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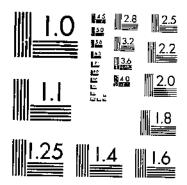
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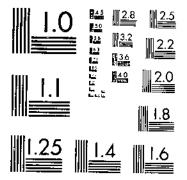
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USDA TECHNICAL BULLETINS
TON OF AMERICAN GUM SPIRITS OF TURPEN
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UNITED STATES DEPARTMENT OF AGRICULTURE WASHINGTON, D. C.

THE FRACTIONATION OF AMERICAN **GUM** TURPENTINE AND EVALUATION OF PINENE CONTENT BY OPTICAL MEANS

By S. Palkin

Senior Chemist, Industrial Furm Products Division, Chemical and Technological Research, Bureau of Chemistry and Soils 1

CONTENTS

	Page	l P	age
Introduction Methods of separating \(\alpha \) and \(\beta \) pinene_ Equipment Procedure	2	Results	11

INTRODUCTION

American gum spirits of turpentine, which is obtained almost entirely from the species Pinus palustris (longleaf) and P. caribaea (slash pine) (22), like that from the French P. maritima is composed very largely of the two isomers α-pinene and nopinene or β-pinene (12, p. 128; 14). The quantity and proportion of these components are of considerable industrial importance in the synthesis of camphor and to some extent in the manufacture of terpin hydrate and α -terpineol (8, 9, 10, 11).

The gradual transition in the southeastern pine belt from the old stands of longleaf to the new growth of slash pine has raised the question as to the possible effect of this on the fundamental character of American turpentine, some influence having already been noted in

the increasingly negative rotation.

Examination of turpentine from slash and longleaf pine (one sample of each) by Dupont and Barraud (14) showed that turpentine from the two sources varies considerably in the proportions of the two isomeric terpenes. Although the authors do not attach any importance to this difference, yet if it is found to hold consistently and if the pronounced decrease in β -pinene content is indicative of a definite change in composition, a change due to the transition, these facts would be of significance in view of the superior reactivity of β -pinene (10, 11) over α -pinene, especially in certain processes (1, for synthetic camphor.

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Drawings of the apparatus were made by R. M. Baker, of the Chemical Engineering Division.

2 Italic numbers in parentheses refer to Literature Cited, p. 12.

The object of the investigation reported in this bulletin was to examine the available means for obtaining fundamental quantitative data on the composition, particularly the major components, of the predominant American gum spirits of turpentine and to obtain data on the composition of turpentines from the well-known sources.

METHODS OF SEPARATING α AND β PINENE

With the possible exception of ozonization (4, 13), no chemical methods for differentiating the two isomers α and β pinene have been found to be quantitative. The well-known classic reactions, for example, oxidation to pinonic and nopinic acids respectively, are productive of low yields and are unsuitable even for approximately quantitative purposes.

Complete quantitative separation of the two from one another and from the tailings by fractional distillation or other physical means, although theoretically possible, would in view of the similarity of

physical properties be impracticable.

Darmois (9, 10) has shown that by means of an effective fractionating column French gum spirits (also American) is resolved, with the exception of a very small quantity at the beginning ("head") and end ("tailings") of the distillation, into the isomers α and β pinene, and mixtures of the two in varying proportions, and that their respective proportions can then be ascertained by the Biot relationship (12, p. 98). By plotting rotatory dispersions Darmois proved that the mixtures contain only the two components. Dupont and Desalbres (15) have found by experimenting with highly purified α -pinenes that inactive pinene tends to concentrate in the top fractions. Chavannes (7), however, by careful fractionation of 30 kilograms of French turpentine has isolated low-boiling unsaturated hydrocarbons.

Dupont applies the same principle for the determination of the composition of the tailings, etc., in terms of β -pinene and nonpinenes, by obtaining the necessary optical data on the dominant "tailings" constituent through fractionation of the end portion in a small

column.

With the aid of a special bubbling column (9, 10) (2 meters long with 35-wire gauze plates), Dupont and his associates, applying the Darmois method, have made a comprehensive study of French tur-

pentine and of others of different origin (2, 12).

