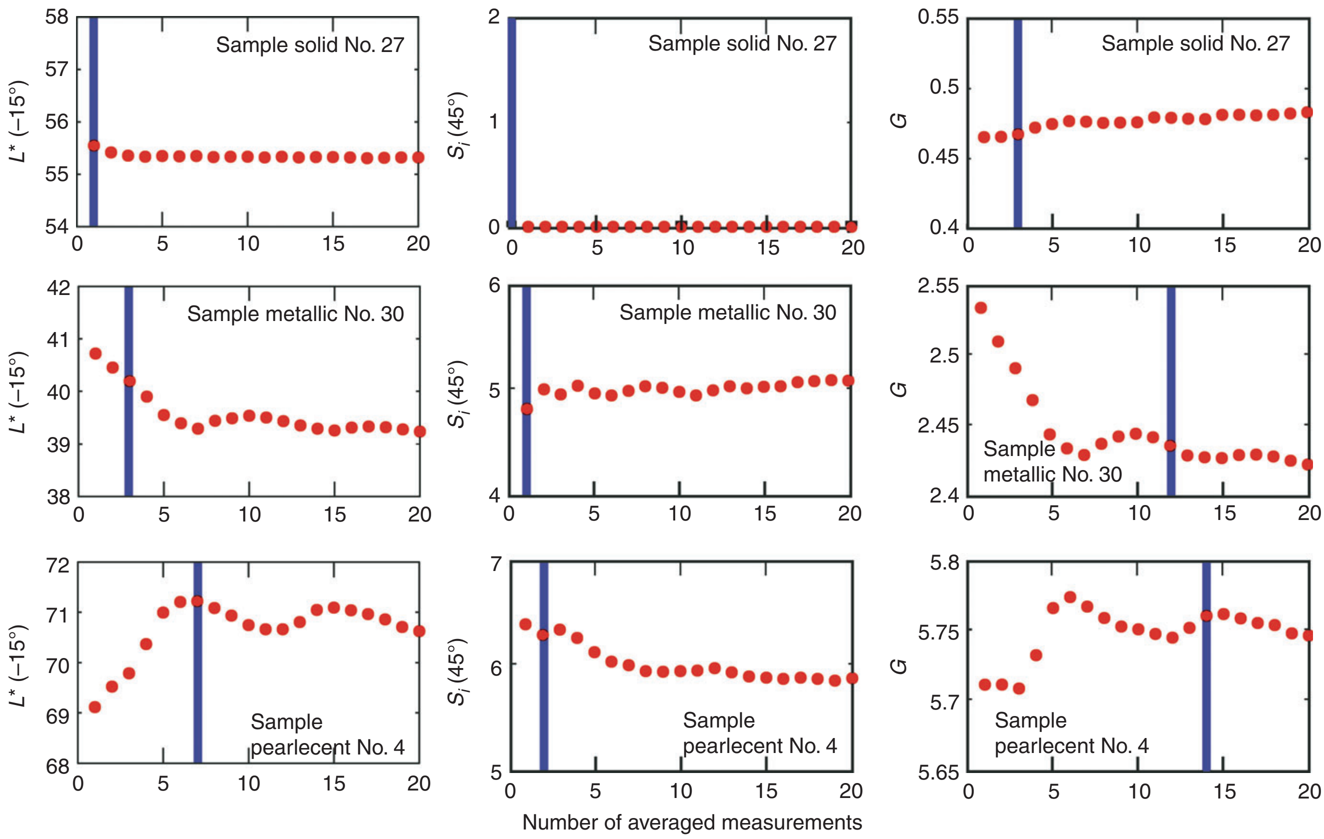

Figure 4 Cumulative mean values for a solid, a metallic, and a pearlescent sample (L^* at -15°, S_i at 45°, and G). The blue line is the proposed minimum number of measurements after applying statistical analysis [Colour figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

not follow a normal distribution, analyses were performed on the untransformed data with the *F*-test α-value set at 0.01. This can be done because ANOVA is sufficiently robust to depart from this assumption, especially when the design is balanced and contains a large number of samples or treatments [17].

