

## MEASUREMENT OF ANTI-STRIPPING AGENT CONTENT IN ASPHALT MIXTURE WITH COLORIMETRIC TEST

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**ABSTRACT:** Stripping can cause premature damage to asphalt pavement. The content of anti-stripping agents in the asphalt mixture needs to control, but the detection and quantification of the content of amine-based anti-stripping agent in asphalt mixture remain a practical problem, therefore research was conducted on how to determine the content of anti-stripping agent contained in the asphalt mixture. Marshall Tests used in this study to evaluate the use of an anti-stripping agent in asphalt mixture through Retained Stability, and Colorimetric Test was held to measure the content of anti-stripping agent contained in asphalt mixture. The Colorimetric Test was developed as a three-step process consisting of amine extraction and trapping through a steam distillation process, sample preparation process and absorbance measurement used spectrophotometer UV/Vis. The anti-stripping agent used in this study was Wetfix-Be with the addition content of 0,2%, 0,3%, 0,4%, and 0,5% of the asphalt weight. The result of the Marshall Tests indicated that the presence of an anti-stripping agent in an asphalt mixture characterized by an increase of the value of the Retained Stability. The addition of 0,4% Wetfix-Be gave the highest increase in Retained Stability, which is equal to 9,25%. The Colorimetric Test result indicated that this test could be used to measure the Wetfix-Be anti-stripping content contained in the asphalt mixture, which in addition to Wetfix-Be anti-stripping 0,4% obtained measured anti-stripping content of 0,365%. This study is critical because the accuracy of the content of anti-stripping agent added to the asphalt mixture will reduce the problem of stripping.

*Keywords: Anti-stripping Agent, Wetfix-Be, Marshall Test, Colorimetric Test, Steam Distillations, Spectrophotometer UV/Vis*

### 1. INTRODUCTION

Moisture damage is often the main factor causing weakening of asphalt pavement in several countries. The most severe impact due to moisture is the loss of adhesion between aggregates and asphalt, which causes a loss of strength and integrity of the pavement structure. This damage is commonly known as stripping, which is a dominant failure due to the separation of the asphalt coating from the aggregate caused by the loss of cohesion of the asphalt cement [1]. One of the efforts made to overcome the occurrence of stripping is by increasing the watertight properties by modifying the asphalt with an anti-stripping agent. General Specifications of Binamarga Division 6.3 have agreed to and require the use of an anti-stripping agent in the asphalt mixture [2].

Just as the asphalt content is the control index in the asphalt mixture design, the anti-stripping agent content is also essential. However, efforts to detect and measure the content of anti-stripping agents in hot asphalt mixtures are still a problem in the field. The most commonly used method for controlling the use of anti-stripping agents is the Indirect Tensile Strength Ratio (ITSR) Test, and the Marshall Retained Stability Test. This test generally requires a long time and is only able to find out the existence

of anti-stripping agents through the measurement of the performance of asphalt mixes without being able to know directly the amount or content of anti-stripping agents contained in the asphalt mixture.

The method of determining the quantity of anti-stripping agent in the asphalt mixture then developed for each type of lime and liquid anti-stripping. Direct measurement of the amount of amine-based anti-stripping in asphalt binder is possible by modifying the ASTM D 2073 Standard Test Method [3]. Furthermore, the development of the Titration Test method can use for asphalt binder and asphalt mixture [4]. However, the analysis with this method is not accurate enough for a tiny amount. For the determination of the amount of liquid anti-stripping agent in asphalt and asphalt mixture, two methods have been identified capable of measuring the content or amount of anti-stripping agent.

The first method is by using a StripScan instrument. The StripScan method involves three main steps, as follows [5]:

1. The asphalt mixture containing an anti-stripping agent is heated so that the anti-stripping agent in it evaporates;
2. The steam then flows through the measurement chamber and will react with litmus paper; this reaction will result in discoloration of the litmus paper;

3. The color of litmus paper was analyzed by a spectrophotometer to measure color changes; significant color changes showed a higher additive.

The second method is the method with spectroscopic analysis through Colorimetric Test. This study will discuss how to measure the content of anti-stripping agents contained in asphalt mixtures using Colorimetric Test.

