LAVOURING MATERIALS

TURAL & SYNTHETIC

A. CLARKE

OXFORD TECHNICAL PUBLICATIONS

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BY

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PREFACE

THIS volume represents the accumulated notes of a number of years during which the author has been connected, in a chemical and technical capacity, with the foodstuff and beverage trades. Several excellent books are in existence which deal in a specialised fashion with certain sections of flavouring materials, *e.g.* essential oils; also a number of contemporary journals deal regularly and systematically with many of the products mentioned. It is intended, however, that the notes gathered herein should form a work of reference, of a more comprehensive type than has yet been published, for the whole of foodstuff and beverage manufacturers, many of whose flavouring materials are drawn from widely varying sources.

Frequent reference to contemporary scientific literature is made in the text with a view to providing a ready means of following up information dealing with any special flavouring material.

A. C.

INTRODUCTION

THE SENSE OF TASTE

Before commencing the treatment of the various substances which are used as flavouring agents it is proposed briefly to describe the physiology of the sense of taste and to indicate the importance of flavouring bodies in the assimilation of food-stuffs.

Closely allied to the sense of taste is that of smell. The two are frequently referred to as the "chemical senses" because their excitation depends upon specific chemical substances. In a great many cases, especially with aromatic bodies, the two senses function together. Experimentally, they may be readily divided. If the sense of smell is suspended either by nasal catarrh or by closing the nostrils many of the aromatic flavours cannot be distinguished. The gustatory sense is, in fact, the most uneducated and backward of the senses in the majority of individuals. The influence of sight is also interesting in this respect; if the suggestion conveyed by the eyes is removed, either by blindfolding the subject or by making the object unusual in appearance, the sense of taste is found to be very erratic.

The four primary or elementary tastes are sweet, bitter, acid and saline, and it is usual for the taste organs of the tongue to be able to distinguish these under any but very abnormal circumstances. Most flavouring bodies are, however, of a compound character, having a tendency towards one or more of the above primary tastes and containing also some aromatic body, either of a volatile or extractive nature. In dealing with such bodies the combined faculties of taste and smell are required, but commonly both are classified as taste.

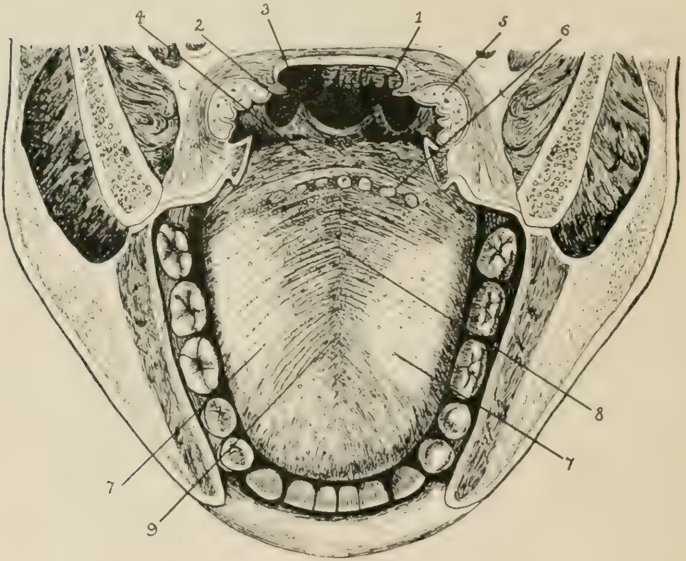

FIG. 1.—Horizontal Section through the Mouth, showing Organs of Taste.

(1) Pharyngo-palatine arch, (2) glosso-epiglottic fold, (3) epiglottis, (4) pharyngeal portion of tongue, (5) palatine tonsil, (6) vallate papillæ, (7) fungiform papillæ, (8) raphe of tongue, (9) conical papillæ. (After Cunningham.)

The dorsal surface of the tongue is divided into two distinct sections, as shown in Fig. 1, by a V-shaped row of eight or nine large circumvallate papillæ. The section extending from the vicinity of the V to the tip is covered with mucous membrane containing innumerable tiny papillæ of the filiform and conical type, while dotted all over the surface and especially at the sides of the tongue are slightly larger papillæ of the fungiform type.

Behind the lingual V at the base of the tongue are lymphatic follicles and mucous glands which are specially concerned with the sense of taste. Besides this area, the soft palate, the anterior pillar of the fauces, the posterior wall of the pharynx, the laryngeal surface of the epiglottis and the inner area of the arytænoid processes of the larynx are endowed with varying degrees of the gustatory sense.

The tiny organs which were found by Loven and Schwalbe, in 1867, to be the agencies which directly registered the taste of a substance are contained chiefly in the epithelial layers of the circumvallate and fungiform papillæ. They are also contained in the areas behind the lingual V mentioned above. In shape each organ resembles a flower-bud, of approximate measurements 80μ in height and 40μ in breadth, the term "taste-buds" being usually applied to them. The nerve fibres lie alongside and between the epithelial cells of the taste-buds.

The taste-sense is not distributed evenly over the entire surface of the tongue, and several workers, among them Kiesow and Hanig, have spent considerable time in the determination of the areas of the greatest sensibility. Further, taking the simple tastes sweet, bitter, acid and saline, it has been demonstrated that the areas of sensibility vary with each. For example, it has been found that the central area of the tongue is comparatively insensitive to most tastes, but that the area of insensibility varies according as to whether the substance applied to it is sweet, bitter, acid or saline.

It has been established that the tip of the tongue is most sensitive to sweet substances and that the base is least sensitive. The sensibility diminishes gradually from the tip to the base and from the sides inwards.

The base of the tongue, near the lingual V, is most sensitive to bitter substances and the tip is the least sensitive. From the base along the sides a rapid diminution in sensibility occurs, while from the sides inwards a gradual essening of perception is evident.

The maximum sensibility to acid has been shown to occur about half-way down each side of the tongue, the sensibility diminishing towards both tip and base. Also, from the edges of the tongue inwards towards the central insensitive area the sensibility to acid gradually diminishes.

The tip and edges of the tongue are most keenly perceptive of saline substances, the base being least sensitive. From the edges inwards the sensibility to salt is fairly constant until the insensitive area is reached.

The sensibility of the taste-organs of the tongue may be modified in many ways. For example, if the tongue is held in iced water for ten minutes it is rendered insensitive to most tastes other than those of an acid description. The action of water at a temperature of 50° C. for a similar length of time has the same effect. Kiesow demonstrated that after treating the tongue with dilute solutions of hydrochloric acid and salt distilled water gives a sweet sensation on the tongue. Many other so-called contrast sensations of this type have been noted. The temporary suspension of the power of taste, for several or all of the elementary tastes, is produced by treating the tongue with certain substances, *e.g.* cocaine, eucaine-B, stovaine, gymnenic acid, etc.

The organs upon which the human being is chiefly dependent for an appreciation of aromatic substances are illustrated in Fig. 2. The olfactory nerves terminate in the mucous membrane of the upper portion of the two nasal chambers, the nerve fibres being disseminated over the upper portion of the cavity.

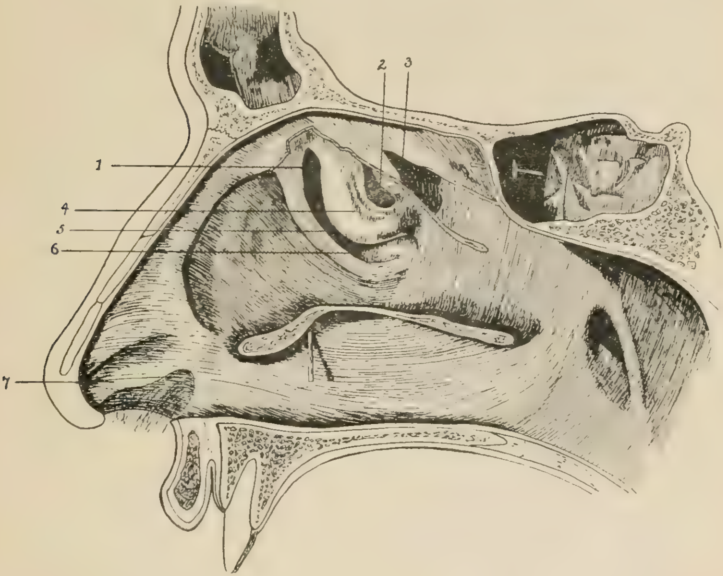

Fig. 2.—View of the Lateral Wall of the Nose.

(1) Opening of anterior ethmoidal cells, (2) opening of middle ethmoidal cells, (3) opening of posterior ethmoidal cells, (4) bulla ethmoidalis, (5) hiatus semilunaris, (6) opening of maxillary sinus, (7) vestibule. (After Cunningham.)

Many theories have been advanced regarding the olfactory sense. One of the latest was propounded by A. Durand at the Paris Academy of Sciences in January 1918. He stated that the sense of smell was dependent on the following conditions: the presence in the air of "ions odorants" which are capable of aiding the condensation of atmospheric moisture, a suitable amount of moisture in the atmosphere and the cooling of the air current which is produced by inspiration (see also *Journ. Chem. Soc.*, 1918, i./88; *Perfumery and Essential Oil Record*, 1917, 8, 336; 1918, 9, 41).

A very interesting field of research, in which there is still the opportunity for a vast amount of work, is that which deals with the relation between chemical constitution and the taste and smell of chemical compounds.

As a consequence of a research upon the chlorides, bromides and iodides of lithium, sodium, potassium and rubidium, Gley and Richet in 1885 formed the opinion that taste is a chemical effect because it takes place in accordance with the ordinary laws of chemical reaction. In 1888 Corin concluded as regards acids that the intensity of the acid taste of a molecule of any acid is dependent on the relation between the weight of the acidic hydrogen—which it is possible to substitute by a metal—contained in the molecule and the weight of that molecule.

Höber and Kiesow in 1898 investigated the possibility of taste being due to free ions in molecules which are capable of ionisation. They showed that the three elementary tastes, sweet, bitter and saline, can be produced by free ions and also that the compound tastes of various bodies is the resultant sum of the tastes of their individual components broken up by the dissociative action of the water.

Haycraft in 1887, Sternberg in the period 1898 to 1903 and Herlitzka in 1908-9 attempted to determine the relations between different tastes and the position of the atoms or groups of atoms in the molecule; most of the researches were, however, carried out with inorganic substances. Barrel and Ranc, in the *Revue Scientifique*, Dec. 7, 1918, summarised the state of knowledge to that date as regards sweet tastes, their conclusion being that considerable ground remained to be covered before the taste of a compound could be deduced with accuracy from its molecular structure.

As regards smell, although several attempts have been made to correlate the olfactory properties of bodies and their chemical constitutions, not very much progress has been achieved. In researches dealing with inorganic compounds, Haycraft, Passy and Zwaardemaker demonstrated, at different times, that the elements which give odoriferous compounds belong almost entirely to the fifth, sixth and seventh groups in the Periodic Classification of Mendeléeff. Also, a definite periodicity occurs within each series of compounds in the appearance of odorous and non-odorous substances.

The comparatively recent development in the production of synthetic odoriferous bodies has emphasised the fact that in homologous series of organic compounds, *e.g.* alcohols, acids, aldehydes, etc., there is frequently a regular change, both in the intensity and quality of the odours, as the series ascend.

The reader who wishes to pursue the study of recent progress and thought on the classification of perfumes and the correlation of odour and chemical constitution will find the following references of interest: *Odour and Constitution*, Austerweil and Cochin, *P. & E.O.R.*, August 1910; *Sense of Smell*, Dr. R. Foerster, *P. & E.O.R.*, September 1911; *The Senses of Taste and Smell*, A. Baudrexel, *P. & E.O.R.*, July 1914; *Classification of Odours*, E. J. Parry, *P. & E.O.R.*, May 1916; *Odour and Constitution*, Dr. H. J. Prins, *P. & E.O.R.*, July 1917; *Constitution and Odour*, R. Marchand; *Chem. Abstr. Am. Chem. Soc.*, 1916, 10, 2383; and also *Journ. Chem. Soc.*, 1921, i./637, 755.

The importance of flavouring compounds in the assimilation of foodstuffs is at once apparent when the fact is realised that the digestion of food depends to a considerable extent upon its palatableness. It has been demonstrated that food which is relished and which stimulates the appetite is usually digested with much greater ease than that which is insipid and flavourless. The over-flavouring of foodstuffs is, of course, an evil practice in that it weakens the perception of the gustatory and olfactory senses for the more delicate flavours.

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FLAVOURING MATERIALS

SECTION I

SPICES, CONDIMENTS AND MISCELLANEOUS VEGETABLE AND ANIMAL FLAVOURING BODIES

CHAPTER I

SPICES AND CONDIMENTS

Allspice or Pimento.—Allspice, Jamaica Pepper or Pimento is the dried, unripe, full-grown berry of the *Pimenta officinalis* (Myrtaceæ), an evergreen tree which is indigenous to the West Indies and is especially cultivated in Jamaica. The berries, which grow in small clusters, are gathered and then either sun- or kiln-dried. After drying they are either brown or grey in appearance.

Allspice is less pungent than either cloves or cassia, its flavour being said to resemble a mixture of cloves, cinnamon and nutmeg. It owes its flavour to a volatile oil, largely composed of eugenol, which it contains to the extent of 3 to 4·5 per cent. principally in its ligneous pericarp. It finds a wide range of application, being used in pickles, sauces, relishes, bitters, liqueurs, and as a flavouring material in both savoury and sweet foodstuffs.

The spice appears on the market both in the whole berries and as a fine powder. In the "whole" condition it is subject to adulteration with the berries of the Pimenta di Tabasco (Mexican Spice) and bayberry fruit (*Pimenta acris*). These berries are rather larger than and different in appearance to the ordinary pimento berries. In the powder form it is most commonly adulterated with ordinary starches, cocoanut shell, clove stems and pea flour. Other substances which have been discovered in ground allspice are exhausted ginger, olive stones, pear stalks (Möller, *Lehr. Pharm.*), cayenne pepper and turmeric.

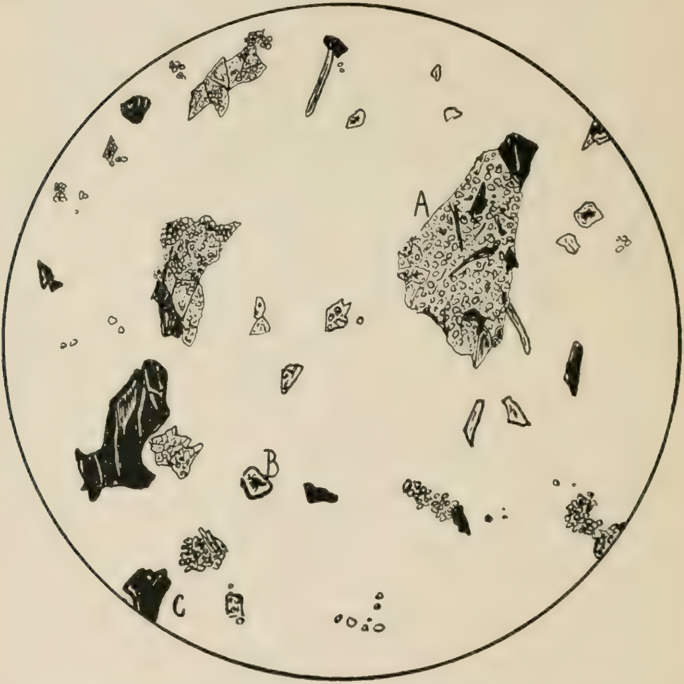

Fig. 3.—Micrograph of Powdered Allspice, showing (A) masses of starch, (B) stone cells and (C) resin.

Analytical Constants.—The moisture content is usually 6 to 7 per cent.; ash should not exceed 5 per cent., of which the portion insoluble in hydrochloric acid (sand) should not exceed 0·5 per cent., the United States standard for ash is not more than 6 per cent.; the total ether extract is about 10 per cent., of which about 4 per cent. is volatile oil; the alcoholic extract varies between 10 and 12 per cent.; crude fibre is present to the extent of about 20 per cent., the U.S. limit being 25 per cent.; the quercitannic-acid content should be not less than 8 per cent. according to U.S. standards. A quantity of starch occurs in pimento, its presence being a distinctive feature of the micro-examination of the spice.

Micro-examination. — The main characteristics of powdered allspice, when viewed under the microscope, are masses of small, almost circular starch granules, large stone cells, appearing either singly or in clusters, sometimes together with fragments of brown parenchyma, and brown or amber-coloured pieces of gum or resin. A few drops of oil are occasionally to be seen and short hairs of a tapering, thick-walled nature.

Caraway.—Caraway fruit, or seed as it is wrongly termed, is the dried, ripe fruit of the *Carum carvi* (Umbelliferæ), which is grown in Holland, England, Germany, Norway and Russia. The English variety is of the greatest value and the Russian of the least value, the latter, being usually mixed with stalks and giving a higher percentage of ash, is used chiefly as a veterinary medicine. The agreeable, aromatic flavour of caraway is due to its content of 3 to 7 per cent. of a volatile oil.

The chief adulterant of caraway fruit is exhausted caraway; this, however, may be detected by its shrivelled appearance, weaker flavour, darker colour and, under the microscope, its ruptured outer cells.

Analytical Constants.—The ash is usually between 5 per cent. and 7·5 per cent.; according to the *British Pharmacopœia* it should not be in excess of 9 per cent., and according to U.S. standards not over 8 per cent. The volatile-oil content is usually 3 to 7 per cent. according to the source of the fruit. The ether extract should be about 10 per cent.

Micro-examination.—The powder shows masses of cells of an oblong structure, from the inner epidermis. Also, there are cells of a pitted, striated nature from the outer epidermis, but no hairs or cell-crystals.

Cardamoms.—These are the dried, fully-ripe seeds of the *Elettaria cardamomum* (Scitaminaceæ), which grows in the East Indies. Other varieties are used as spices, but the *B.P.* recognises only the *Elettaria cardamomum.* Malabar, Mangalore and Mysore cardamoms are the chief commercial varieties. The Malabar and Mangalore types are usually short, well-filled capsules, the Mysore variety being larger and more loosely filled. An interesting description of a very large type of cardamoms is given by E. M. Holmes (*P. & E.O.R.*, 1914, 302). Cardamoms are used chiefly for culinary purposes, for sauces, curries, cordials, etc.

The seeds should not be extracted from the pericarps until required for use. The fruit usually yields about 75 per cent. seeds. In colour the latter are dark brown, they contain 4 to 8 per cent. of a volatile oil and also a quantity of fixed oil.

Analytical Constants.—The *B.P.* requires the ash not to exceed 6 per cent.

Micro-examination.—The distinctive features of ground cardamoms are the deep-tinted cells of the inner covering of the seeds, the perisperm cells containing masses of angular starch granules and calcium oxalate crystals and the thick-walled linear cells with oblique ends of the skin layer. The presence of polygonal cells with straight walls and also the content of small cells containing brown resinous matter denote that the pericarp has been ground with the seeds. Small spiral cells also indicate the presence of the pericarp.

Cinnamon and Cassia.—*True cinnamon* is the dried bark of the *Cinnamomum zelanicum* (Lauraceæ), a tree which is indigenous to Ceylon but is cultivated largely in Sumatra, Java and many parts of tropical Asia. The bark appears on the market in the form of long, cylindrical rolls or quills, several layers usually being found in each quill. In its preparation, the bark is first scraped and then the inner layer is cut into slices and dried. True cinnamon has a light yellow-brown colour with light-coloured lines running the length of the quills. The outer surface is marked with small round spots. The history of the cultivation of cinnamon and cassia are dealt with comprehensively by E. M. Holmes (*P. & E.O.R.*, 1916, 14 and 41).

Cinnamon has greater intrinsic value than cassia, being of a finer and much more delicate flavour. The volatile oil to which cinnamon owes its flavour is contained in the bark to the extent of 0·5 to 1·0 per cent. The oil is composed chiefly of cinnamic aldehyde. Cinnamon is used medicinally as a stimulant and cordial. As a flavouring material it is used chiefly in sweet foodstuffs, also, together with other vegetable flavouring bodies, in the preparation of bitters, liqueurs, sauces, etc.

In the quill form cinnamon is sometimes adulterated with clove stems, guava bark and inferior grades of cinnamon, the flavours of which are rather bitter. Powdered cinnamon is adulterated with walnut-shells (Dyer and Gilbard, *Analyst*, 1895, 129), starches, turmeric, olive stones, pepper, exhausted ginger (Stock, *Analyst*, 1897, 253), sand, sawdust, mustard and the ground bark of common trees, *e.g.* elm.

Analytical Constants.—Moisture is usually about 8 per cent.; ash should not be in excess of 5 per cent. (*B.P.*), although genuine samples sometimes exceed this figure slightly; volatile oil is 0·5 to 1·0 per cent.; fixed oil, about 1·5 per cent.; crude fibre, 30 to 35 per cent.; alcoholic extract, 10 to 13 per cent. Cinnamon contains very small, oval starch granules.

The *Cassia* of commerce is the bark of the *Cinnamomum cassia*, which is grown in India, Indo-China and China. The quills are coarser, about four times thicker and more loosely rolled than cinnamon quills. The outer surface of the quills lacks the light-coloured lines noticeable on cinnamon quills, they have oval leaf markings and small brown excrescences.

Cassia buds are the dried flower-buds of the cassia tree. The best qualities have a strong cinnamon flavour.

Cassia, in both quill and powder forms, is subject to similar adulteration to cinnamon. The Chinese product is sometimes placed on the market in a dirty condition. It contains rather more starch than cinnamon and the starch granules are considerably larger. Its volatile-oil content is approximately 0·5 to 2·0 per cent.

Analytical Constants.—Moisture, 8 to 12 per cent.: ash, 5 per cent., although the United States allows up to 8 per cent., with a sand content of not more than 2 per cent.; volatile oil, 0·5 to 2 per cent.; fixed oil, 1 to 3 per cent.; crude fibre, about 24 per cent.; alcoholic extract, 8 to 10 per cent.; starch varies between the wide limits of 15 per cent. and 30 per cent.

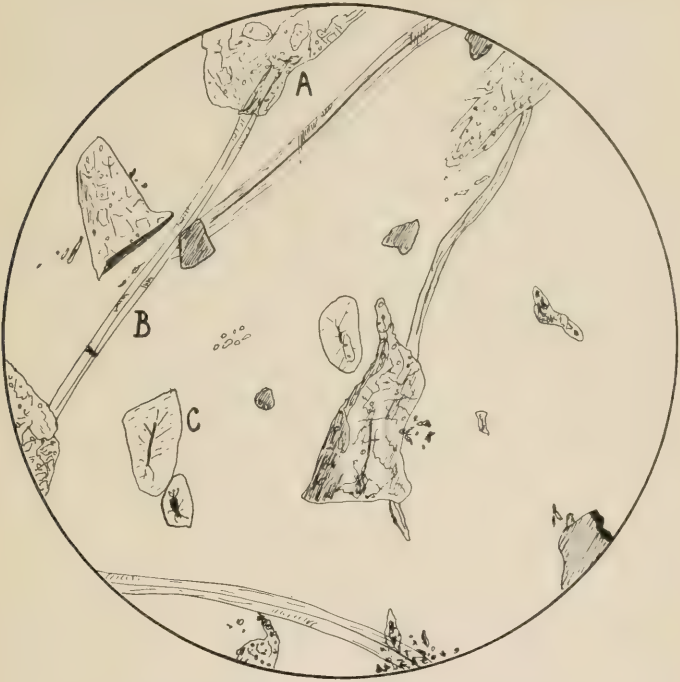

Fig. 4.—Micrograph of Powdered Cinnamon, showing (A) masses of starch granules, (B) bast fibres and (C) stone cells.

Micro-examination. — Cinnamon and cassia are very similar when viewed under the microscope. The chief differences are that the starch grains of cassia are larger and present in greater numbers than in cinnamon. The bast fibres of cinnamon are more slender, longer and thinner-walled than those of cassia. The chief characteristics of both cinnamon and cassia are groups of small starch granules, somewhat resembling allspice starch but usually with fewer granules to each group, yellow or brown tinted cellular tissue containing occasional starch grains, stone cells of yellow-brown shade and somewhat oblong in shape. Cassia also contains long, yellow wood fibres either singly or in bunches.

Cloves.—Cloves are the dried, unopened flowers of the evergreen *Eugenia caryophyllata* or *Caryophyllus aromaticus* (Myrtaceæ), which is a native of the Moluccas. The chief cultivation areas are Amboyna, Zanzibar, Brazil, Ceylon, Mauritius, West Indies and other tropical regions. The flavour of cloves is due to an essential oil which they contain to the extent of about 15 to 19 per cent., eugenol being the chief constituent of the oil. Cloves are used medicinally as a carminative and stimulant; as a flavouring agent they find application in both savoury and sweet foodstuffs, pickles, sauces, bitters, etc.

The most usual adulterant of cloves is the exhausted spice. This may be detected in the whole condition by its shrivelled appearance, also its ether extract is comparatively small and its ash high. Clove stems are sometimes added, but these may be detected microscopically. Clove fruit or "mother of cloves" is also added as an adulterant; this contains sago-like starch granules and stone cells by which it may be distinguished. Allspice is occasionally added as a sophistication in view of its eugenol-content. Cereal and leguminous starches, sand, turmeric, ground olive stones, sawdust and cocoanut shells should be searched for as possible adulterants, but they will rarely be found.

Analytical Constants.—Moisture varies considerably, but is usually about 7 to 8 per cent.; ash should not exceed 7 per cent. and sand not more than 0·5 per cent. (*B.P.*); volatile oil is usually about 15 to 19 per cent.; fixed oil,

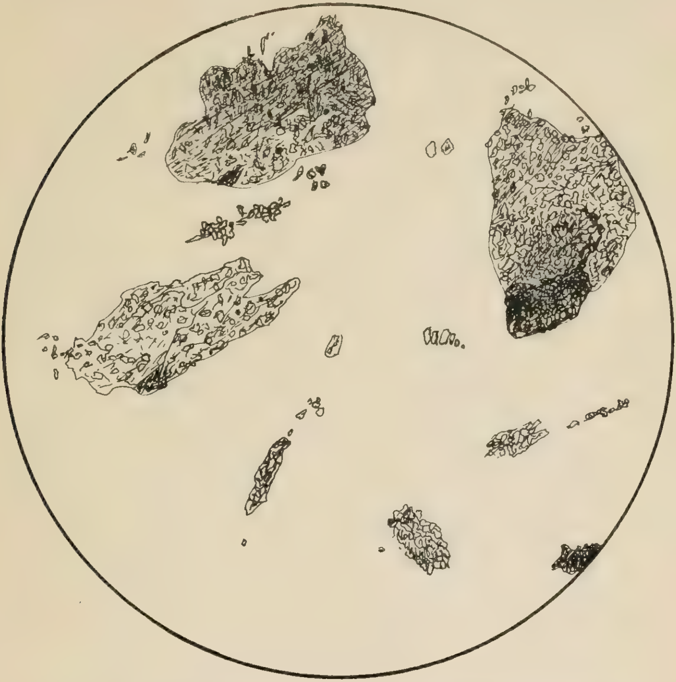

FIG. 5.—Micrograph of Powdered Cloves, showing cellular tissue with occasional oil globules.

6 to 7 per cent.; crude fibre, not more than 10 per cent. (U.S. standard); alcoholic extract, 14 to 16 per cent.; quercitannic acid, not less than 12 per cent. (U.S. standard).

Micro-examination.—The characteristic appearance of powdered cloves under the microscope is shown in Fig. 5.

Masses of cellular tissue, containing brown granular material and occasional oil globules, form the chief objects in the field.

Coriander.—Coriander is the dried, ripe fruit of the *Coriandrum sativum* (Umbelliferæ). In appearance the fruit is globular, 5 mm. in diameter, yellow-brown in colour and ribbed. It is grown chiefly in Holland, England, Germany, Russia, Morocco, etc. A Bombay coriander which is considerably larger than the ordinary variety occasionally appears on the market. The aromatic flavour of the spice is due to its content of volatile oil.

Analytical Constants.—Ash is usually 5 to 6 per cent.: volatile ether extract should not be less than 0·5 per cent.

Micro-examination.—When viewed under the microscope powdered coriander shows oblique linear cells of the endosperm, large thick-walled cells of the mesocarp and six-sided cells of the inner vittæ.

Ginger.—Ginger is the dried, scraped rhizome or rootstock of the *Zingiber officinale*, a native of India and China, which is cultivated in tropical America, Africa, Cochin, Calicut, Australia, etc. Jamaica ginger is considered to have the finest flavour, Japan ginger the poorest. The roots are dug from year-old plants when the stem has withered. They are cleaned and dried in the sun. Usually the skin is removed by scraping, the unscraped root forming what is known in commerce as the "coated" variety. This latter usually comes from Africa and occasionally from Cochin and Bengal. Whole ginger is frequently bleached with chloride of lime solution or sulphur dioxide. The rhizome is also sometimes coated with a wash of chalk or calcium sulphate to preserve it from the attack of insects.

Ginger owes its characteristic flavour to its content of volatile oil (2 to 3 per cent.) and resinous matters. The oleo-resin "gingerine" is obtained by percolating the powdered spice with acetone and evaporating the extract. Recent work on the pungent principles of ginger is referred to in *P. & E.O.R.*, 1917, 64 and 102, *Journ. Chem. Soc., Trans.*, 1917, 769–798.

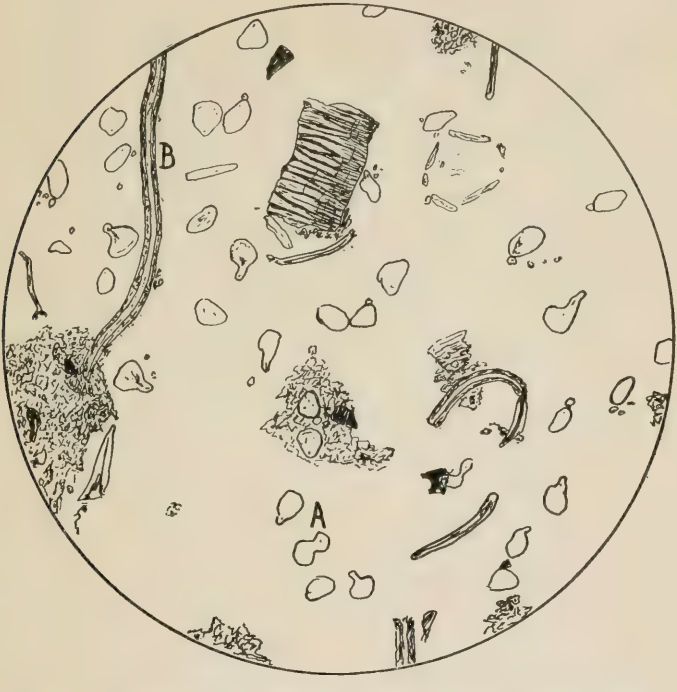

FIG. 6.—Micrograph of Powdered Ginger, showing (A) sack-shaped starch granules, (B) bast fibres, etc.

Ginger has a very wide range of uses; it is a very favourite flavouring agent in sweet foodstuffs as well as, together with other vegetable bodies, in pickles, sauces, etc. The young, soft, peeled roots when preserved in sugar syrup form a very popular conserve.

The body which is used to the greatest extent as an adulterant of ginger, in both whole and powdered forms, is exhausted ginger. This product is the residual material from the manufacture of essences or tinctures of ginger by extracting with water, dilute alcohol or strong alcohol. Ground ginger has also been adulterated with cayenne pepper, turmeric, starch, mineral matter and wood dust.

Analytical Constants.—Moisture, 10 to 14 per cent.; ash, usually 3 to 4 per cent. (*B.P.* gives ash limit of 6 per cent., with not more than 1·5 per cent. of ash insoluble in water); extractive matter soluble in cold distilled water should be about 10 to 15 per cent. (*B.P.* gives a minimum limit of 8·5 per cent., which is decidedly low); extractive matter soluble in 90 per cent. alcohol should not be less than 5 per cent. (*B.P.*); the U.S. standards are as follows: Starch by diastase method, not less than 42 per cent.; crude fibre, not more than 8 per cent.; ash insoluble in hydrochloric acid, not more than 3 per cent.

Micro-examination.—The examination of ginger under the microscope is not of much value in the detection of exhausted ginger. The chief characteristics of powdered ginger are sack-shaped starch granules, which under polarised light show an unsymmetrical cross, together with bast fibres and oleo-resin cells.