To provide the data essential for the Darmois method and procure a distillation picture of diagnostic value, an effective means for fractional distillation of the turpentine is of prime importance. Furthermore, the relatively high boiling points of the components, their proximity, and their susceptibility to isomerization and decomposition render it necessary to carry out the fractionation at reduced pressure.

EQUIPMENT

The assembly is shown in Figure 1. The following fractionating columns were tried in this work:

A gauze-plate bubbling column described by Dupont (12), of 30 plates, approximately 190 cm. long, and 2.5 cm. in outside diameter.

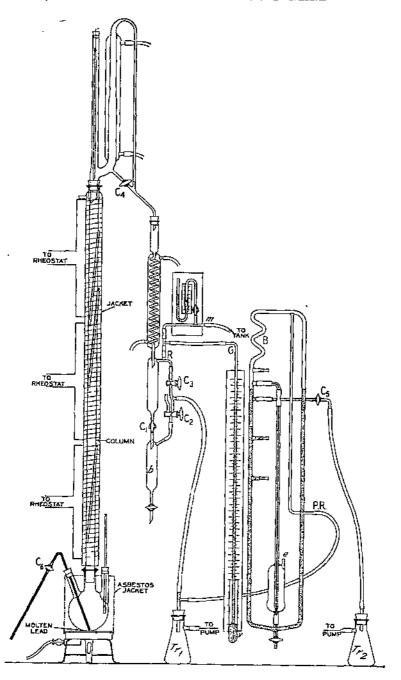


FIG. I

Figure 1.—Assembly for fractionation of turpentine in vacuum, showing column, electrically heated jacket, pressure regulator, dephlegmator-condenser, and other accessory apparatus

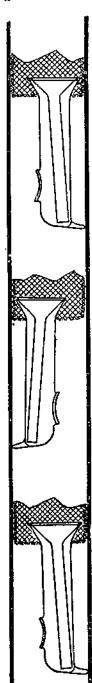


Figure 2.—Improved gauze plate rectifying column, showing special trap tubes

A bubbling cap-plate column devised by Bruun (5), of 15 plates, and approximately 125 cm. long and 2 cm. in outside diameter. This was found to be an effective fractionating device.

A packed column filled with short pieces of glass tubing described by Peters and Baker (20). This column, used only in some of the earlier experiments, was not found so effective as the bubbling-

plate type.

Improved gauze-plate bubbling columns developed in this division (19), one of 15 plates, about 110 cm. long, and one of 32 plates, about 210 cm. long, both 2.5 cm. in outside diameter. (Fig. 2.) These were found to be more resistant to flooding and to function more smoothly in vacuum fractionation than the other columns.

Thermal insulation for the column was provided by an electrically heated jacket, rheostat controlled (20). The superior efficiency of lagged over unlagged columns has been shown by Leslie (16) and

Marshall and Sutherland (18).

· A large reservoir, serving to take up any slight leaks, aided materially in maintaining continuous and smooth operation during the taking of a fraction when the pump was cut off from the column.

Constant pressure in the system was maintained by a Wade and Merriman regulator 3 (24) with modifications to prevent loss of mercury (fig. 1, B), and a trapping arrangement (fig. 1, C₅—Tr₂) made it possible to clean the apparatus (by treatment with

dilute acid) without disturbing it.

The dephlegmator-condenser (fig. 1) used with the gauze-plate column is, with slight modification, the form described by Marshall (17). Division of the product and reflux in these was controlled by stopcock C₄, and the dimensions of the columns were such as to insure ample reflux without interference by ascending vapors.

The Bruun column was provided with an automatic reflux regulator, the reflux ratio being about

Operation of the system, as shown in the diagram of the assembly (fig. 1) is as follows: Connection with the pump is made through an appropriate trapping vessel (Tr₁), which in turn is connected with the pressure regulator at P. R. It is connected with a large reservoir (an old steel oxygen cylinder has been found very serviceable), also with a manometer to indicate pressure in the system (with pump on or off), and to a mercury gauge, G, which also serves as a safety valve at V.

² This has recently been replaced by a mercury regulator and relay.

