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Cryogenic purification of low purity helium with temperature swing adsorption and membrane separation

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Abstract. Ability Engineering Technology (AET) has developed a system capable of receiving helium at very low concentrations (less than 10% by volume) and effectively separating out undesirable gases to yield high purity helium (5.0 – 6.0 grade) at high recovery rates (greater than 90%). This is accomplished with membrane separation and a subsequent cryogenic adsorption process. The operational advantages of utilizing this method for helium recovery will be discussed. The results of field testing this equipment and its implementation for scientific and industrial use cases will also be discussed.

1. Introduction

AET has produced a system capable of separating helium from mixtures where it is present in very low concentrations (single digit percent concentrations by volume) and processing it to very high purities (3.0 – 6.0 grade helium). In this context, the initial processing work is carried out by multiple stages of gas membranes. The post processing work is carried out by a cryogenic temperature swing adsorption-based purifier. The combination of both technologies allows a broad range of processing capability not typically seen in either process independently or in similar processing techniques.

This paper will first address the theoretical potential of such a system. The proposed system used to evaluate this technique will then be discussed, along with brief consideration of the design and construction of these technologies. The testing of these systems will then be discussed, along with their performance. Conclusions will then be drawn as to the feasibility and practicality of such a system, its uses, strengths and weaknesses. Brief mentions will also be made of plans for future development of the technology.

2. Membrane purification

Membranes have been applied in gas separation for decades, particularly in oil and natural gas processing, where they are established for tasks such as nitrogen rejection, natural gas sweetening, and hydrogen recovery [1]. More recently, interest has extended toward helium recovery, with several companies promoting new membrane technologies. However, published studies consistently note that while membrane separation is technically feasible, data on performance for very low-concentration helium feed streams remain limited [2]. As a result,

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AET engaged with a leading global supplier of membranes to evaluate performance. The goal was to assess the symmetry between empirical performance and theoretical data, to design and build systems capable of utilizing the technology in novel ways, and to implement those systems.

2.1 Membrane traditional applications

Membranes have traditionally been deployed in a wide range of gas separation applications, including biogas upgrading, nitrogen generation, hydrogen separation, and natural gas processing, with specialized membrane modules available for each task [1].

2.2 Membrane simulation

Due to confidentiality agreements with the membrane supplier, detailed simulation parameters and performance curves cannot be disclosed. In general terms, the simulation was based on a feed stream containing 8% helium with the balance primarily nitrogen and minor amounts of methane and argon. Using standard stage-cut assumptions and reported He/N_2 selectivity values from the literature [1–3], the model predicted that a two-stage configuration could enrich the helium to approximately 65–70%. These predictions were subsequently used to guide the design of the AET test skid. While the detailed numerical results cannot be published, the experimental results presented in Section 5.1 qualitatively confirmed the enrichment trends observed in the simulation, consistent with previous studies on helium–nitrogen separations [1–3].

2.3 Membrane test skid

AET designed and fabricated a test skid suitable for evaluating the simulation flow. The AET membrane skid is outfitted with a gas dryer for feed gas moisture removal, two horizontally mounted large flow membranes (1st stage), three vertically mounted medium style membranes (second stage), and a buffer tank used for retentate/permeate collection. The skid is additionally outfitted with all the valves and instrumentation necessary to direct feed gas through the various stages and measure their concentrations.

