

# Evaluation of the physical-chemical quality of honey without inspection registration sold in municipalities of Itapira and Mogi Guaçu, State of São Paulo

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### ABSTRACT

Honey, is The valuable food, but its quality can be compromised by adulteration. Brazilian legislation defines quality parameters for honey, including its chemical composition and physical-chemistry characteristics. The objective of this work is to verify whether commercial honeys are not registered in the municipalities of Itapira and Mogi Guaçu meet these legal standards, with the purpose of evaluating the quality and authenticity of the product. In this study, 10 samples of unlabeled honey were collected from the municipalities of Itapira -SP and Mogi Guacu -SP. Physicochemistry analysis were carried out covering reducing sugars, moisture, soluble solids, Ash, apparent sucrose, pH, acidity and color, in addition to adulteration tests, Lugol, Lund and Fiehe reactions, with all analysis being carried out in triplicate. The methodologies adopted followed the recommendations of the Adolfo Lutz Institute. The results were submitted I'm statistical analysis with ANOVA/Tukey test, considering a significance level of 5%. The main results revealed that one of the samples contained reducing sugars below the limit for floral honey, suggesting adulteration. For humidity, disappear samples exceeded the acceptable limit, which affects the quality and durability of the honey. The results obtained from soluble solids are in accordance with works found in the literature. At the mineral impurities were found in the ash content analysis. Some honeys showed high levels of apparent sucrose and acidity, raising concerns about adulteration and impact on flavor and quality. There was variation in the acidity content of the samples, but this remained in the typical range for honeys. Furthermore, analysis of colorimetric parameters revealed significant variations in the L\*, a\* and b\* components for all samples. For adulteration tests, in the Lugol reaction, only 40% of the samples presented negative results, suggesting The high level of adulteration by starch or dextrins. Regarding the Lund reaction, only 30% of the samples revealed protein precipitate within the expected range, indicating that 70% of the samples may have been adulterated by the addition of water or another diluent. In the Fiehe reaction, 70% of the samples demonstrated positive results, indicating adulteration due to the addition of sugar syrups or overheating of the honey. Through the results obtained, it was concluded that compliance with legal standards varies considerably between samples.

Keywords: Adulteration, Beekeeping, legislation, Colorimetric parameters.

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## **INTRODUCTION**

According to current legislation in Brazil, honey is considered a food product resulting from the activity of bees melliferas what collect nectar in flowers or secretions in partsliving plants, including excretions of plant-sucking insects, which are transformed and combined with specific substances of their own. This process occurs in hives, where the honey It is stored It is matured at over time (BRASIL, 2000).

Honey is made up of two types of sugars, glucose and fructose, along with presence of various mineral salts, such as calcium, sulfur, iron, copper, chlorine, sodium, phosphorus and magnesium. He constitutes one complex mixture what includes vitamins, proteins, enzymes, amino acids, polyphenols It is you products resulting from the reaction in Maillard (FILHO et al., 2012).

The chemical composition of honey, as well as its sensory characteristics, such as color, flavor and aroma, may vary due to several factors, such as climate, species of bee involved and the plant of origin of the nectar. This diversity makes it possible to obtain a wide range of honey types in different regions of the country. Furthermore, other elements what influence so much at quality of honey how much in your parameters physical-chemicals include appropriate management of the hives, the selection of the location for installing the apiary, the process of harvest, O appropriate transportation It is to the phases in processing of honey (FERREIRA, 2022).

Per account from the growing demand at the Marketplace It is The growing expectation of the consumers for high quality products, it is crucial that the honey available for sale is a product genuine, according to to the regulations current. At the however, O honey he can to be susceptible The tampering what compromise your integrity, such as The inclusion in sugar conventional, corn syrup, glucose, molasses, invert sugar solution and glucose syrup (DANTAS; SANTOS; SANTOS; SILVA; CARVALHO, 2022).

Despite regulations prohibiting the adulteration of honey, which occurs with frequency due to its relatively high price, the ease of incorporating substances adulterants and the need for laboratory analyzes to identify them, it is common to observe the occurrence of fraudulent practices. According to Decree No. 10,468, dated August 18,2020, which establishes standards for the industrial and sanitary inspection of products of origin animal, consider themselves cheats The action in deprive partial or totally raw material It isproducts of their original characteristics through replacement by inert substances or foreign substances, as well as the addition of ingredients, additives or technological adjuvants to disguise or hide changes, deficiencies in the quality of raw materials or flaws in the process in production, or with The goal in increase O volume, or Weight of product (FERREIRA, 2022).

Among the circumstances, the legislation in force emphasizes the main parameters of physicochemical composition of honey that must be evaluated, including moisture, sugars reducers,

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apparent sucrose, insoluble solids, minerals, ash, acidity, diastasis index and hydroxymethylfurfural (HMF). It also highlights that the detection of fraud and adulteration in honey must be carried out through physical-chemical analyzes stipulated by legislation and the Institute Adolf Lutz (BRAZIL, 2000).

As it is considered of animal origin, honey sold must be registered with the Ministry of Agriculture, Livestock and Supply (MAPA) and have the approval of theFederal Inspection (SIF), responsible for inspecting and ensuring the quality of food products origin animal (ALBUQUERQUE, 2021). O record plays one paper crucial at protectionpublic health, ensuring the quality of honey and promoting ethical business practicesIt is transparent, ensuring the integrity It is security of the honeys sold.

The present work had the general objective of evaluating the physicochemical characteristics of samples of honey without inspection records sold in the municipalities of Itapira-SP and Mogi Guaçu-SP.

# **REVIEW IN LITERATURE**

## HONEY

Among the products resulting from bee activity, honey stands out as the most famous and widely known. Throughout human history, honey has been one of the first foods and also played a significant role as a medicinal resource in many ancient civilizations. Currently, honey is appreciated as a food, recognized so much per your qualities medicinal how put your value nutritional (SILVA, 2006).

According to stipulated at Instruction Normative no. 11/2000 issued for the Ministry from the Agriculture, Livestock It is Supply (MAP), O honey It is categorized with base in your origin, being Divided in two categories main: honey floral It is honey in melate. O honey floral It is collected from flower nectar and can be classified as unifloral or monofloral, depending of the predominance of a single species of flower. This honey variant has properties sensorial, physical-chemical characteristics and distinct microscopic characteristics. For another On the other hand, melate honey is mainly composed of secretions from living parts of plants or excretions of insects that feed on plant sap, which end up accumulating on these plants.

