

ANAEROBIC DIGESTION STABILITY TEST BY SHEWHART CONTROL CHARTDoi: <http://dx.doi.org/10.1590/1809-4430-Eng.Agric.v37n3p618-626/2017>**MICHAEL S. ALCANTARA^{1*}, GEOVANE GRISOTTI², MARIA H. F. TAVARES²,
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ABSTRACT: The anaerobic digestion (AD) operation complexity raise the importance of stability testing to verify whether the operating conditions are under control and whether the process is working as required. The current method of verifying process stability is by a range of the ratio between volatile fatty acidity and total alkalinity (VFA/ TA) within which stable conditions are brought about; these rates vary with the content of organic material. A few indicators as pH or constant biogas production are also used. Therefore, the aim of this study was to propose the use of Shewhart control chart for individual measures as a stability test for AD, as well as to demonstrate its use in poultry litter AD. The method showed to be advantageous since it standardizes a variation range for the process according to the average and verifies the stability by direct measures, such as organic material removal efficiency and biogas production.

KEYWORDS: process monitoring, statistical process control, waste treatment.

INTRODUCTION

Currently, the methods that have been used to evaluate AD stability are indicators as pH, VFA/ TA ratio and constant biogas production.

The pH measure is considered less important than the VFA/ TA ratio for representing large changes in alkalinity, with small variations on a logarithmic scale. Experimental results have shown that, in the event of a drastic pH drop due to VFA accumulation, AD process rarely returns to normal operation (MÉNDEZ-ACOSTA et al., 2010). According to AYMERICH et al. (2013), understanding the dynamic behavior and diffusion pattern of high-solid systems is a key role for the identification of inhibitory levels for different compounds, e.g. VFA, besides detecting periods of imbalance.

Continuously sought attributes for system monitoring have to be low cost, low maintenance, easy to use, and produce fast and accurate responses (JOBILING PURSER et al., 2014). In this context, a statistical process control could be used as stability test for the AD process.

A process can be understood as a system transforming “inputs” (AD) into value-added products, being influenced by natural or random factors, and special or non-random factors. Slight and inevitable variations, which are difficult to trace, result from natural process factors. Special process factors result in traceable changes, whether planned or not, which are characterized by actual changes such as in materials, the environment, or the used method.

Among the most used tools of statistical process control are the control charts. They stand for graphical analysis and monitoring of a process regarding timewise by two characteristics: centralization - the process average and dispersion, estimated by its standard deviation or range of data (HO & QUININO, 2013). The objective of a control chart is to seek for signs of special causes affecting the process stability by visual identification of the behavior of critical quality variables (HACHICHA & GHORBEL, 2012; ABBAS et al., 2013).

According to MONTGOMERY (2009), for one-size samples, i.e. samples are made of

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individual units, a Shewhart control chart could be useful for the monitoring of process average, and using a moving range chart of two successive measurements to estimate the process variability. Equations 1, 2, 3, and 4 are used to calculate the control limits of the Shewhart control chart for individual measurements (MONTGOMERY, 2009):

$$UCL = \mu + k \sigma \quad (1)$$

$$CL = \mu \quad (2)$$

$$LCL = \mu - k \sigma \quad (3)$$

where,

μ : population mean;

k: a specified number of standard deviations;

σ : population standard deviation;

UCL: upper control limit;

CL: center line,

LCL: lower control limit.

According to MONTGOMERY (2009) and MINITAB 15 (2013), the standard to build the chart is by using \bar{X} (sample mean) to μ , $k = 3$, \overline{MR}/d_2 (w) to σ , d_2 as a factor for center line and $w = 2$ to observations number used in the calculation the moving average range (\overline{MR}). However, it is also possible to use control limits narrower than three-sigma ones (3σ) in a Shewhart control chart for individual measurements, enhancing the ability to detect small process shifts.

$$\overline{MR} = \frac{\sum_{i=2}^n |X_i - X_{i-1}|}{n} \quad (4)$$

where,

\overline{MR} : moving average range;

i: 2, 3, ..., n observations,

X: variable under analysis.

When the process shows no special causes of variation of quality characteristics, the process is considered stable. Therefore, the aim of this study was to demonstrate the use of the Shewhart control chart for individual measures in assessing the stability of poultry litter AD.