## **2. LITERATURE REVIEW**

### **2.1 Stripping on Asphalt Pavement**

Stripping is the displacement of asphalt cement films from aggregate surfaces by water caused by conditions under which the aggregate surface is more easily wetted by water than by asphalt [6]. In most cases, stripping starts at the bottom of the asphalt layer and then propagates upwards due to the tensile behavior of the structure and the presence of water above the subgrade [7].

Based on Indian Standards (IS 14982: 2016), stripping value on asphalt containing a maximum of 1.0% anti-stripping agent is required at least 95%, while according to SNI 8139:2015 stripping value for testing using silica aggregate required at least 80% [8].

### **2.2 Anti-stripping Agent**

Anti-stripping material is an additive that may be needed if individual asphalt mixture designs have been proven to be susceptible to moisture damage. Anti-stripping agents made from lime and liquid are the most commonly used types of anti-stripping. Anti-stripping liquid agent is a chemical compound containing amine. Chemical anti-stripping additives divided into two categories: fatty amido-amines and polyamines. Polyamines are mostly used in hot mix asphalt (new or recycled) while fatty amido-amines mostly used in cutback asphalt cement and asphalt emulsions [9].

In the presence of anti-stripping agents, the nature of the aggregate surface changed, and aggregate becomes more lipophilic (oil-loving). Fatty polyamines can form strong chemical bonds even to form permanent chemical bonds with silica. It is what can resist the action of water and significantly increase the adhesion between asphalt and aggregate. Fatty polyamines are a long hydrocarbon chain and an amino group (-NH<sub>2</sub>). The amine group reacts with the surface of the aggregate and hydrocarbons which are hydrophobic in binding to the asphalt. Thus long hydrocarbon chains act as bridges between hydrophilic aggregates and hydrophobic asphalt surfaces to encourage strong bonds between them [8].

The anti-stripping agent used in this study is the type of polyamines with the product name Wetfix-Be. Wetfix-Be is an anti-stripping chemical that contains 100% polyamine condensate tall oil fatty acids with a dosage of use in hot asphalt mixtures of 0.2-0.5% against bitumen content. Wetfix-Be is a liquid additive specifically designed for hot asphalt mixes with excellent heat stability. Wetfix-Be can be stored in hot asphalt for up to 5 days at temperatures up to 170°C without significantly eliminating its effectiveness. Wetfix-Be serves to increase hot-mix attachment to acid aggregates and minimize damage caused by water [10].

### **2.3 Immersion Marshall Test**

To evaluate the use of anti-stripping agents in asphalt mixtures, the Immersion Marshall Test used in this study. The Immersion Marshall Test is a follow-up test from the previous Marshall Test, intending to measuring mixed adhesion resistance to the effect of water and temperature (water sensitivity and temperature susceptibility).

Several ways are used to assess the content of mixed durability, one of them is by looking for Marshall Retained Strength Index (Retained Stability). The Retained Stability analyzed from the stability and flow values of the samples, which divided into two groups: the first group was tested for Marshall Stability by immersion in water at 60°C for 30 minutes and the second group tested after immersion at 60°C for 24 hours. The Immersion Marshall Test uses a Marshall tool, which is a press equipped with a proving ring with a capacity of 22.2 KN (5000 lbs) and flow meter. Proving rings are used to measure the value of stability and flow meters to measure plastic melt or flow, a cylindrical Marshall Test specimen with a diameter of 4 inches and a height of 2.5 inches [11].

### **2.4 Colorimetric Test**

This test is a method of chemical analysis based on the comparison of the color intensity of the solution with the color of the standard solution. This method is part of the photometric analysis. Photometry is a part of optics that learns about the intensity of light (intensity) and the degree of illumination (brightness). Various methods for the spectrophotometric determination of amines in aqueous solution have developed since the 1960s [12]. Silverstein [12] established a spectrophotometric method to measure aqueous concentrations of fatty amines. A spectrophotometer is a tool used in analytical methods that use the principle of energy and physical interactions. A spectrophotometer can be used to determine the concentration of a solution through absorption intensity at a particular wavelength. To determine

the total amine concentration in water, methyl orange was allowed to react with the amines in the presence of an acetate buffer, and the resulting yellow complex extracted into ethylene dichloride. The method was useful for amine analyses in the parts per million (ppm) concentrations range. Larrick [13] improved on Silverstein's spectrophotometric analysis method by combining the buffer and dye reagent into a combination reagent.