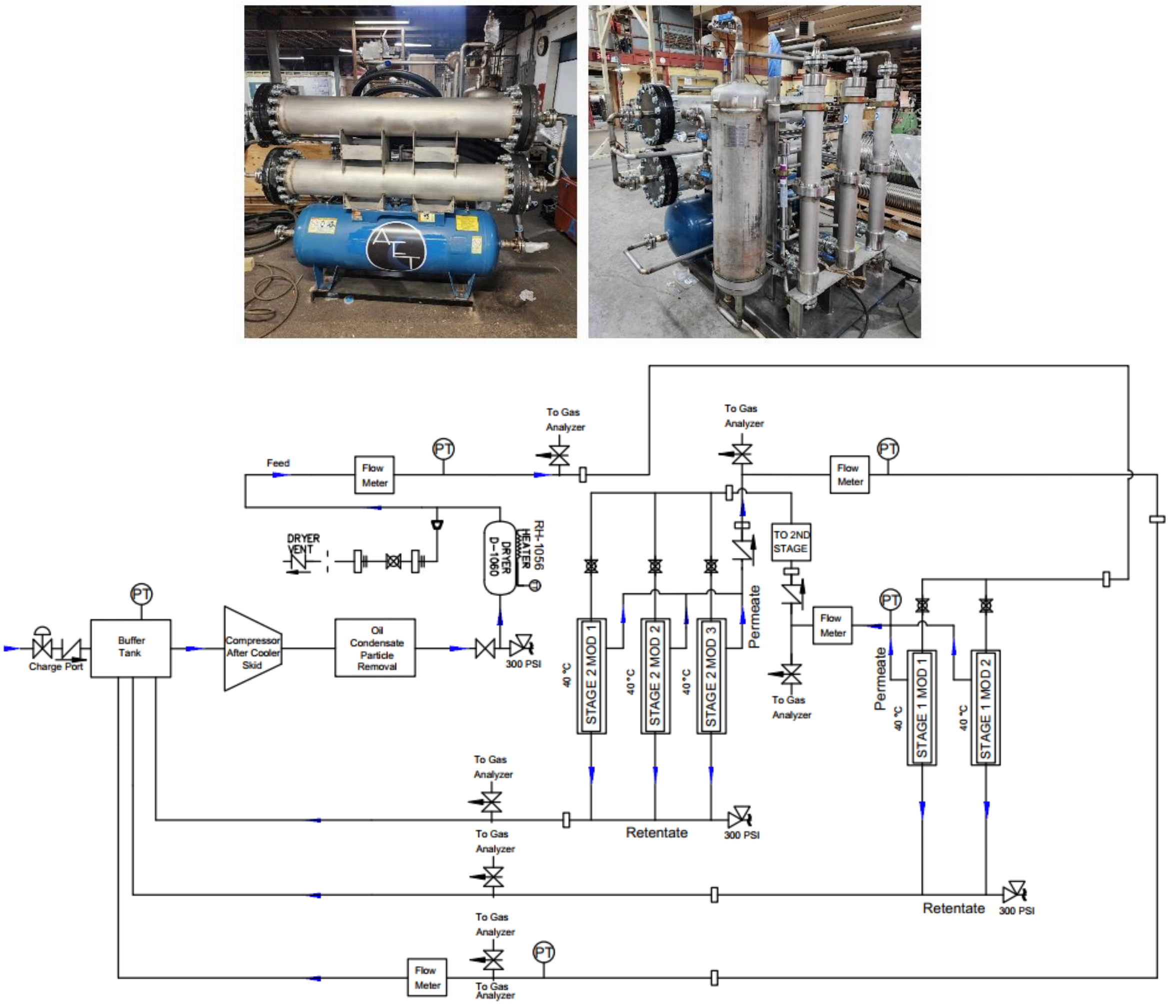

Figure 1. Shown is the AET membrane test skid. The skid is assembled with two stages of gas membranes, a gas dryer, and a buffer tank for gas collection and recirculation. The P&ID shows the overall process of how the test skid works.

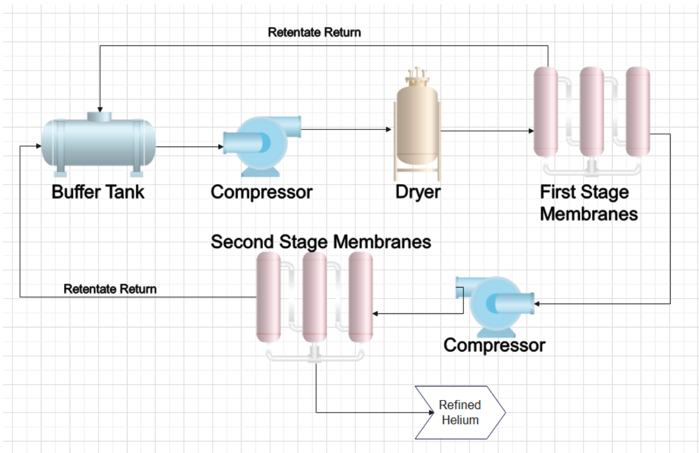

Figure 2. Process flow diagram of a closed-loop helium separation test skid. Low-purity helium feed gas is dried, compressed, and enriched through two membrane stages, with all streams recycled via a central buffer tank until steady state is achieved.