Mustard.—Mustard is obtained from the seeds of the *Brassica nigra*, or black mustard, and *Brassica alba*, or white mustard (*Journ. Chem. Soc.*, 1921, i. 212). The two are powdered and mixed together to form the condiment, most of the fixed oil being removed in the process, together with the outer shell or husk of the black seed. The mustard plants are native to Europe and are widely cultivated in Great Britain and the United States. White mustard seeds measure 2 to 3 mm. in diameter; they are pale yellow in shade and have a granular surface. The black seeds are 1 to 1·5 mm. in diameter, dark brown in colour and have a rough surface.

The constituents of mustard seeds are a fixed oil, a volatile oil (0·5 to 0·75 per cent.) and mucilage. The characteristic properties of mustard are due to the allyl-iso-thiocyanate which is developed by the action of water and an enzyme, myrosin, which is contained in both black and white varieties, on the glucoside potassium myronate, which is contained only in the black seeds. A glucoside, sinalbin, occurs in the white seeds and on hydrolysis this body forms acrinyl-iso-thiocyanate, which like allyl-iso-thiocyanate is a very pungent oil. Mustard seeds in their ripened state are free from starch.

The chief application of mustard is, of course, as a condiment, but it is also used in many cases as the chief agent in the development of piquancy in sauces, pickles, salad creams, etc.

The adulterants which are mostly used in mustard are mustard husk, starch, turmeric, aniline dyes, cayenne pepper, terre alba, chalk, sand and gypsum to a lesser extent. Charlock and rape seeds have been used in place of true mustard. Compound or mixed mustards usually contain wheat or other starch, which is added to reduce the keenness of the condiment. The addition of starch is also said to improve the keeping qualities of the mustard. Any admixture of this type must be labelled "compound" or "mixed" mustard. The abstraction of the fixed oil is regarded by some authorities as an adulteration.

Analytical Standards.—Moisture, 4 to 7 per cent.; ash, 4 to 5 per cent., not over 8 per cent. (U.S. standard);

starch by diastase method, not over 2·5 per cent.; crude fibre, not over 5 per cent.; volatile oil, not less than 0·7 per cent. (calculated as allyl-iso-thiocyanate).

Micro-examination.—The chief characteristics of

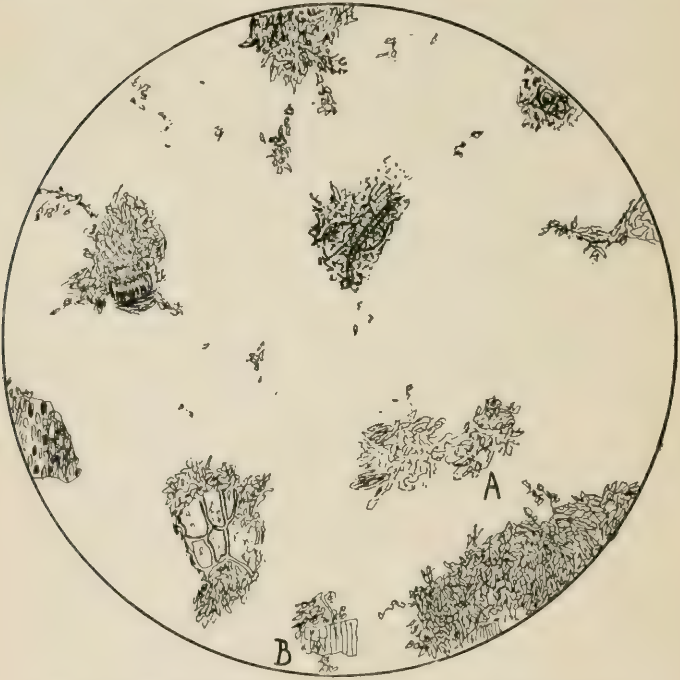

Fig. 7.—Micrograph of Pure Mustard Powder, showing (A) loose cellular tissue and (B) seed skin.

powdered mustard under the microscope are masses of loose, greyish cellular tissue, together with occasional fragments of yellow seed skin.

Nutmeg and Mace.—Both of these spices are derived from the fruit of the *Myristica fragrans*, which is a native of the Malay Archipelago. The chief source of supply is the Banda Islands. Nutmegs comprise the kernels of the fruits, and they are covered by a fibrous arillus, which when dried constitutes the mace of commerce.

Nutmeg.—The flavouring properties of nutmeg are due to a volatile oil (8 to 15 per cent.), besides which it contains albuminous matter, starch, fixed oil and fibre. The spice finds its chief use as a flavouring for sweet foodstuffs, although it is used to some extent in the compounding of bitters, etc.

Nutmegs are usually sold whole, and in this state are not subject to much adulteration. Powdered nutmegs have been adulterated with ground nutshells, starch, etc.

Analytical Standards.—The U.S. standards for nutmeg are as follows: Ash, not more than 5 per cent.; sand, not over 0·5 per cent.; non-volatile ether extract, not less than 25 per cent.; crude fibre, not over 10 per cent.

Micro-examination.—The chief characteristics of powdered nutmegs are loose masses of cellular tissue and numerous starch granules of a uniform size, nearly circular shape and with well-marked hila.

Mace.—When freshly stripped from the fruit, the foliaceous covering which constitutes mace is bright red in colour. After drying, however, it is yellow-brown and brittle. The flavour of mace is due to a volatile oil, besides which it contains a fixed oil, a carbohydrate (amylo-dextrine) and resinous and albuminous matter. Mace is used with other spices in sauces, pickles, etc., and as a flavouring material for savoury foodstuffs.

The chief sophistication of mace takes the form of the substitution of inferior types of mace for the Banda variety. Among these inferior types the chief are Bombay mace, derived from the *Myristica malabarica*, which is useless as a flavouring material, and Macassar mace, which although not so flavourless as the Bombay type has a wintergreen odour and is inferior to the Banda variety. Powdered mace is adulterated with the above-mentioned maces and also with ground olive stones and starches.

Analytical Standards.—The U.S. standards for mace are as follows:—The fixed ether extract should not be under 20 per cent. nor over 30 per cent.; ash, not in excess of 3 per cent.; sand, not over 0·5 per cent.; crude fibre, not over 10 per cent.

Micro-examination.—The main characteristics are loose-textured masses of a greyish-white shade, very small granules of amylo-dextrine and occasional fragments of very light yellow-brown wood fibre.

Pepper.—Pepper is the dried, unripe berry of the climbing shrub *Piper nigrum*, a native of the East Indies, the chief varieties being derived from Siam, Penang, Singapore, Malabar, Cochin, Allippi, Lampong, Tellicherry, Sumatra, Mangalore, Trang, etc. Black pepper is obtained by sun- or fire-drying the berries, whereupon they shrivel and turn black or brown. White pepper is produced from these dried berries by a decortication process, which includes soaking the berries in water to loosen the skins, drying and eliminating the skins by friction. The principal use of pepper is as a condiment. Many pickles and sauces contain black pepper.

The chief constituents of pepper, which impart to it its peculiar properties, are a volatile oil (0·5 to 2 per cent.), a pungent resin and 2 to 3 per cent. of piperin.

In the whole condition pepper is not subject to extensive adulteration, although imitation peppercorns made from clay are said to have been employed.

A. Troccoli and G. Verona-Rinati (*Zeits. Unters. Nahr. Genussm.*, 1912, 736–741) found samples of peppercorns to be adulterated with seeds of *Rhamnus cartharticus* (buckthorn) and *Schinus molle* (American or false pepper). The adulterants most commonly found in ground pepper are inorganic substances, such as sand, clay, chalk, barytes, etc., rice and other farinaceous products, ground olive stones and nut shells, pepper shells and dust, linseed meal, spent ginger, mustard husks, charcoal, graphite, sawdust, turmeric, charred cocoanut shells, etc. A case in which 10 per cent. of steatite was found has been reported. Long pepper, which is the fruit of an Indian plant, *Chavica Roxburghii*, is sometimes used as an adulterant when price permits.

Analytical Constants.—Black Pepper.—Moisture, 8 to 12 per cent.; ash, 3 to 5 per cent., not over 7 per cent. (U.S.); crude fibre, 10 to 11 per cent., not over 15 per cent. (U.S.); starch by diastase method, not less than 25 per cent. (U.S.); non-volatile ether extract, not less than 6 per cent. (U.S.); nitrogen, not less than 3·25 parts in 100 parts of non-volatile ether extract (U.S.); volatile oil, 0·5 to 1·8 per cent.; piperin and resin, 7 to 8 per cent.; albuminoids, 7 to 12 per cent.

White Pepper.—Moisture, 8 to 12 per cent.; ash usually 1 to 2 per cent., not more than 4 per cent. (U.S.), with sand not more than 0·5 per cent.; starch by diastase method, not less than 50 per cent. (U.S.); non-volatile ether extract, not less than 6 per cent. (U.S.); nitrogen, 4 parts in 100 parts non-volatile ether extract; piperin and resin, 7 to 8 per cent.; volatile oil, 0·5 to 1·8 per cent.; albuminoids, 8 to 10 per cent.

Micro-examination.—The most prominent features of black pepper are large masses of starch and pieces of the coloured parenchyma and epidermis of the shell. Yellow-brown stone cells, with thick walls, are also fairly numerous, as are particles of resinous matter. Needle-shaped crystals of piperin and fragments of stem are also noticeable. Some new interesting features regarding the micro-structure of pepper are given by T. E. Wallis (*Analyst*, 1915, 190).

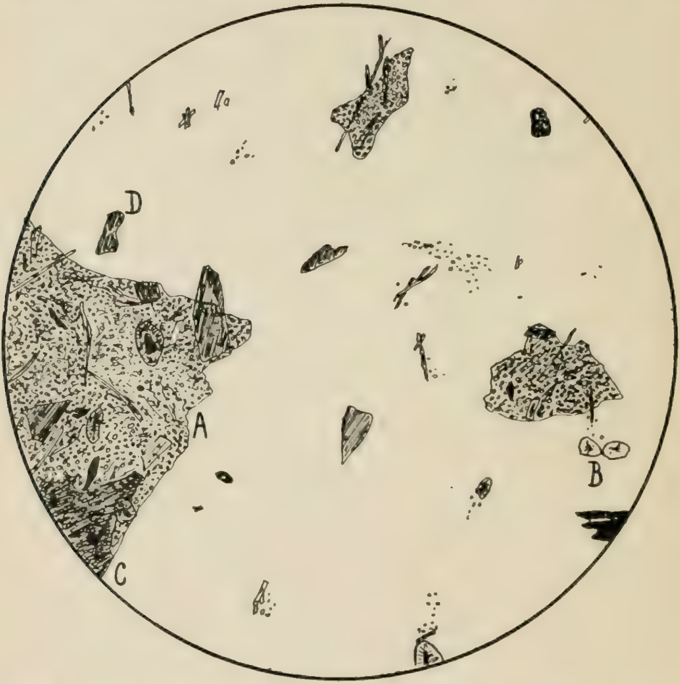

FIG. 8.—Micrograph of Ground Pepper, showing (A) masses of starch granules, (B) stone cells, (C) particles of shell epidermis and (D) resinous matter.

Red Pepper.—Various species of capsica are classed under this head. The type which is official in the *B.P.*

and which is not synonymous with cayenne pepper is called *Capsicum minimum.* This varies in appearance and pungency, as do the other varieties of red pepper, according to the country in which it is grown. For example, the product derived from Sierra Leone is usually yellowish-red and very pungent, that from Zanzibar is redder and has a diminished pungency, while the Japanese variety has a very brilliant red colour and very much less pungency. According to the United States definition, red pepper is the red, dried fruit of any species of capsicum, most commercial cayenne pepper being, however, of the *C. annuum* and *C. fastigiatum* types. A comparison of the different varieties of red pepper is made by L. M. Tolman and L. C. Mitchell in *Bull.* 163, *U.S. Dept. of Agric., Bureau of Chem.*, May 1913, pp. 1–32.

Cayenne pepper of commerce is chiefly obtained from Natal, Egypt, Nepaul, Zanzibar, Sierra Leone, Spain, Hungary and Japan. Nepaul cayenne pepper is supposed to have the finest flavour, it has a red-brown colour and an odour resembling violets. Spanish and Hungarian peppers, which are known respectively as "Pimiento" and "Paprika," the latter being derived from *C. tetragonum*, have very little pungency and give rise to the so-called "Bird Pepper."

Cayenne pepper contains a volatile alkaloid, resin, fat, a red colouring matter and capsaicin. The latter compound is a very acrid body which is soluble in alcohol, ether, benzene and acetic acid. The flavour of cayenne pepper deteriorates on exposure to damp. The pepper is used chiefly in sauces, pickles, cordials, etc., and also in bitters.

The following are the adulterants which are alleged to have occurred in cayenne pepper: brick dust, oxides of iron, red lead, vermilion, gypsum, ground redwood, coal tar dyes, turmeric, ginger, nutshells, rice starch, olive stones, mustard hulls and exhausted capsica. The colour of paprika and pimienta is occasionally deepened by grinding with olive oil.

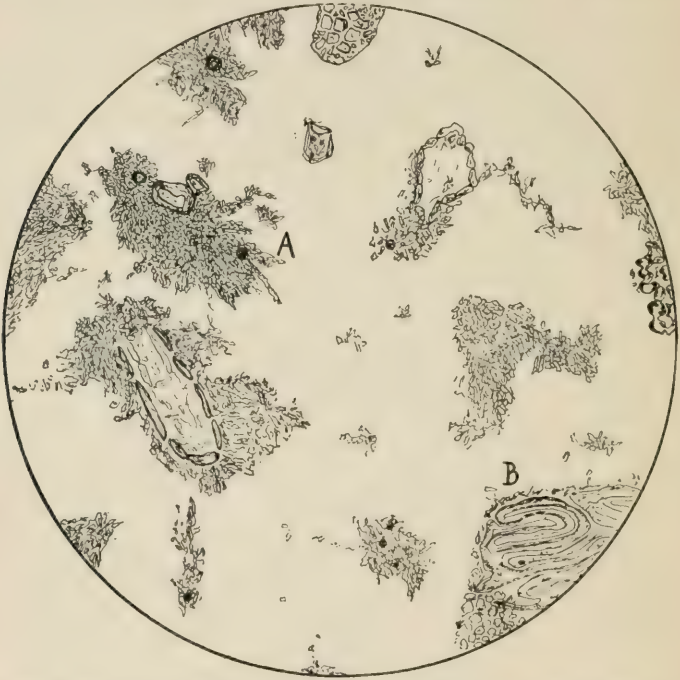

Fig. 9.—Micrograph of Cayenne Pepper, showing (A) Cellular Masses containing Oil Drops and (B) Intestine-like Cells from the Epidermis of Seed Shell.

Analytical Constants.—Moisture should not exceed 10 per cent., 5 to 7 per cent. being a fair average; ash should not exceed 6 per cent., usually varying between 4 per cent. and 6 per cent.; the amount of ash insoluble in hydrochloric acid is usually 1 to 2 per cent.; ether extract is usually about 18 per cent., although variations between 17·5 per cent. and 22 per cent. are frequently met with: in most cases the non-volatile ether extract is about 90 per cent. of the total ether extract; crude fibre should not exceed 28 per cent.; starch, by the diastase method, should not exceed 1·5 per cent.

Micro-examination. — The chief characteristics are cellular masses containing occasional brilliant red oil drops, but no starch. Cells from the epidermis are sometimes to be observed, as are also remarkable intestine-like cells from the skin of the seed shell.

CHAPTER II

ADULTERANTS OF SPICES AND CONDIMENTS

THE following are the approximate analytical constants of the chief substances which are used as adulterants of spices and condiments:

Ground Almond Shells.—Ash, 2·5 to 3 per cent.; sand, 0·05 per cent.; volatile oil, 0·15 to 0·18 per cent.; non-volatile ether extract, 0·5 to 0·75 per cent.; alcoholic extract, 5 to 6 per cent.; starch by diastase method, 0·5 to 1 per cent.; crude fibre, 45 to 50 per cent.

Powdered Brazil Nut Shells.—Ash, 1·5 to 1·75 per cent.; sand, 0·1 to 0·2 per cent.; volatile oil, 0·05 to 0·1 per cent.; non-volatile ether extract, 0·4 to 0·6 per cent.; alcoholic extract, 1·0 to 1·5 per cent.; starch by diastase method, 0·6 to 1·0 per cent.; crude fibre about 50 per cent.

Cocoa Shell Powder.—Ash, 8 to 9 per cent.; sand, 0·5 to 1 per cent.; volatile oil, 1 to 1·25 per cent.; non-volatile ether extract, 3 to 3·5 per cent.; alcoholic extract, 4·5 to 5 per cent.; starch by diastase method, 3 to 3·5 per cent.; crude fibre, 14 to 15 per cent.; total nitrogen, 2·5 to 2·75 per cent.

Ground Cocoanut Shells.—Ash, 0·5 to 0·8 per cent.; sand, nil; volatile oil, nil; non-volatile ether extract, 0·2 to 0·4 per cent.; alcoholic extract, 1 to 1·5 per cent.; starch by diastase method, 0·6 to 0·9 per cent.; crude fibre, 55 to 58 per cent.

Ground Walnut Shells.—Ash, 1·2 to 1·5 per cent.; sand, trace; volatile oil, 0·1 to 0·2 per cent.; non-volatile ether extract, 0·5 to 0·7 per cent.; alcoholic extract, 1·5 to 2 per cent.; starch by diastase method, 1 to 1·5 per cent.; crude fibre, 55 to 60 per cent.

Ground Buckwheat Hulls.—Ash, 1·5 to 2 per cent.; sand, about 0·01 per cent.; volatile oil, 0·05 to 0·08 per cent.; non-volatile ether extract, 0·3 to 0·5 per cent.; alcoholic extract, 2 to 2·5 per cent.; starch by diastase method, 1·25 to 1·5 per cent.; crude fibre, 40 to 45 per cent.

Clove Stems.—Ash, 7 to 8 per cent.; sand, 0·6 to 0·7 per cent.; volatile oil, 4 to 5 per cent.; non-volatile ether extract, 3·8 to 4·1 per cent.; alcoholic extract, 6·5 to 7 per cent.; starch by diastase method, 2 to 2·2 per cent.; crude fibre, 15 to 19 per cent.; total nitrogen, 0·92 to 0·95 per cent.; quercitannic acid equivalent, 18 to 20 per cent.

Ground Date Stones.—Ash, 1 to 1·5 per cent.; sand, 0·03 to 0·05 per cent.; volatile oil, 0·2 to 0·5 per cent.; non-volatile ether extract, 8 to 8·5 per cent.; alcoholic extract, 16 to 17 per cent.; starch by diastase method, 2 to 2·5 per cent.; crude fibre, 5 to 6 per cent.

Ground Olive Stones.—Ash, 0·5 to 1 per cent.; sand, 0·25 to 0·5 per cent.; volatile oil, 0·5 to 0·75 per cent.; non-volatile ether extract, 0·2 to 0·4 per cent.; starch by diastase method, 1·5 to 2 per cent.; crude fibre, 50 to 60 per cent.

Pressed Linseed Meal.—Ash, 5 to 6 per cent.; sand, 0·5 to 0 6 per cent.; volatile oil, 0·02 to 0·05 per cent.;

non-volatile ether extract, 6 to 7 per cent.; alcoholic extract, 9 to 10 per cent.; starch by diastase method, 13 to 15 per cent.; crude fibre, 8 to 10 per cent.; total nitrogen, 5 to 6 per cent.

Fig. 10.—Micrograph showing appearance of (A) Ground Cocoanut Shell, (B) Ground Olive Stones, (C) Wood Dust and (D) Cocoa Shell Powder.

Oak Wood Dust.—Ash, 1 to 1·5 per cent.; sand, trace; volatile oil, 0·05 to 0·1 per cent.; non-volatile ether extract, 0·5 to 1 per cent.; alcoholic extract, 6 to 7 per cent.; starch by diastase method, 1·5 to 2 per cent.; crude fibre, 45 to 50 per cent.; quercitannic acid equivalent, 12 to 13 per cent.

Red Sanderswood Dust.—Ash, 0·5 to 1 per cent.; sand, 0·5 to 0·8 per cent.; volatile oil, 1·0 to 1·5 per cent.; non-volatile ether extract, 10 to 13 per cent.; alcoholic extract, 19 to 22 per cent.; starch by diastase method, 1·0 to 1·2 per cent.; crude fibre, 50 to 55 per cent.

Spruce Wood Dust.—Ash, 0·2 to 0·4 per cent.; sand, nil; volatile oil, 0·5 to 0·8 per cent.; non-volatile ether extract, 0·6 to 1 per cent.; alcoholic extract, 1 to 1·5 per cent.; starch by diastase method, 1·0 to 1·2 per cent.; crude fibre, 60 to 65 per cent.

Pea Flour.—Moisture, 15 to 16 per cent; ash, 2·3 to 2·5 per cent.; carbohydrates, 57 to 59 per cent.; non-volatile ether extract, about 2 per cent.; proteins, 22 to 23 per cent.

Bean Flour.—Moisture, 11 to 12 per cent.; ash, 2 to 2·5 per cent.; carbohydrates, 57 to 58 per cent.; non-volatile ether extract, 2·4 to 2·5 per cent.; proteins, 26 to 26·5 per cent.

Corn Flour.—Moisture, 13 to 14 per cent.; ash, 1·4 to 1·5 per cent.; carbohydrates, 64 to 65 per cent.; non-volatile ether extract, 6·5 to 7 per cent.; proteins about 10 per cent.

Wheat Flour.—Moisture, 13 to 14 per cent.; ash, 0·6 to 0·7 per cent.; carbohydrates, 71 to 72 per cent.; non-volatile ether extract, 1·4 to 1·6 per cent.; proteins, 13 to 13·5 per cent.

Rice Flour.—Moisture, 10 to 11 per cent.; ash, 0·5 to 0·55 per cent.; carbohydrates, 83 to 85 per cent.; non-volatile ether extract, 0·8 to 1·1 per cent.; proteins, 5 to 5·5 per cent.

Turmeric.—This substance is important both as an adulterant and as a condiment. In the latter quality it is used extensively in curry powder.

Turmeric, *Curcuma longa*, is a member of the same species of plants as ginger. It is indigenous to Cochin China and the East Indies. Its chief constituent is the yellow colouring matter "curcumin," besides which it contains a volatile oil, gum, starch and cellulose. Curcumin is insoluble in cold water, but soluble in alcohol and ether. Turmeric is used as an adulterant, particularly in mustard and ginger, to cover, by means of its yellow colour, admixtures with starches.

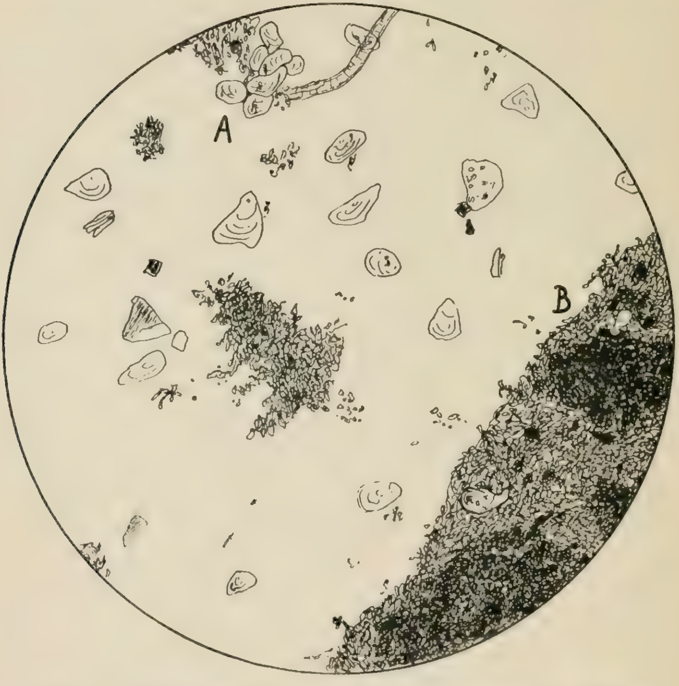

FIG. 11.—Micrograph of Powdered Turmeric, showing (A) Starch Granules and (B) Cellular Tissue containing Yellow Resin.

Analytical Constants.—Moisture, 8 to 10 per cent.; ash, 6 to 9 per cent.; total ether extract, 10 to 12 per cent.; volatile ether extract, 2 to 5 per cent.; alcoholic extract, 6 to 10 per cent.; crude fibre, 4 to 6 per cent.; starch by diastase method, 30 to 40 per cent.

Micro-examination. — The chief characteristics of powdered turmeric when examined under the microscope are the yellow masses of distinctly marked starch granules, cellular tissue and occasional fragments of intense yellow resin.

CHAPTER III

ANALYTICAL METHODS

The following are the most commonly used general chemical methods for the analysis of spices and condiments. Particular methods relating to special articles are dealt with later.

Moisture.—2 to 3 gms. of the spice are dried to constant weight in an air oven at 110° C. The loss in weight represents moisture and volatile oil, the latter being deducted after determination from the ether extract.

Ash.—2 to 3 gms. are incinerated in the usual manner in a platinum crucible and the ash weighed. The amount of *ash soluble in water* is estimated by treating the ash with 50 to 100 c.c. of boiling water, filtering on to a tared filter paper or Gooch crucible, drying and weighing. The amount of *ash soluble in hydrochloric acid* (sand) is estimated by boiling the ash with 40 to 50 c.c. of 10 per cent. hydrochloric acid, filtering on to a tared Gooch crucible, washing with hot water, drying and weighing. Inorganic bodies used as adulterants are estimated in the ash by the usual methods of quantitative analysis.

Ether Extract.—2 to 3 gms. of the dried powder from the moisture estimation are extracted with pure, dry ether in the Soxhlet apparatus. The ether is evaporated and the extract desiccated over sulphuric acid for a day. The extract is weighed for the total ether extract figure.

The container is then heated very gradually to 110° C. in an air oven and the heating continued until the weight is constant. The residue gives the non-volatile ether extract figure and the loss in weight the volatile oil figure.

Volatile oil is also estimated by distilling a mixture of the spice and water, the distillate being collected in a graduated cylinder, in which the amount of volatile oil can be read off. In cases where the volatile oil is slightly soluble in water special treatment is required. The water and volatile oil are separated and the aqueous portion extracted with petroleum ether. The extract is evaporated at a low temperature in a tared vessel and the amount of oil remaining added to the volume of oil read off in the cylinder.

Alcoholic Extract.—2 to 3 gms. of the spice are shaken occasionally for a day with 100 c.c. of 95 per cent. alcohol, the solution allowed to stand overnight and then filtered. A measured quantity of the filtrate is evaporated to dryness on a water bath and heated to constant weight at 110° C.

Starch.—A preliminary extraction is usually performed, except in the case of cassia and cinnamon, with ether and alcohol on 4 to 5 gms. of the spice. The residue is then transferred to a flask and the starch estimated by the diastase method or the acid conversion method. The diastase method is specially suitable for spices or condiments which contain only small quantities of starch.

Crude Fibre.—2 to 3 gms. of the spice are extracted with ether and then boiled successively with dilute sulphuric acid and dilute caustic potash. The liquor is diluted and centrifuged, the final residue being dried to constant weight at 110° C. in an air oven.

Nitrogen.—The Kjeldahl method is usually employed except in special cases.

Micro-examination.—The following methods of clearing dense tissues are sometimes used in the preparation of spices for examination under the microscope: (1) Standing in chloral hydrate solution for a day. (2) Successive treatment with boiling dilute acid and alkali. (3) A drop or two of weak alkali added to the water-mount of the substance.

A drop of a solution of iodine in potassium iodide introduced under the cover-glass is useful in distinguishing starch granules and amylo-dextrine. Starch granules turn black with iodine, amylo-dextrine granules, red.

SPECIAL METHODS

Cloves. — The *Tannin Equivalent*, *Oxygen Absorbed Figure*, or *Quercitannic Acid Content* of cloves is estimated by first extracting the spice with ether and then finding the oxidising power of potassium permanganate on an aqueous extract of the residue, proceeding in a similar manner as in the Lowenthal tannin estimation. 10 c.c. $\frac{N}{10}$ oxalic acid are equivalent to 0·0623 gms. quercitannic acid or 0·008 gms. oxygen absorbed.

Ginger.—Sophistication with cayenne pepper may be detected by boiling a small quantity of the sample with alcohol, filtering the extract and evaporating to dryness. The pungent taste of cayenne is at once apparent in the extract if the sample is adulterated with this substance.

Dyer and Gilbard (*Analyst*, 1893, 199) indicate the value of the *alcoholic extract* and *water-soluble ash* in detecting adulteration with exhausted ginger. In the analysis of pure ginger the alcoholic extract averaged

2·8 per cent., while exhausted ginger gave only 1·2 per cent. The water-soluble ash was 2·7 per cent. with pure ginger and 0·35 per cent. in the case of exhausted ginger.

Stock, in the *Analyst*, 1894, 312, makes some interesting remarks on the grinding of ginger roots for analytical purposes.

Mace.—Several chemical tests are in existence for the detection of the presence of Bombay mace in the Banda or true variety. When basic lead acetate is added to the alcoholic extract a red precipitate is obtained if Bombay mace is present; true mace gives a white precipitate. Although the petroleum ether extracts of Bombay and true mace are similar, Southall Brothers and Barclay have demonstrated that a subsequent extraction with ether gives 35 per cent. from Bombay mace compared with 1·7 per cent. from true mace. Solstein (*Zeits. Öffentl. Chem.*, 3, 253) recommends a similar process for detection and estimation of Bombay mace. Further chemical tests are given by Schneider, *Journ. Pharmacol.*, 4, 57; Hefelmann, *Pharm. Zeit.*, 1891, 122; Busse, *Zeits. Unters. Nahr. Genussm.*, 1904, 590; Cripps, *Analyst*, 1909, 519.

Macassar mace may be tested for by Griebel's method (*Zeits. Unters. Nahr. Genussm.*, 1909, 202) as follows: A small quantity is shaken with light petroleum, filtered and mixed with 2 c.c. glacial acetic acid. Concentrated sulphuric acid is carefully poured down the side of the tube to form a layer under the acetic acid solution, a red ring forming at the junction of the two solutions when Macassar mace is present in excess of 20 per cent.

Mustard.—D. Raquet (*Ann. Chim. Anal.*, 1912, 174–178) suggests the use of dilute alcohol in place of water in the distillation method of estimating essential oil in mustard as giving more concordant results. The following percentages of mustard oil (as allyl-thiocarbimide) were found in samples of black mustard of different origin: English, 1·39 per cent.; Greek, 1·2 per cent.: French, 1·08 per cent.; Sicilian, 0·99 per cent.; Italian, 0·99 per cent.; Bombay, 0·81 per cent.

A note on the *Iodine Values* of mustard oils is given in the *Analyst*, 1918, 216.

Cayenne pepper is detected in a similar manner to that mentioned above under ginger.

When turmeric is present in mustard the addition of ammonia produces an orange-red colour. Bohrisch, in the *Analyst*, 1904, 29, 372, gives an alcoholic extraction method for the detection of turmeric and aniline dyes in mustard. E. Sievers (*Zeits. Unters. Nahr. Genussm.*, 1912, 393) proposes a modification of Bohrisch's process for turmeric as follows: A few gms. of mustard are moistened with alcohol and 10 c.c. of ether added. The mixture is shaken, allowed to settle, the ethereal layer poured on to a strip of filter paper and tested for turmeric in the usual manner with boric acid.

Kreis (*Chem. Zeit.*, 1910, 34, 1021) has devised a method for the estimation of starch in mustard in which the starch is weighed directly.

Pepper.—Many chemical methods have been suggested for the detection of olive stones or "poivrette" in pepper, most of these depending on colour reactions.

Chevreau makes use of aniline acetate, which colours olive stones yellowish-brown and leaves the pepper greyish or white.

D. Martelli (*Analyst*, 1895, 181) uses phloroglucinol in hydrochloric acid, which when heated with olive stones imparts to them a brilliant red colour.

Jumeau employs a solution of iodine in equal parts of alcohol and ether. This gives a deep brown colour with pepper and yellow with olive stones.

Neuss treats the sample with hydrochloric acid, pepper being turned yellow, while most adulterants remain unchanged.

A method devised by A. W. Stokes (*Analyst*, 1889, 82) for the microscopic detection of poivrette and long pepper is as follows: A sample is warmed with ammonia, then washed with water and examined under the microscope in glycerine, using polarised light. With pepper it is possible to obtain an entirely dark field by rotating the prisms, this being impossible with poivrette, long pepper or rice.

CHAPTER IV

MISCELLANEOUS FLAVOURING MATERIALS

VEGETABLE BODIES

Agaric.—This is a white, spongy fungus, *Polyporus officinalis,* which grows on old larch trees in Eastern Europe and Siberia. Its chief constituent is agaracin (agaricic acid), a white crystalline powder which melts at 138° C., is insoluble in water, but soluble 1 : 140 in 90 per cent. alcohol. It possesses an astringent taste, but no characteristic odour, its chief usage as a flavouring agent being in beverages of a bitter nature.

