

Rainbow initiated a phased test work programme at ANSTO Minerals in Sydney, Australia to develop a feasible, rare earth extraction, flowsheet for processing the phosphogypsum (“PG”) deposit located at Phalaborwa, South Africa.

The ANSTO Minerals test work programme was supported by various other studies and test campaigns at various facilities. The key programs can be summarised as follows:

- Filtration test work at Roytec Global in Johannesburg, South Africa
- Nano filtration desktop study at Chimerical in Cape Town, South Africa
- Site water analysis and basic neutralisation testing at SGS Johannesburg, South Africa
- K-Tech rare earth purification and separation desktop study carried out in Florida, USA

The initial test work programme, at ANSTO Minerals, has been designed as a two phased programme with the phase 1 programme completed July 2021.

The phase 1 programme covered the following milestones:

- Material characterisation and mineralogy
- Radiological classification
- Leach conditions and lixiviant selection
- Resin in leach evaluation
- Preliminary metallurgical variability response

Material characterisation and mineralogy

- Mineralogical evaluation indicates 96 weight % (wt.%) as calcium sulphate and a 3 wt.% calcium aluminium, rare earth rich, fluoride phase that contains the bulk of the rare earth minerals. The remainder of the rare earths are associated with refractory phases, for example monazite, and no rare earth association with the calcium sulphate phase was detected.
- Rare earths are not preferentially upgraded to any size fraction and are evenly distributed with particle size.
- Maximum rare earth recovery possible at mild to medium acid conditions will be in the range of 70 to 75% of contained total rare earths, with a similar response for the light rare earth group.

Radiological classification

- The gamma results of the phosphogypsum sample confirm a very low uranium content of 1.7ppm that is not in secular equilibrium.
- The thorium content also very low at 48ppm and in secular equilibrium, probably associated with the detected monazite phase.
- The IAEA International guidelines for exemption from regulation pertaining to radioactivity is that all radionuclides should be less than 1 Bq/g, so from this perspective the phosphogypsum would not be subject to the requirements of any regulations.

Leach conditions and lixiviant selection

- Hydrochloric and sulphuric acid stand out as the lixiviants of choice and outperform the nitric acid and calcium nitrate leach system, as previously postulated by Mintek.
- Hydrochloric acid leaching, as expected, results in partial gypsum dissolution, and as indicated by the mineralogy, does not improve the rare earth extraction compared to less costly sulphuric acid.

- Moderately elevated temperatures do not impact on the total extraction extent but significantly improve the dissolution kinetics.
- Increased slurry density in the leach impacts marginally on the extraction of rare earths due to suspected solubility constraints.
- Sulphuric acid control level in the leach must be in the region of 110-150g/L to ensure rare earth stability in the leach system but requires further optimisation in the phase 2 programme.

Resin in leach (“RIL”) evaluation

- Various resins were evaluated and were effective in rare earth absorption at mild acid conditions.
- High impurity co-loading, mainly calcium, resulted in very low rare earth loadings.
- In order to improve selectivity, the slurry pH needed to be increased and this impacted on the stability of the rare earths in the solution phase.
- The forecast rare earth loadings indicate an impractically large resin movement rate and associated elution system.

Preliminary metallurgical variability response

- 14 spatially distributed drill core samples were subjected to a standard sulphuric acid leach test with resin addition. Resin, at this stage, was still a contender but did not impact on the metallurgical response.
- The overall metallurgical response yielded an average total rare earth oxide extraction of 66% with associated minimum at 60% and maximum at 70%.
- Further variability work will be concluded in future but from an early stance the overall metallurgical response appears to be consistent, indicative of a homogeneous deposit.