Results

Figures 5–7 give box plots of the results for the three studied variables: colour, sparkle, and graininess. The bottom and top of the box are the first and third quartiles, and the black band inside corresponds to the median. The ends of the whiskers represent the minimum and maximum values of all data. Any data not included between the whiskers are plotted as an outlier with an asterisk. These results enable us to decide when a statistical study is necessary for each individual case, and to recommend a minimum number of measurements for the characterisation of colour, sparkle, and graininess parameters. In an ideal case it is expected to require only a single measurement for characterisation of colour, sparkle, and graininess. However, the special properties of the pigments will require more than one measurement in some cases.

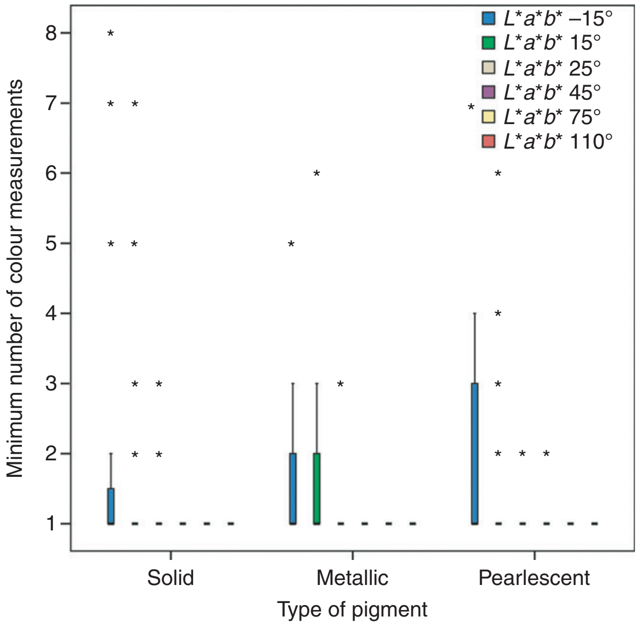

Figure 5 Box plot of the minimum number of colour measurements in samples with different types of panel colour recipe for different measurement geometries [Colour figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Colour measurements

Figure 5 gives a box plot of the minimum number of colour measurements for a set of 30 samples with solid pigments, as well as samples with metallic and pearlescent pigments. The results showed that a single measurement, using a BYK-mac, was needed to determine the colour in four geometries (25, 45, 75, and 110° from the specular angle), regardless of the type of pigment recipe, given that the height of the box matched the width of the whiskers. This means that the upper and lower quartile were both 1 and match with the box, i.e. they required just one measurement. In addition, the data were not analysed by means of an ANOVA test because all values were constant.

On the other hand, in the two geometries closest to the specular angle (−15 and 15°), the boxes became higher and the whiskers became wider, meaning that a larger number of measurements were necessary to determine the colour for all types of sample. For these geometries, the boxes of pearlescent samples were higher than the boxes of metallic samples, and even higher than the boxes of solid samples. Therefore, it can be concluded that a greater number of measurements were needed to determine the colour for metallic samples than for solid samples. An even greater

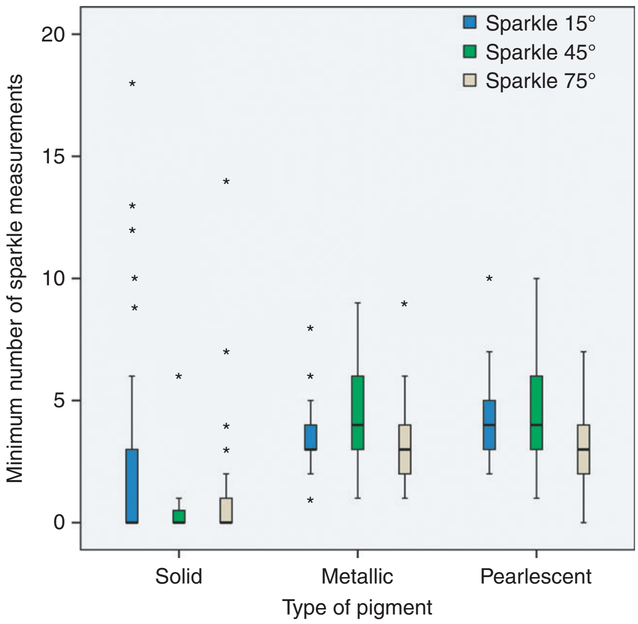

Figure 6 Box plot of the minimum number of sparkle measurements in samples with different types of pigment for different measurement geometries [Colour figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