Aloes.—These are derived from *Aloe chinensis, A. vera* and *A. Perryi*, known commercially as Socotrine, Zanzibar, or Curacao aloes. Cape aloes are not official and are used chiefly for veterinary purposes. Aloes occur in hard, brown masses, having a dull fracture. The chief supplies are obtained from the West Indies. They possess a characteristic, disagreeable odour and a bitter taste, being used chiefly in bitters. The chief constituents of aloes are aloin, emodin or trioxymethylanthraquinone, a small amount of gallic acid, resin and a trace (0·0003 per cent.) of volatile oil. Tests: Ash, not over 5 per cent.; moisture, not over 10 per cent.; 1 gm. of aloes warmed with 5 c.c. of 95 per cent. alcohol should give after cooling an almost clear solution. For tests for adulterants see Allen's *Commercial Organic Analysis,* 1913, vol. vii. 138, 146. Aloin tests are contained in the *Pharm. Journ.,* ii./10, 235.

Angelica Root and Seed.—The two main types are the European variety *Angelica archangelica,* and the American variety *Angelica atropurpurea.* The roots have the characteristic, aromatic angelica taste, the American being lighter coloured and less aromatic. The fruits or seeds are oblong, $\frac{1}{4}'' \times \frac{1}{8}''$, round at the ends and whitish in appearance. *Archangelica* contains 0·3 to 1·0 per cent. of an essential oil. Angelica has a large variety of uses as a flavouring agent; it is used in bitters and in liqueurs of the Vermouth, Chartreuse, Benedictine, Carmel Monks, etc., types.

Angostura Bark or Cusparia Bark.—This is the dried bark of *Cusparia febrifuga.* It occurs on the market in slightly curved and quilled pieces about $\frac{1}{8}''$ in thickness. It is yellowish in outer appearance, has a bitter flavour and is used as a bitter tonic and stimulant. Its properties are due to the alkaloids cusparine, cusparidine, galipin and galipoidine. In the *U.S.D. Arch. d. Pharm.,* 1911, 174, the account of an examination is given which revealed a content of cusparine, cuspareine and galipoidine (see also *Journ. Chem. Soc.,* 1921, i. 121).

Anise and Star Anise.—Anise is the dried ripe fruit of *Pimpinella anisum.* It is obtained chiefly from Spain, Italy, Russia, Malta and Germany. It has a sweet taste and characteristic odour, its chief use being as a stomachic, carminative and aromatic flavouring agent for many purposes. The flavour of anise is due to its content of an essential oil (1·5 to 6·0 per cent.). The fruits should be free from sand and earthy matter. Conium has occasionally been detected as an adulterant, its presence being revealed by the evolution of a mousy odour when the anise is ground with potash.

Star anise is the fruit of *Illicium verum*, which is obtained chiefly from China. It has a characteristic aromatic taste due to its content of volatile oil. Aniseed oil of commerce is, in fact, produced almost wholly from *Illicium verum*. *I. religiosum*, originating from Japan, is occasionally used as an adulterant. It has a flavour resembling turpentine. Anise and Star anise are used in liqueurs of the type of Anisette de Hollande, Anisette de Bordeaux, Absinthe, etc., also in cake-flavouring essences, bitter beverages, flavours of the "cough" type, etc.

Arnica.—This is derived from *Arnica montana* and consists of the dried flower-heads of the plant. Previous to 1914 the root was official in the *B.P.* It is obtained from the mountainous regions of Central and Southern Europe. Its taste is acrid and bitter, resembling apple to some extent, and its use is chiefly as a stomachic and weak stimulant. It contains a volatile oil to the extent of 0·5 to 1·0 per cent. Occasional adulteration occurs with the flowers of *Inula Britannica.*

Balm.—This is the fresh herb *Melissa officinalis*, which is grown in Mediterranean countries. It is used as a digestive, having been introduced by the Carmelite monks of Paris in 1611, in the form of Eau de Melisse des Carmes. A note on the historical interest of balm appears in the *Chem. & Drug.*, ii./09, 819. It has a flavour resembling lemon. The essential-oil content of balm is 0·014 to 0·104 per cent. It is used in bitter beverages, etc.

Buckbean.—This is the leaves of *Menyanthes trifoliata*, of the gentian species. It has a very bitter flavour, but no odour, being used chiefly as a bitter tonic and carthartic, in some cases along with wormwood and gentian.

Burdock.—This is the herb *Arctium lappa*, the whole herb, root and fruits being used. It has a sweet, mucilaginous taste and is used along with bitters in herbal beverages. The rhizome contains about 0·17 per cent. and the leaves 0·28 per cent. of essential oil.

Calamus or Sweet Flag.—This is the rhizome of *Acorus calamus*, grown in China, Japan and Russia. The roots are pale yellowish-brown in colour and have a bitter, aromatic flavour. The peeled root, which often appears on the market in a split condition, is less aromatic. It contains 0·8 to 3·5 per cent. of volatile oil. It is used in the compounding of bitters.

Calisaya.—This is a non-official cinchona bark derived from *Cinchona calisaya* or Yellow Cinchona. The bark, which is sold in quilled pieces, has a bitter flavour and slight odour. It is employed in the preparation of bitter beverages.

Caroba.—This consists of the leaves of *Jacaranda lancifoliata.* The leaves have a bitter taste, somewhat resembling tea, but no odour. According to the *Brit. Med. Journ.*, i./85, 327, they contain aromatic resin, but no alkaloid.

Cascarilla.—This is the dried bark of *Croton eluteria*, grown in the Bahamas, which occurs on the market in the form of short quills of about ½″ in diameter, having a chalky surface. The chief constituents are 1·5 to 3 per cent. of a volatile oil, tannin, resins, and also the bitter, crystalline substance cascarillin. The latter substance is most easily extracted by means of acetone. It is used like columba and gentian as an aromatic tonic possessing a bitter aromatic flavour. Tests: Ash is usually about 8 per cent. The *B.P.* ash-limit is 11 per cent.

Celery Seeds.—These are the seeds of *Apium graveolens*, cultivated in European countries. They are used chiefly as a culinary flavour, also in pickles, etc., and for their tonic properties combined with such drugs as damiana, coca and kola. They contain 2·3 to 5 per cent. of a volatile oil.

Centaury.—This comprises the whole herb *Erythræa centaurium.* It has a bitter flavour and a slight odour, which, however, disappears on drying the herb. It is used in bitters.

Chiretta.—This comprises the entire plant *Swertia chirata*, which is collected when in flower and dried. It is derived from Northern India, where it is extensively used as a bitter. It resembles in properties gentian, columba, etc. The active principles are chiratin, a bitter substance, and ophelic acid. No tannin is present. It is used as a bitter principle in beer.

Colocynth.—This is the dried pulp of *Citrullus colocynthis*, or bitter apple, free from seeds. The pulp is yellowish white, spongy and very bitter. It is obtained from Smyrna, Trieste, France, Spain, Egypt, Cyprus and Persia. Its chief constituents are a glucoside, colocynthin, and resinous matter. It is used in very small quantities in bitters. Tests: Ash, not *less* than 9 per cent.; aqueous extract should not give blue colour with iodine. The *B.P.* requires not more than 2 per cent. of fixed oil.

Columba.—This consists of the dried root of *Jateorhiza columba* or *J. palmata* cut into transverse slices. It is obtained from East African forests and has a bitter taste and musty odour. The chief constituents of columba root are a white crystalline substance columbin, an alkaloid berberine, columbic acid and starch. It contains 0·005 per cent. of an essential oil. It is used as a bitter principle in beverages.

Cubebs.—This is the dried, full-grown, unripe fruit of *Piper cubeba*, which resembles black pepper in many respects. It occurs on the market as dried berries, usually with the stalks attached. Java, Sumatra and Borneo are the chief sources of supply. It has a pleasant, aromatic odour, a rather bitter, pungent taste, and should be free from a mace-like flavour. Eastern peoples use cubebs as a condiment; it has, however, many applications both as a flavouring material in bitter beverages and as a stimulant and stomachic medicine. Its chief constituents are 8 to 18 per cent. of a volatile oil, about 20 per cent. of an oleo-resin (*Chem. & Drug.*, ii./09, 579), cubebic acid, cubebin, and a small amount of piperin. It is sometimes adulterated with similar shaped fruits of the *Rhamnus* and *Piper* species (*Chem. & Drug.*, ii./05, 797). Tests: Ash, not over 8 per cent. (*B.P.* limit); the ether extract should be about 20 per cent.

Dandelion.—This consists of the smooth, yellowish-brown root of *Taraxacum officinale*, collected in the autumn. It is a bitter like columba. Its chief constituents are taraxacin, asparagin, resins, salts, inulin and mannite. The dried roots are liable to attacks from insects, and should not be kept for more than a year; the juice of the root, also, undergoes alteration on exposure to air. Dandelion is a bitter tonic used in beverages, bitters, etc. It is also roasted, ground and used in place of coffee or as a dandelion coffee. Dandelion root resembles pellitory root, but has not its bitterness.

Elder Flowers.—These are the dried flowers of the European plant *Sambucus nigra*, separated from the stalks. The flowers blacken quickly if left in the undried condition. They contain about 0·037 per cent. of a volatile oil and are used as a household remedy for catarrh. Their taste is mucilaginous, and their odour slight, but characteristic. Elder bark is also used as a bitter principle to some extent; its taste is sweet at first, then bitter.

Elecampane Root.—This is derived from *Inula helenium*, which is grown in European countries. The root resembles belladonna in shape and appearance, but on scraping does not show white as does belladonna. The flavour is bitter and aromatic, the odour resembling camphor and orris root. Its chief constituents are inulin and helenin. It is used in the compounding of bitters.

Fennel.—This is the greenish-brown, dried, ripe fruit of *Fœniculum vulgare*, which is grown in Southern Europe. The fruit has a sweet aromatic flavour resembling anise, which, however, varies slightly according to derivation. Fennel is obtained from France and Germany and also India, Persia and Japan. Its chief constituent is its volatile oil, which it contains to the extent of 4 to 6 per cent. It is sometimes adulterated with the exhausted fruit. Its chief usage as a flavouring agent is in liqueurs of the anisette type, bitters, etc. Tests: Ash should not exceed 11 per cent., usually being between 8 per cent. and 10 per cent.

Galanga.—This is the dark red root of *Alpinia officinarum* and the *A. galanga*, which is obtained chiefly from the East Indies. Its flavour is spicy and pungent, resembling ginger. It is used in bitters of the angostura type and in liqueurs, such as vermouth.

Garlic.—This is the bulb of *Allium sativum*, which is grown in Spain, Portugal and Italy. It has the well-known flavour of onions, but much intensified. The essential-oil content of garlic is 0·005 to 0·01 per cent. It is used in culinary dishes, pickles, etc.

Gentian.—This comprises the dried rhizome of *Gentiana lutea,* the exterior of which is yellowish-brown, the interior being of an orange tint. It is obtained from the mountainous regions of Central and Southern Europe. Its flavour is sweet at first, then exceedingly bitter. The chief constituents are a very bitter glucoside gentiopicrin, gentianic acid, a sugar gentianose and a trace of volatile oil. Gentian has occasionally been adulterated with the root of the *Rumex alpinus,* and in the powdered state with ground olive stones. It is used as a bitter principle in beer and herbal beverages. Tests: Ash, not over 6 per cent. (*B.P.* limit); not less than 33 per cent. of the ash must be soluble in water; the aqueous extract of gentian should total 30 to 40 per cent.; for further tests see the *B.P.*

Ginseng.—This name is applied to the roots of several Asiatic plants, and also to *Panax quinquifolium* or False Ginseng. The Chinese ascribe to it extraordinary medicinal properties. It has a sweet, slightly aromatic taste and demulcent properties. The *Pharm. Journ.,* ii./10, 575, gives an account of its culture in Kentucky and its conveyance to China (see also *Journ. Chem. Soc.,* 1921, i. 296).

Guaiac Wood.—This comprises the heartwood of *Guaiacum officinale* or *G. sanctum,* which is of a dark, greenish-brown colour and heavier than water. It is grown chiefly in Jamaica and San Domingo, the wood being marketed in the form of shavings or raspings. It has an aromatic, biting taste and is used in certain bitter beverages. Its constituents are 26 per cent. of resin, containing guaiacic, guaiaconic and guaiacinic acids, also two saponins, a neutral guaiac-saponin and guaiacsaponic acid. The wood and resin have stimulating and diaphoretic properties. Tests: Ash, 1 to 2 per cent.; the alcoholic extract of the wood gives a blue coloration with dilute ferric chloride.

Holy Thistle.—This is the herb *Carbenia benedicta.* Its flavour is extremely bitter, but it possesses no distinctive odour.

Hops.—These are the flowers of *Humulus lupulus,* which are usually treated with sulphurous acid to prevent them going brown and developing an odour of valerian. The flavour of hops is bitter and aromatic; they are used chiefly as a flavouring agent in beverages. The bitter principles are in greatest predominance in the yellow glands of the axil of the fruit, and these, when separated by sifting, form the substance lupulin. Tests: Ash, 5 to 7 per cent.; ethereal extract, 9 to 15 per cent.; when distilled with water, should yield 0·9 per cent. volatile oil; the benzol extract is a good criterion of the quality of hops, usually varying from 12 to 19 per cent.

Horehound.—This is the herb *Marrubium vulgare,* which is used largely in the preparation of herbal beverages. It has a pleasant, aromatic, bitter taste and characteristic odour, also it possesses tonic properties.

Hyssop.—This is the herb *Hyssopus officinalis,* which is grown in European countries. It has an aromatic, bitter flavour and camphoraceous odour. Its essential-oil content is 0·3 to 0·9 per cent. The herb is used in the compounding of liqueurs of the Chartreuse, Benedictine, Carmel Monks, etc. types.

Juniper Berries.—These are the ripe fruits of the European plant *Juniperus communis.* They are nearly black in colour, with a greyish bloom. The flavour is characteristic, being of an aromatic, turpentine nature. The chief constituent is an essential oil (0·5 to 1·5 per cent.), composed mainly of terpenes. It is used in beverages.

Kola.—This is the dried fleshy seeds of *Cola vera,* obtained from West Africa and the West Indies. The seeds contain 1·0 to 2·5 per cent. of caffeine and also a glucoside, kolanin. They have an astringent flavour and are used in the manufacture of certain beverages. See also *Brit. Med. Journ.*, i./90, 969, and *Chem. & Drug.*, ii./09, 580.

Laurel.—Both the leaves and the fruits of the laurel, *Laurus nobilis,* are used. This laurel is grown chiefly in Europe, Syria and Asia Minor. The flavour is aromatic, its chief constituent being about 1 per cent. essential oil. The fruits contain about 30 per cent. of a fixed oil.

Lavender Flowers.—These are the flowers of *Lavandula vera* separated from the spikes. They are grown in England, France, Spain and Italy. Their characteristic lavender flavour is due to an essential oil which they contain to the extent of 0·5 to 1·0 per cent.

Liquorice.—This is the root of *Glycyrrhiza glabra,* obtained from Spain, Russia, Italy, Persia, Arabia, etc., and also grown to some extent in England. The root has varying degrees of acridity according to its source. It owes its characteristic flavour to the active principle glycyrrhizin, which occurs in the root in combination with ammonia. Other constituents are resin, starch, gum, grape sugar, asparagin and malic acid. Aqueous extraction of the root usually gives 24 to 38 per cent. solid extract. The following references deal with the analysis of liquorice root and extract: *Chem. & Drug.*, ii./09, 580, i./10, 21, i./11, 133; *Pharm. Journ.*, i./06, 494, i./13, 365, 370; *Am. Journ. Pharm.*, Dec. 1912; see also *B.P.* and *U.S.P.* for extraction tests. Ash, 3 to 4 per cent., not over 6 per cent. (*B.P.*). Liquorice has a very widespread use as a flavouring material; it is also used in beverages to produce a froth heading.

Lovage.—This is the grey-brown root of *Levisticum officinale.* It has a very thick, white spongy bark. Its flavour is slightly bitter, resembling fenugreek. It contains 0·3 to 1·0 per cent. of a volatile oil.

Manna.—This is the pale, yellow, concrete saccharine exudation from *Fraxinus ornus* and *F. rotundifolia.* It has a sweet flavour similar to honey and consists mainly (70 to 80 per cent.) of mannite, sugar, extractive matter and 10 per cent. moisture. Mannite is not fermented by yeast, also it does not reduce Fehling's solution.

Marjoram.—This is derived from the herb *Origanum majorana*, which is grown in European countries. It is used chiefly as a culinary flavour, being agreeably aromatic in taste. It contains 0·7 to 0·9 per cent. of a volatile oil.

Musk Seeds.—These are the greyish-brown seeds of *Hibiscus abelmoschus*, which is grown in the East and West Indies and Java. They are used as a coffee flavour and in the East as an adulterant for musk. Their flavour is greasy and distinctly of a musk type. They contain about 0·2 per cent. volatile oil.

Orris Root.—This is the root of *Iris florentina.* It has a fine, violet odour, being used in many flavouring essences and in liqueurs of the anisette type. The best quality of orris root is Florentine. It contains "butter of orris," a sweet, violet-scented fatty material, to the extent of 0·10 to 0·20 per cent.

Paradise Grains.—These are the seeds of *Amomum melegueta*, which is grown in West Africa. They are used in bitters and contain 0·3 to 0·7 per cent. of a volatile oil.

Pellitory Root.—This is the dried, brown root of *Anacyclus Pyrethrum*, derived from Morocco and Algeria. It has a pungent, acrid taste, a characteristic odour, and is used as a salivatory stimulant in bitters, etc. Its chief constituents are a volatile oil, resins and inulin. It has been adulterated with the root of the *Corrigiola telephiifolia*, which is similar in size and is obtained from Morocco.

Peppermint.—This is the herb *Mentha piperita*, which is grown in America, England, France, etc. Its chief constituent is a volatile oil rich in menthol and menthyl esters, which is distilled from the fresh, flowering herb. The flavour is aromatic, with cold pungency. It has a widespread usage in many types of flavours.

Polypody.—This consists of the root and leaves of *Polypodium vulgare*. The root is slender and dark brown in appearance, its taste is slightly acrid and sweet, but it is devoid of odour.

Pomegranate.—This is the dried root and root-bark of *Punica granatum*. It has an astringent flavour, but no odour. Its chief constituents are 0·5 to 0·7 per cent., and even as high as 1 per cent. of alkaloids, composed chiefly of pelletierine, etc., and 20 per cent. of tannin. It is used to some extent in the compounding of bitters. Tests: Ash, 5 to 13 per cent., not over 15 per cent. See Squire's *Comp. to B.P.*, p. 671, for summary of further tests.

Prickly Ash Berries and Bark.—These are the fruit and bark of *Xanthoxylum americanum*, or Toothache Tree. They have a very pungent, bitter taste and cause a flow of saliva. They are used in herbal beverages.

Pyrethrum Root.—See *Pellitory Root.*

Quassia.—This is the wood of *Picræna excelsa*, obtained from Jamaica. The wood is sold in yellowish-white chips or raspings, it has a very bitter taste, resembling, but much more bitter than, sassafras and columba. It is used as a bitter principle in beer. Its chief constituents are the bitter principle "quassin" and an essential oil. Quassia is sometimes adulterated with the exhausted wood. Tests: Ash about 3 per cent., not over 4 per cent.

Red Peruvian Bark.—This is the bark of *Cinchona succirubra,* which is usually sold in quilled pieces. It is bitter and astringent in flavour and has a slight odour due to its containing a small quantity of essential oil. Its chief constituents are quinine, quinidine, cinchonine and cinchonidine. Good red bark should yield 5 to 6 per cent. of alkaloids, not less than half of which should be quinine and cinchonidine. It is used in bitter beverages of a tonic character. Tests: Ash, 2 to 4 per cent.; for alkaloid-strength tests see *B.P.*, *U.S.P.*, etc. Assay methods are also given in *Pharm. Journ.*, ii./05, 579, and *Chem & Drug.*, i./08, 21. The alpha-naphthol test for cinchona alkaloids is described in *Am. Journ. Pharm.*, 1913, 502; *Pharm. Journ.*, ii./13, 881; and *Chem. & Drug.*, i./14, 84.

Rhubarb.—This is the root of *Rheum palmatum*, *R. officinale* and other species. It is obtained from China, Turkey and the East Indies. It has a nauseous, astringent taste and is used as a flavouring matter in bitters. Notes on the constituents of rhubarb are to be found in *Journ. Chem. Soc., Trans.*, 1911, 946; *Pharm. Journ.*, i./13, 403. Tests: Ash, 7 to 12 per cent., although Chattaway and Moor (*Analyst*, 1903, 207) found nearly 30 per cent. The *B.P.* ash limit is 15 per cent. A colorimetric method for the assay of rhubarb is given in *Pharm. Journ.*, ii./05, 580.

Roman Chamomile.—These are the buff, dried, expanded flower-heads of *Anthemis nobilis,* which is grown in

Europe and America. The flavour is bitter and aromatic, the flowers being used in the preparation of herbal beverages. They contain 0·8 to 1·0 per cent. of volatile oil. The constituents of the flowers are given in *Journ. Chem. Soc., Trans.*, 1914, 1844. Tests: The ash should not exceed 5 per cent.

Rosemary.—This is the herb *Rosmarinus officinalis*, which is grown in Spain, France and Italy. It has an aromatic taste and characteristic odour, due to its content of 1 to 2 per cent. of a volatile oil. It is used in many flavouring essences, also in the compounding of bitters, etc.

Sage.—This is the herb *Salvia officinalis*, which is grown in European countries. It is used chiefly as a culinary flavour. Its content of volatile oil amounts to 1·3 to 2·5 per cent.

Sarsaparilla.—This is the brown root of *Smilax ornata.* Several varieties are to be found on the market, *e.g.* Guayaquil, Mexican, Honduras, Red Jamaica, Orange or Native Jamaica, Lima, etc. The root has long been used as a tonic and blood purifier, particularly in the form of bitter beverages. The flavour is slightly acrid. It contains a saponin glucoside, sarsasaponin, and a phytosterolin (*Journ. Chem. Soc., Trans.*, 1914, 219). Tests: Ash, 5 to 7 per cent.

Sassafras.—This comprises the dried root of *Sassafras officinale* or *S. variifolium*, which is grown in North America. The root bark has a rust-brown colour, is aromatic, sweet and slightly astringent in flavour. The whole root is sold in the form of chips. The flavour is due to the presence of 3 to 9 per cent. of a volatile oil, which is usually distilled from the root bark. Sassafras is used as a flavouring material for herbal beverages and in the compounding of liqueurs of the anisette type. Tests: Ash, about 2 per cent.

Saxifrage.—This is the herb *Pimpinella saxifrage.* It is cucumber-like in flavour, but has not much odour. It is used in the preparation of bitter beverages.

Senega. — This is the dried yellowish-grey root of *Polygala senega*, which is obtained from North America. It has an acrid, slightly acid flavour, resembling methyl salicylate, and a rather rancid odour. Its chief constituents are senegin, a glucoside of a saponin nature, and polygalic acid; the volatile-oil content of the root is 0·25 to 0·33 per cent. Tests: Ash, 2 to 5 per cent.; when extracted with ether, acidified with hydrochloric acid, water added and the ether evaporated off, the aqueous solution should give a violet-red colour with a drop of dilute ferric chloride solution. The following roots have some resemblance in appearance to senega: Valerian, arnica, serpentary and green hellebore.

Senna.—This consists of the leaves and pods of *Cassia angustifolia* (Tinnevelly or East Indian Senna) and *C. acutifolia* (Alexandrian Senna). The latter are usually preferred as being milder flavoured; the taste is sickly sweet, with a resemblance to tea. Senna is used in the compounding of certain bitters. The chief constituents of senna are carthartic acid, sennacrol, sennapicrin, chrysophanic acid, emodin and cartharto-mannite (sugar). See *Pharm. Journ.*, ii./13, 741, and *Chem. & Drug.*, ii./13, 743, for further description of constituents.

Simaruba Bark.—This is the root bark of *Simaruba officinalis* or Mountain Damson. It is very bitter and astringent to the taste, but has no odour. The chief constituents are a crystalline, bitter glucoside, a yellow resin and a brown resin (*Chem. & Drug.*, i./08, 600).

Squaw Vine or Partridge Vine (*Checkerberry*).—This is the herb *Mitchella repens.* It has a bitter, astringent flavour, but no odour. It has been used in flavouring various materials, *e.g.* herbal beverages.

Tamarinds.—These are the reddish-brown fruit of *Tamarindus indica*, freed from the brittle outer pericarps. Several varieties are on the market, *e.g.* West Indian, East Indian and Egyptian. The West Indian type are preserved in sugar; the East Indian are not preserved and the Egyptian are marketed in a pressed condition. The latter two varieties are used largely in pickles, sauces, etc. The chief constituents are tartaric acid, citric acid, potassium tartrate, acetic acid and sugar. Tests: They should contain acid equivalent to 10 per cent. calculated as tartaric acid, also the fruits should be free from copper.

Tansy.—This is the flowering herb, *Tanacetum vulgare*, which is grown in Europe and North America. It has a strong, unpleasant odour and a bitter, aromatic taste. Its content of essential oil is 0·1 to 0·3 per cent.

Thyme.—This is the fresh herb *Thymus vulgaris*, which is grown in European countries. It has a characteristic aromatic flavour and is extensively used for culinary purposes. Its flavour is due to its content of 0·3 to 2·6 per cent. of a volatile oil.

Tonka Beans.—These are the seeds of *Dipteryx odorata*, which is indigenous to tropical South America. They contain the well-known and widely used flavouring material coumarin, to the extent of about 1·5 per cent., and also sugar, malic acid, fixed oil, gum and fibre (*Journ. Pharm. Chim.*, ii. 480). The beans vary in size and appearance according to derivation. Surinam beans are grey and large; Angostura are slenderer, long and shining, Para being shorter and smaller. In their preparation for the market, the beans are soaked in rum and this causes them to shrink and assume a grey efflorescence composed of crystals of coumarin. The odour of both tonka beans and coumarin resembles that of new-mown hay. The beans have a wide range of applicability in flavouring materials suitable for use in confectionery, tobacco, etc., as well as being occasionally employed to cheapen vanilla extract.

Valerian.—This is the dried, erect root of *Valeriana officinalis* and *V. wallichii* collected in autumn. The plants are grown chiefly in Europe and Asia. The root has a strong, unpleasant odour which develops during drying, and a camphoraceous, bitter taste (*Journ. Chem. Soc.*, 1921, i. 488). Its volatile-oil content is 0·5 to 1·0 per cent. Tests: Ash, 8 to 10 per cent.

Vanilla.—Vanilla beans of commerce are the dried, fermented pods of the climbing orchids *Vanilla planifolia* and *V. pompona*, which are indigenous to tropical America. They are chiefly cultivated in Mexico, Reunion, Tahiti, Java, the Seychelles, Fiji and the West Indies. The fruit is gathered when ripe in the autumn, and it is fermented and dried, either by the sun or in warm rooms, the method of process depending on the customary practice in the production areas (*Agric. News*, vi. 291).

The beans, as they appear on the market, are brown-black in colour, 8″ to 10″ long and $\frac{1}{4}''$ to $\frac{3}{8}''$ thick. The longest varieties, *e.g.* Mexican, are usually the best, these frequently having a coating of fine vanillin crystals, which is termed frost or "givre." Busse, *Zeits. Unter. Nahr. Genussm.*, 1899, 519, gives the following figures as regards vanillin-content for vanillas of various derivations: Mexican, 1·69 to 1·86; Bourbon, 1·91 to 2·90; Java, 2·75; Ceylon, 1·45; German East Africa, 2·16; Tahiti, 1·55 to 2·02. Other constituents of vanilla beans are vanillic acid, resin, sugar, fat and mineral matter. Exhausted vanilla beans coated with crystals of benzoic acid, which may readily be detected by the microscope, are sometimes offered on the market.

Vanilla extract is prepared from vanilla beans, alcohol and sugar in the following approximate proportions: Vanilla beans sliced, 10: sugar, 20: alcohol, 65; water, 35. Sometimes glycerine is substituted for the sugar, the colour of the extract being thereby deepened owing to more extractive matter being drawn from the beans. Dilute alkali is also occasionally used in extracting vanilla beans, an economy in the amount of alcohol required being effected; the quality of the essence, however, suffers considerably.

Vanilla extracts are sophisticated by the use of coumarin, tonka bean, caramel, acetanilide, etc. Vanilla extract gives a heavy precipitate with lead acetate; extracts which do not contain natural vanilla usually give no precipitate or only give a slight cloudiness due to the presence of caramel. Hess (*Journ. Am. Chem. Soc.*, 1899, 21, 721) precipitates the resinous matter from the extract by means of dilute hydrochloric acid. After separation, this resin is re-dissolved in alcohol and a few drops of ferric chloride added. If the extract is genuine no coloured precipitate should be obtained.

Several methods have been devised for the estimation of vanillin and the detection and estimation of coumarin in vanilla extracts. The following is an example: 50 gms. of the extract are diluted with 30 c.c. of water, evaporated to 50 c.c. on the water bath, again diluted with 30 c.c. of water and evaporated to 50 c.c. Twenty-five c.c. of lead acetate (containing 8 gms. of crystalline lead acetate per litre) are added and the solution made up to 100 c.c. After shaking, the mixture is allowed to stand for a day and filtered. Fifty c.c. of the filtrate are treated with 65 c.c. of ether, in small quantities at a time, in a separating funnel. The ethereal extract is shaken several times with 2 per cent. ammonia solution (up to 20 c.c. being used). The residual ether extract is evaporated in a tared dish at 20° to 25° C., dried over sulphuric acid and weighed as *Coumarin*. The coumarin should be soluble in petroleum ether; if, however, a residue remains after treatment with this solvent it should be weighed and tested for *Acetanilide*. The above-mentioned ammoniacal solution, after separation from the ether extract, is slightly acidified with hydrochloric acid and shaken in a separating funnel with ether as in the previous extraction. The ether extract is evaporated at a low temperature, dried over sulphuric acid and weighed as *Vanillin*.

T. von. Fellenberg has devised a process, which is given in the *Journ. Chem. Soc.*, 1916, ii. 355, for the colorimetric estimation of vanillin in vanilla extract.

For the detection of coumarin in vanilla extract, Wichmann distils the extract, evaporates the distillate to dryness and fuses the residue with caustic potash, thus converting the coumarin into potassium salicylate, which can be estimated colorimetrically, using ferric chloride solution. Dean, in the *Journ. Ind. Eng. Chem.*, 1915, 519, proposes a method which is not interfered with by saccharine or salicylic acid (see also *Analyst*, 1918, 325).

Winton, Albright and Berry (*Journ. Ind. Eng. Chem.*, 1915, 516 to 519) give the results of the estimation of acidity and ash of a number of samples of vanilla extract prepared from several varieties of vanilla beans. Methods for the estimation of vanillin in flavouring extracts are given in the *Analyst*, 1912, 501; 1917, 17, 208.

Virginia Snake Root.—This is the root of *Aristolochia serpentaria* or serpentary rhizome, which is obtained from the southern regions of North America. It is a bitter, with stomachic properties, after the type of columba; its taste is aromatic and bitter, resembling to some extent valerian, camphor and turpentine. Its essential-oil content is 1·0 to 2·0 per cent. Tests: Ash, 7 to 10 per cent.

White Cinnamon.—This is the fawn-coloured, quilled bark of *Canella alba*, which is grown in the West Indies. It has a biting, aromatic odour, resembling cinnamon. Its volatile-oil content amounts to 0·75 to 1·25 per cent.

Wild Cherry Bark.—This is the greenish-brown bark of the North American plant *Prunus serotina* or Virginian Prune, collected in the autumn. It contains amygdalin and emulsin, which develop hydrocyanic acid when mixed with water. Its flavour, when soaked in water, is of a bitter almond type. It is said to yield 0·2 per cent. of volatile oil and 0·075 per cent. of hydrocyanic acid. A syrup prepared from wild cherry bark is used for covering the taste of nauseous drugs. The bark is also used in herbal beverages. Tests: Ash, 3 to 4 per cent., not over 6 per cent. For the identification of spurious cherry barks, see *Pharm. Journ.*, i./09, 192, ii./10, 604.

Woodruff.—This is the herb *Asperula odorata.* It has a flavour resembling coumarin.