Key decisions and conclusions

- Phosphogypsum will be hydraulically reclaimed from the stacks and pumped to the processing facility.
- In order to manage the leach slurry density and allow for reagent recycling, pre- and post-leach filtration steps will be included. The filterability of the material will need to be confirmed.
- Significant fluoride levels in the leach due to the targeted dissolution of the rare earth rich calcium aluminium fluoride phase.
- Fluoride removal and or control needs to be considered in the phase 2 test work campaign
- Sulphuric acid was initially selected as the lixiviant of choice due to cost, materials of construction, availability in the region and targeted dissolution of the rare earth rich calcium fluoride phase without significant gypsum dissolution.
- Although ambient leach conditions yielded acceptable results over a 24-hour leach period a moderate temperature adjustment may impact positively on capital reduction and risk mitigation and will be further considered during the phase 2 test work campaign.
- Resin in leach, as a concentration step, will not be further investigated due to the excessive impurity co-loading that was evident from these early tests.

At the conclusion of the phase 1 leach programme at ANSTO Minerals, Roytec Global in Johannesburg, South Africa was commissioned to complete pressure and vacuum filtration testing on the phosphogypsum material.

Filtration test work at Roytec Global

A composite sample similar in nature to the tested composite at ANSTO Minerals was divided and half the material was leached employing elevated temperature sulphuric acid leaching.

The two samples were then handed over to Roytec Global to perform pre- and post-leach filtration tests. The following key findings emanated from this test work:

- Material performed as expected and in line with original phosphoric acid process filters at Phalaborwa.
- Improvement in reclamation solids concentration will have a positive impact on the filter operation and sizing.
- Pre- and post-leach filters will yield an expected 25-27% cake moisture and a competent cake.
- Cake washing was tested and a three-stage counter current wash was modelled, indicating an expected >99% recovery of soluble rare earth elements.
- The use of filters allows for acid recycling over the leach circuit as well as the possibility of dry stack tailings deposition, that will have positive tailings management and environmental implications in terms of reduced capital and operating costs (“capex” and “opex”, respectively).

The phase 1 test work campaign and successful filtration testing highlighted the importance of acid recovery and recycling with an associated reduction in Pregnant Leach Solution (“PLS”) stream volume and improved rare earth solution tenor to feed to a downstream purification and separation circuit.

Nano filtration was selected as a technology to recover and recycle sulphuric acid and upgrade the downstream rare earth PLS grade. Chimerical Engineering, in Cape Town, South Africa, was commissioned to complete a desktop study to evaluate the application and performance of such a system using typical leach PLS stream compositions from the phase 1 ANSTO test campaign.

Nano filtration (“NF”) desktop study

The focus of the study was to model NF performance using speciation models that Chimerical has developed and proved on similar projects with actual feed stream compositions anticipated for Phalaborwa at the time of study.

The desktop study confirmed the following key findings:

- Nano filtration, subject to confirmatory test work, will recover 65% of the sulphuric acid for recycling to the front end of the leach circuit at similar strength than the PLS feed stream.
- Rare earth recovery to the retentate (concentrate) stream will be 99%.
- Rare earths will be concentrated by a factor of 3 in the concentrate stream.
- Fluoride will report with the acid to the permeate stream and thus recycle to the leach circuit.
- Test work will need to be completed to confirm saturation limits and maximum acid recovery, possible system scaling issues and membrane selectivity under actual conditions.

The technology provides an opportunity to recycle acid to the leach circuit that is critical due to the high levels of free acid required in the leach system. It also allows for fluoride management options on a rare earth barren stream and thus reduces the risk of rare earth losses during fluoride removal treatment.

Pending test work confirmation, it should be possible to further reduce the downstream processing volumes employing NF and optimise the overall circuit design. Test work will be initiated once an optimum leach regime has been established.

The desktop study allowed Rainbow Rare Earths to confirm a plausible conceptual design for the front-end processing circuit at Phalaborwa. The circuit will employ the large volumes of typical stack water on site as process water used for hydraulic reclamation and the bulk of the process water requirements.