Colorimetric Test consists of 3 stages of the process: the process of extracting and capturing amine through steam distillation, the process of sample preparation, and measuring the absorbance of the sample with a UV/Vis spectrophotometer. In this study, the amines water solution was obtained using a steam distillation process. The use of steam distillation method in boosting amine compounds from asphalt mixture based on the physical properties of the Wetfix-Be anti-stripping agent, that is, insoluble in water, higher boiling point and lower vapour pressure than water.

### 3. RESEARCH METHODS

#### 3.1 Samples

The materials to be used in this study, consisted of asphalt mixture such as coarse aggregate, fine aggregate, filler, asphalt Pen 60/70 from Iran asphalt production, Wetfix-Be anti-stripping agent with a content of 0%, 0.2 %, 0.3%, 0.4% and 0.5% of the asphalt weight; and the ingredients for Colorimetric Test such as methyl orange, sodium acetate trihydrate, potassium chloride, glacial acetic acid, ethylene dichloride, and distilled water.

#### 3.2 Design of Specimens

Based on RSNI-M-01-2003, requires a minimum of 3 specimens for the standard Marshall Test per variable. The total specimens obtained for the Marshall Test are 54: consisting of 24 specimens for Marshall Test (before determining the Optimum Asphalt Content), and 30 specimens for Marshall Test (after using the Optimum Asphalt Content and by adding an anti-stripping agent), where each of the 6 specimens made for each addition of the Wetfix-Be anti-stripping (3 specimens for Marshall Standard and 3 specimens for 24-hour immersion). The total specimens for the Colorimetric Test were 25 specimens, which consist of 15 specimens for the standard curve and ten specimens for validation. So the total specimens were 79 specimens.

The aggregate method used is ideal gradations. The aggregate combination of the mixture obtained with the proportion of coarse aggregate (CA) 57%, fine aggregate (FA) 36.5%, and filler 6.5%. In determining the Optimum Asphalt Content of the

plan, the formula is used:  $P_b = 0.035 (\% CA) + 0.045 (\% FA) + 0.18 (\% \text{ filler}) + \text{constant}$ . Used value constant 1 because  $K = 0.5-1.0$  for Asphalt Concrete. The calculation results obtained by the Optimum Asphalt Content content ( $P_b$ ) are 5.8%.

#### 3.3 Marshall Test Phase

Marshall Test consists of 2 phases, phase I: testing the specimens without adding anti-stripping agent by using Marshall Apparatus set, to get the data characteristics of Marshall, which aims to determine the Optimum Asphalt Content; and phase II: testing the specimens by adding Wetfix-Be anti-stripping with variations of content 0%, 0.2%, 0.3, 0.4% and 0.5% of the asphalt weight, using Marshall Apparatus sets. Then, do the Marshall Immersion Test to get Retained Stability values of the specimens after soaking them at 60°C for 24 hours.

#### 3.4 Colorimetric Test Phase

Before doing the Colorimetric Test, the colour reagent is prepared first [14] as follows:

- dissolve 0.1 gram of methyl orange in 100 ml of deionised water,
- dissolve 29.6 grams of sodium acetate trihydrate and 50 grams of potassium chloride in 100 ml of other deionised water,
- combine the two solutions, add 100 ml of glacial acetic acid, then
- dilute 300 ml of all the solutions to be 500 ml with deionised water.

The Colorimetric Test is carried out in 3 phases of a process which consists of the process of extraction or separation of amine from asphalt mixture by using steam distillation, the process of sample preparations, and process of measuring absorbance with a Spectrophotometer. All stages of the process are as follows:

##### 1. The process of extraction:

- Prepared asphalt mixture specimens with the addition of Wetfix-Be, using the same mixed design with Marshall Test specimens. This process consists of: (a) asphalt heating at  $\pm 90^\circ\text{C}$ , (b) addition of Wetfix-Be to the asphalt, (c) aggregate heating at  $\pm 110^\circ\text{C}$ , (d) pouring asphalt + Wetfix-Be into the aggregate, (e) mixing aggregate and asphalt + Wetfix-Be, and (f) inserting the asphalt mixture into a distillation flask and then put in the oven at  $170^\circ\text{C}$  for  $\pm 1$  hour.
- Prepare and ensure a series of distillation devices installed, where a kettle containing  $\frac{3}{4}$  of water from the height of the kettle has installed, and the hot plate has turned on so that the water in the kettle has boiled. Furthermore, the flask contains a mixture of

asphalt which has heated in an oven installed into a series of distillation devices. Then it is left and waited until the amine vapour from the distillation flask flows to a 100 ml distillate container.