Wormwood.—This comprises the leaves and flowering tops of the herb *Artemisia absinthium*, which is grown in Europe and North America. It has a very bitter taste and an odour resembling thujone. It contains a bitter principle absinthin, which is slightly soluble in water, but easily soluble in alcohol, ether and chloroform. Its volatile-oil content is 0·5 per cent. It is used in bitters and, together with anise, coriander, etc., in the liqueur absinthe, also, along with angelica, etc., in vermouth.

Yarrow.—This is the herb *Achillea millefolium.* It has an insipid flavour and a very weak odour. It contains about 0·23 per cent. of a volatile oil.

Zedoary Root.—This is the root of *Curcuma zedoria*, which is grown in Ceylon. It has a bitter and camphoraceous flavour, and an odour resembling ginger and cardamoms. It is used in bitters. Its volatile-oil content varies between 0·06 and 0·25 per cent.

BALSAMS, RESINS, ETC.

Benzoin.—This is a resin derived from the tree *Styrax benzoin*, which is grown in Sumatra, Siam, Penang, Java, Saigon, Palembang, etc. The resin exudes from incisions in the trees and is exported either in masses or tears. Cocking and Kettle at the British Pharmaceutical Conference (see *P. & E.O.R.*, 1914, 331) gave the analytical constants of benzoin of various derivations (see also *Journ. Chem. Soc.*, 1921, i. 351, 352). Sumatra benzoin contains up to 18 per cent. free and combined benzoic acid and rather more free and combined cinnamic acid. Siam benzoin contains up to nearly 40 per cent. free and combined benzoic acid. Other constituents of benzoin are vanillin, phenyl-propyl-cinnamate, styrol, etc.

Peru Balsam.—This is a dark brown, thick liquid, which is obtained from the trunk of *Myroxylon pereira*, a tree growing in the forests of San Salvador. The method of extracting the balsam is described in the *P & E.O.R.*, 1918, 281. It has a bitter taste and an aromatic odour. Its chief constituent is cinnamein (55 to 65 per cent.), which consists of a mixture of benzyl benzoate and benzyl cinnamate. It also contains resins and alcohols. A description of the methods of testing Peru Balsam are given in the *P. & E.O.R.*, 1912, 203 (see also *Journ. Chem. Soc.*, 1921, ii. 226). The *B.P.* specific gravity limits are 1·140 to 1·158. As regards the alcohol contained in the balsam, namely, peruviol, this was isolated in 1899 by Thoms, and has been investigated by Schimmel & Co. It has the following characters: specific gravity, 1·1200; optical rotation, 0° 55′; refractive index, 1·5718; acid value, 36·4; ester value, 228·2.

Styrax.—This oleo-resin is derived from the inner bark of *Liquidambar orientalis,* which grows in forests in south-west Asia Minor. Its chief constituents are cinnamic acid, both free and combined, and cinnamic alcohol. Much styrax has been sold from which the odorous constituents have been extracted. The oleo-resin is grey in colour and contains 20 to 30 per cent. water. Its content of cinnamic acid is 20 to 30 per cent. It is purified by dissolving in ether or alcohol, filtering and distilling off the solvent. See *Analyst*, 1912, 499, for a description of the examination of styrax.

Tolu Balsam.—This is obtained by making incisions in the trunk of *Myroxylon toluifera*, which grow in New Granada and Venezuela. It is at first soft, but later becomes harder and finally becomes brittle. Its chief constituents are free and combined cinnamic acid (about 35 per cent.), benzyl benzoate, benzyl cinnamate, vanillin, a volatile oil and resin. The *P. & E.O.R.* gives analytical notes in the following issues: 1911, 277; 1912, 202; 1913, 293; 1915, 89, 124.

ANIMAL PRODUCTS

Ambergris.—This is an excretion of a morbid nature from the sperm whale, *Physeter macrocephalus.* It is obtained chiefly from the coasts of Sumatra, Java, and Madagascar. The grey variety, which is the most valuable, is opaque, solid, and with a granular fracture. It is frequently streaked with yellow. The black variety is plastic to the touch. Ambergris contains about 85 per cent. ambreine, which is soluble in alcohol; it also contains benzoic acid. It has a specific gravity of 0·91.

Castoreum.—This consists of the dried, preputial follicles of the beaver, *Castor fiber*, and is obtained from Canada, Russia, Siberia, etc. It is placed on the market in sacs, 2″ to 3″ in length, the contents of which are reddish-brown in colour and have a characteristic odour. Castoreum varies in composition according to derivation. The chief constituents are a resinous body (40 to 70 per cent.) which is soluble in alcohol, a volatile oil, benzoic acid, castorin (*Bull. de Pharm.*, 1897), salicin, salicylic aldehyde, albuminous matter, inorganic salts, and mucus. Adulteration is sometimes practised with resin, dried blood, wood fibre (*P. & E.O.R.*, 1913, 14), etc. Castoreum is liable to deterioration unless kept perfectly dry and in a cool place.

Civet.—This substance is obtained from the civet glands of several species of cats. The bulk of the civet of commerce is obtained from the African or Abyssinian civet cat, *Viverra civetta*: other species yielding civet are the Indian cat, *V. zibetta*; the Burmese cat, *V. megaspila*; the Malabar cat, *V. civettina*; the Chinese cat, *V. malaccensis*; the Javan cat, *V. tangalunga*, etc. Civet is usually semi-solid and dark-coloured when it arrives on the market. It also has a very disagreeable odour. Its use is practically confined to fixing other perfumes, being employed in very small amounts in certain extracts and essences. It is subject to adulteration with fats of both mineral and vegetable origin, and also banana pulp. When dried and extracted with hot acetone it should yield 80 per cent. extract. The following are the chief characters of civet: Moisture, 10 to 30 per cent.; ash, 1 to 2 per cent.; petroleum ether extract, 80 per cent. upwards (*P. & E.O.R.*, 1913, 348); acid value of petroleum ether extract, 130 to 150; melting point, 43° to 46°; saponification value, 130 to 150.

Musk.—This is the dried secretion of the preputial follicles of the musk deer, *Moschus moschiferus*, obtained from Central Asia. The most valued variety is Tonquin musk. "Grain" musk is obtained by drying the contents of the pods, which are cut from the animals. The odour of natural musk is due to its content of 0·5 to 2 per cent. of a ketone-like substance muskone (*P. & E.O.R.*, 1916, 133), besides which is present also fatty matter, proteins and resins. Tests: Ash, not more than 8 per cent.; moisture, about 30 per cent. The *U.S.P.* limits moisture to 15 per cent., the figure being obtained by drying over sulphuric acid. 10 to 12 per cent. is soluble in 95 per cent. alcohol, and 50 to 75 per cent. is soluble in water. The *Swiss P.* states that about 10 per cent. is soluble in 90 per cent. alcohol and 50 per cent. in water.

SECTION II

ESSENTIAL OILS

CHAPTER V

THE PREPARATION AND ANALYSIS OF ESSENTIAL OILS

The three chief processes used in the recovery of essential oils are (1) distillation, (2) extraction and (3) expression. The process used varies according to the material under treatment, the particular nature of the oil and its resistance to varying degrees of heat.

Distillation.—The majority of essential oils are obtained by distillation processes. The old method of distillation consisted in heating over an open fire a large still containing the vegetable material and water. This has been displaced to a great extent by steam distillation, using either saturated steam under pressure or superheated steam. The general principle of such distillation is to heat the material in the still by means of a steam coil and at the same time to blow steam through the mass. The vegetable material is usually separated from the bottom of the still by a false bottom, through the perforations of which live steam is blown from a perforated coil situated beneath.

Distillation under reduced pressure is practised in some cases where the volatile constituents of the oil are subject to decomposition. Terpeneless and sesquiterpeneless oils are produced in this manner.

Certain processes of continuous distillation have been devised for the production of essential oils. The distillation of essential oils is, however, a matter of considerable skill and experience; variations have to be made in the methods of distillation according to the material under treatment. The preparation of the vegetable material for distillation also requires experience; for example, seeds, barks, rhizomes, etc., are usually dried and ground, woods are rasped or ground, and green, soft material is distilled in many cases without preliminary treatment.

Extraction.—The preparation of essential oils by extraction from flowers, etc., with volatile solvents is described later in Section III.

Expression.—This method is used particularly in the case of the citrus oils, *e.g.* lemon, bergamot, orange, etc. The procedures adopted vary in different countries. In some cases the rinds of the fruits are placed in cloths and subjected to great pressure. This squeezes out a cloudy fluid which is allowed to stand in tall vessels for several days for separation to occur, the atmosphere being excluded as much as possible.

One of the chief expression processes is the sponge, *éponge* or *spugna* method, which is used principally in Sicily and Southern Italy. This consists in ejecting the essential oil from the glands just under the skin of the fruit by pressing the convex pieces of rind against a sponge until they become practically flat. The sponge absorbs the oil and is squeezed periodically. The *Scorzetta* method is similar to the above, except that the fruits are halved, and the skins after pressing remain unbroken, being subsequently packed in brine and exported for use in the production of preserved and candied peels.

The *écuelle* method of obtaining citrus oils, which is practised in France and Northern Italy, consists in rolling the fruits in a bowl-shaped vessel, the interior of which is covered with short spikes. The oil cells are ruptured and the oil is collected from the apparatus in a tube.

Several machine processes for the production of citrus oils have been invented and worked with varying degrees of success. The *P. & E.O.R.*, 1916, 73, makes reference to a method of extracting lemon oil in which the lemons in a broken condition are treated with a certain acid which causes the oil to separate and rise to the surface. About 2·5 per cent. more oil is said to be obtained by the process than by the sponge method.

GENERAL ANALYTICAL METHODS

Physical Tests.—*Specific Gravity.*—This is usually determined on the Westphal balance at a temperature of 15° C. In the case of small quantities the determination is made by means of a specific gravity bottle, Sprengel tube or pycnometer. Oils which are solid at 15° C., such as aniseed and otto of rose, are tested at 20° C.

Optical Rotation.—This is determined at 20° C., using a 100 mm. tube in a polarimeter reading in angular degrees. The specific rotation or $[\alpha]_D$ is calculated by dividing the angular rotation by the density of the oil.

Refractive Index.—This is usually determined at a temperature of 20° C. on an instrument of the Abbé type. It is sometimes more convenient to make the test on some oils at a temperature of 25° C.; these oils are noted in Chapter II.

Congealing-Point.—This is a test which, along with the melting-point, is of great importance in the testing of aniseed oils. The determination is made by taking 5 c.c. of the oil in a test tube and immersing same in a cooling bath at a temperature of 10° C. It is stirred by means of a thermometer graduated in tenths of a degree. When the oil begins to solidify, the tube is removed and placed in a bath at a temperature of 14° C. and the oil stirred continuously. The temperature rises and then falls again, the maximum point reached being the congealing-point. Several tests are made and the mean taken. When a similar determination is to be made on fennel oil it is usual to employ a freezing mixture of ice and salt or to maintain the temperature at 0° or lower.

Melting-Point.—This is determined by placing the previously congealed oil contained in a test tube in a water bath at a temperature approaching that at which the oil should melt, and very gradually increasing the temperature of the water bath. The temperature at which the oil is just melted is read off on a thermometer graduated in tenths.

Boiling-Point.—This is determined by distilling a small quantity of the oil in an ordinary distillation flask, noting the temperature registered and correcting to a pressure of 760 mm. Extreme accuracy is obtained if the portion of the thermometer which is not surrounded by the vapour is allowed for according to the formula

$$T = T' + n\,(0{\cdot}000143\,t)$$

T' being the temperature observed, n the number of degrees exposed and t the temperature of the exposed part of the thermometer.

CHEMICAL TESTS

General Methods.—*Esters.*—The ester value of an essential oil is the difference between its acid value and its saponification value. The *acid value*, which is the number of milligrams of potash required to neutralise 1 gm. of oil, is estimated by dissolving 3 to 5 gms. of the oil in 15 to 20 c.c. of neutral alcohol and titrating with N/10 alcoholic potash, using phenol phthalein as indicator. The *saponification value* is estimated by boiling 3 to 5 gms. of the oil with 20 c.c. normal alcoholic potash under a reflux condenser for one hour and titrating the excess of alkali with normal sulphuric acid. The result is expressed in terms of the number of milligrams of potash absorbed by 1 gm. of the oil. The percentage of esters in an oil, which is usually expressed as the ester present in predominating quantity, is calculated from the formula

$$\frac{M \times N \times 100,}{1000 \times W}$$

M being the molecular weight of the ester, N the number of c.c. normal caustic potash absorbed and W the weight of the oil.

An interesting report is given in the *P. & E.O.R.*, 1915, 218, of a research on the rate of saponification of linalyl, terpinyl and geranyl acetates. Terpinyl acetate saponifies much more slowly than geranyl and linalyl acetates. The necessity of using efficient condensers in the saponification process is pointed out in the *P. & E.O.R.*, 1914, 251.

Alcohols.—The usual method of estimating the percentage of alcohols in an essential oil is by acetylation followed by saponification. The method is as follows: Ten c.c. of the oil are boiled with 20 c.c. of acetic anhydride and 2 gms. of anhydrous sodium acetate, under a reflux condenser, for two hours. The liquid is diluted with about 100 c.c. of water and allowed to stand for several hours in the water bath. The oil is then washed in a separating funnel with several lots of distilled water or with brine containing 1 per cent. of caustic potash. Finally, the oil is dried over anhydrous sodium sulphate and filtered. The percentage of esters in the acetylated oil is then estimated by a similar saponification process to that given above for *Esters.* The percentage of alcohols is calculated from the formula

$$\frac{M \times N}{10\,(W - 0{\cdot}042N)}$$

M being the molecular weight of the alcohol, N the number of c.c. of normal caustic potash absorbed and W the weight of the acetylated oil taken. The formula only answers when the original oil contains no esters. T. T. Cocking has an interesting method of calculating the percentage of alcohols in presence of esters in the *P. & E.O.R.*, 1918, 37.

It is important if concordant results are to be obtained that the acetic anhydride and anhydrous sodium acetate used are of a high degree of purity. It is advisable that freshly fused sodium acetate be used and that the acetic anhydride should have a specific gravity of at least 1·080 and contain upwards of 95 per cent. of acetic anhydride. Low results are given by some alcohols, *e.g.* linalol and terpineol, owing to decomposition. Also, it is possible to estimate citronellal by this method as this aldehyde is converted quantitatively into isopulegyl acetate by acetylation.

The determination of citronellol and citronellal in essential oils by a process of formylation has been used quite extensively of late years. C. T. Bennett, in the *P. & E.O.R.*, 1921, 12, 351, points out, however, that this formic acid method is absolutely untrustworthy.

Aldehydes and Ketones.—(*a*) The old method consists in absorbing the aldehyde or ketone by means of a 35 per cent. solution of sodium bisulphite. Five to 10 c.c. of the oil are measured into a 200 c.c. flask, which has a neck graduated in 1/10th c.c. One hundred and fifty c.c. of the bisulphite solution are added and the mixture heated on the water bath for one to two hours with constant shaking, or until the solid bisulphite-aldehyde (or -ketone) compound formed is dissolved. The uncombined oil is forced into the graduated neck by adding more solution. The flask and its contents are allowed to cool for twelve hours and the volume of uncombined oil read off. A simple calculation gives the percentage of aldehyde or ketone which has been absorbed by the bisulphite solution. This method is used for cassia and lemon-grass oils.

(*b*) The *B.P.* 1914 recommends the following neutral sulphite method for the determination of aldehydes in cinnamon and lemon-grass oils: 5 c.c. of the oil are carefully measured into a similar flask to the one used in method (*a*). Twenty-five c.c. of a 20 per cent. solution of sodium sulphite and several drops of phenol-phthalein solution are added. The mixture is heated on a water bath with constant shaking, a 10 per cent. solution of acetic acid being added from time to time in sufficient quantity to keep the contents of the flask neutral. When no further alkali is liberated, which is usually the case at the end of about fifty minutes, the unabsorbed oil is forced into the graduated neck of the flask and measured after cooling for twelve hours.

Phenols.—Five to 10 c.c. of the oil are placed in a flask similar to that used in the determination of aldehydes and ketones and 100 c.c. of 5 per cent. caustic potash solution added. The mixture is heated in a water bath for one hour, with frequent shaking. The unabsorbed oil is then forced into the graduated neck of the flask and measured after cooling for twelve hours. The "absorbed" portion of the oil represents phenols and a simple calculation gives the percentage contained in the oil. The phenols contained in pimento, clove, thyme, origanum, bay, cinnamon leaf, etc. oils are estimated in this manner.

Adulterants.—The chief adulterants used in the sophistication of essential oils are resin, mineral oil, alcohol, acetone, chloroform, artificial esters such as terpinyl acetate, etc., turpentine oil, fatty oils, terpenes, copaiba oil, gurjun balsam oil, cedarwood oil, etc. *Resin* may be detected and estimated in cassia oil by adding a solution of lead acetate in 70 per cent. alcohol to a solution of the oil in 70 per cent. alcohol and weighing the precipitate formed (*P. & E.O.R.*, 1914, 264). *Mineral Oil* is less soluble in alcohol than most essential oils; it is also unsaponifiable and has a low refractive index and specific gravity. *Alcohol, acetone and chloroform* distil at comparatively low temperatures. Alcohol and acetone give the iodoform reaction and chloroform the phenyl iso-nitrile test. *Artificial esters*, such as terpinyl acetate, glycerine acetate, ethyl tartrate, citrate, phthalate, etc., distil in the last fractions of the oil. Terpinyl acetate can be identified by a method (*P. & E.O.R.*, 1915, 148) based on the protracted period which is necessary to complete its saponification by alcoholic potash. A special method has been devised by Schimmel & Co. (*P. & E.O.R.*, 1915, 147) for the detection of terpinyl acetate. *Turpentine* frequently reduces the specific gravity of an oil and influences the optical rotation of the first fractions which are obtained when the oil is distilled. *Fatty oils* leave an oily stain when the oil is allowed to evaporate on blotting-paper. Also, with the exception of castor oil, they are insoluble in alcohol. *Terpenes*, which are sometimes added to lemon, orange, etc. oils, lower the specific gravity and increase the optical rotation. *Copaiba, gurjun balsam* and *cedarwood* oils have high specific gravities and distil in the last fractions of the oil. When several drops of nitric acid are added to gurjun balsam oil a deep violet colour is obtained.

Special Methods.—These are very numerous and are being continually added to, so that it is proposed to summarise the most important and briefly refer to several recently introduced methods, leaving the reader to scan current literature dealing with analytical chemistry if it is desired to keep pace with the most modern methods.

Citral in Lemon Oil.—This is estimated according to the method given in the *B.P.*, 1914. Twenty gms. of the oil are weighed into a 200 c.c. flask, 20 c.c. of absolute alcohol and 20 c.c of N/2 hydroxylamine hydrochloride in 80 per cent. alcohol added. This hydroxylamine hydrochloride solution contains 35 gms. hydroxylamine hydrochloride per litre and is standardised against semi-normal acid. Eight c.c. normal alcoholic potash are added, the mixture is boiled under a reflux condenser for thirty minutes and then cooled The mixture in the flask is diluted with 250 c.c. of distilled water, which are added through the reflux condenser with a view to washing same. A few drops of phenol-phthalein are added to the solution and normal caustic potash added until the solution is neutral. The contents of the flask are then titrated with semi-normal hydrochloric acid, using methyl orange as indicator. A blank experiment is carried out and the number of c.c. of semi-normal hydrochloric acid required by this blank test, in excess of those required by the main test, represents the amount of hydroxylamine which has been taken up by the citral. When this is multiplied by 0·076 the amount of citral in the oil taken is obtained.

Citral in Terpeneless Lemon and Lemon-grass Oils.—This is best determined by the neutral sulphite method given under "Aldehydes." In the case of terpeneless lemon oils the bisulphite method gives erratic figures and in the case of lemon-grass oil higher results, by about 4 or 5 per cent., are obtained using the bisulphite method than when the neutral sulphite method is employed.

In the *Analyst*, 1910, 125; 1918, 379, the Hiltner method of estimating citral by colorimetric means, together with a modification of the same, is described. The reagent employed is metaphenylenediamine hydrochloride, with the addition, in the modification, of oxalic acid.

Cineol in Eucalyptus and Cajuput Oils. — Several methods have been suggested for this estimation, the one in most common usage is, however, that involving the use of phosphoric acid. Five c.c. of phosphoric acid (S.G. 1·75) are added to 10 c.c. of the oil contained in a porcelain basin, which is cooled by means of a freezing mixture. The contents of the basin are stirred with a glass rod until a solid mass is formed. This is placed in calico and pressed between blotting-paper in a strong press for several hours. The solid cake is then transferred to a graduated measure, 25 c.c. of cold water added and, after standing twelve hours, the volume of oil is read off. *Arsenic acid* may be used in a similar manner to phosphoric acid for the determination—it has the disadvantage, however, of being extremely poisonous.

A method is given in the *Analyst*, 1920, 457, for the estimation of cineol in eucalyptus oil by the determination of the solidifying point of the oils. T. T. Cocking also proposes to estimate cineol by means of the solidifying point of a mixture of the oil and o-cresol.

The *B.P.* test for phellandrene in eucalyptus oils is as follows: Two c.c. of the oil are mixed with 2 c.c. of glacial acetic and 2 c.c of saturated sodium nitrite solution. If phellandrene is present crystals of phellandrene nitrosite separate out. A more sensitive test is obtained by the addition of 5 c.c. of petroleum ether to the mixture.

A method of testing for cineol in phellandrene oils is given in the *Journ. Soc. Chem. Ind.*, Aug. 31, 1918. The reagent used is iodol, which gives a crystalline compound with cineol.

Citronellal and Geraniol in Citronella Oils.—These are estimated by Dupont and Labaune's method as follows: Ten gms. of the oil are shaken for two hours at 15° to 18° C. with a mixture made up as follows: 10 gms. hydroxylamine hydrochloride are dissolved in 25 c.c. water and 10 gms. of potassium carbonate, dissolved separately in 25 c.c. water, added, the mixture being then filtered. The oil is separated, dried over anhydrous sodium sulphate and acetylated with acetic anhydride and fused sodium acetate for one and a half hours under a reflux condenser. The resultant oil is washed, dried and neutralised and about 2 gms. taken and saponified with alcoholic potash. The total "geraniol and citronellal" figure is obtained from the total acetylisable constituents, which are estimated in the ordinary manner, and the quantity of citronellal, which is converted into nitrile in the above estimation and remains unaffected by the potash, is calculated by difference, the geraniol being calculated by the usual formula.

Prussic Acid in Oil of Bitter Almonds.—Two methods are in general use—(1) which is gravimetric, is as follows: 1 to 1·5 gms. of the oil are dissolved in about 10 c.c. alcohol, 10 c.c. of ammoniated alcohol added and the mixture allowed to stand for half an hour. A small quantity of nitric acid is then added to acidulate the mixture and the cyanide precipitated with silver nitrate. The solution is filtered and the precipitate dried to constant weight in a water oven and weighed. The percentage of prussic acid is calculated from the formula

$$\frac{\text{Weight of silver cyanide} \times 20{\cdot}179}{\text{Weight of oil taken}}$$

(2) The volumetric method of estimating the amount of prussic acid consists in titrating a 10 per cent. solution of the oil in alcohol with N/10 silver nitrate solution, using potassium chromate in alkaline solution as indicator, after allowing for the chloride present in the reagents employed.

Artificial Benzaldehyde.—This is usually detected in a natural oil by means of the trace of chlorine which it nearly always contains.

Lead in Cassia Oil.—A rapid method for the colorimetric estimation of lead in cassia oil by means of ammonium sulphide is given in the *Journ. Soc. Chem. Ind.*, 1920, 39, 35–36.

CHAPTER VI

SYSTEMATIC SURVEY OF THE ESSENTIAL OILS

In the following systematic survey of the essential oils the usual method of classification is adopted, namely, according to the botanical derivation of the oils.

Coniferæ.—*Cedar Oil.* — This is obtained from the Virginian red cedar, *Juniperus virginiana.* The *wood* oil is distilled from the refuse of pencil factories, the yield being 2·5 to 5 per cent. The chief constituents of the oil are the sesquiterpene cedrene, about 7 to 10 per cent. of cedrol, which is a crystalline body melting at 78° to 80° when pure, and cedrenol (*P. & E.O.R.*, 1912, 107). Characters: Specific gravity, 0·940 to 0·960; optical rotation, −25° to −44°; refractive index, 1·495 to 1·510; soluble in 10 to 20 vols. of 90 per cent. alcohol; 80 per cent. of the oil distils between 125° and 155° at a pressure of 14 mm. (Schimmel).

The *leaf* oil contains limonene, borneol, cadinene, bornyl esters, etc. Characters: Specific gravity, 0·883 to 0·888; optical rotation, +55° to +65°. Oils distilled from the leaves of *Thuja occidentalis* and *Chamæ cyparis sphæroidea* are often sold as cedar leaf oils.

Juniper Berry Oil.—This is obtained, to the extent of about 0·8 to 1·5 per cent., by distillation of the beans of *Juniperus communis*, which is indigenous to Greece, but most extensively grown in Italy and Hungary (*P. &*

E.O.R., 1915, 63). The chief constituents of the oil are 25 to 60 per cent. pinene, 15 to 25 per cent. cadinene, juniper camphor (a sesquiterpene alcohol melting at 165° to 166°) and a small quantity of esters boiling at about 180°. Juniper oil is largely used in the flavouring of artificial gin. Characters: Specific gravity, 0·862 to 0·890; optical rotation, −3° to −15°; refractive index at 25°, 1·472 to 1·488; soluble in 4 vols. of 95 per cent. alcohol when fresh. The specific gravity increases and the solubility decreases with age (*P. & E.O.R.*, 1913, 402; 1914, 5).

Juniper Wood Oil, which is used as a substitute for the *berry* oil, is usually a mixture of the latter with turpentine.

Pine-Needle Oil.—This is distilled chiefly from the leaves of *Pinus pumilio* and *P. sibirica*, the oil from the latter being the Ol. Abietis of the *B.P.*, 1914. The oil from *P. pumilio* is obtained principally from Hungary and the Austrian Tyrol; its constituents are: sylvestrene, l-phellandrene, small amounts of l-pinene, cadinene, dipentene and 5 to 7 per cent. of esters, chiefly bornyl acetate. Characters: Specific gravity, 0·863 to 0·875; optical rotation, −6° to −14°; refractive index at 25°, 1·475 to 1·485. The oil distilled from *P. sibirica* is obtained chiefly from Northern Russia and the Urals. Its chief constituents are 30 to 45 per cent. esters (*Chem. & Drug.*, ii./11, 68), these being chiefly bornyl acetate, also pinene, dipentene, camphene, phellandrene, etc. Characters: Specific gravity, 0·900 to 0·930; optical rotation, −30° to −45°; refractive index at 25°, 1·467 to 1·476; soluble in an equal volume of 90 per cent. alcohol (*P. & E.O.R.*, 1915, 94). Pine-needle oils are largely used in the flavouring of medicated sweetmeats, etc. See *P. & E.O.R.*, 1915, 166, for characters of other pine-needle oils, e.g. *P. pectinata*, *P. sylvestris* and *P. canadensis.*

Turpentine Oil.—This is distilled from the oleo-resin which exudes from several species of *Pinus.* American turpentine oil, derived from *Pinus australis* and *P. tæda*, is the chief of these products on the English market. It contains chiefly pinene and a small amount of dipentene. Characters: Specific gravity, 0·855 to 0·877; optical rotation, +1° to +6°; refractive index, 1·465 to 1·480; 80 to 85 per cent. distils between 155° and 165°. French turpentine is derived from *P. pinaster.* Its chief constituent is l-pinene, the characters of the oil being as follows: Specific gravity, 0·855 to 0·874; optical rotation, −18° to −40°; refractive index, 1·470 to 1·480; 75 to 90 per cent. distils between 155° and 165°. Russian and Swedish turpentine oils are derived from *P. sylvestris* and *P. ledebourdii.* They contain principally sylvestrene and have a very disagreeable odour. Characters: Specific gravity, 0·855 to 0·874; optical rotation, +5° to +16°; refractive index, about 1·479; 30 to 70 per cent. distils between 155° and 165°. The various turpentine oils are of no interest as flavouring agents; from the American oil, however, is obtained the liquid Terebene, which is sometimes used in the flavouring of medicated lozenges, etc.

Terebene.—This is a colourless liquid produced by the action of sulphuric acid on turpentine, followed by distillation. It consists principally of dipentine together with other hydrocarbons. It has a specific gravity of 0·862 to 0·866, is optically inactive, boils at 160° to 170° and is soluble in 5 vols. of 90 per cent. alcohol.

Gramineæ.—*Citronella Oil.*—This is obtained, to the extent of 0·5 to 1·0 per cent., by distilling the grass

Cymbopogon nardus (*Andropogon nardus*), which is grown chiefly in Ceylon, the Straits Settlements and Java. The chief constituents of the Ceylon oil are geraniol, citronellal, dipentene, limonene, camphene, and small quantities of methyl-heptenone, methyl-eugenol, linalol, borneol and sesquiterpenes. The Java oil contains similar constituents, the citronellal, however, predominating. Characters: Ceylon—Specific gravity, 0·900 to 0·920; optical rotation, −7° to −16°; refractive index, 1·471 to 1·481; total acetylisable constituents (as geraniol), 58 to 64 per cent.; soluble in 3 to 10 vols. 80 per cent. alcohol. Java—Specific gravity, 0·885 to 0·910; optical rotation, 0° to −10°; refractive index, 1·4650 to 1·4730; total acetylisable constituents (as geraniol), 80 to 92 per cent.; soluble in 3 to 10 vols. 80 per cent. alcohol. Citronella oil is used chiefly for the perfuming of cheap soap; it is, however, interesting from the flavouring standpoint, as are other members of the Gramineæ, by reason of its geraniol content. Much adulteration has been practised with mineral oil, this having given rise to the controversy regarding the Schimmel solubility test of the oil in 80 per cent. alcohol. The determination of the total acetylisable constituents in the oil is, however, now regarded as the most satisfactory test. The following references deal with the valuation of Java citronella oil: *P. & E.O.R.*, 1911, 201; 1912, 123, 250, 334; 1913, 13. For methods of estimating the geraniol and citronellal contents, see *P. & E.O.R.*, 1912, 106; 1914, 275.

Ginger-grass Oil.—This is derived from the ripened grass *Cymbopogon martini* var. *sofia*. It is inferior to palma-rosa oil (see later), containing more geranyl acetate and having a distinct ginger odour. The chief constituents of the oil are geraniol, dihydrocuminol, geranyl acetate and small quantities of dipentene, limonene, carvone and an aldehyde. Characters: Specific gravity, 0·930 to 0·945; optical rotation, usually between +20° and +30°, but as wide variations as −30° to +54° have been observed; refractive index, 1·478 to 1·489: total alcohols (as geraniol), 35 to 70 per cent.; esters, 5 to 25 per cent.; soluble in 3 vols. 70 per cent. alcohol.

Lemon-grass Oil (Indian Verbena).—This is derived from the grasses *Cymbopogon citratus* and *C. flexuosus*, which are grown in Malabar and Cochin (*C. flexuosus*), and in Ceylon, the Straits Settlements and the West Indies (*C. citratus*). The chief constituents are citral, geraniol, citronellal, methyl-heptenone, limonene and dipentene, with additionally in the West Indian variety an olefinic terpene which rapidly resinifies. Lemon-grass oil is the chief source of citral, which it contains to the extent of 70 to 85 per cent. (*P. & E.O.R.*, 1916, 95). Characters: West Indian Oil—Specific gravity, 0·877 to 0·887; optical rotation, −2° to 2°: refractive index, 1·483 to 1·489; citral, 75 to 85 per cent.; not soluble in 90 per cent. alcohol. East Indian Oil—Specific gravity, 0·895 to 0·905; optical rotation, −3° to +3°; refractive index, 1·483 to 1·488; citral, 70 to 80 per cent.; soluble in 3 vols. 70 per cent. alcohol (*P. & E.O.R.*, 1914, 330, 398; 1916, 333, 394). The *P. & E.O.R.*, 1918, 96, gives a note regarding the constituents of a Formosan lemon-grass oil.

Palma-Rosa Oil (Indian Geranium Oil).—This is derived from the young bluish-white grass *Cymbopogon martini* (*Andropogon schœnanthus*), which is grown chiefly in India. It has a rose-geranium odour and contains chiefly geraniol (75 to 95 per cent.), geranyl acetate, geranyl caprylate and traces of dipentene, citronellol and methyl-heptenone. Characters: Specific gravity, 0·885 to 0·895;

optical rotation, $-2°$ to $+2°$; refractive index, 1·474 to 1·485; total alcohols (as geraniol), 75 to 95 per cent.; esters, 5 to 11 per cent.; soluble in 3 vols. 70 per cent. alcohol. It is occasionally sophisticated by the addition of mineral oil, fatty oils, turpentine, gurjun balsam and cedarwood oils.

