Emergency analytical testing: things to consider

C.J. Pretorius
Council for Scientific and Industrial Research (CSIR), Pretoria, Gauteng, South Africa

ABSTRACT: Circumstances may dictate that samples from mining operations are analysed for unknown compounds that are potentially harmful to humans. These circumstances may be out of the ordinary, unique or isolated incidents. Emergency analytical testing may be part of an investigation following a safety incident or part of a risk assessment. The outcome of the testing is usually required within a short time and the testing needs are not always clearly defined. The main concern is generally the health and safety of mine employees and various disciplines from the mine's operations that may be involved in the investigation. This paper provides guidance to individuals tasked with such an investigation or risk assessment on what to consider when emergency testing is required, particularly on how to take samples to protect sample integrity, what type of testing to request from the analytical facility, and how to treat the results and outcomes of such testing.

1 INTRODUCTION

In an emergency situation it may be necessary to take a grab sample from an area or a suspicious source. The situation may be an incident, part of an investigation or a research project. Examples of such situations may be after a spillage of an unknown liquid, after an explosion where the usual gases were not detected, after a fire where residue or gases persist in the workplace environment long after the incident, when new products are introduced into a process or when mine employees are effected by an unknown stressor in the workplace air. The type of sampling may not be standard and may not be included in routine continuous or compliance monitoring. In a situation like this, the sampling and analysis may enforce interim and final decisions with potentially costly and far-reaching consequences. It is therefore important that sampling is conducted in the best possible way for obtaining value from the analytical outcomes.

2 BACKGROUND

Over the past few years the researcher has been involved in situations where unconventional sampling and non-routine analysis have been required in order to find a solution to a problem. This type of sampling has presented a few challenges and the outcomes have revealed the need to provide guidance on how to conduct sampling under unconventional circumstances but still maintain sample integrity. Once the sample is handed over to an analytical facility, the real challenge starts: which analytes to test for!

In an emergency situation in a mine, specific health and safety procedures are followed. This paper does not focus on these procedures but rather on the taking and treatment of samples and the required testing afterwards.

3 OBJECTIVE

The objective of this paper is to provide points to consider on the sampling and analysis of samples under emergency or unconventional circumstances.

4 APPROACH

Different types of grab samples that may need to be taken are:

- *Solids such as soil, rocks, burnt residue (e.g. timber, conveyor belt or clothing);*
- *Liquids such as water, oil or unknown suspensions;*

- *Fine to ultrafine particulates such as airborne dust, fumes or vapours; and*
- *Gases – usually unknown and potentially com*plex gases.

4.1 *Consideration #1: Planning the sampling*

The first step will be to obtain background information from more than one source to ensure that the facts of the incident are recorded accurately. This will direct the person that will do the sampling, and allow for the timeous gathering of preparatory information and planning of best approaches. Sampling and method handbooks such as the National Institute for Occupational Safety and Health's (NIOSH) *Method finder* (NIOSH 2003), the United States Environmental Protection Agency's (US-EPA) *General field sampling guidelines* (US-EPA 2013) and the Health and Safety Executive (HSE) guidance for *Methods for the Determination of Hazardous Substances* (HSE 2017) are useful as quick references.

4.2 *Consideration #2: Taking a sample safely from a hazardous environment*

When going into a potentially unsafe and hazardous environment, the maximum health and safety precautions should be taken. Always presume that the sample to be taken is toxic and harmful to human health. The safety of the sample may only be confirmed by the testing laboratory.

Do not touch the sample with bare hands; do not smell the sample or taste it.

4.3 *Consideration #3: Taking a sample that represents the original source*

The sample should be representative of the original source. To ensure this, the sample should be taken as soon as possible from the most likely origin. If one needs to investigate the cause of an incident or the potential long-term effects afterwards, the source should be pin-pointed as soon as possible and samples taken at or near the source. This may include sampling an area to determine the footprint of the source identified as the potential cause of the incident and "background" or reference samples. Gases dissipate over time, liquids can change composition and even solids may change their form. The sooner any sample can be taken, the better.

4.4 *Consideration #4: Finding a suitable container to protect sample integrity*

Use a suitable container. This is one of the most important factors to consider as the container has to protect the integrity of the sample – without contaminating or affecting the sample's composition (Budowle et al. 2006). Use a container made from an inert material that will not react with the sample (e.g. glass, Teflon (PTFE), poly propylene (PP) or polyphenylene ether (PPE)). This may be the biggest challenge in an emergency situation, where a suitable container may not be readily available.

