



## BLACK – THE FINE DETAILS

The measurement of deep-black coatings represents an enormous challenge for measurement technology and for the preparation of samples, requiring minute attention to detail. By Kai Krauss, Andrea Höpke, and Markus Mahn, Orion Engineered Carbons.

**Measurement devices are primarily used to analyse coloured surfaces. Errors in the calibration of the devices when measuring close to zero reflection range can lead to erroneous test results. As this is often not recognised, erroneous measurements are common.**

**W**ith most people, colourimetry conjures up the image of bright and shining colours. Producers of black colors naturally have a different perspective. Their occupation inherently makes them think of black colours and coatings. Now and again, grey joins their palette. This might literally sound rather monotonous, but it really isn't in practice. Grey tones particularly demonstrate significant variations when it comes to the so-called undertones. Deep-black coatings also all have their own undertone, which can be highly relevant to the end user. As a general rule, a difference is made between blue and brown (reddish) undertones, revealing the colourful world hiding underneath the deep-black cover.

In addition to this colour spectrum, there's another exciting and challenging obstacle to master: The measurements are done on surfaces with virtually no reflection. This places great demands on the diligence of the work process and the measurement technology used. This topic has been of interest for a very long time and has inspired, among other things, the development of a method for the measurement of

the hue-dependent blackness value and undertone ( $M_c$  and  $dM$ ) [1]. These have developed into the standard of deep-black coating measurements of colours and coatings.

### BLACKNESS VALUES $M_c$ , $M_v$ , $dM$

The measurement of black coatings takes place within the  $L^*a^*b^*$  colour space, with a value lower than five on the  $L^*$  axis. This is equal to a light reflection of lower than 0.1 percent. When venturing into the realm of deep black, such as the standard topcoats used by automotive OEMs, for example, we quickly reach a range of  $L^* < 1$ .

The colourimetric properties of a black coating system can be described using the hue-independent blackness value  $M_v$  (jetness). The measuring method is specified in DIN 55979. This is joined by the absolute contribution of hue  $dM$ , which is also often called the undertone. The blackness value determines the black content, in other words how deep the black is from a colourimetric/optical perspective. The undertone describes the colourimetric/optical perception of the colour shade. The undertone is called blue if  $dM > 0$  and brown if  $dM < 0$ . The blue undertone is generally preferred for technical applications, especially in automotive topcoat systems, as it yields a more lush result and an impression of brilliant colour. Brown undertones on the other hand tend to be perceived as warm and are thus pre-

**RESULTS AT A GLANCE**

- The measurement of deep-black coatings places great demands on sample preparation as well as the measurement technology and calibration. The use of calibration plates usually does not suffice, as they are not black enough. Instead, a so-called black hollow body, which absorbs virtually all the light, is used. As deep black can only be measured on high-gloss and clean plates, any contaminants must be very thoroughly removed and the plates must be absolutely free of scratches, finger prints, and similar before measuring.
- If these specifications are followed, replicable measurements of deep-black coatings are possible and information about the blackness/jetness  $M_Y$  and undertone  $dM$  can be obtained.
- The undertone itself can either reinforce the impression of blackness (in most cases with a blue undertone) or decrease it (in most cases with a brown undertone).

Figure 1: Jetness  $M_Y$  as a function of reflection [2].