This implies that continuous water treatment will be required to improve the water quality for use in the circuit. Water treatment options range from simple two stage neutralisation that produces more gypsum that requires long term stacking and/or focused treatment to yield saleable products. It is envisaged at this early stage that a hybrid water treatment facility will provide all the water requirements for the project whilst improving the overall site wide water quality over time.

Water neutralisation test work

Stack water samples from site were collected and sent to SGS Johannesburg to perform basic neutralisation test work employing either slaked lime or limestone. The intent was to evaluate water quality improvement at various pH intervals as well as volumes of precipitate formed and reagent consumptions.

The following key findings emanated from this volume of work:

- Neutralisation improves water quality and significantly reduces fluoride present in the stack water.
- Various water qualities are possible with a multistage circuit.

With all of the above in mind, the phase 2 test work campaign was initiated at ANSTO Minerals to investigate the following objectives and scope:

- Pre-leach impurity control
- Optimise leach parameters
- Optimise PLS quality and quantity

Pre leach impurity control

The key focus was to reduce impurities, mainly phosphates and fluoride, prior to the leach circuit. The intended approach would then allow these impurities to be dealt with in the water neutralisation circuit and not the leach circuit.

Key findings can be summarised as follows:

- Hydraulic reclamation will alter the hydration state of the long term stored phosphogypsum prior to the leach.
- Hydraulic reclamation and pumping at a 25 wt.% solids concentration coupled to pre-leach filtration will significantly reduce potassium, magnesium and sodium levels as well as to a lesser, but still significant extent, the phosphates and fluorides present in the raw phosphogypsum feed. This step will also reduce impurities present in the pore water in the stacks.
- Historical work completed by Mintek postulated a fluoride removal step that consisted of adding concentrated sulphuric acid to the filter cake to reduce the fluoride through

volatilisation. This technique did not prove to add any value and no fluoride could be reduced through this process.

Optimise leach parameters

The key leach parameters with respect to sulphuric acid were investigated and optimised.

- The acid concentration in the leach can be reduced to 110 g/L without impact on the rare earth extraction or stability in the leach circuit.
- The optimum temperature is 40°C that will result in a 12-hour leach period significantly reducing Capex and Opex.
- The practical designed leach slurry density will be 30 wt.% solids.

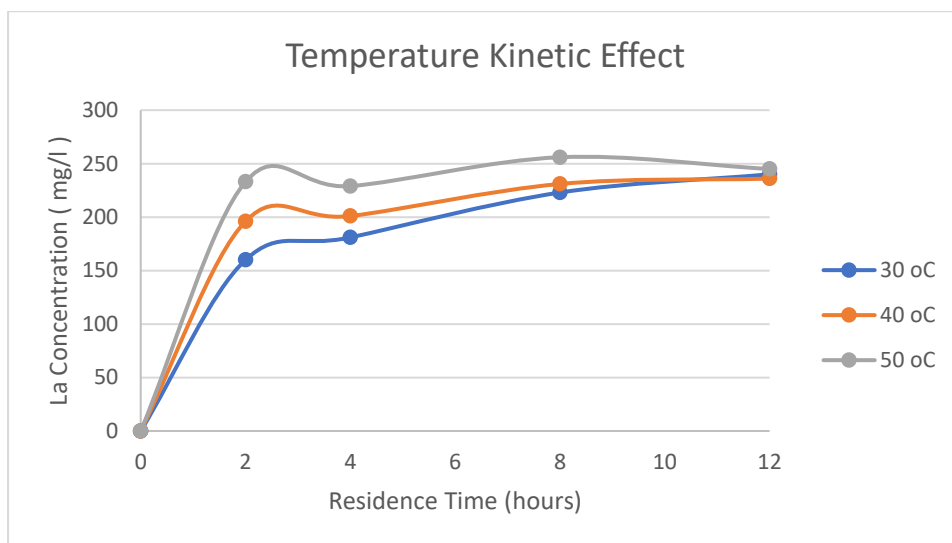


Figure 1: Temperature Impact on leach kinetics

The above graphical representation illustrates, within experimental error, how the increased temperature improves leach kinetics with both the 40 and 50 °C leaches essentially completed after 8 hours. All three leaches reached a similar extraction after 12 hours.