O honey It is one substance composed per several sugars, with predominance of the monosaccharides glucose It is fructose. In addition from that, contains proteins, amino acids, enzymes, acidsorganic, minerals, pollen It is others substances, including sucrose, maltose, maleitosis It is others oligosaccharides (including dextrins). May also contain small amounts of fungi, algae, yeast It is others particles solid resulting of process in collect of honey (MENDES et al., 2009).

The honey production process involves collecting nectar from flowers, in the case of honey



floral, or secretions from living parts of plants and excretions from insects that feed on sap in plants, at the case of honey in melate, what they are then stored at vesicle honey ofbees. In the hive, the nectar goes through a transformation process that includes two stages, one physics and one chemistry. The physical stage consists of reducing the moisture content, occurring thanks to the mandibular movements of bees, which involve ingestion and regurgitation of the nectar, as well as the flapping of wings, reducing the moisture content to about 17% to 18%. A chemical step involves the addition of specific enzymes, such as invertase, glucose oxidase and diastasis (FERREIRA, 2022).

A invert convert part from the sucrose of nectar collected in glucose It is fructose It is to be continuedacting until the honey ripens. This sucrose hydrolysis reaction produces a solution concentrated sugar content, which contributes to the honey's resistance to fermentation, ensuringits quality. Glucose oxidase generates gluconic acid and hydrogen peroxide from glucose gift at the honey, substances what also help The avoid O growth in microorganisms, especially in the early stages of the process, when the moisture content is still high. Diastase is the enzyme responsible for breaking down starch and can be used as one indicator in overheating of honey, then It is unstable in temperatures high (FERREIRA, 2022).

To the characteristics of honey, as flavor, aroma, color, viscosity It is properties medicinal products, are intrinsically related to the source of nectar and the species of bee that produces it. produced (DAMASCENO, 2012). O honey It is considered one food in high quality nutritional due The presence in vitamins, minerals It is your high value energy, these nutritional characteristics, along with its medicinal properties, have contributed to one growing interest on part of consumers (MACEDO, 2007).

### PRODUCTION NATIONAL OF HONEY

A history from the beekeeping at the Brazil started in 1839, at the state of River in January, whenFather Antonio Carneiro introduced *Apis mellifera bees* with authorization from Dom PedroII, per quite of Decree no. 72 in 12 in July in 1839. Posteriorly, immigrants Europeans contributed to the introduction of other bee species, mainly in the South andSoutheast. At that time, beekeeping was more of a hobby than a professional activity. It is economic (DAMASCENO, 2012).

In the 1950s, Brazilian beekeeping faced a significant crisis due to pests and diseases that led to the extinction of 80% of hives in the country. Professor Warwick Estevan Kerr, with support from the Ministry of Agriculture, carried out research in Africa to find African queen bees with good productivity characteristics and resistance to illnesses, aiming to improve The beekeeping at the Brazil. At the however, one incident at the apiary experimental in They are Paul resulted at escape in bees queens African for The nature, due to their aggressive behavior. This led to sensationalist reports and the abandonment of activity per many beekeepers, while others if adapted The techniques in

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management for to leadwith to the "bees Africanized " (TREVISOL; BUENO; OLIVEIRA; MACEDO, 2022). Second Damascene (2012), The introduction of bees Africanized he was one point in turn forThe beekeeping Brazilian. After O development in techniques suitable in management at decade in70, The beekeeping if returned an activity strong all over the country.

Honey productivity in Brazil is relatively low compared to others countries. According to the 2017 Agricultural Census, Brazilian beekeepers recorded a average in 19.8 kilos in honey per hive per year. A China leads O ranking worldwide in production honey, followed by Türkiye, Iran and Argentina. Brazil occupies tenth position on the list of largest producers, with 51 thousand tons in 2020 (TREVISOL; BUENO; OLIVEIRA; MACEDO, 2022).

According to the Brazilian Association of Bee Studies - ABELHA (2022), in 2021, O Brazil achieved one record at production in honey, with 55.8 thousand tons, representing an increase of 6.4% compared to 2020. The value of production also increased, reaching R\$ 854.4 millions, one addition in 34.8% in relationship The 2020. O price average of honeyrose from R\$ 12.07 for R\$ 15.30 per kilo.

In 2022 data released by IBGE, honey production in Brazil reached 60.97 thousand tons, overcoming you numbers of year previous. O state of River Big of South highlightedas the main producer, contributing 9 thousand tons, generating revenue of R\$ 957,811 thousand. The municipality of Bagé, in this state, led the production, contributing R\$ 8,100 thousand. In Paraná, production totaled R\$ 138,993 thousand with a quantity produced of 8.6 tons. O bigger County producer he was Arapoti, contributed with R\$ 16,363 thousand At the Piauí,production reached R\$121,715 thousand with a quantity of 8.3 tons. Saint Raymond Nonato, in this state, he was the municipality with bigger production, generating R\$ 11,198 thousand. In Minas Gerais, production reached R\$89,307 thousand with a quantity of 6 tons. The municipality of Ant He stood out as O bigger producer, generating R\$ 4,230 thousand. They are Paul occupied The fifthposition, with production valued at R\$73,158 thousand and 4.8 tons. Botucatu was the County more prominent, generating R\$ 9,113 thousand, while Itapira It is Mogi Guaçu contributed with R\$ 147 thousand and R\$ 111 thousand respectively.

## LEGISLATION

The physicochemical parameters for honey in Brazil are established by the Ministry from the Agriculture, Livestock It is Supply (MAP) with O purpose in standardize O processing of products of animal origin. This aims to ensure equal conditions and total transparency in the production and marketing of these products. Normative Instruction no. 11, dated 20 of October 2000, approved O Regulation Technician of Identity and Quality of honey. That regulation define The identity, classification, designation, compositionIt is you requirements relative to the characteristics physical-chemical, sensory, conditions in storage, additions, control in contaminants, conditions hygienic,

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criteria macroscopic It is microscopic, weights It is measures, labeling, sampling It is methods in analysisto be followed (BRASIL, 2000). This instruction is in accordance with the guidelines of the Ministry ofHealth, according to The Resolution MERCOSUR/GMC/RES. No. 89/99 (BRAZIL, 1999).

