from Fick's law of diffusion applied to this case where x_d is comparable in magnitude to r.

The zinc wires used dissolve 2 to 2.5 times as fast, per unit area, as flat disks 1.2 cm. in diameter, having only one face exposed to the solution at the same distance from the stirrer. They also dissolve 20 to 25 per cent faster than metal cylinders of the same diameter as the stirrer and rotating at the same speed.

Nomenclature

- A = surface area of wire, sq. cm. (in Table III, average of initial and final values)
- $C_{\text{reagent}}, M = \text{concn. of reagent, gram moles/liter (unless otherwise})$ noted)
- specific heat, cal./gram, ° C. c =
- Ď diffusivity of reagent, sq. cm./sec. = Ğ
- -
- rate of mass flow, grams/sq. cm. sec. heat transfer coefficient, cal./sq. cm. sec. ° C. h == =
- jChilton-Colburn heat and mass transfer factors, dimensionless \boldsymbol{k}
- dissolution rate coefficient, cm./sec.
 thermal conductivity, cal./cm. sec. ° C. K

- radius of wire, cm. (in Table III, average of initial and final values)
- turbulent motion normal to interface
- effective film thickness in dissolution process x_d -
- x_h effective film thickness in heat transfer constant with dimensions of 1/sec. -
- α ۵
- density, grams/cc. viscosity, poises ц

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Controlling Gin Flavor

TROM a technical point of view, the beverage alcohol or distilling industry in the United States has been stagnant for at least thirty years. During the years of prohibition the industry in the United States was legally dead and, for fifteen years prior to that time, large inventories, tradition, and fear of governmental interference and prohibition were not conducive to research and development.

During this thirty-year interval other industries experienced marked advancement along all technical lines. Aca-



MECHANICAL CONTROLLING AND RECORDING EQUIPMENT ON A GIN STILL

H. F. WILLKIE, C. S. BORUFF, AND DARRELL ALTHAUSEN Hiram Walker and Sons, Inc., Peoria, Ill.

Gin production in the past has been characterized by lack of control over many of the important variables such as quality of spirits, quantity and quality of flavor in the various botanicals used, variable types and methods of operating the stills, etc. Critical study of these variables disclosed valuable information which led to standardization of spirits and operations which, with proper selection of botanicals and regulation of the quantity of each ingredient used in the formula in accordance with its flavor value, now permits the production of gin under technical control which guarantees uniformity and quality of final product.

demic and pure research findings had been adopted by the majority of industries, and a technic of industrial research had been developed that had been accepted by practically all plants. This acceptance led to new products, modernization, improvement in quality and uniformity, and greater profits to the management. The liquor industry, which possesses a heritage that dates back beyond recorded history, has always shunned research and development, accepting only those advances that were imperative, such as the classical work of Pasteur, the development of the Coffey still, and a few others. The industry and the consuming public were again robbed of the benefits of industrial, chemical, and biological advances of the age by re-establishing in most plants the old generation of distillers in power following repeal. The industry as a

JANUARY, 1937

whole is a paradise for a technical man, but the present management and nontechnical-minded operating staffs found in most plants are seriously objecting to the introduction of present-day modern methods of production and control. Since repeal, certain new plants have been built as exact replicas of distilleries of thirty to fifty years ago and are operating without the services of one qualified chemist.

In contrast to these general conditions, with the advent of repeal a few distilleries adopted modern production methods and employed control chemists and chemical engineers; one or two established functioning research departments and placed technical men in operating, control, and administrative positions. It has been the policy of one of these organizations to build upon its 103 years of continuous experience in the distilling industry, to investigate from a modern technical point of view all traditional manufacturing methods and equipment, to replace them, if necessary,

with modern methods, and also to remove the black coat of secrecy and empiricism from this business. All engineering, biological, and chemical phases of the operations are under technical control; all have been and are still being studied.

These statements are not intended to be disparaging to others in the distillery business but rather as "food for thought" and encouragement to those who desire to do similar work for this industry, but who, so far, have not been able to convince their management of its merits. The industry as a whole has been inoculated with modern "technical mindedness," and many a chemist awaits with interest the end of the incubation period.

Periods in Gin Production

The results of studies on gin production will be discussed, and the manner in which certain medicinal, physiological, chemical, organoleptic, and engineering features enter into its production will be demonstrated.