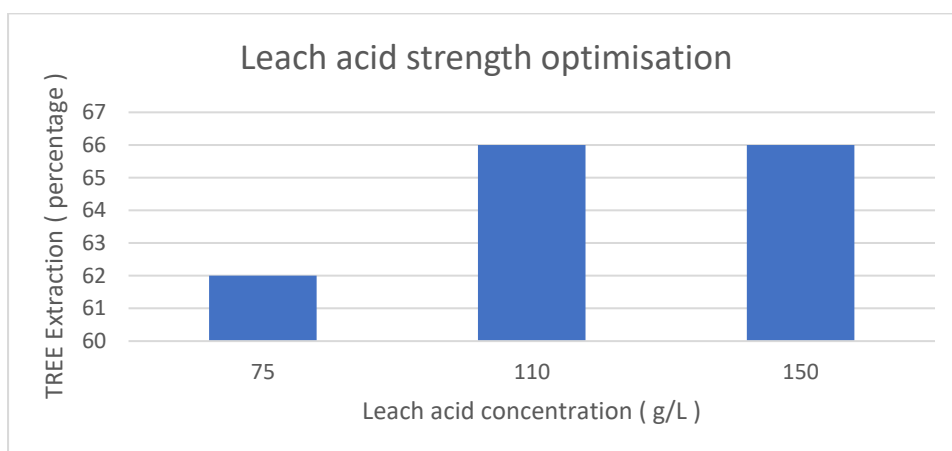


Figure 2 Leach acid strength optimisation

Figure 2 illustrates the leach acid strength optimisation and the selection of 110 g/L as the control level for the leach.

With these parameters confirmed the focus shifted to improving the PLS quality and quantity through PLS recycle testing.

Improve PLS quality and quantity

Under optimised conditions, and using pre-washed phosphogypsum as feed, the leach was tested in closed mode employing five cycles. The leached slurry was filtered, and the primary filtrate used to repulp fresh feed for the next cycle leach. Reagent levels were measured and adjusted as required.

The following are the key observations:

- Key impurity levels spiked, most notably the fluoride levels, as could be expected since the target mineral leached is a calcium aluminium fluoride phase.
- These impurity levels impacted on the rare earth extraction.
- Offline precipitation tests were conducted employing process solution that was dosed with HF and H₃PO₄ to simulate various impurity levels. It was established that the fluoride had the most pronounced effect on the rare earth stability in solution.

This test work clearly highlighted the importance of fluoride management in the leach and recycling solutions to successfully improve the PLS quality and quantity from a downstream circuit efficiency and economic perspective.

Key decisions and conclusions

- Pre-leach impurity control will be critical in the final flowsheet and all future work will be completed with phosphogypsum washed with gypsum saturated process water.
- Testing that incorporates actual neutralised stack water as process water will be scoped in the phase 3 programme.
- Key, sulphuric acid, leach regime parameters established and confirmed.
- PLS recycling or other counter current leaching systems will need to be critically investigated in a trade-off study to improve downstream PLS volumes and grade in conjunction with fluoride control strategies.
- Alternative fluoride control strategies will be tested over the next phase of test work started in the week of 31st January 2022 to finalise the front-end flowsheet for Phalaborwa.

In order to purify and separate the target rare earth elements and produce final products for sale, a host of technologies have been investigated by Rainbow Rare Earths. K-Technologies from Florida in the USA has been selected as the best fit partner, to employ their technology, for the development of a downstream solution for Phalaborwa.

K-Tech rare earth purification and separation desktop study

K-Tech was commissioned and delivered a desktop study late December 2021. The report covered PLS stream concentration, purification and separation of the targeted rare earth elements namely neodymium, praseodymium, dysprosium and terbium up to final separated oxides.