The following are useful points:

- Where possible, use a clean, dark, glass container. In the event that organic compounds may be tested, a glass container is recommended as certain organics may adhere to the sides of a plastic container. The dark container limits ultra-violet rays from sunlight breaking down certain compounds.
- If a previously used container is used, make sure that the container is washed and dried before sampling. Rinse or "clean out" the container with some of the sample in question to get rid of possible contaminants. A good rule of thumb is that one shouldn't be able to smell what was in the container prior to taking the sample.
- Liquids: a glass bottle may be suitable for liquid samples. Fill the bottle to the top, close the bottle securely with a cap and ensure that the cap seals tightly to prevent leakage. Make sure that the bottle does not leak. Place the bottle in a bag and seal the bag. A volume of two to five litres should be sufficient, especially when very low concentrations of analyte are to be tested.
- Keep the sample cool (below 5°C) and get it to the testing facility as soon as possible. Micro-organisms have to be tested within 24 hours of the sample being taken as they decompose and break down fast. Keeping the sample cool assists with the preservation of certain compounds.
- Solids: a plastic bag with an airtight seal may be suitable for this type of sample. Use two bags for the sample and another one to secure the bags that contain the sample. If any reaction occurs with the plastic bag on contact, rather use a glass container that can seal air tight. An equivalent quantity of two to three handfuls of sample should be sufficient, especially when very low concentrations of analyte are to be tested (US-EPA 2015b).
- Gases: unfortunately the best way to take a sample of gases is to use a Tedlar bag (US-EPA 2001) or a stainless steel canister (US-EPA 2015a) designed for gas sampling. The right type and size of bag/canister and sampling equipment should be kept at the mine in preparation for an emergency. Normal plastic bags or containers are not suitable as gas molecules may diffuse through the material. Gases should immediately be sent to testing facilities for analysis before any loss or degradation of the gas occurs. The manufacturer's instructions should be fol-

lowed for each type of bag or container. Use the biggest, most practical bag or container possible. If the situation allows, take more than one sample in a bag or container.
- Dust or airborne particulates can be sampled using the standard gravimetric sampling train and membrane filters.
- Human tissue, blood and urine can only be handled by medical professionals and should not be sampled by any other person.
- Seal the container with tape or cable ties to prevent the sample from being tampered with. The objective should be to protect and maintain the sample integrity.
- The container or package should be accompanied by the necessary paperwork to identify the content and all critical sample and sampling information. Examples of information to include are facts about the incident and the contact person; the date and time that the sample was taken; unambiguous sample information; clear labelling; and photos of the sampling site where possible. The paperwork is essential for transport and handling so that the person handling the package is aware of the content and may take the necessary health and safety precautions. If the sample forms part of an investigation, the paperwork can take the form of a chain of custody; i.e. it specifies who takes ownership of and responsibility for the sample during each handover.

4.5 *Consideration #5: Recording contextual information from the environment and the sample*

Make notes of the observations from the environment and about the sample. This information may be very important during an investigation but may also assist during the analysis of the sample. Where relevant, the information should be included in the necessary paperwork accompanying the sample to the testing laboratories. The following guidelines are provided:
- Take note of personal observations using your senses (without making deliberate contact with the sample): what was seen or heard (e.g. foam that was bubbling); was there a noticeable smell (e.g. smelt like vinegar); could something be felt (e.g. temperature, vibrations) (e.g. close to the unknown material it was warm and there was a slight tingling to the skin); was there a distinct taste in your mouth when inhaling the air (e.g. metal taste)?
- Answer the 5W questions about the sample: who took the sample (e.g. a mine employee); what was sampled (e.g. powder residue); where was the sample taken (e.g. scraped of the hanging wall in a closed-off area); when was the sample taken (e.g. two days ago); why was the sample taken in other words what was the reason or the incident that led to the sample being taken (e.g. mine employees are getting a rash on their skin after contact with the residue; residue is as a result of a fire incident three weeks ago)?
- Record how the sample was taken (e.g. material was scraped of the hanging wall with a plastic spoon).

4.6 *Consideration #6: Choosing the appropriate testing facility*

Submit the sample to an appropriate testing facility as soon as possible. It may be useful to keep a list of testing facilities and their capabilities. Find out the administrative and technical requirements of the facilities and, if possible, implement measures in advance to prevent unnecessary delays on requests in the event that emergency testing is required.
A few facilities (amongst others available) are listed below:
- Mine Rescue Services are equipped with instrumentation to test for certain gases.
- In general, micro-biology laboratories have a narrow scope for micro-organism testing (e.g. E.coli, faecal coliform or total coliform count).
- If there is a suspicion that human pathogens are involved, only pathology laboratories should do the testing (e.g. Ampath or Lancet). Involve the medical doctor from the start to follow the correct procedures. Please note that these facilities may be able to test samples only in a particular form or matrix (e.g. only urine or blood and not water). It is important to find out what their capabilities are and to be prepared.
- The Council for Scientific and Industrial Research (CSIR) has various laboratories that can characterise liquids, solids, gases and dust samples.
- The Council for Mineral Technology (Mintek) specialises in mineral processing and extractive metallurgy.
- The National Nuclear Regulator (NNR) and the National Energy Corporation of South Africa (NECSA) are capable of testing for radio-active materials.
- The National Institute of Occupational Health (NIOH) may provide testing on aspects related to toxicology and medical matters.
- The University of Pretoria has a forensic testing laboratory.