2. The process of sample preparation:

- add 5 ml of colour reagent into a 100 ml water from amine vapour extraction and left on for 10 minutes
- add 20 ml of ethylene dichloride and shake for 5 minutes
- let the layer separate for 5 minutes
- extract 1-2 ml of ethylene dichloride (bottom layer) and put it in a vial glass

The whole process is carried out for asphalt mixture specimens with other content of Wetfix-Be.

3. The process of measuring absorbance :

Using a spectrophotometer measured the absorbance of ethylene dichloride samples immediately at 420nm wavelength. Ethylene dichloride extract from samples that do not contain anti-stripping agents used as the baseline absorbance measurement or zero on the spectrophotometer.

Furthermore, a standard curve (regression equation) is made describing the relationship between the content of the addition of an anti-stripping agent and the absorbance value produced by reading a spectrophotometer.

The absorbance value of the validation sample is calculated to the regression equation obtained.

**4. RESULTS AND ANALYSIS**

**4.1 Property Test Results of Material**

The results of the examination of aggregate characteristics provide specific gravity values that meet the requirements, the asphalt coating to aggregate percentage is >95%, and the abrasion value is 22.05%. Regular bitumen tests performed on the base bitumen binder to determine its physical properties. The results presented in Table 1.

**4.2 Marshall Test Results**

*4.2.1 Marshall Test Results to get Optimum Asphalt Content (OAC)*

The results of all Marshall parameters, presented in Table 2, and the OAC value of 5.7% obtained.

*4.2.2 Marshall Test Results with the Addition of Wetfix-Be Anti-stripping (after using the OAC)*

Results calculation of all parameters of Marshall with the addition of Wetfix-Be anti-stripping presented in Table 3.

Table 1 Properties of the Base Bitumen

Test	Standard Method	Specification Limits		Results
		Min	Max	
Penetration (0.1 mm)	SNI 06-2456-1991	60	70	65,2
Softening Point (°C)	SNI 2434:2011	48	-	48
Thin Film Oven Test (TFOT) (% Weight)	SNI 06-2411-1991	-	0,8	0,038
Ductility (cm)	SNI 2432:2011	100	-	> 140
Specific Gravity	SNI 2441:2011	1	-	1,024

Table 2 Marshall Test Results to get OAC

Marshall Parameters	Test Results					Specification
	Asphalt Content (%)					
	5	5,5	6	6,5	7	
Density; t/m <sup>3</sup>	2,293	2,295	2,294	2,289	2,282	-
V I M; %	5,27	4,53	3,87	3,39	3,02	3,0 – 5,0
V I M PRD; %	-	3,25	2,64	2,21	-	> 2,0
V M A; %	15,02	15,41	15,88	16,50	17,20	> 15
V F B; %	64,95	70,63	75,61	79,46	82,47	> 65
Stability; kg	1070	1096	1128	1062	992	> 800
Flow; mm	4,22	3,84	3,74	4,87	5,21	2,0 – 4,0
MQ; kg/mm	254	285	302	218	191	> 250

Table 3 Marshall Test Results with the addition of Wetfix-Be

Marshall Parameters	Test Results					Specification
	Anti-stripping Content (%)					
	0,0	0,2	0,3	0,4	0,5	
Density; t/m <sup>3</sup>	2,292	2,299	2,306	2,306	2,308	-
V I M; %	4,37	4,05	3,78	3,75	3,67	3,0 – 5,0
V M A; %	15,69	15,40	15,16	15,14	15,08	> 15
V F B; %	72,24	74,05	75,16	75,28	75,66	> 65
Stability; kg	1153	986	990	1003	982	> 800
Flow; mm	3,32	3,41	3,40	3,38	3,51	2,0 – 4,0
MQ; kg/mm	347	289	291	297	280	> 250

Marshall Test results obtained that asphalt mixture with the addition of 0%, 0.2%, 0.3%, 0.4% and 0.5% Wetfix-Be anti-stripping all by Marshall parameters requirement determined by Binamarga Specifications 2010 Rev.3. From the test results obtained:

- The addition of Wetfix-Be anti-stripping in asphalt mixture generates a lower stability value than asphalt mixture without the addition of anti-stripping. This decrease may occur because of the primary function of using anti-stripping not to raise stability but rather to minimize the asphalt mixture damage against the influence of water by changing the nature of the aggregate surface so that the aggregate becomes more lipophilic so that strong chemical bonds formed between the asphalt and the aggregate surface.
- The addition of Wetfix-Be anti-stripping in asphalt mixture resulting in an increase inflow value, but will decrease the MQ value. It shows that the addition of anti-stripping will produce a bit more plastic asphalt mixture.
- The addition of the Wetfix-Be anti-stripping in the asphalt mixture causes the density value to increase. It shows that the use of anti-stripping causes the asphalt mixture to be slightly denser than without the use of anti-stripping.
- The addition of Wetfix-Be anti-stripping in asphalt mixture affects the value of the VIM, VMA, and VFB. The higher the content of the addition of anti-stripping will reduce the cavity between aggregates and in the mixture and will increase the cavity containing asphalt. It shows that the addition of anti-stripping causes the

mixture to be more waterproof and air, so that water is difficult to enter the cavities in the mixture and ultimately make the mixture more durable.

4.2.3 Marshall Immersion Test Results

The value of the Retained Stability after soaking for 24 hours presented in Table 4 and the graph showed in Figure 1. From the Marshall Immersion Test results, it seems that there was an increase in the Retained Stability value after the addition of Wetfix-Be anti-stripping to the asphalt mixture. The mixture without the addition of Wetfix-Be anti-stripping results in a Retained Stability value that does not meet the requirements of the Binamarga Specification 2010 Rev.3. The results of this study indicate that the use of anti-stripping in the asphalt mixture will produce a mixture that is more resistant to damage caused by water. However, the increasing use of anti-stripping does not make the mixture better. It indicated by a decrease in the Retained Stability value at the addition of 0.5%.

The addition of 0.4% Wetfix-Be has high effectiveness on stripping compared to other additional content. It is by the research of Hesami, E. and Mehdizadeh, G. (2017) with the same type of anti-stripping, namely polyamine [15] and in following the study of Nazirizad, M., et al. (2015) with the use of the anti-stripping Interline In/400-S [16].

Table 4 Marshall Immersion Test Results with the addition of Wetfix-Be

	Specification	Results Test				
		Anti-stripping Content (%)				
		0,0	0,2	0,3	0,4	0,5
Initial Stability (S1); kg	>800	1153	986	990	1003	982
24-hour Immersion Stability (S2); kg	-	987	905	919	938	914
Retained Stability (S2/S1); kg	>90	85,60	91,78	92,82	93,52	93,07

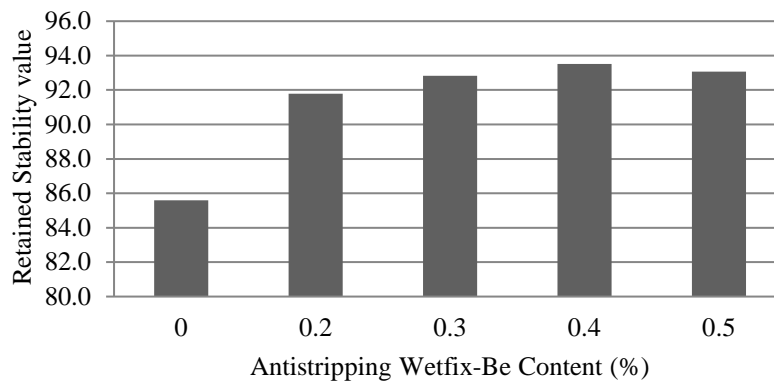


Fig. 1 Correlation between Anti-stripping Wetfix-Be Content and Retained Stability Value

### 4.3 Colorimetric Test Results

Organic anti-stripping absorbance values of extract results from asphalt mixture samples containing 0%, 0.2%, 0.3%, 0.4%, 0.5% Wetfix-Be anti-stripping through the reading of a spectrophotometer at a 420nm wavelength shown in Table 5; and the standard curve in relation to the content of the addition of Wetfix-Be anti-stripping with the absorbance value shown in Figure 2. Based on Table 5, it seems that the absorbance value of the

solution produced by the anti-stripping extract in the asphalt mixture increases with the addition of anti-stripping. It shows that the steam distillation method can extract amine compounds which have been mixed in the asphalt mixture to allow the measurement of anti-stripping in the asphalt mixture through Colorimetric Test. Based on Figure 2, the  $R^2$  value in the calibration curve is  $> 0.97$ . It shows a strong correlation between the absorbance value and the level of addition of Wetfix-Be anti-stripping.

