ACHIEVING RADIOMETRIC

RESULTS WITH GAUGES

Dr Madeleine Stoll, Berthold Technologies GmbH & Co. KG, Germany, provides an overview of radiometric gauges in the potash industry, from potassium detection to density and bulk flow measurement.

Alongside nitrogen and phosphorus, potassium is one of the three main macronutrients for plants. Long-term agricultural cultivation inevitably leads to a reduction of those nutrients in the soil, for which reason the use of potassium-based fertilizers is indispensable.

In order to obtain the raw material, potassium-bearing salts are extracted from evaporite deposits, which are over millions of years deeply buried in the earth's crust. The most economically advantageous ores include sylvinite (KCl), carnallite ($KMgCl_3 \cdot 6H_2O$) and, if available, kieserite ($Mg[SO_4] \cdot H_2O$).

In the potash industry, it is important to use robust and reliable measuring devices. It is not just dust and dirt that can damage measurement gauges; highly alkaline and abrasive suspensions also exclude many measurement techniques. Therefore, non-contacting and non-intrusive radiometric gauges have been used for years, since they are immune to such rough conditions in the factories and deliver reliable results in real time.

Radiometric potassium analysers

To measure the potassium concentration online, radiometric potassium analysers are specifically designed and manufactured for the potash industry. In nature, the element potassium is both stable – potassium isotopes K-39 and K-41 – and radioactive – represented by the radioisotope K-40. Stable potassium and K-40 are always present in a fixed ratio. K-40 makes up only about 0.01% of the total potassium. With the help of radiometric gauges, it is possible to make use of this constant ratio. By measuring the concentration of K-40, the total concentration of potassium and, thus, KCl can be determined.

However, due to its low proportion, the detection of K-40 requires an extremely sensitive and long-term stable measuring system. Moreover, to achieve an optimal signal-to-noise ratio, the system must be able to suppress additional occurring background radiation, which is highly increased in potash factories and in mining galleries below ground. For this purpose, detector shields are applied to reduce the sensitivity against background radiation. Radiometric potassium analysers are made of a detector unit connected to a transmitter. The detector can be installed inside a tube, which is immersed into the respective vessel or the detector can be mounted to the tank's outer wall. Thus, the setup can be adapted to the conditions present in the factory. Under specific circumstances, there is also the possibility to measure the potassium mass flow in combination with a radiometric bulk flow measurement at conveyor belts.

The main components of a detector unit are the scintillator, the photomultiplier, and the electronics. When a detector receives gamma radiation originating from radioactive decay – i.e. in this instance, of K-40 – gamma rays interact with the scintillator material by producing light flashes. These faint flashes of light are transformed into electrons by the photocathode of the photomultiplier. While leaving the photocathode, the electrons are accelerated to the anode and multiplied by the photomultiplier, so that a well measurable current pulse can be detected (Figure 1). The amplitude of this signal is proportional to the energy deposited by the gamma ray in the scintillator. With the help of the transmitter, which is typically installed in the control room, the signal is evaluated, and the total potassium (usually displayed as K_2O equivalent) or potassium chloride concentration is quantified.

