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Spectrophotometric determination of trace amount of Vanadium in rice and flour using 1-[(4-antipyl azo)] 2-Naphthol as new chromogenic spectrophotometry

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Abstract. The study included follow-up of the interaction of the prepared reagent 1- (4 -Antipyril-Azo) 2- naphthol (APANOL) with vanadium (V) ion, which gave a greater wavelength of absorption (533) nm as well as a study of the best conditions of the size and concentration of the reagent (1×10^{-3}) molar and acidic function (pH = 3) and the stability of the complex for more than two hours and temperature from 10 to 40 degrees Celsius and the linearity range falls within the concentrations (0.10-4.00) ppm and correlation coefficient ($R^2 = 0.9945$) and the value of molar absorption coefficient ϵ (1.02×10^4) l. Mol⁻¹ cm⁻¹ and sandal allergy (0.004) micrograms cm⁻². And determined the accuracy and accuracy of the analytical method followed using standard solutions of ion with a standard concentration of the reagent, so the frequency (RSD %) with respect to concentrations (2.00, 3.00) ppm of vanadium ion (V) equal to (0.9 and 0.9), Erel. %) Was equal to (0.2, 0.3) %

Keywords. 1-(4- antipyril azo) 2-Naphthol, Vanadium (V) determination, spectrophotometry.

1. Introduction

Pyrazolones dye polygonal heterocyclic, that have very momentous purpose in chemistry and biology together owing to the inordinate pliability and assorted structural facets , a vast variety pyrazolon azo have been prepared and their complexation performance examined [1-5]. Furthermore, pyrazoles fond of to a sulphanilamido Rejection Across an azo connection have been acquainted to display biological action [6]. It is recognized that heterocyclic azo components are on a large scale second hand due to of their chelatogenic properties. The strength of pyrazolone-azo is due to its strength on complexity with the effects of mineral ions present in biological arrangements [7-12]. Determination of Vanadium (V) was developed by different ways. These processes require expensive and complicated instruments, consequently, development of Vanadium (V) in different samples was desirable [13-15]. The target of the present labor is to promote rapid, an easy method for the limitation of Vanadium (V). The method is dependent on reaction of 1-[(4-antipyl azo)] 2-Naphthol, with V (V) ion to produce colored complex.

2. Experimental

2.1. Preparation of the reagent (APANOL)



Prepare the detector in two steps: -

The first step: the formation of diazonium salt resulting from the dissolution of 1-Phenyl -2,3-dimethyl 4-amino -pyrazolone - (5) using ethanol alcohol and then adding concentrated HCl acid in the presence of sodium nitrite at a degree (0-5 Co).

The second step: the pairing process that was made between the diazonium salt prepared in the first step with an alcoholic base solution of (2-Naphthol) taking into account the degree cooling (C5-0) to complete the pairing process to obtain the organic APANOL reagent whose solution color is reddish orange. As shown in the following equations:

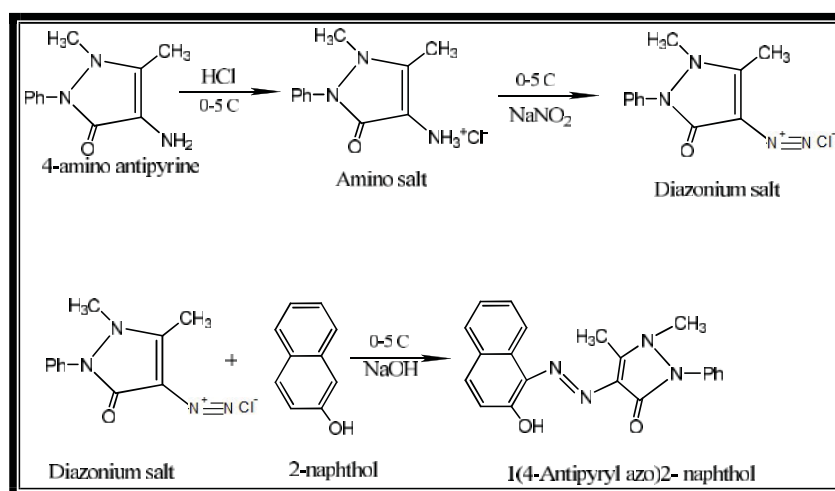


Figure 1. Preparation of reagent (APANOL)

2.2. Preparation of Vanadium (V) complex

The component of V(V) was synthesized by equivalent measurement quantity from reagent in 100 ml of deionized water then affix teardrop sage with flipping to an equal amount 1:2 for V(V) in 100 ml deionized water. The complex thus created off washed with $\text{CH}_3\text{CH}_2\text{OH}$ and desiccated.

