The state of organized sampling work for minerals in China since WCSB9

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The state of organised sampling work for ore products in China since WCSB9 is introduced in this paper, including national standards, industry standards and group standards in China. In particular, the group standards organized by CSTM Material Sampling and Blending Technical Committee are introduced, meanwhile, the development of automatic sampling and sample preparation system, slurry on-line samplers and multiplexer dividers, on-line sampling and LIBS analysis technology integration system in China in recent years are introduced.

Key words: Chinese standards, sampling work, mineral, automatic sampling and sample preparation system, on-line sampling

Preface

In May 2019, WCSB arrived for Asia and China for the first time. The industry's top event shared the cutting-edge sampling and blending theory knowledge, covering mining, food, pharmaceutical, agriculture, biology, environmental protection and many other aspects. Participants learned valuable experiences and cases from internationally renowned experts and promoted academic and practical exchanges.

The actual demand and market space for sampling theory and technology in China is huge. The relevant sampling research in China has just started, which is far from meeting the needs of reality and lags far behind the research development and practical application of international advanced sampling theory. WCSB9 promoted the research of sampling theory and practice in various related fields in China.

In the past two years, China has made some achievements in the standardization of sampling and preparation, especially the group standards organized by CSTM Material Sampling and Blending Technical Committee, which is committed to the unification of sampling terms and supervision and management standards in the Chinese inspection market.

Chinese standards for sampling and preparation of minerals since WCSB9

Table 1 lists the major standards for the sampling and preparation of minerals in China After 2019. The table mainly describes the sampling methods of precious metal concentrate, ferroalloy products, waste palladium carbon, zirconium and zirconium alloy, gold nugget, copper, lead anode mud, bulk flotation copper concentrate, etc.

Table 1. Chinese standards for sampling & sampling preparation of minerals since WCSB9

rank	Chinese standards
1	YS/T 1328-2019 Methods for sampling and sample preparation of precious metals concentrates
2	GB/T 13247-2019 Methods for sampling and testing the grain size of ferroalloys products
3	GB/T 39292-2020 Methods for sampling and sample preparation for analysis of palladium carbon wastes
4	YS/T 1466-2021 Methods of sampling and sample preparation for chemical composition analysis of zirconium and zirconium alloy
5	GB/T 13449-2021 Method for sampling and sample preparation of gold lump ores
6	YS/T 87-2021 Methods for sampling and sample preparation of copper anode mud and lead anode mud
7	YS/T 96-2021 The method for sampling and sample preparation for gold and silver contents assaying in the bulk flotation copper concentrates
8	HG/T 5965-2021 Waste printed circuit board—Sampling and sample preparation procedures

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9	T/CGA 025—2021 Method of sampling and sample preparation for tailing of gold mine
10	T/CSTM XXXX-202X Sampling and sample preparation terms and definitions of minerals
11	T/CSTM XXXX-202X System for supervision and administration of sampling and sample preparation of mineral products
12	T/CSTM XXXX-202X Intelligent Robotic Preparation System of Solid mineral
13	YB/T 4849-2020 The technical specification of on-line automatic sampling, sample preparation, particle size analysis and the determination of tumbler strength for sintering ore

YS/T 1328-2019 ‘Methods for sampling and sample preparation of precious metals concentrates’ specifies the procedures and methods for sampling, blending and moisture determination of precious metal concentrates. The quality fluctuations are divided into three types: large, medium and small, ≥2.5% is large, 1.0%～2.5% is medium, ≤1.0% is small, obtained the minimum number of sampling at the desired precision at 95% probability is given. The method conforms to the quality fluctuation of the goods and the obtained samples by this method are more representative, which is suitable for the sampling and preparation of precious metal concentrates transported by conveyor belts, trucks, bins, ships, piles, and container bags. The standard emphasizes that the sample meets the requirements of analysis particle size.

GB/T 13247-2019 ‘Methods for sampling and testing the grain size of ferroalloys products’ is a revision of the old standard. The old standard was adopted by ISO 4551:1987 ‘Ferroallys-Sampling and sieve analysis’. Deoxidation and alloying of molten steel are realized by adding ferroalloy. This process has been changed from manual or feeding car to automatic feeding tube. Due to the limitation of automatic feeding system, the particle size of ferroalloy is strictly controlled. The old national standard GB/T 13247-1991 didn’t have specific testing procedures. Through the collection of relevant standard data, collection of large steel mills ferroalloy particle size sampling method, manganese thin ferroalloy particle size analysis test and sieving test, etc. The revised national standards mainly added "normative reference documents", "equipment and tools", "crushing" operation, particle size 10mm and the following test screening requirements, manual screening operation requirements, mechanical screening operation, inspection report, etc., and modified sampling shovel sampling operation requirements, screening results dissent treatment, etc. The revised standard refines the granularity detection procedure and makes it easier to operate.