MATERIAL AND METHODS

Experimental digester

The experiment was conducted at the Laboratory of Biological Reactors and the analyses were performed at the Laboratory of Environmental Sanitation, both in the State University of Western Paraná (UNIOESTE), in Cascavel – PR, Brazil.

Figure 1 show the digester, built in polyvinyl chloride, with the dimensions of 0.60 x 0.15 m (length x diameter), operating horizontally with a 5.75 L working volume. Temperature was maintained at 27 °C (± 2 °C) by an air conditioning system.

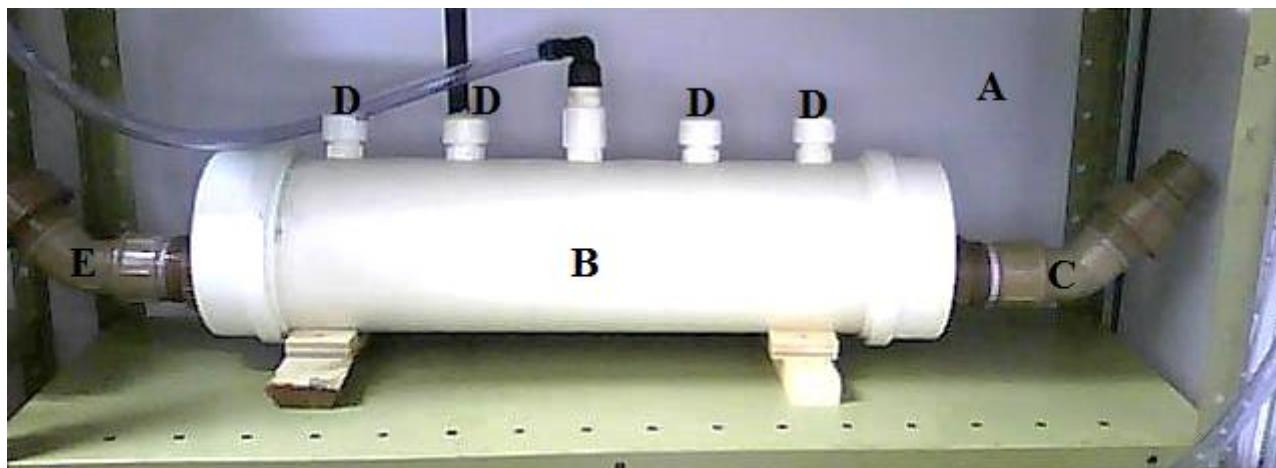


FIGURE 1. (A) Air-conditioned environment, (B) Digester, (C) Effluent input, (D) Sampling points and biogas output, (E) Effluent output.

AD was considered stable during the statistical process control by Shewhart control chart for individual measurements, with 2.5 standard deviations from the average, using MINITAB 15 software. Organic load consisted of 3 kg total volatile solids per m^3 of digester working volume per day ($\text{kg TVS m}^{-3} \text{ day}^{-1}$) and 10-day hydraulic retention time (HRT) in a semi-continuous reactor system, being fed once a day. The AD was evaluated by the organic material removal efficiency, which was measured by the amount of total volatile solids consumed (TVS_{cons} , %) and by the biogas production, being expressed by the ratio between these variables ($\text{m}^3_{\text{biogas}} \text{ kg}^{-1} \text{ TVS}_{\text{cons}}^{-1}$).

Biogas volume was daily quantified by a gasometer; and the measurements were corrected for the Standard Temperature and Pressure (STP), 273.15 K and 101,325 Pa. Also, the VFA/ TA ratio of the effluent was determined weekly by the simple comparison with the results of Shewhart control chart on biogas production and on organic material removal efficiency in testing the stability of poultry litter AD.

The physicochemical parameters were analyzed by procedures described by APHA (2012) for TS and TVS (2540B method), and pH (4500B method). Yet the parameters volatile fatty acidity and total/ partial alkalinity were evaluated by methods described by JENKINS et al., (1983) and RIPLEY et. al. (1986).

Preliminary Statistical Analysis

An overview of data behavior can be obtained through preliminary analysis, calculating measures of central tendency, dispersion, asymmetry, and kurtosis.