Second The Instruction Normative no. 11, in 20 in October in 2000, to the analytics what must be performed routinely on honey include the quantification of reducing sugars, humidity, apparent sucrose, water-insoluble solids, ash, acidity, diastasis activity and content in hydroxymethylfurfural (HMF). O Regulation Technician in Identity It is Quality of Honey(BRASIL, 2000) strictly prohibits the addition of any sugars or substances that do not do part from the composition original of honey, It is require what all honey intended to the consumption humanmeets minimum quality requirements. The established physicochemical parameters are described at Table 1.

	on Brazilian	
lications		
Honey Floral Me		
Variable		
Minimum 65	Minimum 60	
Maximum 6	Maximum 15	
Maximum 0.1		
Maximum 20		
Maximum 0.6	Maximum 1.	
Maximum 50		
Minimum 8		
Maximum 60		
	Varia Minimum 65 Maximum 6 Maximu Maximu Maximu Maximu Maximu Minimu	

Source: Adapted in BRAZIL (2000).

Although not included in Brazilian legislation, some guidelines are used to identify adulterations or conservation problems in honey. The standards established for the Lugol, Lund and Fiehe reactions can be consulted in Table 2. These determinations are not specifically included in Normative Instruction no. 11, dated 20 October in 2000, but they are recommended for the Institute Adolfo Lutz as practices auxiliaries atassessment of quality of honey.

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Table 2: Standards settled down for the Institute Adolfo Lutz for to the reactions in Lugol, Lund It isField		
Parameters	Limits settled down	
Des des Los el	Need	
Reaction Lugol	Negative	
Reaction Lund	maximum 3.0 mL It is minimum 0.6mL.	
Reaction Fiehe	Negative	

Source: Adapted in IAL (2008).

In addition to the inclusion of unauthorized substances, the legislation also prohibits the honey overheating process, which involves raising the temperature to values superiors The 60°C (BRASIL, 2000). That practice in overheating It is oftenemployee with O purpose in reuse honeys what already demonstrate signals in fermentation initial, reduce to tendency to crystallization or to facilitate the process filling (FERREIRA, 2022).

## METHODOLOGY

## **OBTAINING FROM THE SAMPLE**

The bee honey used in this work comes from the municipalities of Itapira- SP (5 samples) It is Mogi Guaçu -SP (5 samples). They were 10 samples collected in warehouses, emporiums, open fairs and markets, in August 2023. They were in the formin liquid honey, devoid of any labeling.

The samples were taken to the Bromatology Laboratory of the Federal Institute of South of Minas Gerais (IFSULDEMINAS) - Inconfidentes campus, where they were duly packaged The temperature environment It is to the shelter in light in your packaging designated, until The conclusion in all to the analytics physicochemical. These analytics they were executed during O monthin September of 2023.

## ANALYTICS PHYSICAL CHEMISTRY

To the analytics of this work they were carried out at the Laboratory in Bromatology, of InstituteFederal southern from Minas General- campus Inconfidentes (IFSULDEMINAS).

They were conducted analytics physical chemistry for The determination in sugars reducers, moisture, solids soluble, ash, sucrose apparent, pH, acidity It is color, in addition of the tests of adulteration, including The reaction in Lugol, reaction in Lund It is The reaction in Fiehe. All to the analyticsthey were carried out in triplicate, following The methodology recommended for the Institute Adolfo Lutz(IAL, 2008).

## **Sugars reducers**

Reducing sugars were determined according to method A (176/IV) of the Instituto Adolfo Lutz (IAL, 2008). In this technique, the product is hot titrated using solution in Fehling A It is B, at

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presence in blue in methylene, no overtaking O period in threeminutes.

## Moisture

A determination from the moisture he was carried out in refractometer analog, brand INSTRUTHERM, with scale in 0 The 95 °Brix, getting O index in refraction The 20°C. He was followed the 173/IV method of the Adolfo Lutz Institute (IAL, 2008), obtaining the percentage of humidity according to Table Chataway.

### **Solids soluble**

You solids soluble they were determined in refractometer analog, brand INSTRUTHERM, with scale in 0 to 95 °Brix, getting O index in refraction The 20 °C, second Omethod 010/IV of Adolfo Lutz Institute (IAL, 2008).

### Ashes

Ash determination was carried out by heating the sample, following the method 018/IV of the Adolfo Lutz Institute (2008), where the material was incinerated at 550 °C and cold The temperature environment in one desiccator. To the operations they were repeated until obtaining constant weight.

### **Sucrose apparent**

The determination of apparent sucrose was carried out by titration. The samples were hydrolyzed It is treated with acid hydrochloric, followed for the neutralization with hydroxide in sodium. The titration of Fehling's solutions A and B, in the presence of methylene blue, was performed quickly, not exceeding three minutes, according to method A (178/IV) established by the Institute Adolfo Lutz (IAL, 2008).

## pН

O pH he was determined in meter in pH in bench, brand Alakit, model AT-355, second O Institute Adolfo Lutz. (IAL, 2008) according to O method 017/IV- Determinationelectrometric of pH.

## Acidity

The acidity of the honey solution was determined by titration, after diluting the honey in phenophytalein was added to water, and the titration was carried out with sodium hydroxide solution. sodium, until reaching pH 8.5. The pH measurement was made with a benchtop pH meter from the brand Alakit, model AT-355, following O method 016/IV of Institute Adolfo Lutz (IAL, 2008).

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## **Parameters colorimetric**

A analysis in parameters colorimetric he was carried out in samples in 30 mL at the colorimeter Konica Minolta (Model CM-2300). He was used O system CIELab (L\*, The\* It is B\*), at the which L\* represents O component in brightness, varying in 0 The 100. The parameters The\* It is b\* correspond to the two color components, ranging from -100 to 100. The a\* componentspans the scale from green to red, while the b\* component ranges from blue to yellow .

### **Reaction in Lugol**

Lugol reaction analysis was performed using diluted honey samples. After a process of heating in a water bath, followed by cooling, 0.5 mL was addedfrom the solution in Lugol. A occurrence in one change for shades reddish-brown orblue indicated The presence in dextrins, or starch at sample, according to to the guidelines of InstituteAdolfo Lutz (IAL, 2008) established by method 184/IV.