The production of gin may be said to have passed chronologically through three periods. First came the period of the "coctum," the "philter," and the "pot-stills," dating from the age of its origin in the seventeenth century (26) to the advent of the column still (7). During these years some of the gins must have been fiery potions as a typical formula will show (16):

Corn spirits at proof, gallons	80
Oil of turpentine, pint	1
Culinary salt, pounds	7-8
Water, gallons	30 - 40
Distill to 100 gallons	

Occasionally "oil of vitrol" (15) was used in place of the salt when a really "hard liquor" was desired. Not even the venerable distillers of Schiedam, Holland, would have described this concoction as a food product.

The second period runs from the beginning of the day of rectification to the present time. Throughout these decades the value of "clean" ethyl alcohol was appreciated and essence de genièvre was abandoned in favor of redistillation of ethyl alcohol in contact with or through botanicals. That is, formulas specified a given number of pounds of juniper berries, coriander seeds, cardamom, etc., and, in addition, highly rectified ethyl alcohol (15, 26). This period was also the heyday of secret formulas. Texts, the trade, and the press continue



Adding Botanicals to a Gin Still

to display the following type of announcement: "This same renowned secret formula has been in use in our family for decades and has been handed down from generation to generation." An illustrative formula of this period might read as noted in Table II.

The third period which is proposed for adoption by the food and beverage industry is one in which chemistry and chemical engineering take the place of traditional, secretive, and ruleof-thumb methods. The writers propose an era of flavorcontrolled gins. Producing flavor-controlled gins orients itself around three primary considerations, all technically regulated—namely, the use of (a) ethyl alcohol of definite specifications, (b) a standardized botanical charge, and (c) mechanically controlled distillation.

Ethyl Alcohol of Definite Specifications

Today most distillers of gin pay particular attention to the type and kind of alcohol used. The majority of American distillers are using a grain mash as the source of their alcohol for gin. According to Herstein and Gregory (15), objection exists in Europe to the use of molasses alcohol due to its harsh top notes, but apparently this is not so true in America although we must admit that there is some prejudice against the use of anything except grain alcohol. Recently, the question has been raised as to whether synthetic alcohol, now illegal for beverage purposes, might serve as a source of alcohol for gin, etc. The presence or absence of alleged toxic constituents (4, 18, 19, 20, 25) in synthetic ethyl alcohol is still a debated question. It is to be regretted that an editorial (17) and a reliable article appearing recently on this subject (4) carried no claims relative to the therapeutic action of commercial grades of synthetic ethyl alcohol, but reported only upon c. P., or laboratory quality, which they concluded to be comparable in physiological effect to potable grain spirits. Alcohol from grapes is as yet far too expensive to be considered in America for the manufacture of gin.

Alcohol for this purpose must be free from all but traces of odoriferous impurities which occur in minute quantities in the commercial product and owe their source to the raw materials from which they are manufactured (8). Potato alcohol, even though highly rectified, gives a different bouquet from rice alcohol. Likewise, the other commercial grades of alcohol can be identified best by their characteristic bouquet.

The tools for use in the liquor industry are sadly lacking for any specific determination for these impurities; consequently, the indices developed for identifying "pure spirits" are still somewhat relative. It is largely a matter of good taste rather than intelligence that permits the discrimination between these various spirits. However, when the specifimade of it. In addition to the evaluation of the quantity of oil present, the character or flavor type of the botanical must be controlled by means of specifications. After selection and purchase of the botanicals, they should be warehoused under regulated humidity and temperature conditions to preserve the desired flavorings.



METHOD (STEAM DISTILLATION)

cations given in Table I are required, a high grade of "Cologne spirits" suitable for a mild gin, is obtained.

The methods employed in all these determinations except the permanganate time and bouquet are found in the official methods of the A. O. A. C. (2). That for the permanganate time is given by Allen (1), and the technic of bouquet analysis is a tradition of the industry.

A comparative analysis of commercial samples of alcohol from different sources (Table I) indicates how closely grain and molasses alcohols approximate the proposed standards.