3. Cryogenic purification

Cryogenic temperature swing adsorption has been around for many years and has become one of the fundamental methods for helium recapture and purification for both industrial and scientific purposes. AET has specialized in cryogenic purification for many decades. As a result, AET has been able to serve the scientific community, providing helium purifiers for laboratories conducting large scale helium refrigeration, liquefaction and recovery activities. AET has also been a big contributor to the industrial community, providing purification and recovery equipment to specialty gas manufacturers, hypersonic wind tunnel operators, semi-conductor component assemblers, and many others.

With such a diverse background in helium recovery and purification, AET has made many advancements in the world of purification technology. As a result, AET has specialized the design of a unique gas purification product series. As detailed on the AET website [4], this purification product series targets users both large and small and covers a range of capability.

3.1 Temperature swing adsorption

Temperature swing adsorption works off the Van der Waals principle. As temperature decreases, gas molecules become less energetic and have a higher tendency to adhere to surfaces with vast surface area due to molecular surface tension. This fundamental adsorption behavior, well established in the literature [5], is the basis for the use of materials such as activated charcoal, molecular sieve, and silica gel in gas purification. Conducting this process at cryogenic temperatures dramatically increases the adsorption capability of these materials, making TSA a reliable method for removing undesirables from a flow stream and producing high purity gas [6]. This also makes the reusability case viable, as reversing the process by heating the adsorption material effectively allows all trapped substances to be desorbed and removed during regeneration.

3.2 AET's approach to cryogenic purification

AET purifiers bring in a flow of gas that is first dried by an ambient temperature, adsorbent based gas drying vessel. The temperature of the feed gas is then incrementally reduced to liquid nitrogen temperatures via strategically placed heat exchangers. Larger heat exchangers exchange heat between the inbound and outbound flow streams, while the smaller heat exchangers exchange heat between the gas stream and a flow of liquid nitrogen. Liquid nitrogen is the main consumable in this operation. Once the feed gas has reached nitrogen temperatures, it is passed over the adsorbent, at which point all impurities are effectively adsorbed from the gas stream. The gas then returns to the primary heat exchanger and leaves the purifier near ambient temperature, consistent with conventional cryogenic TSA purification schemes described in the literature [7,8].