## **Reaction in Lund**

A reaction in Lund he was determined according to to the guidelines of Institute Adolfo Lutz (IAL,2008), following method 182/IV. In the procedure, the honey samples were diluted and subsequently, 0.5% tannic acid and water were added. After rest, it was observed training or absence of precipitate.

## **Reaction in Fiehe**

A reaction in Fiehe he was determined second to the guidelines of Institute Adolfo Lutz (IAL, 2008), using method 183/IV. In the procedure, the honey samples were diluted in ether. The resulting solution was then transferred to a test tube containing solution hydrochloric in resorcino It is left in rest. O emergence in one coloring red indicatedThe presence in glucose commercial or overheating in honey.

## ANALYSIS STATISTIC

The results obtained in the physicochemical and colorimetric analyzes were subjected to analysis in variance (THE NEW) with O test in Tukey The 5% in probability. That procedurestatistical analysis was carried out using the Sensomaker® computer program, developed byPinheiro, Nunes and Vietoris (2013). The results obtained were compared with the legislation in force, specifically Normative Instruction no. 11 of 2000, which establishes the requirements and standards for honey in Brazil (BRAZIL, 2000).

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## **RESULTS AND DISCUSSION**

## ANALYTICS PHYSICAL CHEMISTRY

You results of analytics physical chemistry in sugars reducers moisture, solids soluble, ash, sucrose apparent, acidity and pH, are presented in the Table 3.

Sample	Sugars reducers	Moisture (%)	Solids soluble	Ashes (%)	Sucrose	Acidity	pН
no.	(g/100g)		(°Brix)		apparent	(mEq/kg)	
					(g/100g)		
01	75,01°±1,18	$22,00^{b} \pm 0,01$	78,00 ° ±0,00	$0,32^{b}\pm0,20$	$6,29^{d}\pm0,66$	14,86 <sup>e</sup> ±0,91	3,59 <sup>b</sup> ±0,08
02	$61,23^{h}\pm0,57$	$22{,}50^a{\pm}0{,}02$	77,50 <sup>f</sup> ±0,02	$0,23^{b}\pm0,26$	$15,50^{b}\pm0,34$	$18,45^{d,e}\pm0,07$	3,59 <sup>b</sup> ±0,02
03	73,19 <sup>c,d</sup> ±1,57	22,50 <sup>a</sup> ±0,02	77,52 <sup>f</sup> ±0,01	0,45 <sup>a</sup> ±0,19	$1,20^{e}\pm0,56$	$63,52^{b}\pm6,21$	3,77 <sup>a</sup> ±0,01
04	85,96 <sup>a</sup> ±0,43	$21,00^{d}\pm 0,02$	79,00 ° ±0,04	$0,07^{c}\pm0,07$	$2,82^{e}\pm 1,63$	$27,69^{d}\pm1,26$	$3,76^{a}\pm 0,00$
05	75,39°±1,43	22,33 <sup>a,b</sup> ±0,28	77,67 $^{\rm e,f}\pm0,28$	$0,44^{a}\pm 0,02$	$6,61^{c,d}\pm 2,29$	$18,84^{d,e}\pm 1,51$	3,66 <sup>b</sup> ±0,02
06	79,37 <sup>b</sup> ±1,26	20,00 <sup>e</sup> ±0,03	$80,00^{b}\pm0,04$	$0,46^{a}\pm 0,00$	$2,59^{e}\pm0,67$	93,59 <sup>a</sup> ±1,23	2,79 <sup>e</sup> ±0,01
07	68,49 <sup>e,f</sup> ±0,46	20,17 <sup>e</sup> ±0,27	79,83 <sup>b</sup> ±0,25	$0,04^{c}\pm0,00$	$10,05^{\circ}\pm0,94$	48,63°±8,28	$3,04^{d} \pm 0,01$
08	$66{,}97^{\rm f,g}\pm\!\!0{,}68$	$18,50^{\rm f}\pm0,03$	81,50 <sup>a</sup> ±0,03	0,41 <sup>a</sup> ±0,05	$19,24^{a}\pm0,74$	43,59°±1,78	3,31° ±0,01
09	70,59 <sup>d,e</sup> ±0,76	20,00 <sup>e</sup> ±0,02	80,02 <sup>b</sup> ±0,01	0,43 <sup>a</sup> ±0,29	$13,69^{b}\pm1,09$	69,28 <sup>b</sup> ±2,39	$3,09^{d} \pm 0,01$
10	64,94 <sup>g</sup> ±0,73	21,50° ±0,03	78,51 <sup>d</sup> ±0,02	$0,29^{b}\pm0,18$	$4,\!85^{d,e}\pm\!1,\!50$	59,77 <sup>b</sup> ±2,00	$3,80^{a} \pm 0,01$

 Table 3: Results of analytics physical chemistry of samples in honeys commercialized us counties in Itapira- SP It is

 Mogi Guaçu -SP

\*means followed by the same letter, in the same column, do not differ from each other at  $p \le 0.05$  using the Tukey Test. Source: Authors (2023).

### **Sugars reducers**

Upon completing the analysis of reducing sugar levels in honey samples, stands out This one parameter as one indicator fundamental from the composition of that product. Table 3 presents notable variations between the samples, evidenced by the averages followed by distinct letters, indicating statistically significant differences ( $p \le 0.05$ ), according to observed at the Test in Tukey. A sample what presented O bigger content inreducing sugars was number 04, statistically differing from all other samples analyzed. It can be seen that the sample of No. 02 had the lowest sugar content reducers, statistically differing in all other samples analyzed.

According to the guidelines of current legislation (BRASIL, 2000), the minimum content acceptable for reducing sugars is set at 65% for floral honey. In this study, one can observe at Table 3, what just The sample in no. 02 presented contents below ofstipulated limit. However, it is worth noting that legislation allows honey melate possess one content Minimum in 60%. Of that form, although O honey in question exceed the limit stipulated for floral honey, it does not violate the values established for honey melate. The specific categorization of honey, due to the lack of labels in the analyzed samples, remains unknown. All remaining samples comply with current legislation, so much in what if refers to the floral honey how much to honey in melate.

Lopes (2015) analyzed honey from the jatai bee (*Tetragonisca angustula*), collected in the district of Felisberto in the city of Curiúva-PR and identified, when analyzing the rates of sugars

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reducers, values maximums reaching 63.43% It is minimums in 54.55%. In significantly, all samples analyzed demonstrated that they did not comply with thestandards established by Brazilian legislation.

