# REVIEW OF INDUSTRIAL APPLICATIONS OF ANALYSIS, CONTROL, AND INSTRUMENTATION

# ANALYTICAL Chemistry

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REVIEW OF INDUSTRIAL APPLICATIONS

# Air Pollution

## KINGSLEY KAY

Occupational Health Division, Department of National Health and Welfare, Ottawa, Canada

THE present paper covers the period 1952 to the latter part of 1956 and supplements a previous review (224) published in 1952.

Among the significant scientific events of the new review period was the formation of the ACS Committee on Air Pollution, under the Chairmanship of H. F. Johnstone. This committee facilitates interchange of scientific data on the subject. It has organized a number of symposia; the most recent one was held at the 130th meeting of the AMERICAN CHEMICAL SOCIETY in Atlantic City, September 1956. Analytical programs on air pollution have also been presented during recent years by Stanford Research Institute, the Air Pollution Control Association, and the American Industrial Hygiene Association, which in 1955 formed a special committee on air pollution.

Certain publications during the review period warrant reference in this paper. McCabe continued his column in Industrial and Engineering Chemistry and edited a volume of papers from the United States Technical Conference (277). The Manufacturing Chemists' Association of the United States compiled an Air Pollution Abatement Manual (293) containing a section on analysis. Various aspects of the subject have been dealt with in reviews (81, 216, 278, 403, 433, 434). An air pollution bibliography was issued by the U.S. Bureau of Mines (449) in 1954. An encyclopedia of instrumentation in industrial hygiene (488) appeared

in 1956. The second edition of a pamphlet on instruments for studying atmospheric pollution was recently issued by the Committee on Air Pollution Controls of the American Society of Mechanical Engineers (10). Stanford Research Institute has continued to publish proceedings of the National Air Pollution Symposia (412) and has edited a handbook on air pollution (174). The Air Pollution Foundation has described the methods used in an aerometric survey of Los Angeles Basin (362). The annual reports of the Los Angeles County Air Pollution Control District (271) and the International Joint Commission Reference on Pollution in the Windsor-Detroit area (198) have provided substantial information on methods and instrumentation. In 1954, Cholak (78) drew attention to the diversified nature of urban contaminants and to the growing measurement problem in this field.

### STUDY OF AEROSOLS

The general approach to physical analysis of aerosols in the air pollution field continues to center on such characteristics as mass concentration, size distribution, and particulate density for following the course of pollution changes. Increasing use of atomic energy has stimulated widespread investigation of health effects of radioactive pollution and has required new aerosol collection and assessment techniques. Developments in chemical analysis have been limited. The main concern has been in relation to carcinogenic hydrocarbons which have been demonstrated in air-borne particulate matter of a number of cities (235, 398, 464).

Close health scrutiny of urban aerosols began after the Donora, Pa., disaster, when a theory arose that particulates not toxic per se, might act in combination with gaseous pollutants through chemical change, adsorption, or synergism to produce a respiratory load of toxic power (224). Subsequently the London disaster of 1952 occurred with health effects bearing a striking similarity to those in Donora. While no evidence of joint action between gaseous and particulate constituents of the air was revealed, the particulate load was exceptionally high, whereas the gaseous load in terms of sulfur dioxide did not reach extraordinary levels (388). This renewed interest in the theory of joint action and a recent laboratory study by Labelle and coworkers (239) has indicated that under appropriate conditions, augmented toxicity of certain gases can be produced when inhaled in combination with submicron particulates which penetrate to the lung. In this connection Hatch (183) stresses that dust sampling for health evaluation should correspond to the range of sizes reaching the lungs. These health findings have already encouraged aerosol studies, particularly in respect to particle size and interaction distribution with gaseous constituents of the atmosphere.

