

# Benzene Concentration in the Phases of Tropical Soils

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**Abstract.** This work evaluates tropical soil adsorption capacity of a hydrophobic compound (benzene). With such purpose, a lateritic soil poor in organic matter and a hydromorphic soil rich in kaolinite with a higher organic content were studied. The lateritic soil, rich in Al and Fe oxides, presented a higher sorption capacity in grain size terms for having a higher clay fraction which consequently favored a greater surface contact area, and in mineralogical terms for containing micro-aggregates of Al and Fe oxides, which may confine hydrophobic compounds. This study also compared the lateritic soil retardation factor with Batch Test sorption data. It is shown that the retardation factor for benzene may overestimate the concentration of the adsorbed phase, and thus underestimates the concentration in the effective dissolved phase. Also, a simplified model is presented to calculate benzene concentration in the various phases (free, dissolved and adsorbed) and in the pore-fluid of a lateritic soil in a saturated environment.

**Key words:** adsorption, benzene, retardation factor, model.

## 1. Introduction

Benzene is an important aromatic hydrocarbon present in various industrial petrochemical products, being petroleum and coal its main sources. In spite of its industrial importance, benzene is a compound which is highly toxic to human health, either through inhalation, contact with the skin or by ingestion, causing damages to the central nervous system and the possibility of cancer (Malansky & Malansky, 1997). Cairney *et al.* (2002) reported that human occupational activity exposed to volatile hydrocarbons in gasoline via excessive inhalation may produce cognitive or neurological effects.

Since benzene is one of the components of fuels, namely gasoline and diesel, in the event of accidents at gasoline stations, or during the transportation of these materials, serious environmental damage may be caused. This hydrocarbon is considered a volatile compound which is highly soluble in comparison with others compounds, such as benzene, toluene, ethylbenzene and xylenes (BTX), and may easily infiltrate the soil and reach water tables, seriously affecting the water quality (Ulrich, 1999). According to Corseuil & Fernandes (1999) the alcohol added to Brazilian gasoline favors co-solvency, increasing the solubility of BTX hydrocarbons.

The flow of volatile hydrocarbons contaminants such as benzene, through the preferential flow channels and pores in the soil is partitioned in free, water, volatile and adsorbed phases. This partition in adsorbed phases occurs

through the adsorption of minerals and organic matter that comprise the soil. Each of these processes may occur in a higher or lower degree, depending on the physical and chemical characteristics of the soil, as well as a function of the contaminant volume and of the soil matrix geometry (Zytner, 2002, Farias, 2003, Fetter, 1993).

The flow mechanism, which generally orients chemical partition processes (dissolution and adsorption), is a slow diffusive flow, *i.e.*, less than or equal to  $10^{-9}$  m.s<sup>-1</sup> (Rowe *et al.*, 1995, Donahue *et al.*, 1999).

The clay minerals and organic material in the soil (adsorbent) may adsorb hydrocarbons (adsorbate) weakly or strongly, depending on the intensity of interaction between adsorbent/adsorbate. Strong interaction indicates chemical adsorption or chemisorption, which are covalent bonds or electrostatic bonds between the molecule and the surface. This process may require high activation energy, which may be relatively slow and not very reversible. However, in physical adsorption the interaction is weak (as occurs with aromatic hydrocarbons such as benzene). The bonds are of low energy activation and the process is easily reverted. Sorption may occur at the liquid/solid or vapor/solid interfaces. The latter form of sorption also occurs with benzene, due to its high volatility (Shaw, 1975; McBride, 1994).

The *Batch Test* experiment evaluates absorption by estimating the partition coefficients between equilibrium solution, which is called adsorption coefficient or adsorption constant. The adsorption coefficient may be deter-

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mined by the Freundlich isotherm, given by the following equation:

$$\frac{X}{M} = K_f C_e^{1/\omega} \quad (1)$$

where  $X$  = mass of the compound adsorbed by the soil ( $\mu\text{g}$ );  $M$  = mass of the adsorbent (g);  $K_f$  = equilibrium constant which indicates adsorption capacity ( $[\mu\text{g.g}]/[\text{L.mg}]^{1/\omega}$ );  $C_e$  = concentration of the equilibrium solution after adsorption (mg/L) and  $\omega$  = constant indicating the intensity of sorption.