In one study about The solubility from the glucose in solutions in fructose, similarAt the concentrations found in honey, it was observed that glucose became more soluble with O increase from the concentration in fructose. That observation he was explained for the balancebetween anhydrous glucose and monohydrate glucose (form present in granulated honey). In high levels of fructose, the balance favors the anhydrous form of glucose, which is more soluble in water. The relevance of this balance to granulation was supported by evidence that in solutions saturated with fructose, the transition from glucose monohydrate to glucose form anhydrous occurs below in 30°C. One can infer what honeys with tall contents in fructoseIt is lows contents in glucose they are any less prone The crystallization, one phenomenon what affects Theacceptance of these natural products by consumers (MOREIRA; MARIA, 2001).

According to highlighted per Silva (2006), The presence in sugars at the honey playsone paper crucial at your conservation, one turn what contributes for The creation in one pressureosmotic in the medium, which, in turn, prevents the development of bacteria, mold, yeasts and other unwanted microorganisms. This suggests that honeys that presentcontents in sugars below of limit Minimum allowed for the legislation they can to createconditions more favorable to the proliferation of these microorganisms, which lights up a signal in alert regarding The quality It is security of these products.

### Moisture

To the finish The assessment in analysis in moisture in samples in honeys, he was considerable variability was identified, as can be seen in Table 3. note that samples No. 02 and 03 obtained the highest moisture contents, not statistically differing from sample no. 05. The sample that presented the lowest was no. 08 differing statistically from all other samples analyzed. Among the results obtained, presented in Table 3, only samples No. 06, 07, 08, and 09 they are according to The current legislation, the which establishes a limit maximum of 20%.

The importance of maintaining the moisture content of honey within these parameters legal is directly linked to the quality of the product and its durability. This is due to the fact that, when honey has a high moisture content, there is a greater susceptibility The Law Suit in fermentation. That vulnerability occurs due The possible contamination by microorganisms, which are naturally found both in the area of honey extraction and in the bodies of bees, which play a crucial role in production of this precious liquid (SOUZA, 2016).

Second Mora-Escobedo et al. (2006), The moisture initial of honey also It is one of thefactors



influential at your crystallization, then how much smaller The moisture bigger The concentration of monosaccharides (glucose and fructose), resulting in a higher level of saturation and, consequently, greater probability of crystallizing. This crystallization varies depending with several factors, such as the concentration of sugars, the water content in its composition Natural, The origin floral of nectar, O handling during your processing, good asto the conditions of storage (COMFORTI et al., 2006).

The water content in honey is influenced by several factors, ranging from conditions climate at the day from the harvest until O level in maturation of bees responsible for its production. Honey is a hygroscopic product, that is, it has the ability to absorb water of environment. A amount in water gift at the honey it is directly related The occurrence in fermentation unwanted, one turn what how much bigger The amount of water present, the greater its susceptibility to this process. When the moisture content exceeds the limit of 20%, this indicates that the honey has undergone addition of water, has been subjected to inadequate processing or has been harvested before reaching maturity. internship in adequate maturation (MEIRELES, Cçado, 2013).

### **Solids soluble**

Concluding the investigation into the analysis of soluble solids in samples of honeys, notable variations were found between samples, evidenced by the averages followed by distinct letters, indicating statistically significant differences ( $p \le 0.05$ ), according to introduced at Table 3. One can observe what The sample in no. 08 gotthe highest content of soluble solids, statistically differing from all other samples analyzed. To the samples in no. 02 It is 03 presented you minors contents in solid solubleno statistically differing from the sample in no. 05.

At the what it says respect to the values medium in solids soluble, It is important observewhat The legislation Brazilian no includes This one parameter in its guidelines. How much bigger for the sugar content and the longer the maturation period of the honey, the greater the presence of soluble solids. Therefore, the analysis of this parameter was conducted with the purpose of enrich The gamma of variables for comparing results.

The soluble solids values obtained in the samples analyzed in this work, ranged from 77.50 to 81.50° Brix, as indicated in Table 3. These results are aligned with you found per Barbosa (2013) in your search about honeys marketed in the fairs from the city in Empress, at the state of Maranhão, what presented variations of 77.31 a 81.36° Brix.

In the work carried out by Vieira et al. (2017), which performed the physical- chemistry of honeys produced in the state of Mato Grosso do Sul, Brazil, for the analysis of soluble solids content (°Brix), the observed values ranged from 75 to 80 °Brix, with value average in 76.05±1.5 °Brix, you values found in this study, they were Upcoming to the obtained by Santos et al. (2010) in honeys produced in the State of Ceará whose indexes found were 73.80 to 80.05 °Brix.

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Total soluble solids are not a parameter established in legislation, but its content is directly related to the sweetness of the honey, as it reflects the amount of sugar present. The evaluation of this characteristic is relevant due to the preference of the consumer per honeys with levels lowest of sugar (SILVA et al., 2009).

## Ashes

Upon concluding the analysis of the ash content in honey samples, this parameter as a fundamental indicator of the composition of these products. Accordingly with Table 3, as observed in the Tukey Test, the samples that presented you bigger contents in ashes they were to the in no. 03, 05, 06, 08 It is 09, no differing statistically among themselves. It can be observed that samples No. 04 and 07 showed you lower levels medium for the content in ashes, no statistically differing between yes.

According to Brazilian legislation, the maximum permitted limit for ash content in floral honey it is established at 0.60% (BRASIL, 2000). Therefore, based on these data, all the honeys analyzed in this study they are according to the limit stipulated.

The ash content is indicative of the presence of minerals in honey, mainly compounds per abundance in potassium (K), sodium (At), calcium (Here) It is magnesium (Mg), in additionin small quantities in aluminum (Al), iron (Faith), copper (Ass), manganese (Mn) It is zinc(Zn), along with traces of arsenic (Ar), iodine (I) and fluorine (F) (GOIS et al., 2013). As highlighted by Marchini et al. (2004), the ash content represents the minerals present in honey, which can be used as a criterion to evaluate its quality, and it is related to your botanical origin It is geographic.