During distillation all parts of the apparatus are kept in communication with the pump and the pressure-regulating system and reservoir, through the stopcocks. When taking a fraction, compartment b is closed to the rest of the apparatus by stopcock C_1 and is brought to atmospheric pressure through a 2-way stopcock, C_2 , the pump remaining in communication with the rest of the system. When the fraction has been taken, communication of column to pump is closed by stopcock C_3 , and compartment b is evacuated by opening cock C_2 to the pump (the proper pressure point is automatically determined by the pressure regulator), and the whole system is again brought into intercommunication by opening cocks C_3 and C_1 .

PROCEDURE

Fractionations were made with the apparatus shown in Figure 1 at constant pressures, some at 20 mm., others at 65 mm. Operations at constant pressure added greatly to the convenience and smoothness of the distillations and facilitated making sharp cuts. Approach to the pure components was indicated readily by the distilla-

tion temperatures.

The fractionation of each sample was divided into two steps. All but the last 50 to 100 gm. of sample was distilled in the large apparatus (fig. 1); the remainder, comprising the tailings and some β -pinene, was transferred to a small unit to complete the fractionation. In this the column proper was similar to the Vigreux column (23), with thermal insulation, pressure regulator, and other accessory apparatus the same as that described for the large column.

Rotation measurements were made in the green ($V=546 \text{ m}\mu$) and yellow ($J=578 \text{ m}\mu$), and occasionally also in the yellow ($D=589 \text{ m}\mu$), with a Schmidt and Hänsch instrument equipped with a monochromator. The small quantities of various components in the highboiling fractions are given as tailings. No attempt was made in this

investigation to determine its several constituents.

The quantitative data were determined by the Darmois-Dupont optical method on the basis of the Biot relationship (12, p. 98; 9, 10):

 $X_1 = \frac{(\alpha)_0 - (\alpha)_2}{(\alpha)_1 - (\alpha)_2}$

for any given fraction containing the two terpenes in which-

 $(X)_i$ = the proportion of α -pinene,

 $(\alpha)_{\alpha}$ = observed rotation in that fraction.

(α)₁=rotation of the pure α-pinene (determined through isolation of this constituent by careful refractionation of the top fractions and occasionally further checked by the plotting method of Darmois).

 $(\alpha)_2$ =rotation of the β-pinene, which, when obtained from turpentine, regardless of origin (12), is found to have a constant value, $\alpha_1 = -19.62^{\circ}$ (10-cm. tube); specific rotation, $(\alpha)_1 = -22.44^{\circ}$.

Beyond the point of pure β -pinene in the series of fractions, the proportions of β -pinene and the nonpinene constituent or tailings were determined from the relationship

$$X_{2} = \frac{(\alpha)^{3} - (\alpha)^{3}}{(\alpha)^{3} - (\alpha)^{3}}$$

where $(\alpha)_a$ =rotation of the dominant tailings constituent, X_a =proportion of β -pinene having the rotation $(\alpha)_a$, the observed rotation of the fraction being $(\alpha)_a$.

of the fraction being $(\alpha)_0$. Strictly speaking, specific rotations should be used. As density differences involved are negligible, however, in practice actual rota-

tions are used (9).

As pointed out by Dupont, slight errors in the latter calculations (12, p. 121) are introduced, as the rotation of the dominant tailings constituent is not obtained with great accuracy, probably owing to the presence of more than two components in the last few fractions. These fractions, however, are too small to affect the general accuracy of the determinations.

RESULTS

Analytical data on the various turpentine samples examined are given in Table 1. Samples 2, 8, 9, 10, and 11 were obtained from authentic oleoresins by steam distillation in the usual manner.

Table 1.—Analytical data on turpentine samples from commercial and authentic sources