GB/T 39292-2020 ‘Methods for sampling and sample preparation for analysis of palladium carbon wastes’ stipulates the general procedures, general rules, sampling pretreatment, sampling, sample preparation, marking and storage requirements of sampling and sample preparation for palladium carbon wastes. In the process of transportation and sampling, it is difficult to obtain homogeneous and representative samples because there are moisture and organic volatilization, light specific gravity, large volume, mixed liquid and solid, integral or partial pulping of palladium carbon waste. This standard provides for the use of incineration before sampling, high temperature carbon removal can make the weight and volume greatly reduced to 1% of the original, at this time sampling becomes easy, and can be very good to obtain a uniform representative sample.

GB/T 13449-2021 ‘Method for sampling and sample preparation of gold lump ores’ is a revision of the old standard. The relationship between quality fluctuation, maximum particle size and sample size is increased. The parallel sampling method of unloading truck in the material yard and the sampling method of bagged bullion ore are added. Sample size requirement changed from -150 mesh to 0.074mm.YS/T 87-2021 ‘Methods for sampling and sample preparation of copper anode mud and lead anode mud’ is a revision of the old standard too. In recent years, anode mud as an important by-product of copper and lead smelting enterprises, its trade share in the market is increasing. Due to the characteristics of copper and lead electrolysis production process and the complex composition and high value of anode mud, the original YS/T 87-2009 sampling method can no longer meet the current demand of trade, especially the lack of sampling method of ton bag packaging anode mud, resulting in the inspection dispute between the supply and demand sides in the anode mud trade. Through the revised standard, the inspection batch, sampling tools and sampling methods of anode mud in different packaging forms were standardized, to play a guiding role in the internal and external delivery inspection of anode mud, and to control inspection risks and reduce trade disputes by improving the representativeness of samples. The stratified oblique insertion sampling method is added in the new standard.

YS/T 96-2021 'The method for sampling and sample preparation for gold and silver contents assaying in the bulk flotation copper concentrates'. The standard is applicable to sample preparation for analysis of associated gold (Au:1g/t ~ 30g/t) and silver (Ag:20g/t ~ 2000g/t) in bulk flotation copper concentrate. The copper concentrate with gold and silver content not in its range can follow this method. The determination precision β_M, sample preparation precision β_p and sampling precision β_S values were determined by precision test. The total precision β_{SPM} in the basic batch was finally determined by comprehensive test data and the safety and economy of sampling, which were modified according to international practice and GB 14260. The total precision of bulk flotation copper concentrate with gold grade of 1g/t ~ 15g/t is 1.6 and that with gold grade of ≥15g/t~30g/t is 2.5. The total precision of silver grade in 20g/t~500g/t is 20, and that of silver grade in ≥500g/t~2000g/t is 20~80.

'Sampling and sample preparation terms and definitions of minerals', 'System for supervision and administration of sampling and sample preparation of mineral products', 'Intelligent Robotic Preparation System of Solid mineral' are group standards organized by CSTM Material Sampling and Blending Technical Committee. These standards are still in the development stage and only have draft versions at present, which are expected to be approved in November 2022.

The purpose of the standard 'Sampling and sample preparation terms and definitions of minerals' is to unify the definition of terms, the name of each process and the specification of equipment involved in the process of mineral product sampling and preparation, standardize the technical indicators of the intelligent system currently used in the market, improve the operation consistency of sampling personnel, and improve the representativeness and uniformity of test samples. Standardize the English translation of terms, in line with international advanced standards, to facilitate the unification and communication of sampling and preparation in international trade.