Standardized Botanical Charge

The assay and regulation of the weights of the various ingredients used in accordance with their flavor value is an innovation in the field of liquor manufacture. Whereas in the past all gin formulas read only in terms of pounds of each ingredient per unit quantity of alcohol (Table II), the research department of Hiram Walker & Sons, Inc., and associated companies developed a method of flavor assay and now select their botanical ingredients and modify the poundage formulas in accordance with the flavor value of each ingredient as noted in Table III. Disclosed in this tabulation is the main factor in flavor control. Since the essential oils and associated volatile constituents of the crude drugs communicate the intensity of flavor and character to the gin, in spite of the opinion of one well-known gin manufacturer to the contrary (3), the addition of a regulated quantity of each botanical so as to give the same amount of oil in each batch will help to duplicate the flavors, provided the same type of botanical (same character of oil) is always used. The correct weight of botanical to be used in the charge (column C, Table III) is calculated by dividing the weight of that particular oil noted in the standardized formula (A) by the percentage of oil noted in the botanical (B) and multiplying by 100.

Botanicals from identical sources vary in oil and flavor concentration from year to year. Likewise, they vary in type, quality, and quantity of flavor in accordance with geographical source. Flavor value also changes with uncon-ditioned storage. Thus it is necessary to standardize the herb, berry, or root, as the case may be, before any use is

Since juniper oil is the chief flavoring constituent of all gins, the requirements of their assay will be discussed in detail, dismissing all other botanicals with the summary statement that they are likewise rigorously examined.

It is believed that many of the specifications here disclosed are unique and not to be found in the literature by virtue of the fact that data are ordinarily gathered from freshly harvested products and are not the same as those noted for seasoned (aged) botanicals. Caraway seed, for example, as harvested and distilled in Holland, is reported to yield around 6 per cent oil (12). When what is reported as legitimate Dutch caraway seed arrives in America, weeks distant from the day of gathering, distillers find about 2.5 per cent oil. We may attribute this difference to shipment of "drawn" seeds or to volatilization of part of the oil by aging. At any rate, the recipient of the aged seeds never realizes the same value as was present when the seeds were harvested. This same situation applies to other botanicals (11, 13). Thus a modern gin manufacturer should assay all of his crude drugs.

Juniper Berries

The following specifications for juniper berries have been developed and found satisfactory by the writers for preparing a uniform high-grade potable gin:

1. Ripe berries of the Juniperus communis from the Chianti region of the Apennine Mountains of immediate harvest past; maximum age. one year.

2. Not more than a total of 4 per cent unripe stock, oxycedrus berries, damaged goods, dead berries, and other adulterants shall be present.

The moisture content shall not be less than 7 per cent by weight as evaluated by the Dean-tube method at 20° C. on a sample previously dried in a desiccator for 16 hours over 66° B6. sulfuric acid. The total moisture as determined by the loss in

the desiccator and the Dean tube shall not exceed 14 per cent. 4. The acid number shall be 13 to 17 when determined on the fruit as calculated on a 7 per cent moisture basis and no more than 3 when determined on the volatile oil steam distilled therefrom.

The per cent volatile oil shall not be more than 1.6 or 5. less than 0.9 when determined by the Hiram Walker method.
6. The per cent volatile oil shall be 0.9 to 1.2 when determined

by the Clevenger method.

7. Average size of the berries shall be 8 to 10 mm., and the color shall be steel blue.

Average number of berries per gram shall be 8 to 10.

9. Specific gravity of the oil shall be 0.8650 to 0.8792 at $20^{\circ}/20^{\circ}$ C.

10. Refractive index of the oil at 20° C. shall be 1.4840 to 1.4870.

One part of oil shall be completely soluble in 5 parts or 11. less of 90 per cent alcohol. 12. The unpurified natural oil is to possess "juniperol"

bouquet, not a terebinthinate or turpentine odor. 13. A distillate of an alcoholic infusion of the juniper berries

shall possess a type of flavor which checks with standards held in the laboratory files. 14. Juniper berries, when combined with the remaining ingredients called for by the formula and when added in accordance with the oil value established for each ingredient, must give a gin that corresponds with standards held in the laboratory files.

The authors wish to point out that they do not propose these constants as the last word in assay. Experience may show the necessity of modifying some of them and likewise better means of evaluating all. They are serving their purpose very well in standardization and may act as sign posts along the way for others to secure better botanicals.