	Rotation (1	0-em. tube)	Refrac- tive index $\binom{n^{25}}{D}$	Den- Sity (d ^{15.6°})	Composition		
Source and sample No.	αj=578mμ,	a,=5:i6mμ,			a-pinene	β-pinene	Tail- lags
Commercial from mixed fields of slash and longleaf pine: G-7. G-15. G-16. B-1. Samples from authentic sources: Slash pine 3, old. Longleaf 1, fresh Longleaf pine 2, fresh Gum dip 8, slash Gum dip 9, longleaf. Scrape 10, slash Scrape 11, longleaf.	+12.20 -22.58 +11.0	-2. 2 -9. 98 +2. 5 -12. 58 -22. 56 +16. 27 +14. 34 -24. 9 -27. 8 +8. 85	1. 4697 1. 4692 1. 4680 1. 4701 (1. 4698 1. 4663 1. 4691 1. 4692 1. 4690 1. 4700 1. 4700 1. 4681	0. 8707 8062 8661 8692 8667 8624 8680 8680 8687 8657 8657 8657	Per cent 63.0 61.9 62.2 59.2 \$ 58.0 62.5 61.0 62.5 58.8 67.0	Per cent 31. 5 32. 6 28. 8 36. 9 35. 6 28. 6 33. 3 33. 7 34. 4 33. 6 29. 2	Per ccn. 5.3 6.1 3.9 6.4 9.5 3.3 1.7.6 6.3.8

Longleaf pine No. 1 represents a commercial turpentine obtained from an area in Mississippi known to be exclusively longleaf.

Slash pine No. 3 represents a sample from an authentic source

which had been in storage for about four years.

Samples G-7, G-15, and G-16 were obtained from commercial turpentines which had been kept in storage in tanks for several years (approximately closed from the atmosphere).

Sample B-1 represents a commercial turpentine obtained from

mixed longleaf and slash gum.

Typical detailed fractionations of a sample each of slash and long-leaf pine are given in Tables 2 and 3. Data on rotations, rotatory dispersions, refractive indices, and densities of the fractions, plotted against weight, are shown in Figures 3, 4, 5, and 6. Normal inflections are noticeable with the change from the relatively pure α -pinene

⁴Prepared by W. C. Smith, of this division, from oleoresins made available by the Forest Products Laboratory in connection with another investigation.

to the mixtures with β -pinene, and from these to the β -pinene. Influence of the tailings on the properties of the β -pinene is shown by the sharp inflections just beyond the β -pinene points of the respective curves.

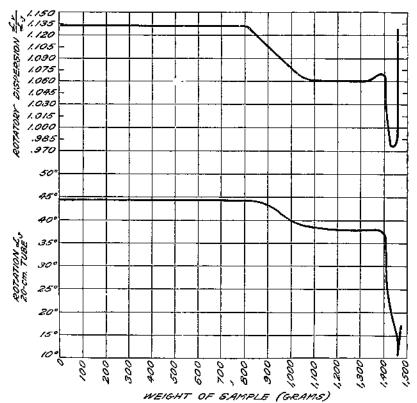


Figure 3.—Rotations and rotatory dispersions of fractions from slash-pine turpentine

A break in the continuity of rotatory dispersions of longleaf pine turpentine occurs at the point (fraction 10) of change from dextro α -pinene to levo β -pinene and is not indicative of a new component as shown by the continuity of the rotations, refractive indices, and densities in the corresponding regions.

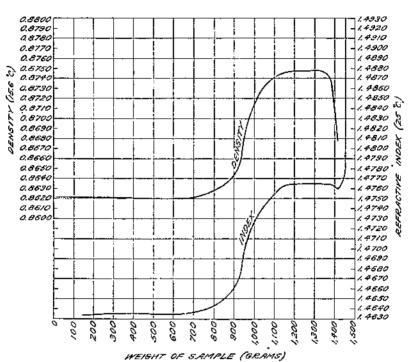


Figure 4.—Densities and refractive indices of fractions from slash-pine turpentine

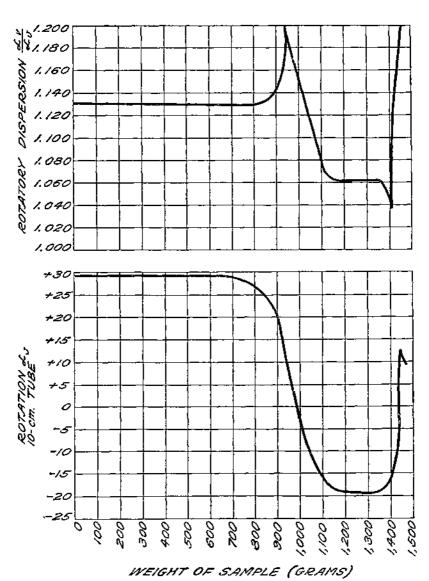


FIGURE 5.—Rotations and rotatory dispersions of fractions from longlent-plue turpentine