High  $1/\omega$  values indicate a greater affinity between adsorbate and adsorbent. Thus, when  $\omega$  is equal to 1, the equation of the isotherm describes a linear partition or partition between the two phases, called  $K_d$ . However, when  $1/\omega$  is different than 1,  $K_d$  becomes specific for the concentration in which is determined, and thus  $K_f$  becomes more adequate to describe the sorption.

The contribution of organic matter to sorption in the soil may be evaluated through the standardized adsorption coefficient for organic carbon content ( $K_{oc}$ ). This may be calculated by the following relation for  $K_d$  or  $K_f$ :

$$K_f = K_{oc} f_{oc} \quad (2)$$

where  $f_{oc}$  ( $\text{g.kg}^{-1}$ ) is the mass of the fraction of organic carbon. The organic material (OM) mass percentage values may be converted to organic carbon content (OC), through the following expression:

$$K_{oc} = \frac{K_f \times 100}{MO\%} \times 1.724 \quad (3)$$

The conversion factor of 1.724 is produced by the average percentages of organic carbon of hummus composition, when the percentage of OC in the humus is 58%. However, some studies use a factor of 1.923, which corresponds to 52% of organic carbon (Kiehl, 1979).

Humus, which is developed organic matter, has a high molecular weight polydispersed in the soil matrix. Its composition rich in polymeric and aromatic compounds influences the wide superficial area of humus propitiating the adsorption of fulvic and humic acids. These compounds may also favor the adsorption of hydrophobic compounds (Chiou *et al.*, 1983; Karickhoff *et al.*, 1979; Murphy *et al.*, 1994). The specific surface of the minerals and of the organic material may influence the adsorption process due to its area of contact. The specific surface area of the organic matter, the oxides of iron, gibbsite and kaolinite have the following respective values: 700, 400, 100 and  $10 \text{ m}^2.\text{g}^{-1}$  (Kiehl, 1979; Sposito, 1984).

Studies of the adsorption of hydrophobic compounds in soil profiles, conducted by Njoroge *et al.* (1998), presented decreasing results for  $K_d$  at depths, as a function of the nature and quantity modification in the organic matter

of the profile. In deeper horizons hydrophilic organic compounds (fulvic and humic acids) decrease adsorption.

The retardation factor is one of the internationally used parameters in numeric models to determine the retention of contaminants in soil, being determined by the following equation (Rowe *et al.*, 1995):

$$R = 1 + \frac{\rho K_d}{n} \quad (4)$$

where  $R$  is the retardation factor;  $\rho$  is the apparent dry density;  $K_d$  is the sorption coefficient;  $n$  is the total porosity.

This study aims to present a contribution towards understanding the adsorption process of hydrophobic compounds in tropical soils, using the Freundlich isotherm to determine the mass of soil contaminated by dissolved organic compounds. This provides data for the estimates of the adsorbed phase concentration which may be used in decision-making processes aimed at the remediation of soil contaminated by benzene. Furthermore, a simplified model is presented to estimate the partition of hydrophobic compounds in a saturated medium evaluated by a quantitative comparison and retardation factors.

## 2. Materials

Two samples of soil typical of the *Cerrado* (savanna) region in Brazil were selected. The first sample is a lateritic soil, of reddish color, collected from B horizon at a depth of 4 m. Its texture is silty clay, with a large quantity of granular structure and small pores. The other sample is a hydro-morphic soil from A horizon collected at a depth between 0.5 and 10 cm. This superficial horizon is rich in organic matter, characterized by a black color, due to a silty clay texture and a small quantity of aggregates with few roots. The two soils are visually homogeneous and isotropic. Table 1 presents geotechnical properties of the materials studied. The degree of flocculation is obtained by percentage difference between ultrasound and water-dispersible clay fractions. The degree of dispersion is obtained by percentage water-dispersible clay fraction.

## 3. Methods

The *Batch Test* was initiated with the preparation of a stock solution of  $250,000 \text{ mg.L}^{-1}$  of benzene diluted in methanol. Two grams of air-dried (2 mm) soil was weighed and placed in 5 amber glass flasks. Diluted benzene from the stock solution was added to each flask, all reaching a final volume of 10 mL. This test was conducted in triplicate. All the flasks were adequately sealed to retain benzene. Then, the samples were submitted to agitation with a shaker at a constant temperature of  $23 \text{ }^\circ\text{C}$  for 4 h (Brusseau *et al.*, 1991; Saison *et al.*, 2004). This shake for 4 h instead 24 h was conducted to reduce microbiological benzene degradation.