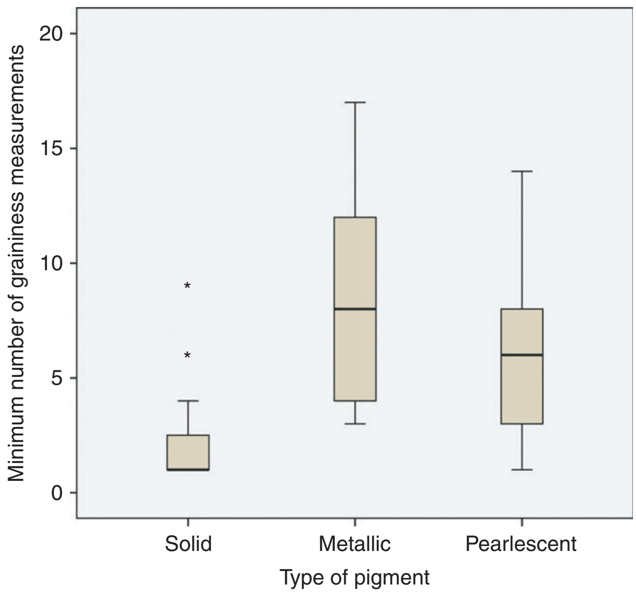

Figure 7 Box plot of the minimum number of graininess measurements in samples with different types of pigment [Colour figure can be viewed in the online issue, which is available at wileyonline library.com.]

number of measurements were necessary to determine the colour for pearlescent samples. An analysis of variance was carried out in this case to study whether there were significant differences between the means. The results of a one-way ANOVA test with type of pigment as a factor for the minimum number of colour measurements are shown in Table 1.

As can be seen, the *P*-value was greater than 0.01 for the two geometries closest to the specular angle (−15 and 15°), which means that there were no significant differences between the averages of the minimum number of colour measurements. Therefore, these results indicated that two measurements were necessary for colour characterisation using these geometries. Because the average results of measurements were always decimal numbers, they were rounded up to the nearest whole number.

Sparkle measurements

The same steps described in the previous section were used for sparkle measurements. Figure 6 shows the box plot of the minimum number of sparkle measurements for the same data.

In this case, a larger number of measurements were needed to determine the sparkle, given that the boxes became higher and the whiskers became wider in all geometries and for all types of pigment. Moreover, the results showed that the minimum number of measurements was always lower for the solid samples than for the metallic and pearlescent samples, which is consistent with the fact that solid samples do not exhibit a sparkle effect. It was for this reason that the ANOVA test was only carried out with metallic and pearlescent data.

Table 1 Results of analysis of variance (one-way ANOVA) (factor – type of pigment: solid, metallic, or pearlescent) for the minimum number of colour measurements

	−15°			15°		
	df[a]	F[b]	*P*-value	df	*F*	*P*-value
Type of pigment	2	1.1420	0.324[c]	2	0.0840	0.919[c]
Transformation	log(x + 1)			[d]		

a df: degrees of freedom.
b *F*: *F*-test.
c Level of significance $P < 0.01$.
d No transformation.

On the one hand, in spite of the fact that the solid samples do not have sparkle, sometimes the measured sparkle values are not precisely zero because of measurement uncertainty. Not surprisingly, the first quartile and the median are both at zero for solid colours. We use the third quartile to indicate the minimum number of measurements for minimising uncertainty.