Downloaded via DALHOUSIE UNIV on September 4, 2018 at 15:08:49 (UTC). See https://pubs.acs.org/sharingguidelines for options on how to legitimately share published articles A recent contribution has been made by Gordieyeff (148). It is reasonable to suppose that the work on techniques of aerosol analysis may be widely exploited as the health position of the aerosols in urban centers becomes more clearly established through biological research.

### AEROSOL COLLECTION TECHNIQUES

Classical methods of aerosol collection have been critically examined by Cadle and Schadt (58), who used nearly monodisperse aerosols to reveal the extent to which theoretical efficiencies were attainable for different particle sizes. A number of significant discrepancies between theoretical and observed efficiencies were found. Sampling for air-borne radioactivity was reviewed in 1955 by Heslep and Bellamy (189) and in 1956 by Burnett and Hatch (54). German instruments and methods have been surveyed by Hasenclever (182). In the whole field of aerosol collection the impressive feature is the general emphasis being placed on theoretical aspects, opening the way to new advances in collector design.

Gravity Settling. This long-established method of collection for estimation of gross pollution load continues to be widely used in urban centers, though meteorological variables and the effect of collector design limit the extent to which cross comparison of data can be made from center to center. Munger (312) has developed an example permitting estimation of particulate fall in relation to wind direction. Pond and Paxton (344) have examined water-containing jars and greased plates for dustfall measurements. Comparison of the two procedures shows that greased plates give more reproducible results and higher dustfall rates. The effect of design differences in collectors has been measured by Newbury (316) and Fisher (127). The latter author has shown that the collector proposed by the Air Pollution Control Association of the United States yields estimations of dustfall as much as 80% higher than the standard British design. Gummed films for collection of settled particulates have been considered by Machta (289).

Filtration. The current range of filtering media has been discussed by Chambers (66, 67). Several comprehensive studies on collection efficiency of these media have been carried out. Harris and coworkers (179) and Avy and Railleres (22) studied filter papers. Smith and Surprenant (409) compared the properties of various filter media using atmospheric dust and dioctyl phthalate. Fitzgerald and Detwiler (128) tested papers with Duralumin sol of diameter 0.005 to 2.0 microns and found high efficiencies above 0.2 micron, with a sharp decrease below this size. In a comprehensive investigation of the efficiency of papers and millipores for lead fume collection, La Torre and Silverman (252) studied effects of loading and filtering velocity on collection efficiency and particle size retention. In these tests electron microscopy was employed. It was found that collection efficiency varied widely over the range of media tested.

High-volume sampling through filter paper or glass fiber cloth has been widely employed for mass concentration and other estimations in urban air pollution surveys. Hall (169) designed a continuous filter paper sampler for assessing the atmospheric particulate load. Correlation between deposit absorbance and visibility was claimed if corrections were made for differences in relative humidity. Clayton and Giever (83) have reviewed an extensive range of automatic filter paper collection devices to provide material for soiling and mass concentration determination by light transmission, absorbance, or color comparison. Katz, Sanderson, and Ferguson (222) and Johnson (205) have assessed the reliability of the optical approach. The effect of wind speed and other aspects have also been critically examined (155, 186, 217).

Molecular filter membranes have been a most important new medium and are now extensively exploited in micro sampling. Goetz (144), who introduced these membranes in America, dealt with a number of their applications. First and Silverman (124) have determined efficiency for quartz dust, sulfuric acid mist, and metal fumes. Mercer (303) used these filters in atmospheric monitoring for alpha emitters. Langer and Fisher (250) employed membranes for weight sampling of cigarette smoke. Most recently, Johnson (205) has found membranes superior to paper in mass concentration estimations by the optical method. Goetz (145, 146) has designed an apparatus in which particulates in liquid suspension are collected on filter membranes according to a graded scale proportional to concentration, thus eliminating conventional mass determination.

A method for removal of dust samples from paper filters has been described by Joos (210). A further trial of soluble filters was reported by Avy (21), who employed a mixture of tri- and tetrachloronaphthalene.

