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Stability Analysis of Feed Gas Based on Pressure using Gas Chromatography at PT Badak NGL

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Abstract: As one of the LNG processing companies in Indonesia, Badak NGL plays an important role in product QA and QC. Laboratory and Environment Control PT Badak NGL obtained ISO 17025 as a testing and calibration laboratory. In clause 7.7 of ISO 17025, stated that each laboratory is required to improve competence, one of which is by holding interlaboratory meetings. Currently, PT Badak NGL is participating in the implementation of the Inter Laboratory Meeting (ILM) in East Kalimantan. In the implementation of the ILM 112th and 113th in 2022, several laboratories provide outlier results in natural gas sample testing. Various factors affect the results of natural gas sample testing, one of them is the sample pressure. This research aims to determine the minimum pressure limit in testing feed gas samples. The test is carried out by designing a series of two cylinders to test the repeatability value. The sampling process is based on GPA 2166 and the measurement of repeatability values is based on GPA 2261. This method is effective for determining the limit of repeatability values as a reference for the minimum sample pressure before being injected into Gas Chromatography.

Keywords: Feed gas; Pressure; Repetability; Gas Chromatography.

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1. Introduction

PT Badak Natural Gas Liquefaction or known as PT Badak NGL is the largest liquefied natural gas processing company in Indonesia and one of the largest LNG refineries in the world. The company is located in Bontang, East Kalimantan, and has 8 process trains (A - H) capable of producing 22.5 Mtpa LNG (million metric tons of LNG per year).

PT Badak NGL is a non-profit company that acts as an operator where natural gas is shipped from producers. Annual report PT Badak NGL 2022 mentioned that Badak LNG obtained feed gas from Muara Badak and supplied by Chevron Indonesia, Pertamina Hulu Sanga-sanga, Pertamina Hulu Mahakam, ENI. Gas from these producers is then piped to a gas processing facility.

As an non-profit company, PT Badak NGL has an important responsibility in reporting the results of testing the compotition of feed gas sent by producers. The result of testing the compotition of the delivered feed gas affects the sale and purchase contract between the producer and the buyer. Therefore, PT Badak NGL must be fully responsible for the implementation of feed gas testing.

PT Badak NGL succeeded in obtaining ISO 17025 accreditation as a testing and calibration laboratory with the scope of accreditation for testing Natural Gas (NG) and Liquefied Natural Gas (LNG). ISO 17025 is an international standard given to laboratories in conducting calibration and testing activities throughout the world. ISO 17025 is able to become a benchmark for laboratories in proving valid test and calibration results that can be widely trusted.

In clause 7.7 ISO 17025, Ensuring the Validity of Results, it is stated that the laboratory must have procedures to monitor the validity of the results. Data generated should be recorded in such a way that trends can be detected and, whenever possible, statistical techniques should be applied to review the results. One of the methods that mentioned in ISO 17025 to ensure the validity of the test results is by holding an interlaboratory meeting. In this case, PT Badak NGL joined the Inter Laboratory Meeting (ILM) in East Kalimantan. The ILM implementation was attended by all natural gas producers, distributors and consumers consisting of 10 laboratories.

ILM is held periodically every 3 months. In the practice of ILM, the series of cylinders used consists of 16 sample cylinders arranged in series. Where two sample cylinders, specifically cylinders no. 1 and 16 are used as verification cylinders, 4 sample cylinders are used as a reserve, and 10 cylinders are distributed to related laboratories. During the implementation of the 112th ILM in June 2022 and the 113th in September, several laboratories provided outlier sample feed gas test results. An outlier result is a condition where the test results of a laboratory are very different from other laboratories according to statistical calculations.

The results of the outlier feed gas sample testing can be caused by several factors, both from the feed gas sampling process, conditioning sample feed gas, and the sample testing process using Gas Chromatography. During the implementation of ILM, all sampling procedures were witnessed by all participants and confirmed according to the GPA 2166 standard concerning Obtaining Natural Gas Samples for Analysis by Gas Chromatography. Furthermore, the homogeneity of the sample cylinder series has also been tested from sample cylinder 1 and sample cylinder 16. Therefore, the cause of the outlier test results can be caused by conditioning the sample cylinder or the testing process with Gas Chromatography.