The samples should be representative of the process; therefore, data independence and normality have to be checked. These are premises for the use of the Shewhart control chart. If these characteristics are unfulfilled, the results can be misleading due to possible false alarms (MONTGOMERY, 2009). Another premise for the use of this test is that data must be identically distributed.

Normality Test

Prior to the Shewhart control chart, data normality should be tested using methods such as Anderson-Darling, Ryan-Joiner, Shapiro-Wilk, and Kolmogorov-Smirnov. If the normal distribution is rejected, the data must be transformed for normalization, making use of transformation tools such as Box-Cox. Thereafter, transformed data should be tested again to verify normality.

Autocorrelation

Another principle for Shewhart control chart use is the lack of data autocorrelation. In this case, autocorrelation may affect the performance of control charts by confusing process special

causes with common ones. Autocorrelation is the correlation coefficient between observations lagged in time; it can be easily observed by an autocorrelation chart since when the dashed lines (limits of two standard deviations) are exceeded, autocorrelation exists (MONTGOMERY, 2009).

RESULTS AND DISCUSSION

The Shewhart control chart for individual measurements was chosen to assess the AD stability because it is applicable for cases with low availability of measurements taken from the same process, and with small differences between samples at short time intervals, such as the process concerned.

The preliminary analysis was performed using descriptive statistics as shown in Table 1. Greater incidences of high-efficiency organic material removal and low biogas production were found in the analysis, being characterized by negative and positive skewness, respectively. Both variables were classified as leptokurtic since kurtosis values were lower than 0.263, i.e. a degree of flatness (sharpness) of the distribution lower than the reference degree of the Gauss curve. The organic material removal efficiency was uniform with a variation coefficient lower than 20%. On the contrary, the biogas production was non-uniform with a variation coefficient of 43.32%, being explained by the peaks of biogas production (positive skewness). The average standard deviation indicates the precision for the average estimates of both organic material removal efficiency and biogas production, being of 3.7 and 8.9%, respectively, and the sample standard deviation represents the range of sample variation.

TABLE 1. Descriptive statistics of the values of organic material removal efficiency (TVS_{cons}, %) and biogas production (m³_{biogas} kg⁻¹ TVS_{cons}⁻¹).

Variables	TVS _{cons} (%)	m ³ _{biogas} kg ⁻¹ TVS _{cons} ⁻¹
Average Standard Deviation	2.5	0.0245
Sample Standard Deviation	12.3	0.1198
Coefficient of Variation	18.2	43.32
Minimum	42.1	0.1199
Quartile 1	55.7	0.1877
Average	67.6	0.2766
Median	71.2	0.2653
Quartile 3	76.3	0.3437
Maximum	85.8	0.5369
Skewness	-0.50	0.72
Kurtosis	-0.86	-0.42

By the distribution analysis of quartile 1 and quartile 3 compared to the average, we may infer a normal distribution for both organic material removal efficiency and biogas production. However, according to MONTGOMERY (2009), even if there is a supposed normality, it must be checked when using the control chart. Thenceforth, normality was assessed by normality tests, being confirmed by p-values, which should be greater than the significance level (α) for normal data, as in Table 2. When one of the tests shows normality, the data distribution is already considered normal.

TABLE 2. Normality tests for organic material removal efficiency (TVS_{cons}, %) and biogas production (m³_{biogas} kg⁻¹ TVS_{cons}⁻¹).

Normality Test ($\alpha = 5\%$)	TVS _{cons} (%)	m ³ _{biogas} kg ⁻¹ TVS _{cons} ⁻¹
Anderson-Darling	0.097	0.083
Ryan-Joiner	>0.100	>0.100
Kolmogorov-Smirnov	>0.150	0.095

Data were normal according to the Anderson-Darling's, Ryan-Joiner's, and Kolmogorov-Smirnov's tests at 5% significance. MONTGOMERY (2009) stated that for more than 30 data per population, worries about sample distribution are discarded. As assured in the Central Limit Theorem (CLT), with an around 30 data, sample means tend to have a normal distribution. MONTGOMERY (2009) said that, in many times, the CLT in itself is a justification of the assumed normality.

However, MARTÍNEZ-ABRAÍN (2014) stated that the effect of a large-sized sample on distribution pattern depends both on sample size and on variability between populations. Therefore, researchers must explore beforehand the variance profiles of populations to assure they are within a safety zone (golden rule, $n = 30$), or whether sample size should be increased. There is no exact number of data to test for normality, the assessed values just have to show a normal distribution; however, using values equal to or greater than 30 help achieve to normality.