Anacleto et al. (2009) observed that the percentages of ash varied between 0.21% and 0.60%, with an average of 0.39%, and these values are within the limits established by the Ministry of Agriculture and Supply. Souza (2017) also adds what The determination of content in ashes he can to reveal possible irregularities at thehoney, such as contamination caused by lack of decantation or filtration at the end of product extraction process, as well as adulteration, serving as an indication of its quality. Vieira et al. (2017) in their study on physical-chemical characterization of honey produced in the State of Mato Grosso do Sul, reported ash contents that ranged from 0.06% to 0.55%, with an average of  $0.25\% \pm 0.17\%$ , in accordance with the legislation in force.

## **Sucrose apparent**

According to The Table 3, you results in sucrose apparent in samples in honeys withoutrecord commercialized in Itapira It is Mogi Guaçu demonstrated be difference statistic in between you scores medium for This one parameter. A sample what presented O biggersucrose content was no. 08, statistically differing from all other samples analyzed. Note what to the samples in no. 03, 04 It is

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06 presented minors contents medium for content in sucrose apparent, no different from the sample in no. 10.

According to current Brazilian legislation (Brazil, 2000), the maximum parameters established for apparent sucrose levels are 6% for floral honey and 15% for molasses honey. It is observed that samples No. 02 and 08 do not comply with thementioned legal limits, since they exceed the values stipulated for both honey floral how much for O honey in melate. Per other side, to the samples identified by the no. 01,05, 07 and 09 are within acceptable limits for melate honey, however, exceed the tolerated values for floral honey. Finally, it is worth highlighting that the samples with no. 03, 04 It is 06 they were to the unique what if framed us Limits settled down for the honey floral, as recommended by legislation in force.

Sodré et al. (2007) found values for apparent sucrose between 0.16 and 7.63, for honeys analyzed at the state of Ceará. Studies in Rue et al. (2004) about qualityin honeys of County in Santana of Cariri, at region from the Chapada of Araripe, describedvalues between 0.84 and 8.19%. The percentages of apparent sucrose in honey samples analyzed ranged from 5.80% to 19.96%. According to Melo et al. (2016), 60% of samples analyzed introduced themselves with values superiors to the what determines The Instruction Normative no. 11 (BRASIL, 2000), regarding his work on honeys sold in region in Uberlândia-MG.

The apparent sucrose content is of critical importance in evaluating the origin It is quality of honey, unraveling if to the bees were fed with sugar at thebeginning of flowering or if there was direct adulteration of the honey by adding sucrose, such as highlighted by Silva (2007). This analysis reveals crucial information about the process of production of honey, well like integrity of the final product.

### Acidity

Concluding the acidity analysis in the honey samples, it was evident the presence of distinct values, as indicated in the Tukey Test. As can be seen in the Table 3, sample no. 06, had a higher acidity content, statistically differing from all samplesothers samples analyzed. A sample what presented O smaller content in acidity he was The no. 01,no statistically differing of the samples of no. 02 and 05.

Brazilian legislation establishes a maximum limit of 50 milliequivalents of acidity per kilogram in honey (mEq/kg) (BRAZIL, 2000). In this study, it was found whatsome samples presented values above this limit. As can be seen in Table 3, specifically, to the samples in no. 03, 06, 09 It is 10 they are in disagreement with The current legislation, demonstrating one discrepancy in relationship to the standards settled down.

Acidity is an extremely important parameter when evaluating honey, as what exercises

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influence direct about your flavor It is your capacity in conservation. That occursbecause the acidity is significantly affected by processes as fermentation, as pointed per Silva et al., (2008).

A origin from the acidity gift at the honey drift in miscellaneous sources, such as The variation in organic acids from different nectar sources, the action of enzymes, the paper played per bacteria during O process in maturation of honey It is, additionally, the amount of minerals present in the composition of the honey, as discussed by Evangelista et al., (2005). This complex interaction of factors demonstrates as The acidity of honey he can to be one indicative valuable from the your origin It is quality, while also highlights The importance of greeting of the standards settled down for the legislation for ensure integrity of the final product.

In the study conducted by Soares, Aroucha and Góis (2011), which addressed honeys wild animals in the State of Rio Grande do Norte, high levels of acidity, varying in between 26.73 It is 126.77 mEq/kg. Second to the discoveries in Lopes (2015), The variable acidity presented one average in 56.44 meq/kg, with values Minimum It is maximumin 47.41 meq/kg It is 65.00 meq/kg, respectively. Such results suggest what some samples they are in disagreement with you standards settled down for the legislation Brazilian.

### pН

The pH values obtained from the 10 honey samples analyzed, presented in Table 3 indicate that samples No. 03, 04 and 10 presented the highest values, not statistically different from each other. It is noted that sample number 06 presented the smaller value, differing statistically in all others analyzed samples.

There is no indication of pH analysis as mandatory in Brazilian legislation forquality control of honey samples, however, proves to be beneficial to assist in indication of fermentation or adulteration of honey (ELLER, 2022). Floral honey has values below 4.0 and melate honey superiors The 4.5.

To the variations in pH found in this work, introduce themselves nearby to thevalues obtained per Pereira et al. (2015), what, to the analyze O pH in 10 samples in honeys, in the municipality of Maringá, PR, they obtained results of 2.86 to 4.17. Just like, Finco, Moura It is Silva (2010) what found value average in pH in 3.7 in 24 samples in honeys in many different flowery.

Pinto and Lima (2010) who, when evaluating 26 honey samples from the Vale do Steel, MG, got results in pH in between 2.41 The 4.53. Lily et al. (2015) to the analyze24 samples of wild honey sold in the State of Rio de Janeiro obtained results for pH between 3.80 and 4.90, which are values close to those found by Andrighetto et al. (2009), with a pH of 3.79 to 4.67, in the city of Santo Augusto, RS, with four samples of honeys.

A variation of the values in pH in honeys no he can to be underestimated, one turn what it suits

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as an indication of various conditions that may have a significant impact about the quality and authenticity of honey. Adulteration of honey with syrups or other sugars, for example, can result in a decrease in pH, giving it a greater acidity, as evidenced by Araújo, Silva It is Sousa (2006).

## PARAMETERS COLORIMETRIC

You results of analytics of the parameters colorimetric of samples in honeys they are presented at Table 4.

