

# Novel Diglycolamide Extractant?s Performance in Liquid-Liquid Separations for Pilot Scale Application

August 2022

Addyson Lyn Barnes, Mitchell Greenhalgh, Ramedy G Flores, Kevin L Lyon, Mac Foster, Bruce Moyer, Philip Keller, Santa Jansone-Popova, Katie Johnson





### DISCLAIMER

This information was prepared as an account of work sponsored by an agency of the U.S. Government. Neither the U.S. Government nor any agency thereof, nor any of their employees, makes any warranty, expressed or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness, of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. References herein to any specific commercial product, process, or service by trade name, trade mark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the U.S. Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the U.S. Government or any agency thereof.

### Novel Diglycolamide Extractant?s Performance in Liquid-Liquid Separations for Pilot Scale Application

Addyson Lyn Barnes, Mitchell Greenhalgh, Ramedy G Flores, Kevin L Lyon, Mac Foster, Bruce Moyer, Philip Keller, Santa Jansone-Popova, Katie Johnson

August 2022

Idaho National Laboratory Idaho Falls, Idaho 83415

http://www.inl.gov

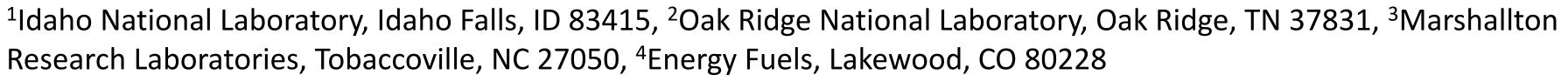
Prepared for the U.S. Department of Energy Under DOE Idaho Operations Office Contract DE-AC07-05ID14517



### Novel Diglycolamide Extractant's Performance in Liquid-Liquid Separations for Pilot Scale Application

### **Enhanced Separations of Critical Materials**

Addyson Barnes<sup>1</sup>, Santa Jansone-Popova<sup>2</sup>, Mac Foster<sup>3</sup>, Ramedy Flores<sup>1</sup>, Mitch Greenhalgh<sup>1</sup>, Philip Keller<sup>4</sup>, Bruce Moyer<sup>2</sup>, Kevin Lyon<sup>1</sup>, Katie Johnson<sup>2</sup>





### Background

- Modified diglycolamide (DGA) extractants show high affinity for light lanthanides and improved separation factors compared to phosphonic acids used commercially.
- Previous studies with DGAs included flowsheet design and implementation into solvent extraction equipment but with low extractant concentration. Modifications on the alkyl chains of previous DGA extractants led to a competitive product for light rare earth separation with a higher extractant concentration.
- The scope of this project is to test an optimized DGA extractant for application in pilot scale solvent extraction equipment.



Longer residence time of approx. 3 min

Larger hold up volume: 600 mL

Separation of phases caused by gravity



### **Centrifugal Contactor**

Residence time on the order of seconds

Minimal holdup volume: 200 mL Phase separation aided by centrifugal

force

## **Equipment Testing**

Previous dispersion studies led to variable and subjective values. Phase disengagement compatibility with solvent extraction equipment was therefore determined experimentally.

### **Phase Carryover**

- Caused by entrainment or slow disengagement. Typically, <1% carryover is acceptable for operating conditions.
- Conditions: O/A=1, Aqueous feed  $[H^+]=3$  M
  - Mixer Settler: 0.2% aqueous carryover
  - Centrifugal Contactor: 0.1% aqueous carryover
- Extractant dispersion performance was fair, so centrifugal contactors may be a more ideal equipment type due to added separation forces.

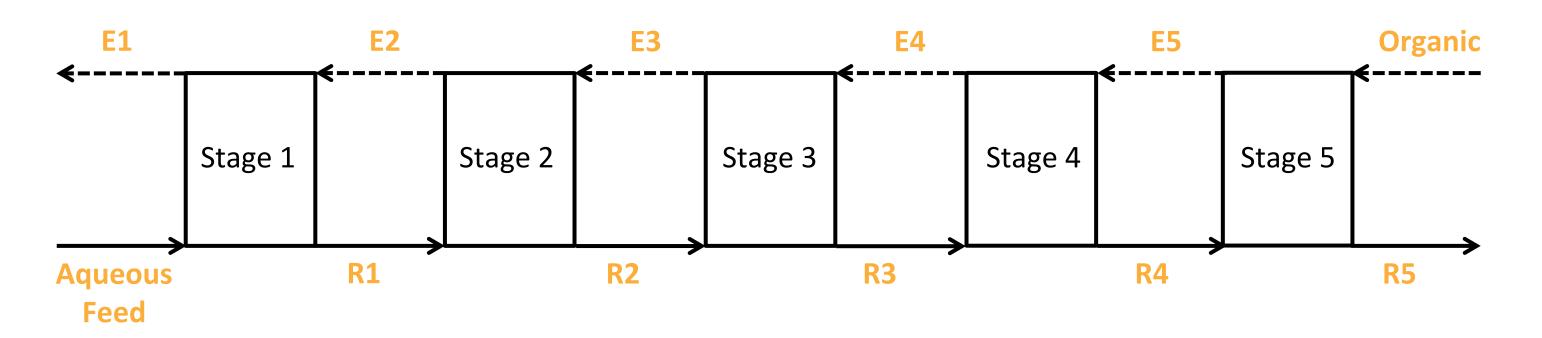
### Selective Scrubbing

- Single stage scrubbing tests with loaded organic and simulated strip product feed were performed to demonstrate the exchange of co-extracted La and Ce with heavier lanthanides.
- An associated bench scale, acid dependency test with the same parameters showed an increase in Pr and Nd extraction but a decrease in La and Ce scrubbing with an increase in acid concentration.

	Mixer Settler	Centrifugal Contactor
% lost of La+Ce	32%	61%
% gain of heavies	131%	43%

### **Counter Current Batch Contacts**

- Matrices of batch shakeouts simulate steady state results of a five-stage extraction cascade and a five-stage scrub cascade.
- Distribution ratios were extracted and implemented into flowsheet modeling to calculate optimal number of stages.



### Distribution Ratios for Extraction -La -Ce -Pr -Nd 10.00 **Distribution Ratio** 1.00 0.10 0.01

Stage 3

Stage 4

Stage 5

Stage 2

Stage 1

### **Future Work**

- Implementation into a cascade of 30 mixer-settlers to test flowsheet model and verify pilot scale performance
- Consider life cycle of extractant and any consequent modifications necessary to maintain integrity for large scale application
- Collaborate with other industries to demonstrate the modified DGA's extraction capabilities