K-Tech also considered various alternative options that can support the front-end leach circuit pending further test work confirmation.

Key findings can be summarised as follows:

- It is critical to reduce PLS volumes and improve rare earth grades in the PLS to ensure a feasible long-term processing solution.
- Product options that can be considered range from NdPr oxide, Dy oxide, Tb oxide, low cerium mixed rare earth concentrate or a low cerium Nd,Pr,Dy,Tb product. The balance of the rare earths can be stored for future use.
- The estimated opex and capex can be significantly improved if the PLS volume can be reduced to the 40-60 m³/h range feeding K-Tech.
- The desktop study includes a duplicate NF system to further reduce the volumes predicted by Chimerical. K-Tech is confident that further upgrading using NF should be possible based on their commercial experience.
- They forecast, pending final test work, good purity levels for the oxide products in the range of 99.5 to 99.9%.
- They assumed a conservative circuit recovery of 80% for the oxide products but are confident that this can be improved once final test work has been concluded.
- It might be possible to employ a cheaper carbonate intermediate product and tailor the product suite to phases of project implementation with deferred capex and opex impacts.
- Can produce a cerium depleted mixed rare earth carbonate product with significant capex and opex improvements as a starter project with a phased move to the refined products.
- K-Tech supports a pre-wash prior to the leach as currently planned for Phalaborwa.
- Support the use of the existing gypsum stack water post neutralisation as process water for large portion of the process (adding to the ESG credentials of the Phalaborwa project), with the exception of high purity water requirements for certain aspects in the continuous ion exchange ("CIX") and continuous ion chromatography ("CIC") sections. The high purity requirements are low in terms of overall volumes.
- K-Tech can also consider their water treatment technology that can remove impurities and generate saleable products, and not just a mixed waste that require storage, to improve opex costs.
- K-Tech recommends the following to further optimise the capex and opex of the Project ;
 - Investigate counter current leaching to improve PLS tenor and reduce volumes
 - PLS recycle to improve PLS volumes
 - Alternative concentration technique that can negate the use of NF all together or implicate a lot smaller NF circuit than the present anticipated circuit
 - Consider various water treatment options
 - Kick-off a bench testing campaign to improve on the desktop study and firm up assumptions that form the basis of the desktop study.

The detailed test work programme dealing with all the trade-off suggestions highlighted above as well as test the K-Tech technology, namely continuous ion exchange and continuous ion chromatography, for purification and separation of the target products for Phalaborwa is already underway at K-Tech.

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Glossary of terms

Bq/g	Becquerel per gramme, a unit to measure radioactivity
Cake washing	Wash water applied to filtered solid to remove impurities or target solution
Continuous ion chromatography (CIC)	Rare earth separation as individual groups or elements in continuous fashion.
Continuous ion exchange (CIX)	Extract the rare earths from the PLS stream and produce a concentrated rare earth solution
Filter cake	Solid product produced as a result of filtration
H ₃ PO ₄	Boric Acid
HF	Hydrofluoric Acid
Hydraulic reclamation	To recover a solid from a storage facility through the use of high pressure water
Leach circuit	System of tanks where a valuable element is recovered
Lixiviant	A liquid medium in hydrometallurgy used to selectively extract the desired metal from a mineral
m ³ /h	Measurement of flow in cubic meters per hour
Nano filtration	Pressure driven filtration through a membrane
Neutralised stack water	Gypsum stack water that has been treated with a neutralising agent like lime
Phosphogypsum	Gypsum produced as a result of phosphoric acid production
Pore water	Water contained or locked in solids
ppm	Parts per million
Pregnant Leach Solution (PLS)	A solution that contains the extracted valuable metal
Water Neutralisation	Water treatment employing lime or limestone
wt.%	Measurement of composition based on mass, weight percentage