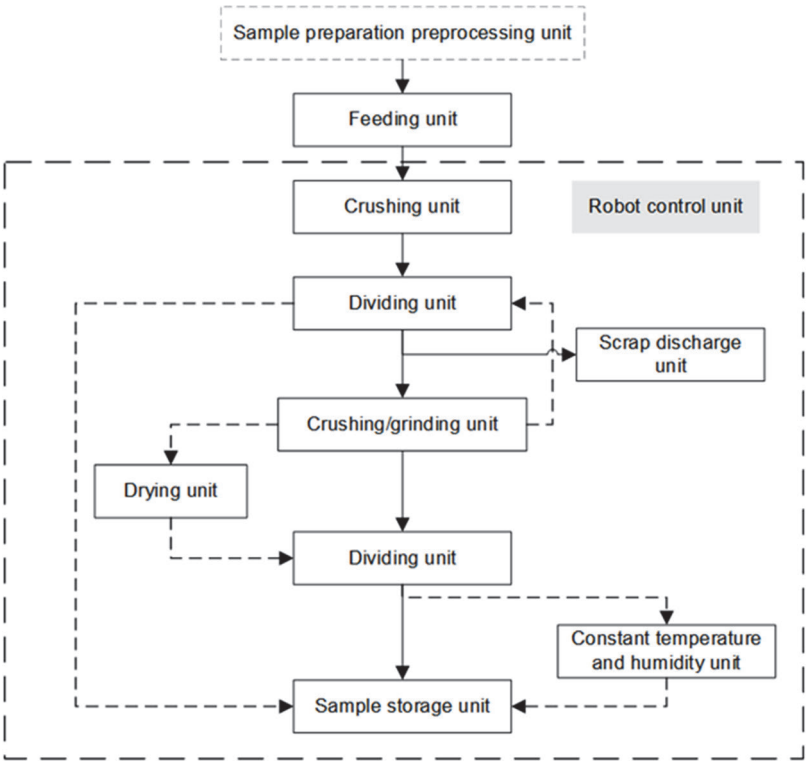

Figure 1. Schematic diagram of solid mineral robot sample preparation system

The purpose of the standard 'System for supervision and administration of sampling and sample preparation of mineral products' is to recommend the use of a sound sample sampling supervision system, to avoid the purposeful sampling and biased sampling. The standard mainly from the following eight aspects of standardization requirements, the formation of the whole sampling process supervision system:

1. Barcode encryption system
2. Automatic sampling system
3. Video surveillance system
4. Preparation before sampling
5. Sampling on site
6. Sample transfer
7. Sample preparation monitoring system
8. Personnel management

The standard 'Intelligent Robotic Preparation System of Solid mineral' has two parts. Part 1 is 'General Technical Specification'. This part specifies the system composition, use environment, workflow, marking, packaging, transportation, and storage requirements of solid mineral robot sample preparation system. Schematic diagram of solid mineral robot sample preparation system is outlined above in Figure 1. Part 2 is 'Performance Inspections'. This part specifies the terms and definitions, test items, test preparation, test methods and requirements, test period, test report and other requirements in performance test and acceptance of solid mineral robot sample preparation system. The main projects of performance test of solid mineral robot sample preparation system include:

1. Stand-alone test
2. Water adaptability test
3. Processing capacity test
4. Precision test of the whole machine
5. Bias test

The performance test shall be valid for two years and shall be conducted immediately in the following cases:

1. Design, production, or commissioning of a new system
2. When the system feeding, crushing, dividing, drying, pulverizing and other key components are replaced;
3. Higher requirements or doubts on sample preparation precision or bias;
4. When the variability of coal increases significantly.

The Development of Automatic Sampling and Sample Preparation System

In recent years, the cargo volume of bulk ore products in China is on the rise, and the sampling and detection are heavier, so it is particularly important for the automatic sampling and preparation equipment and system of all kinds of minerals at the port. With the rapid development of mechanical automation and internet of things technologies, the functions of the automatic sampling and preparation equipment are more diversified, the technology are more standardized, and the operation more intelligent, which have been promoted in more and more ports, and the business environment at ports has been significantly.

Sensor of iron ore mechanical sampling facility

With the development of intelligent technology, new sensing technology has been introduced in iron ore sampling facilities. Programmable controller (PLC) of iron ore mechanical sampling and sample preparation facilities, to issue action instructions to the equipment, it must get the status information and relevant measurement information of each component. This information is generally obtained through low-voltage electrical appliances and sensors installed in various parts of the sampling and sample preparation facilities. Commonly used sensors include weighing sensors, position sensors, moisture sensors, etc., which respectively undertake the position information and weighing information of the sampling and sample preparation facilities. In principle, the component equipment of the sampling and sample preparation facilities can be controlled separately, and each equipment is equipped with machine side control facilities, and the design is the machine side priority.

The sensor technology of sampling and sample preparation facilities has been used in iron concentrates grade sensing technology, quality fluctuation check prediction technology, microwave technology, isotope nuclear technology, video and image recognition technology, etc. The development of these intelligent technologies makes some processes of the facilities replace the original work that requires a lot of physical strength, improve the hardware sensing to the level of software sensing, and makes the iron ore mining sensing technology more stable and reliable, reduce the cost and improve the efficiency.

The 4th generation of mobile intelligent sampling vehicle

The Port mobile intelligent sampling vehicle (3rd Generation) with automatic feeding function has solved the problem of sampling in any location and area. However, when it's working in the small space, the body cannot be moved well. This leads to a single product sampling point, which does not represent the product very well, and it has an impact on the results. The 4th generation of intelligent sampling vehicle is designed to solve this problem. The vehicle can collect samples at multiple points and operate in a narrow space, with the small sampling error, well representative, high efficiency, and saving manpower. The third and fourth generation sampling vehicles are respectively shown in Figure 2.

