

April 19, 2017

▪ **TEST REPORT** ▪

**PN 134565**

PO CC

**Chemical Analytical Services**

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Rev 041916



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**Subject:** Analytical testing on samples submitted by the above referenced customer

**Received:** One (1) container of carbon black

**IODINE ADSORPTION NUMBER; ASTM D 1510, Method A**

A weighed sample of carbon black was treated with a portion of standard iodine solution. The mixture was shaken and the carbon black was separated from the iodine. The excess iodine was titrated with standard sodium thiosulfate solution. The absorbed iodine is expressed as a fraction of the total mass of carbon black.

**TABLE 1 - IODINE ADSORPTION NUMBER**

<u>SAMPLE ID</u>	<u>IODINE ADSORPTION NUMBER, g/kg</u>
Carbon Black	109

**DIBUTYL PHTHALATE ABSORPTION NUMBER (DBP); ASTM D 2414, METHOD B**

DBP was added by means of a constant-rate buret to the carbon black in a mixing chamber. As the DBP was absorbed, the viscosity of the mixture increased. When the viscosity reaches a predetermined torque level, the buret and mixer shut off. The volume of DBP used was reported by the software. The result is expressed as the volume of DBP needed for 100g of sample.

**TABLE 2 - DIBUTYL PHTHALATE ABSORPTION NUMBER (DBP)**

<u>SAMPLE ID</u>	<u>DBP ABSORPTION NUMBER cm<sup>3</sup>/100g</u>
Carbon Black	93.5

**CARBON BLACK ASH; ASTM D1506, METHOD A**

A pre-dried sample was weighed before and after combustion for 1.5 hours at 550°C. The remaining ash was weighed and calculated as a percent of the dried sample.

**TABLE 3 - CARBON BLACK ASH**

<u>SAMPLE</u>	<u>ASH, %</u>
Carbon Black	24.08

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**TOTAL SULFUR; LECO METHOD<sup>^</sup>**

Sample was combusted in an oxygen atmosphere where the sulfur oxidized to SO<sub>2</sub> gas. The gas was measured by an infrared cell and the results calculated by the microprocessor.

**TABLE 4 – TOTAL SULFUR**

SAMPLE	TOTAL SULFUR, %
Carbon Black	2.43

**CARBON BLACK TYPE; ASTM D3849-95a**

**Application**

This method applies principally to carbon black obtained by the pyrolysis of vulcanized rubber compounds or molded parts. The technique can be used on raw, uncompounded black as well.

**Sample Preparation**

The sample was vacuum pyrolyzed for one hour and was treated with hydrofluoric acid (HF) to remove silica and other mineral fillers. The carbon black was then dispersed in 1 ml of chloroform, diluted, and a single drop of the dispersed black was placed on several carbon-coated 200 mesh copper grids for analysis by Transmission Electron Microscopy (TEM).

**Analysis**

The pyrolyzed carbon black was examined by TEM using a Philips Model CM12 interfaced with a Gatan Model 780 Dual Vision imaging system. Individual particles were examined and only those particles with at least half a diameter visible were measured using Jandel Scientific software (SigmaScanPro<sup>®</sup>). The data was transferred to an Excel spreadsheet, and statistics and a histogram were obtained. Table 5 is a summary of the sample particle size data. For more detailed descriptive statistics of the carbon black primary particle size data, see Table 2. Figure 1 is a particle size histogram for the sample.

**TABLE 5 – Emergent Waste P/N 134565 – Sample: Carbon Black**  
Summary of Carbon Black Primary Particle Size Data

Sample	Average Particle Size, nm	Standard Deviation, nm	n (Number of Particles Counted)	Maximum Particle Size, nm	Minimum Particle Size, nm	Estimated Type	ASTM Std. nm
Carbon Black	24.19	8.72	200	46.09	6.68	N200	20-25



**Discussion**

The pyrolyzed black was processed and imaged according to the latest version of ASTM D3849, "Standard Test Method for Carbon Black-Morphological Characterization of Carbon Black Using Electron Microscopy." Carbon black particle diameters were measured according to ARDL Procedure 3803. The resulting statistics include the mean particle size, standard deviation and size range for the sample.

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The right-most column in Table 5 indicates which traditional ASTM grade the results are comparable to based strictly on particle size. This is simply a suggestion to assist the rubber compounder since it corresponds to the sample's effective particle size.

Keep in mind that statistical analysis comparison to known particle size carbon black types are only suggestions as to what carbon black was used in the sample. There is considerable overlap in the primary particle size between different carbon black types from different suppliers. The precise carbon black to be used in a reverse-engineered or reconstructed compound should be determined by the ultimate physical properties desired in the final product.