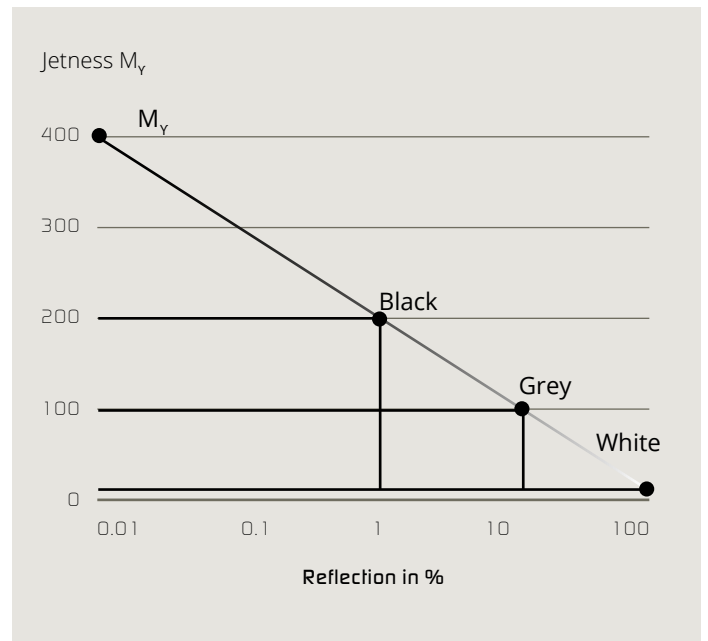
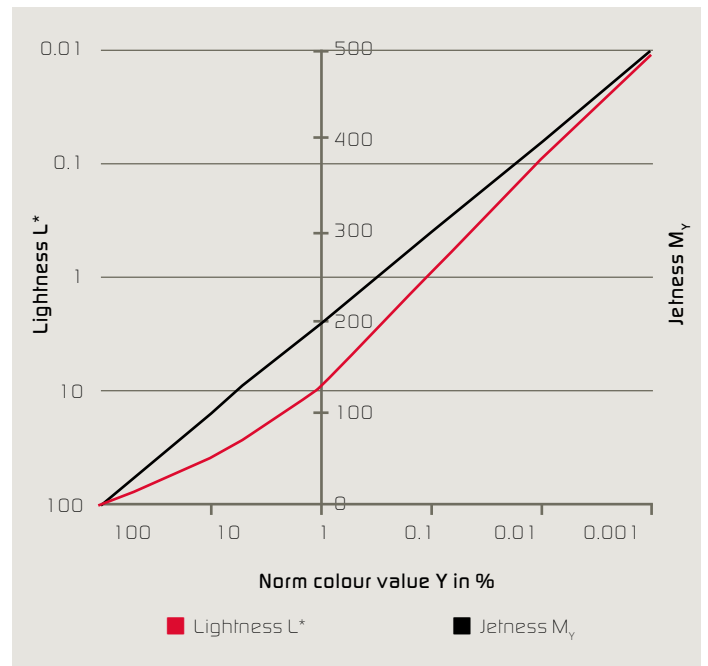


Figure 2: Correlation between lightness  $L^*$  and blackness value (jetness)  $M_Y$ .



ferred for interior applications and above all wood coatings. Dependence of jetness  $M_Y$  as a function of reflection is depicted in Figure 1 [2].  $M_Y$ ,  $M_C$  and  $dM$  can be calculated using the following formulas [3]:

$$M_Y = 100 \cdot \log\left(\frac{100}{Y}\right)$$

Blackness value

$$M_C = 100 \cdot \left[ \log\left(\frac{X_n}{X}\right) - \log\left(\frac{Z_n}{Z}\right) + \log\left(\frac{Y_n}{Y}\right) \right]$$

Hue-dependent blackness value

$$dM = M_C - M_Y$$

Absolute contribution of hue

**CIE L\*A\*B\* COLOUR SPACE**

The CIE  $L^*a^*b^*$  colour space was developed to achieve a better correlation with human colour perception. The lightness  $L^*$  and the colour coordinates  $a^*$  and  $b^*$  make up this colour space. They are calculated on the basis of the tristimulus values X, Y, and Z. The standard colour values required, when using light source D65 and an observer at a 10° angle, are defined as  $X_n = 94.81$ ,  $Y_n = 100.0$ , and  $Z_n = 107.34$  [3]. Here, Y represents the relevant value for the perception of the lightness, which correlates with the  $L^*$  value in the CIE  $L^*a^*b^*$  colour space, whereas X and Z are necessary to calculate the  $a^*$  and  $b^*$  values. The relevant formulas are as follows:

$$L^* = 116 \cdot \sqrt[3]{\frac{Y}{Y_n}} - 16$$

Lightness

$$a^* = 500 \left[ \sqrt[3]{\frac{X}{X_n}} - \sqrt[3]{\frac{Y}{Y_n}} \right] \quad b^* = 200 \left[ \sqrt[3]{\frac{Y}{Y_n}} - \sqrt[3]{\frac{Z}{Z_n}} \right]$$