2.2.1. Apparatus

Spectrophotometric measurement using UV – visible T80double beam spectrophotometer using glass cells of 1,00 cm. On the test scan Shimadzu FT.IR 8000 sequence, vibrational spectra were registered. PH measurements were performed using Inolab pH – meter WTW 720equipped with a glass – combined saturated calomel electrode Melting points of both ligand and complex were obtained with an electrothermal melting point apparatus 9300. The conductivity was calculated with an Inolab conductivity model -WTW 720 in DMSO (10^{-3}) solution.

2.2.2. Reagents

All chemicals used were of analytical grades.

Vanadium (V) stock solution ($100 \mu\text{g} \cdot \text{ml}^{-1}$)

Prepare by dissolving 0.0459g of ammonium vanadate with hot distilled water in a beaker capacity (5.00) ml , add .002 ml of concentrated nitric acid and transfer the solution quantitatively to a volumetric volume of (200) ml , complete the volume to the mark with distilled water, and from this Standard solution Other standard solutions were prepared by successive dilution with distilled water.

1-(4antipyryl azo) 2- Naphyhol (1 mM)

Foreign ion solutions ($10 \mu\text{g} \cdot \text{ml}^{-1}$)

These solutions were prepared by dissolved an amount of the compound in deionized water sequel the size in a volumetric flask.

2.3. General Procedure

A 10 ml bottle of volumetric flask are taken and each volume of the standard solution of the Vanadium (V) is set at 10 ppm. Each vial of 3 ml of the 1mM reagent solution is modified. The acidic function of the solution is adjusted to 3.00 and the complex volume is computed to 10 ml. The absorption of the solutions formed at length wave form for maximum absorption against the reagent solution under the same conditions except the Vanadium (V).

3. Results and Discussion

Properties of (APANOL) and its metal chelate

APANOL is a shared by or involving three parties, with coordination of carbonyl group, hydroxyl group and azo group nitrogen; it has the Back construction:

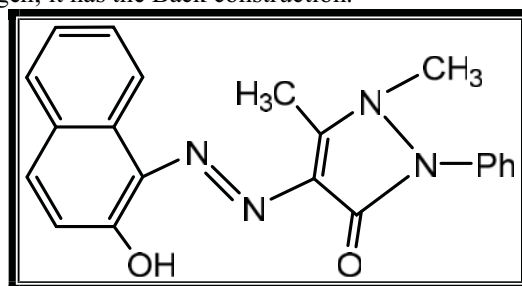


Figure 2. Structure of APANOL

Because of the large joint system, the component shows stellar ability to form an internal metallic chelate. APANOL and its metallic chelating can be dissolved (in aqueous solution) easily.

3.1. Spectra

The consequence of this realization particular which the reactions of V(V) with 1(4 –pyrazolon azo) 2-Naphthol Produces extremely soluble colored compounds that can be used as a convenient challenge test of V(V). The electronic absorption of APANOL and its complex in deionized water was registered in wavelength (190 – 800) nm. Figure 3 shows the absorption spectrum of APANOL (1×10^{-3} M) dissolved in deionized water versus deionized water as a blank analysis , This colored reagent has an extreme a absorption at 494 nm , while the colored complex has a Abs(max) at 533 nm for V(V) ,the blank at this wavelength offer zero absorbance Figure 4.