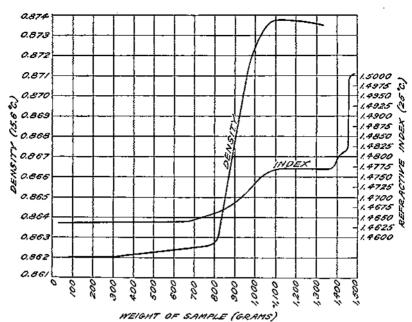


FIGURE 6.—The fractionation of American gum spirits of turpentine and evaluation of its pinene content by optical means

Table 2.—Fractional distillation of 1,500 grams of turpentine (3, stash pine, old, Table 1) and properties of fractions

[Original turpentine:
$$a_1 = -40.1$$
 (20-cm. tube) $n_D^{25} = 1.4698$ $a_V = -45.12$ (20-cm. tube) $d^{15.6} = 0.8667$]

Fraction No.	Boiling tempern-	Weight	Rotation (20-cm. tube)		Disper-	Refrac- tive index	Density
2120000110	ture at 20 mm.	** cight	α ₁ =578mμ	α,≃546mμ	$\binom{\alpha_T}{\alpha_j}$	$\binom{n^25^\circ}{D}$	(d15,0°)
2 	° C. 0 51. 0 51. 0 51. 0 51. 4 51. 2 51. 5 51. 8 58. 1 58. 2	Grams 33 45 53 87 119 97 137 90 150 160 80 85	- 44, 45 - 44, 46 - 44, 7 - 45, 10 - 46, 2 - 45, 15 - 45, 15	-50, 17 -50, 15 -50, 45 -50, 45 -50, 9 -51, 95 -50, 95 -40, 95 -41, 17 -41, 17 -41, 18	1. 129 1, 130 1, 130 1, 129 1, 129 1, 120 1, 120 1, 128 1, 102 1, 078 1, 002 1, 063	1. 4633 1. 4635 1. 4635 1. 4635 1. 4635 1. 4634 1. 4634 1. 4637 1. 4847 1. 4736 1. 4736	0, 8020 8620 8620 8624 8628 8628 8679 8722 8737 8733
4 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5	58. 2 58. 4 68. 0 68-60. 0 67. 8 69. 2 93. 8 **91. 0 **120	86 78 61 20 20 19 12 11 6 11	-37.75 -37.55 -38.3 -29.8 -19.9 -16.5 -10.70 -17.10 -50.0	-40.00 -30.8 -31.1 -31.3 -19.9 -15.8 -10.5 -19.3 -50.0	1. 000 1. 061 1. 073 1. 050 1. 00 . 976 . 981 1, 129	1, 4766 1, 4705 1, 4772 1, 4814 1, 4816 1, 4817 1, 4830 1, 5005	. 8737 . 8730 . 8582
		337	-44. 4 -38. i	-50, 25 -40, 5	1. 132 1. 0604	1.4635 1.4768	. 8622 . 8735

¹ Composition as calculated from data in table: α-plaene, 58.0 per cent; β-pinene, 35.0 per cent; tailings 6.4 per cent. Rotation of a-pinene: ap=-45.2° (20-cm. tube).
70 grams, representing remainder of the sample, was transferred to small apparatus and fractionaled, giving fractions 18 to 22.

At 12 mm.

TABLE 3 .- Fractional distillation of 1,500 grams of turpentine 1 (longleaf 2, Table 1) and properties of fractions

 $n_D^{25^\circ} = 1.4692$ Original turpentine: $\alpha_1 = +12.29$ (10-cm. tube) $d^{15.6^{\circ}} = 0.8694$] $\alpha_{2} = +14.34$ (10-cm. tube)