Figure 2. The 3rd generation (left) and fourth (right) of mobile intelligent sampling vehicle

The 4th generation of intelligent sampling vehicle mainly includes the feeding manipulator, lifting and feeding device, automatic blending device, conveying device, rotary division device, discarded sample output device and collector set on the vehicle body.

The feeding manipulator takes part of the bulk or powdery material as the sample material. The receiving hopper with the sample material rises from the bottom of the lifting and feeding device to the top of the device for unloading. The sample material received by the receiving hopper flows out of the outlet of the automatic blending device and is transported by the conveying device to the rotary division device with an up and down sampling trough. The discarded sample output device and the sample receiving conveyor are located in the lower position of the device. The sample discharged by the rotary division device is transported to the collector, and the sample material that is not sampled flows to the output of the discarded sample output device.

The 4th Generation Intelligent Sampling Vehicle adopts a 6-axis manipulator, with a shovel, grabber or spiral sampling head for sampling. Realizing the random determination of the number and location of sampling points, and automatically complete the processes of sampling, lifting, primary division, sample abandonment and secondary division. The samples are more representative without artificial errors.

Manipulator sample preparation system

The manipulator automated sample preparation system is designed to solve the problems of large number of samples collected, low efficiency of manual collection alone, unstable sampling performance, etc.

The system comprises at least two sets of sample preparation units and a movable manipulator located between the two sets of sample preparation units, the sample making unit comprises a sequentially set of the first crusher, the first dividing machine, the second crusher, the second dividing machine, the dryer, the weighing device, the third dividing machine, the grinding machine, the bagging machine, the manipulator can be moved to the first crusher, the first dividing machine, the second crusher, the second dividing machine, dryer, weighing device, the third dividing machine or grinder position feeding or unloading.

The sample preparation system is mainly suitable for the preparation of a large number of samples, fully realizes the automation of material sampling, can randomly determine the number of sampling points and locations, automatically complete the sampling, sample preparation, crushing, dividing, sampling, residual material return, air transmission system, automatic storage cabinet and other whole process, without any human factors involved, the sampled products are strongly representative, can correctly reflect the authenticity of material quality, and have obvious advantages in safety, environmental protection, energy saving, high efficiency and other aspects.

Linear and Ring manipulator sample preparation system as shown in Figure 4 are suitable for processing a large number of samples of the preparation, It fully realizes the automation of material sampling and preparation

without any human factors. Up to 9 particle size levels can be measured simultaneously. The sieving and granulation level could adjust flexibility and good sample representation.

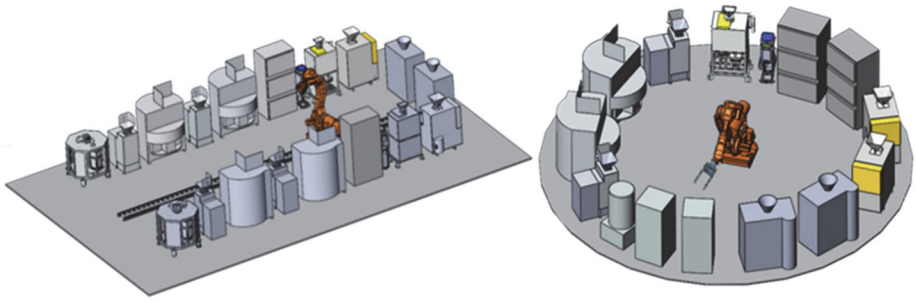

Figure 4. Linear manipulator (left) and Ring manipulator (right)

Automatic robot sample preparation system

The Figure 5 shows the automatic robot sample preparation system. Sample preparation scheme is flexible. The whole process of sample entry and exit can be weighed, sample preparation is transparent and visible, less residue, less error. The sample can be automatic packaged and transmission.

Automatic robot sample preparation system has built-in various sample preparation schemes such as sample preparation process, drying time, division ratio etc. Automatic and flexible adjustment according to the sample parameters, remote setting and the system automatically select the sampling scheme. The built-in mixing device can ensure the representativeness of the sample.