Source and Age of Juniper

Through the assistance of others, twenty-five different sources of juniper berries (24) have been brought under this assay. They differed as to type and original geographical origin. Some of the samples were dismissed because of unreliable markets, limited supply, or toxic nature (23). After selection of the types that would bear investigation for flavor values, 10 kg. of each of five geographic kinds were obtained and have been studied week after week for a period of over one year. This study is being continued. When these berries arrived in the United States (not more than 6 months after harvest) the surface moisture, volatile fraction

TABLE I. SPECIFICATIONS FOR AND ANALYSIS OF COMMERCIAL GRADES OF ALCOHOL Specifica- tions — Analysis of Commercial Alcohol—					
Source Proof at 60° F. (15.6° C.) Bouquet	190–192 Neutral	Grain 192.0 Neutral	Molasses 190.0 Neutral	Ethylene 190.5 Objection- able	Wine 183.5 Fruity
KMnO₄ time, min.	60 or greater	65	60	40	20
	G	rams per	100 Liters	at 100 Pro	of
Aldehyde as AcH Esters as Et acetate Acids as acetic Fusel oil as amyl alcohol Extract	<2.0 <7.0 <1.2 <7.0 <0.7	$1.2 \\ 3.7 \\ 0.6 \\ 6.0 \\ 0.5$	1.52.60.99.00.8	2.0 3.2 1.4 4.7 0.6	$5.7 \\ 45.0 \\ 0.9 \\ 18.6 \\ 1.0$

TABLE II. STANDARD TYPE OF GIN FORMULA®

(In pounds pe	r charge of 30	00 wine gallons at 100 pro	of)
Ingredient	Pounds	Ingredient	Pounds
Juniper berries Coriander seed Cinnamon bark	100 50 10	Angelica root Lemon peel Cardamom	10 1 1 1
^a This formula is use sent a true potable gin		lustrative purpose and doo ormula.	es not repre-

TABLE III.	FLAVOR-CONTROLLED GIN FORMULA ⁴
/1	

	(in pounds	per charge	01 3000	wine galions	s at 100 l	oroqi)	
					\boldsymbol{A}	В	С
					Lb. of	Volatile	Caled.
					Volatile	Oil in	Lb. of
					Oil in	Botani-	Ingredient
Code		_	-	Date of	Standard	cal,	per
No.	$\mathbf{Ingredient}^{a}$	Type	Source	Purchase	Charge	%	Charge
520	Juniper berries	Italian	W-1	5/4/36	1.0	1.2	83.3
521	Coriander seed	Moroccan	K-1	6/10/36	0.5	1.0	50.0
522	Cinnamon bark	Ceylon	K-1	7/1/36	0.15	1.5	10.0
523	Angelica root	Saxonian	C-3	8/15/35	0.15	1.0	15.0
524	Lemon peel	Sicilian	C-3	6/16/36	0.001	0.2	0.5
525	Cardamom	Mysore	C-3	7/7/36	0.02	2.0	1.0
	formula is used pu	rely for illus	trativ e pu	rpose and do	oes not rep	resent a t	rue potable

gin production formula.

at 110°C., acid number of the fruit, per cent oil, nonvolatile fraction in the oil at 110° C., Dean-tube moisture, gravity, and refractive index of the oil were measured, and a pilot still distillation was made of each type of berry. Polarimetric measurements were not made because of the large quantity of oil required for this study and the limited amount of samples available. The junipers studied included the following:

Variety of Juniper ^a	Locality of Origin
Tyrolean Yugoslavian No. 4 Yugoslavian No. 5 Italian Czechoslovakian	Assling Valley at 1300 meters Region of Maribor Region of Maribor Tuscany Carpathian Mountains
^a All berries of October, 1934, harve	est.

In addition to these data, nearly five hundred samples have been analyzed during the last two years under their reported definitions of variety and source. These include Russian, Czechoslovakian, Swedish, German, French, Italian, Yugoslavian, and Austrian juniper berries. The Italian berries from the Chianti region gave the best flavor. Those Yugoslavian berries raised along the forty-third parallel of latitude or near Zara are nearly as good. If a sharp-flavored gin is desired, German juniper should be used. Gin producers may prefer different types of finished product; hence many differ not only as to the formula but also as to the source and desired age of the juniper. Some distillers prefer to use juniper berries two or three years old. Later the writers will show (in connection with the oil content) specific reasons why they have no interest in two-, three-, or fouryear-old berries. A year-old berry is one that has matured one year after the date of harvest. Granted that geographical source and age have much to do with the type, quality, and quantity of flavor found in juniper berries, it becomes necessary for each gin producer to have definite source and age specifications for his berries if he is to produce a uniform product.

