

SCOPE

The analysis of low silicone coating on plastic is demonstrated.

BACKGROUND

Barrier Coatings (Packaging & Specialty Plastics)

Thin silicone coating is excellent at reducing ingress of water and oxygen into packaged products. Applications include plastics used for food packaging as well as pharmaceutical and medical packaging. Specialty plastics are also often coated with a thin silicone coating used as a barrier coating or release coating.



Thermoformed Plastics

Thermoformed plastics are used globally for food as well as consumer goods packaging. Polymer films when in contact with each other as a roll or stack have a tendency to block or stick together. Coating with a very low coat weight of silicone prevents this giving them the de-nesting characteristics the industry desires. In the logistics of producing, storing and using thermoformed plastic undercoating causes the plastics to stick together. Over-coating has the potential to negatively impact the clarity of the packaging (product inside the packaged is blurred when viewed). A typical coat weight is less than 0.2 g/m² (200 mg/m²).

Rigaku NEX QC+ analyzer meets the challenges of the coated plastics industry with several modern features:

- No need to use He purge for typical coat weights
- No special sample preparation or sample cup requirements
- Sample tray option for high throughput applications such as the labeling market

INSTRUMENTATION

Model:	Rigaku NEX QC ⁺
X-ray tube:	4 W Ag-anode
Detector:	High Performance SDD
Sample Type:	2X2 inch coupons
Safety Film:	Prolene (3.5um Mylar also suitable)
Analysis Time:	100 seconds
Environment:	Air
Aperture:	Flat window ring with aluminum hold-down ring
Options:	6-position Autosampler



SAMPLE PREPARATION

A coupon is cut approximately 2X2 inches square and simply placed in the analysis chamber over the aperture, coating side down. Any size sample can be used, as long as it is flat and covers the 26mm diameter aperture opening. Alternately, 32mm diameter coupons can be punched for batch analysis using the 6-position autosampler tray.

QUANTIFICATION UNITS

Coat weight is expressed as mass/area, the amount of silicone per unit area of plastic. While typical coat weights in the label industry in the range of 0.2-2.0 g/m² (also called gsm), the lower silicone levels used as barrier coatings and in thermoforming are mg/m² and typically fall in the range 50-500 mg/m² (0.05-0.5 g/m²).

QUANTIFICATION and CALIBRATION

To demonstrate the principle of quantifying the coat weight a simple 2-point calibration was made using two samples of known silicone coat weight of 50 and 500 mg/m². Blank uncoated BOPP was used to develop a background correction for accurate subtraction of background for each sample measured.

REPEATABILITY (Precision)

To demonstrate repeatability (precision), the low and high coat weight samples were chosen from the set of calibration standards. Each sample was measured in static position for ten repeat analyses using a total analysis time of 100 sec per measurement, with typical results shown below.

Silicone Units: mg/m ²				
Sample ID	Standard Value	Average Value	Std Dev	% Relative
50	50.0	50.1	0.5	1.0
500	500.0	500.8	2.2	0.4