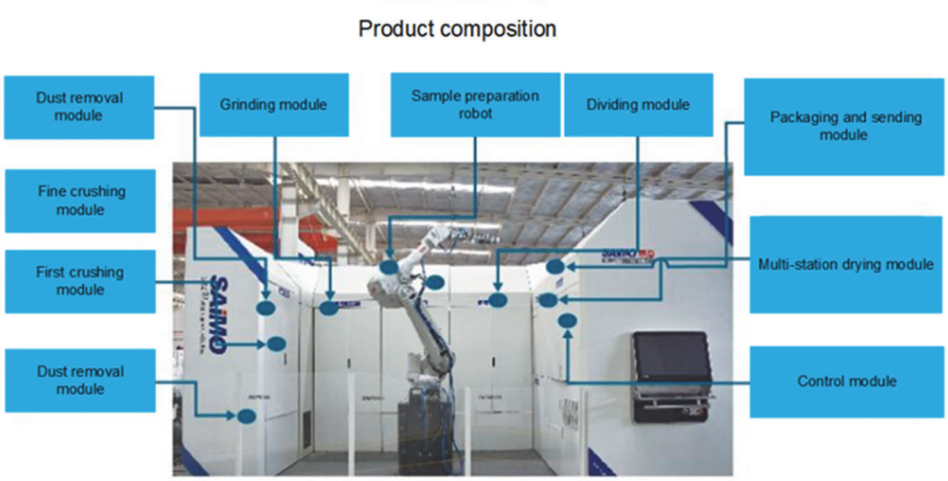

Figure 5. Automatic Robot sample preparation system

The open size and rotation speed of the divider are adjustable. Automatic calculation of division ratio and automatic adjustment according to the weight of the sample to ensure the constant quality of retained samples. The feed flow of the divider can be automatically adjusted and can be fed evenly. Before grinding, wash the grinder and the inlet and outlet sample line. Sample containers are cleaned automatically with a gas and cleaning device. The pipeline and sample preparation equipment are automatically cleaned by compressed air. Built-in full-moisture online analysis module can upload results automatically. The automatic robot sample preparation system can ensure the sample is representative enough and meet the precision requirements.

Application of automatic sample preparation system in bauxite industry

The automatic sample preparation system is applied in bauxite. The Process is shown in Figure 6. The user only needs to import the sample into the warehouse of the hoist and put the IC card circulating with the sample on the card reader of the upper industrial control machine for information leading to start the automatic sample preparation.

To meet the fast-paced sample preparation, the system is divided into 10 main units, such as primary crushing unit, secondary blending-division, crushing unit, drying unit, powder production unit, bottle conveying unit, sample and abandoned sample transmission unit, packaging writing card unit, electrical control unit, pneumatic control unit and dust removal unit. The single sample stays in different stages of crushing, dividing, each drying station and pulverizing for no more than 4 min, ensuring that the single sample preparation time is less than 6 min during continuous operation.

The automatic sample preparation system from the loading to crushing, dividing, drying, milling until bottling, code writing and other whole process to achieve automation. To prevent the loss of samples after sample preparation failure, a temporary storage mechanism is set up before the 3mm analytical samples and storage samples are prepared.

For the case of excessive water content, the pre-drying unit is used to replace the field drying, and the lost water is automatically included in the sample moisture data, which improves the efficiency of sample preparation.