The problem of a critical aerosol size for maximum penetration of filters has been subjected to continuing investigation by LaMer (241), Ramskill and Anderson (351), Davies (94), and Chen (72). No substantial experimental evidence for the existence of a critical size was produced. The theoretical position was summarized by Chen (72) in 1955. More recently, Thomas and Yoder (431, 432) have presented experimental data leading to the conclusion that an aerosol size for maximum penetration of fiberglass, sand, and lead shot exists. Rossano and Silverman (373) have investigated electrostatic effects in fiber filters for aerosols. Inertial impaction in relation to filtration has been examined theoretically and experimentally by Wong, Ranz, and Johnstone (484, 485).

Isokinetic Sampling. The role of isokinetic conditions in ensuring representative sampling of suspensions was observed in the design of stack sampling equipment by Hardie (176) as long ago as 1937. Nevertheless the practical importance of the principle has not been widely recognized in the aerosol field. Some experimental proof of the effect of deviation from isokinetic sampling of aerosols was provided in 1952 by Watson (467). In 1954, Hemeon and Haines (185) dealt with stack sampling errors and suggested a new procedure (167). Isokinetic sampling has been most recently reviewed by Wilcox (476) who has stated that errors become significant for particle sizes around 1 micron and over.

Impingement. Theoretical and experimental studies were made by Ranz and Wong (352, 353) on impaction efficiencies. These authors showed that with a given set of operating conditions for a jet impactor, impaction separation occurs for approximately the product of the square root of particle density times an effective particle diameter. Ammonium chloride and sulfuric acid aerosols were employed in the experimental work. Johnstone, Field, and Tassler (207) have extended basic knowledge of the Venturi atomizer, establishing that in aerosol collection by this technique inertial impaction of the aerosols on droplets is the main mechanism operating. Boucher (38) has succeeded in collecting very small particles with adaptations of the Venturi principle involving, among other considerations, similarity of diameter between the captor particles and those to be captured.

Recent developments in impingement apparatus have been concerned with modifying cascade impactor designs to provide for collection of large particles at velocities sufficiently low to avoid disassembling aggregates, and to provide for improved collection efficiencies at the submicron level. In the latter instance it has been necessary to establish a compromise as between the ideal jet dimension for submicron collection, and the equally important consideration of conformity with over-all flow rate and maintenance of adequate sampling volume. One solution to this problem was produced by Wilcox (475)for the extended particle size range 0.2

to 100 microns in diameter. Five stages of impaction were employed. Samples of several types of heterogeneous clouds were reported to have been size graded effectively. Those having variation in density in addition to particle size were manageable, but size grading was not as effective as for samples of uniform density. In France, Boucher and Lenoble (39) attempted to overcome the lower diameter limit of collection of the May impactor by the adjunction of a Venturi stage containing a plastic grid. These authors claimed that particles smaller than 0.5 micron can be collected in their true state of dispersion.

Another practical problem in impingement is overloading of adhesive collectors. Where the adhesive surface has become loaded, succeeding particles bounce off. To counteract this effect, Davies, Aylward, and Leacey (95) developed a moving slide design. This was employed by Langer and Fisher (250) in studies of cigarette smoke, and was compared with thermal and electrostatic precipitators. It was found to yield collection efficiencies of over 90%. Lately May (301) has described an improved design of moving slide impinger which permits greater ease in changing slides.

It has been long recognized that impingement disassembles aggregates of particles. The effect of this phenomenon on particle counting was shown quantitatively for coal dust by Davies and coworkers (95) who found impingement counts as high as 7 to 10 times those obtained with thermal precipitation. In dust counting for health control it is desirable to eliminate large aggregates which do not in practice reach the lung. May and Druett (302) developed a preimpinger for removal of such aggregates. Another two-stage instrument has been proposed by Hamilton and Walton (173).

The annular impactor of Tait (423)was designed for collection of dusts containing long-lived alpha emitters. This instrument is capable of sampling at high rates, around 700 liters per minute, by using a jet in the form of an annular ribbon. Multiple strokes have been shown to reduce the apparent particle concentration obtained with the Owens jet dust counter (104).

