

- 1972 (6), 70 (*Chem. Abstr.*, **80**, 78053r).
- (219) Vialatte, B., *C.R. Acad. Sci., Ser. C*, 1972, **275** (23), 1407 (*Chem. Abstr.*, **78**, 79369u).
- (220) Vialatte, B., *J. Radioanal. Chem.*, 1973, **17** (1-2), 301 (*Chem. Abstr.*, **81**, 32933v).
- (221) Vinogradov, A. V., Zemlyanukhina, N. A., Pavlova, I. V., Dronova, M. P., Lopatina, N. N., *Zavod. Lab.*, 1973, **39** (2), 148 (*Chem. Abstr.*, **78**, 154549h).
- (222) Vrbsky, J., Fogl, J., *Sb. Vys. Sk. Chem.-Technol. Praze, Anal. Chem.*, 1971, H7, 75 (*Chem. Abstr.*, **77**, 109082u).
- (223) Walton, R., Report 1973, GEPP-114 (*Chem. Abstr.*, **80**, 124025h).
- (224) Woolley, J. F., *Int. Congr. At. Absorpt. At. Fluorescence Spectrom., Pap., 3rd*, 1971, **2**, 871 (*Chem. Abstr.*, **79**, 132570b).
- (225) Yamaguchi, M., Tomida, S., Shio, H., *Nagoya-Shi Kogyo Kenkyusho Kenkyu Hokoku*, 1973 (50), 27 (*Chem. Abstr.*, **81**, 20491m).
- (226) Yamauchi, T., Somiya, S., *Seramikkusu*, 1973, **8** (7), 523 (*Chem. Abstr.*, **80**, 18785w).
- (227) Yoshimori, T., Umezaki, S., Tanaka, Y., *Bunseki Kagaku*, 1972, **21** (10), 1381 (*Chem. Abstr.*, **78**, 52147h).
- (228) Yoshimori, T., Ishiwari, S., Watanabe, Y., Harada, T., Yamada, S., *Trans. Jap. Inst. Metals*, 1973, **14** (5), 396 (*Chem. Abstr.*, **80**, 123815d).
- (229) Yoshimura, W., *Bunseki Kagaku*, 1972, **21** (4), 475 (*Chem. Abstr.*, **77**, 69753e).
- (230) Yudelevich, W. G., Vall, G. A., *Tr. Novosibirsk. Inst. Inzh. Geod., Aerofotos'emki Kartogr.*, 1972 (28), 51 (*Chem. Abstr.*, **78**, 168129h).
- (231) Zhukova, M. P., Morein, N. G., Shashura, M. V., Ozerskaya, F. A., *Sb. Tr. Tsent. Nauch-Issled. Inst. Chern. Met.*, 1972 (79), 61 (*Chem. Abstr.*, **77**, 69677h).
- (232) Zinchenko, V. A., Vasil'eva, L. S., *Tr. Vses. Nauch.-Issled. Proekt. Inst. Alyum., Magn. Elektrod. Prom.*, 1972 (81), 135 (*Chem. Abstr.*, **80**, 66378n).
- (233) Zinov'eva, L. D., Gladysheva, K. F., *Zavod. Lab.*, 1973, **39** (1), 23 (*Chem. Abstr.*, **78**, 118878f).
- (234) Zmijewska, W., *J. Radioanal. Chem.*, 1973, **14** (2), 267 (*Chem. Abstr.*, **80**, 10013x).

Essential Oils and Related Products

Ernest Guenther, Gilbert Gilbertson, and Roman T. Koenig

Fritzsche Dodge & Olcott Inc., New York, NY 10011

This fifteenth review of the analysis of essential oils and related products covers the literature from September 1972 to August 1974, inclusive. It follows the general pattern previously established (194).

Analytical investigations of the compositions of essential oils have continued at an intensive pace over the past two years. In addition, many analyses of the volatile components of foods and flavors have been undertaken. Much of this work was made possible by the continuing refinement of instrumental methods and by innovations conceived by the investigators. As before, the authors have attempted to include all papers dealing directly with the analysis of essential oils. It was impractical to review all investigations into the structure of essential oil components, but a selected number have been included to give a general view of the progress in this field.

As in recent years, nearly all investigations have been conducted using a combination of gas chromatography with mass spectrometry or other instrumental methods, together with physical and chemical methods. The combination of methods employed is often not mentioned in the review, since the reader can generally surmise what methods were employed, or he may refer to the original article when required.

Official Compendia. The National Academy of Sciences (388) issued the first "Supplement to the Food Chemicals Codex Second Edition," effective January 15, 1974. This supplement contains modifications of the aldehyde test and the heavy metals test, as well as numerous modifications of previous monograms and a number of new monograms. The Essential Oil Association of the USA (140, 141) issued EOA No. 1-1D-3-1, which describes a standardized gas chromatography analytical procedure. They also published EOA FP-2, which is a list of closed cup flash-points of 225 aromatic chemicals and isolates.

Books and Articles. An introduction to the chemistry and nomenclature of terpenes, including monoterpenes, sesquiterpenes, diterpenes, and others was edited by Newman (393). Fischer (147) wrote a review of the analysis of

essential oils and related products, wherein the 19 principal German reference works on the subject are described. Merkel (364) published a small work describing many of the commonly employed fragrant substances; and an informative general review of recent progress in perfumery materials, including the jasmones, musk odor compounds, and essential oils was written by Bedoukian (50).

The CRC press published three handbooks which contain much data of interest to the analyst of essential oils and related products. The "CRC Atlas of Spectral Data and Physical Constants for Organic Compounds," edited by Grasselli (190), contains data on 8000 organic compounds, including physical properties, IR, UV, NMR, and mass spectra. The "CRC Handbook of Spectroscopy" edited by Robinson (459), is a reference book of spectroscopy data available on the most important materials in various fields of spectroscopy. The "CRC Handbook of Chromatography," edited by Zweig (609), contains data on 12,000 compounds, while it also suggests quantitative methods of analysis in gas, liquid, paper, and thin-layer chromatography.

General Procedures. The instruments and methodologies for the isolation, fractionation, and identification of volatile flavor compounds, with special emphasis on their isolation, was discussed by Chang (91). Von Rudloff (576) considered the scope and limitations inherent in the gas chromatography of terpenes in chemosystematic studies, and Wellner (585) reviewed the techniques employed in quality control of essential oils and other volatile flavoring materials.

By interpolating the efficiency of a gas chromatograph based on an analysis of citral, its performance for the analysis of essential oils and fragrances could be predicted in respect to separating power, decomposition tendency, column polarity, and sensitivity by Brandauer and Ziegler (73). Blumenthal and Chang (62) described a method for obtaining reproducible quantitative gas chromatograms by first saturating the chromatographic column with at least seven consecutive runs. McGugan and Howsam (339) devised a test for detecting the loss of volatile components in a gas chromatographic column. The sample was recovered from the effluent for comparison with the original sample. Prevot et al. (440) investigated four trapping techniques as

Authors have not been supplied with free reprints for distribution. Extra copies of the review issue may be obtained from Special Issues Sales, ACS, 1155 16th St., N.W., Washington, DC 20036. Remit \$4 for domestic U.S. orders; add \$0.50 for additional postage for foreign destinations.

related to panel test threshold values. They found an enrichment recycling technique to be the most suitable.

The Perkin-Elmer F 21 gas chromatograph was fitted to its outlet with a thin layer plate moved by the recorder synchronously with the paper. This method enabled Still and Knauer (520) to identify the peaks from the gas chromatograph. Ryhage (469) discussed improvements in the gas chromatograph-mass spectrometer interface, and the recording of chromatograms and mass spectra, together with computer analysis.

A procedure whereby unknown compounds eluted from a gas chromatograph are trapped and subsequently identified by colorimetric functional group analysis at concentrations lower than had previously been possible, was described by Cronin and Gilbert (109). Kaminski et al. (254) utilized a functional reaction technique for detecting volatile organic compounds by gas chromatography. Functional group fractionation was accomplished by Palmer (416), who first eluted the mixed flavor components from coffee through silica gel with Freon, and then applied gas chromatography and mass spectrometry to the aroma-bearing fractions which were thus obtained. Kubeczka (309) employed preliminary dry-column chromatography to first separate an essential oil into fractions of different polarity, whereupon the fractions were studied by gas chromatography, followed by other procedures. The preliminary separation resulted in an eventual more definite identification of the components. Dimitrov and Jennings (130) constructed a glass apparatus to facilitate a new method for extraction of volatile compounds. The apparatus assured good reproducibility, precision, and sensitivity, and the extract obtained was suitable for gas chromatographic investigation.

Advantages and disadvantages of thin-layer quantitative analysis were discussed by Kirchner (283), who gave some suggestions for the minimizing of errors. Liquid-liquid reversed phase partition chromatography was employed by Kozhin et al. (297) for the analysis of essential oils and separation of components. Walradt and Shu (580) utilized a simple gel-permeation chromatographic method for the liquid chromatographic determination of the extent of oligomer or polymer formation in flavor chemicals.

Nuclear magnetic resonance was used to detect interaction of fragrances with the substances in which they were incorporated, especially proteins, by Lemberg (330), who employed D₂O solution to broaden selectivity of the NMR signal. Bose and Brambilla (71) studied the potential of carbon-13 labeling for biosynthetic studies by observing the ¹³C NMR spectra for citronellol and related terpenes, obtained by using off-resonance decoupling and other techniques.

An automatic procedure for sorting IR spectra with a programmable desk-top calculator was developed by Rann (447).

Characteristic functional group reactions, previously used for colorimetric and fluorimetric determinations of organic compounds, were adapted to UV spectrophotometric determinations of functional groups by Pesez and Bartos (427).

Zone melting was employed by Dugacheva et al. (135) for the purification of aromatic chemicals.

An improved apparatus for the quantitative estimation of essential oil in a plant product was devised by Hendriks (210). The apparatus allowed a shorter distillation time and the direct estimation of the oil quantity. Stanislas et al. (518) also described an improved apparatus for the volumetric determination of essential oil by steam distillation from plant material. The apparatus incorporated two exchangeable tubes, connected through ball and socket joints,

for the collection of oil. Takaishi (530) employed a steam distillation apparatus for the determination of essential oil in micro quantities. The distillation apparatus was used in conjunction with gas chromatography. An increased yield of oil was obtained in the extraction from distillation waters by liquid-liquid chromatography on a production scale by Kozhin et al. (295, 296). They also perfected another method for extracting oils from distillation waters by filtration through a porous material containing a chlorinated solvent. Strobel (522) invented a unique method for separating components, which consists of pulsing wet steam through a zone containing the material to be separated. The aroma concentrate is collected in a trap held at the temperature of liquid nitrogen. The changes in the dielectric constants and other properties of several essential oils during aging were examined by Luedde (337). Dielectric constants were determined by Panenko et al. (417) for a number of essential oils and extracts, including rose, sage, and lavender. Chapard et al. (93) analyzed the oxidation products of several oils and found that it was simpler to identify the oxidation products than it was to identify the original oils.

Essential Oils—General. The difficulties in identifying and controlling the quality of essential oils, due to variations in their chemical composition, caused by preparation methods, geographic origin, seasonal variations, and differences in chemotypes, were related by Granger et al. (187). Eiserle and Rogers (138) described the composition of volatile oils derived from oleoresins. The bactericidal properties of essential oils and aromatic substances were compiled by Fuehrer (153). Chkhaidze et al. (98) studied the effect of ionizing radiation on the odor and properties of several essential oils. Livshits (335) gave details of the composition and preparation of several substitute synthetic essential oils, mentioning main components employed in synthetic bergamot, patchouly, vetiver oils, and others.

The character and chemical composition of numerous Afghanistani essential oils were ascertained by Younos et al. (602-604).

Brazilian essential oils were investigated by Peyron (429), who studied cabreuva, palmarosa, citronella, petit-grain and patchouly oils; and by Mancini et al. (344, 345), who determined the best thin-layer chromatography system of analysis for the essential oils which are official in the Brazilian Pharmacopeia.