Veti-vert or Cuscus Oil.—This is distilled, to the extent of 0·4 to 0·9 per cent., from the roots of *Vetivera zizanoides* (*Andropogon muricatus*), which is grown in Bengal, Punjaub, Réunion, Mauritius and the West Indies (*P. & E.O.R.*, 1913, 42). The chief constituents are sesquiterpenes. Characters: Specific gravity, 1·015 to 1·030 (Réunion oil, 0·980 to 0·995); optical rotation, $+25°$ to $+45°$; refractive index, 1·517 to 1·520; esters, less than 10 per cent.; soluble in 2 vols. 80 per cent. alcohol. The oil is sometimes adulterated with fatty oils.

MONOCOTYLEDONS

Aroideæ.—*Calamus Oil.*—This oil is obtained, to the extent of 1·5 to 3·5 per cent., by distillation from the fresh rhizome of *Acorus calamus* or Sweet Flag, which is grown in Europe, North America, China, Japan, Cochin China and the East Indies. The chemistry of the oil is not very well known. It contains eugenol, calamene (a bicyclic terpene), asarone, calameon (similar in constitution to cineol, melting-point, 119°), traces of pinene and oxygenated bodies. Characters: Specific gravity, 0·960 to 0·970; optical rotation, $+10°$ to $+35°$; refractive index, 1·506 to 1·508; soluble in 2 to 3 vols. 90 per cent. alcohol.

Iridaceæ.—*Orris Oil.*—This is obtained, to the extent of 0·1 to 0·2 per cent., by steam distilling the rhizome of *Iris florentina*, *I. germanica* and *I. pallida*, which are grown in Mediterranean countries. It is a yellowish-white mass, the chief odorous constituent of which is the ketone irone, this being accompanied by about 85 per cent. myristic acid and traces of esters. Characters: Acid number, 200 to 220; melting-point, 38° to 50°; saponification value, 190 to 210; slightly dextro-rotatory; soluble in 16 vols. of 90 per cent. alcohol (*P. & E.O.R.*, 1917, 275). Orris oil is used in many flavouring essences as a substitute for or fixative of natural violet perfumes.

Liliaceæ.—*Garlic Oil.*—This is obtained, to the extent of 0·005 to 0·01 per cent., by distillation of the whole fresh plant *Allium sativum*, which is cultivated extensively in Spain, Portugal and Italy. It is used in Continental countries as a flavouring agent. It contains chiefly diallyl disulphide, and about 5 to 6 per cent. of allyl-propyl disulphide. Characters: Specific gravity, 1·046 to 1·057. It is optically inactive.

Onion Oil.—This is distilled to the extent of 0·04 to 0·05 per cent. from *Allium cepa*. Characters: Specific gravity, 1·036 to 1·041; optical rotation, about −5°.

Zingiberaceæ.—*Cardamom Oil.*—This is distilled to the extent of 4 to 8 per cent. from the seeds of *Elettaria cardamomum*, which grows in southern India and Ceylon (*P. & E.O.R.*, 1918, 69). The constituents of cardamom oil are cineol, limonene, terpineol, terpinene and sabinene. Characters: Specific gravity, 0·922 to 0·950; optical rotation, +22° to +46°; refractive index, 1·460 to 1·470; soluble in 4 vols. of 70 per cent. alcohol. The *P. & E.O.R.*, 1918, 31, gives a note regarding the characters of a cardamom oil distilled in Mysore.

Curcuma or Turmeric Oil.—This is distilled, with a yield of 5 to 5·5 per cent., from the rhizome of *Curcuma longa*, which is cultivated in India, China and southern Asia. The chief constituents of the oil are an alcohol "turmerol," boiling at 285° to 290°, and a small quantity of phellandrene. The specific gravity of the oil is 0·940 to 0·945.

Galangal Oil.—This oil is steam distilled, to the extent of 0·5 to 1·5 per cent., from the rhizome of *Alpinia galanga* and *A. officinarum*, which are cultivated in Siam and China. It has a sharp taste and camphoraceous odour. The chief constituents of the oil are cineol, eugenol, cadinene and pinene. Characters: Specific gravity, 0·915 to 0·925; optical rotation, $-1°$ to $-4°$; refractive index, about 1·480. Pimento and clove oils are the chief adulterants; these are tested for by solubility. The oil should not be entirely dissolved by an equal volume of 90 per cent. alcohol.

Ginger Oil.—This is distilled to the extent of 1 to 3 per cent. from the rhizome of *Zingiber officinale*, which is grown over a wide area in tropical Asia, the East and West Indies and Africa. Its chief constituents are zingiberine, phellandrene, camphene, and small quantities of borneol, citral, cineol, etc. Characters: Specific gravity, 0·872 to 0·885; optical rotation, $-25°$ to $-45°$; refractive index, 1·488 to 1·495.

Grains of Paradise Oil.—This is distilled, with a yield of about 0·75 per cent., from the seeds of the *Amomum melegueta*. Its odour is aromatic, being very similar to cardamom oil, but not so fragrant.

Zedoary Oil.—This is obtained to the extent of 1 to 2 per cent. by distillation of the roots of *Curcuma zedoaria*. It has a camphoraceous odour due to the presence of cineol. Its specific gravity is 0·990 to 1·010.

DICOTYLEDONS

Anonaceæ.—*Cananga Oil.*—This is derived to the extent of 1·5 to 2 per cent. from the flowers of *Cananga* *odorata*, which is grown in the Philippines. The first fractions which come over during the distillation are sold as ylang-ylang oil (see later), the later fractions forming the cananga oil of commerce. Both oils contain linalol, geraniol, farnesol, nerol, iso-eugenol, cresol, paracresol, pinene, esters, methyl ether, cadinene, and a sesquiterpene alcohol (melting-point, 138°). Characters: Specific gravity, 0·910 to 0·940; optical rotation, $-17°$ to $-55°$; refractive index (20°), 1·494 to 1·504; saponification number, 45 to 100.

Ylang-ylang Oil.—The characters of this oil are as follows: Specific gravity, 0·930 to 0·960; optical rotation, $-38°$ to $-45°$; refractive index (20°), 1·480 to 1·515; saponification number, 95 to 138; soluble in 1 to 2 vols. 95 per cent. alcohol, turbid with more alcohol.

Betulaceæ.—*Sweet Birch or Wintergreen Oil.*—This is now produced almost entirely from the bark of *Betula lenta*, or sweet birch, which is grown in North America. The oil, derived from *Gaultheria procumbens*, which was formerly used, is practically unobtainable. Artificial wintergreen oil or methyl salicylate is sold very extensively in place of the natural product. The yield of oil, which is produced by the action of the ferment "betulase" on the glucoside "gaultherin" occurring in the bark, is about 0·5 to 0·7 per cent. It consists practically wholly of methyl salicylate. Characters: Specific gravity, 1·180 to 1·187; optically inactive; refractive index, 1·534 to 1·539; boiling-point, 218° to 220°; soluble in 6 vols. 70 per cent. alcohol at 25°; gives deep violet coloration with ferric chloride in alcohol. See *P. & E.O.R.*, 1914, 60; 1915, 105, 125; 1916, 344, for the detection of artificial wintergreen oil in the natural product. The oil derived from *Gaultheria procumbens* differs from the

Betula lenta oil by having an optical rotation of −0° 25′ to −1°.

Burseraceæ.—*Elemi Oil.*—This is obtained to the extent of 15 to 30 per cent. by distilling the oleo-resin of *Canarium commune*, which grows in the Philippines. It contains chiefly dipentene and phellandrene. It has a specific gravity of 0·870 to 0·910, and an optical rotation of about + 45°.

Lignaloe Oil.—This is obtained to the extent of 7 to 12 per cent. by distillation of the wood of *Bursera delpechiana*, *B. aloexylon*, and other species, which are grown chiefly in Mexico (*P. & E.O.R.*, 1910, 57). It contains principally linalol (55 to 75 per cent.), geraniol, terpineol and methylheptenone. Characters: Specific gravity, 0·875 to 0·895; optical rotation, −5° to −12°; refractive index, 1·459 to 1·464; linalol (by acetylation), 55 to 75 per cent.; soluble in 2 to 3 vols. 70 per cent. alcohol.

Myrrh Oil.—This is derived, the yield varying between 2 to 10 per cent., from the oleo-resin of *Commiphora myrrha*, *C. abyssinica*, and other species, which grow in Arabia and Somaliland. The oil contains, according to its derivation, varying quantities of myrrholic acid, herabolene, metacresol, etc. Characters: Specific gravity, 0·985 to 1·007; optical rotation, −67° to −90°; refractive index, about 1·536. Oil of Bisabol Myrrh derived from the oleo-resin of *C. kataf*, grown in Somaliland, contains chiefly bisabolene, which is a sesquiterpene identical with that contained in lemon and lime oils. Characters: Specific gravity, 0·883; optical rotation, about −14° 20′ (*The Volatile Oils*, by Gildermeister & Hoffmann (1), 488)

Cannabinaceæ.—*Hop Oil.*—This is distilled to the extent of 0·3 to 1 per cent. from the flowers (strobiles) of *Humulus lupulus*. The oil is more valuable when the flowers have not been sulphured. The chief constituents are dipentene, linalol, tetrahydrocymene, humulene (identical with α-caryophellene, *P. & E.O.R.*, 1911, 176), and an olefinic terpene.

Compositæ.—*Arnica Oil*.—This is obtained by distilling the flowers and roots of *Arnica montana*, which grows in Europe and North America. The oil distilled from the flowers has a bluish colour when fresh. It contains a mixture of ethers (phlorone iso-butyric ether, phlorone dimethyl ether, and thymo-hydroquinone dimethyl ether). Characters: Specific gravity, 0·905 to 0·910 (flower oil), 0·990 to 1·000 (root oil); optical rotation, 0° to −2°.

Chamomile Oil.—This is distilled to the extent of about 1 per cent. from the freshly dried flowers of *Anthemis nobilis*, which is grown in European countries. It has a blue colour when freshly distilled, which, however, gradually turns green, then brown. The chief constituents are amyl alcohol, hexyl alcohol, angelic and tiglic isobutyl ethers, an alcohol anthemol, an ethylene hydrocarbon (melting-point, 63°), and the blue constituent azulene (*P. & E.O.R.*, 1915, 46). Characters: Specific gravity, 0·905 to 0·915; optical rotation, −1° to −3°; refractive index at 25°, 1·442 to 1·465; soluble in 6 vols. 70 per cent. alcohol. The German chamomile oil, which is derived from the flowers of *Matricaria chamomilla*, contains chiefly chamomillol, tri-chamomillol, caproic esters, furfuraldehyde, umbelliferone methyl ether, and solid hydrocarbons. It has a specific gravity of 0·930 to 0·940, congeals at 1°C. and is soluble in 3 vols. 90 per cent. alcohol.

Tansy Oil.—This is distilled to the extent of 0·1 to 0·2 per cent. from the fresh flowering herb *Tanacetum vulgare*, which is grown in European countries and North America. It contains thujone, borneol and camphor. Characters: Specific gravity, 0·925 to 0·955; optical rotation, +25° to +45°; soluble in 3 vols. 70 per cent. alcohol.

Tarragon Oil (*Estragon Oil*).—This is distilled from the flowering herb *Artemisia dracunculus*, which is grown in Europe. It contains methyl chavicol, phellandrene, and para-methoxy-coumarinic aldehyde, and is used in the preparation of sauces, etc. Characters: Specific gravity, 0·890 to 0·960; optical rotation, +2° to +9°; refractive index, 1·516 to 1·518; soluble in 10 vols. 80 per cent. alcohol.

Wormseed Oil.—This is distilled to the extent of about 2 per cent. from the flower buds of *Artemisia maritima*, which grows in South Russia and Asia Minor. It contains thujone, cineol, terpinene, dipentene, terpineol and a sesquiterpene. Characters: Specific gravity, 0·915 to 0·940.

American Wormseed Oil (*Chenopodium Oil*) is obtained to the extent of 0·6 to 1·0 per cent. from the seeds of *Chenopodium ambrosiodes* (N.O. Chenopodiaceæ). This oil has displaced wormseed oil to a great extent. It contains 60 to 65 per cent. ascaridol, 20 per cent. paracymene and camphor, sylvestrene, etc. Characters: Specific gravity, 0·950 to 0·990; optical rotation, −5° to −10°; refractive index, 1·466 to 1·476; saponification number, 246 to 280; soluble in 4 to 10 vols. 70 per cent. alcohol (*P. & E.O.R.*, 1912, 16, 80; 1913, 80; 1915, 25, 104, 125, 189; *Journ. Chem. Soc.*, 1921, i. 259, 797 and 1714).

Wormwood Oil (*Absinthe Oil*).—This is distilled, the yield being 0·2 to 1·0 per cent., from the herb *Artemisia absinthium*, which is grown in Europe and North America. It contains chiefly thujone, thujyl alcohol, esters (acetic, palmitic, and iso-valeric), cadinene, phellandrene, and pinene. Characters: Specific gravity, 0·925 to 0·953; refractive index, 1·460 to 1·474.

Cruciferæ.—*Mustard Oil.*—This is derived to the extent of 0·5 to 0·8 per cent. from the seeds of black mustard, *Sinapis nigra.* The seeds are pressed and then macerated with water, under which treatment the enzyme myrosin acts on the glucoside potassium myronate, both substances being present in the seeds, with the production of allyl-iso-thiocyanate. This latter substance is the chief constituent (92 to 95 per cent.) of mustard oil; accompanying it are carbon disulphide and allyl cyanide. Characters: Specific gravity, 1·014 to 1·032; optical rotation, nil; refractive index at 25°, 1·525 to 1·535.

Geraniaceæ.—*Rose Geranium Oils.*—These are obtained to the extent of 0·1 to 0·15 per cent. by distillation of the leaves of *Pelargonium roseum*, *P. odoratissimum*, *P. capitum* and other species, which are grown in France, Spain, Réunion, Algeria, etc. (*P. & E.O.R.*, 1910, 193; 1912, 243; 1913, 239, 272, 304, 372, 381; 1914, 323). The order of value of the various oils is as follows: Spanish, French, Algerian and Bourbon. The chief constituents are geraniol, esters, citronellol, linalol, traces of acetic, butyric, valerianic and tiglic acids. Among the adulterants which are occasionally used in these oils the following are worthy of mention: Glycerine acetate, ethyl phthalate, tartrate, citrate, succinate, oleate, oxalate, geraniol and palma-rosa oil. Characters: Spanish—Specific gravity, 0·889 to 0·898; optical rotation, −10° to −15°; refractive index, 1·461 to 1·470; esters (as geranyl tiglate), 35 to 42 per cent.; alcohols (as geraniol), 70 per cent.; soluble in 3 vols. 70 per cent. alcohol. French—Specific gravity, 0·895 to 0·905; optical rotation, −7° to −12°; refractive index, 1·461 to 1·470; esters (as geranyl tiglate), 22 to 28 per cent.; alcohols (as geraniol), 75 per cent.; citronellol by formylation, 40 per cent.; soluble in 3 vols. 70 per cent. alcohol. Algerian—Specific gravity, 0·890 to 0·900; optical rotation, −6° to −12°; refractive index, 1·461 to 1·470; esters (as geranyl tiglate), 20 to 29 per cent.; alcohols (as geraniol), 75 per cent.; citronellol by formylation, 33 per cent.; soluble in 3 vols. 70 per cent. alcohol. Bourbon—Specific gravity, 0·890 to 0·895; optical rotation, −8° to −11°; refractive index, 1·461 to 1·470; esters (as geranyl tiglate), 27 to 34 per cent.; total alcohols (as geraniol), 80 per cent.; citronellol by formylation, 44 per cent.; soluble in 3 vols. 70 per cent. alcohol.

Labiatæ.—*Balm Oil* or *Melissa Oil.*—This is distilled from the fresh green herb *Melissa officinalis*, which is grown in Europe and North America. Commercial balm oil is frequently either lemon or citronella oil, or a mixture of the two, distilled over the green herb. The pure oil is very expensive and contains chiefly citral, citronellol and geraniol. Characters: Specific gravity, 0·894 to 0·924; optical rotation, +0° 30′ to −6° 30′.

Hyssop Oil.—This is distilled from the dried flowering herb *Hyssopus officinalis.* It is used to some extent in the compounding of cordials. Its chief constituents are pinene, a ketone pinocamphone, small quantities of cineol, thujyl alcohol and thujone. Characters: Specific gravity, 0·925 to 0·940; optical rotation, −19° to −24°; soluble in 6 vols. 80 per cent. alcohol.

Lavender Oil.—Ordinary lavender oil is obtained to the extent of 0·5 to 1·0 per cent. by distilling in water the fresh flowering herb *Lavandula vera.* The bulk of foreign oil is derived from the mountainous coastal regions in the south of France, the better class of oils being obtained from the plants grown on the higher slopes. English oils, which are produced in the neighbourhoods of Mitcham, Hitchin, Canterbury, etc., are recognised as being the finest in aroma in the world; at the same time they only contain 6 to 10 per cent. of esters as compared with 25 to 45 per cent. in French oils. So that the ester value of lavender oils is no guide to the comparative values of English and French oils as many continental authorities claim. The chief constituents of lavender oil are linalol, nerol, geraniol, linalyl esters, limonene, cineol, a sesquiterpene and a trace of pinene (*P. & E.O.R.*, 1916, 50). Characters: English—Specific gravity, 0·883 to 0·890; optical rotation, $-5°$ to $-10°$; refractive index at 25°, 1·463 to 1·465; esters (linalyl acetate), 6 to 10 per cent.; soluble in 3 vols. 70 per cent. alcohol. French—Specific gravity, 0·885 to 0·895; optical rotation, $-3°$ to $-9°$; refractive index at 25°, 1·461 to 1·465; esters (linalyl acetate), 25 to 45 per cent.; soluble in 3 vols. 70 per cent. alcohol. Lavender oil has been subjected to much scientific adulteration, especially with synthetic esters of the type of ethyl phthalate, citrate, tartrate, glycerine acetate, benzyl benzoate, linalyl acetate, etc.

Spike lavender oil is distilled to the extent of 0·5 to 1 per cent. from the fresh flowering herb *Lavandula spica*, which grows in the mountainous coastal regions of France, Spain and Italy. Its odour is about midway between that of lavender and rosemary oils. Its chief constituents are linalol, camphor, borneol, cineol, d-camphene and a sesquiterpene. Characters: Specific gravity, 0·905 to 0·918; optical rotation, $-2°$ to $+4°$ (or even up to $+6°$ or $+7°$); refractive index, 1·462 to 1·467; total alcohols (as borneol), 25 to 45 per cent.; esters (linalyl acetate), 4 to 6 per cent.; soluble in 6 vols. 65 per cent. alcohol and in 3 vols. 70 per cent. alcohol (see *P. & E.O.R.*, 1916, 239). This oil is occasionally adulterated with turpentine.

Marjoram Oil.—Oil of sweet marjoram is distilled to the extent of 0·3 to 0·4 per cent. from the fresh flowering herb *Origanum majorana*, which is chiefly grown in Spain. The chief constituents of the oil are terpineol, terpinene, terpin hydrate, camphor and borneol. Characters: Specific gravity, 0·890 to 0·910; optical rotation +5° to +18°; refractive index, 1·472 to 1·510: soluble in 3 vols. 80 per cent. alcohol.

Origanum Oils.—These are distilled from several species of *Origanum*. For example, from *Origanum dubium* and *O. majoranoides* (*P. & E.O.R.*, 1918, 70). The oil obtained from Asia Minor is distilled from *O. Smyrnæum* and other species. The Trentino oil is distilled from *O. hirtum* and *O. onites*. As might be expected with oils of such varied derivations the characters have rather wide limits. Characters: Specific gravity, 0·915 to 0·980; optical rotation, −13° to +3°; refractive index, 1·500 to 1·510; phenols, 25 to 85 per cent.; soluble in 3 vols. 70 per cent. alcohol. All origanum oils contain principally carvacrol, together with cymene and sometimes linalol.

Patchouli Oil.—This is distilled to the extent of about 2 to 4 per cent. from the leaves of *Pogostemon patchouli*, which is grown chiefly in the Straits Settlements, the West Indies, Penang, Java, Réunion and Mauritius (*P. & E.OR.*, 1913, 369, 418; 1918, 291). Its chief constituents are 40 to 45 per cent. of cadinene, an alcohol melting at 56° (patchouli alcohol), small quantities of benzoic and cinnamic aldehydes and other bodies. Characters: Specific gravity, 0·950 to 0·995; optical rotation, −40° to −68°; refractive index, 1·504 to 1·520; saponification number, 4 to 18; saponification number (after acetylation), 35 to 80; soluble in 5 to 6 vols. 90 per cent. alcohol.

Peppermint Oils.—These are obtained by steam distillation of one or two species of *Mentha.* The English oil is derived from "black" and "white" *Mentha piperita*, the white variety being superior in quality (*P. & E.O.R.*, 1910, 6; 1911, 175). The following are the characters of the English oil: Specific gravity, 0·900 to 0·910; optical rotation, −22° to −33°; refractive index, 1·460 to 1·465; total menthol, 55 to 70 per cent.; esters (as menthyl acetate), 3 to 15 per cent. ("black" oil usually contains 7 to 10 per cent. esters and the "white" oil 11 to 15 per cent.); soluble in 3 to 4 vols. of 70 per cent. alcohol. Most peppermint oils improve with storing for a period. The oils have a very widespread use both as flavouring agents and in pharmacy.

American peppermint oil is derived from *Mentha piperita*, which is grown chiefly in Wayne County, Kalamazoo, etc. (*P. & E.O.R.*, 1910, 7; 1917, 60). It differs from English oil in that it usually contains a lower percentage of esters. The following are the characters of American peppermint oil: Specific gravity, 0·900 to 0·920; optical rotation, −18° to −33°; refractive index, 1·458 to 1·467; total menthol, 50 to 65 per cent.; esters (as menthyl acetate), 6 to 10 per cent.; soluble in 3 to 4 vols. 70 per cent. alcohol when double-distilled, otherwise it usually gives a cloudy solution in these proportions.

Japanese peppermint oil is derived from *Mentha arvensis* (*P. & E.O.R.*, 1911, 7; 1918, 32). It has a very woody taste, and is used chiefly as a source of menthol, which it contains to the extent of 70 to 90 per cent. The dementholised Japanese peppermint oil on the market usually contains 40 to 45 per cent. menthol. A rectified dementholised Japanese peppermint oil has been offered on the market during the last few years, the woody flavour having been eliminated to an extent which permits the use of the oil for blending purposes. The following are the characters of Japanese peppermint oil: Specific gravity, 0·900 to 0·915; optical rotation, −30° to −42°; refractive index, 1·462 to 1·468; total menthol, 70 to 90 per cent.; esters (as menthyl acetate), 3 to 6 per cent.; soluble in 3 to 4 vols. 70 per cent. alcohol (*P. & E.O.R.*, 1918, 207).

The chief constituents of peppermint oils of all derivations are menthol, menthyl acetate, valerianate, etc., menthone, phellandrene, pinene, limonene, cadinene, cineol and traces of dimethyl sulphide, aldehydes and acids. Adulteration by means of copaiba oil, gurjun balsam, cedar oil, etc., may be determined by fractionation; glycerine acetate can be extracted with water or 5 per cent. alcohol.

Rosemary Oil.—This is distilled to the extent of 1 to 2 per cent. from the dried leaves of *Rosmarinus officinalis*, which is grown chiefly in Spain, Northern Africa (*P. & E.O.R.*, 1916, 214), Italy and France. The French oil is considered to be of superior quality (*P. & E.O.R.*, 1910, 190; 1911, 109, 274). The chief constituents of the oil are pinene, cineol, camphor, borneol, camphene and bornyl acetate. Adulteration is sometimes practised with camphor oil. Characters: Specific gravity, 0·895 to 0·920; optical rotation, −3° to +10° (or even up to +15°); refractive index at 25°, 1·465 to 1·475; total alcohols, as borneol, 10 to 18 per cent.; esters, as bornyl acetate, 2 to 6 per cent.; soluble in 10 vols. 80 per cent. alcohol.

Sage Oil.—This is distilled to the extent of 1·25 to 2·5 per cent. from the fresh flowering herb, *Salvia officinalis*, which grows in Mediterranean countries (*P. & E.O.R.*, 1916, 369). It contains chiefly cineol, pinene, borneol and thujone. Characters: Specific gravity, 0·910 to 0·930; optical rotation, +10° to +25°; refractive index, 1·462 to 1·467; esters, 2 to 6 per cent.; total alcohols, 20 to 25 per cent.; thujone, 40 to 50 per cent.; soluble in 3 vols. 70 per cent. alcohol. Other oils of sage are derived from *Salvia triloba* (Spanish sage) and *S. sclarea* (Muscatel or "clary" sage). The former has a specific gravity of 0·925 to 0·932; optical rotation, +13° to +19°; total esters (as linalyl acetate), 9 to 21 per cent.; total alcohols, 23 to 29 per cent.; soluble in 6 vols. of 70 per cent. alcohol. The latter has a specific gravity of 0·907 to 0·928; optical rotation, −19° to −24°; and a content of 40 to 50 per cent. linalyl acetate. It possesses an odour resembling lavender.

Spearmint Oil.—This oil is distilled to the extent of 0·2 to 0·3 per cent. from the fresh green herb *Mentha viridis* (American spearmint), and also from *M. crispa* (German spearmint). Its chief constituents are carvone, pinene, limonene and traces of esters. Characters: Specific gravity, 0·920 to 0·940; optical rotation, −30° to −52°; refractive index at 25°, 1·481 to 1·487; carvone, 50 to 60 per cent.; soluble in an equal volume of 90 per cent. alcohol, goes turbid with more alcohol.

Thyme Oils.—Red thyme oil is obtained to the extent of 0·3 to 2·6 per cent. by distillation from the fresh and dried herb *Thymus vulgaris*, which is grown in Mediterranean countries. It contains chiefly carvacrol, thymol, cymene, pinene, linalol and borneol. Characters: Specific gravity, 0·905 to 0·935; optical rotation, 0 to −4°; refractive index, 1·485 to 1·498; phenols, 20 to 50 per cent.; soluble in 3 vols. 80 per cent. alcohol.

Lemon thyme oil (Spanish) is distilled from the flowering herb *Thymus hyemalis.* It contains citral in addition to carvacrol. Characters: Specific gravity, 0·900 to 0·920; citral, 10 to 20 per cent.; phenols, 8 to 12 per cent. (see also *P. & E.O.R.*, 1912, 212; 1915, 187). Wild thyme oil is derived to the extent of 0·15 to 0·6 per cent. from *Thymus serpyllum*, which grows in Europe, North America and Asia. It contains chiefly cymene and terpenes, together with a trace of phenols. Characters: Specific gravity, 0·890 to 0·920; optical rotation, −10° to −21°. For oil of *Thymus mastichina* or wood marjoram, see *P. & E.O.R.*, 1911, 40: 1915, 187.

Lauraceæ.—*Bois de Rose Oil (Male).*—This is distilled from *Ocotea caudata*, which is grown in French Guiana. The oil is similar in composition and odour to linaloe oil. The "femelle" oil is obtained from *Protium altissimum* and contains geraniol, nerol, terpineol, methylheptenone, etc. Characters: Specific gravity, 0·870 to 0·880; optical rotation, −15° to −17°; soluble in 3 vols. 70 per cent. alcohol.

Camphor Oil is obtained by distillation of the wood of *Cinnamomum camphora*, which grows chiefly in Formosa, Japan and China. The process is subsidiary to the manufacture of camphor. Two oils are obtained during the distillation, namely, "light" and "heavy." The light oils contain chiefly terpenes, *e.g.* pinene, limonene, phellandrene, dipentene, terpineol, cineol, borneol and camphor. The heavy oils contain safrol, eugenol, camphor and cadinene. Characters: Light Oil—Specific gravity, 0·860 to 0·950; optical rotation, +12° to +32°; refractive index, 1·465 to 1·481. Heavy oil—0·950 to 1·100; optical rotation, 0 to +12°; refractive index, 1·500 to 1·508.

Cassia Oil.—This is distilled to the extent of 0·5 to 2 per cent. from the bark, leaves and twigs of *Cinnamomum cassia*, which is grown chiefly in China. The chief constituents are 70 to 90 per cent. of cinnamic aldehyde, cinnamic esters, terpenes, salicylic aldehyde, coumarin (*P. & E.O.R.*, 1916, 7), and phenylpropyl-acetate. Characters: Specific gravity, 1·050 to 1·065; optical rotation, $-1°$ to $+1°$: refractive index, 1·585 to 1·605: soluble in 3 to 4 vols. of 70 per cent. alcohol. The chief adulterants are resin and mineral and fatty oils. The former particularly has a very extensive use in this respect, although improvement has been made of late years (*P. & E.O.R.*, 1916, 323). Lead, derived from the containers in which the oil is shipped, must also be looked for in the oil.

Cinnamon Oil.—This is distilled to the extent of 0·5 to 1 per cent. from the bark of *Cinnamomum zelanicum*, which is grown chiefly in Ceylon. The oil contains 55 to 75 per cent. of cinnamic aldehyde, together with pinene, cymene, phellandrene, cuminic aldehyde, benzaldehyde, nonaldehyde, methyl-amyl-ketone and a small quantity of eugenol. Characters: Specific gravity, 1·000 to 1·040; optical rotation, 0 to $-1°$; refractive index at 25°, 1·565 to 1·590; soluble in 3 vols. of 70 per cent. alcohol (see *P. & E.O.R.*, 1910, 169).

The oil distilled from the leaves contains 75 to 90 per cent. eugenol and also a quantity of safrol. Its cinnamic aldehyde content is very small. Characters: Specific gravity, 1·045 to 1·065; optical rotation, $-1°$ to $+1°$; refractive index at 25°, 1·530 to 1·540: soluble in 3 vols. 70 per cent. alcohol.

The oil distilled from the roots contains eugenol, camphor, safrol, benzaldehyde, benzoic esters, etc.

Laurel Oil.—This is distilled to the extent of 1 to 3 per cent. from the leaves of *Laurus nobilis*, which is cultivated in Mediterranean countries. It has an odour resembling cajuput oil and contains chiefly cineol, geraniol, eugenol, pinene, methyl-chavicol, esters, a sesquiterpene and a sesquiterpene alcohol. Characters: Specific gravity, 0·920 to 0·936; optical rotation, −15° to −18°; ester value, 47; cineol, 25 to 50 per cent.: soluble in 3 vols. 80 per cent. alcohol.

Shiu Oil.—This is obtained from Japan and is chiefly interesting because of its high content of linalol (*P. & E.O.R.*, 1912, 111, 124, 239). Characters: Specific gravity, 0·870 to 0·883; optical rotation, −14° to −16°; refractive index, about 1·4606; total alcohols by acetylation, 47 to 62 per cent. Another interesting Japanese oil is "Kuromoji," which is obtained from the leaves and twigs of *Lindera sericea* (*P. & E.O.R.*, 1912, 263). This oil contains 40 to 50 per cent. linalol, 10 per cent. esters (as geranyl acetate), terpineol, l-carvone, dipentene and d-limonene. Its odour resembles that of coriander oil. Characters: Specific gravity, 0·890 to 0·915; optical rotation, −2° to −14°: soluble in 4 vols. 70 per cent. alcohol.

Sassafras Oil.—This is distilled to the extent of 3 to 9 per cent. from the root-bark of the smaller roots of *Sassafras officinale*, which is grown extensively in North America. Its chief constituent is 80 to 90 per cent. safrol, other constituents being pinene, camphor, phellandrene, cadinene and eugenol. Its most common adulterant is camphor oil (which see). Characters: Specific gravity, 1·065 to 1·095; optical rotation, +1° to +4°; refractive index, 1·523 to 1·530; soluble in 2 vols. 90 per cent. alcohol.

Leguminosæ.—*Copaiba Oil.*—This is derived, the yield being about 40 to 65 per cent., from the balsam or oleoresin of *Copaifera Langsdorffii* and other species, which are grown in the northern coastal sections of South America. The constituents are chiefly sesquiterpenes (caryophyllene). Characters: Specific gravity, 0·895 to 0·918; optical rotation, −7° to −25°; refractive index at 25°, 1·493 to 1·500.