Table 4: Results of the parameters colorimetric of samples in meis commercialized in the municipalities of Itapira- SP and Mogi guaçu -SP

Sample no.	Luminosity	Component a	Component b
01	50,37 <b>a</b> ±1,74	1,24 <sup>c</sup> ±0,15	$16,41^{b} \pm 0,51$
02	45,99 <sup>a,b,c</sup> ±1,06	2,36° ±0,29	24,27 <b>a</b> ±0,19
03	33,53 <sup>e</sup> ±2,57	10,30 <b>a</b> ±1,99	10,39° ±0,77
04	$40,59^{d}\pm1,03$	8,59 <sup><b>a</b></sup> ±0,13	24,70 <sup><b>a</b></sup> ±0,85
05	$43,42^{b,c,d}\pm0,77$	$5,54^{b} \pm 1,01$	23,89 <sup><b>a</b></sup> ±2,91
06	40,75 <sup>c,d</sup> ±0,88	7,50 <sup>a,b</sup> ±0,79	23,27 <b>a</b> ±1,42
07	43,65 <sup>b,c,d</sup> ±1,04	1,19 ° ±0,06	8,65 ° ±0,17
08	26,22 <sup>f</sup> ±2,49	2,73 ° ±0,38	$1,22^{d}\pm0,40$
09	48,07 <sup>a,b</sup> ±0,75	0,73 ° ±0,18	23,42 <sup><b>a</b></sup> ±0,18
10	$34,16^{e}\pm1,20$	7,96 <sup>a,b</sup> ±0,65	15,47 <sup>b</sup> ± 0,17

\*averages followed by the same letter, in the same column, do not differ from each other after  $p \le 0.05$  for the Test Tukey. Source: Authors (2023).

Among you values obtained in each component of system, The Luminosity varied in between 26.22 It is 50.37, as power to be observed at Table 4. To the samples what presented the highest values for this component were numbers 01, 02 and 09, not differing statistically among themselves. It can be seen that sample number 08 obtained the lowest value, differing statistically in all other samples analyzed.

The a\* component varied from 0.73 to 10.30, as demonstrated in Table 4. The samples that presented the highest values for this component were samples no. 03 and 04, not statistically different from samples no. 06 and 10. It is noted that samples No. 01, 02, 07, 08 and 09 obtained lower averages, not differing statistically between yes.

For O component B\* he was obtained one variation in 1.19 The 24.70 according to presented in Table 4. It can be seen that samples No. 02, 03, 04, 05 and 09 obtained higher values for this component, not statistically different from each other. The samples that presented the lowest averages were numbers 03 and 07, not differing statistically in between yes.

The color of honey may be related to the processing and storage of honey, your composition chemical, to the factors climatic during O flow of nectar It is The temperatureat which honey ripens at hive (SOUSA et al., 2020).

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For some researchers, the variation in the original color of honey is not considered a significant indicator of its quality, since aging is a factor that can intensify this characteristic (LACERDA et al., 2010). However, Azeredo et al. (1999) conducted a study on the physicochemical characteristics of honeys in São Fidelis, at the state of River in January, It is your results indicated what no there was variationin color, both in absorbance and in visual observation of the samples, throughout analytics carried out at intervals regular over 365 days.

The study carried out by Oroian et al. (2012) included 15 samples of acacia honey, linden and sunflower, classified according to floral origin based on their color. Using the CIELab system, which includes luminosity coordinates (L\*), color axes (a\* and b\*), hue angle (H) and color intensity (C\*). The colorimetric parameters were statistically different between samples, highlighting the usefulness of the CIELab system at classification of honey with base in your origin floral. Analytics colorimetric carried outby Sousa et al. (2020) using the CIELab method indicated that *S. bee honey. Bipunctata* did not present a statistically significant difference in relation to honey produced for the bee *M. Quadrisfasciata*.

At the study conducted per Oroian, Ropciuc It is Buculei (2016), didicated The authentication of different types of Romanian honeys, physicochemical and chemometrics. Five varieties different they were analyzed: acacia, sunflower, linden, honeydew and polyfloral. In the context of colorimetric analysis, it was found that honey acacia had the highest luminosity value (L\*), followed by linden honey, sunflower, polyfloral and honeydew. Acacia and linden honeys proved to be clearer, characterized per values more high in L\*, in comparison with to the too much varieties, while O honey in honeydew revealed itself O more dark, presenting you minorsvalues in L\*. You honeys originating of flowers in sunflower It is linden stood out for the moreelevated purity in color, indicating one intensity or saturation significant from the tone, disregarding The influence from the luminosity. In contrast, the honey in acaciarevealed itself to be less pure in color, suggesting a less intense tone or saturated.In relationship to the components in color, sunflower It is linden Showed O bigger yellow component (b\*). As for the red components (a\*), sunflower and linden registered values high, while linden exhibited one component green more weak.

### **TESTS IN TAMPERING**

The results of adulteration tests on honey samples are presented inTable 5.

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Sample no.	Reaction in Lugol	Reaction in Lugol Reaction in Lund Reaction	
01	Positive	Absent	Positive
02	Negative	Absent	Positive
03	Negative	Present	Negative
04	Negative	Present	Negative
05	Positive	Absent	Positive
06	Positive	Absent	Positive
07	Positive	Absent	Positive
08	Positive	Absent	Positive
09	Positive	Absent	Positive
10	Negative	Present	Negative

Table 5: Results of adulteration tests on honey samples sold us municipalities of Itapira- SP and Mogi guaçu -SP

Source: Authors (2023).

## **Reaction in Lugol**

A reaction in Lugol It is one method qualitative employee for identify The presence of starch or dextrins in honey. After the addition of Lugol's solution, a notable change in color, varying in brown reddish The blue, or same in red-violetThe blue, as described by Périco et al. (2011)

Among the samples analyzed, in relation to the Lugol reaction, as can be seen at Table 5, just to the samples in no. 02, 03.04 It is 10 got O result negative for the Lugol reaction, that is, 60% of the samples analyzed in this study had results positive, showing a high level of adulteration by starch or dextrins in the honeys inquestion.

In one study conducted per Gomes et al. (2017) for to assess The quality of honeysold in western Pará, the Lugol test was performed on honey samples coming from bees of the genus *Melipona* and the species *Apis mellifera*. It was observed that honey samples from bees of the genus *Melipona* presented the highest percentagein adulteration. That he took The conclusion in what that practice he can be related The smallerproductivity of these bees, together with O price in sale more high incomparison with *Apis mellifera* honey.