The essential oils of the Georgian SSR were analyzed and described by Yakobashvili (593).

Seven wild aromatic plants growing in Greece were distilled by Skrubis (507), who determined the major components of each oil obtained.

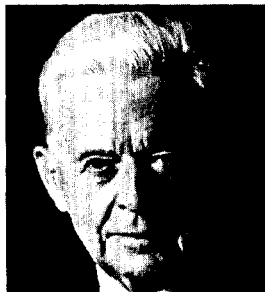
The essential oils of India and their analytical properties were discussed by Sood (512). Baslas and Baslas (48) related the occurrence, characteristics, and properties of the major commercial Indian essential oils, and Sinha and Singh (506) conducted chemical examinations of oils of *Cymbopogon distans*, *Erigeron multiradiatus*, and *Mosla ocimoides*, which were obtained from Kumaon.

The contents and physical properties of Vietnamese essential oils from three plants, including a type of citronella grass, were determined by Lucius and Alder (336).

Yugoslavian essential oils and their physicochemical characteristics were delineated by Tusakov (562).

Individual Essential Oils. The cortical essential oil of *Abies balsamea* was analyzed by Lee et al. (328), who identified many components, some for the first time. Hunt and Von Rudloff (236) differentiated between *A. balsamea* and *A. lasiocarpa* by the relative quantity of terpenes, piperitone, methylthymol, and thymol. The seed volatile oils

Ernest Guenther, born in Munich, Germany, attended the Technische Hochschule there as well as the University of Munich. He took his PhD at the University of Zurich in 1920. Associated with Fritzsche Dodge & Olcott, Inc., since 1924, he is now Executive Vice President and Technical Advisor of the company, recently retired. An expert in the field of essential oils, Dr. Guenther has published approximately 150 papers in various scientific journals and is author of six volumes of "The Essential Oils." His American Chemical Society activities include numerous lectures before local chapters throughout the United States and Canada. He expects to continue doing field investigations in various parts of the world, making films for subsequent lectures.



Gilbert Gilbertson is Technical Advisor for International Operations at Fritzsche Dodge & Olcott, Inc., directing the flow of technical information to the foreign divisions. He is also concerned with patent applications, foreign food laws, and biological testing. He is a member of the Fritzsche-D&O Quarter-Century Club and in the past has held various positions with the company in production, the Flavor Laboratories, the Analytical Laboratories, and research and development. Serving in the U.S. Navy, he studied electronics at the U.S. Naval Research Station in Washington, D.C. He studied chemistry at Pratt Institute, Brooklyn, N.Y., and received his BS from Seton Hall University, South Orange, N.J.



Roman T. Koenig has been in charge of Technical Information and the Libraries at Fritzsche Dodge & Olcott, Inc., since 1959. He is also Editor of the *Fritzsche-D&O Library Bulletin*, a monthly publication comprising current technical articles, which services the essential oils, perfume, and flavor industries all over the world. Previously, Dr. Koenig was a member of the staff of the Technical Information Division of Esso Research and Engineering Co., and prior to that he had been engaged in physicochemical research in industry and taught at universities both in the United States and England. Born in Poland, he obtained his PhD in chemistry in 1946 at the Imperial College of Science and Technology of the University of London. He is a member of the American Chemical Society.



from various *Abies* species, European, American, and Japanese, were differentiated by Cermak et al. (88). Tyukavkina et al. (564) isolated maltol from the needles of *A. sibirica*.

Sacco et al. (472) identified many constituents of various *Achillea* from the west-central Alpine region, and Bohlmann and Zdero (65) characterized several polyacetylenic compounds from *Achillea* species.

The oil of *Aegle marmelos* was demonstrated by Bhandari and Gupta (60) to contain ethyl *n*-amyl and methyl *n*-heptyl ketones among many other constituents.

Fujita and Fujita (162) proved the main compounds of oil of *Agastache rugosa*, among many others identified, to be methyleugenol and methylchavicol.

The oil of *Ageratum conyzoides* was investigated by Kasturi et al. (270), who determined the structure of two new components; Rao and Nigam (450), who identified its main constituents, and Sood (510) who examined the oil from the flowers.

The oils from Ajowan fruits and herbs were distilled by Balbaa et al. (37), who compared their characteristics and chemical composition.

Thin-layer and gas chromatography resulted in the eluci-

ation of the composition of the oil of *Aloysia triphylla* by Montes et al. (371).

By chemical and instrumental methods, Fujita and Yamashita (155) newly detected 46 compounds in the oil of *Alpinia speciosa*.

Oil of *Andropogon iwarancusa* was shown by Garg and Nigam (169) to contain high percentages of carene, piperitone, piperitol, and perillyl alcohol.

Oils from angelica and lovage, obtained by combined extraction and distillation and having superior odor characteristics and solubility, were described by Kulesza and Podlejski (313).

The oils from numerous *Aniba* species were examined by Alpanse de Morais et al. (14), and nitrophenylethane, phenylethyl benzoate, and trimethoxyallylbenzene were found.

Truly positive differentiation among oils of anise, star anise, and fennel, was accomplished by Herriset et al. (212) with thin-layer chromatography. Ballarian and Ballarin (38) used a combination of color reactions and chromatography for the same purpose. Kravets et al. (303) estimated anethole content by a simple determination of the refractive index and other methods.

Furan, pyrrole, and phenolic compounds were identified among the volatile products in heated apple juice by Brule (79).

The terpenoid composition of several *Aralia* species was investigated by Yoshihara (601).

The chief components of the essential oils from several *Arnica* species were identified by Willuhn (589, 590). He reported that phlorol isobutyric acid and phlorol methyl ether could not be confirmed in the rhizome oils of *A. montana*.

In the oil from *Artemisia abrotanum*, Radu et al. (444) detected the presence of 28 components and confirmed eucalyptol. Fesneau (145) characterized the essential oil of *A. coerulescens* and determined the phenols, aldehydes, ketones, and cineol content. Enanthic acid and creosol, among others, were identified by Dudko et al. (134) in the oil from *A. compacta*. Two new monoterpene lactones were isolated, and characterized, from oil of *A. filifolia* by Torrance and Steelink (556). Matsuo et al. (357) determined 15 components from the neutral portion of the oil *A. fukudo*. Its main components were α - and β -thujone. The oil of *A. herba alba* was analyzed by Cohen et al. (104). Rovesti (464) reported that the oils from *A. rehan* and *A. pallens* cultivated in Ethiopia yielded essential oils with odors similar to that of Indian oil of davana. He also reported the main components of the oils. Oil of *A. santonica* f. *citralifera* growing in the Crimea contained 58% of α - and β -citral, according to Khort and Gogol (278). Detailed descriptions, including physicochemical properties and many of their main components, were written for several *Artemisia* species growing in Siberia by Berezovskaya et al. (58). Kozhin et al. (294) identified 13 compounds in oil of *A. taurica* and 8 in oil of *A. monogyna*. Shiskov et al. (502) showed that *A. taurica* contained 75% α - and β -thujene and 18% cineole.

A method for the determination of the volatile oil content of *Asafetida*, based upon oxidation, was evolved by Abraham et al. (3).

The oils from several *Asarum* species from Europe and Asia were analyzed by Endo and Ogino (139), who identified new sesquiterpene alcohols as well as linalool in these oils.

At least 53 components were separated from Chinese Oil of *Atractylia ovata* by Schmidt and Muehlstaedt (487) and 31 of these were identified.

Copalic and other related acids were isolated from oil *Balsam copaiba* and characterized by Mahajan and Ferreira (342). Akisue (12) identified the main components in oil Balsam Peru.

A summary of the 300 volatile constituents of banana, which have been identified, was given by Dupaigne (136). Tressl and Drawert (559) also discussed the components of banana, many of which are esters and carbonyls.

Oils obtained from European type basil were analyzed by Zola and Garnero (608), who found that French and Italian oils were characterized by a large amount of methylchavicol whereas Moroccan oils had a high eugenol content. Kotlyarova and Salakaya (292) compared the efficacy of the chemical and spectrophotometric methods for the determination of eugenol in basil oils. Peyron et al. (430) demonstrated that oil ylang-ylang was often employed in the adulteration of Reunion type basil oils. The antifungal effects of Bulgarian basil oil and its fractions were described by Toleva et al. (552).

An organoleptic evaluation of a low-temperature distillate of canned beef was correlated by Persson and Von Sydow (426) with instrumental analysis, and 95 compounds, including sulfur compounds, aldehydes, ketones, alcohols, and furans were identified. Chang (92) identified 59 compounds in a boiled beef extract, but none of these by itself was characteristic of beef flavor.

In the analysis of the sesquiterpene fraction of bergamot oil, Calabro and Curra (84) employed preparative and analytical gas chromatography. Besides sesquiterpenes, certain paraffins were also identified. Huet and Dupuis (233) observed changes in the main components of bergamot and clementine oil with ripening of the fruit.

Among constituents identified by combined gas chromatography and mass spectrometry in blackberry essence, Gulan et al. (195) reported dimethoxy- and trimethoxybenzene and phenylethyl formate.

Several hydrocarbons and alcohols were found in oil of *Boenninghausenia albiflora* by Sood (511).

Mulders et al. (380, 381) used several combinations of techniques to discover numerous volatile components of white bread, many of which had not been reported previously.

Lamparsky and Schudel (322) proved by synthesis the structure of a newly isolated component of buchu leaf oil, *p*-methan-8-thiol-3-one.

Urbach et al. (566) showed the presence in butter oil of many volatile lactones, phenol, indole, skatole, and others.

The rate of decrease in quantity and change in properties of oil in calamus roots during storage was observed by Jukneviene (247). Niwa et al. (406) established the stereochemistry of two new selinane-type sesquiterpenes isolated from the oil.

Based on cineole content and the yield of oil from *Callistemon speciosus* grown in Brazil, Wasicky and Saito (583) suggested the best time for harvesting to be January to August. Moreira et al. (373), in a similar series of volatile oil determinations, narrowed the best time for harvesting to February through April.

Oil of *Calyptranthes spruceana* was shown by Correa et al. (107) to contain 25% perillaldehyde, whereas oil from an unidentified species contained 98.7% methylchavicol.

Oil from the camphor tree, *Cinnamomum reticulatum*, growing in Taiwan, was found by Fujita and Fujita (163) to contain 96.8% *l*-linalool and no camphor or phenol ethers. Fujita et al. (164, 165) also examined the oil from *C. camphora* at various growing stages. The content of camphor was 30–43% in the fruits, and 77–91% in shoots of varying

maturity. Oils from *C. camphora* var. *linaloolifera* were similarly analyzed. Ioshida (240) isolated and determined, by IR and NMR, and by hydrogenation, the structure of two new alcohols in oil of *C. camphora*, and also identified many other constituents.

New constituents of oil of *Cannabis sativa* were identified with mass, IR, and NMR spectra by Stahl and Kunde (517). Malingre et al. (343) also characterized a number of new substances including several cannabinoid components.

The quality of caraway oil was evaluated using thin-layer chromatography by Machovicova et al. (340). Rothbaecher and Suteu (461) separated *trans*- and *cis*-carveol, by formation of complexes, from Rumanian caraway oil.

The composition of Alleppy cardamom oil was clarified by Barua et al. (43), employing two different gas chromatographic techniques for the high- and low-boiling fractions.

Some of the main constituents in oil of *Carissa fraxandras* were identified by Chandra (89). Coumarin was the most abundant component of 96 isolated and identified in oil of *Carphephorus odoratissimus* by Karlsson et al. (266).

The oil from the above-ground growth of carrots was examined by Bakina et al. (34). Many of its components were identified, the most abundant being geraniol and its acetate. Chelovskaya and Nikolaev (95) analyzed the oils from wild, and cultivated carrot seed and found that the wild variety contains more simple terpenes such as geraniol, whereas the cultivated carrot seed oil contains more complex bicyclic terpenes. Sparenburg (516) utilized a gas chromatographic method for the determination of carotol. *n*-Heptadecane was employed for verifying the internal standard.