Adulterants

These studies have shown that such specifications are necessary because of the flagrant adulteration so prevalent in botanicals shipped into the United States and subsequently dispensed to the commercial trade. Juniper may be found on the market today with as high as 40 per cent of unripe fruit, oxycedrus, or other contaminants that are harmful to good flavors.

Another reason for care in eliminating the unripe or adulterating berries is shown by the effect of various quantities of these materials on certain physical constants:

	25%Adulteration	4% Adulteration
Austrian berries: Per cent oil Refractive index Acid No.	$1.80 \\ 1.4817 \\ 21.27$	$1.20 \\ 1.4780 \\ 16.65$
Italian berries, acid No.	38% Adulteration 19.26	4% Adulteration 13.89

Since we wish to rely upon a certain range in chemical and physical factors for flavor control, obviously these constants must be fixed. Unripe and oxycedrus berries are a beautiful rich brown, whereas the communis berry is gun metal or midnight blue. Hand-selected berries are chosen now by some discriminating distillers.

Moisture Content

Obviously, unless the distiller is to consider water as a variable in his scheme of assay, he must select berries of the same range of moisture



FIGURE 2. CONTROLLED CONDENSATION GIN EQUIPMENT

content, both as regards surface moisture (water lost on drying in a desiccator) and Dean-tube moisture. Very dry berries are synonymous with very old berries.

Popov and Pashkevich (21) claim that the maximum total moisture content of freshly harvested juniper berries should not be over 16 per cent and that 12 per cent is about normal. Several types of juniper berries were received with a maximum age of 8 and a minimum of 6 months. These berries were warehoused under uncontrolled conditions for 12 months, and the moisture content was measured weekly. The data obtained indicate that humidity, storage temperature, and the surface texture of the berry affect moisture values. The maxima and minima data for the several types examined are as follows:

~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~		ight ^a
6 mo.	12 mo.	18 mo.
8.6	6.6	4.5
9.3	6.5	4.1
8.8	7.2	5.6
10.1	6.4	4.7
10.1	8.2	4.5
	6 mo. 8.6 9.3 8.8 10.1	8.6 6.6 9.3 6.5 8.8 7.2 10.1 6.4

^a Moisture content by Dean-tube method after drying 16 hours over  $66^{\circ}$  Bé,  $H_1SO_4$  to eliminate surface moisture.

Numerous analyses and data collected on 100,000 pounds of plant stock conclusively demonstrate that a minimum moisture content of 7 per cent is justifiable for an Italian berry not more than 12 months old.

The above figures and Figure 1 indicate loss in moisture noted in berries stored in an unconditioned warehouse for various lengths of time. Temperature- and humidity-controlled warehousing should reduce loss in moisture and retard oil and flavor losses. There is also a practical side of this question—namely, that the drying of the berries with the attendant loss in oil (Figure 1) is a total loss in investment at the rate of the cost of the berries, which runs from 5 to 9 cents per pound. This loss may amount to 20 to 33 per cent for undried berries held 52 weeks in unconditioned warehouses:

Type	Moisture, %	Weight, %
Tyrolean	12.98	29.4
Czechoslovakian	15.14	21.9
Italian	15.11	20.0
Yugoslavian No. 4	17.51	32.7
Yugoslavian No. 5	14.00	19.5

. .....

Economical and flavor control data, therefore, justify the fixing of specification 3.

#### Acid Number

Naturally when the distiller seeks a beverage that is mild and bland in taste he wishes to eliminate those tones responsible for the rasping top notes in bouquet and the astringent character. This is best done by selection of berries or herbs low in terpenic constituents and low in acid number. A low acid number is not always the entire story. Along the forty-third and forty-fourth parallels of latitude in Yugoslavia and Italy grow very good junipers of the communis variety. Northward near the forty-sixth parallel to the Assling Valley, Tyrolean berries are found. Still farther north, the German and far to the west in the tradition-infested Carpathian Mountains, the Roumanian, Polish, and Czechoslovakian berries are found. The Tyrolean berries are easily differentiated from these others because of their high acid number:

$\mathbf{Type}$	Age		id Number ^a Maximum	Av.
Tyrolean Czechoslovakian Italian Yugoslavian Nos. 4 and 5 German American Russian	6-18 mo. 6-18 mo. 6-18 mo. 6-18 mo. 1-3 yr. Fresh 1-3 yr.	$17.6 \\ 14.8 \\ 14.9 \\ 13.5 \\ 10.0 \\ 6.0 \\ 2.0$	$19.0 \\ 16.3 \\ 16.7 \\ 15.8 \\ 13.0 \\ 8.0 \\ 4.0$	$     18.3 \\     15.5 \\     15.8 \\     14.7 \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     \\     . \\     . \\    $

^a Calculated to 7.0% moisture basis.