GPA 2166 has explained the process of conditioning cylinder sample feed gas prior to injection and testing using Gas Chromatography. The document explains that the sample needs to be heated to 11 C above the sample temperature to ensure all components are vaporized before flowing into the detector in Gas Chromatography. The reference document also explains that the flow sample flowing towards the column in Gas Chromatography is set at 3 bubbles per second. Furthermore, the document does not regulate the minimum sample pressure that is injected in Gas Chromatography.

Sample pressure is an important parameter that needs to be controlled in conducting tests using Gas Chromatography. The sample pressure affects the diffusion rate of each component in the column (Scott, Raymond P.W., 2020). As we know that Natural Gas has the most complex components (N_2, CO_2, and alkanes) so that the sample pressure is very influential in giving strength to each component to diffuse to the stationary phase in the Gas Chromatography column.

The complexity of the components possessed by the sample feed gas also affects the stability of the components in repeat testing. Each component gives different test results. These differences are influenced by relative molecular masses, boiling points, intermolecular attractions, and the presence of major and minor components in the sample (G. Gilbert, Seymour, 2021)

This study aims to analyze the stability of sample feed gas based on pressure as alternative strategy to determine the minimum sample pressure limit in feed gas testing and analyze the effect of intercomponent stability in sample feed gas.

This research was conducted at PT Badak NGL. The scope of this research includes the sample feed gas used in the test is limited to only the sample feed gas Train E and the sample cylinder capacity used is 300 cc according to the cylinder in the ILM implementation. The sampling process was carried out based on GPA 2166 with the purging fill and empty method. The sample conditioning process is also based on the document, in which the sample cylinder is heated to a temperature of 11°C above the sample temperature

within 2 hours. The flushing process was carried out for 5 minutes according to the vendor's recommendation and modification of the sample loop on the Gas Chromatography used. The stability value is based on the repeatability value according to GPA 2261.

2. Materials and Experiment Methods

This study will be carried out systematically by comparing the repeatability value of each component according to the limits given in GPA 2261. Initially, two cylinders were assembled in series using identical cylinders. The purpose of this cylinder assembly is to obtain two identical samples as a test material and compare the trends of the two cylinders to ensure the accuracy of the repeatability values.

Before the sample cylinder is assembled, the cylinder is ensured to be clean by washing it and flowing pure water to prevent liquid and impurities from entering the cylinder. The cylinder is also checked for leaks between connections using a liquid leak detector.

After the sample feed gas from Train E is obtained, the sample cylinder is conditioned first by heating the sample cylinder using a belt heater. Figure 1 ilustrate the set up between cylinder sample, belt heater, and Gas Chromatography Agilent 6890N. The sample cylinder is heated to a temperature of 11°C above the sample temperature for 2 hours. When the sample cylinder is heated, the connection between the outlet valve cylinder can be connected to the sample loop on Gas Chromatography.

After the conditioning process is complete, the sample cylinder outlet valve is opened and gas is flowed into the sample loop to ensure that there are no leaks at the connection between the sample cylinder and Gas Chromatography. This is enabled so that sample readings can be representative and no air enters. The flushing process is carried out by flowing the sample for 5 minutes with a constant flow. After that the sample can be tested and the pressure recorded when the flushing process is complete. The sample pressure is varied between 550 psig to 100 psig. Figure 2 ilustrate the overall methodology of experimental.