Colour Coordinates

The relationship between the lightness  $L^*$  and the hue-independent jetness  $M_Y$  as a function of the tristimulus value Y is depicted in Figure 2 [3]. This logarithmic presentation has the advantage of significantly spreading out the region of very low reflection ( $Y < 1$ ) which more clearly emphasizes the distinction between the black and deep-black ranges. Without using this logarithmic depiction, differences that are readily discernible to the eye cannot be adequately represented in numbers. ➔

- The correlation between the absolute contribution of hue  $dM$  and the colour coordinates  $a^*$  and  $b^*$  is depicted in Figure 3. A negative  $b^*$  value here represents a blue undertone with  $dM > 0$ . With a positive  $b^*$  value in the yellow range, the  $dM$  value is negative and corresponds to a brown undertone.

Depending on the absorption and reflection properties of the coating, this can be divided into various colour ranges, as depicted in Table 1. Colours are only classified as black at less than one percent reflection, for deep black, this value drops even lower to less than 0.1 percent reflection. Carbon blacks are subdivided into various degrees of black following generally applicable classifications. In Table 1, the coatings which are produced with the designations Regular (RCG), Medium (MCG), and High Color Gas Blacks (HCG) following this nomenclature are also listed. The correct measurement of deep-black coatings requires great diligence.

Table 1: Colour depending on the absorption and reflection of light.

Absorption in %	Reflection Y in %	Jetness MY	Colour	Carbon black Type
0	100	0	white	
90	10	100	grey	
99	1	200	black	
99,9	0,1	300	deep black	
99,99	0,01	400	deepest black	
<hr/>				
99,60 - 99,75	0,40 - 0,25	240 - 260		RCG
<hr/>				
99,84 - 99,92	0,16 - 0,08	280 - 310		MCG/HCG
<hr/>				
99,92 - 99,95	0,08 - 0,05	310 - 330		HCG

Figure 3: Absolute contribution of hue  $dM$  displayed in the CIE  $a^*b^*$  colour coordinate system.