4.7 *Consideration #7: Providing useful information to the testing facility*

When submitting the sample to the testing facility, provide appropriate and sufficient information. The

information gathered under Consideration #5 may be useful here as it will direct the testing but also explain the results i.e. possible contamination. Be aware that asking for a specific test instead of explaining the problem may lead to incorrect tests being conducted and valuable time being wasted (during which sample degradation and changes to the composition can occur). Where possible, refrain from reaching a conclusion until after the investigation has been completed.

As new information becomes available, feed it through to the laboratory as soon as possible to assist with the testing as this may have time, quality and cost implications for both parties.

4.8 *Consideration #8: Requesting the appropriate analyses*

The type of analysis to request will vary with each sample and the situation it is associated with.
As a rule of thumb, it is wise to start with qualitative screening methods, for example:

- Organic screening for volatile and semi-volatile organic compounds (VOC and SVOC) using methods such as gas chromatography mass spectroscopy (GC-MS) and Fourier-Transform Infrared (FTIR);
- Organic screening for non-volatile organic compounds (NVOC) using methods such as High-performance liquid chromatography (HPLC) and Ultra-performance Liquid Chromatography (UPLC) for very low concentrations;
- Inorganic screening for inorganic elements (e.g. iron, lead, arsenic) using methods such as atomic absorption spectroscopy (AAS), inductively coupled plasma (ICP) and X-Ray Fluorescence (XRF);
- Inorganic screening for mineral compounds using X-Ray Powder Diffraction (XRD); and
- Micro-organism screening for common bacteria such as E.coli, faecal coliform and total coliform.

These tests provide a general overview of the sample and may provide sufficient information on which to focus the continued analysis. Speciation methods are available to distinguish between particular pollutants (e.g. hexavalent chromium vs total chromium).

Analysis methods have a specified time associated with them. For this reason it is important that sufficient quantities of the sample are available for parallel analysis to be undertaken and that the correct information is provided to the facility from the start.

4.9 *Consideration #9: Interpreting the outcomes of the analysis within the context of the incident*

What to do with the outcomes of the analysis? Most test reports state that the results relate only to the samples analysed. The results from the samples taken should be an indication of what the problem is. Refrain from extrapolating the results and making assumptions based on the results of a few samples. Use the outcomes of the analysis within the context of all the information from the incident.

The information needs to be accurately interpreted. When submitting samples to a testing facility there is a certain perception or expectation of the outcomes. If the analysis information is misunderstood, the investigation of the incident may be dealt with incorrectly.

5 CONCLUSION AND RECOMMENDATIONS

In an emergency situation or after an incident, it is important to ensure that a representative sample is taken, if required. The container should be inert and able to protect and maintain the integrity of the sample. The sample should be kept cool and sent to a testing facility as soon as possible. Detailed contextual information should be recorded from the environment in which the incident occurred and from the sample itself. When requesting analyses from a facility, it is recommended that screening methods are chosen first before specific methods are selected as a time-saving mechanism. If sufficient quantities of the sample are provided, different screening methods can be analysed in parallel.

The most important consideration is to be prepared. Establish or review current emergency sampling procedures and implement improvements that could prove to be invaluable on the day that they are required.

ACKNOWLEDGEMENTS

The author wishes to acknowledge the CSIR scientists Calie Adlem, Nial Harding, Paul Steenkamp and Mellisa Govender-Kirkpatrick for their inputs and contributions.

REFERENCES

Budowle, Bruce, Schutzer, Steven E., Burans, James P., Beecher, Douglas J., Cebulla, Thomas, Chakraborty, Ranjit, Cobb, William T. et al. 2006. Quality sample collection, handling, and preservation for an effective microbial forensics program. *Applied and Environmental Microbiology*,

72(10):6431-6438, October. United States. doi:10.1128/AEM.01165-06.

Health and Safety Executive. Methods for the Determination of Hazardous Substances (MDHS) guidance books. www.hse.gov.uk/pubns/mdhs/index.htm (Accessed 24/05/2017)

National Institute of Occupational Safety and Health (NIOSH). 2003. *Manual of analytical methods. Method finder*.

United States Environmental Protection Agency (US-EPA). 2001. Standard Operating Procedure 2120. Tedlar Bag Sampling. August 2001.

United States Environmental Protection Agency (US-EPA). 2013. Standard Operating Procedure 2001. General Field Sampling Guidelines. July 2013.

United States Environmental Protection Agency (US-EPA). 2015a. Standard Operating Procedure 1704. SUMMA Canister Sampling. November 2015.

United States Environmental Protection Agency (US-EPA). 2015b. Standard Operating Procedure 2019. Incremental Sampling Methodology For Soil. December 2015.