Sixteen low-boiling components of cascarilla oil were separated and identified by Motl and Trka (379). The presence of a C-13 ketone was also detected. In addition to many known constituents of cascarilla oil, Claude-Lafontaine et al. (103) isolated and identified a new sesquiterpene phenol, cuparophenol.

Three novel keto-alcohols were isolated and characterized by Varshney et al. (568) from leaf oil of *Cassia auriculata*.

The essential oils from *Cedrus deodara* and other cedar trees cultivated in the Crimea, were evaluated by Akimov and Kuznetsov (11). Fourteen identical components were found in all of the oils. Shankaranarayanan et al. (496) isolated and established the structure of deodarone from *C. deodara*.

Fractional distillation, chromatography, and mass spectrometry were employed by Motl and Trka (378) to establish the chemical composition of oil from *Cedrela odorata*.

The essential oils from *Chaerophyllum hirsutum* from flowers, fruit, foliage, and rhizome, were distilled by Kudrzycka-Bieloszabska and Glowniak (312). The properties and some of the chemical components were determined.

A method for the differentiation between Roman and German chamomile oils, utilizing UV, IR, and Raman spectra, was described by Herisset et al. (213). The chemistry of chamazulene in German chamomile oil was investigated by Evodkimoff et al. (143). Kerekes et al. (274) extracted chamomile oil from distillation waters with activated carbon and studied the composition of the oil. Pomini (436) described the botany and composition of the essential oil of Roman chamomile. The quantity and composition of the oils from 9 different strains of German chamomile, especially with regard to azulene, was described by Schaefer and Stein (484), and the characteristics of oil from German chamomile grown in Hungary, were investigated by gas and thin-layer chromatography by Tyihak et al. (563). Schilcher (485, 486) suggested an improved method for the deter-

mination of essential oil in chamomile flowers, and differentiated 4 chemical types of chamomile oil, based on the occurrence of (-)- α -bisabolol, and isomers of bisabolol oxide.

The volatile aroma from cheddar cheese was gas chromatographed by Manning and Robinson (346), who reported that the aroma was chiefly due to 4 compounds, H₂S, methanethiol, dimethylsulfide, and diacetyl. Ney and Wiratama (395) found, by different chromatographic methods, numerous components in the aroma of a German blue mold cheese.

About 30 hydrocarbons were detected and 25 identified by Rustembekova et al. (468) in the oil *Chenopodium botrys*.

The constituents of several essential oils from chrysanthemums native to Japan, were ascertained by Matsuo et al. (359), using gas chromatography, IR, NMR, and mass spectra. Bohlmann et al. (67) identified a new acetylenic compound in *C. crassifolium*, and established its structure.

The yield and composition of oil cinnamon Ceylon from various parts of the plant, was investigated by Angmor et al. (23). The rootbark yielded the highest amount of oil; however, camphor was the major component of that oil.

The essential oil of *Cinnamosma fragrans* from Madagascar showed the occurrence of 34 substances according to an investigation by Schulte et al. (489).

A number of distinct chemical differences between the oils of citronella Ceylon and citronella Java were demonstrated by Wijesekera et al. (588), using gas chromatography. Nigam and Datta (396) also pointed out chemical differences among the oil of various species of Cymbopogon, including Ceylon and Java citronella oils. Sixteen terpenoids were identified by Razdan and Koul (451) in the oil of citronella Java type. Carvone was reported for the first time. Soular and Fanghaenel (513) studied a citronella oil produced in Cuba and established that it was the Java type. Gulati and Sadgopal (197) also conducted an investigation into the properties and chemical composition of oil citronella Ceylon.

Citrus oils from Florida and the methods of analyzing them were described by Kesterson et al. (275). Herisset et al. (216) utilized a combination of thin-layer chromatography with UV, IR, and Raman spectrography for the differentiation among citrus oils. The analysis of essential oils from fruits, leaves, and flowers, of citrus plants was discussed by La Face (320), with references to the determination of purity. Goretti and Liberti (183) used gas chromatography to analyze various citrus oils and found that irrespective of the column used, about 300 components were found in each of them. The physicochemical properties of Italian bergamot-orange was ascertained by De Leo (117). Di Giacomo (121-126) outlined criteria for quality evaluation of essential oils from citrus fruits. Particular mention was given to methods for ensuring the purity of the oils, and the characteristics and composition of the nonvolatile residue of expressed citrus oils. The chemical composition of citrus oils was also delineated by Di Giacomo and Calvarano (127). The peel oil of citrus iyo prepared by expression was analyzed by Hirio and Takaoka (221), who isolated 52 compounds, 15 of which had not been found previously in citrus oils and some which had not been previously found in nature, such as *trans*-2,6,6-trimethyl-2-vinyl-5-hydroxytetrahydropyran. The leaf oils from 10 different citrus taxa and 8 citrus hybrids were analyzed by gas chromatography by Kamiyama and Amaha (255), who identified 43 compounds. Karawya et al. (263) analyzed leaf oils from citrus plants growing in Egypt. Thiebaut (544) described citrus essential oils from the Ivory Coast.

Huet (232) adapted a new method for determining essential oils in a small disk from the peel of citrus fruit by distillation and the bromide-bromate titration method of Scott and Veldhuis. Silica gel plates and color reactions were employed by Sardi and Retamar (480) for distinguishing aldehydes in citrus fruit essential oils. Sato (482) studied the products evolved by the exposure of lemon and orange oils to air. Qualitative fluorimetry of coumarins and furocoumarins was employed by Martin and Berner (350) to identify various citrus oils.

Huneck (235) reported the presence of 2-hydroxy-4,6-dimethoxy-5-methylacetophenone in clove oil.

More than 300 volatile compounds were identified in roasted cocoa beans by Keeney (272), who also discussed the effect of fermentation and roasting on the flavor compounds.

Claggett et al. (102) determined many of the low-boiling components in *Comptonia asplenifolia* oil. The chemical composition of several Bulgarian coniferous essential oils was found by Nikolov et al. (402, 403) to be very similar, and physical constants cannot be used to identify individual oils. They also obtained the physical constants for concrete and absolute oils obtained from Bulgarian pine trees. Ognyanov and Tsankova (410) identified many of the chemical components of the oils from several Bulgarian coniferous trees.

The yield of oil from coriander seed, as related to moisture content, was determined by Berstovaya (59). Ponomarev and Shlyapnikova (437) determined the loss in oil resulting from drying and crushing the seed. This oil could be collected by adsorption on aluminum oxide. Goi (181) evolved a rapid determination of linalool, using the interferometer ITP-2. Lishtvanova and Moskalenko (334) identified 12 components in coriander oil and studied the determination of linalool by gas chromatography. Meerov et al. (361) obtained a coriander oil by extracting the seeds with condensed CO₂. The essential oil was steam-distilled from the extract. Euk'yanov and Berestovaya (338) studied the oil content and composition in various parts of the coriander plant during its growing cycle; whereas the oil content in the vegetative parts changed considerably and decreased towards the end of the growing period, the oil in the seeds remained constant in quality and quantity.

The essential oil content of several varieties of cumin seeds and their chemical composition was studied by Georgiev et al. (171).

A definite distinction between two types of oil of curcuma was established by Herisset (211), using thin-layer and gas chromatography, but especially with spectrophotometric methods.

Sensory evaluation and gas chromatographic analysis of the volatile components from black currants were correlated, using a statistical approach involving computer techniques, by Karlsson-Ekstrom and Von Sydow (267).

The components of the essential oil of *Cymbopogon goerin* were examined by Fujita and Fujita (160), who identified 2 new sesquiterpene ethers. Rovesti (462) examined the yield of oil of *C. sennarensis* over the seasons and determined some of its components. The odor of the oil has similarities to peppermint and palmarosa.

Four components were isolated by Auterhoff and Momberger (31) from the higher boiling fractions of oil of damiana leaves. Preparative gas chromatography and physical methods were employed.

The chemical differences between oils of European and Indian dill were demonstrated by Baslas and Baslas (45-47), and a detailed analysis of the composition of the European type was reported. Irinchev et al. (241) investigated

the composition of Bulgarian dill oils, and correlated it with weather conditions.

Dubovenko et al. (133) distilled and identified some main components of the oil of *Dracocephalum foetidum*.

The physicochemical properties and some major components of an oil distilled from *Entandrophragma cylindricum* were determined by Talalaj (532).

Thin-layer chromatography served Bonzani da Silva and Grotta (70) to isolate eleven components from the oil of *Erechtites valerianefolia*.

Gas chromatographic analysis by Suemitsu et al. (523) of oil of *Eriobotrya japonica* indicated that nerolidol and farnesol were the main components among many identified.

Oil of *Eucalyptus citriodora* cultivated at Jorhat and analyzed by Barua et al. (44) contained 77.9% aldehyde as citronellal. Cardoso do Vale and Proenca da Cunha (86, 87) identified many components of oil of *E. globulus* collected at Torres Vedras. They suggested that distillation procedures in Portugal should be improved to yield colorless oils with higher cineole content. Yllera Camino (600) reviewed the composition, adulteration, and analysis of oils of *E. globulus* and *E. camaldulensis* in Spain. Prakash and Sinha (438) published a chemical study of oil from the fruits of *E. globulus*. Southwell (515) studied the compositional variations in oil of *E. punctata*. Brasil e Silva and Bauer (75) found 52 to 66% cineole in oils from three varieties of eucalyptus growing in Brazil, and Chkhaidze et al. (97) reported that the main component of a Russian eucalyptus oil was geranyl acetate.

The major constituents of leaf oil from *Eugenia bracteata* were identified by Rao and Nigam (449).

Several of 21 compounds present in oil of *Euphorbia monostula* were identified by Karryev and Mamedov (268). Thymol was found at 39%.

A greater quantity of fennel oil was obtained by Kasimovskaya et al. (269) by distilling the entire above-ground plant than by distilling only the seeds. The anethole content of the oil was also comparable to that from the seed oil. German and Rumanian fennel were examined by Trenkle (558), who distilled oil from various parts of the plants and compared them. The root oil was examined for the first time. Paukov et al. (420) analyzed fennel oils obtained after experimental treatment of the seeds and the plants.

The techniques of steam distillation, solvent extraction, trapping the volatile essence, molecular distillation, preparative and analytical gas chromatography, and others, were all employed by Kinlin et al. (282) to identify the volatile components in roasted filberts. He reported 187 new constituents for the first time.

The composition of the oil from several Turkish fir species was elucidated by Okay (411). Oil from the wood of Douglas fir was investigated by Sakai and Hirose (475, 476), who established the absolute configuration of (+)-methyl todomatuate and its newly discovered analogs, and todomatuic acid, isolated from the oil. Snajberk et al. (508) determined the composition of volatile oil from the cortical oleoresin of Douglas fir.

The composition of the terpene fraction from galbanum resin was investigated by Naves (390), who also confirmed propenyl butyl sulfide by IR and NMR spectrometry. Riezobos (453) detected minute quantities of alkoxyalkylpyrazines in galbanum oil and described their importance to the odor.