MEASUREMENT TECHNOLOGY

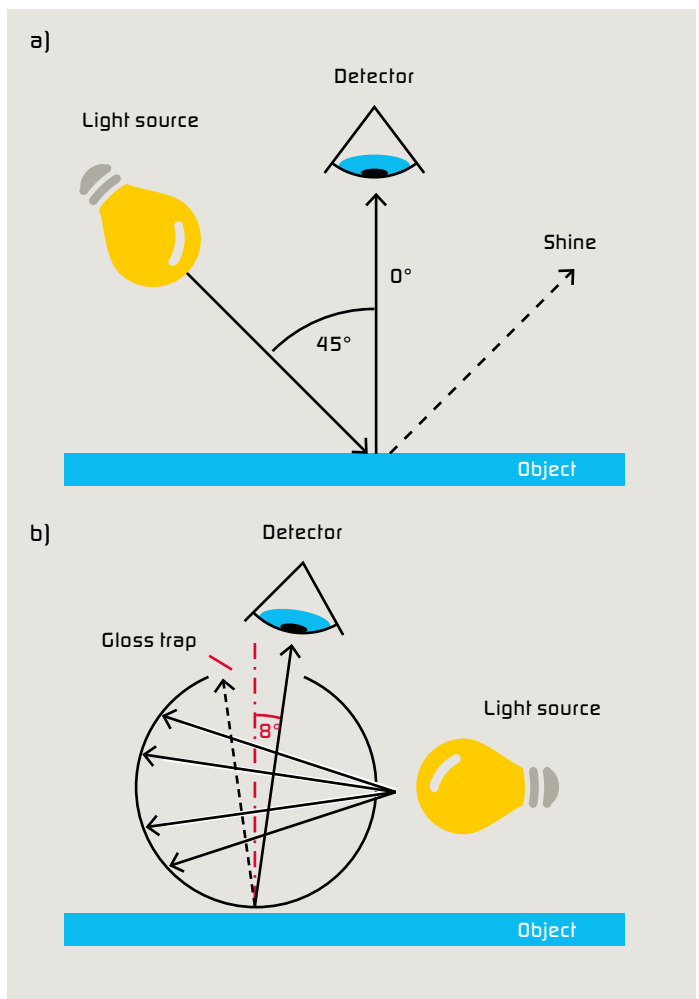
The measurement described in DIN 55979 was introduced to facilitate a better differentiation in the region of lowest reflection. This makes it possible to also determine the blackness of black coatings with a high colour depth. In addition to the suitable calibration, which is described in more detail below, this places great demands on the measurement devices themselves. In general, the measurement geometry can be varied between  $45^\circ/0^\circ$  or  $0^\circ/45^\circ$  and  $d/8^\circ$  or  $d/0^\circ$ . Which geometry is selected depends on the type of black coating and its surface texture.

The measurement of deepest-black coatings on a glossy surface, necessitates a measurement geometry of  $45^\circ/0^\circ$  (Figure 4, top). A large aperture is necessary to ensure that the measurement is as accurate as possible: it is important to detect the greatest amount of "potential" reflection. In addition, the measurement device must guarantee an accuracy in the reflection values of at least four decimal places and the software used must be able to process this as well. Replicate measurements should be characterized by very small standard deviations to keep the "device noise" to a minimum. The relevant calibration standard should be a black hollow body (light trap).

INFLUENCE OF SURFACE REFLECTION

With coatings that are less deeply black and matt or very structured surfaces, a  $d/8^\circ$  geometry can be used, for example. In this case,

Figure 4:  $45^\circ/0^\circ$  geometry (a) and  $d/8^\circ$  (b) geometry for measuring the blackness value and the undertone.



a spherical measurement geometry – the so-called integrating or Ulbricht sphere – is used to generate diffuse light (Figure 4, bottom). A gloss trap is included with this measurement geometry. In its closed condition (gloss is included in the measurement), surface-independent objective colour values are generated; something the human eye is not capable of. In its opened configuration (gloss excluded), values determined approach the perception of the human eye. These values are also particularly significantly influenced by matt and structured surfaces. In Table 2, it becomes clear that the difference between d/8° and 45°/0° measurement geometries is particularly pronounced for the deepest-black plates 3 and 4.

Gloss is an optical property that is described as the capacity of a surface to reflect strongly focused light, similar to a mirror (Figure 5). If the

gloss trap remains closed, all reflected and scattered light becomes part of the detected light. This leads to a lower blackness value with glossy samples. This is clearly recognizable when comparing the  $M_v$  values with the d/8° geometry in Table 2.

**PRACTICAL MEASUREMENTS**

As has already been mentioned in the introduction with regards to blackness values  $M_v$ ,  $M_c$ , and  $dM$ , the measurement of black coatings takes place in the  $L^*a^*b^*$  colour space with a value lower than five on the  $L^*$ -axis, or even at values lower than one for deep-black coatings. In addition to the great demands this places on the measuring technology, the most minor contaminations or even scratches on the plates to be measured can lead to large deviations between measurement results. Deviations in the reflection of as low as 0.005 can already result in great variations in the measurement result. To give an example: measuring the same plate once with and once without a fingerprint leads to significantly different measuring results. Simply wiping away the fingerprint also fails to produce the same results as an initial measurement on the clean coated surface. This becomes apparent in Figure 6 and Table 3. The table shows that the differences aren't as pronounced in areas of lower colour depth (Plate 1) as they are in higher colour depth regions (Plate 2). Only after very carefully cleaning the surface multiple times with isopropanol/water can the correct results be repeated. To assess the cleaning results, we advise to observe it under a very strongly directional light, as discreet streaks cannot be detected under normal/diffuse light (Figure 7). When measuring various samples, it should additionally be assured that the samples are always positioned in the same manner.