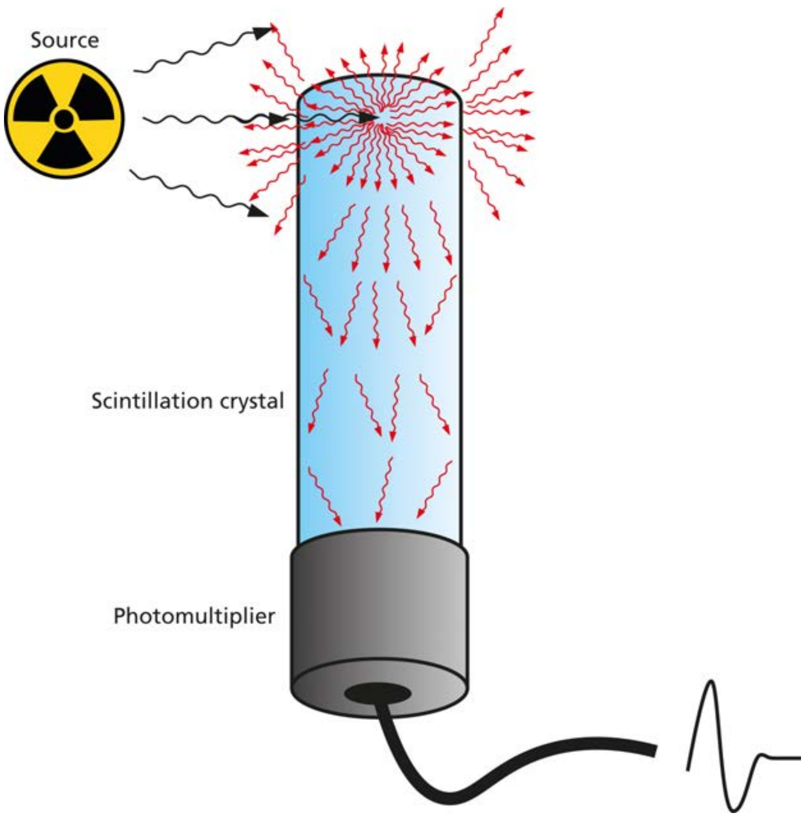

Figure 1. Detector makeup.

Radiometric density and bulk flow gauges

Besides the potassium concentration, density and bulk flow are preferably measured by radiometric gauges. For both methods not only a detector is needed, but also a radioactive source. Source and detector are typically installed in one line, and the vessel, pipe or conveyor belt to be examined in between. The principle behind radiometry is based on attenuation. When radiation passes media, depending on the density and material of the media, a material-specific amount of the radiation is attenuated. The detector then detects the remaining radiation. The resulting signal can be assigned to a respective process value by means of an earlier calibration.

Both density and bulk flow measurement can be installed on existing systems. In the case of the density measurement, the installation can be done without any downtime of the process. When the system is calibrated once, a future calibration or maintenance is not needed, since the measurement works non-intrusively and without any contact.

Shielded and encapsulated sources

Today's radiometric detectors are highly sensitive to gamma radiation, so that very low radioactive source activities are required. This is important for the user's health and safety, as well as being a major cost-saving factor. Caesium-137 (which has a half-life of 30.2 years) or Cobalt-60 (which has a half-life of 5.3 years) are used as radioactive sources. With the help of a tight capsule, the radioactive material is always sealed safely, even under extreme and rough conditions. For radiation protection, the capsule is enclosed in a housing called the shield. The shield is made of steel or stainless steel, which is filled with lead, tungsten, or both. Only a small opening in the shield (not in the capsule) serves as a narrow exit and collimation point for the gamma radiation beam used in the measurement. Designed to ensure optimal radiation protection during transport and installation, this opening always remains closed by a special shutter. This shutter is only opened when the measurement is put into operation. To be able to measure the respective process in the best possible way, either point or rod sources can be used. The usual operation time of a radiometric source lasts between 10 – 15 years.

Detectors

Depending on the conditions on-site and on the measuring task, point and rod detectors are available. Point detectors usually come either with a NaI(Tl)-crystal or a polymer as scintillator material. NaI crystals are more sensitive at the same size, however, they are more expensive compared to polymer scintillators. Due to the long scintillator rod, rod detectors are only available in a polymer version. Both scintillator materials are long-term temperature stable between −20 °C and 60 °C. To protect the scintillator and the electronics at temperatures above 60 °C, the use of a water-cooling jacket is mandatory.

Bulk flow measurements at conveyors

For bulk flow measurements, a measuring frame is required, which is installed around the conveying system, e.g. conveyor belts, screw conveyors or bucket elevators (Figure 2). Typically, the measuring frame carries a point detector at the top and a rod source at the bottom, but there are different arrangements possible depending on the conveying system present. The measuring frame additionally serves as radiation protection since it includes lead-strengthened sides. To measure the bulk flow, the material on the conveyor system is irradiated from below by the gamma radiation source. The radiation is absorbed depending on the weight per unit area, i.e. area x density. The remaining radiation is detected by the point detector. To receive the bulk flow process value, the weight-per-unit-area value is multiplied with the conveyor speed. In comparison to conventional bulk flow measurement systems, no mechincally moving parts are installed, which results in a maintenance-free system, without future re-calibration. Moreover, dirt, encrustations, vibration, belt tension, and wind cannot impair and influence the measurement.