Oil of geranium was examined with gas and thin-layer chromatography by Gogiya and Ivanova (178, 179) and its physicochemical characteristics and chemical composition were reported. They also similarly analyzed the oils from two other geranium species, *Pelargonium capitatum* and

P. radula. Ivanova and Gogiya (242, 243) further explored the variation in oil content of these two species over the growing season, and identified additional components. Kravchenko and Rik (301) demonstrated that photoionization mass spectrometry is more convenient for the analysis of geranium oil than electron excitation. Raguenaud (445) analyzed fractions taken from geranium oil during commercial distillations. De la Torre and Retamar (115) established the percentage of the main constituents in oil of *P. hortorum*, and Nikolov et al. (401) reported the physical constants of oils extracted from *Geranium macrorrhizum* with petroleum ether and alcohol. Zhelev (607) conducted a more extensive analysis and identified many components, including germacrene and oxygen-containing sesquiterpenes which comprised 90% of the oil. Hefendehl (207) found that the chief component of oil of *Pelargonium tomentosum* was (-)-isomenthone. This contradicted earlier reports by other investigators. Pesnelle et al. (428) established the stereochemistry of 10-epi- γ -eudesmol, isolated from African geranium oil. A rapid method for determining menthone using IR spectrometry and measuring the absorption band at 1720 cm^{-1} was proposed by Kachakhidze et al. (248), and Kotlyarova et al. (291) demonstrated an improved oximation assay including pretreatment of the oil with alkali to diminish differences in results obtained from hot and cold oximation procedures. The citronellol and geraniol content of Egyptian geranium oil was reported by Osman (414).

In oil of ginger, Kami et al. (253) identified several alkanes, carbonyl compounds, alcohols, sulfides, esters, terpenes, and cineole. Mathew et al. (355) studied the odor quality of oils produced from Australian ginger varieties by several methods. Masada et al. (353) investigated the pungent principles in Japanese ginger and reported new gingerols not previously known.

Some important aromatic chemicals were identified in muscat grapes, and their influence on the aroma was shown by Terrier et al. (543).

Odoriferous resins having volatile components which are of value in perfumery were obtained from several species of *Grindelia* by Kapelev (257).

Terpenes and 1,2-methylenedioxy-3,6-dimethoxy-4-allylbenzene were found in oil of *Heckeria umbellata* by Brasil e Silva and Bauer (74), using thin-layer chromatography and IR spectroscopy.

Bohlmann and Zdero (66) characterized a new azulene isolated from *Helichrysum bracteatum*.

The terpenes in the oil of *Heterothalmus psiadioides* were identified by Brasil e Silva and Bauer (76), who concluded that the oil could be of commercial importance, since the plant is abundant.

The oil of *Hibiscus syriacus* was distilled and analyzed by Hanny et al. (203).

An essential oil was produced by Bakhtadze and Dembitskii (33) from honeycombs, and the main components of the oil, which had an odor of honey and roses, were determined. Krotova et al. (307) conducted a more detailed analysis and reported at least 57 aromatic compounds in honeycombs, of which many were identified.

Some varieties of hops were distinguished by Naya and Kotake (392) on the basis of their essential oil composition. Shaw et al. (499) described the mass spectra of humulones and related compounds, and discussed which ions appear to be diagnostic for particular structures.

Thirty-two compounds were identified in the oil of *Houttuynia cordata* by Kameoka et al. (251). The characteristic odor appeared to be due to 3-keto-decanal, methyl nonyl ketone, methyl lauryl sulfide, and aliphatic alcohols.

Isolation and characterization of constituents from oil of *Hymenatherum tenuifolium* were reported by Krishnappa (304), and hymenetherene was shown to be a mixture of myrcene and other compounds.

The composition of jasmine oil, with emphasis on five new nitrogen-containing compounds, was discussed by Polak (434). Asaturova and Chkhenkeli (26) ascertained the physicochemical properties of oils from large-blossom jasmine obtained by dynamic sorption.

The oil of *Juncus roemerianus*, a marsh grass, analyzed by gas chromatography-mass spectrometry by Miles et al. (366), contained benzene and naphthalene derivatives, including tetrachlorobenzene and benzyl cyanide.

Junionone, the first vegetable monocyclic cyclobutane monoterpenoid ever found, was isolated from juniper berry oil and characterized by Thomas and Ozainne (548). Significant variations were found by Hoerster (225) in juniper leaf oils from different places. Talwar et al. (533) identified some main constituents in oil of *Juniperus macropoda*. Tatro et al. (536) investigated the differences among juniper leaf oils of three varieties. Banthorpe et al. (41) determined the main components in the leaf oils from eleven different junipers and concluded that juniper species are remarkable for producing such a small number of structural type components in their essential oil. The composition of the oils from various Asian junipers was ascertained by Goryaev et al. (185), and Tomita and Hirose (553) demonstrated that cadinenol from *J. rigida* is identical to (\pm)-epi-cubanol.

The essential oil from the wood of *Lansium anomalay-anum* was shown by Krishnappa and Dev (305) to consist primarily of three sesquiterpenes. The previously reported "chigadmarene" was actually impure α -gurjunene.

Forty components were found by Kolesnikova et al. (288) in the oil of larch.

Greek laurel oil of good quality similar to the leaf oil was obtained from stems and waste plant material by Pruidze and Kekelidze (441). Pruidze et al. (443) also studied the loss of essential oil with storage of laurel leaves, but found that the composition of the oil remained basically the same. Hogg et al. (228) discovered dehydro-1,8-cineole in laurel leaf oil. This was the first report of its natural occurrence.

The composition of oils of lavender and lavandin from Dalmatia was discussed by Devetak (119). The lavender oils had a cineole-like by-note which makes them inferior. The main components of Spanish lavender oils were determined by Martin Mesonero and Cabo Torres (352). Linalyl acetate was observed in only one of several oils. Adzet (5) quantitatively assayed many components of a type of lavender oil from plants collected in the Pyrenees. Picci and Manunta (433) analyzed the oils from lavender grown in 8 areas of Sardinia and correlated composition with climatic conditions. The oils differed from those from the same species grown in France. Several experimentally bred lavenders produced oils of differing composition, with linalyl acetate content ranging from 34 to 67%, as determined by Kravchenko and Bronshtein (302). The quantitative composition and physicochemical properties of Bulgarian lavender oil was ascertained in detail by Vlahov et al. (573), and Karetnikova and Kustova (265) elucidated the hydrocarbon composition of Russian lavender oil. Oil of spike lavender was examined by instrumental methods by Wobben et al. (592). Many compounds were determined, but geraniol was not found, and diethyl phthalate, present in some samples, was judged to indicate adulteration.

New strains of lavandin being developed in Italy and yielding an essential oil closer to that of lavender were de-

scribed by Giusti (175). Martin and Zola (349) also examined oils from new hybrids of *Lavandula*. One of the oils had less eucalyptol and more linalool. A new component of lavandin was proved through spectroscopic data, and confirmed by synthesis, by Belsten et al. (54), to be *trans*-5-hydroxy-2-isopropenyl-5-methyl-3-hexenyl acetate. Mookherjee et al. (372) isolated the carbonyl compounds from lavandin oil with Girard T reagent and identified 29, of which ten aldehydes, four ketones, and three bifunctional compounds were found for the first time. A gas chromatographic procedure for assaying linalool and linalyl acetate in lavandin oil was devised by Ognyanov and Panaiotova (409). Chingova-Boyadzhieva and Staikov (96) ascertained which type of lavandin gives the best yield and quality of oil when grown in the Kazanluk area. Damjanic et al. (112) described various terpeneless lavandin oils obtained by adsorption on SiO₂ and other solid adsorbents. Senic and Filipovic (493) derived correction factors for physical constants of lavandin oil with temperature variations.

The structures of some aliphatic monoterpenoids isolated from oil of *Ledum palustre* were determined by Von Schantz et al. (578) on the basis of their IR, NMR, and mass spectra.

The volatile compounds in lemon and lime juice essences were separated by gas chromatography and identified with mass spectrometry and IR spectroscopy by Moshonas et al. (375). Of 27 compounds identified in lemon essence, 25 were newly found, and all 34 volatile compounds from lime essence had not been previously reported therein. Pennisi et al. (424) studied the properties and chemical composition of "Femminello comune" lemon oil over the growing season and as affected by lime treatment of the peels. Calvarano and Di Giacomo (85) derived indications of the amount of oxidized compounds in, and, in some cases, the geographic origin of lemon oils by IR absorption. Bonaccorsi et al. (69) used gas chromatography to detect adulteration of lemon oil with the new synthetic material being used in recent years. Di Giacomo and Calvarano (128) found the ratio between CD value and absorbance at maximum wavelength useful for detecting adulteration. Fincke and Maurer (146) proposed an extraction and gas chromatographic method for the analysis of lemon oil and the changes in its composition when incorporated in non-fatty sweets.

The essential flower oil from Meier lemon was distilled by Topadze et al. (554). The properties of the oil and the chemical composition of the terpene and sesquiterpene fraction were determined.

Flower oils of lily of the valley and lilac were prepared by Mack and Keopsel (341) using sophisticated techniques involving trapping at liquid N or methanol-Dry Ice temperatures. In lily of the valley oil, 27 components were identified, of which 20 had not previously been reported, and 12 previously reported components could not be found. The oil contained 80% of cinnamyl alcohol. In lilac oil, 16 components were found, of which 12 had not previously been reported, while 10 previously reported components were not found.

The essential oils of two varieties of lilies were analyzed by Okazaki et al. (412, 413). The oils contained 2,6,6-trimethyl-2-vinyl-5-ketotetrahydropyran, whose stereochemistry was established, as well as linalool and oxidation derivatives, phenols, and many other compounds.

The changes in lime oil resulting from ecological conditions was studied by Rovesti (465) for oils from fruit grown at various altitudes in Ethiopia. Fanghaenel et al. (144) identified many coumarins in the essential oil from Cuban lime. This oil was also investigated by Tapanes et al. (535)

who analyzed its terpene fraction and reported a higher than usual content of γ -terpinene and terpinolene, and Perez Zavas (425) described its distillation from centrifuged oil and the properties of the resulting product. Ames et al. (16) described lime oils produced in Gambia and identified citral a and b, and the physicochemical properties of a lime oil produced in India were ascertained by Hlaing et al. (222).

Oils distilled from *Lippia adoensis* and *L. schimperi* were analyzed by Rovesti (463). Linalool, its acetate, and *d*-carvone were identified among the chief components.

Many sesquiterpene hydrocarbons were isolated and identified in oil of liverwort by Matsuo et al. (358).

The major components of oil of mace, including six aromatic ethers, were identified by Forrest et al. (150).

Oil of *Magnolia obovata* was shown by Fujita et al. (156, 157) to contain as much as 87% eudesmol. The structure of honokiol, an isomer of magnonol, was established. Fujita and Fujita (158) found high percentages of methyl chavicol, safrole, and methyleugenol in oil of *M. salicifolia*.

Kovats (293) conducted an analysis of the oil of mandarin, identifying its components after gas chromatographic separation. Kutateladze and Tsanova (317) determined the amino acid content of mandarin fruits and the fruits and leaves of related hybrids.

The volatiles in the headspace air and in the essential oil of marijuana were identified by Hood et al. (230).

The essential oil of marjoram experimentally grown in the Drome was assayed by Lamy (323), and Salehian and Netien (479) compared the chemical composition of marjoram oils from Hungary, Egypt, the Drome, and the Provence. The seasonal variation in the oils produced in Egypt was studied by Abou-Zied (2), and Marczal and Vermes Vincze (347) compared the composition of marjoram oil with oil of origanum.

The oil composition of *Mentha aquatica*-*M. longifolia* hybrids and *M. dumetorum* was compared by Murray and Lincoln (384) with that of the parent plants. Very notable quantitative differences were observed.

Among the oils from various *Mentha* species analyzed by Chladek et al. (99), the highest alcohol content was observed in oils from *Mentha arvensis* and *M. piperascens* from Brazil. Belafi-Rethy et al. (51) separated 34 components and identified 21 representing 99.3% of the oil from *M. arvensis*. Chobanu (100) determined the composition of dementholized oil. Sacco and Nano (470) ascertained the quantitative makeup of two varieties of *M. arvensis* oils which were entirely different from the normal, and Gill et al. (174) similarly found that great variations existed in the composition of the essential oils from *M. arvensis* grown in North America. Four distinct chemotypes were observed, but could not be correlated with any morphological variations. The growing of *M. arvensis* in India and the physicochemical properties of the oil obtained was related by Gulati et al. (196). Sakata and Hashizume (477, 478) isolated and characterized (+)- β -methyladipic acid, (-)-isopulegol, dehydroxymenthofurolactone, and 2,3-dimethyl-4-hydroxy-2-nonenic acid lactone. Virmani and Datta (571) tabulated properties of oil of *M. arvensis* related to geographic sources. Ubertain et al. (565) studied the changes occurring in mint oil with controlled air oxidation.