	Boiling tempera-		Rotation (1	0-cra. tabo)	Disper-	Refrac-	Density
Fraction No.	ture at 20 mm.	Weight	αj=578 tnμ	α _τ ≂510πμ	$\binom{\alpha_{\tau}}{\alpha_{J}}$	(n ^{25°} D)	(d ^{15.6})
	• С.	Grams					
	5t. 0	112	+29, 12	- -32. 78	1.128	1, 4634	į
	51.2	103	+29. 54	-\-33, 20 -\-33, 15	1, 126	1.4634	i i
	51.4	90	+29, 44 +29, 39	+33, 18	1, 126 1, 129	1, 4634 1, 4634	0.8622
	51.6 51.6	102 102	+29. 40 +29. 40	+33, 17	1, 123	1.4634	!!
		102	+29, 98	¥32, 72	1, 120	1, 4635	
		102	+28.51	-1-32, 17	1, 128	1, 4636	8620
		85	+26.71	-1-30, 24	1, 130	1, 4642	. 8629
		90	+21.07	+24, 94	1.135	1,4656	, 8637
0		52	+8, 54	+10, 24	1, 199	1, 4691	. 8674
1	57. 2	98	-10.10	-10, 30	1.010	1.4740	. 8728
2		89	-18.66	-19.81	1.062	1, 4761	ii
3	58.4	29	-19, 34	20, 50	1,060	1.4763	8747
4	[58.6	76	-19.31	-20.46	1.060	1, 4763	
.5		51	-19,03	20, 24	1,003	1. 4761	Į)
B 2	59. 5	45	-14.71	-15.26	1.037	1, 4758	
<u>7</u>	76.0	24	-9.41	-9.58	1.018	1.4754	
8	101.0	10	+12, 49	+14.39	1. 152 1. 177	1, 4824 1, 4955	
<u>9</u>	³ 60-71	8	+12.38	+14,58 +9,88	1. 219	1. 4935	
Residue		9 21	+8.10	TV. 88	1.219	1.4000	
tesique			+29, 22	+33,065	1, 131	1.4635	. \$622
2 to 15 (combined)		324	-19.08	-20, 225	1. 000	1. 4760	8747

¹ Composition as calculated from data in table: α-pinene, 62.5 per cent; β-pinene, 33.3 per cent; tallings, 4.2 per cent. Rotation of α-pinene: α; = +30.5° (10-cm. tube).

72 grams, representing remainder of sample, was transferred to small apparatus.

The distillation temperatures for the fractions representing respectively the a and β pinene components are in fair agreement with the temperatures for these compounds corresponding to a vapor pressure of 20 mm. as reported by Pickett and Peterson (21).

Reasonable agreement in the other properties of the a and β pinene fractions with those of the pure compounds reported in the literature was generally obtained by the one fractionation. The refractive indices for the a-pinene components, although in agreement with those reported by several authors, were all somewhat higher than those reported by Dupont and Desalbres (15) for the pure compounds, but several refractionations showed no appreciable change. Rotatory dispersion is of particular diagnostic significance. The values given for the two terpenes are: α -pinene=1.133; β -pinene=1.0615 (1 $\hat{\delta}$).

SUMMARY

An assembly is described, comprising plate rectifying column, pressure regulator, and other accessory apparatus, which has been found to function very smoothly and satisfactorily for the fractionation of turpentine in vacuum.

By distilling at fixed pressures, distillation temperatures become reasonably accurate reference points in the approach to the pure components, and closer correlation of distillation temperatures with com-

position of the respective fractions becomes possible.

Data relating to the composition of a number of turpentines with respect to the a and \$\beta\$ pinene and tailings are given, their proportions

having been ascertained optically by the method of Darmois and

Dupont.

No great variation was noted in the total of the two components, a and β pinene. Although material differences were noted in their proportions, such differences do not seem to be ascribable to the variety of pine (longleaf or slash) or to the dip or scrape form of either.

As regards the tailings, however, as well as may be judged from the limited number of samples examined, a generally higher content is indicated in the slash-pine turpentine than in the longleaf, and as may be expected, the old turpentine contains more than the fresh turpentine. In view of the influence of the tailings components on yields in industrial syntheses, this observation, if found to be true generally, would have a greater significance than would at first be apparent.

There is apparently no indication, however, that the increase of slash pine as a source of American turpentine will tend to diminish

its β -pinene content.

The increase of turpentine of negative rotation with the increasing use of slash pine is doubtless due to the negatively rotating character of the α -pinene in the latter and not to any material change in the proportion of α and β pinene.

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14

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