By contrast, MONTGOMERY (2009) also stated that even when normality assumption is violated until a moderate level, this control chart may still work fairly well. Nevertheless, if the independence of observations is unsupported, the results will be misleading, as many false alarms will occur by the positive correlation of the data. There was autocorrelation between the observed values by the sample autocorrelation function showed in Figures 2 (A) and 2 (B).

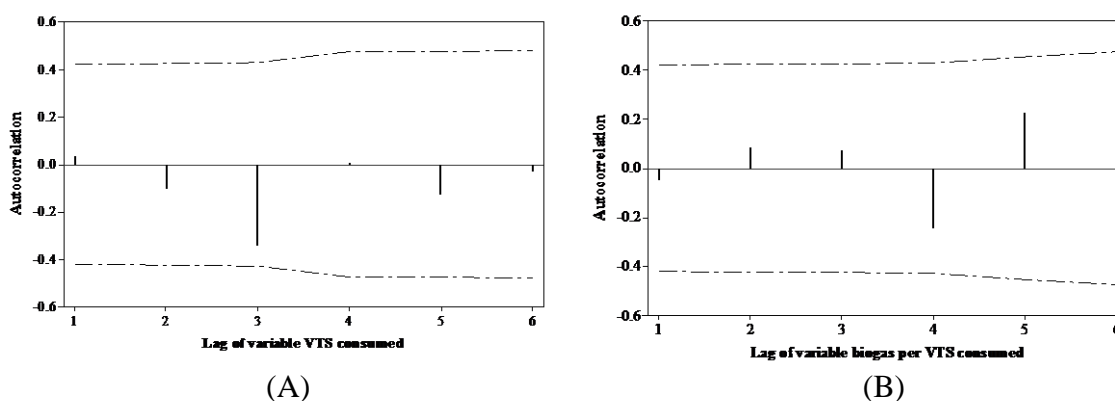


FIGURE 2. Graph of the sample autocorrelation function for the variables: (A) organic material removal efficiency and (B) biogas production.

Since there were no correlation coefficients outside the limit of two standard deviations, the values of organic material removal efficiency and biogas production are independent. If there was autocorrelation between the observations, correlation structure should be removed from the time series that best identifies the process, such as ARIMA and SARIMA, being the analysis of the control chart through residuals.

The Shewhart control chart for individual measurements can be used for evaluation of AD stability since the observations satisfy the premises. The analyses of the Shewhart control chart and of the moving average range chart for the control of process stability depends on the desired accuracy for one or more criteria. The criteria 1, 2, 3 and 4 are suggested by the WESTERN ELECTRIC COMPANY (1956) and the others by MONTGOMERY (2009):

1. One or more points outside of the control limit (3σ);
2. Two out of three consecutive points outside the two-sigma (2σ) warning limits but still within the control limits;
3. Four out of five consecutive points beyond the one-sigma limits (1σ);
4. A run of eight consecutive points on one side of the center line;
5. Six points in a row steadily increasing or decreasing;

6. Fifteen points in a row between one-sigma (1σ) and the center line, both above and below the center line;
7. Fourteen points in a row alternating up and down;
8. Eight points in a row on both sides of the center line with none between one-sigma (1σ) and the center line;
9. An unusual or nonrandom pattern in the data;
10. One or more points near a control limit.

In the analysis of AD stability through Shewhart control charts, presented in Figures 3 and 4, all the criteria and the control limits were used as 2.5σ from the average to obtain increased strictness in stability; however, only the criteria 1, 4, and 5 are frequently used in the statistical process control.