Figure 5: Diffuse and specular reflection of light on plane surfaces.

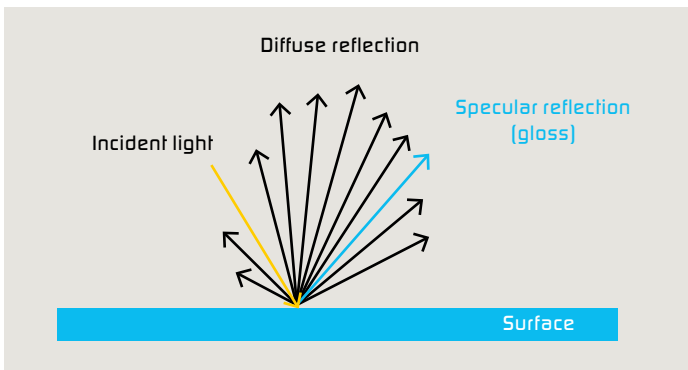


Figure 6: Comparison of a coated panel without and with finger prints and following inadequate cleaning (from left to right).

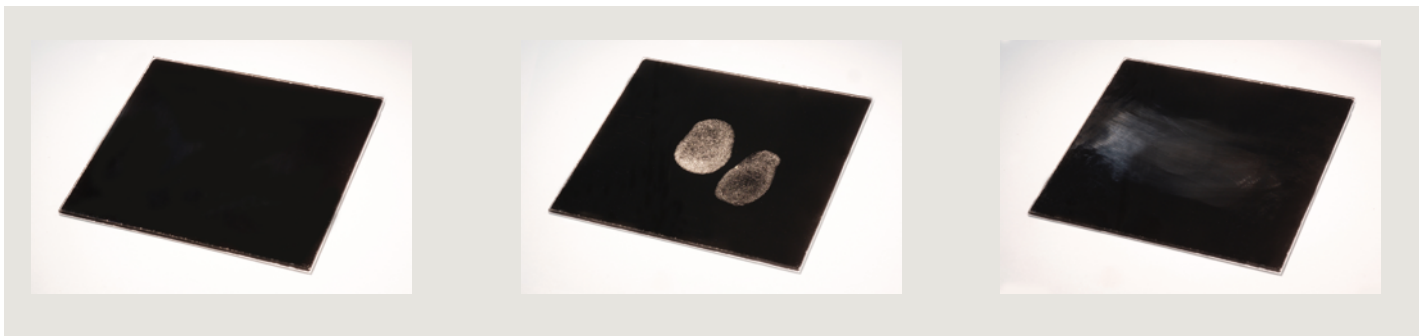
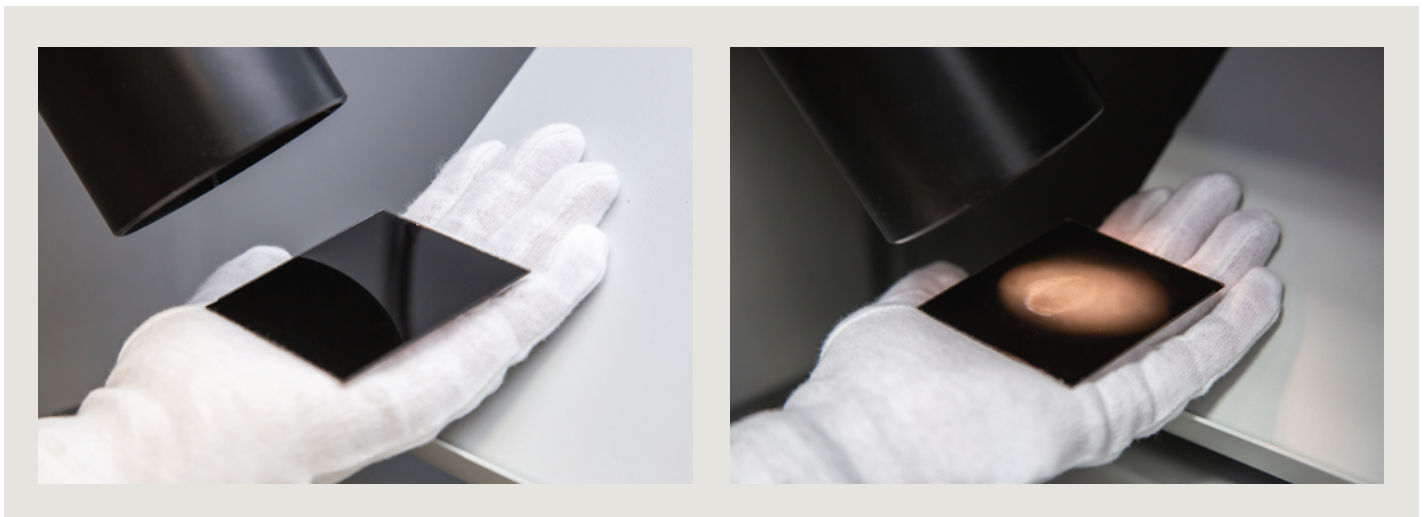