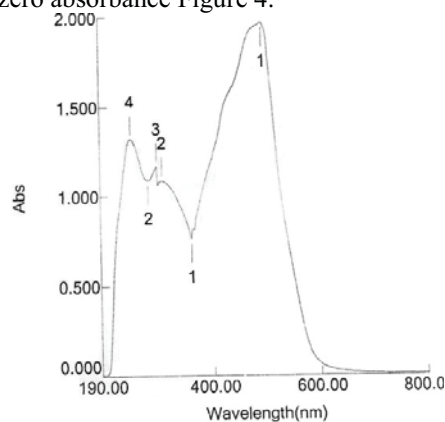


Figure 3. Absorption spectra of the reagent [APANOL]

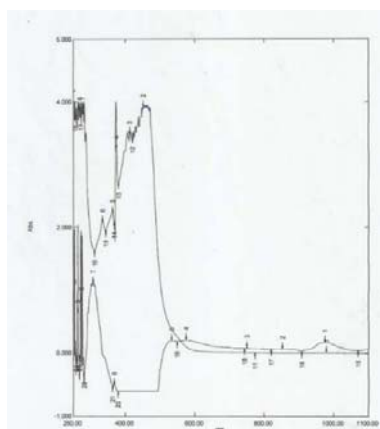


Figure 4. Absorption spectra of [V(V) + APANOL]

The influence of different parameters on the absorption strength of the problem produce was studied and the reaction status were improved.

3.2. Effect of pH

To determine the fastness of the complex of the reaction of the Vanadium ion (V) with the reagent, the minimal of the blank, and the rate of reaction velocity, studied the effect of pH from (1.00 - 10.00). Only pH 3.0 was locate to be finest for Vanadium (V). HCl and NaOH outcome in low sensitivity and was not steady for V(V). A small amount of HCl was added at a concentration of 0.05 molar and the same concentration of NaOH was used to study the effect of pH change on the electron absorption spectrum of the complex.

The absorption spectrum of the complex with the ion under study gave a concentrated package at 533 nm. The bands show in the domain of 248-306 nm for V(V) is assign to $\pi \rightarrow \pi^*$ transmission. The another band spotted in the district of 533 nm is assign to $n \rightarrow \pi^*$ electronic transmission [17-19] for V(V) Figure 5, Table 1.

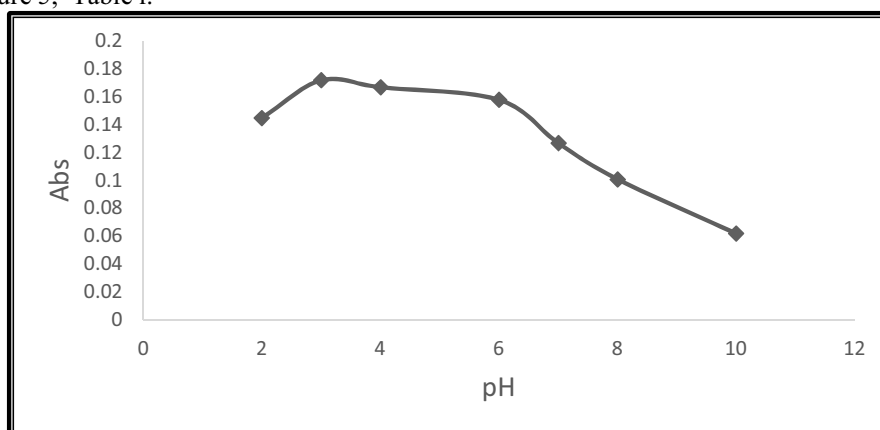


Figure 5. Effect of pH on complex V-APANOL

Table 1. Analytical characteristics of Co-complex

Characteristic	V(V) – complex
Absorption maximum (nm)	533

Beer's law range (ppm)	(0.1– 4.0)
pH range	(3.0)
Sandell's sensitivity $\mu\text{g. cm}^{-2}$	0.0049
Molar absorptivity ($\text{L. mol}^{-1} \text{ cm}^{-1}$)	1.02×10^4
Melting point for reagent	(235) C 0
Melting point for V (V) – complex	(240.5) C 0

3.3. Impact of Reagent's concentration

Study the effect of the reagent size on the formation of the complex which shows the absorption values of the complex increased to a peak at 3.00 ml of reagent solution at a concentration of (1×10^{-3}) molar with one milliliter of vanadium ion solution (V) at a concentration of (10.00) ppm to give the best color intensity, and then the absorption values begin to decrease when the detector volume increases.