Density and mass flow measurements at pipes

A density measurement system consists of a point detector and a point source. By using a narrow radiation beam, product density, concentration and solid content can be measured contact-free and non-intrusively in vessels and pipes. The mass flow in pipes can be determined in combination with a flow meter. The measurement is neither influenced by pressure or temperature, nor by abrasive or acidic conditions. For an optimal measuring result, it is important that the pipes are completely filled and that air bubbles or material sedimentation at the bottom are avoided.

Applications in potash factories

Due to the variety of different production, processing, and refinement techniques, this article will focus on the methods of processing solid potash salts obtained from mining, which apply radiometric gauges as essential monitoring devices.

After mining the material below ground, the crude salt is first pre-crushed in the gallery and then transported to transport shafts by conveyor belts. Already on the conveyor systems, bulk flow and potash content are determined online and in real time. Thus, crude salt masses from different mining spots below ground can be mixed into a mass of optimal potash concentration for further processing. As the material reaches the surface, the crude

Figure 2. Radiometric bulk flow measurement system installed at an outside conveyor belt.

Figure 3. Radiometric density gauge installed at the outlet of a floatation tank.

Figure 4. Potassium analyser monitors the potassium concentration after electrostatic separation.

salt is milled down to obtain monomineralic fragments as far as possible. Since the crude salt is a mixture of valuable potassium bearing salts, other salt types and further impurities, separation techniques need to be applied to remove undesirable admixtures and to concentrate the valuable salts.

There are three different separation techniques used in the potash industry. One of the most common techniques is flotation. This method is based on the different binding capabilities of the substances in crude salt to chemical additives. Depending on the deployed chemical and the desired salt type, the fragments of the respective salt are hydrophobised. They float to the top of the flotation tanks with the help of mechanical or pneumatical produced air bubbles and form a concentrated salt foam, which is then collected. Remaining, undesired salt types and impurities either stay in the water or sink down to the bottom of the tank. This residue is then removed from the tank via an opening at the bottom. To reuse the floatation water, the solid material and the water are separated by filtration. To monitor the density of the residue before filtration, radiometric density measurements are installed at the outlets of the tanks (Figure 3). Since the foam still includes a high amount of impurities and thus, an insufficient percentage of the desired salt type, several flotation steps are needed to maximise the yield. The same is performed with the residues, preventing the loss of valuable material. The final steps include washing and draining of the foam concentrate.

Another separation technique is hot dissolution with subsequent cooling crystallisation, which makes use of the different solubilities of salt components during heating. It has the advantage that highly purified products with equal grain-size can be produced. To separate potassium chloride (KCl) from crude salt, a sodium-chloride (NaCl)-saturated solution (mother liquor) is heated up to ≈120˚C. In the next step, crude salt is added. Since the solubility of KCl increases with increasing temperature and the mother liquor is saturated with NaCl, KCl dissolves, while NaCl and other insoluble components remain nearly completely solid. Those solids are then removed by filtration. Afterwards, the hot solution is cooled down, enabling KCl to crystallise and then be separated from the solution. Radiometric potassium analysers are installed at the vessels of the hot dissolution process to monitor the progress. The detectors are either mounted at the wall of the vessel, which protects the scintillator and the electronics from the heat of the solution, or immersed into the vessel via a dip tube, in which case the detector needs to be cooled additionally, either by fresh air or by water cooling.

Finally, the electrostatic separation method is a technique invented and patented by K+S Minerals and Agriculture GmbH. This method does not require the use of water, and therefore no liquid residues are produced. Prior to the separation procedure, the monomineralic salt fragments received from the aforementioned grinding process, are conditioned by special surface-active substances enabling the salt types to be electrostatically charged differently. A freefall separator, which generates a high voltage field, separates the salt types. The yield is optimised by repeating this procedure several times. After the separation process, the potassium concentration is determined online by radiometric potassium analysers (Figure 4). At fully closed drag chain conveyors bulk flow measurement systems are installed. Due to the absence of water, salt dust is present, which increases the background radiation. Therefore, the detectors need to be shielded in order to decrease this influence.

In addition to the application locations discussed, there are several other locations in potash factories, where radiometric bulk flow, density and potassium measuring devices are installed.

Conclusion

Radiometric gauges have become an indispensable part in the production of potash fertilizers. With the help of those systems, processes are monitored in real time and thus process flows can be optimised. The non-contacting and non-intrusive measuring technology resists the rough and aggressive conditions without requiring further maintenance and re-calibration. Where other measurement methods fail, radiometry provides reliable and reproducible results. WF

Note

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