Table 5 Absorbance Value of Asphalt Mixture Contains Wetfix-Be

Anti-stripping Content (%)	Absorbance Values		
	Test I	Test II	Test III
0	0	0	0
0,2	0,0058	0,0080	0,0053
0,3	0,0148	0,0136	0,0181
0,4	0,0264	0,0237	0,0205
0,5	0,0319	0,0289	0,0334

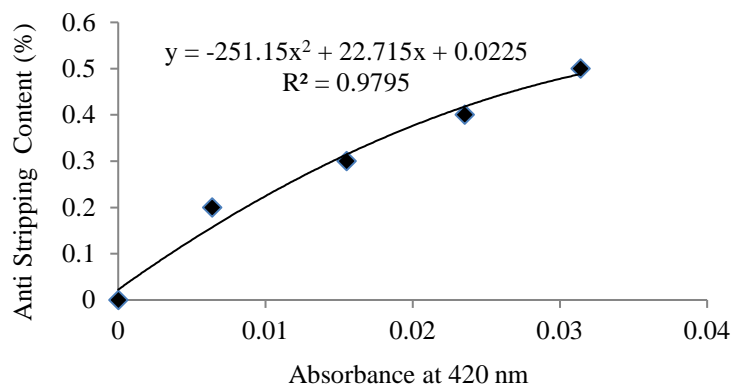


Fig. 2 Standard Curve of Anti-stripping Content vs Absorbance

Ten asphalt mixture samples containing 0.4% Wetfix-Be made, to validate the Colorimetric Test Method in detecting and measuring the anti-stripping content contained in the asphalt mixture. Then measured the absorbance value of the sample and substituted it into the regression equation in Figure 2, namely:  $y = -251.15 x^2 + 22.715 x + 0.0225$ ; where, y is the measured anti-stripping content (%), x is the absorbance value. The measured Wetfix-Be anti-stripping content shown in Table 6. Based on Table 6, the measured Wetfix-Be anti-stripping content is in the range of 0.279-0.396% with an average value of 0.365%, standard deviation of 0.035, and a variation coefficient of 9.482%. These results indicate that the Colorimetric Test performed can be used to measure the content of Wetfix-Be anti-stripping contained in the asphalt mixture. It is following the study of Chen, C. (2007) who also used Colorimetric Tests but the method for extracting amine compounds did not use a distillation process but using a specially designed series of amine traps.

## 5. CONCLUSION

The presence of anti-stripping in the asphalt mixture characterized by an increase in the Retained Stability value. The addition of 0.4% Wetfix-Be anti-stripping content to the asphalt mixture provides the highest increase in Retained Stability value, which is equal to 9.25%. The measured Wetfix-Be anti-stripping content is significantly close to the content of the addition of anti-stripping in the asphalt mixture sample, wherein the addition of 0.4% Wetfix-Be anti-stripping, the measured anti-stripping content was 0.365%. It proved that the Colorimetric Test carried out can be used to measure the contents of Wetfix-Be anti-stripping contained in the asphalt mixture. This study is critical because the accuracy of the content of anti-stripping agent added to the asphalt mixture will reduce the problem of stripping.

Table 6 The Measured Wetfix-Be Anti-stripping Content in Asphalt Mixture

Specimen No.	Absorbance Value	Measured Anti-stripping Content (%)
1.	0,0208	0,386
2.	0,0132	0,279
3.	0,0216	0,396
4.	0,0170	0,336
5.	0,0214	0,394
6.	0,0201	0,378
7.	0,0211	0,390
8.	0,0204	0,381
9.	0,0189	0,362
10.	0,0177	0,346
Mean	0,0192	0,365
Standard Deviation (s)	0,0025	0,035
Coefficient of Variation (cv) (%)	12,976	9,482

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