However, acid number is not the entire answer to good flavor; for, although Czechoslovakian berries lie within the range of the Yugoslavian and Italian, and data from a number of shipments indicate that German berries give acid numbers ranging from 10 to 13 (average near 12), American 6 to 8, and Russian even as low as 2 to 4, the flavor of these berries is much less sweet than that of the Italian. Obviously, then, the botanical to be used cannot be selected by one single specification; for example, berries of the juniper variety raised much above the forty-fifth parallel, even though high or low in acid number, are terebinthinate and pinene-like. Moreover, in southern France along the desirable forty-third and forty-fourth parallels where the thurifera grows, these products are reported as toxic (22). The inclusion of the acid number of the oil is for the purpose of satisfying U.S.P. requirements (acid number less than 3).

Data indicate that the acid number of the fruit may be considered for all practical purposes to remain unchanged during one year of storage, but that of the volatile oil increases. All acid numbers were determined according to the U. S. P. definition—milligrams of potassium hydroxide needed to neutralize 1 gram of berries. Selection is made from a composite sample of one hundred bags. One-fourth pound (113 grams) is dried over concentrated sulfuric acid and ground, and 2 grams are selected for analysis. This sample is covered with 25 cc. of 190-proof alcohol, allowed to infuse 16 hours, and filtered, and the residue is washed with 25 cc. of alcohol. The alcoholic washings are titrated with 0.05 N potassium hydroxide using phenolphthalein as an indicator.

Allowing for a reduction to 7 per cent moisture, the acid number limits have been fixed at 13 to 17 in order to include both Italian and Yugoslavian and to exclude Tyrolean, German, and American berries. Some distillers would like to shift these figures in order to include German berries.

#### Volatile Oil

From oil analyses (Clevenger method,  $\delta$ ) on old and new stocks of berries held in warehouses and the reported data on the distillation of fresh berries in Europe, an aging curve has been constructed. This is shown as a "best straight line" in Figure 1. This stock had an initial average total moisture content of 14 per cent which decreased to 5 during 48 months of unconditioned storage. Likewise, the oil content decreased from 1.3 per cent on a dry basis to 0.25.

The observed oil values on a number of types of juniper over a 52-week storage period (from 6 to 18 months in age) are as follows:

		-Per Cent Oil-	
Type	Maxima	Minima	Variation
Tyrolean	2,45	1.17	1.28
Czechoslovakian	1.58	0.96	0.62
Italian	1.83	1.55	0.28
Yugoslavian No. 4	1.58	1.18	0.40
Yugoslavian No. 5	1.75	1.04	0.71

These values were determined by a method employed in the Hiram Walker laboratories. This technic is, briefly, as follows: The method involves extracting the crude drug by ether and evaporating the solvent at  $47^{\circ}$  C. That portion of the residue volatile at 110° C. and by steam is calculated as essential oil. There was less loss of oil from the Italian than any of the other berries. These oil data also indicate the error in using a definite poundage of a botanical for a unit quantity of alcohol. For example, 100 pounds of Tyrolean berries might have charged 2.45 pounds of oil or 1.17 pounds of oil, depending upon the age of berry (6 or 18 months), weather, temperatures, and possibly storage conditions. The weight of botanicals used for gin charges must be regulated as shown in Table III.

Oil values on fresh berries from various sources are as follows:

Origin	Steam distn.	Content, %
American Italian Yugoslavian French Swedish and Finnish	$\begin{array}{c} 1.2-1.1 \\ 1.1-1.0 \\ 0.6-0.9 \ (6) \\ 0.5 \ (6) \end{array}$	3.0-4.0 1.8-1.6 1.6-1.2
Russian German (Thuringian) Polish German (West Prussian)	$\begin{array}{c} 4.0-5.0 \ (6) \\ 0.6-0.9 \ (6) \\ 0.6-0.9 \ (6) \\ 0.6 \ (6) \end{array}$	$1.0-1.9 \\ 1.3-1.0 \\ 1.6-1.2 $

Studies indicate that unripe berries give high oil percentages. American juniper from the Rocky Mountains contains as much as 3 to 4 per cent oil, but it is quite turpentinelike in character. Thus, by assay limits on oil analysis, we may exclude many undesirable types of berries as well as old ones.