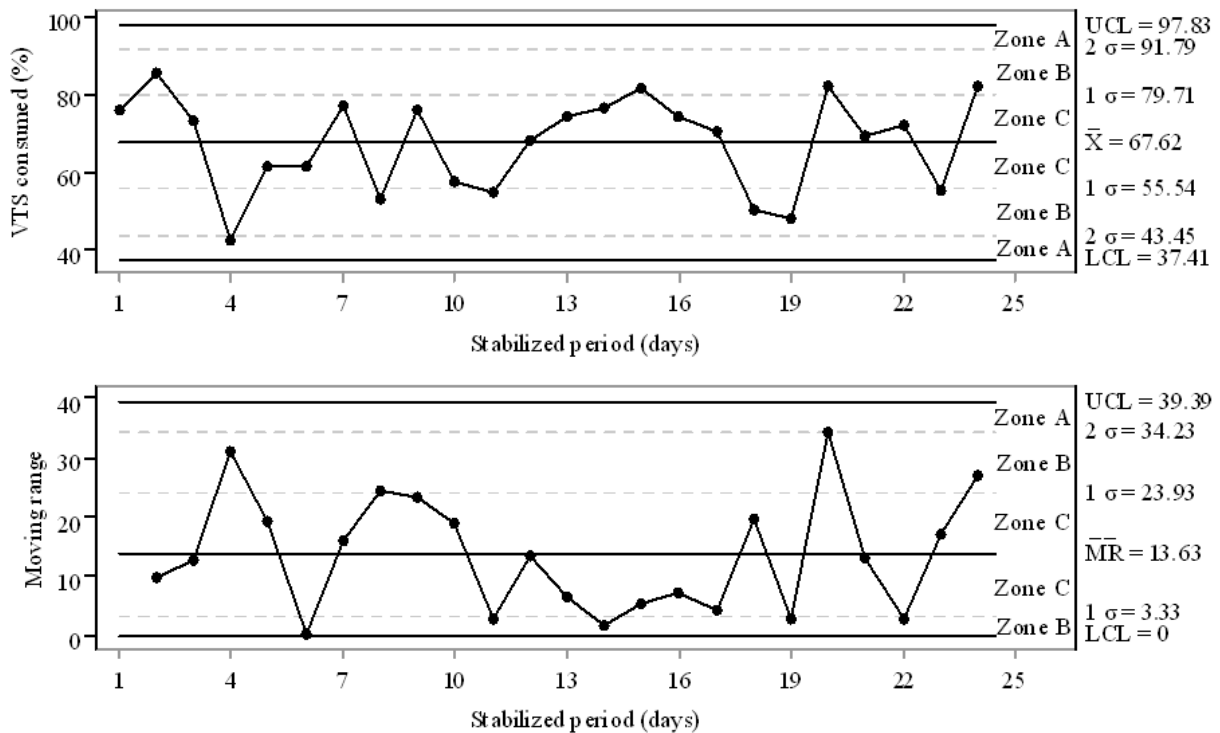


FIGURE 3. Shewhart control chart for individual measurements and moving average range chart as stability test for anaerobic digestion through organic material removal efficiency (TVS_{cons} , %).

The analyzed variables were stable by the Shewhart control chart for individual measurements during the evaluated period since all criteria were satisfied. By analyzing the moving average range chart, the process does not meet criterion 10. However, this chart is applicable only to assist in the process control and decision-making with the Shewhart control chart. According to MONTGOMERY (2009), the moving ranges are correlated and this correlation may often exhibit a pattern of runs or cycles. In the light of this, some care should be used when interpreting these patterns on the moving range chart. Thus, even though the process was stable throughout the evaluated period, by the Shewhart control chart for individual measurements, according to the moving average range chart, greater monitoring is required in the AD process to avoid its destabilization.