In research conducted by Felix (2019) in the State of Paraíba, the Lugol he was applied in five samples in honey marketed. Just one of samplespresented one result positive, indicating one level relatively low in adulteration. In search carried out per Bera It is Almeida-Muradian (2007) they were analyzed 11 samples acquired at the business of state in They are Paul It is all to the samples presented a negative result for the Lugol reaction, indicating that the products did not had adulterations with starch or dextrins. The study carried out by Silva et al. (2018) also got result negative for all samples in honeys analyzed, obtained infairs free from the municipality of Assis Chateaubriand/PR.

## **Reaction in Lund**

Among the samples analyzed, in relation to the Lund reaction, as can be seen in Table 5, only three samples showed protein precipitate within the range expected in 0.6 The 3.0 mL, being these to

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the samples in no. 03, 04 It is 10, It is to the too much samples They present an absence of precipitate, and the absence indicates fraud caused by the addition of water or another diluent.

In study accomplished per Santos It is Barbosa (2011), six samples in honeys purchased in street markets and supermarkets in the metropolitan region of Recife, were subjected to the Lund reaction, five samples showed no precipitate, representing O quantitative in 83.3% in results positive for per addition in water oranother diluter. Richter et al. (2011), in their literature, also found samples outside of the standard, having a representation of 10%, two out of 19 samples analyzed from the city from Pelotas/RS. However, Bera and Almeida-Muradian (2007) reported results within the standards expected for pure honey, when carrying out the Lund reaction in 11 samples obtained in commerce of State in They are Paul, with variation in results.

In a survey involving 20 honey samples from the São José do Rio region, Preto/SP, conducted by Garcia-Cruz et al. (1999), it was observed that 35% of the samples presented results negatives for The reaction in Lund, indicating what those honeys they wereconsidered impure. In counterpart, study accomplished per Aguiar et al. (2016)presented results of rehearsal for O test from Lund, highlighting The presence inalbuminoides and, consequently, proving the integrity and purity in all samples in honey analyzed, your search it was founded at realization in analytics physicist-chemicals in honey samples from the stingless bee species, *Trigonaspinipes* and *Tetragonisca angustula*, collected directly from colonies located in the area rural from the municipality of Acrelândia, State of Acre.

## **Reaction in Fiehe**

Among the samples analyzed, in relation to the Fiehe reaction, as can be seen in Table 5, only three samples demonstrated a negative result regarding fraud, these being samples No. 03, 04 and 10, with the remainder having a positive result for Fiehe reaction, that is, 70% of the samples demonstrated adulteration due to the addition of syrups in sugar or overheating honey.

These results are close to those found by Wrobel and Bonfim (2017), which obtained 66.67% of the samples with formation of an intense red color, indicating in the Fiehe reaction indicating positive results for adulterations, the analysishe was carried out in nine samples in honeys in points commercials It is in sellers street vendors various points in the municipality of Castro-PR.

The Fiehe test is recognized for identifying commercial glucose adulterationsor overheating in the honey. The practice of subjecting honey to high temperatures duringextraction no It is recommended due to the potentials damage associates. A elevation in temperature aims to reduce viscosity, facilitating the filtration stage and preventing crystallization, fermentation and the presence of contaminating microorganisms. However, it is It is crucial to highlight that excessive heating is extremely harmful, such a process he can to result at training in products undesirable, as O 5-

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hydroxymethylfurfural(HMF) (TOSI et al., 2002; TURHAN et al., 2007).

HMF is a cyclic aldehyde that is formed by dehydration of fructose in medium acid, the process of which is accelerated by heat (PASSAMANI, 2005), certainly being aof the most common degradation products in honey, indicating its "aging" (SILVA et al., 2008). It is generally practically absent in freshly harvested honeys andits concentration increases over time, being considered an important indicator of quality (SPANO et al., 2009). Levels high in HMF indicate changes provoked per storage prolonged in conditions inappropriate, overheating or tampering (NOZAL et al., 2001).

Braga et al., (2009) obtained 56% failure in the Fiehe test in honeys sold in the city of Uberaba/MG. However, Almeida-Muradia and Bera, (2007) obtained 100% negative results for the Fiehe reaction in 11 samplesacquired in commerce in the State of São Paulo, proving that there was no tampering by addition of syrups sugar or overheating of honey.

Comprehensive analysis of physicochemical and colorimetric parameters reveals a panorama complex from the quality of honey, highlighting variations significant in several aspects. These results highlight the importance of rigorous and to be continued at the sector beekeeping, aiming to guarantee The conformity of the products with to the normative established.

In addition from that, stands out The need in awareness of the producers It is consumers about to the practices suitable in production It is you scratchs associates The adulteration, aiming to secure The quality It is The trust at the Marketplace in honey. A search perMore efficient analysis methods may be a promising direction for identifyingearly detection of adulteration, protecting the reputation of Brazilian honey and preserving the trust of the consumer.

## CONCLUSION

To the analytics physical chemistry of honey revealed variations significant atconformity with you standards cool in between to the samples. A majority answered to the guidelines for sugars reducers, except The sample in no. 02, what it is outside of the Limits for honeyfloral, but within the parameters for melate honey. Samples No. 06, 07, 08 and 09 they are inside of limit in moisture allowed, while others indicate risk infermentation. To the samples in ashes they are inside of limit, but The sucrose apparentget up concerns about The authenticity, being to the samples in no. 02 It is 08 standing outside of the standards for honey in melate It is floral. Exceedances at acidity they can compromise flavorIt is conservation, but to the samples in no. 01, 02, 04, 05, 07 It is 08 they are inside from the legislation, It is variations at the pH suggest possible fermentations or tampering. You solids soluble are inside from the range common expected. A quality It is authenticity of honey dependof these factors, being crucial to meet to the standards for protect The health of the consumers.

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Colorimetric analyzes showed variation in honey color due to factors as O type in bee, processing It is storage. You tests in adulterationrevealed susceptibility in a proportion significant of the samples, with 60% showing adulteration of starch or dextrin in the Lugol reaction (samples no. 02, 03, 04 and 10) and negative results for samples no. 02, 03, 04 and 10 in the Lund, indicating absence of precipitate. For the Fiehe reaction, only three samples had results negatives for adulteration with addition in syrup in sugar or overheating, being samples no. 03, 04 and 10. After thorough evaluation, it was found that no sample met the established parameters, emphasizing the There is an urgent need for more rigorous standards to guarantee the authenticity and integrity of honey. Guidance for producers to understand and control these variables is essential for guarantee a product authentic ending and in high quality.

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