The foregoing studies, in addition to the fact that the writers prefer Italian or Yugoslavian berries, led to the specifications for volatile oils.

#### **Refractive Index**

The refractive index of authentic samples of juniper oil are as follows:

Source of Berries	Hiram Walker Lab (Berries 6 to 12 Mo. Old)	Guenther (14) (Fresh Berries)	Gildemeister and Hoffmann (9) (Fresh Berries)
Tyrolean	1.4791-1.4816	1.4756-1.4817	
Czechoslovakian	1.4790 - 1.4825	1.4795 - 1.4818	
Italian	1.4812 - 1.4870	1.4790 - 1.4842	1.4790 - 1.4840
Yugoslavian No. 4	1.4817 - 1.4841		
Yugoslavian No. 5	1.4817 - 1.4837		

The Hiram Walker research department selected a refractive index of 1.4840 to 1.4870 as giving a mild, sweet type of juniper flavor. These values are conditioned by the technic of distillation. It is interesting to note that this value serves to exclude the undesirable Czechoslovakian berries and also those from the Tyrol. Age is believed to enhance this index. This enhanced value may be an indication of deterpenification, a desirable quality. Since terpenes are the most volatile fractions of the oils, they escape first. Pine oils have a refractive index of 1.480 or lower, and a sample of terpeneless juniper in the writers' stock has a refractive index of 1.499. Therefore we may accept a certain rise in this value as a good index, whereas too high a value may indicate very old berries.

#### Solubility in Alcohol

Even though alcoholic solubility aids in differentiating old and new stocks and in differentiating one type from another (9, 13), more emphasis is placed upon alcoholic distillation for the selection of the desired berries.

#### Bouquet

To date, the writers have not felt it practical to make the isolation or determination of the percentage of any particular terpenic derivative found in juniper oil (such as alpha-pinene, camphene, borneol, isoborneol, 4-terpineol, or cadinene) the reason for discarding certain junipers, but they are well aware of the possibilities and are conducting studies on the subject. Oil from Juniperus phoenicia L., one of the common species used to adulterate the communis, contains about 90 per cent terpenes, of which the bulk is alpha-pinene (10). The boiling point of alpha-pinene is 156.2° C. The fractional distillations reported on genuine Juniperus communis (13) yield only 1 to 2 per cent of the total distillate boiling below 156° C.

To eliminate all possibility of chance, an individual alcohol distillate of the juniper berries should be made in a laboratory pilot still. This should be followed by preparing a complete flavor-controlled gin in the same still. Seldom do constants on the raw stocks fail to eliminate undesirable botanicals. The bouquet of the pilot-still distillates should check with standards that the distiller has collected and preserved in his files.

The physical constants of oils may be adjusted by adding adulterants, but it is very difficult to adjust several constants of the berries.

Numerical analytical values are also important in case a shipment is rejected, since it becomes possible to offer evidence other than organoleptic properties, as to the authenticity of the crude drugs.

#### **Controlled Condensation**

To obviate cutting heads (removal of low boilers) and to secure a continuously identical series of gin distillations, the writers have installed mechanical devices on the gin still to control the temperature of the vapors in the condenser and thus permit the venting to waste from the still of all lowboiling aldehydes and terpenic products derived from the alcohol and botanicals. This method gives the advantage of controlled temperature of condensation. In this operation also, the oils interact with the alcohol and with themselves to produce desirable flavors not secured by cold compounding or, in other words, in "compounded gins." Likewise, automatic control of the quantity of reflux and rate of distillation adds smoothness and uniformity to the final product. This makes it possible to establish, through automatic steam controllers, the exact optimum time and rate of distillation. Figure 2 shows the general scheme of these two automatic controls.

#### Heads and Tails

The return of the heads and tails is deleterious to flavor control. Recycling of these fractions permits an accumulation of acid constituents and astringent notes. If the heads and tails are drawn, they should be cleaned by rectification. thus permitting the re-use of the recovered alcohol.

Obviously the temperature, at which the collection of potable gin is stopped and the tails are sent to a separate tank, is highly important. By varying the cutting temperature, the quantity of the higher boiling fractions of the volatile oils is modified, and hence the taste and bouquet of the gin are changed. Therefore cutting temperatures must also be fixed.

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