Magnoliaceæ.—*Star Anise Oil.*—This is obtained to the extent of about 3 per cent. from the fruit of *Illicium verum*, which grows in Southern China and Tonkin. The chief constituents of the oil are 80 to 90 per cent. anethol, d-pinene, l-phellandrene, para-cymene, anisic aldehyde, anisic acid, methyl-chavicol, safrol, terpineol, cineol and a sesquiterpene. Characters: Specific gravity (20°/15°), 0·975 to 0·990; optical rotation, −2° to +1°; refractive index at 25°, 1·551 to 1·558; melting-point after solidification, 17° to 19°; soluble in 3 vols. 90 per cent. alcohol. Japanese star anise oil derived from *Illicium religiosum* contains eugenol, safrol and limonene: its specific gravity is about 1·006, and its optical rotation about −8°.

Malvaceæ.—*Oil of Ambrette.*—This is derived, the yield being about 0·2 per cent., from the seeds of *Hibiscus abelmoschus*, which is grown in Java and Martinique. The oil has a musk-like odour, is solid at ordinary temperatures and contains palmitic acid. Characters: Specific gravity, about 0·900; optical rotation, nil or very slightly dextro; melting-point, 30° to 35°; saponification number, 180 to 200.

Myristicaceæ.—*Nutmeg Oil.*—This is distilled to the extent of 7 to 14 per cent. from the seeds of *Myristica fragrans*, which grows in the Moluccas and especially in the Banda and Sunda Islands. Characters: Specific gravity, 0·865 to 0·930; optical rotation, +10° to +30°; refractive index at 25°, 1·475 to 1·484; soluble in 3 vols. 90 per cent. alcohol.

Mace Oil.—This is distilled from the arillus enclosing the seeds of *Myristica fragrans.* It contains chiefly myristicol, which is a mixture of geraniol, terpineol, linalol and borneol, and also 4 per cent. of myristicin, iso-myristicin, myristic acid, safrol, pinene, dipentene, camphene and small quantities of eugenol and iso-eugenol. Characters: Specific gravity, 0·890 to 0·930; optical rotation, +10° to +20°; refractive index at 25°, 1·476 to 1·480; soluble in 3 vols. 90 per cent. alcohol.

Myrtaceæ.—*Bay Leaf Oil.*—This is obtained to the extent of 2 to 3 per cent. by distillation of the leaves of *Pimenta acris,* which is grown in the West Indies (*P. & E.O.R.*, 1914, 425; 1916, 34). The chief constituents are eugenol, methyl-eugenol, phellandrene, an olefinic terpene myrcene, and also traces of citral, chavicol and methyl-chavicol. Characters: Specific gravity, 0·950 to 0·990 optical rotation, 0 to −2°; phenols (eugenol), 50 to 75 per cent.; refractive index, 1·510 to 1·520 (see *P. & E.O.R.*, 1915, 33; 1916, 295).

Cajuput Oil.—This is distilled from the leaves and twigs of *Melaleuca leucadendron,* var. *lancifolia* or *M. minor,* which grows in India, the East Indies and Australia (*P. & E.O.R.*, 1910, 121; 1911, 146; 1912, 243). The chief constituents are cineol (45 to 55 per cent.), pinene, terpineol and small quantities of benzaldehyde and valeric aldehyde. Characters: Specific gravity, 0·919 to 0·930; optical rotation, 0 to −4°; refractive index at 25°, 1·460 to 1·470; cineol, 45 to 55 per cent.; soluble in 3 vols. 80 per cent. alcohol.

Clove Oil.—This is distilled to the extent of 15 to 19 per cent. from the dried flower-buds of *Eugenia caryophyllata*, grown in Zanzibar, Penang, Madagascar, Mauritius, Seychelles, the West Indies, the Moluccas, etc. (*P. & E.O.R.*, 1917, 308). The chief constituents are eugenol (85 to 95 per cent.), esters, furfural, caryophyllene, methyl alcohol, also minute traces of amyl-methyl-ketone and methyl benzoate. Characters: Specific gravity, 1·047 to 1·065 (this increases with exposure to light and air, see *P. & E.O.R.*, 1910, 243); optical rotation, 0 to −2°; refractive index at 25°, 1·528 to 1·540; phenols (eugenol), 85 to 95 per cent.; soluble in 3 vols. 70 per cent. alcohol.

Eucalyptus Oil.—This is distilled to the extent of 0·8 to 1·5 per cent. from the dried leaves of several species of *Eucalyptus*, e.g. *Eucalyptus globulus*, *E. polybractea*, *E. dumosa* and *E. oleosa* (*P. & E.O.R.*, 1913, 281; 1914, 22; 1915, 181), which are grown in Australia, New Zealand, California, Spain and Algeria. The chief constituents are cineol, phellandrene, pinene, cymene, piperitone, aromadendral, a sesquiterpene aromadendrene, eudesmic esters, eudesmol (melting-point, 79° to 80°), also traces of aldehyde, *e.g.* butyric, caproic and valeric, and amyl alcohol. Other eucalyptus oils are derived from *E. macarthuri*, *E. aggregata* and *E. staigeriana*. The first of these contains 60 to 75 per cent. of geraniol principally as geranyl acetate, the second contains upwards of 50 per cent. amyl eudesmate and the last contains l-limonene and citral. Oil of *E. citriodora* contains no cineol, the chief constituent being citronellal together with esters. Oil of *Backhousia citriodora* is composed almost wholly of citral (*P. & E.O.R.*, 1912, 241; 1915, 92, 182). Oil of *E. amygdalina* is composed largely of phellandrene. Characters of Eucalyptus oils: Medicinal Oil (globulus)—Specific gravity, 0·910 to 0·930;

optical rotation, −10° to +10°; refractive index at 25°, 1·459 to 1·470; cineol, 55 to 80 per cent.; soluble in 3 to 10 vols. 70 per cent. alcohol. Amygdalina Oil—Specific gravity, 0·885 to 0·890; optical rotation, −25° to −80°; refractive index, 1·475 to 1·485; cineol, 5 to 25 per cent. Citriodora Oil—Specific gravity, 0·865 to 0·905; optical rotation, 0 to +2°; refractive index, 1·455 to 1·460; citronellal, 80 to 95 per cent.; soluble in 4 to 5 vols. 80 per cent. alcohol. Oil of Backhousia Citriodora—Specific gravity, 0·895 to 0·900; refractive index, 1·4889; citral, 90 to 95 per cent.; soluble in 3 vols. 70 per cent. alcohol (see *P. & E.O.R.*, 1918, 249).

Pimento Oil.—This is obtained to the extent of 3 to 4·5 per cent. from the unripe fruit of *Pimenta officinalis*, which is grown in the West Indies. The chief constituents are eugenol, the methyl ether of eugenol, l-phellandrene, caryophyllene, cineol, palmitic acid and resin. Characters: Specific gravity, 1·040 to 1·055; optical rotation, 0 to −4°; refractive index at 25°, 1·508 to 1·535; phenols (eugenol), 60 to 75 per cent.; soluble in 2 vols. 70 per cent. alcohol.

Oleaceæ.—*Jasmine Oil.*—This is obtained by extraction of pomade, made by the enfleurage process (see later section), with acetone (*P. & E.O.R.*, 1916, 36). *Jasminium grandiflorum* is usually the species used in the South of France for the preparation of the pomade. The composition of jasmine oil is roughly as follows: Benzyl acetate, 65 per cent.; linalol, 16 per cent.; linalyl acetate, 7·5 per cent.; benzyl alcohol, 6 per cent.; together with small quantities of geraniol, methyl anthranilate, para-cresol, indol and a ketone "jasmone." Characters: Specific gravity, 1·006 to 1·018; optical rotation, +2° 30′ to +3° 30′; esters (as linalyl acetate), 90·3 to 95·4 per cent.; esters (as benzyl acetate), 69·1 to 73·0 per cent.

Piperaceæ.—*Cubeb Oil.*—This is distilled in steam to the extent of 10 to 18 per cent. from the dried, unripe fruit of *Piper cubeba*, which is grown chiefly in Java. It is used in the compounding of certain liqueurs. Its chief constituents are l-pinene, camphene, dipentene, cadinene and cubeb camphor (melting-point, 68° to 70°). Characters: Specific gravity, 0·910 to 0·930; optical rotation, $-25°$ to $-40°$; refractive index at 25°, 1·485 to 1·502; soluble in 3 to 10 vols. 90 per cent. alcohol.

Pepper Oil.—This is distilled from the unripe berries of *Piper nigrum*. It contains chiefly phellandrene and sesquiterpenes. Characters: Specific gravity, 0·870 to 0·905: optical rotation, $-5°$ to $+2°$.

Rosaceæ.—*Bitter Almond Oil.*—This is distilled, with a yield of 0·5 to 1·0 per cent., from the seeds of *Prunus amygdalus amara*, after the same have been pressed and macerated with water. Essential oil of apricot and peach kernels is similar in composition to oil of bitter almonds. The essential oil is produced during the maceration process by the action of the enzyme emulsin on the glucoside amygdalin. The chief constituents of the oil are benzaldehyde, hydrocyanic acid and mandelic nitrile. Characters: Specific gravity, 1·045 to 1·060; optical rotation, nil; refractive index, 1·542 to 1·556; soluble in 2 vols. 70 per cent. alcohol. The oil is also sold "free from prussic acid." It is deprived of this body by treatment with ferrous sulphate and lime and redistilling (*P. & E.O.R.*, 1916, 276).

Cherry Laurel Oil.—This oil, which is very similar to bitter almond oil, is distilled from the leaves of *Prunus laurocerasus*, which is grown in Italy, Switzerland, Germany, etc. Its chief constituents are benzaldehyde, prussic acid and a trace of benzyl alcohol. Characters: Specific gravity, 1·050 to 1·065; optical rotation, nil.

Otto of Rose.—The Bulgarian otto is derived from the fresh blooms of *Rosa damascena*, which grows on the Balkan hillsides (*P. & E.O.R.*, 1916, 11, 39; 1918, 277). The chief constituents are citronellol, geraniol, nerol, phenyl-ethyl-alcohol, farnesol and about 10 to 15 per cent. of stearoptene, consisting of mixed paraffin hydrocarbons (melting-point, 34°). Characters: Specific gravity, (30°/15°), 0·851 to 0·862; optical rotation at 25°, −2° to −4°; refractive index at 25°, 1·460 to 1·465; melting-point, 18° to 22°; total alcohols by acetylation (as geraniol), 65 to 75 per cent.; citronellol by formylation, 30 to 38 per cent. Otto of rose which has been deprived of its stearoptene has characters as follows: Specific gravity, 0·882 to 0·885; optical rotation, −3° to −5°; refractive index at 25°, 1·466 to 1·468; total alcohols, 80 to 85 per cent.; citronellol, 40 to 45 per cent.

The use of French otto of rose has been gaining ground during the last few years This otto is produced from various types of roses, *e.g.* Rose de Mai, Ulrich Brunner, Paul Nabouand, etc., so that the proportion of constituents and also the characters vary considerably, a fact which is an advantage in that varying shades of rose odours can be produced. The following are, however, the characters given by one of the leading producers as average representative figures: Specific gravity at 30°, 0·864; optical rotation, −1° to −4°; ester value, 12 to 32; melting-point, 20 to 28; total alcohols, 70 to 80 per cent.: geraniol, about 32 per cent.; citronellol, variable (see also *P. & E.O.R.*, 1917, 6, 220, 279, 330; 1918, 163, 168).

Wild Cherry Bark Oil.—This is distilled to the extent of about 0·2 per cent. from the bark of *Prunus virginiana.* It resembles oil of bitter almonds both as regards constituents and characters.

Rutaceæ.—*Bergamot Oil.*—This is obtained by pressure from the fresh peel of the fruit of *Citrus bergamia*, which is grown in Southern Italy. The oil produced by distillation processes is of a distinctly inferior quality. The oil contains limonene, pinene, octylene, linalyl acetate, linalol, dipentene, camphene, dihydrocuminol, terpineol, limene (bisabolene) and bergaptene (a stearoptene melting at 188°). Characters: Specific gravity, 0·881 to 0·887; optical rotation, +8° to +20°; refractive index, 1·460 to 1·469; esters (as linalyl acetate), 35 to 40 per cent.; non-volatile residue, 5 to 6 per cent.

Buchu Oil.—This is distilled to the extent of 1 to 2 per cent. from the leaves of *Barosma betulina*, *B. crenulata* and *B. serratifolia*, which grow in South Africa. The chief constituents are diosphenol (a crystalline phenol melting at 81° and boiling at 232°), menthone, limonene and dipentene. Characters: Specific gravity, 0·935 to 0·945 (betulina oil), 0·945 to 0·960 (serratifolia oil).

Citron or Cedrat Oil.—This oil is obtained from the peel of the *Citrus medica.* Mixtures of lemon, orange and bergamot oils are occasionally offered on the market as citron oils. The chief constituents are citral, limonene, dipentene and a crystalline body which melts at 144° to 145°. The oil is not much used in commerce. Characters: Specific gravity, 0·850 to 0·870; optical rotation, +65° to +80°; refractive index, about 1·475; citral, 5 to 7 per cent.

Lemon Oil.—This is obtained from the fresh peel of *Citrus limonum*, which is cultivated chiefly in Sicily, Southern Italy, France and Spain. A new process for the recovery of the oil from the peel is mentioned in the

P. & E.O.R., 1915, 91. The chief constituents of the oil are citral, limonene, phellandrene, l- and d-pinene, limene (bisabolene), geraniol, linalol, esters, aldehydes (nonylic, octylic and decylic) and citraptene (melting-point, 143°). Characters: Specific gravity, 0·857 to 0·862; optical rotation, +57° to +65°; refractive index at 25°, 1·473 to 1·476; citral, 3·5 to 5 per cent.

Limes Oil.—The *hand-pressed* variety is obtained from the fruit of *Citrus limetta*, which grows in Italy, the *distilled* variety being obtained from *Citrus medica* var. *acida*, which is cultivated in the West Indies. The two oils differ in both composition and characters. The constituents of the hand-pressed oil are as follows: Citral (6 to 10 per cent.), limonene, dipentene, pinene, linalol, linalyl acetate and a stearoptene. Its characters are: Specific gravity, 0·872 to 0·885; optical rotation, +35° to +40°; refractive index, 1·476 to 1·485. The distilled oil contains chiefly limonene, pinene, dipentene, cymene and limene, but no citral. Its characters are: Specific gravity, 0·856 to 0·868; optical rotation, +34° to +54°; refractive index, 1·470 to 1·478.

Neroli Oil.—The "Bigarade" oil is obtained from the fresh flowers of the bitter orange. The "Portugal" variety is obtained from the flowers of the sweet orange. Both oils are produced chiefly in the South of France, also to some extent in Spain (*P. & E.O.R.*, 1913, 137, 375). The chief constituents of neroli oil are nerol, linalol and geraniol and their esters, terpineol, limonene, pinene, camphene, dipentene, jasmone, methyl-anthranilate, stearoptene and a a trace of indol. Characters: Specific gravity, 0·870 to 0·885; optical rotation, 0 to +5°; refractive index, 1·468 to 1·477; esters (as linalyl acetate), 7 to 20 per cent.; soluble in 3 vols. 80 per cent. alcohol.

Orange Oil.—This is obtained from the fresh peel of *Citrus aurantium* and *C. bigaradia*, the former being the sweet orange oil of commerce and the latter the bitter orange oil. The chief constituents are limonene, terpineol, methyl-anthranilate, linalol, citraptene and less than 1 per cent. of aldehydes (citral, decylic aldehyde, etc.). Characters: Bitter Orange Oil—Specific gravity, 0·848 to 0·853; optical rotation, +91° to +98°; refractive index, 1·472 to 1·478. Sweet Orange Oil — Specific gravity, 0·846 to 0·852; optical rotation, +96° to +99°; refractive index, 1·472 to 1·478. The variety known as Mandarin Oil, which is derived from *C. Madurensis*, contains chiefly limonene, and has the following characters: Specific gravity, 0·855 to 0·859; optical rotation, +65° to +75°.

Petitgrain Oil.—This is derived from the leaves and young shoots of the bitter and sweet orange, the former being known as "Bigarade." An inferior grade is produced in Paraguay, the best oils being obtained from France, Spain and Italy. The chief constituents are linalol and geraniol and their esters, limonene, sesquiterpenes and stearoptene. Paraguay petitgrain oil contains pinene, camphene, dipentene, terpineol and esters of geraniol. The characters of the two types of oil are as follows: Continental—Specific gravity, 0·885 to 0·900; optical rotation, −5° to +3°; refractive index, 1·462 to 1·465; esters (as linalyl acetate), 50 to 80 per cent.; soluble in 3 vols. 70 per cent. alcohol. Paraguay—Specific gravity, 0·884 to 0·895; optical rotation, −2° to +5°; refractive index, 1·462 to 1·465; esters (as linalyl acetate), 35 to 65 per cent.; soluble in 2 to 3 vols. 80 per cent. alcohol.

Santalaceæ.—*Sandalwood Oil.*—This is distilled, using high-pressure steam, from the wood of *Santalum album*, the yield being 2 to 6 per cent. The trees are grown chiefly in India (*P. & E.O.R.*, 1918, 18, 182) and an inferior oil is produced by the natives using open fire stills. Sandalwood oils of other derivations are described in *P. & E.O.R.*, 1911, 11, 79; 1913, 163. The chief constituents are 90 to 97 per cent. of santalol (*Pharm. Journ.*, ii./08, 624; *Chem. & Drug.*, ii./09, 581), which is a mixture of sesquiterpene alcohols, the aldehyde santalal, two sesquiterpenes α- and β-santalene and santene (*P. & E.O.R.*, 1910, 192). Characters: Specific gravity, 0·973 to 0·985; optical rotation, −13° to −21°; refractive index at 25°, 1·500 to 1·510; saponification number, 5 to 15; soluble in 6 vols. 70 per cent. alcohol (see *P. & E.O.R.*, 1916, 47, 116, 210, 254, 278; *Am. Journ. Pharm.*, 1911, 335).

Umbelliferæ.—*Ajowan Oil.*—This is distilled to the extent of 3 to 4 per cent. from the fruits of *Carum ajowan* and *C. copticum,* which are grown in India, Persia, Afghanistan and Egypt. The thymol of commerce is obtained chiefly from ajowan oil. The chief constituents are thymol, which crystallises out on cooling, cymene and terpinene. Characters: Specific gravity, 0·900 to 0·930; optical rotation, +1° to +2°; thymol 40 to 55 per cent. soluble in 3 vols. 70 per cent. alcohol.

Angelica Oil.—This is distilled to the extent of 0·3 to 1·0 per cent. from the dried root, leaves and stems of *Angelica archangelica,* which is grown in Greenland, Russia and Siberia. It contains pinene, phellandrene, cymene, oxymyristinic acid, oxy-penta-decylic acid, valerianic acid and a crystalline lactone melting at 83°. Characters: Specific gravity, 0·857 to 0·918; optical rotation, +16° to +32°; refractive index, 1·479 to 1·482.

Anise Oil.—This is distilled to the extent of 1·5 to 6·0 per cent. from the fruits of *Pimpinella anisum*, which is grown in France, Holland, Germany, Russia, etc. Its chief constituents are 80 to 90 per cent. of anethol, together with methyl-chavicol. Characters: Specific gravity (20°/15°), 0·975 to 0·990; optical rotation, 0 to −2°; melting-point, 15° to 19°; soluble in 3 to 5 vols. 90 per cent. alcohol (*P. & E.O.R.*, 1916, 59). It is sometimes adulterated with fennel oil (see later). As the oil ages its specific gravity and solubility increase owing to the formation of oxidation products.

Caraway Oil.—This is distilled to the extent of 4 to 7 per cent. from the ripe fruit of *Carum carvi*, which is cultivated in Europe and Asia. It contains chiefly carvone and limonene. Characters: Specific gravity, 0·907 to 0·920 (the *B.P.* requires the specific gravity to be not under 0·910); optical rotation, +70° to +82°; refractive index at 25°, 1·485 to 1·497; carvone, 50 to 60 per cent.; soluble in 10 vols. 80 per cent. alcohol.

Celery Oil.—This is distilled to the extent of 2·5 to 3·0 per cent. from the fruit of *Apium graveolens*, which is largely cultivated in European countries. The chief constituents are about 90 per cent. of terpenes, consisting of limonene, together with a sesquiterpene, selinene, and traces of guaiacol, a phenol melting at 66° to 67°, sedanolid (a lactone), sedanolic acid, sedanonic anhydride and palmitic acid. Characters: Specific gravity, 0·870 to 0·895; optical rotation, +67° to +79°; refractive index, 1·479 to 1·481; soluble in 10 vols. 90 per cent. alcohol.

Coriander Oil.—This is distilled to the extent of 0·4 to 1·0 per cent. from the ripe fruits of *Coriandrum sativum*, which is grown in France, Italy, Holland and Russia (*P. & E.O.R.*, 1918, 70). The known constituents of the oil are d-linalol and d-pinene. Characters: Specific gravity, 0·870 to 0·885; optical rotation, +8° to +14°; refractive index at 25°, 1·463 to 1·467; alcohols by acetylation, 45 to 55 per cent.; soluble in 3 vols. 70 per cent. alcohol.

Cummin Oil.—This is distilled to the extent of 2·5 to 4·0 per cent. from the fruits of *Cummin cyminum*, which is grown in Malta, Egypt, Morocco, Syria, India, the East Indies, etc. (*P. & E.O.R.*, 1918, 70). It is also known as Roman caraway oil and contains cuminic aldehyde, cymene and a terpene which boils at 156° (*P. & E.O.R.*, 1913, 43). Characters: Specific gravity, 0·893 to 0·930; optical rotation, +4° to +8°; refractive index, 1·497 to 1·509; cuminic aldehyde, 20 to 30 per cent.; soluble in 3 to 10 vols. 80 per cent. alcohol.

Dill Oil.—This is obtained to the extent of 3 to 4 per per cent. by distilling the fruit of *Peucedanum graveolens*, which is cultivated in England, Germany, the Balkans, etc. It contains 40 to 60 per cent. of carvone, also phellandrene and limonene. Characters: Specific gravity, 0·895 to 0·915; optical rotation, +70° to +80°; refractive index at 25°, 1·477 to 1·488; soluble in 5 to 8 vols. 80 per cent. alcohol and in 3 vols. 90 per cent. alcohol (*P. & E.O.R.*, 1917, 349).

Fennel Oil.—This is distilled, with a yield of 4 to 6 per cent., from the fruits of *Foeniculum vulgare*, which is cultivated in France, Italy, Germany, India, Japan, etc. It contains 50 to 60 per cent. anethol, also fenchone, dipentene and d-pinene. Characters: Specific gravity, 0·960 to 0·990; optical rotation, +6° to +24°; refractive index, 1·525 to 1·550; melting-point (after solidification), 3° to 6°; soluble in 5 to 8 vols. 80 per cent. alcohol.

Parsley Oil.—This is distilled to the extent of 2 to 6 per cent. from the fruit of *Petroselinum sativum*, which grows in many countries of moderate climate. The chief constituents are apiol and terpenes. Characters: Specific gravity, 1·050 to 1·100; optical rotation, −5° to −10°; refractive index, 1·488 to 1·519.

Verbenaceæ.—*Verbena Oil.*—This is distilled from *Verbena officinalis* and *V. triphylla.* It is largely substituted in commerce by lemon-grass oil, which resembles it to some extent. The chief constituents of the oil are citral, limonene, a stearoptene, a sesquiterpene and geraniol. Characters: Specific gravity, 0·900 to 0·925; optical rotation, −8° to −16°; citral, 20 to 38 per cent.; soluble in 3 vols. 80 per cent. alcohol. An oil derived from *Aloysia citriodora* or *Lippia citriodora* is termed Spanish Verbena oil (*P. & E.O.R.*, 1915, 185).

CHAPTER VII

TERPENELESS AND SESQUITERPENELESS OILS

THE production of concentrated essential oils by the elimination of the terpene and sesquiterpene hydrocarbons, which are valueless as regards flavouring capacity, has assumed quite extensive proportions of late years. These concentrated oils are particularly useful in the preparation of soluble essences for the mineral water trade and for purposes which require more soluble flavouring materials than the ordinary essential oils. The manufacture of concentrated oils is, however, a matter of considerable difficulty, requiring much skill and experience; it frequently occurs that the delicate aroma of an oil is spoiled by careless concentration, and it is generally recognised that it is not advisable to carry such concentration too far, the oxygenated constituents which form the odorous principles of the oils being particularly susceptible to heat and to the general conditions involved in the concentration process.

In the purchasing of concentrated oils it is advisable that they should be stipulated "terpeneless" or "terpeneless and sesquiterpeneless," as many oils sold under the label "concentrated" are either ordinary essential oils containing an admixture of additional odorous constituent or purely mixtures compounded from synthetic odorous bodies.

Most terpeneless and sesquiterpeneless oils are produced by fractional distillation of the ordinary essential oils under reduced pressure. The terpene constituents distil over first, then the sesquiterpenes, and finally the odorous constituents, or the latter may be steam distilled after eliminating the sesquiterpenes. Another method of obtaining oils free, to a great extent, from terpenes and sesquiterpenes is to mix them first with weak alcohol and then distil. The distillate separates into two layers, the top being terpenes and the bottom an alcoholic solution of the odorous constituents. The latter is returned to the still and redistilled. At first the alcohol carries with it a proportion of oxygenated odorous bodies, but as the alcoholic strength of the solution in the still diminishes the terpeneless oil separates out in an oily layer. This second process is more troublesome than the ordinary distillation under reduced pressure, but is said to yield concentrated oils of greater delicacy.

The following gives some of the chief points of interest with regard to the principal concentrated oils:

Terpeneless Oils.—*Angelica Oil* has about twenty times the concentration of the ordinary essential oil; its solubility is about 2·5 parts per 100 parts of 70 per cent. alcohol and 85 parts per 100 parts of 80 per cent. alcohol. It has a specific gravity of about 0·951 and a rotation of about −3°.

Aniseed Oil is 1·5 to 2 times as concentrated as the natural oil, its solubility being 3 vols. per 100 vols. 70 per cent. alcohol, and 9 vols. per 100 vols. 80 per cent. alcohol.

Bay Oil is 2 to 3 times as strong as the ordinary oil. It has excellent solubility, 35 vols. being soluble in 100 vols. 60 per cent. alcohol and 90 vols. in 100 vols. 70 per cent. alcohol. Its specific gravity is about 1·03 to 1·04.

Bergamot Oil has 2·5 to 3 times the strength of the natural oil, 9 vols. being soluble in 100 vols. of 60 per cent. alcohol and 50 vols. in 100 vols. of 70 per cent. alcohol. Characters: Specific gravity, 0·883 to 0·888; optical rotation, −7° to −10°.

Caraway Oil is about double the strength of the ordinary essential oil. Its solubility is 25 vols. in 100 vols. 60 per cent. alcohol and 60 vols. in 100 vols. 70 per cent. alcohol. It has a specific gravity of about 0·963 to 0·965 and an optical rotation of +58°.

Celery Oil has eight times the concentration of the natural oil.

Cinnamon Oil is 1·5 to 2 times the strength of the natural oil. It has a solubility of 60 vols. in 100 vols. 70 per cent. alcohol.

Citronella Oil is about twice the strength of the ordinary oil. It has a solubility of 2 parts per 100 of 70 per cent. alcohol and 150 parts per 100 of 80 per cent. alcohol. Its specific gravity is about 0·91 to 0·92.

Clove Oil is 1·5 times the strength of the ordinary oil, 50 vols. being soluble in 100 vols. 60 per cent. alcohol and 100 vols. in 100 vols. 70 per cent. alcohol. The specific gravity is about 1·07, and the optical rotation nil.

Coriander Oil is about 1·5 to 2·5 times the strength of the natural oil. It is soluble 25 vols. in 100 vols. 60 per cent. alcohol, and 60 vols. in 100 vols. 70 per cent. alcohol.

Geranium Oil is 1·5 to 2 times the strength of the ordinary oil, 60 vols. being soluble in 100 vols. 70 per cent. alcohol. It has a specific gravity of 0·89 to 0·90, and an optical rotation of −1° to −2°.

Ginger Oil is 5 to 10 times the strength of the natural oil. It has a specific gravity of about 0·90 to 0·91.

Hop Oil has 8 times the flavouring strength of the natural oil.

Lavender Oil has about 1·75 to 2 times the strength of the ordinary oil. Its solubility is about 15 parts per 100 parts of 60 per cent. alcohol, and 55 parts per 100 parts of 70 per cent. alcohol. The chief constituents are linalol and linalyl acetate. Characters: Specific gravity, 0·893 to 0·898; optical rotation, about $-5°$.

Lemon Oil is the chief of these products and is about 20 times the strength of the ordinary oil. Its solubility is 10 to 20 vols. in 100 vols. 70 per cent. alcohol, and in all proportions of 90 per cent. alcohol. Characters: Specific gravity, 0·896 to 0·900; optical rotation, usually 0 to $-8°$; hydrocarbon content, 20 to 25 per cent. Sachsse & Co. give a process in the *P. & E.O.R.*, 1914, 262, for the estimation of the latter figure, as well as some useful data regarding terpeneless and sesquiterpeneless lemon oils. Much sophistication is practised in the adding of lemon grass citral to the oils. Such oils usually have the characteristic lemon-grass or verbena odour.

Lemon-grass Oil consists principally of citral and has a strength of about 1·5 times that of the natural oil. Its solubility is about 60 vols. in 100 vols. 70 per cent. alcohol. It has a specific gravity of 0·896 to 0·897, and an optical rotation of $-1°$ to $-1° 40'$.

Limes Oil (distilled) is about 12 to 16 times stronger than the ordinary oil. It has a specific gravity of about 0·92 to 0·93, and an optical rotation of about $-2°$.

Mandarin Oil has about 60 to 70 times the strength of the natural oil, its solubility being about 1·5 to 3 vols. per 100 vols. of 70 per cent. alcohol, and in about equal proportions with 80 per cent. alcohol.

Orange Oil is 35 to 50 times stronger than the ordinary oil, its solubility being about 50 parts per 100 parts of 70 per cent. alcohol. The specific gravity is usually about 0·894, but the optical rotation varies between wide limits according to the degree to which the terpenes have been extracted.

Rosemary Oil has about 6 times the concentration of the ordinary oil, its solubility being 20 vols. in 100 vols. 60 per cent. alcohol, and about 60 to 70 vols. in 100 vols. 70 per cent. alcohol. It has a specific gravity of 0·939 to 0·94.

Sassafras Oil is about 1·5 times the strength of the natural oil. It is soluble 4 vols. in 100 vols. 70 per cent. alcohol, and 15 vols. in 100 vols. 80 per cent. alcohol. Characters: Specific gravity, about 1·088 to 1·09; optical rotation, +1° to +2°.

Sesquiterpeneless Oils. — These are in most cases slightly stronger than the terpeneless oils and have increased solubilities, being mostly soluble in all proportions of 60 per cent. alcohol. The following are the characters of a typical sesquiterpeneless lemon oil: specific gravity, 0·9003; optical rotation, −3° 35′; citral (Burgess method), 61 per cent.

SECTION III

FLORAL, FRUIT AND COMPOUNDED FLAVOURS

CHAPTER VIII

THE PREPARATION OF FLORAL PERFUMES

THE two chief methods of preparing floral perfumes are by distillation and by extraction. The former of these methods has been described under the Preparation of Essential Oils, and consequently will not be dealt with further in this section. Distillation is used in the preparation of rose, lavender and orange flower perfumes. For some of the more delicate products, however, this process is too drastic, and extraction by means of volatile solvents or by fixed oils is practised.

EXTRACTION BY VOLATILE SOLVENTS

The apparatus employed for the extraction of the odorous constituents of flowers by means of volatile solvents resembles a large-scale Soxhlet apparatus. The flowers are placed in a suitable chamber and impregnated with the volatile solvent, means being provided for siphoning off the solvent when it reaches a certain level in the extraction chamber. The solvent, containing the odorous constituents of the flowers, together with other bodies such as flower wax, is evaporated at a low temperature, the vapours being condensed and the solvent again supplied to the extraction chamber. In the hands of experts this method of extraction is almost ideal.

The solvent used is frequently petroleum ether of low boiling-point, this liquid fulfilling most of the desideratums of a solvent for such a purpose. It is without chemical action on the odorous material; it has no residual odour after evaporation; it dissolves all the odorous constituents of the flowers; it boils at a very low temperature and is comparatively cheap.

The residual floral perfumes which remain after evaporating the solvent are termed "concretes." They are usually semi-solid and contain, in addition to the odorous bodies, the flower wax and natural colouring matter. From these "concretes" natural flower oils or "absolutes," of great concentration and solubility, are obtained by methods which entail the precipitation of the wax. This wax is of no odour value but retains the perfume of the flowers with very great tenacity, a fact which makes the using of "concretes" a matter of some difficulty. The flowers for which this process is well adapted are as follows: Carnation, cassie, hyacinth, jasmine, jonquil, mignonette, narcissus, orange, rose, tuberose and violet.