A new bicyclic monoterpene was isolated from oil of *Mentha cardiaca* and characterized as 1-vinyl-5,5-dimethyl[2.1.1]bicyclohexane by Hogg and Lawrence (227).

Virmani et al. (572) analyzed oil of *Mentha citrata*.

A hybrid of *Mentha incana* was reported by Mustyats (385) to give a good yield of an oil rich in menthol. It appears commercially promising.

The absolute configuration of 1,2-epoxymethyl acetate isolated from a new strain of *Mentha rotundifolia* was deduced by Shibata and Shimizu (501).

A hybrid of *Mentha sachalinensis* was shown by Yakubovich (594) to yield an oil containing acetates of menthol and its isomers.

Mentha sylvestris and *M. longifolia* were noted by Ku-brak et al. (311) for having a high percentage of linalool.

The oil from a hybrid of *Mentha viridis* grown in Brazil was analyzed by Nano et al. (387), and Sacco et al. (471) determined the composition of a new variety of *M. viridis*, which contained about 75% piperitone.

The main constituent, among 18 identified in oil of *Monarda fistulosa* by Heinrich (209), was thymol.

Muhuhu oil was distilled and investigated by Klein and Schmidt (285) who reported its physicochemical properties and found the main oxygenated component to be brachyl oxide.

Numerous volatile alcohols and esters were reported for the first time in the essence of muskmelon by Kemp et al. (273). To separate the volatiles, they employed a water-recycling apparatus.

The aroma components of raw and cooked mushroom were obtained by simultaneous distillation-extraction in a Likens-Nickerson apparatus. Picardi and Issenberg (432) then identified many compounds including octanol, and also 1-octen-3-one which was formed with cooking. Thomas (545) identified 70 components in mushroom extract, including 9 pyrazines and seven 2-formylpyrroles.

The composition and analysis of oil of mustard was reviewed by Shankaranarayana et al. (495).

An essential oil was distilled from *Myrcianthes callicoma* and its major components were identified by De la Torre and Retamar (116).

Considerable variation in the ratio of sesquiterpenes in myrtle oil was found by Scora (490), and was related to variation in the leaves.

Nigam and Nigam (397) ascertained the quantitative composition of oil of *Nepata hindostana*. Geranyl acetate was present at over 40 percent.

Of the 28 principal components in *Nicotiana alata* oil, Chang and Collins (90) identified 11, including eugenol and heptanoic acid.

Studying the biogenesis of oil of *Ocimum gratissimum*, Dro and Hefendehl (131) found that at first methyl eugenol predominated but, with maturing of the plants, thymol became the major component.

Seventy-seven compounds were identified in the volatile aroma of olive oil by Flath et al. (149).

Studies of the composition and flavor quality of Valencia orange essence oil were conducted by Shaw and Coleman (497, 498). Among many other components, diacetyl was identified. Braddock and Petrus (72) devised a new chemical test involving the measurement of the concentration of malonaldehyde for evaluating the oxidative deterioration of orange essence oil. Moshonas and Shaw (377) identified 7 new orange essence components, and Bielig et al. (61) related the bitter taste developing in canned orange juice to the formation of nootkatone.

Fischer (148) identified several compounds found in limonene from Florida orange oil, including α - and β -ionone. Di Giacomo and De Leo (129) compared Spanish and Italian orange oils by the USP UV spectrophotometric analysis technique, and Lamonica et al. (321) identified many fatty acids in orange oil by chromatography of their methyl esters.

The physicochemical properties and the monoterpene

composition of bitter orange oil produced in Cuba was ascertained by Soular and Fanghaenel (514).

The content of orris oil in roots grown in Moldavia was determined over the season by Pchelintsev and Robel (422), and Nikolaev et al. (400) determined that it contained 25% irone.

The existence in palmarosa oil of nerolidone, α - and β -betulenol, humulene, and compounds related to β -caryophyllene was demonstrated by Naves (391).

Alimukhamedov et al. (13) found allyl-tetramethoxybenzene, myristicin, and 58% apiole in oil from parsley seed grown in Uzbek SSR.

The volatile constituents of passion fruit were analyzed by Murray et al. (383) and by Thomas (418), both of whom identified many components and discussed their importance to the aroma. Winter and Kloeti (591) analyzed the aroma of yellow passion fruit and identified 165 compounds, 161 of which were not previously found in passion fruit, and some of which had not previously been reported in natural flavors.

The chemistry of patchouli oil was elucidated by Teisseire (540), and Teisseire and Maupetit (541), who identified 18 additional components, not previously reported, including a norsesquiterpene alcohol, nordehydropatchoulol, which is largely responsible for the characteristic odor of the oil. Polyakov et al. (435) reported that an oil obtained from patchouli by CO₂ extraction was essentially the same in composition as distilled oil, but that the yield was 2.5% as compared to 0.15–0.38% by distillation.

The quantitative composition of the volatile oil from the rhizomes of *Pavonia odorata* was reported by Dube and Purohit (132).

Pennyroyal oil, aged for nearly three years, was subjected to gas chromatography and IR spectrometry by Fujita and Fujita (159). Many components were identified and seven of them were shown to be oxidation products.

Lamong and Sarawak black pepper could be differentiated by emission spectroscopy as conducted by Kahan and Stahl (249). De Cleyn and Verzele (114) described thin-layer and high pressure liquid chromatography techniques for detecting piperine and its isomers. Gupta et al. (199) identified methyl piperate in an extract from Javanese long pepper.

A total of 85 constituents were quantitatively identified in Oregon peppermint oil by Lawrence et al. (326). Forty-two of these had not previously been reported. Special attention was given to the characterization of trace components. Vlahov and Ognyanov (574) determined the physicochemical properties of Bulgarian peppermint oil, and reported the identification of many components including many sesquiterpenes. Five compounds were reported for the first time. Peppermint oil from the Mitcham type plant cultivated in Piedmint contained viridiflorol. According to Nano et al. (386), this component sweetens the oil's character without changing its taste. Oil from a Sakhalin hybrid of peppermint was found to contain 80% menthol as reported by Ivashenko (244). Hefendehl and Murray (208) gave a detailed analysis of a rather rare limonene-cineole chemotype of *Mentha piperita*. The physicochemical properties of peppermint oils from Shkoder and Korce were tabulated by Gliozheni (176), and Belafi-Rethy et al. (52) determined the main composition of Hungarian peppermint oil. Briggs (77) investigated the effect on the oil when peppermint plants were sprayed with polybutene emulsions, and concluded that menthofuran was reduced and the oils were less desirable. Sviderskaya (525, 526) studied the variation in the alcohols of peppermint oil with maturity of the plants, and described a modified Matthias method

for determining menthol, menthone, and esters. Akimov and Kashchenko (10) reported the optimum purification of peppermint oil with a thin-film evaporator. Shaftan et al. (494) compared three methods for determining menthol and found that all gave identical results except in intensely colored products.

Thirteen components were identified and the physicochemical properties obtained of an oil from *Petasites georgicus* by Toropkina et al. (555).

The hydrocarbons in *Peucedanum palustre* oil consisted mostly of limonene, according to Kozhin et al. (298).

Ashurst et al. (27) found that oil content increased during storage of undried pimenta berries.

A study of pineapple flavor by experts from 5 countries, resulting in a list of 78 components and including determinations of allyl hexanoate, was reported by Chaveron (94). Dupaigne (137) also reviewed the composition of pineapple volatiles.

The composition of the leaf oil of *Pinus pinaster* was established by Pauly et al. (421). Bambagiotti et al. (39) studied the terpene and sesquiterpene components of *P. pinea* oil, and Hannus and Pensar (202) identified 71 terpenes in pine needle oil. Joye et al. (246) compared the composition of the needle oils from five different pine species, and Lishtvanova and Akimov (333) similarly compared the needle oils from five pine species from the Crimea. The volatile components from the bark of *Pinus sylvestris* were identified by Norin and Winell (408), and Rudakov and Poltavchenko (466) proposed a theory for the genesis of some terpenes in oil of *P. sylvestris*.

The physicochemical properties and main constituents of oil of *Pogestemon plectantoides* were ascertained by Nigam and Ramaiah (398, 399), who also isolated two unidentified aldehydes and one alcohol.

Many unusual carbonyl components were identified in the volatile compounds from potato chips by Buttery (80), using gas chromatography-mass spectrometry. Buttery et al. (81) also characterized 42 compounds, mostly pyrazines and aliphatic aldehydes, from baked potato aroma. Among the most significant to the aroma were 2-ethyl-3,6-dimethylpyrazine, methional, and deca-*trans,trans*-2,4-dienal. Buttery and Ling (82) characterized 2-methoxy-3-isopropylpyrazine in the volatile oil of potatoes, and believed that this compound is a major contributor to the earthy aroma.

The mono- and sesquiterpene composition of the oils from five species of prangos was determined and compared by Kuznetsova et al. (318).

Volatile components from the bark of spruce, *Picea abies*, were isolated and identified by Norin and Winell (407).

Over 18 components, including irisolidone and *p*-cumaric acid, were identified in the steam distillate from *Pueraria thunbergiana* flowers by Kurihara and Kikuchi (316).

Belova (53) analyzed two rhododendron essential oils and identified over twenty components.

The preparation and composition of Bulgarian rose oil was discussed by Fuehrer (152). Kapetanovic (258–261) proposed an improved method based on distillation and adsorption for the estimation of oil content in rose flowers. Oils distilled and extracted from *Rosa centifolia* were obtained in good yield and were similar to Bulgarian rose oil. Physical and chemical data of a rose oil obtained from a new source, *R. eglanteria*, were reported by Krzaczek and Chybowski (308). Bakuradze (35, 36) studied the composition of the stearoptenes from Georgian rose oil as well as the constituents of rose oils from various regions of Georgia. Taha and Gomaa (528) showed that the composition of Japanese rose oil is similar to that of Bulgarian. Specifica-

tions were established for Turkish rose oil by Dagecioglu (110), including limits for the elaeoptene and stearoptene fractions. Shlyapnikov and Shlyapnikova (505) examined the effect of different solvents on the yield and composition of rose oil. Peichev-Totev and Dimitrova-Tsaneva (423) found that application of ultrasound to the extraction of oil from rose speeded the process and did not materially change the resulting product. The diffusion coefficients of components of rose extractives were ascertained by Shlyapnikov et al. (503, 504). They also experimented with the extraction of the distillation waters and obtained with CH_2Cl_2 an oil which was very similar to the original oil. Georgiev et al. (172) tested various techniques for the liberation of additional oil from distilled rose petals. Kuppenov et al. (315) used fungal strains to decompose the terpene glycosides and obtained additional rose oil.

Oil of Spanish rosemary was analyzed by gas chromatography by Cabo Torres et al. (83). The results agreed with previous findings. Damjanic and Grzunov (111) obtained a terpeneless rosemary oil by direct absorption from the leaves on silica for 12 hours at 90 °C. The effect of various techniques of distillation and cohobation on the yield and composition of rosemary oil was investigated by Granger et al. (188, 189), as was also the variation in composition of oils from different areas. Hussain (237) found that rosemary oil from Pakistan was quantitatively of a similar composition to oil from Spain and other areas.

The composition, particularly in respect to coumarins, of oil of rue from Moldavia was ascertained by Andon et al. (22). Esteves Reyes and Gonzales Gonzales (142) described new methods for the paper chromatographic determination of coumarins in oil of rue. Kubeczka (310) analyzed the oils from four *Ruta* species and identified several new components in the oil.

The essential oil of saffron was distilled and analyzed by Akhmedov et al. (7, 8). Twenty-seven components were identified, including 26.5% *n*-nonyl alcohol, 5.5% safranal, and 40.5% bornyl acetate.