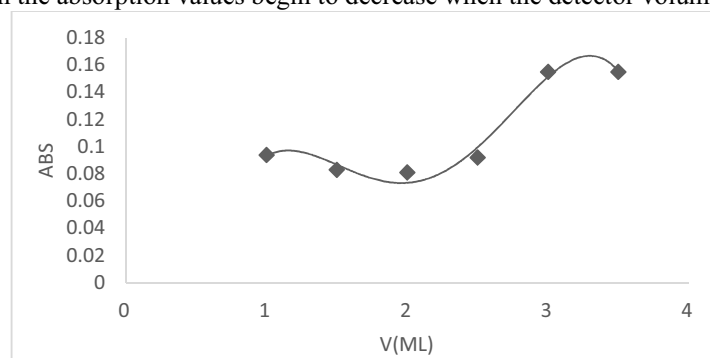


Figure 6. Effect of APANOL concentration

3.4. Impact of reaction time

For the purpose of studying the effect of time on the stability of the prepared complex, the absorption values of the vanadium complex (V) with the reagent (APANOL) were look at the wavelength (533) nm as a function of time under the best conditions for the formation of the complex, through the results shown in Figure 7. The complex formed with high stability and stability can reach (2) hours, and this indicates the advantages of the reagent in estimating vanadium (V) spectrally.

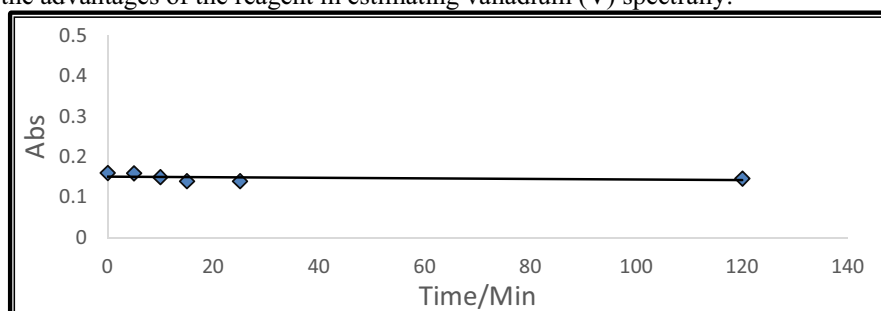


Figure 7. Effect of time on complex of V[APANOL]

3.5. Impact of temperature

To show the effect of temperature on absorption of the complex, a change in the absorption of the complex at different temperatures was observed between (10-50) °C and Figure 8 shows the stability of the complex and the possibility of studying it in the temperature between (10-40) °C. Increase of temperature over these ranges we note that absorption of the complex will gradually decrease due to the fragmentation of the complex and its disintegration in temperatures higher than (40) °C.

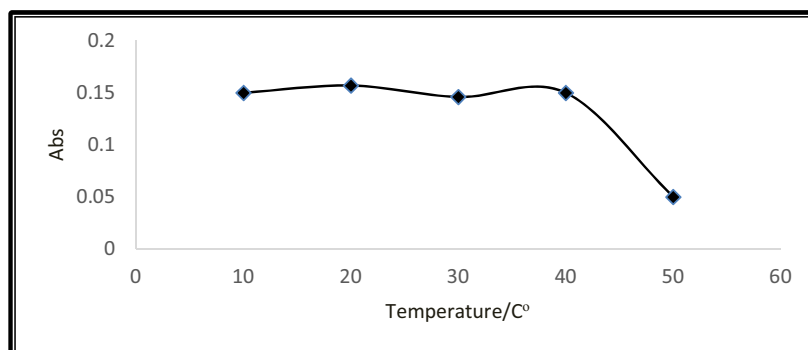


Figure 8. Effect of temperatures on complex V[APANOL]

3.6. Order of addendum of reagents

The order of addendum of the substances was followed to give the best results and only observed that there was a loss in the stability of the complex.

3.7. Calibration graph

The standard calibration curve for vanadium ion (V) with reagent (APANOL) shows that the vanadium complex (V) obeys the Lambert-Bear law for a range of concentrations ranging from (0.1-4.00) ppm under optimum conditions at wavelength (533) nanometers. Correlation coefficient ($R^2 = 0.9945$) with a detection limit of (0.054) parts per million, while the value of molar absorption coefficient (ϵ) was equal to (1.02×10^4) liters. $\text{Mol}^{-1} \cdot \text{cm}^{-1}$. The colored component is steady for 2 hrs.