Figure 1. Experimental Set Up

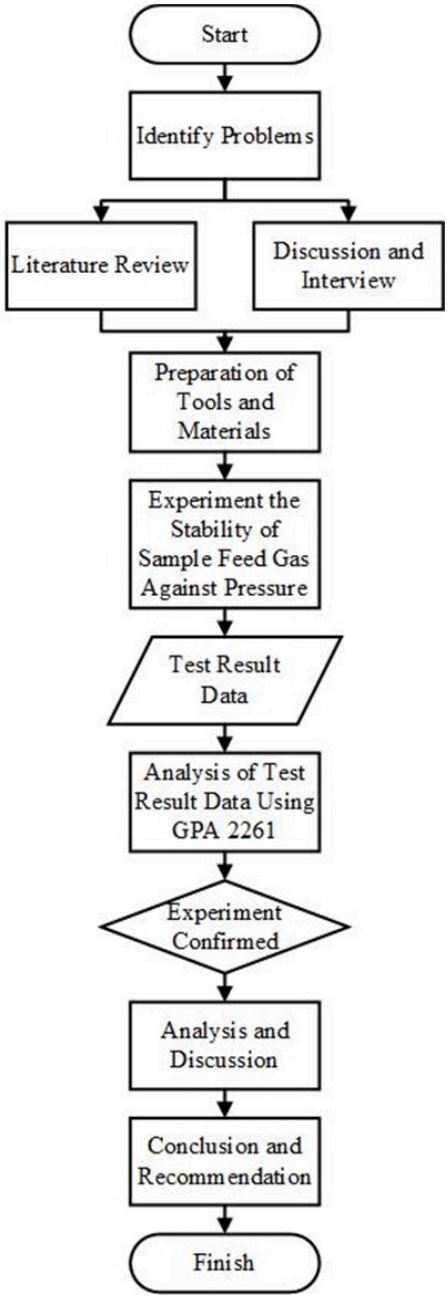

Figure 2. Research Flowchart

Statistical Verification

The verification method used in the test is repeatability in accordance with GPA 2261. There is a formula of statistics for testing repeatability values based on the % mol normalized component which is known from the test results using Gas Chromatography (Table 1). From the results of sample readings in the pressure range, the difference in the largest and highest values can be calculated as well as the average of all readings for each component which is called the x value. The x value can then be substituted into the repeatability formula in Table 1. If the difference value is smaller than the repeatability value, then the sample reading is considered stable and meets the limits, and vice versa. To find out the required minimum sample pressure of feed gas, the value is tested repeatedly from 550 psig to 500 psig, then from 550 psig to 450 psig, and so on until the overall pressure (550 psig to 100 psig).

Table 1. Data Analysis Acceptance Requirements based on GPA 2261

Component	*Range (% mol)*	*Repeatability*
Nitrogen	.02-15	$0.039x^{1/4}$
Methane	50-100	$0.0079x^{1/3}$
Carbon Dioxide	.02-15	$0.0042x^{1/3}$
Ethane	.02-15	$0.0124x^{1/3}$
Propane	.02-15	$0.0084x^{1/8}$
Isobutane	.02-8	$0.01x^{1/5}$
n-Butane	.02-8	$0.0117x^{2/5}$
Isopentane	.02-4	$0.009x^{1/4}$
n-Pentane	.02-4	$0.01x^{1/5}$
Hexane Plus	.02-2	$0.0135x^{1/4}$

3. Results and Discussion

Test Results Data Repeatability Cylinder Sample 1

The repeatability data for the feed gas sample in the first sample cylinder is as follows:

Table 2. Repeatability Sample Feed Gas Cylinder 1

Component (%mol)		N_2	C_1	CO_2	C_2	C_3	i-C_4	n-C_4	i-C_5	n-C_5	C_{6+}
	550	0,054	91,791	3,237	2,152	1,460	0,331	0,400	0,172	0,114	0,289
	500	0,055	91,781	3,240	2,153	1,463	0,332	0,401	0,172	0,112	0,291
	450	0,056	91,768	3,246	2,156	1,466	0,333	0,402	0,172	0,110	0,291
	400	0,055	91,759	3,254	2,151	1,470	0,334	0,402	0,173	0,110	0,293
Pressure	350	0,061	91,716	3,274	2,145	1,482	0,336	0,407	0,174	0,109	0,296
(psig)	300	0,055	91,702	3,282	2,149	1,486	0,337	0,407	0,175	0,109	0,298
	250	0,056	91,677	3,304	2,140	1,496	0,339	0,407	0,176	0,104	0,302
	200	0,066	91,653	3,291	2,173	1,491	0,338	0,409	0,175	0,106	0,299
	150	0,059	91,670	3,299	2,145	1,495	0,340	0,410	0,176	0,106	0,301
	100	0,060	91,640	3,317	2,140	1,504	0,342	0,412	0,177	0,106	0,302
Average normalized mole percent of the component		0,058	91,716	3,274	2,150	1,481	0,336	0,406	0,174	0,109	0,296
***Diff* 1 s/d 10**		0,012	0,151	0,080	0,033	0,044	0,011	0,012	0,005	0,010	0,013
Repeatability		0,019	0,036	0,006	0,016	0,009	0,008	0,008	0,006	0,006	0,010
Result		**PASS**	**NOT PASS**	**NOT PASS**	**NOT PASS**	**NOT PASS**	**NOT PASS**	**NOT PASS**	**PASS**	**NOT PASS**	**NOT PASS**

The formula used in these statistics is mentioned in Table 1. The benefits of the repeatability formula and calculation are used to determine whether in the reading pressure range, the repeatability value of each component still meets the acceptance limits. If there

are components that do not meet these acceptance limits, the pressure reading range can be narrowed and then the repeatability value can be recalculated.