After the agitation process, the samples were stored in refrigerators for 15 min for decantation. Five mL were re-

**Table 1** - Soil Characterization (Farias, 2003).

Test	Lateritic	Gley A
Atterberg Limit		
Liquid limit- $W_L$ (%)	41	46
Plastic limit- $W_p$ (%)	29	35
Plastic Index- $I_p$ (%)	12	12
Granulometry*		
Clay (%)	70	55
Silt (%)	25	30
Sand (%)	5	15
Degree of flocculation (%)	87.1	85.5
Degree of dispersion (%)	12.9	14.5
Chemical		
pH	5.70	5.20
Organic material (%)	0.41	5.44
Exchangeable cations ( $\text{cmol}_c.\text{cm}^{-3}$ )	6.4	159.8
Mineralogy		
Quartz (%)	30.2	42.2
Anatase (%)	1.57	0.93
Kaolinite (%)	24.6	41.4
Gibbsite (%)	25.5	4.6
Goethite(%)	4.6	2.2
Hematite (%)	7.5	0.0
Illite (%)	2.2	3.1
Vermiculite (%)	3.7	5.7
Hydraulic conductivity ( $\text{cm}.\text{s}^{-1}$ )	$3.7 \times 10^{-7}$	$3.8 \times 10^{-8}$
$n$ (%)	55.9	65.0

\*Grain size data obtained by ultra-sound waves using a laser beam grain size analyser.

moved for centrifugation, which was conducted at the Embrapa-Cerrados laboratory, in Brazil, with a refrigerated centrifuge at a temperature adjusted to 0 °C at 2000 rpm for 40 min. The suspension was collected for extraction of benzene in the aqueous phase equilibrium by a modification of the *USEPA* 1996 methodology, as described by Donahue *et al.* (1999). The benzene was extracted with the addition of 1 mL of the suspension of the posterior phase to a 5 mL bottle. Then, 3.3 mL of dichloromethane was added to extract the benzene. An internal standard of 0.2 mL of *p*-fluorotoluene at a concentration of 350  $\text{mg}.\text{L}^{-1}$  was determined. The material was then agitated for a few seconds for the complete extraction of the organic phase. A 2  $\mu\text{L}$  portion was then removed for the quantitative analysis of the benzene in the solution, with an analytical curve as an internal standard, in a gas chromatograph equipped with a Varian Star 3400  $\text{C}_x$  series flame ionization detector (CG/FID).

Cleaning of the columns after the passage of each sample was done with the injection of 1  $\mu\text{L}$  of dichloromethane.

### 3.1. The gaseous chromatography technique

A volume of 2  $\mu\text{L}$  of extract of dichloromethane was injected in the “splitless” mode in a phase DB-5 (30 m x 0.25 mm x 0.25  $\mu\text{m}$ ) cast silicon capillary column under the following chromatographic conditions: the temperature of the injector was 220 °C; the temperature of the detector was 300 °C; the initial temperature of the method was 38 °C, at a heating rate of 5 °C/min, reaching a final temperature at 100 °C (5 min). The carrier gas used was  $\text{N}_2$  5 mL/min and the  $\text{H}_2$  pressure at the head of the column was 68.95 kPa. The quantification was performed using a benzene standard with 98% purity. The internal standard was added to the analytical curve in order to reduce injection and/or volume errors (Leite, 1998).

## 4. Results and Discussion

The benzene partition process to the solid phase, conducted through the *Batch Tests*, presented significant adsorption, as can be verified in Table 2. According to this data, the average percent adsorption values obtained for the lateritic and gley A soil were, respectively, 65.73% and 48.82%. The *Batch Test* assay also produced the standard deviations for the initial and equilibrium concentrations which are associated to the adsorption due to the inherent losses in this type of experiment and also because the adsorbent is highly volatile. Nevertheless, the results were satisfactory.