Figure 7: Coated panel with streaks observed under diffuse (left) and strong focussed light (right).



- The measuring device must ideally be set-up in an air-conditioned and clean environment, as the measurements that are performed here take place “close to device noise”, which means that deviations in the ambient temperature can influence the measurement results. If there are too many dirt or dust particles in the air, their reflection can also lead to lower blackness values. This above all also already applies during the calibration of the device.

### CALIBRATION FOR MEASUREMENTS IN THE DEEP-BLACK RANGE

Naturally, the same discerning demands placed on cleanliness (e.g. preventing fingerprints) must also be placed on the calibration plates and the gloss traps, and generally on the calibration black standard.

When calibrating with the black standard, the device is set to its “zero value”, which serves as the reference value for the samples to be measured. As such, it is of crucial importance that this zero value is determined as exactly as possible. There are various black standards available on the market. In addition to the calibration plates, black traps are common. A further variant is an air measurement that is then used to set the “black standard”. The calibration plates for blacks available on the market usually fall short of the black that one wishes to measure in the deep-black high-gloss range. The main reason for this lies in the continuous development of new, even more intensive black pigments that still need to find their way into the production of calibration plates. We have come to realise that calibration plates, common black traps, and “air” measurements are not suited to our measurement of deep

Figure 8: Colourimetric properties of different Carbon Blacks in water-borne 1K-PU coating. The mean primary particle size increases from left to right.