McCully and associates (282) have conducted experiments on colliding water droplets and particles as an aspect of the scavenging action of rain.

Thermal Precipitation. The superior position of the thermal precipitator in sampling for microscopic examinations of solid particulates has remained unchallenged since it was discussed in the previous review (224). The high efficiency of collection for submicron diameters down to Angström dimensions is as yet unmatched

in any other instrument. The modest forces involved provide best possible conditions for collection of aggregates without dispersion, though Sinclair (405) Cartwright (63), and Walkenhorst (459) have independently drawn attention to the distribution effect of thermal gradients around electron microscope grids when these are used in the instrument for collection of particulates. Brun, Leroux, and Marty (50) have recently analyzed the thermal forces in the vicinity of the grids where precipitation takes place. These investigations suggest that care must be taken to secure good representation in electron microscropic examination of grid precipitates. A further treatment of sampling by thermal precipitation for electron microscopic study was given by Froula and coworkers (136). A design modification for adjusting the location of precipitator hot wires has been described (103). Uniform deposition has been sought by a rotating design (64). Collection of the respirable size fraction is provided for in another model (171).

The thermal forces on aerosol particles in a temperature gradient were examined by Saxton and Ranz (384); a mathematical basis was established upon which designs could be developed. Subsequently Gordon and Orr (149) designed a model with a flow rate of 5 liters per minute suggesting that the main limitation of thermal precipitation, low sampling rate, may ultimately be overcome. Theoretical aspects have been summarized by Popoff (346).

**Electrostatic Precipitation.** This technique has continued to be used where massive collection down to submicron diameters is sought. Commercial designs have proved generally suitable, as evidenced by the limited number of laboratory models described in the past few years. The high efficiency of the commercial type was reconfirmed by Lauterbach and coworkers (254) using sodium-24-enriched sodium chloride aerosol. Fraser (131) has reported on differences in collection efficiency within the submicron range.

Wilkening (478) designed a moving tape precipitator. Bergstedt (32) later described an adaptation for continuous monitoring of radioactive aerosols. A moving aluminum tape is used as the collector and this passes radiation detectors.

In a variation of the principle of electrostatic precipitation, Wilkening (479) collected submicron atmospheric particulates bearing natural radioactive matter by ionization and separation according to their mobilities. Natural radioactivity in the atmosphere was found to attach itself to particles having diameters in the range 0.001 to 0.04 micron. A subsequent paper by Yoshikawa and coworkers (495) detailed an electrostatic particle size analyzer of design similar to that of Wilkening (479) and an earlier worker, Rohmann (369). Particles were charged in a corona discharge according to their size and precipitated by an electrical field. A size range of 1 to 20 microns was claimed for particles of white plastic paint spray. Of related interest is a method for direct measurement of speed of small particles in air currents (276).

Increase in sampling volume and portability have been the characteristics of interest in other special designs. A model with two voltage circuits for sampling dilute aerosols has been built (481). A battery-operated unit for collection of mine dust at a rate of 100 cubic feet per minute has been developed (29). A portable model operating on 110-volt alternating current or 6-volt direct current power, and with arrangements for collection on glass or metal slides, was constructed (194). Another model permits direct microscopic examination of collected samples (486). Low pressure drop at flow rate of 1.5 cubic feet per minute and stainless steel construction have been featured in a precipitator specially suited to stack sampling (374). In Russia, a model for dealing with atmospheres containing up to 10 mg. per cubic meter has been used (267). A dilution method of flow measurement in electrostatic precipitation has been evolved (307).

Progress in understanding the theory of electrostatic precipitation is being made. Walker and Coolidge (461) have provided a semiempirical equation of electrostatic precipitation based on extensive experimental work. Lowe and Lucas (274) summarized theories of behavior of particles in an electrostatic field. An endeavor was made to resolve the discrepancy between theory and practice in the precipitation of particles by considering the molecular and electrical forces acting after deposition on the collecting electrodes. White (474)in 1955 presented a description of recent theoretical and experimental advances.