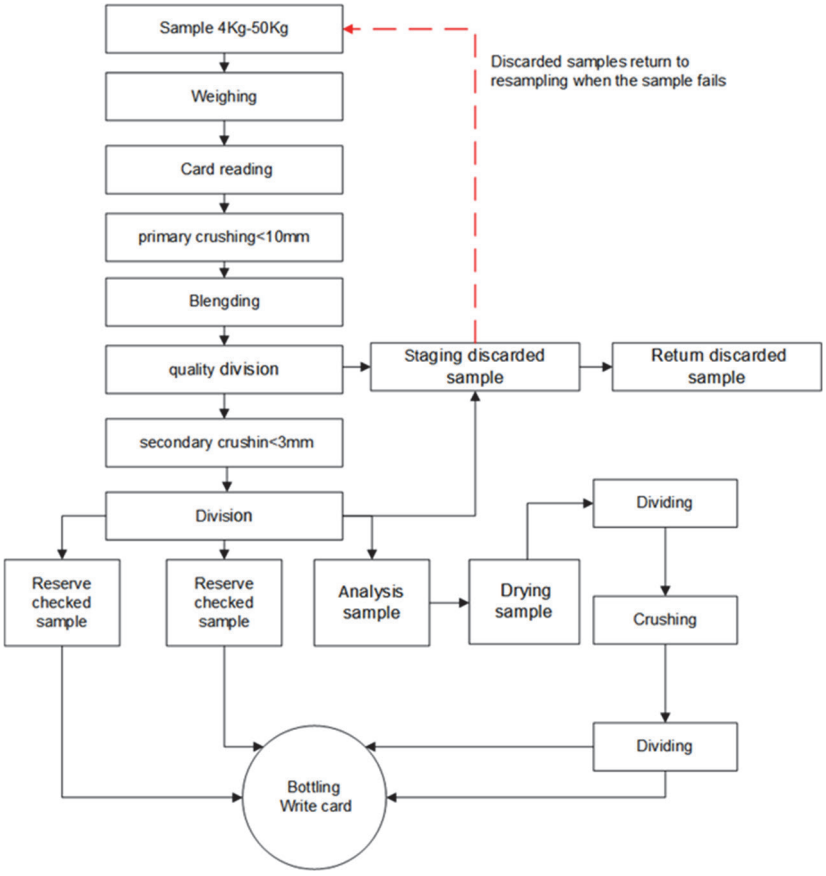

Figure 6. The process of sample preparation in the Bauxite industry

The development of slurry on-line samplers and multiplexer dividers

Pulslurry line sampler and multiple-channel divider

According to the difficult technology of the sampler and the multi-channel divider, a mobile knife-edge type sampler and a multiple-channel divider (Fig.7) were developed. Mobile knife-edge sampler is a device developed for the sampling of pressured or non-pressure (self-flowing) pipes in the flotation process of phosphate rock, providing samples for the grinding fineness and composition of the product in the flotation process.

The instrument is equipped with a sampling tube inside, and the cylinder-driven scraper scraper scrapes up and down once, which is to make the sample intercepted by the sampling tube have a good representation. Due to the particles, corrosiveness and other characteristics of the slurry, the inner wall of the sampling tube is coated with a 5 mm thick wear-resistant ceramic sheet. The upper and lower parts of the sampler are lined with ultra-high polymer materials, which can ensure that the sampler can be wear-resistant, corrosion-resistant and long-

lasting. The sampling pipe can be automatically flushed at regular intervals to ensure that the sampling pipe is unobstructed.

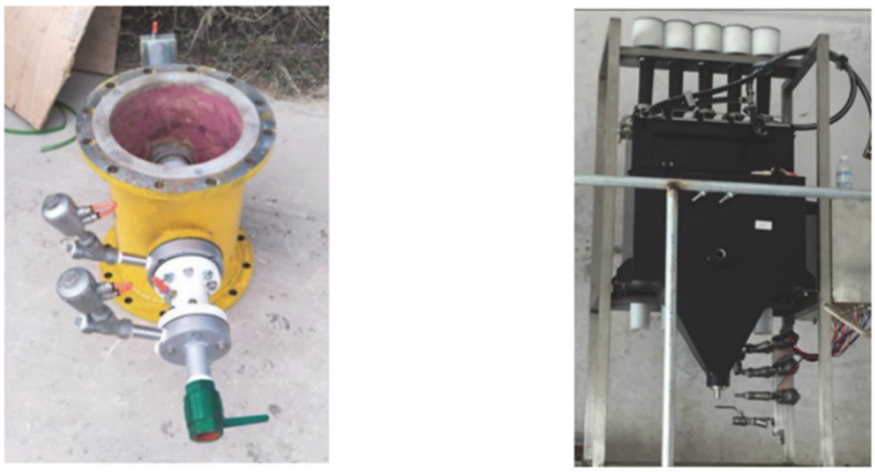

Figure 7. Mobile knife-edge sampler(left) and multiple-channel divider (right)

The multiple-channel divider can realize the mutual switching between multiple slurry runners to achieve the purpose of measuring multiple runners simultaneously by an online analyzer, the entire slurry sample switching process is not used valves, so that the entire measurement process of the slurry sample flows smoothly and is convenient to observe the size of the flow, the slurry samples entering the buffer box are removed by slag, preventing the blockage of the online analyzer measurement window, according to the size of the slurry flow, the tuning fork switch position signal controls the cylinder to push the rubber spinal canal to supplement the buffer box. It can also achieve the effect of stirring the slurry in the buffer box, at the same time, special flushing water is added to ensure that the online analyzer measurement can operate normally.

At present, the sampler and multiple-channel divider developed by this topic have been applied to the 4.5 million tons/year phosphate ore of Yunnan Linhua phosphate concentrator for application demonstration. Figure 8 show in-field pictures of on-line sampler and multiple-channel divider of slurry. The user uses the test report showing that the sampling system continuously and respectively samples the raw ore, concentrate and tailings. The sampling process is stable and representative. It can provide a stable slurry flow for the slurry online LIBS analyzer. The sampling system can also be used in other flotation processes to provide test samples for on-line analytical instruments.

Figure 8. On-line sampler ad multiple-channel divider of slurry

Online sampling and LIBS analysis technology integration system

With the purpose of online monitoring of element components of phosphate ore flotation process, based on LIBS technology, combined with mobile knife-edge type samplers and multiple-channel dividers, it has become an on-line sampling and LIBS analysis technology integration system suitable for phosphate ore flotation process, realizing the rapid analysis of phosphorus, magnesium, iron, silicon and other elements in phosphate ore slurry.

Three knife-edge type samplers are installed at the appropriate position of the raw ore, concentrate and tailings pipelines for intercepting the mineral slurry for testing. The mineral slurry passes through the transportation pipeline to the multiple-channel divider and flows into the multiplex flow stabilization box. At this point, the LIBS analyzer receives a signal that the slurry is in place. The laser starts to stimulate the stable mine slurry column under the steady flow tank flow. After the slurry flow ends, the pump begins to work and automatically clean the sampler, pipeline and multiple-channel divider to ensure that there is no slurry residue in the flow path. The schematic diagram of online sampling and LIBS analysis technology integration system is shown in Figure 9.