EXTRACTION BY NON-VOLATILE SOLVENTS

The solvents used in this case are either vegetable or mineral oils or fats. Perfumed oils or pomades are produced by macerating successive lots of the flowers in warm oils or fats until the latter are saturated with perfume. The process is only suitable for flowers which have a fairly stable perfume, such as cassie, orange, rose and violet. The more delicate flowers are treated by a process which is known as "cold enfleurage." This process entails the use of sheets of glass, or other material, in wooden frames, the whole being termed the *chassis.* Both surfaces of the glass are covered with perfectly odourless fat, and flowers are spread on the upper surface. The chassis are then stacked, so that each layer of flowers has fat above and below it, for a sufficient time to permit the flowers to become exhausted of perfume. The process is repeated exactly as before, using fresh flowers, except that the chassis are reversed, that is, the undersides are now turned uppermost and the flowers strewn upon them; they are then left until the flowers are exhausted, and so on until the fat is saturated with perfume. Up to recent years the operations of the cold enfleurage process were performed entirely by hand. In 1918, however, Messrs. Lautier Fils patented a machine for stripping the flowers from the chassis after exhaustion, and saving thereby much of the perfumed fat (see *P. & E.O.R.*, 1918, 326).

The fats, lards, pomades or perfumed oils are repeatedly extracted with alcohol, which is then chilled to remove the fat, and in this manner floral extracts are obtained which serve as the basis for the preparation of ottos for the flavouring of cachous, creams, etc.

PRINCIPAL FLOWERS AND THEIR ODOURS

Carnation.—The carnation, œillet, or *Dianthus caryophyllus*, is indigenous to Northern Africa and Europe. It is grown extensively in France for its perfume. More than two thousand many-tinted varieties are in existence. The carnation plant is propagated from seeds, layers and suckers. The perfume of the carnation has a decidedly clove-like odour; most synthetic carnation ottos are in fact built up on a basis of iso-eugenol together with methyl iso-eugenol.

Cassie.—Cassie perfumes are prepared from the flowers of *Acacia Farnesiana,* which was indigenous to the West Indies, but is now grown in many countries, for example, America, Africa, India, the Philippines, the Levant and Southern France. The plants are propagated from seeds, and the bushes at maturity reach a height of 15 to 16 feet. The crop of blossoms is taken from October to February, each tree yielding about 1½ to 2 lb. of flowers. Cassie perfumes are produced by extracting the flowers with both fixed and volatile solvents. The pomade is produced by the warm maceration method, and perfumed oils are also produced by extracting the flowers with almond or olive oil. Concretes and absolutes of cassie, produced by extraction with volatile solvents, are offered on the market, and these form convenient articles, from a perfumery standpoint, because of their concentration. Other species of cassie flowers, such as those derived from *A. cavenia* and *A. dealbata* (Wattle), are worked up for their odorous constituents. The constituents which up to the present have been found in cassie extracts are: geraniol, linalol, farnesol, benzyl alcohol, anisic aldehyde, cuminic and decylic aldehydes, methyl salicylate and a violet-like ketone.

Heliotrope.—Many species of heliotrope are in existence, the plant which grows in this country being *Heliotropium peruvianum.* The heliotrope flowers grown in Southern France are macerated for twenty-four hours in melted fat to which they yield their perfume. Heliotrope extract is produced by shaking the pomade with alcohol.

Hyacinth.—The principal country as regards hyacinth culture at present is Holland. Although many hyacinth perfumes and flavourings occur on the market, they are chiefly of synthetic derivation. It has been found to be exceedingly difficult to obtain satisfactory results from the flower. Enfleurage cannot be practised on account of the flowers being gathered damp. Extraction with volatile solvents yields as good results as can be obtained with this flower, and it has been demonstrated that the best yields of concretes are obtained from light-coloured single blooms.

Jasmine.—The jasmine which is grown in Southern France for its perfume is the Spanish variety *Jasminium grandiflorum.* It is grafted upon cuttings of English jasmine, or white jasmine (*J. officinale*). The flowers are harvested from July to October, the midday from 11 a.m. to 3 p.m. being the most suitable time for collection. The cold enfleurage process was until recently mainly used for the extraction of the perfume. Much of the crop is now treated with volatile solvents for the production of concretes and absolutes. The following are the constituents of natural jasmine perfume: benzyl acetate, linalol, linalyl acetate, benzyl alcohol, jasmone, indol and methyl anthranilate. In the *Chem. Zeit.*, 1910, 34, 910, mention is made of the finding of geraniol, cinnamyl acetate, paracresol and p-cresol methyl ether.

Jonquil.—The jonquil (*Narcissus jonquilla*, N.O. Amaryllidaceæ) is cultivated at Grasse in Southern France for its perfume. The bulbs are planted in September and they flower in March. The perfume is extracted by enfleurage and by maceration with oils.

Lily of the Valley.—Most lily of the valley perfumes of commerce are derived from synthetic sources. The natural perfume is chiefly obtained from the *Convallaria majalis*, although the flowers of other plants are sometimes used in the production of "Muguet." The perfume is extracted by the enfleurage process, and on account of the small quantity of odorous material contained in the flowers such pomades are very expensive.

Rose.—Otto of rose, produced by distillation, has been described under Essential Oils. In recent years, however, concretes and absolutes produced by extraction with volatile solvents have been offered on the market.

Tuberose.—The tuberose, *Polyanthes tuberosa*, was indigenous to Mexico, but is now largely grown in Southern France for its perfume. The harvest is gathered from July to October, and the perfume is extracted by both volatile solvents and enfleurage. The perfume obtained by enfleurage contains methyl anthranilate, methyl salicylate, methyl benzoate, benzyl benzoate and benzyl alcohol.

Violet.—The violet, *Viola odorata*, is grown over a very wide area. Its cultivation for perfumery purposes is, however, chiefly carried on near Grasse in Southern France. The crop is gathered in November and December, the flowers being picked in the morning. The flowers are extracted with volatile solvents and with hot oils and fats. Violet leaves are also extracted with volatile solvents to produce a semi-solid, green paste which has the odour of the leaf and which is used in violet perfumes.

CHAPTER IX

NATURAL FRUIT PRODUCTS

Fruit Juices and Syrups.—Natural fruit juices, just as pressed from the fruit, that is, in an unconcentrated condition, but which have been specially treated to prevent deterioration, have a widespread usage in many branches of foodstuff manufacture. The preparation of the juices is mainly as follows: The fruits are first crushed between stone rollers and then pressed to separate the juice from the pulp. The flavours of several fruit juices, such as black currants, gooseberries, blackberries, pears, apples, plums, apricots, etc., are developed by a fermentation process previous to pressing; others, such as strawberry, pine-apple, etc., are spoiled by fermentation, and the fruit must therefore be treated in a fresh condition. When such a fermentation is to be permitted, about 3 per cent. of sugar is added to the crushed fruits and the mass allowed to stand for a week at a temperature of 80° F. The development of alcohol in the mixture assists the precipitation of the pectinous matters, so that when the mass is pressed the resultant juice is clearer than that prepared from fresh fruit. After pressing, the residual pulp is washed with a small quantity of water to remove the remaining juice. The juice is then sterilised at a temperature of 180° F. and stored in hermetically sealed containers or in sulphured casks. During storage the final clarification of the juice occurs.

A recent method of preparing fruit juice is described in *Cal. Citrog.*, 1920, 5, 264. In this method the juice is heated to 85° C. in a special pasteuriser, and then allowed to settle at 50° C. It is then mixed with kieselguhr and filtered through a filter press, again sterilised and finally run into a sterile receptacle. Bulletin No. 241 of the United States Department of Agriculture, entitled "Studies in Fruit Juices," gives the result of some experiments in the preparation and storage of fruit juices.

Syrups are made by mixing 13 parts of sugar with 7 parts of fruit juice and heating to about 170° F. to coagulate the albumen, which is then filtered off. If instead of heating in an open vessel the syrup is brought to the boil in a vacuum still, the vapours which distil over can be condensed, and the first runnings, which contain odorous bodies from the fruit, added to the syrup.

Concentrated Fruit Juices.—Concentrated fruit juices, fruit extracts or "concretes" of fruits are prepared by several methods, most of which involve the concentration under reduced pressure of the juice at as low a temperature as possible. The usual method of making concentrated fruit juice is to distil the ordinary juice in a vacuum still at a low temperature until the required concentration is achieved, collecting the first 10 per cent. of the distillate, which contains the volatile odorous principles of the juice. This first 10 per cent. of the distillate is again distilled under reduced pressure, and the first runnings added to the concentrated fruit juice, which is the residue in the first distillation.

If it is desired to obtain a concentrated juice free from pectin, the ordinary juice is concentrated in vacuum to a certain bulk, an equal volume of alcohol added to precipitate the pectin and the liquid is filtered. The alcohol is then distilled off, the first runnings being redistilled and added to the concentrated juice.

Ger. Pat., 285, 304, 1914, describes the preparation of concentrated fruit juice by first dialysing the ordinary juice in a cold chamber, to reduce its acid content, and then concentrating under reduced pressure.

Fruit Aromas.—Natural fruit aromas or aroma oils are obtained by first extracting the fruit several times with alcohol, then shaking the alcoholic extract with specially purified petroleum ether. The petroleum ether extract is distilled at a low temperature, and there remains as residue the odorous constituents of the fruit, together with fruit wax. The wax is eliminated by dissolving the mass in alcohol and chilling the solution, when the wax separates out and may be filtered off.

CHAPTER X

COMPOUNDED FLAVOURS

NUMEROUS books have been published during the last few years giving hundreds of perfumery and flavouring essence recipes of more or less value, and to these the reader must refer if he is in search of such. In preference to following this procedure it is proposed to indicate the more important types of flavours (the principal ingredients being marked A), making especial note of the bodies (marked B) which are used for varying these types to yield distinctive products. The figures quoted represent quantities which are in all cases given merely to form a basis for the evolutions of new flavours.

Bitters.—These are prepared by macerating mixtures of vegetable bodies with equal quantities of alcohol and water. The following are types of the most important:

Angostura.—(A) Angostura bark 20, bitter orange peel 5, cinnamon bark 5, cinchona bark 5, tonka beans 5, galanga root 5, cardamoms 10, ginger 5, cloves 10; (B) zedoary root, cassia buds, gentian root, carob, calisaya bark, lemon peel, chamomile.

Hamburg.—(A) Cinnamon bark 20, cassia buds 20, ginger 10, gentian root 15, cardamoms 5, bitter orange peel 2; (B) nutmeg, cloves, zedoary root, centaury, galanga root, grains of Paradise, quassia, laurel berries, buckbean leaves.

Hop.—(A) Hops 20, centaury 2, wormwood 1, gentian 1; (B) zedoary, galanga root.

Orange.—(A) Bitter orange peel 20, gentian root 15, citron peel 10, cascarilla bark 5; (B) ginger, cusparia, chiretta.

Essences for bitters are also prepared by the addition of essential oils to the extracts prepared as above from vegetable bodies.

Bouquet Essences. — Natural floral flavours are in many cases strengthened and cheapened by judicious additions of synthetic perfumes, the types of synthetic perfumes mentioned later being suitable for this purpose. Many floral and bouquet perfumes, which are used as flavouring agents, are compounded from combinations of essential oils, floral extracts, animal and balsam tinctures and synthetic products, the following being types:

Ambre Bouquet.—(A) Essence of musk 10, vanillin 1, essence of ambergris 40, benzyl acetate 2, otto of rose 1, tinct. of benzoin 2, tinct. of styrax 2; (B) patchouli oil, geraniol.

Bouquet de Fleurs.—(A) Lemon oil 2, orange oil 2, bergamot oil 3, lavender oil 1, triple extract of rose 4, triple extract of violets 4; (B) tinct. of musk, tinct. of ambergris, vanilla.

Carnation Bouquet.—(A) Triple extract of rose 20, orange flowers 20, violet 10, cassie 10, vanillin 2, oil of bitter almonds 1; (B) oil of ylang-ylang, tinct. of civet, oil of cloves.

Honeysuckle.—(A) Triple extract of rose 40, vanillin 1, benzaldehyde 1, triple extract of jonquil 50, methyl anthranilate 1, oil of orris 10; (B) tinct. of benzoin, esters of geraniol and citronellol, pimento oil, cedrat oil, origanum oil.

Jasmine.—(A) Linaloe oil 1, neroli oil 1, triple extract of jasmine 40, bergamot oil 2; (B) tinct. of ambergris, methyl anthranilate.

Lilac.—(A) Tinct. of civet 10, oil of bitter almonds 1, neroli oil 2, triple extract of tuberose 30; (B) benzyl acetate, ylang-ylang oil, terpineol.

Narcissus.—(A) Triple extract of tuberose 20, otto of rose 1, triple extract of jonquil 20, benzyl acetate 2; (B) tincts. of styrax and tolu, vanillin, heliotropin, neroli oil.

Opoponax.—(A) Neroli oil 1, otto of rose 1, triple extract of jasmine 10, tinct. of musk 5, vanillin 2, oil of bergamot 3, oil of lemon 1; (B) patchouli oil, tinct. of orris, ionone, tinct. of benzoin.

Stephanotis.—(A) Tinct. of musk 20, tinct. of orris 40, oil of bergamot 2, otto of rose 2, neroli oil 2; (B) triple extracts of tuberose, jasmine, linalyl acetate, ylang-ylang oil.

Violet.—(A) Ionone 100 per cent. 10, oil of orris 20, triple extract of violet 30, otto of rose 1, triple extract of jasmine 5; (B) linalyl acetate, triple extracts of cassie and jonquil, tinct. of musk, ylang-ylang oil, vanillin.

Cake Flavours.—These are usually mixtures of essential oils and synthetic materials diluted with alcohol. The following are types:

Butter Ess.—(A) Butyric acid 100 per cent. 20, sweet orange oil 10, benzaldehyde 2; (B) lemon oil, vanillin, coriander oil.

Fruits.—(A) Lemon oil 20, orange oil 30, vanillin 2, strawberry essence 10, neroli oil 1, benzaldehyde 10; (B) coriander oil, cinnamon oil, cardamon oil, clove oil.

Spice.—(A) Cinnamon bark oil 20, clove oil 30, bitter almond oil or benzaldehyde 60, lemon oil 60, sweet orange oil 60; (B) fennel oil, nutmeg oil.

Floral Flavours. — *Natural.* — These consist of the pomades, flower oils, concretes, etc., extracted with or dissolved in the requisite amount of specially purified, odourless alcohol to yield perfumes of the strengths of triple extracts, quadruple extracts, etc., small quantities of benzoin, tolu balsam, Peru balsam, styrax, cinnamic alcohol, benzyl benzoate, musk, vanillin, coumarin, etc., being added to act as fixatives. Essential oils which are extracted from flowers, such as neroli, lavender and rose oils, are in many cases used either alone or in admixtures because of their great strengths.

Synthetic.—Acacia. — (A) Anisic aldehyde 9, benzyl acetate 1, phenyl acetic aldehyde 1; (B) geraniol, citronellol, nonylic aldehyde. Or (A) Yara-yara 10, citronellol 2, nerolin 1, cinnamic alcohol 1; (B) geraniol, methyl anthranilate, cinnamic aldehyde, methyl acetophenone.

Cassie.—(A) Cinnamic aldehyde 12, ionone 100 per cent. 1, indol 1, tinct. of musk 8, tinct. of civet 1; (B) benzyl acetate, methyl eugenol, methyl iso-eugenol.

Carnation.—(A) Eugenol 6, terpineol 8, iso-eugenol 6, tinct. of musk 1, tinct. of benzoin 1; (B) coumarin.

Clover.—(A) Iso-butyl salicylate 20, coumarin 10, heliotropin 10, vanillin 10; (B) linalyl acetate. Or (A) Benzaldehyde 2, eugenol 2, terpineol 6, amyl salicylate 2; (B) geraniol, nerolin, ionone, tincts. of orris, civet and storax.

Cyclamen.—(A) Linalol 10, phenyl ethyl alcohol 2, phenyl acetic aldehyde 1, ionone 2; (B) nonylic aldehyde, decylic aldehyde.

Hawthorn.—(A) Anisic aldehyde 20, vanillin 5, geraniol 6, benzyl acetate 5; (B) heliotropin, coumarin, benzaldehyde, nonylic alcohol.

Hyacinth.—(A) Phenyl acetic aldehyde 10, terpineol 4, cinnamic alcohol 1; (B) methyl acetophenone, benzylidene acetone.

Jasmine.—(A) Benzyl acetate 10, indol 1, linalol 1, methyl anthranilate 1; (B) nerolin, ionone, linalyl acetate, geraniol, citronellol.

Lilac.—(A) Terpineol 2, linalol 6, phenyl ethyl alcohol 3, cinnamic alcohol 1; (B) benzylidene acetone, phenyl acetic aldehyde, decylic aldehyde.

Lily of the Valley.—(A) Linalol 5, benzyl acetate 1, phenyl ethyl alcohol 2, terpineol 3; (B) duodecylic aldehyde, vanillin. Or (A) Linalol 10, nerolin 3, terpineol 10; (B) benzyl acetate, ionone.

Moss Rose.—(A) Geraniol 10, citronellol 2, cinnamic alcohol 1, geranyl valerianate 1, citronellyl valerianate 1; (B) octylic aldehyde, santalol.

Parma Violet.—(A) Ionone 40, vanillin 2, synthetic jasmine 20; (B) violet leaf extract.

Rose d'Orient.—(A) Geraniol 5, citronellol 3, benzyl acetate 1, cinnamic alcohol 1; (B) esters of geraniol and citronellol, phenyl ethyl alcohol.

Tuberose.—(A) Linalol 10, benzyl acetate 8, methyl anthranilate 1; (B) duodecylic aldehyde.

Violet.—(A) Ionone 5, benzyl acetate 2, indol 1, tinct. of musk 4; (B) synthetic cassie, santalol.

White Lilac.—(A) Terpineol 40, phenyl acetic aldehyde 4, methyl anthranilate 6; (B) benzyl acetate, tinct. of benzoin, acetophenone.

White Rose.—(A) Citronellol 10, phenyl ethyl alcohol 10, synthetic rose d'orient 20; (B) nonylic alcohol.

Fruit Flavours.—These may be divided into two main groups: (*a*) for flavouring clear beverages (soluble essences), (*b*) for flavouring confectionery.

Soluble essences are made up by dissolving terpeneless or sesquiterpeneless essential oils in alcohol, by shaking ordinary essential oils, such as citrus oils, with alcohol or mixtures of alcohol and water and separating the alcoholic layer from the terpene layer, by preparing natural fruit extracts in such a manner that they give clear solutions on dilution and by compounding such natural fruit extracts with artificial fruit esters.

Confectionery essences may be derived from natural fruit extracts, from natural fruit extracts with additions of artificial fruit esters and from artificial fruit esters alone. The majority consist of a basis of natural fruit extract strengthened and supplemented by artificial fruit esters, essential oils and synthetic chemicals. Such mixtures of esters, essential oils and synthetic bodies as the following are used for this purpose:

Apricot.—(A) Ethyl butyrate 10, butyl butyrate 10, ethyl acetate 5, ethyl valerianate 5, amyl acetate 7, amyl alcohol 1; (B) benzyl acetate, benzyl propionate, eugenol, anethol, synthetic peach oil, petitgrain oil, vanillin.

Banana.—(A) Benzyl acetate 10, benzyl propionate 10, ethyl butyrate 5, amyl butyrate 10, amyl acetate 5, sweet orange oil 3, lemon oil 4; (B) petitgrain oil, vanillin, ethyl sebacate, cinnamon oil, clove oil.

Cherry.—(A) Amyl formate 10, œnanthic ether 6, ethyl acetate 15, ethyl pelargonate 2, benzaldehyde 1, amyl alcohol 1; (B) clove oil, vanillin, neroli oil, cinnamon oil, ethyl cinnamate, ethyl benzoate.

Peach.—(A) Amyl acetate 10, ethyl acetate 5, synthetic peach oil 5, ethyl butyrate 2, benzaldehyde 1; (B) amyl butyrate, vanillin, lemon oil, orange oil, petitgrain oil, cardamom oil.

Pear.—(A) Amyl acetate 20, lemon oil 2, orange oil 5, bergamot oil 5; (B) ethyl acetate, amyl butyrate, clove oil, vanillin, petitgrain oil, geranyl butyrate, geranyl and citronellyl formates.

Pine-apple.—(A) Ethyl butyrate 20, amyl butyrate 40, amyl acetate 5, ethyl acetate 10; (B) ethyl pelargonate, orange oil, lemon oil, vanillin.

Raspberry.—(A) Amyl acetate 20, amyl butyrate 20, ethyl acetate 5, butyl acetate 30; (B) oil of orris, ionone, petitgrain oil, vanillin, clove oil, orange oil, ethyl nitrite, amyl valerianate, benzyl acetate, methyl salicylate, cinnamon oil.

Strawberry.—(A) Benzyl acetate 10, amyl acetate 20, amyl butyrate 10, ethyl butyrate 20, ethyl acetate 5, methyl salicylate 1, ethyl cinnamate 1; (B) ethyl benzoate, nerolin, coumarin, cinnamon oil, vanillin.

Fruit essences are usually dissolved or reduced to a standard strength in mixtures of alcohol and water or alcohol, glycerine and water. The high duty on alcohol, however, has during the past few years caused a great deal of experimenting to be done with a view to providing other vehicles. For example, fruit essences are now being offered on the market suspended in mucilages or emulsions of gum acacia, tragacanth, etc. For special flavouring purposes, vegetable fat, of a perfectly odourless and tasteless nature, has been strongly flavoured with various flavouring bodies and offered to foodstuff manufacturers, for use in such goods as contain fat.

Herbal Beverage Extracts.—These are prepared by infusing the vegetable matters with hot water and concentrating the extracts under reduced pressure. The following are several types of vegetable mixtures:

Dandelion.—(A) Dandelion root 20, ginger 15, liquorice extract 10; (B) gentian, quassia.

Hop Ale.—(A) Hops 10, ginger 10, quillaia bark 5, liquorice extract 3; (B) wormwood.

Horehound.—(A) Horehound herb 10, ginger 2, liquorice extract 5; (B) lemon peel, chillies, balm herb, gentian, coriander seeds.

Sarsaparilla.—(A) Sarsaparilla root 20, sassafras bark 30, liquorice extract 10; (B) ginger.

Liqueur Essences.—These are prepared by maceration of vegetable bodies with alcohol and water, either with or without the addition of essential oils. The following are two examples of the former type:

Benedictine.—(A) Angelica root 30, angelica seeds 20, cloves 6, balm herb 30, cinnamon bark 5, mace 5, arnica flowers 5; (B) thyme, calamus, vanilla beans, tonka beans. With the addition of (A) oils of peppermint 1, lemon 2; (B) cardamom, orange, coriander.

Chartreuse.—(A) Bitter orange peel 20, ginger root 5, cinnamon bark 3, zedoary root 15, calamus root 10, iva herb 10; (B) balm herb, galanga root, angelica, calisaya bark, anise. With the addition of (A) oils of coriander 1, angelica 1, thyme 1; (B) lemon, orange, œnanthic and acetic ethers.

In some cases also mixtures of essential oils are used, without vegetable extracts, as liqueur flavours.

SECTION IV

ODOROUS CHEMICAL PRODUCTS

CHAPTER XI

ALCOHOLS, PHENOLS AND ALDEHYDES

Alcohols and Phenols.—*Anethol* is similar in odour and flavour to aniseed oil, from which it is obtained by chilling. It is used as a flavouring material in many classes of foodstuffs and beverages. Its melting-point is 22·5° to 23°; congealing-point, 21° to 22°; specific gravity, 0·985 at 25°; boiling-point, 235°; and solubility, 1 vol. in 2 to 3 vols. 90 per cent. alcohol. Anethol has been prepared by several methods. For example, Wallach synthesised it from anisic aldehyde in 1907, while Klages in 1902 devised a synthesis starting from anisol.

Benzyl Alcohol has a faint odour resembling jasmine. In fact, it occurs naturally in jasmine and several other flower oils, and also in small quantities, accompanied by benzyl esters, in Peru balsam. Its chief use is as a solvent in the preparation of non-alcoholic perfumes; especially is it useful in dissolving artificial musks. It is also used as a fixative in floral extracts. Several methods may be employed in its preparation, one of the simplest being the boiling of benzyl chloride with water and lead hydroxide. It is also obtained by reducing benzaldehyde and by boiling Peru balsam with aqueous caustic potash. The pure alcohol boils at about 206° and has a specific gravity of 1·0507 at 15°. It is slightly soluble in water, soluble in alcohol and ether.

Cinnamic Alcohol (Styryl alcohol) has an odour resembling hyacinths to some extent. It is used principally as a fixative for other perfumes in place of storax, from which it is obtained. Cinnamyl cinnamate (styracin) yields cinnamic alcohol when distilled with aqueous potash. Cinnamic alcohol has a melting-point of about 33° C. and a boiling-point of 254° to 255° C.

Citronellol is one of the most important odorous bodies, being used extensively in rose perfumes. Together with geraniol, it forms the product which is known as rhodinol. Citronellol may be obtained synthetically from menthol and also by reduction from citronellal. It is, however, usually obtained from Bourbon geranium and Java citronella oils. It can be separated from geraniol by heating the oil with 95 per cent. formic acid, which decomposes the geraniol and reacts with the citronellol to give citronellyl formate. Other methods of separating citronellol from oils in which it is contained, admixed with geraniol, are by heating the oil to 250° in an autoclave along with water and by heating with phthalic anhydride. In both cases the geraniol is decomposed. The commercial preparation of citronellol from geranium oil is a matter of difficulty. The oil is first saponified with alcoholic potash, the geraniol being partly converted into methyl heptanol. Repeated distillation under reduced pressure is necessary to separate the methyl heptanol owing to the similarity in boiling-points. The separation of the geraniol and citronellol is then accomplished by means of formic acid or, preferably, phthalic anhydride, as indicated above. The citronellol is purified by redistillation.

The characters of citronellol are as follows: Specific gravity, 0·858 to 0·862; refractive index, 1·456 to 1·460; optical rotation, $-2°$ to $+4°$; boiling-point, 225° to 226°. Great variations occur in the odour values of commercial citronellols owing to the difficulty of eliminating impurities.

Decylic Alcohol (n-primary) or Alcohol C_{10} has a fatty odour and is used in small quantities for producing distinctive tones in floral extracts. It is formed by the reduction of capric aldehyde with zinc dust and acetic acid. It melts at $-10°$ to $-8°$ and boils at 110° at 13 mm.

Duodecylic Alcohol (n-primary) or Alcohol C_{12} is similar in properties to decylic alcohol, being used along with Narcissus and Tuberose extracts and also with neroli perfumes as a fixative. It is formed by the reduction of lauric aldehyde with zinc dust and acetic acid. It melts at 13° to 15° and boils at 142° at 13 mm.

Eugenol is the principal odorous constituent of clove oil. It is used as the starting-point in the preparation of vanillin, and also finds employment in many essences and extracts, *e.g.* carnation. Its extraction from oil of cloves is accomplished by treating the oil with potash. The potassium salt of eugenol separates out, is pressed free from terpenes, washed with alcohol and decomposed, with the formation of eugenol, by adding mineral acid. An alternative method utilises the combination of eugenol with sodium acetate to form a separable compound.

The pure product is a colourless liquid boiling at 248°, having a specific gravity of 1·072 and a refractive index of 1·544. It reddens litmus and reduces ammoniacal silver solutions on standing, but not Fehling's solution.

When tested with ferric chloride in alcoholic solution it gives a blue colour. With sulphuric acid it gives at first a brown colour, then purple and finally claret. The *Analyst*, 1900, 25, 265, describes a reagent specially devised for testing for eugenol. It is made up by dissolving 10 gms. of a mercuric salt in 25 per cent. sulphuric acid and diluting to 100 c.c. When 2 c.c. of eugenol are shaken with 5 c.c. of the reagent and allowed to stand, a pale violet colour develops.

Iso-eugenol, which occurs in small quantities in nutmeg and ylang-ylang oils, has a finer odour than eugenol. It is used in compound floral perfumes of the wallflower and carnation types. It is produced by an isomerisation process from eugenol, potash and heat being applied and amyl, methyl, ethyl or butyl alcohols being used as solvent, although processes have been invented of accomplishing the isomerisation by using water alone or by dry-heating out of contact with air. Iso-eugenol is a colourless liquid of boiling-point 262°; specific gravity, 1·090; refractive index, 1·5685 to 1·5720. Ferric chloride in alcoholic solution gives with iso-eugenol an olive-green colour and sulphuric acid a pink colour changing to brown.

Methyl eugenol and methyl iso-eugenol are constituents of cassie and ylang-ylang perfumes. They are used in extracts and essences of the carnation type. Eugenol-methyl ether is prepared from eugenol by the action of methyl iodide and caustic potash. Iso-eugenol-methyl ether is prepared from eugenol-methyl ether by warming it with alcoholic potash, or from iso-eugenol by the action of aqueous potash and dimethyl sulphate. The boiling-points of these two compounds are 247° and 263° respectively.

Geraniol is one of the most commonly used odorous bodies; it forms the basis, along with citronellol, of many rose perfumes. It is obtained commercially from citronella and palma-rosa oils. The fact that geraniol forms a crystalline compound with calcium chloride, which is insoluble in organic solvents but is decomposed by water into geraniol and calcium chloride, is utilised in the preparation of pure geraniol. The following are the characters of geraniol: Specific gravity, 0·883 to 0·886; refractive index, 1·476 to 1·478; boiling-point, 229° to 231°.

Iso-styrone is a body possessed of a distinct rosy odour. It is used to some extent as a fixative and basis for perfumes of a rose and jasmine description.

Linalol is the chief odoriferous principle of several essential oils, *e.g.* linaloe and bois de rose femelle oils. It also occurs in a vast range of natural perfumes, *e.g.* orange flowers, bergamot, ylang-ylang, jasmine, etc. Its odour is about midway between lemon and rose oils, and it is used in a great variety of perfumes of the same nature as those mentioned above. It blends very well with synthetics of the nerolin type. The linalol of commerce is usually obtained by the fractional distillation of linaloe oil. It may also be obtained by acting upon geraniol with hydrochloric acid and saponifying the product. Its specific gravity is 0·870 to 0·874; optical rotation, $-20°$ to $+20°$; refractive index, 1·464 to 1·467; boiling-point, 198° to 199°.

Menthol is a crystalline body obtained by chilling from Japanese peppermint oil. It finds a varied application in the flavouring of many foodstuffs, medicinal preparations and toilet requisites. It has a melting-point of 42°, a boiling-point of 212° and an optical rotation of $-43°$ to $-50°$.

Nerol occurs, chiefly in the form of esters, in neroli, rose, petitgrain, etc., oils. It is a stereo-isomer of geraniol, and is formed along with terpineol and geraniol when linalol is treated with acetic anhydride. Unlike geraniol, it does not form a crystalline product with calcium chloride. It has a specific gravity of about 0·881, boils at 226° to 227° and is optically inactive.

Nonylic Alcohol or Alcohol C_9 has a rose-like, fatty odour, blending well with essence of sweet orange and perfumes of a rose, jasmine and neroli type. It is also used in the production of bouquet perfumes and essences of the millefleurs, eau de Cologne, etc., description. Its caprylic ester occurs in sweet orange oil. It has a specific gravity of about 0·840, a boiling-point of 211° to 213° or 102° at 13 mm. and a melting-point of $-11°$.

Octylic Alcohol or Alcohol C_8 has a fatty odour resembling lemon to some extent. Several octylic esters occur in essential oils, *e.g.* male fern. The alcohol is used in perfumes of the eau de Cologne type. It is obtained from its acetyl derivative. It has a specific gravity of 0·828 to 0·830 and boils at 196° to 197° or 95° at 13 mm. On oxidation it yields caprylic acid, which melts at 16·5°.

Phenyl-Ethyl Alcohol has a honey-rose odour and occurs in otto of rose. It is a constituent of Grasse rose essence and, being very soluble, is particularly to be found in rose waters. During the last few years it has come to be very largely used in synthetic rose perfumes and also for harmonising many floral extracts. The α alcohol is obtained by reducing acetophenone with sodium amalgam. It boils at 203°. The β-alcohol, boiling at 212°, is formed by reducing phenyl-acetic aldehyde with sodium amalgam. The commercial alcohol has a specific gravity of 1·020 to

1·025, a refractive index of 1·530 to 1·535, is optically inactive and boils at about 220°.