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# Emergent Waste P/N 134565 Not Identified Carbon Black Particle Size Distribution

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*Descriptive Statistics, nm*

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Mean	24.19
Standard Error	0.62
Median	23.20
Mode	13.06
Standard Deviation	8.72
Sample Variance	76.06
Kurtosis	-0.15
Skewness	0.50
Range	39.41
Minimum	6.68
Maximum	46.09
Sum	4837.89
Count	200.00

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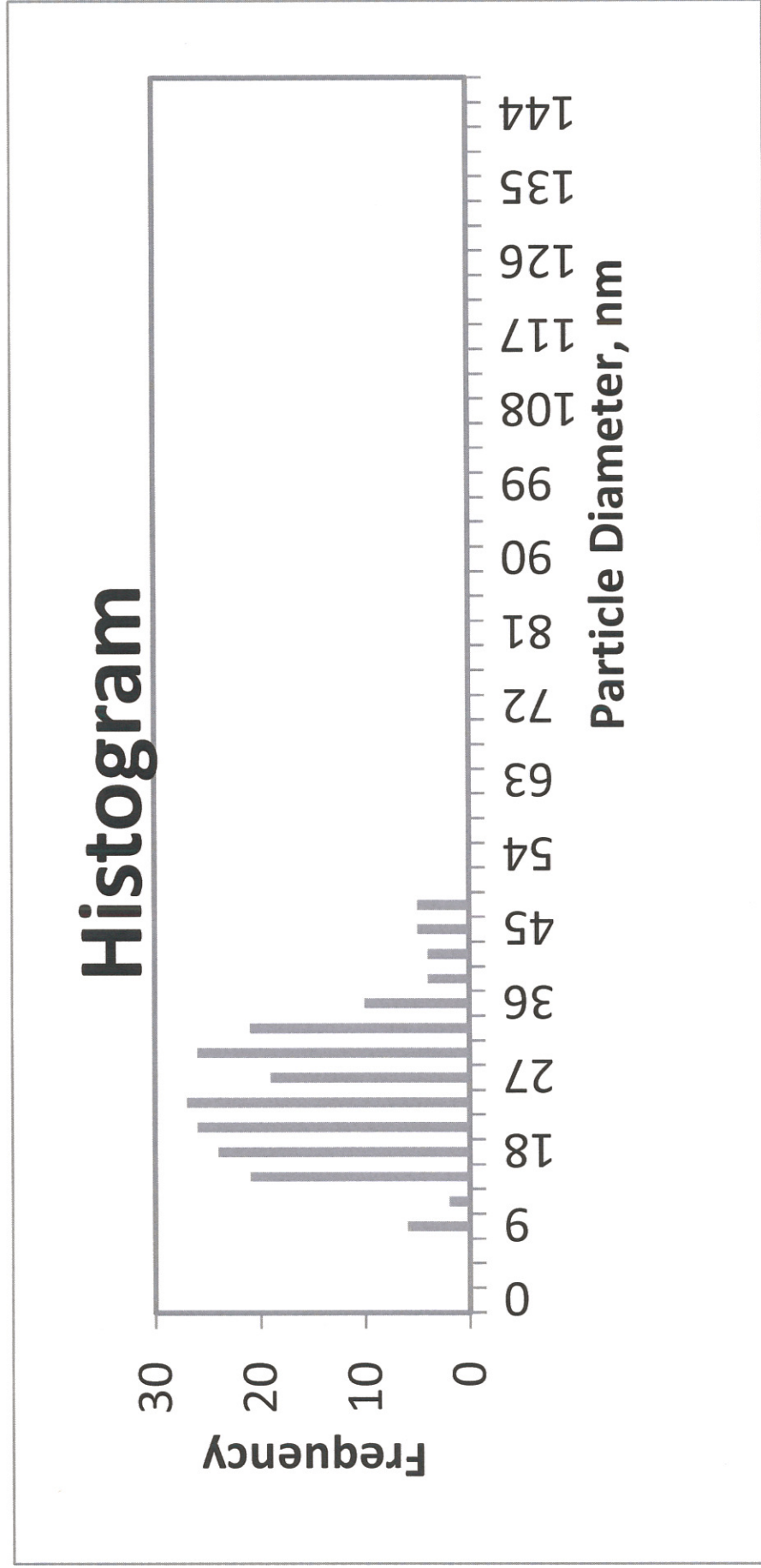


Figure 1



