ColdBlock™ Technologies Inc.

White Paper: Practical Guidance for Gold Digestion Using the ColdBlock Sample Digestion System

Optimizing Exploration Site Workflows While Respecting Regulatory Standards

Abstract:
Gold analysis is inherently challenging due to the uneven distribution of gold, coarse gold particle sizes and the complexity of host mineral matrices [1,3]. Fire assay is a long-standing, established regulatory standard for commercial laboratories and resource reporting [5]. More recently, the commercial development and rollout of the Gamma Activation Analysis (GAA) technology by Chrysos Corporation has provided an alternative analytical technique. Exploration programs and site laboratories are in need of fast, safe digestion methods that can deliver the same accurate, reproducible results to support high throughput decision making. Aqua regia digestions using ColdBlock Technologies Inc. (ColdBlock) digestion system offer an efficient and controlled approach, particularly well-suited to the repeated analysis of well-characterized deposits. This paper provides guidance on method selection, digestion chemistry, sample preparation, and practical workflow considerations for the effective implementation of ColdBlock digestion in site laboratory environments.

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Introduction

The heterogeneous distribution of gold in geological materials results in significant variability between small analytical splits [1]. This nugget effect, combined with the presence of coarse gold particles or sulphide-associated gold, can lead to challenges in achieving reproducibility, particularly when sample sizes are limited [3,4]. Careful consideration of sample mass, homogenization, and digestion chemistry is therefore critical to achieving accurate and reliable measurements [2].

Fire assay is optimized for regulatory compliance and high accuracy gold reporting [5], however, the longer processing time and large sample requirements can limit throughput in exploration and site laboratory settings. While GAA offers faster turnaround and can process samples up to 500 grams, it can be more costly than wet acid digestion methods. Traditional aqua regia provides an alternative approach, and when coupled with the ColdBlock digestion system, it delivers accelerated, consistent heating under controlled conditions. This combination can improve laboratory throughput and reproducibility for well characterized deposits where sample variability is well understood [5,6].

By addressing both the chemical and operational considerations associated with gold analysis, this paper aims to provide practical guidance for laboratories seeking to optimize digestion workflows while maintaining analytical reliability.

Gold Analytical Methods Overview

Fire Assay

- Reference method for regulatory compliance, including reporting under NI-43-101 and many ISO and ASTM procedures. [5]
- Large sample masses, typically 30 to 50 g, improve representativity and reduce the impact of the nugget effect. [1,2]
- Depending on the final measurement, method detection limits can reach as low as 1 ppb, making the method suitable for generative exploration programs and appropriate for low level gold deposits.
- Widely accepted by site and commercial laboratories, and regulatory bodies for resource estimation and formal reporting.
- Time intensive and requires specialized equipment and trained personnel.
- Presents significant safety risks due to elevated temperatures and the generation of toxic fumes.

Gamma Activation Analysis

- New method now accepted by NI-43-101 and JORC.
- Large sample masses, typically 250 to 500 g, significantly improve representativity and reduce the impact of the nugget effect.
- Becoming widely accepted by site and commercial laboratories, and regulatory bodies for resource estimation and formal reporting.

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- Reporting limits may be a limitation for exploration programs or for low grade gold deposits.
- Requires significant investment in equipment, laboratory space, air handling/air conditioning and power infrastructure.
- Significantly improves the environmental and safety footprint of mining labs as there is no need for elevated temperatures and toxic reagents like lead or acids.

Aqua Regia Digestion

- Commonly used for geochemical exploration programs, screening studies, and multi element analysis [5].
- Acid ratios and sample mineralogy, particularly sulphide content, can influence digestion performance [4].
- Not typically used as the primary method for regulatory resource reporting as recovery can be incomplete (80 to 90% for coarse, nuggety gold) [3] and for mineralogical assemblages that interfere with the sample digestion and/or the final gold extraction into the organic layer (for DIBK separation procedures).

ColdBlock Digestion

- Provides rapid heating under controlled conditions for aqua regia digestions.
- Efficiently processes larger sample sizes up to 30 g using CBL large size tubes.
- Improves digestion consistency and laboratory throughput compared with conventional hot plate or block methods.
- Well suited for repeated digestion of similar matrices in exploration or site laboratory environments.
- Provides a significantly safer sample digestion as it requires no lead, high heat or open flames.
- Accurate analysis or lower grade samples requires use of more expensive ICP-MS instruments, or concentration using DIBK (for AAS analysis).

Recommended Applications for ColdBlock Digestion

ColdBlock digestion is particularly useful in exploration and site laboratory workflows where:

- Samples originate from a well characterized deposit or consistent geological setting.
- Gold measurements are repeated across similar matrices during drilling or exploration campaigns.
- Gold method reporting levels of 1 to 5 ppb are required.
- Rapid analytical turnaround is required to support operational decisions.
- Multi element analysis is required and can be performed alongside gold in a single digestion.

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- ICP-MS is available for solution analysis, or solvent extraction like DIBK is available.