Overall, the repeatability test results of the feed gas sample in the first cylinder show that the nitrogen (N_2) and isopentane (i-C_5) components meet the acceptable repeatability range. Other components in the feed gas do not meet the acceptable repeatability range in the pressure range of 550-100 psig.

Based on Table 2, at a minimum pressure of 500 psig, it shows that all components still meet the repeatability acceptance range in accordance with GPA 2261. Meanwhile at a pressure of 450 psig, the CO_2 component no longer meets the repeatability acceptance range. This continues until the minimum pressure or 100 psig. When the sample reading was carried out at a pressure of 400 psig, component C_3 no longer met the acceptable repeatability range. This is followed by component C_1 at a pressure of 350 psig, components n-C_5 and C_{6+} at a pressure of 250 psig, C_2 and n-C_4 at a pressure of 200 psig, and component i-C_4 at a pressure of 150 psig.

Test Results Data Repeatability Cylinder Sample 2

Repeatability testing on cylinder sample 2 was carried out by the same analyst, namely the author. Repeatability analysis on cylinder sample 2 was carried out with the aim of ensuring that the feed gas sample stability test obtained an accurate minimum pressure value. Testing cylinder sample 2 used identical sampling equipment, the same Gas Chromatography, sample pre-treatment and the same Gas Chromatography running process.

Table 3. Repeatability Sample Feed Gas Cylinder 2

Component (%mol)		N_2	C_1	CO_2	C_2	C_3	i-C_4	n-C_4	i-C_5	n-C_5	C_{6+}
Pressure (psig)	550	0,054	91,764	3,252	2,153	1,468	0,333	0,402	0,173	0,108	0,293
	500	0,055	91,764	3,248	2,156	1,467	0,332	0,402	0,172	0,113	0,291
	450	0,054	91,781	3,240	2,152	1,463	0,332	0,401	0,172	0,114	0,291
	400	0,070	91,730	3,270	2,130	1,482	0,336	0,405	0,174	0,112	0,291
	350	0,058	91,713	3,278	2,144	1,483	0,337	0,407	0,174	0,108	0,296
	300	0,057	91,696	3,286	2,148	1,489	0,338	0,408	0,175	0,107	0,296
	250	0,056	91,644	3,319	2,142	1,503	0,341	0,412	0,177	0,101	0,304
	200	0,061	91,668	3,298	2,143	1,495	0,337	0,416	0,175	0,108	0,299
	150	0,075	91,621	3,320	2,141	1,507	0,342	0,413	0,177	0,100	0,303
	100	0,062	91,621	3,325	2,143	1,509	0,342	0,413	0,177	0,103	0,304
Average normalized mole percent of the component		0,060	91,700	3,284	2,145	1,487	0,337	0,408	0,175	0,107	0,297
Diff 1 s/d 10		0,021	0,160	0,085	0,026	0,046	0,010	0,015	0,005	0,014	0,013
Repeatability		0,019	0,036	0,006	0,016	0,009	0,008	0,008	0,006	0,006	0,010
Result		**NOT PASS**	**NOT PASS**	**NOT PASS**	**NOT PASS**	**NOT PASS**	**NOT PASS**	**NOT PASS**	**PASS**	**NOT PASS**	**NOT PASS**

From the repeatability results of the feed gas cylinder 2 sample, it shows that in the pressure range of 550-100 psig, only the i-C_5 component meets the repeatability acceptance limit of GPA 2261. Based on the data in Table 3, it can be seen that at a pressure of 500 psig

all components in the feed gas meet the repeatability acceptance limit of GPA 2261. At a pressure of 450 psig, the CO_2 component no longer meets the repeatability acceptance limit. In the 400 psig range, components C_1, C_2, and C_3 no longer meet the repeatability acceptance limit. Meanwhile, in the 300 psig range, the n-C_5 component does not meet the repeatability limit, followed by the i-C_4, n-C_4, and C_{6+} components at a pressure of 250 psig, and the N_2 component at a pressure of 150 psig.

