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Procedure for sampling, weighing, and analysis of cobalt hydroxides.

GUIDELINE

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The Cobalt Institute (CI) is the global association representing the cobalt industry and value chain. Our Members are producers, users, recyclers, and traders of cobalt. We promote the sustainable and responsible production and use of cobalt in all its forms.

The CI plays a critical role in advancing the interests of our members helping to grow and protect the market for cobalt. We are a knowledge center for governments, agencies, industry, the media, and the public on all matters concerning cobalt and cobalt-containing substances.

Membership of the CI allows organizations to stay abreast of global regulatory and policy developments that will affect their businesses, and contribute to the development and execution of global strategies which focus on:



Global Chemicals Management - The CI is a leader in the human health and environmental science of cobalt in the workplace and the environment - through the CI, members have invested over €30m to ensure regulatory compliance and market access across the globe;



Responsible Sourcing - The CI and its members are playing a leading role in addressing the issues around the sourcing of cobalt and have launched the Cobalt Industry Responsible Assessment Framework (CIRAF) to help members meet current challenges in raw material provenance;



Sustainability - The CI develops crucial data that helps make cobalt a raw material of choice for downstream users and informs the decisions of regulators and policymakers. We have developed the first cobalt industry life-cycle assessment (LCA) and have recently published a Socio-Economic Analysis (SEA) highlighting the economic value of the cobalt industry; and



Government and Public Affairs - Recognised as the authoritative voice of the cobalt industry by many governments around the world, the CI ensures its members have a place at the table when big decisions are made.

Supported by our knowledge base, the CI is a proactive organization which engages with governments, regulators, media, academia, and civil society to positively impact and influence the regulatory environment for cobalt.

1. Introduction

This best practice guideline* details a method to gather representative samples from cobalt hydroxide products to provide material for chemical analyses, including cobalt (Co) content and measurement of moisture content (loss of mass on drying). Cobalt hydroxide products can be physically and chemically heterogeneous, susceptible to segregation during shipping and storage, and are extremely hygroscopic (have a strong tendency to absorb moisture from the air).

This guideline is designed to promote the standardization of weighing and sampling methodology, with a view to minimizing sampling error, increasing the accuracy and precision of chemical analyses of representative samples collected from product lots, and improving quality and reliability for cobalt hydroxide producers, consumers, and others in the industry (hereon referred to as "counterparties").

2. Scope

This guideline applies to semi-refined cobalt hydroxide products packaged in large bags (IBCs - intermediate bulk containers, otherwise known as "big bags").

3. Definitions

- **Lot:** the quantity of material to be sampled - for example, several trucks / containers.
- **Sub-lot:** a smaller portion of the entire body of material under investigation - for example one truck / container.
- **Bag:** one single IBC / big bag from a sub-lot or lot.
- **Increment:** the quantity of material collected in a single operation of the sampling device.
- **Representative sample / sample:** a small quantity (subset) of material that reflects the same properties that exist in the lot with both precision and error in acceptable bounds. The size of a sample may be greater than or less than the size of an increment.
- **Sampling:** method by which a representative sample is collected.

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- **Sampling spear:** used for collecting increments of material from sacks, bags or plastic drums, if these goods are in powder or granulated form. A metal or plastic sampler made of stainless steel or polypropylene (PP). Standard length 1 m.
- **Constant weight:** when the calculated additional loss of mass between two sample weighings separated by an interval no shorter than 4 hours, is less than 0.1% of the initial sample mass.
- **Calibration:** the process of comparing and adjusting measurement systems or procedures against Certified Weights, Temperatures, or against Certified Reference Materials. This process is carried out strictly in accordance with International Standards and should be documented.
- **Error:** the degree to which the result of a measurement, calculation, or specification deviates from the true value.
- **Precision:** the degree to which the result of a measurement is repeatable, reliable, and the same each time.

4. Procedure

The measured characteristics of collected samples are used to infer properties of the lot. How these samples are collected, handled, and stored, will therefore determine how accurately and precisely the samples represent the lot. This guideline seeks to reduce the variability in data obtained by multiple counterparties analyzing the same lot by outlining a best practice common method that can be used for sampling and analysis, and such that the integrity and composition of the samples during sampling, transportation, moisture determination, and sample preparation, be maintained (Figure 1).

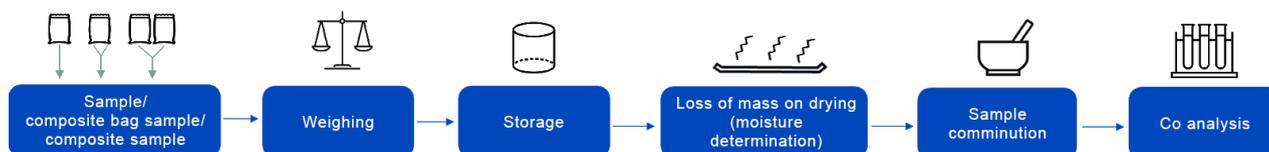


Figure 1. A schematic of steps in sample collection and analysis described in this guideline.

4.1. Sampling

The broader sampling scheme, including the position(s) in the bag where samples are collected, and how many samples are collected from each lot, are outside the scope of this guideline. Best practice is that the numbers of samples collected and analyzed, should adequately reflect the heterogeneity of



the material. In the absence of variance data, the sampling plan for a given consignment should assume high variability of analytes such as Co and moisture. The sampling procedure employed, including the number of samples to be collected, and position(s) samples are collected from in the bag should be agreed, and duplicated by counterparties. An incorrect sampling scheme will increase error in measurements and may decrease precision in measurements made by counterparties.