The adsorption isotherms may be verified in Figs. 1 and 2. The respective coefficient correlation values for the lateritic and gley A soils were respectively 0.8895 and 0.8731, which may be reported to the errors mentioned previously. The linear coefficient is greater for the lateritic soil, indicating greater initial adsorption.

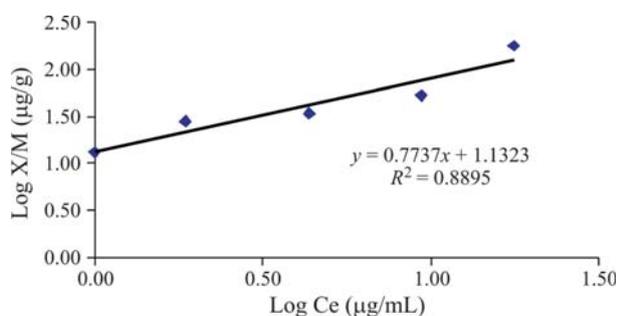
Table 3 shows the data generated by the adsorption isotherms. The lateritic soil presented the highest adsorption coefficient, equal to 13.56  $\text{mL}.\text{g}^{-1}$ , and consequently the highest degree of interaction between adsorbent and adsorbate ( $1/\omega$ ), equal to 0.774. However, based on the adsorption values observed in the literature, it may be stated that the adsorption values found are high for benzene in both soils. With regard to  $K_{oc}$ , a higher value for the lateritic soil was noted, mainly due to the low organic material content in relation to the gley A soil. This consistency regarding  $K_{oc}$  data in comparison with the literature may indicate that organic carbon does not directly control the sorption process of the soils studied.

The higher sorption ( $K_f$ ) for the lateritic soil in comparison with the Gley A may be related to the physical and chemical characteristics of each soil. This may indicate that the grain size distribution of the former are comprised of 70% clay-sized particles, favoring a greater adsorbate/ad-

**Table 2** - Basic *Batch Test* data.

Sample	$C_o$ ( $\mu\text{g/mL}$ ) <sup>1</sup>	$C_e$ ( $\mu\text{g/mL}$ )	$X$ ( $\mu\text{g/mL}$ )	ads (%) <sup>2</sup>	$X/M$ ( $\mu\text{g/g}$ )
Lateritic	$3.65 \pm 0.49^3$	$1.00 \pm 0.3$	2.65	72.60	13.25
	$7.59 \pm 0.31$	$1.85 \pm 0.2$	5.74	75.62	28.71
	$11.17 \pm 0.19$	$4.36 \pm 7.1$	6.81	61.00	34.06
	$19.80 \pm 0.30$	$9.41 \pm 1.56$	10.36	52.47	51.94
	$53.41 \pm 5.06$	$17.64 \pm 1.03$	35.77	66.97	178.83
Gley A	$3.65 \pm 0.49^3$	$1.20 \pm 0.13$	2.45	67.23	12.27
	$7.59 \pm 0.31$	$3.66 \pm 0.67$	3.93	51.80	19.67
	$11.17 \pm 0.19$	$7.10 \pm 0.28$	4.06	36.39	20.32
	$19.80 \pm 0.30$	$9.30 \pm 1.19$	10.50	53.04	52.50
	$53.41 \pm 5.06$	$34.37 \pm 0.80$	19.04	35.65	95.20

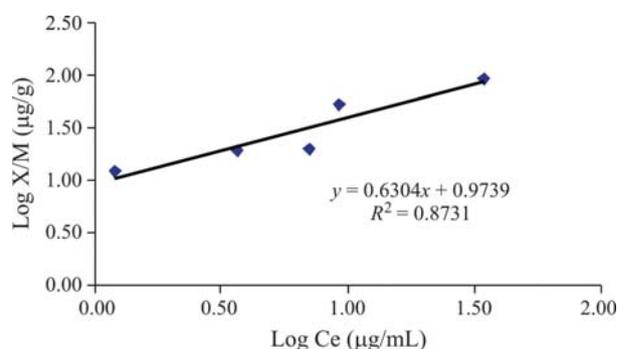
<sup>1</sup>Initial concentration equal for both samples. <sup>2</sup>Percentage of absorbed benzene. <sup>3</sup>Standard deviation.

**Figure 1** - Freundlich isotherm for the Lateritic soil.

sorbent interaction, due to the greater specific surface, which helps creating a larger contact area.