**Centrifugation.** Two methods of aerosol collection based on centrifugation appeared early in the period reviewed. Yaffe and associates (489) proposed a sizing sampler consisting of two Archimedes spirals cut into Lucite, through which particleladen air was passed at high velocity. Harris and Eisenbud (178) suggested a single unit from a multiple high-efficiency industrial cyclone collector for separating aerosols at 1-micron diameter level into two fractions. The fractions corresponded to the size collection characteristics of the upper and lower respiratory tracts.

### ANALYSIS OF AEROSOLS

In analysis of collected aerosols there

has been a notable increase in the attention given to the submicron diameters and this in turn has led to increasing use of the electron microscope. One factor which has influenced this trend has been the interest in fine radioactive particles and their removal from air (128, 129, 189, 401). In the biological field there have been a number of studies of respiratory retention in relation to particle size (14, 239, 326, 349). These have continued to point to the need for taking account of particle size distribution over a broad range in considering the po-tential toxicity of aerosols. The new application of fluorescent tracers in the study of the movement of pollution through the atmosphere has established a practical requirement for particle counting and identification (44, 191, 282, 339).

Several publications of broad interest in analysis of aerosols have appeared. Herdan and Smith (187) have published a volume on small particle statistics, containing an account of statistical methods for investigation of finely divided materials, with a guide to the experimental design of particle size determinations. A manual on particle size determination has been prepared by Cadle (57). In England, Rose has published a book on measurement of particle size in very fine powders (371). The collected papers from the important Conference on the Physics of Particle Size Analysis arranged by the Institute of Physics of the University of Nottingham have been published (46). A new German journal on aerosols has been noted (498). A significant book on electron microscopy by Hall (168) was released in 1953. Swift and Niemann (421) have formulated a semimicro system of qualitative and quantitative elementary analysis for detection of new and unknown chemical warfare agents. This accomplishment in methodology has significance for the air pollution field where exploratory analysis at the trace order of magnitude is frequently necessary.

Particle Size and Frequency. Sedimentation is possibly the simplest method of particle size determination available and continues to be widely employed in analysis of aerosols. Techniques were compared by Jarrett and Heywood (203) using a quartz dust of controlled grading. Mueller (310) also reviewed the method. Joos (209) studied the method according to Andreasen and suggested minor modifications. A particle size analysis apparatus based on sedimentation was reported by Bostock (37). An automatic sedimentation particle size analyzer has been developed by Yagi (491). Last year Whitby (473) contributed a rapid, general-purpose, centrifuge sedimentation approach for determination of size distribution of air

pollutants. The progress of sedimenting particulates has been followed photometrically as a method of size analysis (425).

Elutriation for sizing air-borne dust clouds in continuous flow was considered from the theoretical aspect by Walton (465). A size selecting sampler based on elutriation was reported by Wright (487).

Particle size analysis by optical and electron microscopy is favored by many workers in the aerosol field, partly because of the limited material generally available and for the reason that samples can be sized and counted by direct observation. In addition, samples for microscopy can be collected by techniques such as thermal precipitation, which are least disruptive to the state of dispersion of the particles when airborne.

Mounting techniques for particulates on membrane filters have been described by Cadle and coworkers (60) and by Einbrodt and Maier (109). Mounted filters become transparent when immersed in suitable media. Turbidimetric estimation of quartz in organic solvents has been carried out on samples dissolved from membrane filters (53). Kalmus (212) has evolved a procedure for electron microscopy whereby molecular filter membranes are dissolved in acetone and the particulates transferred to Formvar-coated specimen screens. A method has also been contributed by Yaffe, Paulus. and Jones (490). Hartman and others (181) found that uniformly dispersed specimens for electron microscopy could be prepared by suspending particles in protein solutions. Screens were submerged in these solutions and the streaming away of the solution on withdrawal established good distribution of particles.