On the other hand, the results of the ANOVA test for the minimum number of sparkle measurements for metallic and pearlescent samples (Table 2) showed that there were no significant differences for sparkle characterisation at 15, 45, and 75° between the averages of the metallic and pearlescent samples, as *P*-values of >0.01 were obtained in all cases. We could therefore conclude that four measurements were needed for sparkle characterisation at 15°, five measurements at 45°, and four measurements at 75°, regardless of whether the samples were metallic or pearlescent.

Graininess measurements

The same methodology was also used for graininess measurements. In this case, Fig. 7 gives the box plot of the minimum number of graininess measurements for the same data, and similar conclusions to those obtained for sparkle analysis could be drawn.

The results proved that the minimum number of measurements for the solid colour samples was always lower than for the metallic and pearlescent samples (three measurements). Consequently, the ANOVA test was only carried out with metallic and pearlescent sample data. The results are shown in Table 3.

As can be seen, there were statistically significant differences between the averages of the metallic and pearlescent samples in terms of graininess characterisation,

Table 3 Results of analysis of variance (one-way ANOVA) (factor – type of pigment: metallic or pearlescent) for the minimum number of graininess measurements

	df[a]	F[b]	*P*-value
Type of pigment	1	7.3820	0.00867[c]
Transformation	[d]		

a df: degrees of freedom.
b *F*: *F*-test.
c Level of significance $P < 0.01$.
d No transformation.

Table 2 Results of analysis of variance (one-way ANOVA) (factor – type of pigment: metallic or pearlescent) for the minimum number of sparkle measurements

	15°			45°			75°		
	df[a]	F[b]	*P*-value[c]	df	*F*	*P*-value	df	*F*	*P*-value
Type of pigment	1	6.3270	0.0147[a]	1	0.0150	0.902[a]	1	0.0850	0.772[a]
Transformation	sqrt(x + 1)			[d]			sqrt(x + 1)		

a df: degrees of freedom.
b *F*: *F*-test.
c Level of significance $P < 0.01$.
d No transformation.

meaning that in this case nine or six measurements were needed respectively (Fig. 7).

Discussion and Conclusions

This paper gives a methodology for assessing the minimum number of measurements when characterising gonio-apparent materials using a specific instrument, particularly designed and built to determine some visual appearance attributes, like the current BYK-mac. However, this approach could be applied in future to new visual appearance instruments, even including gloss, goniofluorescence, translucency, etc., for many coloration industries (automotive, coatings, plastics, printing, cosmetics, textile, leather, etc.) using special-effect pigments.

Regarding the characterisation of colour, the results of this study showed that a single measurement was needed to determine the colour at four angles (25, 45, 75, and 110° from the specular angle), regardless of the type of pigment recipe. In addition, two measurements were needed for the characterisation of colour in the two geometries closest to the specular angle (−15 and 15°), which is consistent with the fact that these geometries are very close to the specular angle, which can greatly affect the results.

In terms of sparkle and graininess, the minimum number of measurements for the solid colour samples was always lower than for metallic and pearlescent samples, which is consistent with the fact that solid colour samples lack sparkle and graininess. Therefore, in this case only one measurement is necessary in order to assess the lack of sparkle or graininess.

However, for sparkle characterisation, the statistical study also showed that it was not necessary to make any distinction between metallic or pearlescent samples. In this case, four measurements were needed at 15°, five measurements at 45°, and four measurements at 75°.

Finally, with regard to the characterisation of graininess, the conclusion was that there were significant differences between the means of metallic and pearlescent samples, indicating that nine or six measurements were needed in this case respectively.

In conclusion, this study demonstrates that, using a BYK-mac instrument, although applicable to new multiangle spectrophotometers, more than one measurement is often necessary at each measurement geometry to account properly for colour, sparkle, and graininess effects in samples including special-effect pigments. This can have an impact in some industries, especially in the automotive sector, in which new effect finishes are commonly used.

Therefore, it could be interesting to apply this study once again to other specific panel sets previously designed to evaluate the influence of coating method application, size and shape of special-effect pigments, types of interference pigment, pigment concentrations in recipes, etc.

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