In mineralogical terms, the clay minerals of the lateritic soil contain a larger quantity of gibbsite and iron oxides. The Gley A soil contains a greater quantity of quartz, inert mineral, having few sites for the complex formation of minerals, organic matter, and hydrophobic compounds, which hampers the interaction between adsorbent and adsorbate. It also contains a larger quantity of kaolinite having a lower specific contact surface.

The type of organic material found in the lateritic soil (at a depth of 4 m), albeit low in quantity, may be largely comprised of fulvic and humic acids, which is different than the organic material found at the surface (humus), typ-

**Figure 2** - Freundlich isotherm for the gley A soil.

ical of the Gley A soil. This difference in organic material may influence the type of interaction between the organic mineral complex and the hydrophobic compound (Njoroge *et al.*, 1998).

Another factor to be considered regards the micro-aggregates and related soil micropores formed by the oxyhydroxides of Fe and Al. These may favor the confinement of non-adsorbed hydrophobic hydrocarbons in micropores. Thus, desorption data is superestimated because it included benzene in micropores.

The Gley A soil's exchangeable cations, although in greater quantities, given the higher organic matter content, did not prove to be an ideal parameter to verify adsorption

**Table 3** - Freundlich isotherm data for the lateritic and Gley A soils in comparison with the literature.

Sample	$K_f$ (mL/g)	$1/\omega$	$K_{co}$ (mL/g)	$R^2$	$K_f$ (mL/g) literature*	$K_{co}$ (mL/g) literature*
Lateritic	13.56	0.774	570.2	0.890	2.3-13.8 <sup>a</sup>	83-2300 <sup>a</sup>
					0.20-8 <sup>b</sup>	-
Gley A	9.41	0.630	29.8	0.873	0.17-13 <sup>c</sup>	26-59 <sup>c</sup>

\*Data of various soils. <sup>a</sup>Rowe *et al.* (1995). <sup>b</sup>Donahue *et al.* (1999). <sup>c</sup>Zytner (2002).

capacity. Figure 3 presents the benzene adsorption percentages, indicating the greater propensity of the lateritic soil in comparison with the gley A soil to adsorb (benzene) for the various concentrations observed.

#### 4.1. Simplified model for the estimation of benzene concentration in a saturated soil

A scenario of 1 m<sup>3</sup> of homogeneous lateritic soil was proposed in order to estimate the concentration of Brazilian commercial gasoline, whose composition is 25% ethanol and 0.56% benzene. The soil characteristics were defined as follow: specific weight of 14.45 kN.m<sup>-3</sup>, total porosity of 55.9%, degree of saturation (Sr) of 100%, and the soil's pores completely filled with water. In this situation, biological degradation factors which may influence the concentration of organic contaminants were not considered. In this regard, a leakage is assumed from a fuel tank containing 100 L of free phase of gasoline at the capillary edge interface with a saturated medium forming a Light Non-aqueous Liquid Phase (LNAPL). Thus, with gasoline having a density of 0.7663 g.mL<sup>-1</sup>, there will be a mass of 429.1 g of benzene. The relevant properties of the gasoline were obtained from the National Petroleum Agency (ANP) laboratory in Brasília.

In order to compare the adsorption average from *Batch Test* with retardation factor it was considered 200.0 mg of benzene dissolved in the aqueous phase of 60% volume of water contained in a 1 m<sup>3</sup> soil volume. The benzene partition to the adsorbed phase under field conditions may take days to reach equilibrium due to the residual volume of benzene remaining trapped in the pores (Donahue, 1999). A difference was determined between the initial dissolved and adsorbed phases ( $FD_i$ ) and the effective dissolved phase ( $D_e$ ), or real free concentration. Based on the soil characteristics, the gasoline under study and data in the literature, the effective dissolved phase and the adsorbed phase were calculated. The effective dissolved phase was calculated with the retardation factor, and the adsorbed phase with the percentage of the dissolved phase extracted indirectly from arithmetic average of the adsorption percentages. Therefore, while the average adsorption of the lateritic soil was 66.0% the percentage of the effective dissolved phase was 34.3%. The results presented in Table 4 were obtained from these data.