Correlation of Repeatability Values for Sample Feed Gas in Cylinder 1 and 2

Overall, the results of the repeatability values for cylinder samples 1 and 2 have the same sample stability values. Starting from a pressure of 450 psig, the two samples do not meet the repeatability acceptance value according to GPA 2261 for the CO_2 component. Then, gradually other components do not meet the repeatability value range.

Effect of Pressure on Repeatability Values

Gas chromatography is a chromatographic technique that uses the principle of separating mixtures based on differences in migration speeds of the constituent components (Maráková, K., Opetová, M., & Tomašovský, R., 2023). In gas chromatography, pressure is an important parameter that can influence the retention time and peak shape of the analyte. In gas applications, fluctuations in gas pressure can affect the velocity of particles in the gas. According to the kinetic theory of gases, pressure is influenced by static pulsations between molecules (Handayani, H., 2020). The pressure is caused by collisions between molecules moving at different speeds. In this case, when the pressure in the sample is measured to be large, this indicates that the collisions produced by the particles in it are also large.

Fick's law of diffusion (Flick's Law) describes the relationship between diffusion and other factors. Flick's Law in equation 5 states that the rate of movement of molecules through a material is proportional to the concentration gradient (concentration difference) between the two ends of the material and is inversely proportional to the thickness of the membrane (Won, Y. Y., & Ramkrishna, D., 2019). So according to this law, greater pressure (difference in pressure concentration) will have an increasing effect on the diffusion rate. This is because the greater the pressure difference, the greater the collision force between particles, so that the rate of particle movement will be greater and the rate of diffusion will be greater.

Based on repeatability data on cylinder samples 1 and 2, the lower the pressure, the more the sample composition reading is out of the repeatability acceptable range. The greater the pressure difference between the column cylinder sample and the Gas Chromatography column, the greater the rate of diffusion that occurs in the column. The greater the diffusion rate, the greater the particle movement speed.

Increasing the diffusion rate affects changes in the response of the Gas Chromatography detector. Increasing the diffusion rate will affect the distribution of particles in the detector where the particles will move faster so that the resulting retention time will be smaller. A substance with a faster diffusion rate will reach the detector more quickly in a given time than a substance with a slower diffusion rate.

Low particle movement speed as a result of a decrease in sample pressure can affect the reading time of components in the detector. The lower the pressure, the lower the particle speed. This results in the retention time read by Gas Chromatography being longer. Peak retention time is one of the components that influences quantitative and qualitative analysis in Gas Chromatography. When there is a change in retention time that exceeds the normal retention time, this results in errors in the reading and calculation of the sample area. As a result, the calculation of the read sample composition is not representative and has a repeatability value outside the permitted range.

Qualitative analysis is generally reflected in the chromatographic peak retention time, which requires peak height and peak area parameters. Generally, the width of the chromatographic peak will be influenced by variable retention times. The longer the retention time, indicates that the particles move more slowly and spread out in the column.

This will have the effect that the width of the chromatogram peaks formed will be wider. The relationship between retention time and baseline peak width of the chromatogram, namely when the retention time is short the peak width is small, has a high peak, and the peak area is relatively small. Meanwhile, the retention time is long, has a large chromatographic peak width, low peak height, and relatively large peak area.

The relationship between retention time and peak width of the resulting chromatogram influences the theoretical plate number (N). In accordance with the theoretical plate formula in equation 3, when a small pressure results in a larger retention time and a larger baseline peak chromatogram width, this will result in a smaller number of theoretical plates. On the other hand, when the pressure is large, it results in a faster retention time with a smaller baseline peak chromatogram width, resulting in a larger number of theoretical plates.

Gas Chromatography columns that have a large number of theoretical plates are more efficient at separating samples compared to columns with a low number of theoretical plates. The concept of plate count as a measure of efficiency is based on separation by distillation. The ability to separate by distillation is reflected in the number of plates, where each plate has a different equilibrium. The greater the number of plates, the better the separation potential.

In accordance with the theory that has been described, the effect of decreasing pressure has a related effect between one parameter and another in reading samples using Gas Chromatography. When the pressure is low (the pressure gradient is small), it can result in a smaller number of theoretical plates, thus indicating a worse component separation efficiency. As a result, the reading of the sample composition in chromatography will experience changes and cause the repeatability value to not meet the acceptance range according to GPA 2261.