The detected slurry and cleaning water return to the corresponding position, such as the original ore to the original tank, the concentrate to the concentrate pond, the tailings to the tailings pond, to avoid the waste of pulp, and the whole analysis and test process can form a closed loop.

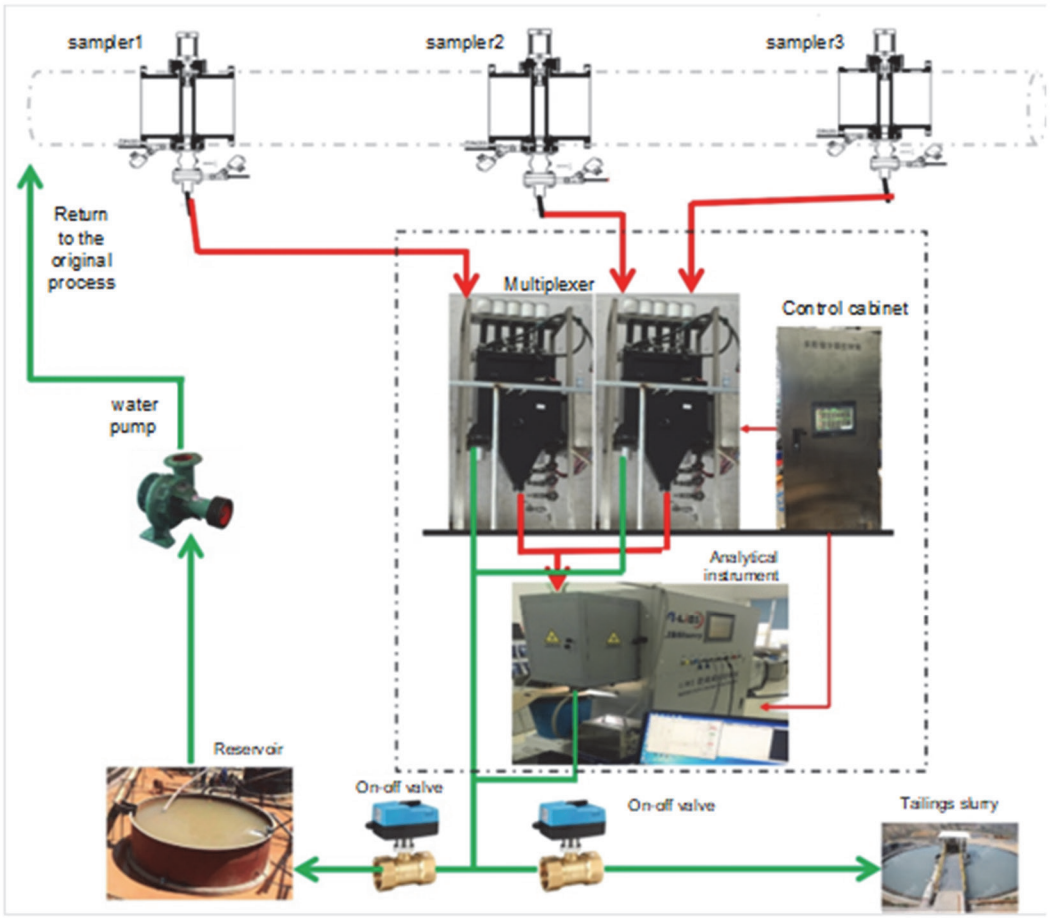

Figure 9. Online sampling and LIBS analysis technology integration system

The development status and trend of sampling preparation system

Automatic sampling and sample preparation system

Guangxi Iron and Steel Group Co., Ltd. has invested and built a new set of automatic sampling and sample preparation system in the Fangchenggang iron and steel base terminal. The process flow chart of automatic sampling and sample preparation system is shown in Figure 10. The system is designed according to the national standards for iron ore and coal sampling and sample preparation. Large robotic hands simulate manual

operation for sample preparation of moisture content, particle-level distribution, physical properties, and sample preparation of chemical composition analysis.

The operating load capacity and process of the sampling and sample preparation system are fully compatible with the port loading and unloading main system. The system is composed of four parts: primary sampling, secondary sampling, robot automatic online sampling preparation unit and material return mechanism. The human error caused by manual sampling is avoided, and the representativeness of the sampling is guaranteed.