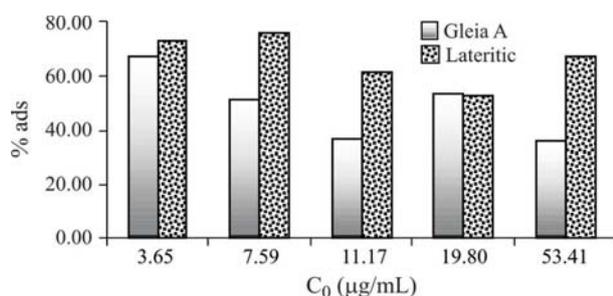


Figure 3 - Percentage of adsorbed benzene for the soils.

Table 4 - Calculations of the quantity of benzene partitioned in the soil and water.

$FD_i$ (mg)	$D_e$ (mg)	Adsorbed phase (mg)
200.0	5.55 <sup>a</sup>	194.45 <sup>a</sup>
200.0	68.54 <sup>b</sup>	131.46 <sup>b</sup>

<sup>a</sup>Calculation with the retardation factor. <sup>b</sup>Calculation with the average percentage obtained from the *Batch Tests*.

It may be seen in Table 4 that, from the result of the effective dissolved phase calculated by the retardation factor, it was possible to determine the adsorbed phase from the difference between  $FD_i$  and  $DE$ . Further analysis of the value obtained for the adsorbed phase indicates that it is overestimated, considering that maximum sorption in extreme conditions is empirically obtained by the *Batch Test*. However, maximum sorption would be 131.46 mg, which corresponds approximately to an average sorption of 66.0% in the *Batch Test*. From this perspective, the effective dissolved phase is underestimated when calculated by the retardation factor.

In order to calculate the concentration in the fluid-pore it is necessary to begin with the equivalent concentration for the soil, considering a contaminant mass in the initial dissolved phase (200.0 mg) in a given soil volume, which is expressed by the following equation:

$$C_E = \frac{M_{fd}}{V\rho} \quad (5)$$

where  $C_E$  = equivalent concentration (µg/g);  $M_{fd}$  = mass of the contaminant in the environment (mg);  $V$  = volume of the contaminated soil (m<sup>3</sup>);  $\rho$  = density of the soil (kg/m<sup>3</sup>).

Lastly, for the pore-fluid concentration the following equation is used:

$$C_p = \frac{C_E}{\frac{\theta_w}{\rho_w} + K_d} \quad (6)$$

where  $C_p$  = concentration of the pore-fluid (µg.mL<sup>-1</sup>);  $\theta_w$  = volumetric water content;  $\rho_w$  = water density (g.mL<sup>-1</sup>);  $K_d$  = partition or adsorption coefficient (mL.g<sup>-1</sup>).

The concentration in the pore-fluid is estimated at 0.010 µg.mL<sup>-1</sup> for  $K_d$  of 13.56 mL.g<sup>-1</sup> and the water density equal to 1 g.mL<sup>-1</sup>. These estimates, besides being applications of the Freundlich isotherms, are important to support rapid decision making in the event of an accident involving contamination by hydrocarbons. The concentration in the pore-fluid may also be important for diffusive flow studies, or to better understand a non-saturated medium where the water content is lower or scarce.

## 5. Conclusions

The adsorption isotherms for both soils studied presented higher results in comparison with data in the literature. However, for the two soils studied, the one presenting the best result was the lateritic soil. This result differs from other studies that demonstrate greater sorption as a function of the high organic matter content. Nevertheless, the greater sorption may be related to the type of interaction formed among the mineral, the type of organic matter and the adsorbate. The texture of the soil may be one of the factors influencing adsorption in clay rich soils by favoring a greater interaction with adsorbate as a function of the greater specific surface. Another important aspect to be considered is the role of the micro-aggregates of Fe and Al oxides, which may confine the particles of adsorbate. In this study, a larger quantity of exchangeable cations did not influence sorption, although this was expected due to the absence of interaction between polar elements and hydrophobic compounds. Despite soil gley A having a larger specific surface, due to the larger amount of organic matter, its results for benzene adsorption were smaller than those of the lateritic soil. However, the adsorption process was greater in lateritic soil, probably because the influence of Fe and Al oxides micro-aggregates.

From the simplified model developed to estimate the concentrations of benzene partitioned in the various phases, it was observed that the retardation factor may overestimate the concentration in the adsorbed phase.

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