Intercomponent Stability in Cylinder 1 and 2

Based on the results of repeatability calculations for cylinder samples 1 and 2, the results showed that the carbon dioxide (CO_2) component was the first component to experience reading instability at a pressure of 450 psig. Continuously, reading the sample gives results beyond the acceptable limit of repeatability up to the smallest pressure (100 psig). Meanwhile, other components gradually experience repeatability instability up to a pressure of 100 psig.

The influence of component stability on the composition identification process using Gas Chromatography is closely related to the diffusion process that occurs therein. Graham's law states that the rate of diffusion of a gas is inversely proportional to the root mass of its particles. In other words, at the same temperature and pressure, the speed of gas diffusion is inversely proportional to the root of its density. In the diffusion process, the smaller the particle size, the faster the particle will move, so the diffusion speed is higher.

Apart from that, the diffusion process is also influenced by intermolecular attractive forces. CO_2 compounds and alkane compounds are both nonpolar covalent compounds. As a result, both compounds have zero dipole moment. However, according to organic chemistry theory, if two nonpolar compounds are close together, an attractive force occurs between the negatively charged electrons of one molecule and the partial positive charge in the other molecule.

In the sample feed gas component, CO_2 is the first component to experience reading instability. This is influenced by the type of compound in each component. CO_2 is an inorganic compound, while the hydrocarbon alkane compound which is the major component is an organic compound. In accordance with basic theory, Gas Chromatography is generally used to separate volatile compounds. If the analysis target is not volatile, it will generally be derivatized (reacted) to become a volatile compound. Carbon dioxide (CO_2) is a non-volatile gas and is not considered a volatile organic compound. Therefore, even

though CO_2 has a low boiling point, namely -78.46°C, this compound is difficult to separate and evaporate by the Gas Chromatography component.

Apart from the volatile nature of the CO_2 compound, the influence of the chain shape of the CO_2 compound also has an influence on the rate of diffusion and the London force that occurs. The CO_2 compound has the same molecular mass as the C_3 compound. However, compound C_3 has a much longer and straighter compound structure. As a result, the attractive force that occurs in the C_3 compound is much greater than the attractive force in the CO_2 compound. This attractive force influences the rate of diffusion that occurs, the greater the attractive force, the slower the resulting diffusion rate. Therefore, the resulting retention time when the pulling force is large is much shorter, so that the deviation in the peak width of the chromatogram will be smaller and the reading will be much more stable.

Although in Graham's theory it is stated that components that have a lighter molecular weight have a faster diffusion rate, this is also influenced by the major factors of composition and shape of the compound chain. In the feed gas sample, the component that has the smallest molecular mass is the C_1 component. However, this component is a major component where 91% of the feed gas component is dominated by methane. As a result, the changing reading value does not have such a significant effect compared to other components that have a lighter molecular weight than C_1. In this case, components C_2 and C_3 are the components that are more unstable than component C_1. Small value changes in components C_2 and C_3 have very influential results on the repeatability range, because these components are minor components where the range for C_2 and C_3 is 1-5%. In addition, the n-pentane component also shows unstable values compared to the values for the n-butane and iso-butane components. This is because the n-pentane component is much smaller than the n-butane and iso-butane components in the overall feed gas component.

4. Conclusions

The repeatability calculation process in accordance with GPA 2261 has its own limits for each component, where the variable used in the calculation is the x value which represents the normalized mol % component. Based on the results of research conducted, the minimum pressure limit for testing feed gas is 450 psig. At this pressure, all components meet the repeatability acceptance limit, where the difference between the largest and smallest reading composition values is smaller than the calculated repeatability value. At this point, the difference value obtained in the composition of C_1 in cylinder 1 was 0.006 and the value of C_1 in cylinder 2 was 0.001, which was smaller than the calculated repeatability value of 0.019. therefore, the component meets the acceptance limits and so do the other components. Pressure has a significant influence on the repeatability results of component readings. Based on Ficks' Law, the greater the pressure gradient, the greater the influence on the acceleration of the rate of diffusion that occurs. As a result, the particles will move faster towards the detector and the peaks formed will be taller and slimmer. The peak shape and retention time influence the calculation of the number of theoretical plates used as an indicator of separation efficiency in the column. The slimmer and faster the retention time, the greater the number of theoretical plates. Therefore, the separation process that occurs is more effective so that the components have good repeatability values.

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