ColdBlock digestion may be less suitable when:

- Samples are highly variable, or originate from multiple deposit types.
- Samples are impacted by high nugget effects and very large sample sizes are required.
- Samples contain refractory gold (Carlin-type arsenian pyrite) and/or preg robbing effects from naturally occurring carbon in the sample matrix.
- The mineralogy of the sample contains elements that will interfere with the partitioning of gold in the DIBK layer (for methods employing Flame AAS measurement), i.e. high iron, manganese, copper.
- Formal regulatory reporting requires fire assay or GAA for gold determination [5].

ColdBlock Gold Digestion – Practical Guidance

Sample Selection and Preparation

Gold analysis is sensitive to both sample size and homogeneity. For well characterized deposits, 10-30 g sample sizes are recommended for ColdBlock digestions. Larger masses help reduce variability caused by coarse or unevenly distributed gold particles [3]. Samples should be finely ground and thoroughly homogenized to improve representativity [2].

Digestion Chemistry

Gold dissolution in aqua regia relies on the combination of an oxidizing agent and a chloride source [5]. Nitric acid serves as the oxidizer, converting metallic gold to $Au3^{+}$ ions, while hydrochloric acid provides chloride ions to form the soluble tetrachloroaurate complex $(AuCl_4)^{-}$. The overall reaction can be represented as:

$$Au\ (s) + 3\ HNO_3 + 4\ HCl \rightarrow HAuCl_4 + 3\ NO_2 + 3\ H_2O$$

ColdBlock's use of controlled, infrared heating during digestions accelerates standard aqua regia reactions.

For samples containing significant sulphide phases, the nitric acid is added first in a controlled, gradual manner. This allows the nitric acid to oxidize sulphides and other reactive minerals before chloride is introduced. The reaction produces NO_x vapors, which can be observed as brown-reddish fumes in the fume hood. In some cases, nitric acid may be added in excess to ensure complete oxidation before hydrochloric acid is introduced [4]. Only after this oxidation step is hydrochloric acid added, to provide chloride enabling the formation of the tetracloroaurate complex and ensuring gold remains dissolved in solution.

This staged addition approach:

- Reduces the risk of incomplete digestion caused by sulphide interference.

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- Improves reproducibility and recovery of gold.
- Controls reaction vigor, making the process safer and more consistent.

Aqua Regia versus Reverse Aqua Regia

Standard aqua regia uses a fixed HCl:HNO3 ratio, typically 3:1. In sulphide-rich or complex matrices, a reverse aqua regia approach, with initial excess nitric acid, may be employed to ensure thorough oxidation before adding HCl [4]. This approach is particularly effective for site laboratories processing well characterized deposits, where reproducibility and throughput are priorities.

DIBK Flame AAS vs ICP-MS Measurement

The use of DIBK as an organic extraction reagent is a well-established method for preconcentrating the digested gold in an acid solution, and enabling flame AAS to achieve method reporting limits as low as 5 ppb. This is a low-cost method that requires relatively inexpensive instrumentation and reagents, and only basic laboratory infrastructure. However, the addition of DIBK to the gold acid digestion workflows presents the following limitations:

- The addition of another step in the analytical method that requires staff training and diligent performance to ensure quality results.
- Disposal of the used organic solvent/acid mixture via licensed hazardous waste companies is expensive.
- Interferences can occur when partitioning gold into the organic layer, particularly in samples containing elevated concentrations of iron, manganese, copper, and other competing matrix elements.

Alternatively, final measurement of the acid solutions by ICP-MS is an excellent way of providing ultratrace method detection limits for gold, sub 1 ppb, as well as providing the full range of aqua regia digestible multielement sites including, Ag, Cu, Ni, Pb, Zn, Co, Fe, As, etc. However, the ICP-MS analysis method suffers from the following limitations:

- Expensive hardware and lab infrastructure required to support an ICP-MS instrument.
- Cost of the ICP-MS instrument, although all in costs for ICP-MS instruments have significantly decreased in the last 5-years.
- Need for experienced and well-trained spectroscopists and chemists and instrument specialists to operate the ICP-MS instruments and provide high quality data, and service technicians should the instrument experience issues.
- Need for high purity and expensive reagent grade Argon gas to fuel the ICP-MS.

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Solution Stability and Gold Complexation

Once Au^{3+} is formed and complexed with chloride, it remains stable in solution as $AuCl^{4-}$ [5,6]. Maintaining sufficient chloride concentration helps stabilize this complex and reduces the likelihood of adsorption losses onto container surfaces or sample introduction components. For best results, digested solutions should be analyzed without extended storage, and matrix conditions between standards and samples should remain consistent [6].

Method Limitations

While ColdBlock aqua regia digestion is highly effective for many exploration workflows and site labs, some limitations should be considered.

- Aqua regia does not fully dissolve all refractory mineral phases; gold hosted in arsenian pyrite, tellurides, or other refractory minerals may not be completely recovered [4].
- The nugget effect may influence results when coarse gold particles are present.
- Increasing sample mass and thorough homogenization helps reduce variability but cannot eliminate it entirely [1,3]. For regulatory resource reporting, fire assay and GAA remain the accepted reference methods [5].