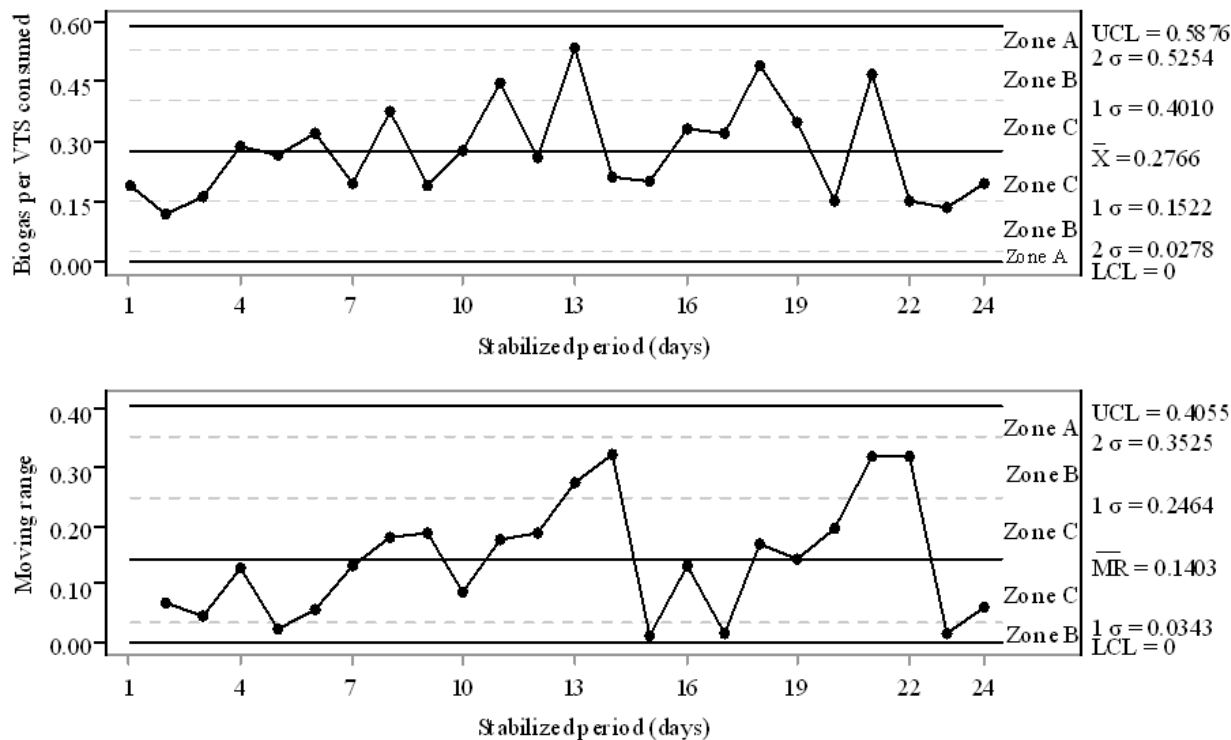


FIGURE 4. Shewhart control chart for individual measurements and moving average range chart as stability test for anaerobic digestion through biogas production ($\text{m}^3_{\text{biogas}} \text{kg}^{-1} \text{TVS}_{\text{cons}}^{-1}$).

In this study, the experimental values of biogas production were similar to other studies, showing the test applicability. GANGAGNI RAO et al. (2013) evaluated poultry litter AD using effluent reuse in a self-mixed anaerobic digester under high organic loading rate (4 kg TVS/ m^3 /day and 24 HRT days) and obtained $0.23 \text{ m}^3 / (\text{kg TVS}_{\text{added}})$ of biogas production. COSTA et al. (2012) studied poultry litter AD with 1% total solids in a batch system and achieved a methane (CH_4) production of $0.145 \text{ m}^3 / (\text{kg TVS}_{\text{added}})$.

While AD was stabilized according to the Shewhart control chart, VFA/ TA ratio of the effluent output showed fluctuations (Table 3), hindering an accurate determination of the period of stability of the AD process by this variable. Furthermore, the effluent pH values remained near 7.0 during the period, which also impaired evaluation or monitoring of the AD by such variable, as there were no differences.

TABLE 3. VFA / TA ratio of anaerobic digestion under stabilized period.

Stabilized Period (days)	9	12	18	23
VFA/ TA	1.43	0.26	0.40	0.40
pH	7.10	7.23	7.09	7.10

If compared to the VFA/ TA ratio, the Shewhart control chart is more advantageous for assessing AD stability. This is because it can be used throughout the waste treatment process, with the variable of interest reaching the assumptions of the test without needing a previous study of VFA/ TA stability bands. Therefore and according to MONTGOMERY (2009), this technique can also be applied to other processes. AD startup or operation with disturbs and toxic compounds can be tested by the Shewhart control chart as long as the analysis variable fulfills the test premises. This highlights the importance of the Shewhart control chart to evaluate the AD stability.

CONCLUSIONS

The Shewhart control chart for individual measures is applicable to testing the AD stability for any period in which the analysis variable fulfills the test premises. This test has the following advantages:

- 1) It evaluates the stability of process directly by the interest variables: organic material removal efficiency and biogas production;
- 2) It standardizes a variation limit for the process, according to the interest variables' average.

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