4.1.1. Frequency of samplings from lots and number of samples to be collected

The frequency of bags to be sampled and the number of increments to be collected from each bag, is to be agreed between counterparties. Whether multiple increments are to be combined to produce a composite bag sample, a composite sub-lot sample, and / or a composite lot sample is to be agreed between counterparties. If a natural state sample of the cobalt hydroxide material is to be held in reserve should there be an error, or a dispute arise, is also to be agreed between counterparties.

4.1.2. Sample collection

Samples of increment mass should be drawn from a full cross section of the bag by sampling spear. Other mechanical devices may be used to collect samples provided the method permits sampling of a full profile of the bag. Due to the hazardous properties of the material, best practice is to gather increments from a full vertical profile of the bag from a natural opening and without piercing or otherwise compromising the integrity of the bag. Care should be taken to fully account for potential stratification of the material within the bag.

4.1.3. Material subdivision (if applicable)

To follow best practice, increment(s) collected should not be subdivided or split prior to moisture determination (see section 4.4.). This approach best enables analysis of the full bag profile and minimizes sample handling. In cases when moisture analysis is possible only on a subsample of the collected material, and / or when a natural state sample of the cobalt hydroxide material is to be held in reserve, and / or a duplicate sample is needed to calculate milling time from a sacrificial sample, the material should be homogenized by an appropriate method prior to division of the material and weighing of the formed samples.

4.2. Weighing of samples / subsamples

Samples should be weighed immediately upon collection to 0.1 g using a certified and calibrated commercial balance. Best practice is to weigh the sample directly into an appropriate pre-weighed vessel (see section 4.3.).



4.3. Storage of samples

Samples should be stored and transported in clean and ideally air-tight vessels, for example aluminium foiled air-tight bags, with a minimum of airspace. This is to mitigate potential changes in moisture content.

4.4. Loss of mass on drying (moisture determination)

4.4.1. Sample handling

The stored sample should be mixed in the air-tight vessel to recover moisture lost to vessel surfaces, and immediately weighed. The sample weight should be compared to the weight of the sample measured immediately after sample collection to calculate moisture lost during storage. When moisture analysis is possible only on a subsample of the collected material, comparison of moisture determination results of two duplicate samples is also recommended as best practice.

4.4.2. Drying

The sample(s) should be distributed evenly on a clean stainless-steel tray to a maximum depth of 3 cm. Large lumps of material should be broken up with an appropriate device, and the material should then be dried to constant net weight in calibrated commercial ovens at $105 \pm 5^\circ\text{C}$ (see section 3. Definitions). After an initial drying period of 24 hours, or a longer empirically determined and mutually agreed initial period, check weighing should be performed thereafter in intervals no shorter than 4 hours until total moisture loss has been verified. All weighing should be done with hot samples (weighed immediately from ovens) and using a tared commercial and calibrated balance with precision of at least 0.1 g. This will minimize moisture gain from the ambient air during cooling which may otherwise interfere with accurate measurement of the material (dry) weight. A heat-proof mat is recommended for use with balances.

Loss of mass on drying (%) is calculated according to equation (1):

$$(1) \text{ Loss of mass (\%)} = ((\text{weight at } t_{\text{start}} - \text{weight at } t_{\text{end}}) / \text{weight at } t_{\text{start}}) \times 100$$

Where t = time

Data should be expressed to two decimal places. The acceptable difference between the two duplicate moisture samples is $< 0.3\%$.



4.5. Sample comminution

The dried material should be milled, screened, mixed thoroughly, and then subdivided to form subsamples for Co analysis. The whole sample must be processed. Milling should achieve a particle size of > 95% less than 150 µm. Oversize and screened material must be recombined and thoroughly mixed before subdivision of material to form subsamples for Co analysis. Best practice is to use a sacrificial sample / subsample to empirically determine the milling time required to achieve this particle size. The sacrificial material should be separate to the main sample and should be discarded after analysis.

4.5.1. Sample storage for chemical quality analyses

After weighing, the dried subsamples are to be collected into air-tight vessels for later chemical analyses.

4.6. Co analysis

It is best practice that common samples be used for both moisture determination and measurement of Co content. The numbers of subsamples to be analyzed for Co content, should be agreed between counterparties.

4.6.1. Sample handling

To correct for possible moisture gain in the time between sample comminution and subdivision (see section 4.5) and Co analysis of subsamples, subsamples should be dried again in an oven to remove the hygroscopic moisture content for a period of 16 hours at $105 \pm 5^\circ\text{C}$. Dried subsamples should be immediately sealed and retained within a suitable laboratory desiccator until ready to be used for the chemical analysis. Alternatively, the moisture content of the subsample may be determined concurrently with the analysis of Co content in the subsample e.g., with a halogen lamp moisture analyzer.

4.6.2. Sample Co measurement

The Co concentration in subsamples of the material should be determined by appropriate means using calibrated instrumentation. Best practice technique is to apply hygroscopic moisture correction principles concurrent with the weighing of the test sample correcting the final Co value for the hygroscopic moisture. Examples of analytical methods used for the determination of Co are the classical potassium ferricyanide potentiometric 'back' titration (incorporating manganese correction), wavelength dispersive X-ray fluorescence (WDXRF) using fused borax glass bead and internally standardized inductively coupled plasma optical emission spectroscopy (ICPOES).



4.6.3. Data handling

The Co content of the material (and content of other elements, if measured) should be reported on a dry weight basis, having been corrected by the moisture determination result.

5. Safety information

This guideline involves materials and processes that can be hazardous. It is the responsibility of users of this guideline to assess and implement appropriate safe working practices prior to use.





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