Bernard and coworkers (35) have reported an electron microscopic procedure for particle size determination on fine powders. Results were in agreement with nitrogen adsorption measurements. Size estimation and other aspects of the electron microscopy of monodisperse latexes as outlined by Bradford and Vanderhoff (42) deserve reference. Cartwright (63) developed a combined optical and electron microscopic procedure using quartz and coal dust in the size range 0.3 to 10 microns. It was shown that differences in assessment in the overlap range of the two instruments were due to the greater resolving power of the electron microscope. Wilcox and Van Antwerp (477) have also investigated this aspect. McGinn (284) used a double-image micrometer to expedite size frequency work on aerosols by light microscopy. Counting and sizing aerosols by microscopy have been considered by a number of other investigators (71, 90, 91, 132, 365, 460, 497). Recent applications of the electron microscope to particle size determination in the aerosol field are numerous and include a study of uranium ore toxicity (482), filter testing (252), and effects of synthetic ceramic fiber dust (154). Bentley, Cartwright, and Gordon (31) have estimated the approximate size of liquid droplets from an atomizer by the simple procedure of spraying developer on exposed photographic plates.

The effect of the electron microscope beam on specimens under observation is a factor to be considered in this technique. Decomposition of inorganic specimens was observed by early investigators, and lately by Fischer (125) and Talbot (424). Examples include such substances as calcium sulfate, dihydrate and others known to occur in the atmosphere. A replica technique has been evolved by Fischer and Ellinger (126) for materials known to be unstable during direct observation.

Specimen contamination leading to growth of particles under observation and to deposition of artifacts has been recognized by many electron microscopists. The origin of these phenomena was investigated by Ennos (112)and ascribed to the interaction of electrons with oil vapor from diffusion pumps. Measures for prevention were evolved. Subsequently Yasuzumi and others (493) identified carbon black artifacts having their origin in combustion of vaporized oil or burnt tungsten filaments. In view of the exploratory nature of most current applications of electron microscopy to air pollution studies, the factors discussed above may be of special significance.

Surface area measurement has not been employed extensively for particle size determination in air pollution investigations, presumably because of the complexity of the nitrogen adsorption method, and the difficulty of accurate measurements with fine powders by the permeability method. The latter aspect has been dealt with by Kamack (213). A thorough treatment of the subject has also been provided by Barrer (26) who is well known for his fundamental work in this field.

Measurement of particle surface area by microscopy has been dealt with in a paper by Pidgeon and Dodd (343). These workers noted that measurement of specific surface area of nonporous particulates by microscopy generally depends on determination of a characteristic linear dimension such as average particle diameter. Empirical shape factors must be employed and a proper statistical method of averaging size distribution from microscopic observations must be evolved. Area and volume measurements as described by Kenrick (227), Tooley and Parmalee (437) avoid such uncertainties but are time-consuming. Pidgeon and Dodd

(343) have developed a more rapid method dependent on random orientation which they can secure by a new mounting technique. Another contribution to the subject was made by Robins (366), who employed a new method for measuring thickness of particles (367). In conjunction with measurement of the dimensions of projected images, this permitted surface and volume shape factors to be calculated.

Hemeon (184) has determined the surface area of particulate matter collected at Donora during the smog episode, in order to estimate quantities of adsorbed sulfur dioxide. The nitrogen adsorption technique was employed. As yet the toxicological significance of surface area versus particle diameter has been explored only in respect to quartz, where it has been proposed that surface area may provide a better assessment of hazard (28). It appears likely that the gathering momentum of fundamental air pollution research will ultimately bring surface area measurement into a more prominent role in the field.