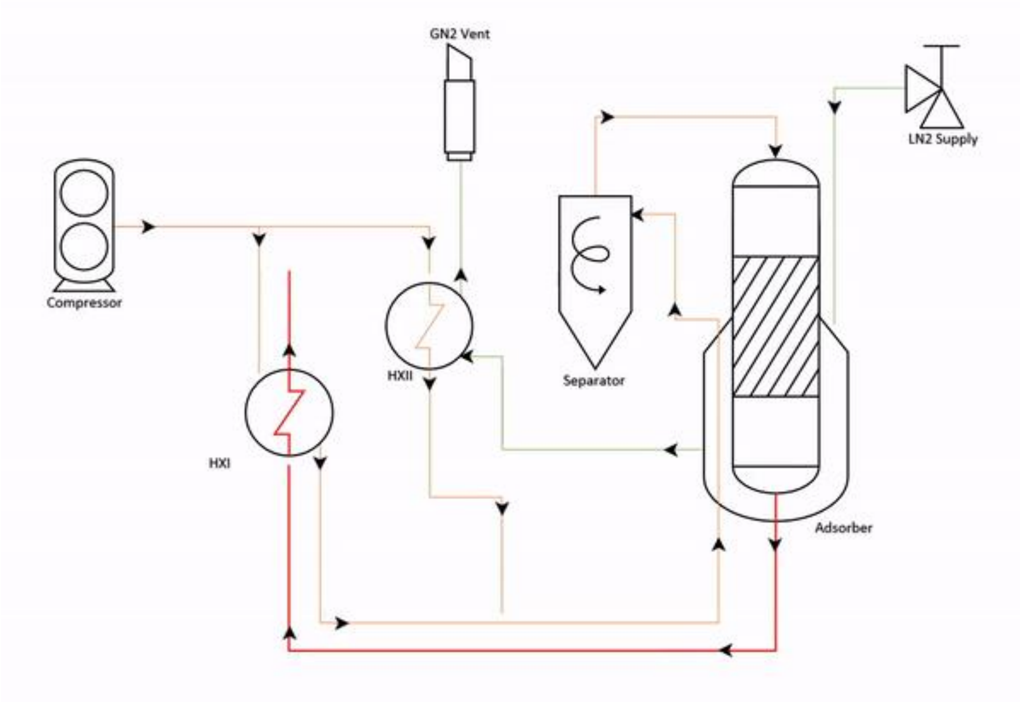

Figure 3. Shown is a flow diagram of the basic purification process in AET purifiers.

3.3 Cryogenic purification – Traditional Performance

AET's cryogenic purifiers range from large to small, with flowrates as low as 10 scfm to as large as 3200 scfm for helium, pressures from 10 bar to 200 bar, and outlet purities as high as 99.99999% (7.0 grade).

Traditionally, cryogenic TSA purification systems require inlet helium concentrations of no less than 90–95% to consistently guarantee 5.0–6.0 grade helium at the outlet. Relatively few suppliers of gas purifiers will guarantee even this performance. Similar limitations have been reported in large-scale scientific facilities, where cryogenic adsorption units are employed for helium recovery and purification [7].

3.4 AET HP Series cryogenic gas purifier

To address the issue of providing equipment capable of producing high purity grade helium from lower concentrations, AET designed and constructed the HP (High Pressure) series purifier, in both standard and UHP (Ultra High Pressure) variants.

The HP series purifier has been designed to be capable of receiving helium concentrations as low as 70% and concentrating the outlet helium to purities of 99.99% – 99.9999% (4.0 – 6.0 grade). The UHP version can achieve 99.99999% (7.0 grade). The HP series typically operates between 1000 – 3000 psig, with flowrates between 50 NM3/hr – 400 NM3/hr, as documented in the manufacturer's technical specification [4] and consistent with the operating envelopes reported for cryogenic purification systems in fusion and large-scale research facilities [7].

Equipment in the marketplace with this capability is not widely available or known. To date, the HP series has been extensively tested with both Helium and Hydrogen gas at normal inlet concentrations (90 – 95% helium purity) with proof of 5.0 – 7.0 grade performance at the outlet via gas chromatography, but the system had yet to be tested at the extremities of its performance range prior to the commencement of this research.

3.5 AET HP series test unit

To rigorously test the HP series' capabilities, an experimental unit was designed and constructed by AET for use in processing gas mixtures for a hypersonic wind tunnel facility. The gas recovery system at this wind tunnel test facility serves as the perfect environment for research tests against the HP series performance, as the facility typically has varying gas mixtures to process regularly with very high concentrations of nitrogen gas.

This HP unit was commissioned to receive helium at 85%, with the balance being nitrogen gas. The target purity for the system was only 99.9% (3.0 grade). The system was designed and constructed with special measures and components specifically meant to achieve this, which it does adequately. Based on further design enhancements—including increased cooling capacity in the adsorption circuits and improved operating capability of the liquid air separator—it was anticipated that the unit could also operate effectively at lower inlet helium concentrations while still achieving high outlet purity. This expectation, while not yet verified at the time, provided the rationale for testing the system beyond its original design specification.

Figure 4. Shown is the AET HP series purifier built for processing gas mixtures at a hypersonic wind tunnel facility. This HP series was designed to receive 85% He/15% N_2 mixtures and purify to at least 99.9% (3.0 grade). Based on design enhancements to the adsorption circuits and liquid air separator, the system was anticipated to handle lower inlet helium concentrations while maintaining comparable outlet purity.

4. Low concentrated helium to high purity grade helium

The goal of this research is to combine the capabilities of the two technologies discussed thus far. Membrane purification can theoretically take helium gas from single digit percent concentrations by volume and bring it up to concentrations of 60 – 70% or higher depending on the circumstances. Cryogenic purification systems, such as the HP series, can theoretically take helium gas from concentrations of 60 – 70% by volume, and purify it to an outlet grade in the 4.0 – 6.0 region. As a packaged system, both technologies working together should be able to receive single digit percent concentrated helium and process it to 4.0 – 6.0 grade helium.