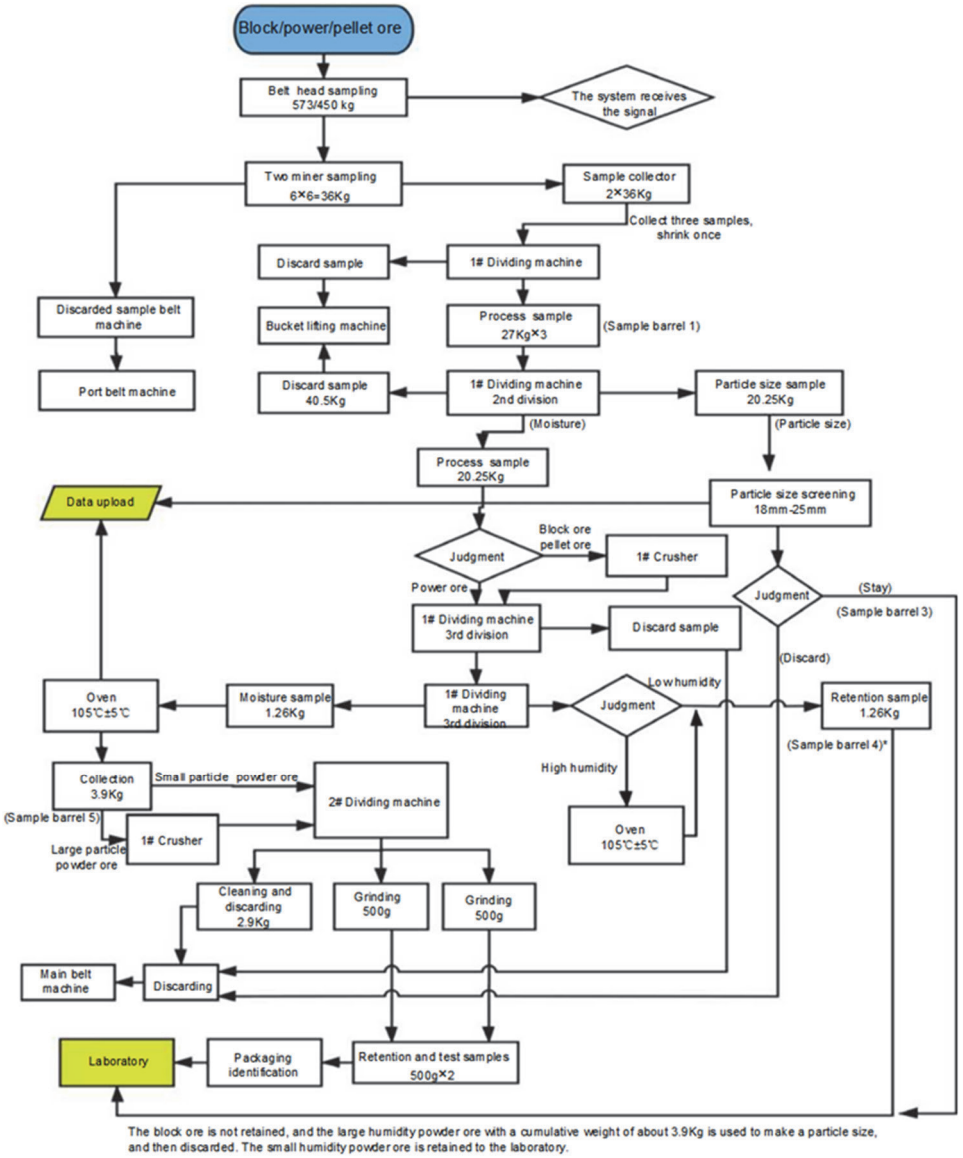

Figure 10. Process flow chart of automatic sampling and sample preparation system

Application of mobile internet of things in the optimization of mineral product inspection and testing process

The internet of things is a huge network formed with the Internet by collecting any information that needs to be monitored, connected, or interactive through a variety of information sensing devices. Its purpose is to realize the connection between objects and things, things and people, all objects and the network, to facilitate the identification, management and control.

The whole process of minerals sampling and sample preparation uses mobile internet of things technology to comprehensively perceive, safe transmission and intelligent processing to optimize the sampling system and process and solve the problems in a series of mineral inspection and testing process. The developed mobile internet of things technology includes the computer operating system, mobile terminal APP and WeChat public

account program. The three methods are simultaneously compatible and optimize the existing inspection workflow of minerals. Each link is better seamless docking, including the information transmission process with customers, more transparent, timely, and can improve the work efficiency of inspection. At the same time, in terms of quality supervision, the whole process of minerals inspection is traced, which improves the quality of inspection.

At present, there is no blind number editing, and equipment traceability only traces the equipment model and equipment status that cannot automatically collect data, and not studies the data collection of automatic collection and detection results. Meanwhile, the security and confidentiality agreements involved in the internet of things process are not discussed, which needs to be further improved later.

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