Bulgarian sage clary oil was investigated by Chorbadzhiev et al. (101). By the use of fractional distillation, paper and gas chromatography, many of its components were identified. Shevchenko and Tikhomirova (500) reported some main components found in oil of sage clary from plants grown in Crimea.

The isolation from oil sandalwood East Indian of *exonorbicycloekasantalal*, and the determination of its chemical structure was accomplished by Gibson and Barneis (173).

Ten commonly available samples of summer savory oil were examined for purity by Herisset et al. (217). Only 2 samples were entirely pure. Tavberidze and Tavberidze et al. (537-539) examined the oil content of spike-bearing savory at different growth stages and with storage, and also analyzed the oils.

Terhune et al. (542) isolated from *Schinus molle* oil a new sesquiterpene hydrocarbon, β -spatulene, and established its stereochemistry.

The oil of *Schizonepeta tenuifolia* was analyzed by Fujita and Fujita (161), who identified many of its components.

The volatile flavor components in aqueous smoke condensates from various woods were identified and compared by Fujimaki et al. (154). They found that condensates from oak and bamboo gave the most acceptable smoke flavor. Kim et al. (279) identified 98 constituents from smoke condensates, of which 31 had not previously been reported. The most important flavor constituents, aside from the phenolic compounds, seemed to be carbonyls and lactones with higher boiling points. Kornreich and Issenberg (289)

characterized the phenolic compounds from wood smoke condensates, and Rijk and Van Battum (454) developed a fast method for determination of benzo[*a*]pyrene in smoke aroma.

A variety of spearmint oil was distilled from *Mentha gentilis nm. hirtella* by Von Schantz et al. (577). The oil contained 36% carvone, and 28 additional compounds were identified. Lawrence et al. (327) analyzed the oils from 4 *Mentha* hybrids from Canada and reported their carvone content as well as terpenes and other constituents. The isolation and structure determination of 6-hydroxycarvone from spearmint oil was accomplished by Tsuneya et al. (561).

In 4 different strawberry essential oils, Vereshchagin (569) found 78 compounds and discussed the influence of some of them on the aroma of the fruit. Soboleva et al. (509) correlated odor evaluation with gas chromatography of strawberry volatile compounds and identified 21 of 66 peaks observed.

The stability of the oil from *Tagetes minuta* was tested by Pruidze et al. (442) in contact with salt, acids, and storage, and under various conditions. Only in alcoholic solution was the oil very stable under all conditions.

A qualitative and quantitative analysis of tangerine oil was reported by Moshonas and Shaw (376), and 17 major constituents were identified, including carbonyl components which had not previously been systematically investigated. Coleman and Shaw (105) compared the composition of tangerine essence oil with that of peel oil. Gas chromatographic analysis of the 20 main constituents showed the two oils to be similar in composition.

Gas chromatographic analysis of oil tarragon by Zarhami and Russell (606) revealed aromatic compounds such as methyl chavicol, methyl eugenol, eugenol, and others.

Forty-seven compounds were identified, 11 of them for the first time, in the essential oil from Georgian black tea by Kozhin and Treiger (299). Saijo (473, 474) compared the volatile components of several black teas, aged, fresh, and a type manufactured from mature leaves, as well as Indian, Ceylon, and Japanese black teas.

He also reviewed the formation of aldehydes in black tea and the biogenesis of terpenes. Ceylon black tea aroma constituents obtained by steam distillation and by adsorption on charcoal, were evaluated by Wickremasinghe et al. (586). Yamanishi et al. (595-597) identified methyl jasmonate and lactones, including jasmine lactone, among Ceylon tea volatiles. They also identified a total of 57 compounds in Ceylon tea aroma and determined the approximate composition of its topnote. A similar analysis wherein 66 compounds including 21 pyrazines were identified, was conducted on roasted green tea aroma. Thin-layer chromatography was employed by Tirimanna (550) to study the composition of the essential oil of black tea. Among the many compounds identified was indole. Kozhin et al. (300) employed mathematical analysis of quantitative gas chromatography to determine the effect of various components on the total tea aroma. The organic acids in tea essential oils were ascertained by Tkeshelashvili and Gogiya (551). Carbonyl compounds in black tea essential oil were quantitatively identified before and after heat treatment by Bokuchava et al. (68). The identity and quantity of phenols in green tea and processed tea were investigated by Goliya and Tkeshelashvili (180). In the high-boiling components of tea oil, Ina and Eto (239) identified several unusual components. Takano and Ikeda (531) isolated and characterized a component from Japanese red tea which imparted a peach flavor when added to foods or beverages.

Fujita et al. (166) differentiated among 3 species of *Thea senensis* by the composition of their essential oils.

Various thyme oils were differentiated by Herisset et al. (214, 215) according to UV, IR, and Raman spectra of their essential oils. In other experiments, a similar differentiation was accomplished by two techniques of gas chromatography. Thyme oils from the Catalonia region were classified by gas chromatography into chemical types by Adzet Porredon (6). The physicochemical properties and the main components of thyme oil from plants growing in Albania were examined by Asllani (28), who compared the oils to those from other countries. Murav'ev and Nazarov (382) estimated the oil content in thyme, based upon a determination of thymol and carvacrol. The oil from a different variety of thyme, *Thymus quinquecostatus*, was obtained with steam distillation by Kameoka et al. (252), who also identified its components.

The preparation of essential oil of tobacco and its separation into basic, acidic, acid hydrolyzable, phenolic, and neutral fractions was described by Richter (452). Roberts and Rohde (456) isolated and identified 105 compounds in the oil of burley tobacco. Fifty-six of these were not previously reported in tobacco, and 11 were new compounds. Takahara et al. (529) characterized several novel components from the steam volatile carbonyl compounds in the neutral fraction of the methanol extract of a yellow leaf tobacco. The compounds had the sweet aroma characteristic of the tobacco. Aasen et al. (1) determined the structure of five isomeric megastigmatrienones isolated from tobacco. Austin et al. (30) evaluated two methods for the collection of tobacco head space volatiles. Demole et al. (118) isolated and characterized two new flavor compounds from burley tobacco, solanofuran and spiroxabovolide. Kimland et al. (280, 281) isolated and identified 100 volatile acids from sun-cured Greek tobacco, and about 50 compounds from the volatile, neutral constituents.

A number of important volatile constituents of tomato were isolated and identified by Seck and Crouzet (491). Guadagni et al. (192) evaluated some components of fresh tomatoes and their mixtures for their contribution to fresh tomato aroma, the only one which was significantly useful was *cis*-hex-3-enal.

Thin-layer and gas chromatography was employed by Stepanenko et al. (519) to elucidate the composition of the oils from four Umbelliferae plant species.

The chemical composition of *Valeriana collina* essential oil was determined from detailed photometric and electron-microscopic studies by Sarkany et al. (481). Sesquifenchene was isolated from oil valerian root and characterized by Paknikar and Kirtany (415).

Garnero (170) discussed the composition of vetiver oil. Hanayama et al. (201) established the stereochemistry of zizanoic acid and related constituents in vetiver oil. Kaiser and Naegeli (250) described a number of new components and proposed a biogenetic scheme for the formation of (+)- α -cedrene and (+)-zizarene. Klein (284) revised the structure of vetivenic acid. Maurer et al. (360) established and confirmed the stereochemistry of C-12 ketones in oil of *Vetiveria zizanioides*. Fuehrer (151) described some components of *V. zizanioides* and suggested synthetic substitutes for vetiver oil.

Several volatile monosulfides were detected in the hydrolyzate from *Wasabia japonica*, black mustard, and horseradish by Kojima et al. (286, 287), as well as numerous other components including isothiocyanates.

The physicochemical properties of oils of *Wisteria floribunda* and *Cytisus scoparius* were reported by Mitsuhashi et al. (370).

A quantitative gas chromatographic determination of ascaridol in oil of wormwood was conducted by Rochat et al. (460), and compared with the iodometric determination. Akhmedov et al. (9) characterized a new lactone, arabsin, isolated from wormwood oil. Berezovskaya et al. (57) increased the yield of azulene from Roman wormwood by alkali treatment before distillation of the oil, and Mashanov (354) found that lemon wormwood oil had a high content of citral and geraniol.

The quantitative composition of oil of *Xanthoxylum acanthopodium* was given by Sen et al. (492). It contained 60% linalool.

The sesquiterpenes of Bulgarian zdravetz oil were identified by Tsankova and Ognyanov (560).

Some chemical characteristics of oil of zeodary, as well as several unidentified ketones, were described by Haque and Rashid (204).

Aromatic Chemicals-General. The analysis and biogenesis of acyclic and cyclic monoterpenoids were described by Thomas (546, 547). Mathieson (356) discussed various methods for structure elucidation of mono-, sesqui-, and di-terpenes. Widen (587) characterized the carbon skeletons of unknown essential oil components by vapor-phase reduction and dehydrogenation followed by gas chromatographic identification. The biogenesis, structure determination, and stereochemistry of acyclic and cyclic sesquiterpenoids were discussed by Roberts (457, 458).

Acids. Acids extracted from fruits were passed through an ion exchange resin to eliminate interfering compounds, then esterified and analyzed by gas chromatography by Yamashita et al. (598). A rapid methylation of microamounts of nonvolatile acids was devised by Levitt (331).

Natural and synthetic acetic acids were distinguished by their content of ^{14}C by Kaneko et al. (256).

Micro quantities of iron were determined in benzoic acid by Tikhonov and Popova (549), using a photometric method.

Cinnamic acid was determined by an oxidation method with aqueous iodine catalyzed with mercuric salts by Kharat and Bose (276).

A specific color reaction was employed by Lang and Lang (324) for the direct identification of formic acid.

Kwapniewski and Szota (319) separated maleic and fumaric acids by thin-layer chromatography, using a mixture of toluenes.

The absorption and emission spectroscopy of pyruvic acid and its esters were investigated by Arnett et al. (25).

Gushchina and Buloshnikova (200) developed a photometric procedure for the determination of iron in sebacic acid.

The organic acids in vanilla were analyzed by Schoen (488), using the AOAC method. He devised an improved method for interpreting the data obtained.

Aldehydes and Ketones. A titrimetric method for the determination of aromatic aldehydes by oximation in an ethanolic 0.05N piperidine solution was proposed by Gal'pern et al. (167). Morishita and Kojima (374) developed a laser-pyrolysis-gas chromatographic method for the characterization of carbonyl compounds through semi-carbazones. Yamazaki et al. (599) separated 2,4-dinitro-phenylhydrazones of aromatic aldehydes by partition two-dimensional, thin-layer chromatography. The structures and configurations of oximes and lactams were determined with NMR and IR spectroscopy by Zabza (605).

The structure of albanone was established by Lansbury and Boden (325).

The absolute configuration of (-)-campherone, (+)-epicampherone, and related compounds were established

by Hodgson et al. (223), using chemical reaction procedures.

Piatkowski and Siemieniuk (431) investigated the stereochemistry of carvone epoxide.

A colorimetric assay of citral and citronellal in volatile oils was illustrated by Karawya et al. (264).

The absolute configuration of the sesquiterpene (-)-cryptomerin was elucidated by Hodgson et al. (224).

Elgenone was analyzed by gas chromatography by Min'kovskii (367).

5-Hydroxymethyl-2-furaldehyde was determined in honey by several different methods, including colorimetric and spectrophotometric techniques, by Dhar and Roy (120).

Mehta and Kapor (362) established the configuration at C-3 in isolongifolene ketones.

trans-2-Nonenal was determined in coffee by Parliment et al. (419), who discussed its importance to coffee aroma.

On the basis of its NMR spectrum, the structure of a nootakatone derivative was delineated by Leitereg (329).

A rapid oxime determination of salicylaldehyde, in which salicylic acid and phenol do not interfere, was proposed by Vulterin and Draha (579).