Phenyl-Propyl Alcohol is contained in storax, from which it is obtained in the manufacture of cinnamic alcohol. It is also prepared synthetically. Its hyacinth odour is less pronounced than is the case with cinnamic alcohol, resembling more nearly phenyl-ethyl alcohol. It is used for similar purposes to phenyl-ethyl and cinnamic alcohols and also in extracts of a reseda type. Its preparation is accomplished by reducing cinnamyl alcohol with sodium amalgam.

Safrol is contained in sassafras and camphor oils. It is prepared from the latter by chilling out the camphor and repeatedly fractionating the remaining oil. In the pure condition it is a liquid with a congealing-point of 11°, a specific gravity of 1·106, a refractive index of 1·5385 and a boiling-point of 233°.

Iso-safrol is prepared from safrol by heating the latter with sodium ethylate to 200°. It has a specific gravity of 1·127, a refractive index of 1·580 and a boiling-point of 253°.

Santalol is obtained by the fractional distillation of sandalwood oil. It is used in the production of essences and in medicine. Commercial santalol is a mixture of two santalols, α-santalol and β-santalol. α-Santalol has a specific gravity of 0·980 to 0·984, an optical rotation of 0 to $-1°$, a refractive index of 1·505 to 1·509 and a boiling-point of 300°. The characters of β-santalol are as follows: Specific gravity, 0·985 to 0·986, optical rotation, $-51°$ to $-56°$; refractive index, 1·505 to 1·509; boiling-point, 309° to 310°.

Terpineol is very largely used as the basis of cheap lilac, lily of the valley and similar perfumes. It is necessary that the terpineol should be of a high degree of purity when used for perfumery purposes. Rose, geranium and ylang-ylang oils blend well with terpineol, as also do benzyl acetate, geraniol, citronellol, heliotropin, etc. Terpineol is obtained by the action of alcoholic sulphuric acid upon pinene, or by distilling terpin with very dilute hydrochloric acid. The commercial article is a thick liquid with a specific gravity of 0·940, a boiling-point of 216° to 218° and a refractive index of about 1·480.

Thymol is extracted from thyme and ajowan oils. It is a powerful antiseptic, is soluble in 1100 parts of cold water, in 900 parts of boiling water and readily in alcohol, ether, chloroform, acetic acid, etc. It melts at about 51° and boils at about 233°.

Undecylic Alcohol or Alcohol C_{11} has a powerful and retentive, fatty odour. It is used as a fixative because of its stable nature. It melts at $-12°$ to $-11°$ and has a boiling-point of 128° at 13 mm.

Aldehydes and Ketones.—*Acetophenone* is a white crystalline body melting at 21° to 22°. It has a powerful odour and is sometimes used in perfumes of the lilac, mimosa, syringa, new-mown hay, etc., types. It is, however, rather acrid and is frequently substituted by methyl-acetophenone, which is of a much more pleasant nature. It blends well with terpineol, benzyl acetate, etc. Acetophenone may be obtained by distilling calcium benzoate with calcium acetate. It is, however, usually prepared by the interaction of benzene, acetyl chloride and aluminium chloride.

Anisic Aldehyde or Aubépine occurs in small quantities in fennel and aniseed oils. It has the odour of hawthorn blossom, is soluble in all proportions of alcohol and fatty oils and forms an excellent basis for perfumes of the hawthorn and new-mown hay types, blending well with orange, petitgrain, ylang-ylang and neroli oils and also with heliotropin, coumarin, etc. When exposed to the air it oxidises rapidly to anisic acid, which has no odour value, also it is affected by light and heat and consequently it is necessary to keep it in full, coloured bottles in a cool place. It is prepared by the methylation of p-oxybenzaldehyde or by the oxidation of anethol. It boils at about 246° and has a specific gravity of 1·125 to 1·126.

Benzaldehyde is the chief odorous constituent of essential oils of bitter almonds, peach kernel and apricot kernel. For flavouring purposes it is necessary that the product should be free from chlorine, and, as most artificially produced benzaldehyde on the market contains traces of chlorine, this impurity should be tested for very rigorously. Benzaldehyde is used as a substitute for bitter almond oil in maraschino and other flavours. It is a colourless liquid which rapidly oxidises to benzoic acid on exposure to the atmosphere. Its specific gravity is 1·051 to 1·055, it is optically inactive, its refractive index is about 1·545 and its boiling-point, 177° to 179°.

Benzaldehyde may be prepared by many methods. The Lauth and Grimaux process is as follows: Benzyl chloride is boiled with nitrate of lead or copper under a reflux condenser for eight hours. The product is subsequently purified by combining it with bisulphite of soda, decomposing with sodium carbonate and steam distilling. Several syntheses commence with benzal chloride, *e.g.* Hermann-Schmidt oxidises this compound with manganese dioxide and water, Caro heats it with milk of lime under pressure and Bigot employs lead oxide and water. When either benzyl or benzal chloride is used as raw material, the finished benzaldehyde contains traces of chlorine which are difficult to remove. Such, however, is not the case if the product is manufactured by the direct oxidation of toluene according to the following patents: *Ger. Pat.*, 101221, 1897, using finely divided manganese dioxide in presence of sulphuric acid, *D.R.P.*, 127388, 1900, using nickel oxide at 100° and *Ger. Pat.*, 175295, 1903, using manganese sulphate. An interesting process for the catalytic production of aldehydes is also described in *Berichte*, 1918, 51, 585. In this process palladinised barium sulphate, containing 5 per cent. of metal, is used, and excellent results are said to be obtained.

Chlorine may be tested for in benzaldehyde by heating the latter with sodium and testing for chlorine as in ordinary organic analysis, or by heating the benzaldehyde with chlorine-free slaked lime to redness. The mixture is then cooled, a small quantity of water added and then pure nitric acid used as solvent, the acid solution being tested for chlorine with silver nitrate. For a comparison of the various methods of estimating chlorine in benzaldehyde, see *P. & E.O.R.*, 1917, 41.

Benzaldehyde is best assayed by means of saturated sodium bisulphite solution. The U.S.P. method makes use of the phenyl hydrazone, but both this and the neutral sodium sulphite method give unsatisfactory end reactions.

Benzylidene-acetone (Cumaranol or Cinnamyl-ketone) has a very powerful and retentive coumarin-like odour. It is used in perfumes of the sweet-pea, new-mown hay, lavender, syringa, etc., types. It is formed when benzaldehyde and acetone are condensed according to Claisen's reaction, using caustic soda as catalyst. Benzylidene-acetone is soluble in alcohol and ether and forms a crystalline compound with bisulphite of soda. It gives an orange-coloured solution with concentrated sulphuric acid.

Carvone is contained in caraway, dill and spearmint oils, from which it is extracted. It has a usage in the flavouring of certain liqueurs. Its characters are as follows: Specific gravity, 0·964; optical rotation, −60° to 60°; refractive index, 1·498 to 1·500; boiling-point, 225° to 230°.

Cinnamic Aldehyde, which occurs chiefly in cinnamon and cassia oils, is made synthetically by what is termed the croton condensation between benzaldehyde and acetaldehyde in the presence of caustic soda and other alkalies, alcohol being occasionally added to the mixture to assist the reaction. Cinnamic aldehyde is a colourless to light yellow liquid, having a pure cinnamon odour and the following characters: Specific gravity, 1·055 to 1·058; refractive index, about 1·6195; boiling-point, 248° to 252° (with decomposition), or 129° to 130° at 20 mm. pressure.

Citral is the chief odorous principle in lemon and lemon-grass oils. The product on the market is prepared from both sources; the quality of that prepared from lemon-grass oil is very much inferior in delicacy of odour to that prepared from lemon oil, and the price is proportionately less. Citral has a very large range of usage for all manner of flavouring purposes where a suggestion of lemon is required. It is especially used in the preparation of soluble essences for use in the mineral water trade. So-called "synthetic lemon oils" are produced using lemon terpenes and lemon-grass citral; these, however, are deficient in delicacy of aroma and can usually be detected by their odour. Citral is interesting in that it is the starting-point for the synthesis of violet perfumes of the ionone type. It has the following characters:

Specific gravity, 0·895; optically inactive; refractive index, about 1·488; boiling-point, 228° to 229°. An interesting series of optical determinations on citral and ionones is detailed in *J. pr. Chem.*, 1921, ii. 102, 305–331.

Citronellal occurs in citronella oil. The pure product has about three times the odour value of the latter oil. It has an intense melissa odour and is used in lemon, geranium, eau de Cologne, etc. essences. The following are its characters: Specific gravity, about 0·855; optically inactive; refractive index, 1·444 to 1·448; boiling-point, 207° to 208°.

Hydroxycitronellal is a fresh-smelling product which is specially suitable for use in perfumes, such as lilac, lily of the valley, cyclamen, etc. To be of any value it should be pure and free from terpenes. 5 c.c. of the compound should be soluble when shaken with 100 c.c. of 40 per cent. sodium bisulphite solution.

Decylic Aldehyde or Aldehyde C_{10} occurs in exceedingly small quantities in oils of sweet orange, orris, neroli, lemon-grass, etc. It has a soft, fatty odour and a medium volatility. Its boiling-point is 75° to 76° at 2 mm., and in a pure state it is a crystalline body melting at 23°. It is prepared, as are most of the fatty aldehydes of this series, by distilling under reduced pressure the calcium or barium salt of the corresponding fatty acid with barium formate.

Duodecylic Aldehyde or Aldehyde C_{12} is a very interesting aldehyde, having in a diluted state a very fresh odour. It blends well with essence of orange and violet perfumes. Its boiling-point is 101° to 102° at 2 mm., and its melting-point 24°. It usually appears on the market dissolved in a solvent, *e.g.* benzyl alcohol.

Heliotropin is a white crystalline body possessing a pleasant heliotrope odour. It melts at about 36·5° and is soluble in 5 parts 95 per cent. alcohol. It is manufactured from safrol. This latter body is converted into iso-safrol by treatment with caustic soda at 105°, and the iso-safrol is then oxidised with potassium dichromate and sulphuric acid, heliotropin being formed.

Heptylic Aldehyde (Œnanthol) or Aldehyde C_7 is a body possessing an intense, even offensive odour. It must be used with great discretion and in small quantities. Its chief property is that it gives a note of freshness to other odours.

Ionone, Methyl-Ionone and Artificial Violet Perfumes find a very extensive use in the preparation of floral ottos, extracts, etc. These latter contain both ionone itself and the derivatives of α-ionone and β-ionone, which are combined in ionone. The delicacy of the finished artificial violet perfume depends to a large extent on the purity and derivation of the raw materials. Citral, derived from lemon-grass oil, is the chief of these, and is obtained from the oil by careful fractionation. Pseudo-ionone, or homologous bodies, is first prepared by shaking with acetone, or homologues of acetone, in presence of baryta or sodium ethylate for two or three days. After the separation and purification of the pseudo-ionone, it is converted into ionone by an isomerisation process, using sulphuric acid, hydrochloric or phosphoric acid, according to whether α- or β-ionone is required in predominance. As regards odorous properties, β-ionone is rather more acrid than α-ionone, approximating more nearly to natural violet perfumes than the latter, which has an orris odour.

Methyl Acetophenone is possessed of a finer odour than acetophenone; it resembles coumarin, but is much stronger.

Its odour blends well with heliotropin, terpineol, geranium oil, cassie extract, etc., and forms the basis of mimosa perfumes.

Methyl-Nonyl Acetaldehyde or Aldehyde C_{12} has an odour of the lemon or mandarin type: it is used in eau de Cologne and other bouquet perfumes.

Nonylic Aldehyde or Aldehyde C_9 is a fine, fatty odorous body which occurs in small quantities in oils of rose, orris, mandarin, and probably in limes. It boils at 69° to 70° at 2 mm., and melts at 5° to 7°.

Octylic Aldehyde or Aldehyde C_8 occurs in oils of lemon, orange and jasmine. It should only be used in extracts in very small quantities. Its boiling-point is 56° at 5 mm., and its melting-point $-13°$ to $-10°$.

Phenyl Acetaldehyde (toluylic aldehyde), or Jacinthea, is a liquid possessing an intense odour resembling hyacinth. Its chief drawback is that it has a tendency to polymerise quickly, so that it is necessary to keep it in full bottles in a cool, dark place, or to dilute the pure liquid with an equal volume of alcohol. In any case it is advisable to specify that any of the product supplied is freshly manufactured. It may be produced by an isomerisation process from acetophenone, or from phenyl acetic acid by heating with calcium hydroxide. It is, however, produced in bulk from cinnamic or lactic acid. Jehl has devised a process for its preparation by treating methyl cinnamate in methyl alcohol solution with bromine followed by caustic soda. The mixture is subsequently neutralised with sulphuric acid, sodium carbonate added and steam distilled, the phenyl acetaldehyde in the distillate being extracted with ether. It boils at 209° to 210°.

Vanillin is probably the most important and widely used of all the synthetic flavouring materials. It is extensively used in place of vanilla beans. The chief method of production is by the oxidation of iso-eugenol. It is also formed, however, by the oxidation of coniferyl alcohol or coniferin, and by heating guaiacol with chloroform and caustic soda solution. It is said that a 95 per cent. yield and an exceedingly pure product is obtained by oxidising eugenol, iso-eugenol, coniferin or coniferyl alcohol with air and ultra violet rays simultaneously at a temperature of 50° to 60° (*Journ. Soc. Chem. Ind.*, 1910, 1036).

Vanillin occurs in white monoclinic needles. It has a melting-point of 81° and a boiling-point of 170° at 15 mm. In hydrochloric acid solution it gives well-marked colour reactions with ketones, phenols and other substances contained in essential oils; such a solution is in fact used in the identification of essential oils. Vanillin is soluble in 100 parts of water at 14°, and readily soluble in alcohol and ether. With ferric chloride it gives a blue coloration.

The adulterants which have been found in vanillin are acetanilide, coumarin, heliotropin, acetyl iso-eugenol, acetyl-salicylic acid, phthalic acid, sugar, guaiacol carbonate and benzoic acid. Acetanilide may be detected by boiling with potash, aniline being formed if acetanilide is present. Vanillin is estimated by conversion into its bisulphite compound, decomposing the same with acid and extracting the vanillin with ether. The *Chem. Zeit.*, 1914, 38, 402, describes in detail the detection of adulterants in vanillin, and this description is abstracted in the *P. & E.O.R.*, 1914, 150.

CHAPTER XII

ESTERS AND UNCLASSIFIED ODOROUS BODIES

Esters.—*Amyl Acetate* has a powerful odour resembling "pear" and is known as *pear essence.* Its preparation is accomplished by the reaction of amyl alcohol and sodium acetate in presence of sulphuric acid. A method for the commercial preparation of amyl acetate from the monochlor derivatives of saturated hydrocarbons (pentyl and hexyl chlorides) is described in *Journ. Am. Chem. Soc.*, 1916, 38, 1368. Amyl acetate has a specific gravity of 0·860 to 0·863, a refractive index of about 1·400 and a boiling-point of 138° to 139°.

Benzyl Acetate is an important constituent of oils of jasmine, ylang-ylang, neroli and essence of cassie. The acetate prepared from benzyl alcohol and acetic acid is usually less acrid than that prepared from other sources. The product is offered on the market free from chlorine. Synthetic jasmine oils and essences are based on benzyl acetate, which is one of the most widely used synthetic perfumes. It has a specific gravity of 1·061 to 1·064, a refractive index of 1·500 to 1·504 and a boiling-point of 205° to 206°.

Bornyl Acetate is contained in pine-needle oils, of which it has the characteristic odour. It is prepared by heating together borneol and acetic acid in presence of sulphuric acid. Its specific gravity is 0·990 to 0·993 : optical rotation,

−39° to −37°; refractive index, 1·4655 to 1·4665; and boiling-point, 98° at 10 mm. For recent work on the preparation of bornyl esters, see *Journ. Chem. Soc.*, 1921, i. 115.

Citronellyl Acetate has an odour of a fruity type resembling lavender to some extent. It is used for shading the perfumes of rose, lavender, bergamot and also in some bouquet extracts. It boils at 119° to 120° at 15 mm.; its specific gravity is 0·892 to 0·901; and its refractive index, 1·4455 to 1·4490.

Ethyl Acetate finds a widespread application in the preparation of artificial fruit essences. It has a characteristic, pleasant, ethereal odour. Its preparation is accomplished by distilling alcohol and acetic acid in presence of sulphuric acid, or by heating together fused sodium acetate, alcohol and sulphuric acid. It boils at 77·5°, has a specific gravity of 0·903 to 0·906 and a refractive index of 1·370 to 1·375.

Ethyl-Phenyl Acetate is a body having a honey-like odour. It is used in perfumes, such as honey, and in essences requiring a suggestion of wax.

Geranyl Acetate has an odour of a rose-lavender type. It is used in rose, geranium, lavender, ylang-ylang and rosemary perfumes. Its specific gravity is 0·914 to 0·918; refractive index, 1·462 to 1·464; and boiling-point, 242° to 244°.

Linalyl Acetate is a very widely used odorous body. It is contained in very large proportions in bergamot, linaloe, bois-de-rose femelle, etc., oils. Its chief usage is in blended perfumes of the lavender, bergamot, etc. types. Artificial bergamot oil contains linalyl acetate together with lemon, orange or bergamot terpenes, and sometimes terpinyl acetate. Linalyl acetate is usually made from oils of

Mexican linaloe and bois-de-rose femelle, the latter yielding the finer product. It may be produced in a pure state by first preparing linalol-sodium and acting upon the same with acetic anhydride. Linalyl acetate boils at 105° to 106° at 11 mm. or at 220° with decomposition; it has a specific gravity of 0·905 to 0·912 and a refractive index of 1·452 to 1·453.

Phenyl-Ethyl Acetate is a compound which has a marked fruity odour resembling peach to some extent; it is used in conjunction with phenyl-ethyl alcohol in floral perfumes. It has a specific gravity of 1·038 and a boiling-point of 232° to 233°.

Phenyl-Propyl Acetate is possessed of a flowery odour resembling, to some extent, linalyl acetate.

Terpinyl Acetate is used for the same purposes as geranyl acetate in perfume blending. It occurs in several essential oils, such as bergamot and lavender, and is produced commercially from both turpentine and terpineol, the latter yielding the finer product. It is particularly used in cheap eau de Cologne and bouquet perfumes, and also in essences for the perfuming of tobacco. It may be produced in a pure state by boiling terpineol with acetic anhydride and sodium acetate under a reflux condenser. Its specific gravity is 0·955 to 0·960; refractive index, 1·464 to 1·465; and boiling-point, about 220°.

Methyl Anthranilate occurs in neroli, tuberose, ylang-ylang, gardenia and jasmine perfumes. It is used chiefly in the blending of perfumes of this type, its chief disadvantage being that it colours the extracts in which it is used. Methyl anthranilate may be obtained by fusing indigo with caustic potash, the potassium salt of anthranilic acid being formed. The anthranilic acid is set free and esterified by passing dry hydrochloric acid gas through its solution in methyl alcohol. It may also be obtained from ortho-nitro-toluene by oxidation to o-nitrobenzoic acid, this being then reduced to anthranilic acid. Methyl anthranilate has a specific gravity of 1·168 and a melting-point of 24°.

Amyl Benzoate has an amber-like odour and is used as a fixative in the blending of perfumes. It is prepared by passing dry hydrochloric acid gas through a mixture of amyl alcohol and benzoic acid. It has a specific gravity of 1·002 to 1·003 and a boiling-point of 258° to 260°.

Benzyl Benzoate is a practically odourless body melting at 20° to 21°. It occurs naturally in Peru balsam and is used as a substitute for the same in the fixing of extracts. It is a good solvent for artificial musk. Its specific gravity is 1·120 to 1·123; refractive index, 1·570; and boiling-point, 324°.

Ethyl Benzoate is similar in odour to methyl benzoate (see later), being used in essences such as new-mown hay, meadowsweet, etc. It is prepared by passing dry hydrochloric acid gas through a solution of benzoic acid in alcohol. Its characters are as follows: Specific gravity, about 1·050; refractive index, 1·504 to 1·505; boiling-point, 210° to 213°.

Methyl Benzoate (Artificial Niobe Oil) is used in essences such as new-mown hay and meadowsweet. It is obtained by distilling methyl alcohol and benzoic acid in presence of sulphuric acid. Its characters are as follows: Specific gravity, 1·094; refractive index, 1·518; and boiling-point, 200°.

Amyl Butyrate has an ethereal odour resembling pine-apple to some extent. It is used in fruit essences of the pine-apple, apricot type. Its preparation is accomplished in a similar manner to that of ethyl butyrate (see later), using, however, in this instance fusel oil in place of alcohol. It has a specific gravity of 0·866 to 0·867, a refractive index of 1·413 and a boiling-point of 170° to 180°.

Benzyl Butyrate has an odour resembling jasmine, like other benzyl esters. It is occasionally used in fruit essences, *e.g.* apricot, banana.

Ethyl Butyrate of commerce is a liquid possessing a pungent odour resembling pine-apple. It is used largely in the manufacture of fruit essences. Its preparation is achieved by distilling butyric acid and alcohol in presence of sulphuric acid. Its boiling-point is 110° to 121°; specific gravity, 0·883 to 0·884; and refractive index, 1·392.

Geranyl Butyrate has a fine, rosy odour. It is used in giving distinctive tones to rose, geranium, etc., perfumes. It boils at 142° at 13 mm.

Phenyl-Ethyl Butyrate has a distinctive odour resembling tea-rose. It is used in the blending of floral extracts.

Phenyl-Propyl Butyrate resembles benzyl butyrate in odour, but is not so powerful.

Benzyl Cinnamate is used for similar purposes to ethyl cinnamate. It is a solid body with a characteristic odour resembling Peru balsam. Its boiling-point is 337° to 340° and its melting-point 32°.

Ethyl Cinnamate has a fine odour resembling apricot. It is used in the preparation of perfumed waters and eau de Cologne, in the latter case acting as fixative. It has a specific gravity of about 1·054, a refractive index of 1·557 and a boiling-point of 260° to 265°.

Methyl Cinnamate is a body of balsamic odour which is used for similar purposes to ethyl cinnamate. Its specific gravity at 36° is 1·041, its melting-point is 36° and its boiling-point, 263°.

Benzyl Formate is an aromatic liquid of a sweet, cinnamon-like odour. It is used in heliotrope, jasmine, etc. perfumes and in certain fruit flavours.

Citronellyl Formate has a rose-bergamot odour, which makes it invaluable in the shading of perfumes of the lavender, rose, eau de Cologne types. It has a specific gravity of 0·905 to 0·913, a refractive index of 1·450 to 1·453 and a boiling-point of 98° to 100° at 10 mm.

Ethyl Formate is used in artificial rum essences. It is prepared by distilling a mixture of alcohol, glycerine and oxalic acid. It is a very volatile liquid, boiling at about 54·5°.

Geranyl Formate has an odour resembling wild-rose. It is used along with other geranyl esters and geraniol in producing distinctive types of rose perfumes. It has a specific gravity of about 0·925, a refractive index of 1·451 to 1·460 and a boiling-point of 113° to 114° at 15 mm.

Phenyl-Ethyl Formate is possessed of a very sweet, pungent odour of the chrysanthemum type.

Phenyl-Propyl Formate has a distinctive odour recalling honey and cinnamic alcohol.

Amyl Heptylate is possessed of a strong, fruity odour and is used in extremely small quantities to create perfumes of a distinctive type.

Benzyl Propionate has a fruity odour and is used to some extent in fruit essences of the banana, apricot, etc. types. It is also employed in jasmine perfumes to add a distinctive tone to the benzyl acetate.

Citronellyl Propionate has a fresh rosy odour and is used to some extent in rose perfumes.

Geranyl Propionate has an odour resembling bergamot. It is used in perfumes such as lavender, bergamot, rose, etc.

Phenyl-Ethyl Propionate is possessed of a fruity odour of a rose type and is used for giving distinction to floral extracts.

Amyl Salicylate is a liquid with a distinct clover-like odour. It is used extensively as the basis of perfumes, such as Californian poppy, clover, trèfle, orchidée, etc., and blends well with musk ambrette. Its specific gravity is 1·050 to 1·053; refractive index, 1·505 to 1·507; optical rotation, about + 1° 30′; and its boiling-point, 270° to 277°.

Ethyl Salicylate has an odour resembling methyl salicylate, or oil of wintergreen (see later). It is, however, more delicate, sweeter, and altogether finer. It has a specific gravity of 1·135, a refractive index of 1·523 and a boiling-point of about 230°.

Methyl Salicylate (Artificial Oil of Wintergreen) is used in meadowsweet and new-mown hay perfumes. It is the chief constituent of oil of wintergreen. Its characters are as follows: Specific gravity, 1·185; refractive index, 1·537; and boiling-point, 224°. The *Journ. Am. Chem. Soc.*, 1917, 39, 820, gives an optical method for detecting impurities in methyl salicylate, using the benzoyl derivatives of the ester. See also *P. & E.O.R.*, 1914, 60, for a colour test to distinguish synthetic and natural oils of wintergreen.

Ethyl Sebacate is used fairly extensively in fruit essences. It has a specific gravity of 0·962 to 0·963 and a refractive index of about 1·437.

Ethyl Succinate has the following characters: Specific gravity, about 1·050; refractive index, 1·42 to 1·43; boiling-point, 210° to 215°.

Amyl Valerianate is possessed of a pronounced apple-like odour: it is in fact known as apple oil and is used in fruit essences of an apple type. It is produced by oxidising amyl alcohol, or by distilling sodium valerianate with amyl alcohol in presence of sulphuric acid. It has a specific gravity of about 0·857 to 0·860, a refractive index of 1·410 to 1·417 and a boiling-point of about 188°.

Benzyl Valerianate has a flowery odour somewhat resembling phenyl-ethyl acetate.

Bornyl Iso-Valerianate is a colourless liquid possessing a powerful, camphor-like odour. It is used to a small extent in perfumery. Its preparation is achieved by esterifying borneol with iso-valerianic acid. Its physical properties are as follows: Specific gravity, 0·954 to 0·956; refractive index, about 1·462; optical rotation, about −35°; and boiling-point, 255° to 260°.

Ethyl Valerianate has the characteristic odour of pineapple and is used in fruit essences of many types. It is prepared by distilling sodium valerianate with alcohol in presence of sulphuric acid. Its specific gravity is about 0·895 and its boiling-point 133° to 134°.

Geranyl Iso-Valerianate has an odour of a rose type and is used for giving distinction to rose perfumes. It is prepared by esterifying geraniol with iso-valerianic acid. It boils at 135° to 138° at 13 mm.

Phenyl-Ethyl Valerianate is a liquid with a pronounced rose-leaf odour.

Styrolyl Valerianate has a strong odour of jasmine, and is used in very small quantities in jasmine and narcissus perfumes.

Other Odorous Bodies.—*Coumarin* is a white crystalline body with an odour resembling new-mown hay. It is the chief odorous constituent of tonka beans and has an extensive usage, both alone and in combination with other odorous bodies, as a flavouring agent. The starting-point in the preparation of coumarin is phenol, this being condensed with chloroform by heating to a temperature of 60° to 65° in the presence of caustic soda to form salicylic aldehyde. This latter substance is purified and heated with acetic anhydride and sodium acetate, coumarin and aceto-coumaric acid being formed. The mixture is heated with water and the aceto-coumaric acid is decomposed, giving coumarin and acetic acid. Coumarin is recovered from the reaction mixture by steam distillation. Other methods of preparing coumarin are by heating phenol with malic acid and zinc chloride or sulphuric acid and by treating o-oxy-phenyl propionic anhydride with bromine at 170°. Coumarin has a melting-point of 67° and a boiling-point of 290°. The *P. & E.O.R.*, 1918, 81, cites a case in which terpin hydrate was discovered as an adulterant of coumarin to the extent of 30 per cent.

Brom-styrol (Hyacinthine) is a body having an intense hyacinth-like odour. It has the advantage of being more stable than phenyl acetaldehyde. It blends very well with, and has a distinct freshening effect upon, lilac, heliotrope, new-mown hay and rose perfumes. Alpha-brom-styrol is prepared by heating dibrom-hydrocinnamic acid with water. The auto-oxidation of brom-styrol to brom-acetophenone is explained in *Compt. Rend.*, 1921, 172, 162–165.

Diphenyl Ether is a crystalline body which has an odour resembling geranium. It is formed by extracting a molecule of water from two molecules of phenol. Sabatier and Mailhe achieved this by dropping melted phenol into thorium oxide at 400°. This method, which was published in 1913, is very interesting in that many similar compounds having geranium odours can be produced in the same manner, *e.g.* phenyl-cresyl ether, etc. (see also nerolin).

Another method of producing diphenyl ether is to heat sodium phenate with monobrom-benzene in presence of powdered copper at a temperature of 200°, the diphenyl ether being recovered by distilling in steam. Diphenyl ether melts at 28° and boils at 253°.

Diphenyl Methane has an odour of green geranium leaves. It is used in the production of cheap and lasting geranium perfumes. It may be made by heating together benzene and paraformaldehyde in presence of sulphuric acid, or by the action of benzene on methylene chloride in presence of anhydrous aluminium chloride. Its commercial preparation is usually accomplished by the ordinary Friedel Crafts reaction of benzene and benzyl chloride in presence of anhydrous aluminium chloride. Diphenyl methane melts at 26·5° and boils at 261°.

Indol is contained to some extent in neroli, jasmine, tuberose and other perfumes. It is very powerful in odour and requires to be used with great care. It has the drawback of colouring essences and extracts of which it forms a component. The auto-oxidation of indol and skatol has recently been the subject of a research, for which see *Journ. Chem. Soc.*, 1921, i. 127. It is synthesised from anthranilic acid, also it is formed when o-nitrocinnamic acid is distilled with caustic potash and iron filings, or when oxindol is distilled with zinc dust. The usual method of preparation is by heating together aniline and dichlorether. Indol is a colourless crystalline substance, melting at 52° and boiling at 253° with partial decomposition. It is soluble in boiling water and distils easily in steam.

Nerolin, Yara-yara and Bromelia.—These bodies consist of the ethyl and methyl ethers of naphthol. The compounds, which are usually sold under the names nerolin and bromelia, are the ethyl ether, and have an agreeable odour somewhat resembling neroli. Yara-yara is the methyl ether and has a powerful odour resembling acacia. Nerolin and yara-yara are used in cheap eau de Cologne perfumes and sachet powders. They can be produced by several methods, *e.g.* β-naphthol, hydrochloric acid and ethyl or methyl alcohol are heated together in an autoclave for eight hours, or the sodium salt of β-naphthol is heated on a water bath with ethyl or methyl bromide or iodide, in ethyl or methyl-alcohol solution. A method devised by Sabatier and Mailhe makes use of thorium oxide as a catalytic agent. β-naphthol in ethyl or methyl-alcohol solution is passed over the thorium oxide at a temperature of 240° to 260°. Nerolin melts at a temperature of 37°, while yara-yara melts at 70° and boils at 294° to 295°.

Para-Cresol-Methyl-Ether is a constituent of natural ylang-ylang and canaga oils. It has a powerful odour and forms the basis of synthetic perfumes of this type. It is produced by methylating the sodium salt of para-cresol with dimethyl sulphate. Its specific gravity is 0·975 and its boiling-point 176·5°.

Skatol is the β-methyl derivative of indol. It is present in narcissus perfumes and also in natural civet and musk. Its odour is most repulsive in a pure state and, like indol, it has to be used in very small quantities. It occurs in the products of decomposition of substances containing albumen or albuminous materials. It may be prepared by fusing egg albumen with caustic potash or by the natural decomposition of pancreas.

Synthetic Musks.—Musk Xylol has a very extensive use in perfumery and flavouring materials. Its chief drawback is its comparative insolubility in alcohol. It dissolves in the proportion of 6 gms. per litre of 95 per cent. alcohol.

Its solubility in benzyl benzoate is about 280 gms. per kilo. and in ethyl phthalate about 135 gms. per kilo. A frequently occurring impurity is trinitro-xylol, and this is tested for by shaking, in a closed tube, equal weights of the product and benzol. When trinitro-xylol is absent, the product is totally soluble. Its melting-point is 112° to 113°. Musk xylol is the original synthetic musk prepared by Baur by condensing pure meta-xylene and tertiary butyl chloride in the presence of aluminium chloride, and nitrating the hydrocarbon produced, after purification, with a mixture of fuming nitric and sulphuric acids.

Musk Ketone resembles natural musk in odour more nearly than does musk xylol. It melts at 130°, and is prepared by condensing butyl toluene and acetyl chloride in the presence of carbon disulphide and aluminium chloride and nitrating the product.

Musk Ambrette melts at 85°. It is much more powerful in odour than musk xylol and musk ketone, and has, in addition, a pleasant suggestion of ambrette. It is prepared by condensing meta-cresol with butyl chloride, nitrating the product and converting it into its methyl ether.

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