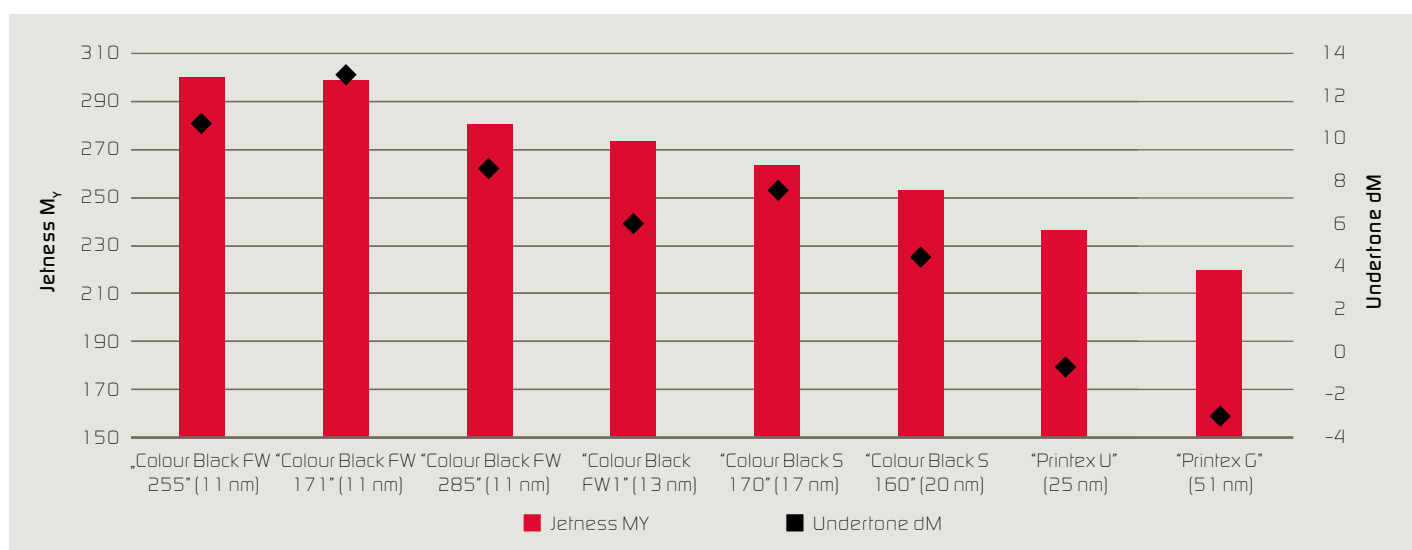


Table 2: Comparison of measurement values of plates with different blackness levels, measured with two measurement devices (45°/0° and d/8°), and comparison of measurement with and without gloss trap.

45°/0°	X	Y	Z	$M_y$	dM
Plate 1	0,4985	0,5220	0,5156	228	-3,9
Plate 2	0,0957	0,1010	0,1026	300	-2,4
Plate 3	0,0258	0,0283	0,0374	355	10,6
Plate 4	0,0237	0,0248	0,0308	361	6,0
d/8° Measurement without gloss (gloss trap open)	X	Y	Z	$M_y$	dM
Plate 1	0,4725	0,4973	0,4928	230	-3,6
Plate 2	0,0878	0,0938	0,0967	303	-1,2
Plate 3	0,0169	0,0187	0,0290	373	18,0
Plate 4	0,0171	0,0183	0,0258	374	12,4
d/8° Measurement with gloss (gloss trap closed)	X	Y	Z	$M_y$	dM
Plate 1	4,3771	4,6215	4,9224	134	-0,3
Plate 2	3,9904	4,2192	4,5352	137	0,2
Plate 3	4,4093	4,6693	5,1565	133	1,4
Plate 4	3,9832	4,2121	4,5404	138	0,3

blacks. We therefore use a custom-built black hollow body instead. This metal cylinder has an opening on one side, with a sphere mounted inside on the base. The interior is completely coated in matt black. Now, if the measurement aperture of the measurement device seals off this black hollow body, it can be assumed that virtually no light reaches the detector.


After calibration, a test is performed on black measurement plates, that correspond to the colour depth range of the sample to be measured. Measurement of the commonly used basic standard does not suffice for the measurement range requirements here; therefore, a set of six plates in the  $M_V = 228$  and  $M_V = 360$  range is used. Here, it is crucial that the entire measuring range can be detected by the measuring device before proceeding with the actual measurements.

The measurement results should always be evaluated relative to the measurement series, since using other independent calibrations in the deep-black range will introduce some discrepancies. It is thus necessary to always and immediately include the selected standard into the measurement and not to simply use it as a data template.