A study of different chromatographic techniques for the determination of vanillin and ethylvanillin was conducted by Martin et al. (351), to determine which of these techniques provides the best resolution and highest efficiency.

Alcohols and Phenols. The parameters affecting the separation and quantitation in gas chromatography of geraniol and other terpene alcohols and their trimethylsilyl ethers and acetates were investigated by Watts and Kekwick (584). Gueldner et al. (193) characterized the carbamates of terpene and straight-chain alkyl alcohols by IR and NMR spectrometry, and separated them by gas and thin-layer chromatography. R_f values were given for many terpene alcohols. Lin et al. (332) conducted NMR studies of several sesquiterpene alcohols and their oxidation products. Rik and Kravchenko (455) reported the mass spectra of geraniol, linalool, citronellol, α - and 4-terpineol, and methol. Infrared absorption spectrophotometry was employed by Gorska and Gluzinska (184) for the direct determination of phenols.

Agerol, obtained from the flowers of *Achillea ageratum*, was investigated by Grandi et al. (186), who determined its absolute configuration.

Bakuchiol, a novel monoterpene phenol, was isolated and characterized by Mehta et al. (363). The structure of the same alcohol was also studied by Rao et al. (448).

Gulyaeva and Blokh (198) devised a determination of microquantities of sulfur in butyl alcohol.

The stereochemistry of cubebol was elucidated by Tanaka et al. (534).

An atomic-absorption determination of silver in ethyl alcohol was conducted by Mikhel'son and Ivanov (365).

Several isomers of eugenol and isoeugenol were separated with gas chromatography by Rodul'fi et al. (467). UV and IR spectra were given.

Geraniol was determined by gas chromatography in oil of citronella, as described in the *Analyst* (18).

The stereochemistry of gymnomitrol, isolated from *Gymnomitrium obtusum*, was deduced by Connolly et al. (106).

The ring-chain conformational preference was determined for 7-*cis*- β -ionol based on NMR studies by Ramamurthy et al. (446).

Banthrope et al. (40) established the stereochemistry of isolongifolene alcohols.

Linalool was assayed with IR spectroscopy by Grushchanskii and Borona (191).

The relative and absolute configurations of menthol and neomenthol were illustrated by Barry (42), and Kheifits et al. (277) determined traces of thymol in menthol.

The structure of myliol, tetracyclic sesquiterpene alcohol isolated from *Mylia taylorii* was confirmed by Benesova et al. (55) with NMR spectra and chemical studies.

Narchinol A was characterized by Hikino et al. (218).

Suga et al. (524) supplied further proof of a revised structure for (+)-occidentalol.

Paradisiol was shown by Huffman and Zalkow (234) to be identical to intermediol.

The structure of patchouli alcohol was elucidated through a stereospecific synthesis by Mirrington and Schmalzl (368).

A novel sesquiterpene alcohol from *Picea polita* was characterized by Sayama and Kyogoku (483).

The shifts of methyl proton resonance of pinanols and related compounds induced by benzene were found by Hirata (219) to be useful in establishing both the location and stereochemistry of the methyl protons near the OH or AcO function.

The similarity of the structure and fragrance of three terpene cyclohexanols to those of santalols was discussed by Aul'chenko and Kheifits (29).

Sylveterpins were investigated by Abraham and Verghese (4), and their structures were established.

Thymol was determined in micro quantities by oxidative coupling with *p*-phenylenediamine by Taha and Gomaa (527). Alpanse de Morais et al. (15) found high thymol contents in the oils from two Amazonian plants.

Esters and Lactones. The structure of archangelolide was determined from NMR spectra and chemical correlation by Holub and Samek (229).

Arteannuin B was isolated and characterized by Jeremic et al. (245).

The characterization of chlorohyssopifolin A and B, two new sesquiterpene lactones, was accomplished from their IR, NMR, and mass spectra by Gonzales Gonzales et al. (182).

A two-column gas chromatographic assay for coumarin and dihydrocoumarin was presented by Hoffmann (226). Haskins et al. (205) determined coumarin in deer's tongue, Kroeller (306) determined the coumarin content in cigarette smoke, and Valutskaya et al. (567) checked the coumarin contents of the oils from various Siberian Umbelliferae. Hoque and Dutta (231) obtained the retention times for 22 natural coumarins.

Dialkyl phthalates were assayed in cosmetic preparations using an improved gas chromatography method by Godly and Mortlock (177).

Methyl acrylate reactants were quantitatively analyzed in process by an NMR method developed by Kulkarni and Pansare (314).

Ethers, Oxides, and Peroxides. Dipole moments and Kerr constants confirmed the *trans* structure of chrysanthenone oxide as shown by Arbuzov et al. (24).

A gas chromatographic method for the assay of 1,8-cineole in essential oils, proposed by the "Essential Oils Subcommittee, Society, for Analytical Chemistry" in England was published in the *Analyst* (17). Gandini et al. (168) determined the stereochemistry of 1,8-cineole derivatives.

Two new furanosesquiterpenes were isolated and described by Hayashi et al. (206).

Terpenes and Hydrocarbons. Better separation of terpene olefins on silica gel thin-layers impregnated with AgClO_4 was obtained by Prasad et al. (439). Sesquiterpene hydrocarbons in *Bazzania trilobata* were identified by Andersen and Huneck (19). Bohlmann (63) discussed the biogenesis and structure of natural acetylenes.

Reconsideration of the physical and chemical properties of (-)- γ -amorphene by Briggs and White (78) indicated that it is in fact identical with (-)- γ -muurolene.

The configurations of bicyclogermacrene and isobicyclogermacrene were established by Nishimura et al. (405).

Questions of confirmation in the transyclononene ring of caryophyllene were considered by Warnhoff and Srinivasan (582). β -Caryophyllene and 5-kaurane were isolated by Bohlmann and Rao (64) and their structures established.

The conformation of humulene was studied by Cradwick et al. (108).

Compounds of the *o*-menthene series were investigated by Bazyl'chik et al. (49).

The stereochemistry and circular dichroism of several pinane derivatives were investigated by Hirata (220). Naumov and Bezzubov (389) conducted electron diffraction studies of the structure of β -pinene.

(+)- α -Selinene was shown by Andersen et al. (20) to be an enantiomeric eudesmane.

The stereochemistry of seychellene was established by a total synthesis by Mirrington and Schmalzl (369).

The chemistry and structure of thujopsene and humulene were investigated by Dauben (113).

Zonarene and its epimer were identified in 9 essential oils by Andersen et al. (21), and their absolute configurations were established. Iguchi et al. (238) confirmed the stereochemistry of zonarene by synthesis.

Miscellaneous. The chemical composition of different varieties of ambergris and its relation to quality was investigated by Korzh and Strigina (290).

Capsaicin and allied compounds were isolated and identified by Awasthi and Singh (32). Trejo-Gonzales and Wold-Altamirano (557) evolved a new UV absorption method for the determination of capsaicin.

When L-cysteine or L-cystine were reacted with carbonyl compounds, Kato et al. (271) found that many sulfur-containing compounds, such as thiazoles and thiophenes, which are naturally found in foods, resulted.

Dimethyl sulfide was identified in the volatiles from asparagus by Ney and Freytag (394).

Halogens in organic substances were determined by Volodina et al. (575), using electric glow discharge.

The detection of musk ambrette with a gas chromatographic method was described by Karasek and Keller (262).

The volatile components of protein hydrolyzates from soybeans were analyzed by Markh and Vinnikova (348).

The formation of pyrazines from thermal treatments of some amino-hydroxy compounds were studied by Wang and Odell (581).

The assay of sulfides and disulfides with phenyl-iodosodiacetate was described by Verman and Bose (570).

The mass spectrometry of organic sulfur compounds and their fragmentation mechanism were studied by Nishimura et al. (404).

2,4,6-Trichloroanisole was identified in several essential oils by Stoffelsma and De Roos (521).

Synthetic substances were identified in vanilla extracts and essence with thin-layer chromatography and color reactions by Benk and Bielecki (56).