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5. System test

A full systems test is devised to evaluate the operational performance and capability of both membrane and cryogenic purification systems working together to bring ultra-low purity helium to high purity grades. The main goal of this test is to prove feasibility. The membranes should perform as expected based on the proposed simulation data, and the HP series purifier should outperform its traditional use cases based on AET's design improvements. The full systems test should clearly show the membrane skid receiving ultra-low pure, single digit percentage grade helium gas, the HP series purifier should produce 4.0 – 6.0 high purity grade helium, and the membrane skid's final helium gas concentration shall be the HP series purifier's inlet concentration.

5.1 AET membrane skid system test

A test set up is derived from the membrane simulation results and the AET membrane test skid's design of construction. The test involves two hermetically sealed, rotary screw helium compressors, one for each stage of membranes. A blend of specialty mixed gas is purchased to feed both the membrane skid and the first stage compressor. The gas mixture represents a close match to the initial starting gas mixture defined in the membrane simulation.

Table 1. The table shows the purchased mix gas components and concentrations. This gas mixture will be fed to the AET membrane test skid to evaluate performance capabilities.

Component	Concentration Requested	Actual Concentration
Carbon dioxide	1%	0.9998%
Methane	1%	1.0071%
Argon	1%	0.9969%
Helium	8%	8.000%
Nitrogen	Balance	Balance

<u>Certified Standards</u>
Preparation Tolerance: +/- 2 % of Component
Certification Tolerance: +/- 2% of Component

Figure 5. Shown is the AET membrane skid test setup. The membrane skid is shown connected to two rotary screw helium compressors.

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The equipment is controlled and monitored by a simple PLC mounted to the membrane skid, and two additional independent PLC's on each compressor skid. The membrane skid PLC monitors feed, permeate and retentate pressures across each stage of membranes. Sample taps are placed at each feed, permeate and retentate stream across the skid so helium concentrations can be measured in each location. The compressor PLC's keep both compressor skids running, while gas bypass flow control is adjusted by hand.

The raw mixed gas is fed to the first stage compressor, and the compressor is brought online. As the first stage compressor approaches 250 psi discharge pressure, it is allowed to gradually feed the first membrane stage on the test skid. As the pressure builds at the first stage of membranes, the retentate flow is allowed to expand back into the central buffer tank located on the test skid. Gas is then gradually fed back to the suction of the first stage compressor from the buffer tank, creating a semi-closed loop flow. As this is happening, the first membrane stage begins to slowly permeate gas to the suction of the second compressor stage. A handheld helium analyzer is placed on the first stage permeate sample tap, and 21% – 30% helium concentrations are observed at this location during start-up.

As the first stage permeate gas feeds over to the second stage compressor's suction, the second stage compressor is also brought online. The second stage compressor is allowed to gradually build its discharge pressure. As it does, the compressor discharge is gradually fed to the second stage of membranes on the test skid at approximately 165 psi. The second stage retentate is allowed to expand back into the buffer tank, feeding the first stage compressor suction. As this happens, the second stage permeate begins to generate gas, which is also directed back to the buffer tank feeding the first stage compressor.

The second stage permeate gas sample tap is accessed with the handheld helium gas analyzer. As the pressures and flowrates build to expected operating conditions at each membrane stage, the final permeate sample tap reaches >60% helium gas concentration, with the balance of the mixture being nitrogen. It should be noted that the permeate concentration at this location did not remain at a steady value. Because compressor bypasses and stage pressurization were adjusted manually, the system was subject to transient fluctuations in pressure and flow. As a result, the permeate concentration often oscillated between 40–60% and occasionally spiked above 60% when conditions were briefly optimal. These spikes were observed in real time during operation, although capturing them photographically proved difficult due to the delay between valve adjustments and reaching the analyzer screen. The representative photograph in Figure 6 shows a reading of 57.8% during a downward fluctuation, taken immediately after a higher value had been observed.