### COLOURIMETRIC PROPERTIES IN FULL TONE COATINGS

The jetness  $M_V$  and the undertone  $dM$  of coatings pigmented with carbon blacks are influenced by a wide range of physical and chemical parameters. Illustrative hereof are the average primary particle size, pigment concentration, the functionalisation of the pigment surface, stabilisation with additives, and of course the aforementioned measurement conditions.

Assuming that the optimal measurement conditions laid out in this article are met, the average primary particle size is the main factor influencing the jetness  $M_V$  and the undertone  $dM$ . This applies irrespective of the fact that the dispersion of carbon blacks never produces the primary particles, but only produces the superordinate structure, the aggregates. In order to measure the differences, the carbon black particles must also be well dispersed in the system. To this end, they must be stabilized in an equally appropriate manner within the binder system. In coating systems that contain organic solvents, the use of surface-modified, polar carbon black is advised. In water-based systems, non-surface-treated, non-polar carbon black is usually better suited.

Figure 8 depicts the dependence of jetness  $M_V$  and the undertone  $dM$  on the primary particle size in a water-based PU coat. Finer particles usually result in a higher black value with a blue undertone in covering coats. Coarser particles result in a lower blackness value and a brownish undertone. It is worth mentioning that the effect on the undertones is precisely the opposite with transparent colourings and grey blends. Carbon blacks with finer particles result in brown undertones, and carbon blacks with coarser particles in blue undertones. 

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Table 3: Comparison of colourimetric data of a perfectly clean plate, the same plate with finger print and after several cleaning steps.

Sample	X	Y	Z	$M_V$	$dM$
Plate 1 clean	0,5005	0,5241	0,5185	228	-3,9
Plate 1 with finger print	0,5510	0,5775	0,5816	224	-3,0
Plate 1 cleaned once	0,5099	0,5343	0,5341	227	-3,4
Plate 1 cleaned twice	0,5050	0,5291	0,5271	228	-3,5
Plate 1 cleaned three times	0,5040	0,5283	0,5257	228	-3,6
Plate 1 cleaned four times	0,5002	0,5242	0,5180	228	-3,9
Plate 2 clean	0,0232	0,0249	0,031	360	7,2
Plate 2 with finger print	0,1618	0,1707	0,1979	277	3,3
Plate 2 cleaned once	0,0930	0,1003	0,1509	300	15,6
Plate 2 cleaned twice	0,0286	0,0305	0,0420	352	11,4
Plate 2 cleaned three times	0,0289	0,0310	0,0425	351	11,3
Plate 2 cleaned four times	0,0233	0,0248	0,0312	360	7,4

“The requirements placed on correct measurement are highly discerning, but they produce very good results.”



**Dr Kai Krauss**

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### 3 questions to Dr Kai Krauss

**You write that deep-black coatings must be measured repeatedly with a minor deviation.**

**How often do you measure and what is the extent of this deviation?** When assessing the calibration, we usually measure test plates approximately five to ten times on the same spot. Only then can we guarantee that the correct value has been measured for this particular spot. The average value that is determined in this manner may only have a max. deviation of +/- 0.003 to the average Y value of this test plate that is determined beforehand using an extensive number of measurements.

**Is only one physical spot of the test coating measured or do you measure various spots of the coating to compensate for potential imperfections in the coating film?** After making sure that a measurement on a certain spot is accurate, you should additionally perform repeat measurements (5 to 10 repeats) on different spots of the coating film. How many spots you should measure is highly dependent on the quality of the coating surface. To this end, we use special glass plates, onto which we apply the coating film, after which we measure through the glass to exclude surface effects to the greatest possible extent.

**If the primary particle size influences the blackness value, the distribution of particle sizes should also play a role. Is this relevant in practice or rather negligible?** For a defined blackness value, it is crucial that the particle size distribution is very narrow. In addition to the primary particle size distribution, this also applies to the aggregate size distribution, as carbon black is always present in the form of an aggregate in the coating film. The blackness value will significantly decrease in the presence of a larger number of coarser particles. ↻

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## LITERATURE

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