LITERATURE CITED

- (1) Aasen, A. J., Kimland, B., Almqvist, S. O., Enzell, C. R., *Acta Chem. Scand.*, **26**(6), 2573 (1972).
- (2) Abou-Zied, E. N., *Pharmazie*, **28**(1), 55 (1973).
- (3) Abraham, K. O., Shankaranarayana, M. L., Raghavan, B., Sastry, L. V. L., Natarajan, C. P., *Flavour Ind.*, **4**(7), 301 (1973).
- (4) Abraham, P. M., Verghese, J., *Indian J. Chem.*, **10**(2), 140 (1972).
- (5) Adzet, D. T., *Rev. Real Acad. Farm. Barcelona*, **1972**(5), 17; *Chem. Abstr.*, **79**, 9752k (1973).
- (6) Adzet Porredon, T., *ibid.*, **3**; (6) 29; *Chem. Abstr.*, **79**, 35041x (1973).
- (7) Akhmedov, A. I., Chogovadze, Sh. K., Goryaev, M. I., Dembitskii, A. D., *Maslo-Zhir. Prom.*, **1973**(1), 26; *Chem. Abstr.*, **79**, 83384h (1973).
- (8) Akhmedov, A. I., Goryaev, M. I., Chogovadze, Sh. K., Dembitskii, A. D., *Izv. Akad. Nauk Kaz. SSR, Ser. Khim.*, **22**(5), 56 (1972); *Chem. Abstr.*, **78**, 33775v (1973).
- (9) Akhmedov, I. S., Kasymov, Sh. Z., Sidiyakin, G. P., *Khim. Prir. Soedin.*, **1972**(2), 245; *Chem. Abstr.*, **77**, 85567c (1972).
- (10) Akimov, Yu. A., Kashchenko, G. F., *Tr. Vses. Nauch.-Issled. Inst. Efirnomaslich. Kul't.*, **3**, 171 (1971); *Chem. Abstr.*, **80**, 30593q (1974).
- (11) Akimov, Yu. A., Kuznetsov, S. I., *Rast. Resur.*, **8**(4), 562 (1972); *Chem. Abstr.*, **78**, 75769b (1973).
- (12) Akisue, G., *Rev. Farm. Bioquim. Univ. Sao Paulo*, **10**(2), 151 (1972); *Chem. Abstr.*, **79**, 57558y (1973).
- (13) Alimukhamedov, S. A., Maksudov, N. A., Goryaev, M. I., Sharipova, F. S., *Khim.-Farm. Zh.*, **6**(9), 15 (1972); *Chem. Abstr.*, **78**, 33787a (1973).
- (14) Alpande de Moraes, A., Andrade da Mata Rezende, C. M., Von Bulow, M. V., Mourao, J. C., Gottlieb, O. R., Marx, M. C., Da Rocha, A. I., Magalhaes, M. T., *Acta Amazonica*, **2**(1), 41 (1972); *Chem. Abstr.*, **80**, 149023u (1974).
- (15) Alpande de Moraes, A., Mourao, J. C., Gottlieb, O. R., Leao da Silva, M., Marx, M. C., Maia, J. G. S., Magalhaes, E. M. T., *ibid.*, **45**; *Chem. Abstr.*, **80**, 124560d (1974).
- (16) Ames, G. R., Matthews, W. S., Thain, E. M., *Mezhdunar. Kongr. Efirnym Maslam, [Mater.]*, **4th 1968**, **1**, 445 (1971); *Chem. Abstr.*, **78**, 128349y (1973).
- (17) *Analyst (London)*, **98**(1169), 616 (1973).
- (18) *ibid.*, (1172) 823.
- (19) Andersen, N. H., Huneck, S., *Phytochemistry*, **12**(7), 1818 (1973).
- (20) Andersen, N. H., Shunk, B., Costin, C. R., *Experientia*, **29**(6), 645 (1973).
- (21) Andersen, N. H., Syrdal, D. D., Lawrence, B. M., Terhune, S. J., Hogg, J. W., *Phytochemistry*, **12**(4), 827 (1973).
- (22) Andon, T. M., Belova, N. V., Denisova, G. A., *Herba Hung.*, **11**(2), 21 (1972); *Chem. Abstr.*, **79**, 123795b (1973).
- (23) Angmor, J. E., Dicks, D. M., Evans, W. C., Santra, D. K., *Planta Med.*, **21**(4), 416 (1972).
- (24) Arbuzov, B. A., Vereshchagin, A. N., Gubkina, N. I., Sadykova, I. M., Vul'fson, S. G., *Izv. Akad. Nauk SSSR, Ser. Khim.*, **1972**(6) 1288; *Chem. Abstr.*, **77**, 101911t (1972).
- (25) Arnett, J. F., Larson, D. B., McGlynn, S. P., *J. Am. Chem. Soc.*, **95**, 7599 (1973).
- (26) Asaturova, V. N., Chkhenkeli, B. N., *Tr. Gruz. Nauch.-Issled. Inst. Pishch. Prom.*, **1971**(5), 191; *Chem. Abstr.*, **78**, 47642m (1973).
- (27) Ashurst, P. R., Lee, D. A., Lewis, O. M., *J. Sci. Res. Council. Jam.*, **3**(1), 50 (1972).
- (28) Asllani, U., *Bul. Shkencave Natyr., Univ. Shtetor Tiranes*, **27**(12), 111 (1973); *Chem. Abstr.*, **80**, 149025w (1974).
- (29) Aul'chenko, I. S., Kheifits, L. A., *Tr. Vses. Nauch.-Issled. Inst. Sin. Natur. Dushist. Veshchestv*, **1971**(9), 115; *Chem. Abstr.*, **78**, 88510k (1973).
- (30) Austin, D. J., Roeraade, J., Kimland, B., Aasen, A. J., Enzell, C. R., *Beitr. Tabakforsch.*, **7**(1), 8 (1973).
- (31) Austerhoff, H., Mombberger, H., *Arch. Pharm. Ber. Deut. Pharm. Ges.*, **305**(6), 455 (1972); *Chem. Abstr.*, **77**, 79470z (1972).
- (32) Awasthi, D. N., Singh, B. P., *Proc. Indian Acad. Sci., Sect. B*, **77**(5), 196 (1973).
- (33) Bakhtadze, T. K., Dembitskii, A. D., *Tr. Gruz. Nauch.-Issled. Inst. Pishch. Prom.*, **1971**(5), 172; *Chem. Abstr.*, **78**, 62047q (1973).
- (34) Bakina, L. A., Senchenko, G. G., Meshcheryuk, G. I., Kozhina, I. S., *Rast. Resur.*, **8**(2), 252 (1972); *Chem. Abstr.*, **77**, 92732f (1972).
- (35) Bakuradze, N. S., *Tr. Sukhum. Opyt. St. Efirnomaslich. Kul't.*, **1971**(10), 194; *Chem. Abstr.*, **78**, 47643n (1973).
- (36) Bakuradze, N. S., Nazarenko, M. V., *Mezhdunar. Kongr. Efirnym Maslam, [Mater.]*, **4th 1968**, **1**, 21 (1971); *Chem. Abstr.*, **79**, 83392j (1973).
- (37) Balbaa, S. I., Hilal, S. H., Haggag, M. Y., *Planta Med.*, **23**(4), 312 (1973).
- (38) Ballarín, C., Ballarín, J., *Pharmazie*, **27**(8), 544 (1972); *Chem. Abstr.*, **78**, 7738r (1973).
- (39) Bambagiotti, A. M., Vincieri, F. F., Coran, S. A., *Riv. Ital. Essenze, Profumi, Piante Off., Aromi, Saponi, Cosmet., Aerosol*, **45**(12), 875 (1972).
- (40) Banthorpe, D. V., Curtis, A. J., Fordham, W. D., *Tetrahedron Lett.*, **1972**(36), 3865.
- (41) Banthorpe, D. V., Davies, H. S., Gattford, C., Williams, S. R., *Planta Med.*, **23**(1), 64 (1973).
- (42) Barry, J., *J. Chem. Educ.*, **50**(4), 292 (1973).
- (43) Barua, A. K. S., Bhagat, S. D., Salkia, B. K., *Analyst (London)*, **98**(1164), 168 (1973).
- (44) Barua, A. K. S., Gupta, N. K., Bhagat, S. D., Mathur, R. K., Gopinath, K. W., Iyengar, M. S., *Flavour Ind.*, **3**(8), 416 (1972).
- (45) Baslas, B. K., Baslas, R. K., *Riechst., Aromen, Koerperpflegem.*, **22**(5), 155, 158 (1972); *Chem. Abstr.*, **77**, 66100d (1972).
- (46) *ibid.*, (6), 200, 202; *Chem. Abstr.*, **77**, 85546v (1972).
- (47) *ibid.*, (8), 270; *Chem. Abstr.*, **77**, 130489s (1972).
- (48) *ibid.*, (12), 421, 424, 426, 428; *Chem. Abstr.*, **78**, 75764w (1973).
- (49) Bazyl'chik, V. V., Bardyshev, I. I., Ryabushkina, N. M., Polyakova, N. P., Fedorov, P. I., *Vestsi Akad. Navuk Belarus. SSR, Ser. Khim. Navuk*, **1973** (6), 104; *Chem. Abstr.*, **80**, 83264q (1974).
- (50) Bedoukian, P. Z., *Cosmet. Parfum.*, **88**(4), 31 (1973).
- (51) Belafi-Rethy, K., Iglewski, S., Kerenyi, E., Kolta, R., *Acta Chim. (Budapest)*, **76**(1), 1 (1973); *Chem. Abstr.*, **80**, 6772b (1974).
- (52) *ibid.*, (2) 167; *Chem. Abstr.*, **80**, 19365w (1974).
- (53) Belova, N. V., *Mezhdunar. Kongr. Efirnym Maslam, [Mater.]*, **4th 1968**, **1**, 31 (1971); *Chem. Abstr.*, **79**, 83386k (1973).
- (54) Belsten, J. C., Bramwell, A. F., Burrell, J. W. K., Michalkiewicz, D. M., *Tetrahedron*, **28**(13), 3439 (1972).
- (55) Benesova, V., Sedmera, P., Herout, V.,

- seki Kagaku, **21**(9), 1223 (1972); *Chem. Abstr.*, **78**, 11275e (1973).
- (600) Yllera Camino A., *Ion(Madrid)*, **32**(370), 273 (1972); *Chem. Abstr.*, **77**, 130484m (1972).
- (601) Yoshihara, K., Hirose, Y., *Phytochemistry*, **12**(2), 468 (1973).
- (602) Younos, Ch., Lorrain, M., Pelt, J. M., *Plant. Med. Phytother.*, **6**(3), 178 (1972); *Chem. Abstr.*, **78**, 33785y (1973).
- (603) *Ibid.*, (4), 251; *Chem. Abstr.*, **78**, 75766y (1973).
- (604) Younos, Ch., Mortier, F., Pelt, J. M., *ibid.*, (3), 171; *Chem. Abstr.*, **78**, 33784x (1973).
- (605) Zabza, A., *Pr. Nauk. Inst. Chem. Org. Fiz. Politech. Wroclaw*, **1971**(3), 1; *Chem. Abstr.*, **78**, 111524u (1973).
- (606) Zarghami, N. S., Russell, G. F., *Chem., Mikrobiol., Technol. Lebensm.*, **2**(6), 184 (1973).
- (607) Zhelev, Z. G., *Mezhdunar. Kongr. Efirnym Maslam, [Mater.]*, 4th 1968, **1**, 97 (1971); *Chem. Abstr.*, **79**, 83394m (1973).
- (608) Zola, A., Garnerio, J., *Parfums, Cosmet., Savons Fr.*, **3**(1), 15 (1973); *Chem. Abstr.*, **78**, 115116v (1973).
- (609) Zweig, G., Sherma, J., "CRC Handbook of Chromatography", CRC Press, Cleveland, Ohio, 1972, Vol. 1, 784 pp., Vol. 2, 343 pp.

Pesticide Residues

Wayne Thornburg

Del Monte Corporation Research Center, Walnut Creek, CA 94598

Publication in the field of pesticide methodology has continued at a high level during the two-year review period from November 1972 through November 1974. The author has attempted to review articles which will be most useful to the residue analyst and, when possible, papers were selected that were published in journals which are readily accessible in major libraries.

This review follows the general format and pesticide nomenclature used in the 1973 biennial review of Thornburg (323).

Frear's "Pesticide Index"(103) lists the common, trade, and chemical names of many pesticides, and the author has tried to use names found therein. Common names that appear in the Environmental Protection Agency tolerance regulations have been used where possible.

Another useful publication edited by Shepard (284) is the "Dictionary of Pesticides 71" which is a compilation of the pesticides available commercially in the United States and throughout the world. Trade names, common names, and chemical names are cross-referenced wherever possible. Names of basic producers are also listed for each pesticide.

"Residue Reviews," under the editorship of Gunther (124), continues to be an excellent source of information on pesticide methodology. A total of 54 volumes has now been published.

Zweig has continued his editorship of "Analytical Methods for Pesticides, Plant Growth Regulators, and Food Additives" with the publication of Vol. VI, "General." This volume contains the cumulative index to volumes I-VI (355). Zweig and Sherma have co-edited Vol. VII of this series, "Thin Layer and Liquid Chromatography and Analysis of Pesticides of International Importance" (356).

The following symposia were published:

ACS Symposium on "Pesticide Identification at the Residue Level," *Advances in Chemistry Series* **104** (3), and an ACS Symposium on "The Fate of Organic Pesticides in the Aquatic Environment," *Advances in Chemistry Series* **111** (4).

The proceedings of the 2nd International Congress on Pesticide Chemicals (317) contains a number of excellent articles on pesticide residue analysis.

Ebing (88) compiled the second volume of a comprehensive

reference book of the GLC pesticide literature of the world in "Gas Chromatography of Plant Protection Agents, Vol. II" covering the period from 1970-72.

Fishbein (100) published a book on the chromatography of pesticides.

Bourke and coworkers (35) described a pesticide residue data information retrieval system.

Safe and Hutzinger (273) described the up-to-date uses of mass spectrometry in "Mass Spectrometry of Pesticides and Pollutants."

This review period has been characterized by improvements and automation of instrumentation with increased reliability.

Many improvements have been made in liquid chromatography, but it still lacks the ease of use and sensitivity that is expected of gas chromatography. However, research is under way to combine separation techniques of liquid chromatography with quantitation by gas chromatography.

Eissenbeiss and Sieper (91) described the use of high-performance liquid chromatography in residue analysis. These authors concluded that high-performance liquid chromatography (HPLC) can be regarded as an alternative or a supplementary method to conventional methods such as GLC.

Seiber (278) studied the reverse-phase liquid chromatographic retentions of 17 pesticides on Vydac reversed-phase in water-methanol and mixed solvents.

Sampling, sample preparation, extraction, and cleanup of the extracts are still a very important part of successful pesticide analysis. A number of solvents and reagents, especially purified for pesticide analysis, are available and their use is highly recommended.

Narayanaswami and coworkers (227) described the use of TLC in the detection of poisoning by pesticides.

Mallet and Surette (208) investigated the fluorescence of pesticides by treatment with acid and base on TLC plates.

Almost every pesticide residue analytical procedure requires at least one concentration step involving partial or complete removal of an organic solvent. Ott and Liebig (241) modified a commercial multi-tube solvent evaporator so that it could be used for the concentration of pesticide residue-containing solutions.

Voss and Blass (332) developed a solvent-saving extraction-evaporation apparatus for residue analysis of pesticides.

Suzuki and coworkers (313, 314) described a systematic separation and identification of pesticides using a combina-

Authors have not been supplied with free reprints for distribution. Extra copies of the review issue may be obtained from Special Issues Sales, ACS, 1155 16th St., N.W., Washington, DC 20036. Remit \$4 for domestic U.S. orders; add \$0.50 for additional postage for foreign destinations.