Figure 6. The handheld gas analyzer is shown connected to the second stage membrane permeate stream. At the time this photo was taken, the permeate stream had reached 57.8% purity. Due to manual operation and transient pressure/flow fluctuations, helium concentrations at this point varied significantly, with real-time readings occasionally exceeding 60%.

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5.2 AET HP series

To set up the HP series purifier for testing, a gas mixture is prepared to have 57.6% helium concentration with the balance of the gas being nitrogen. This helium concentration is less than what the membrane test skid achieved during its test run. Enough feed gas is prepared so that the purifier can run for 8 – 12 hours.

The purifier's operating pressure and flowrate are adjusted to match the best-case scenario for proper nitrogen separation from the helium gas. Due to the partial pressure of nitrogen in the mixture, the best run conditions are selected so that the purifier's air separator can perform optimally. Several checks are made with calculations and proposed flow scenarios to ensure that the nitrogen remaining in the purifier after separation isn't enough to overcome the adsorption capability of the unit's primary cryogenic adsorption vessel.

The purifier is backfilled with pure helium and cooled down to cryogenic temperature with liquid nitrogen. The unit is allowed to cool long enough such that the primary adsorber vessel reaches a low steady state temperature. Once achieved, the unit is brought online, and helium is allowed to flow through the cryogenic purification circuit.

The gas mixture entering the purifier is maintained between 57%-58% helium concentration consistently. Operating temperatures, pressures and flowrates are maintained during the run cycle. The purifier is allowed to operate for at least 2 hours continuously before the first sample is taken. This is done to ensure nitrogen separation and adsorption achieve full optimal and steady conditions, and the purifier's outlet piping is sufficiently purged. Multiple gas samples are taken on the purifier's pure gas sample tap using grab sample cylinders, a trace nitrogen in helium analyzer, and a trace moisture analyzer. On average, the purifier shows outlet purity of approximately 2ppm N2 and 4ppm moisture. With 6ppm impurities total, the purifier demonstrates its ability to generate helium at >99.999 purity (5.0 grade) under these operating conditions.

Figure 7. Pictured above are three separate gas analysis results related to the HP series purifier test run. Pictured on the left is the purifier inlet analyzer which shows helium gas entering the purifier at 57.6% purity. On the right are the trace nitrogen in helium analyzer along with the moisture analyzer. The purifier outlet sample reads 2.06 ppmV on the nitrogen analyzer, and 4.4 ppmV on the moisture analyzer.

6. Result conclusions

The AET membrane skid and the AET HP series purifier have successfully demonstrated the ability to purify helium gas from 8% concentration in nitrogen to >99.999 (5.0 grade) purity. This test shows the viability of the membranes to effectively separate fast diffusing helium particles from larger, slower diffusing molecules such as nitrogen. The test also shows the capability of the AET HP series purifier to effectively separate large quantities of nitrogen gas and maintain optimal adsorption performance throughout. The two systems working together demonstrate a viable two-step separation process where the membranes do the initial gross removal of nitrogen while the cryogenic purifier accepts the membrane outlet gas and does final gross removal and polishing of the helium.

7. Future plans

Future test runs are planned to further optimize the performance of both the membranes and the purifier. Changes will be made to how the helium compressors are brought online, and improvements will be made for handling gas bypass flows and optimal flowrates to each membrane stage on the test skid. This should yield performance closer to expected outlet helium purity, which was expected to be 68.5% instead of 60%. More analysis data will be collected and analyzed to determine the ultimate helium losses incurred, and the exact deviation of membrane performance against the simulation. The modular nature of the test skid's larger membranes along with the skid's capability to accept a third stage of membranes for further gas refinement will also be explored.

Enhancements will also be made to the HP series to optimize its performance capability. Additionally, improvements to the gas sampling technique and system will be implemented, as it is likely that the moisture readings observed were due to introduction of air at sample connection points, and improper purging.

8. Conclusions

AET has successfully developed and demonstrated the operation of a system capable of taking a mixture of 8% helium in nitrogen and purifying the helium up to >99.999% (5.0 grade) purity. This has been accomplished with membrane separation and cryogenic temperature swing adsorption. Helium recovery rates are expected to be near 90% but further testing and analysis will need to be conducted to further verify recovery numbers and anticipated helium losses.

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