3.8. Composition of the component

Two methods were adopted to determine the ratio of the metal ion to the reagent: the method of molar ratios and the method of continuous changes and by applying the best conditions for the formation of vanadium complex (V) with the reagent (APANOL) at ($\text{max}\lambda = 533 \text{ nm}$), as observed from Figure (7) Which represents the method of molar ratios and the Figure (8) which represents the method of continuous changes that the ratio of vanadium ion (V) to the reagent (APANOL) is (1: 2) at the best acidic function of the complex formation.

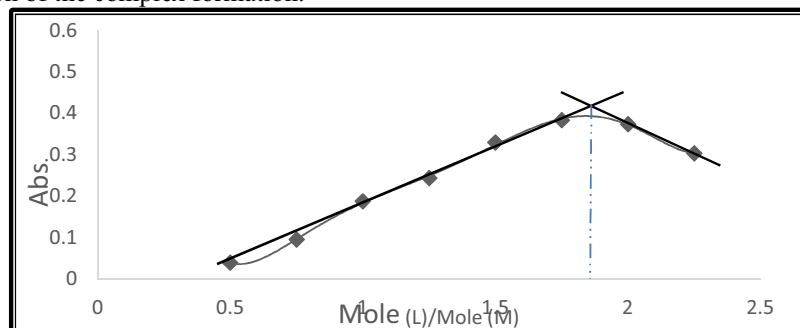


Figure 9. Mole-ratio method for V[APAOL] complex

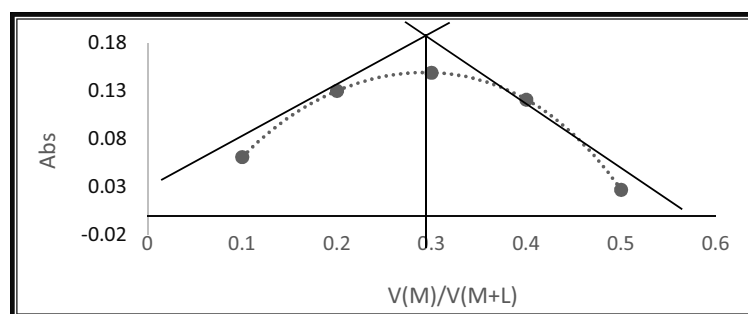


Figure 10. Job's method for V[APANOL] complex

3.9. Interferences

The aim of this study is to demonstrate the effect of the interference of a group of positive and negative ions on the absorption of vanadium complex (V) and its spectral estimate according to the best conditions obtained during this study, as a group of positive two and three ions were selected, The results are shown in the table, It shows that the presence of some positive and negative ions during the formation process of the complex resulting from the interaction of vanadium ion (V) with the reagent (APANOL) affects a different effect on its absorption value and this depends on the nature of the interfering ion and its concentration.

Table 2. Impact of foreign ions

Seq.	Ion	Conc.µg/ml	E%
1	Zn+2	100	-54.11
2	Co+2	100	-394.7
3	Ni+2	100	-125.5
4	S2O8-2	100	-53.4
5	I-	100	-65.6
6	Br-	100	-31.3
7	Cl-	100	-26.1
8	Pt+2	100	-83.1
9	Cu+2	100	-217.4
10	Hg+2	100	4.6
11	Pd+2	100	-260.4
12	Cl-	100	-26.1
13	SO4-2	100	-65.1

3.10. FT.IR of reagent and it's complex

The release ligand FT.IR and its metal chelate were attained in the range (500-4000) cm⁻¹ Figures 11, 12. The IR bands of the congregation (APANOL) and its V(V) with their probability are given in Table 3. The ligand IR spectrum provides a wide band at 3433 cm⁻¹, which the OH group can be assigned to. Due to the non-polar aspect of the bond found at 1456 cm⁻¹. This band is moved to a low density scale at 1404 cm⁻¹ frequency value Based on the complexation Leads to chelation using (M-N) [22]. The ligand's IR spectrum showed a sharp band at 1635cm⁻¹ attributable to pyrazole azo γ (C = O). On complexation this band disappears [23, 24]. The bonding of oxygen to the metal ion is Submitted at 520 cm⁻¹ on behalf of the occurrence of the band as a consequence of γ (M-O) [25, 26].