Analytical Workflow in Site Laboratories

ColdBlock digestion is commonly integrated into exploration laboratory workflows where large numbers of samples must be processed quickly. A typical workflow includes sample drying, grinding, homogenization, digestion, dilution, and ICP-MS analysis.

Figure 1. A typical exploration laboratory workflow incorporating ColdBlock digestion and ICP-MS analysis.

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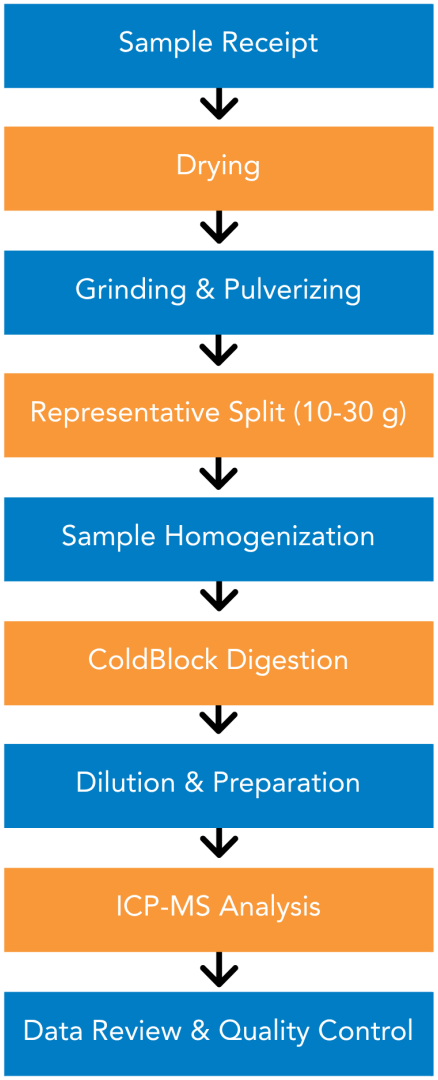

Because ColdBlock digestion allows larger sample masses to be processed rapidly, laboratories can balance representativity and throughput. Multiple samples can be digested simultaneously under controlled heating conditions, reducing variability between digestion batches and improving reproducibility compared with traditional hot plate digestion.

Gold Quantification and Analysis

By combining ColdBlock digestion with ICP-MS analysis in helium collision mode and using internal standards, laboratories can achieve rapid, accurate, and reproducible gold measurements even in complex matrices with high TDS or interfering elements.

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Potential Interferences

Spectral overlaps are the primary concern for gold analysis by ICP-OES. Iron and other matrix elements can produce emission lines that overlap with gold, making ICP-OES unreliable for trace measurements.

Even in ICP-MS, polyatomic species such as $^{181}Ta^{16}O^{+}$ can interfere under certain conditions, which is why helium collision mode is used to minimize these effects [7].

Matrix Considerations

Large sample mass digestions can produce solutions with elevated total dissolved solids, particularly when analyzing geological materials rich in iron, aluminum, calcium, and magnesium. Higher matrix concentrations can supress the gold signal, affect plasma stability, and accelerate contamination of interface cones.

Dilution of digests, the use of high matrix introduction techniques, and regular monitoring of internal standard recovery help control these effects and maintain consistent gold measurements. Common ISTDs used for gold analysis include ^{193} Ir or ^{209} Bi [7].

Carryover and Memory Effects

Gold can exhibit mild memory effects in sample introduction systems, particularly when concentrations vary widely between samples. Rinse solutions containing dilute aqua regia or hydrochloric acid (~20 % v/v) help remove residual gold and minimize carryover between samples.

Quality Control Considerations

Quality control procedures are critical for maintaining confidence in gold measurements. Exploration laboratories should incorporate method blanks, calibration verification standards, and duplicate samples within each analytical batch.

Certified reference materials representative of the deposit being studied should also be included where possible. Monitoring internal standard recovery and calibration verification results throughout the run helps identify matrix effects, instrument drift, or digestion inconsistencies [7].

Regular evaluation of these quality control samples ensures that ColdBlock digestion and ICP-MS analysis remain stable and reproducible across large exploration sample sets.

Conclusion

Gold analysis presents unique challenges due to heterogeneous distribution, coarse particle size, and complex mineral matrices. While fire assay remains the established standard for regulatory reporting

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and commercial laboratories, exploration programs often require faster analytical workflows to support operational decision making.

ColdBlock aqua regia digestion provides a rapid and controlled approach for processing larger sample masses in site laboratory environments. When applied to well characterized deposits and combined with careful sample preparation and ICP-MS analysis, the method provides reproducible and reliable gold measurements while significantly improving laboratory throughput.

By understanding the chemical, analytical, and operational considerations discussed in this paper, laboratories can effectively integrate ColdBlock digestion into exploration workflows while maintaining analytical integrity.

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Additional Resources

Application Note – Summary of Gold Digestions by Aqua Regia (Large Sample Size)

Application Note – OREAS 239 – Gold and Base Metals (Large Sample Size)

Video – Digestion of Copper Gold Concentrate for Gold Analysis using ColdBlock

Article – ColdBlock Digestion Technology Optimizes Assaying Process at Canadian Gold Copper Mine

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