TYPICAL DETECTION LIMITS

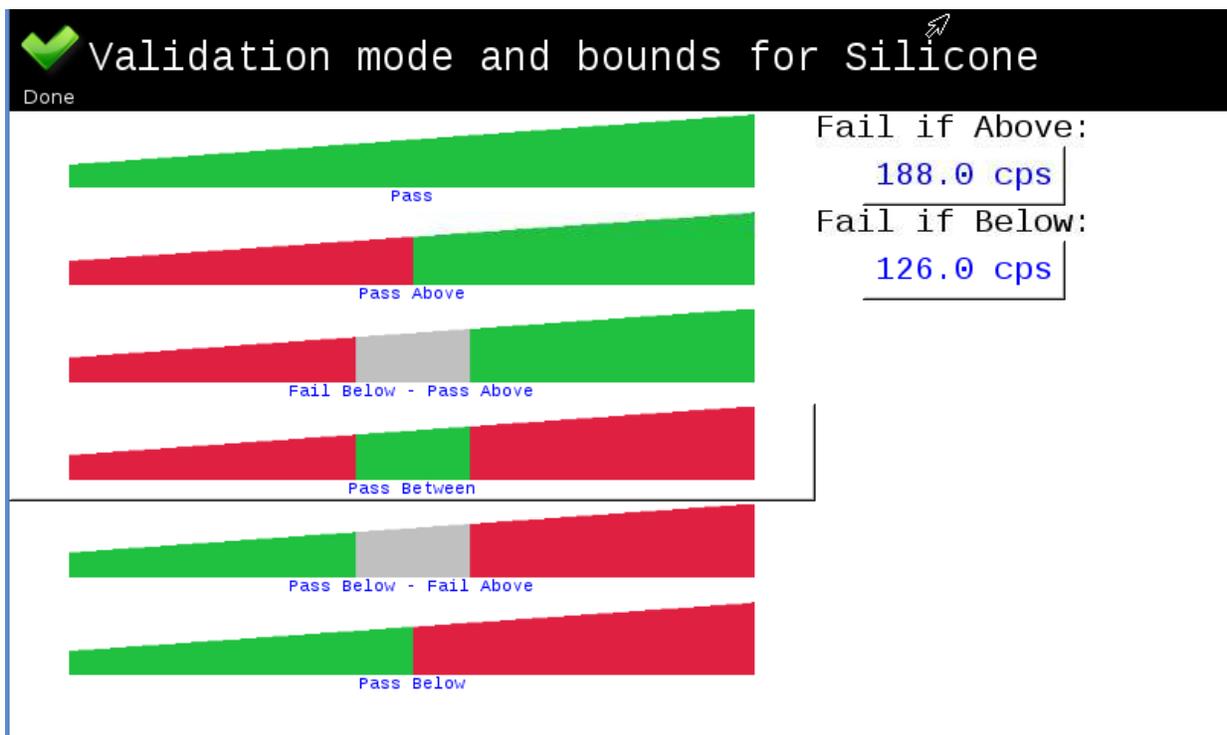
To determine the Lower Limit of Detection (LLD) using the empirical method, ten repeat analyses of an uncoated blank sample is measured and the standard deviation calculated. The LLD is then defined as three times the standard deviation.

Silicone LLD @ 100 sec	Silicone LLD @ 50 sec	Silicone LLD @ 25 sec
0.5 mg/m ²	0.7 mg/m ²	1.0 mg/m ²

SIMPLE TREND ANALYSIS

Often in the industry it is not necessary to quantify the silicone coat weight, but the need is to simply know has the silicone been applied or not. This type of trend analysis can be easily done on NEX QC+ by simply measuring the Si X-ray intensity and using the NEX QC+ Validation feature. For trend control only one sample is required with a silicone coating considered to be the good target level. The mg/m² coat weight does not need to be known. This sample is measured as a 1-point calibration and the Validation feature enabled.

In this example a good sample is used and measured to have 157 cps Si. The Pass Between Validation scheme is chosen with typical bounds entered as +/-20%.



TREND ANALYSIS RESULTS

The following examples demonstrate use of the Validation feature for trend control without the need to quantify silicone coat weight.

Sample ID:			Sample A
Timestamp:			16:00:56 2016-02-26
Instrument:			NEX QC S/N QC1283
Application Name:			Low Si cps on Plastic
Total Time:			100 sec
ID	QC	Result	
Silicone	Fail	-0.1 cps	

Sample A failed due to undercoating. Trend validation helps prevent product rejection.

Sample ID:			Sample B
Timestamp:			16:07:38 2016-02-26
Instrument:			NEX QC S/N QC1283
Application Name:			Low Si cps on Plastic
Total Time:			100 sec
ID	QC	Result	
Silicone	Pass	162.5 cps	

Sample B passes, indicating the desired amount of silicone coating has been applied.

Sample ID:			Sample C
Timestamp:			16:20:42 2016-02-26
Instrument:			NEX QC S/N QC1283
Application Name:			Low Si cps on Plastic
Total Time:			100 sec
ID	QC	Result	
Silicone	Fail	1819.6 cps	

Sample C failed due to over-coating, indicating cost overruns in the use of excess silicone material.

CONCLUSION

The typical results detailed here show the exceptional performance of the Rigaku NEX QC+ for a fast and simple measurement of silicone coatings on plastic. It is a modern and affordable tool for reducing your running costs, product rejection and product waste.