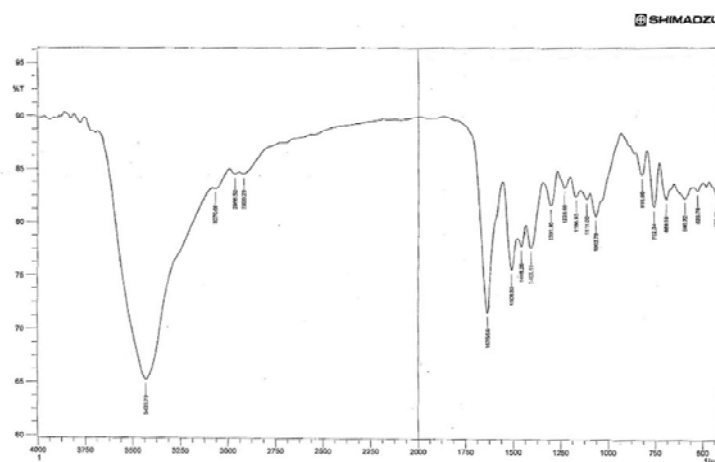


Figure 11. FT-IR of reagent (APANOL)

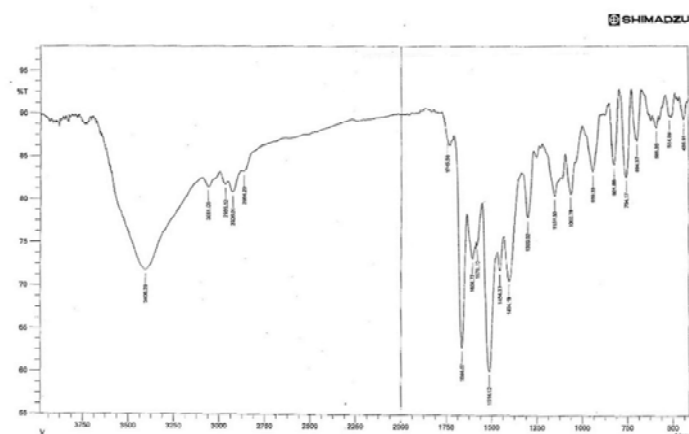


Figure 12. FT-IR of component (APANOL) with V (V)

Table 3. Chosen FT-IR data of (APANOL) and it's complex with V (V)

Compound	$\nu(\text{OH})$	$\nu(\text{C-H})$	$\nu(\text{N=N})$	$\nu(\text{C=O})$	$\nu(\text{M-O})$	$\nu(\text{M-N})$
HL	3433	arom.	1456 m	1635 s	-	-
[V(APANOL)]	3406	3070	1404 m	1664	520 w	422 w

S: sharp ; m: medium ; w: weak

4. Applications

4.1. Definition of Vanadium (V) in practical samples

Vanadium ions were estimated in laboratory-prepared models for sample preparation for reagent analysis (APANOL). The results that we obtained for the mentioned ions were estimated by (methods spectrophotometer), and therefore the determination of vanadium ions (V) in this way is highly sensitive and can be used to estimate these ions in various models for ease of completion and speed as well as accuracy.

Table 4. Determination of V (V) in synthetic samples

Quantity taken of V(V) p.p.m	Recovery%	*RSD%
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1.0	99.35	0.810
2.0	99.56	1.026

*For five determinations

4.2. Definition of Vanadium (V) in Rice and Flour samples

Put 1 g from sample in a hot cuvette, then heat them to high temperature (about 800 ° C) for 3.0 hou, and after the sample is turned into black ash, dissolve it in little of HNO₃, then heat it until drying, then the mixture must be filtered. The filtered is taken, Put it in volumetric flask (25 ml) and complete the volume by deionized water.

Table 5. Determination of V(V) in *Rise and Flour* sample

Sample.	Con. of sample	*R.S.D%	Re. %
Rise	µg / g	0.86	99.46
Flour	0.915	0.45	99.38s

*For five determination-

5. Conclusion

From the above results, its can be concluded that APANOL is a potential reagent for the determination of Vanadium (V). The reagent is simple to preparation using available chemicals. Moreover, this work simple, rapid and sensitive for the determination of Vanadium (V) without the need of heating or extraction.

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