Literature references to the use of xray techniques in particle size determination of urban air pollutants have not been noted. However, many other submicroscopic materials have been dealt with. The method yields most readily an average diameter, suits ranges around 0.1 micron and under, and only micro samples are required. The subject has been reviewed by Bernal and Carlisle (34) among others. Yudowitch (496) has outlined theory of the small angle scattering technique. Line broadening in diffraction patterns is an alternative procedure which has been exploited by many workers. On magnesium oxide particles, Birks and Friedman (36), for example, have found good agreement between electron microscopy and line broadening. An interesting comparison of this method with electron microscopy and the nitrogen adsorption procedure has been made by Franklin and coworkers (130) using fine ferromagnetic powders. These authors also obtained good agreement except in the case of the nitrogen adsorption technique.

# PHYSICAL MEASUREMENTS ON SUSPENDED PARTICLES

Physical measurements on particulates in suspension continue to be persistently investigated. The approach is usually based on the light-scattering property. Steady progress has been made in understanding of the variables which must be dealt with in design of direct particle size distribution apparatus, but it is evident that substantial fundamental work will be required before instruments fully capable of handling multidisperse aerosols will be created. Meanwhile a better conception of the relation of particle size, mass concentration, and chemistry of aerosol pollutants to their toxicity is being secured through the accelerating research programs and this should ultimately aid in equipment design.

Mass Concentration by Scattering. Some years ago LaMer (240) drew attention to the forward angle Tyndallometer. Based on this principle, an air pollution instrument for direct continuous measurement of mass concentration was developed by Sinclair (406). It is claimed that a moderate change in average particle size or distribution does not have a marked effect on scattering. For highest accuracy it was suggested that the instrument should be calibrated with the aerosols found in a given locality. Chen and associates (73) and Broomhead and Burdekin (47) have attempted to exploit Tyndallometry for dust assessment.

**Visibility Instruments.** Practical instrumentation for visibility measurement has been derived from photometric principles established by Steffen and Rubin (413) and Coleman and Rosenberger (85). One of these recording visibility meters was developed by Chaney (69). Another has been described by Marynowski and Littman (295).

A visibility instrument based on transmission has been developed by Douglas (105). This was tested in Detroit by Clayton and Giever (83), who have reported that visibility assessment, as derived from transmittance readings, was not found to be correlated with the soiling power of the atmosphere or the mass weight of air-borne particulates.

Direct Counting and Sizing. In 1954, Gucker and Rose (161) described a forward-scattering counter and sizer with dark-field illumination greatly improved over the original models designed by Gucker and his group of investigators (157-9). This counter, tested on dioctyl phthalate aerosol, gave linear response over the range 0.25 to 0.5 micron. A counting efficiency of 95  $\pm$  8% was realized. In 1955, a new instrument was described by O'Konski and Doyle (322). The functions of a recording light-scattering photometer and electronic particle counter were combined in a single instrument employing right-angle scattering and single electron photomultiplier tube. With uniform test aerosols produced from polyvinyltoluene and polystyrene latex hydrosols, it was found that pulse amplitudes for 0.33-, 0.5-, and 1.0-micron diameters were proportional to the square of particle diameter. Lower limit of sensitivity for single particles was 0.3 micron. Lower concentration limit of sensitivity was 3 imes

 $10^{-11}$  gram per liter of a 0.3-micron diameter test aerosol. This proved to be thirty times greater than the sensitivity achieved in 1947 when the first model was tested (158). As a result of the availability of polyvinyltoluene and polystyrene latex hydrosols, previous uncertainties due to the difficulty of producing uniform aerosols have been overcome and the present tests have indicated that a light-scattering particle counter is feasible for rapid determination of size distribution in aerosol systems. A review of the work of these investigators in design of aerosol particle counters has been given by Gucker and Rose (160). Gucker and Doyle (156) have studied the possibility of determining particle size of aerosols by measurement of sonic amplitude, and conclude that further exploration is warranted.

The LaMer group has made further contributions. LaMer and Gendron (243) reviewed work on monodisperse aerosols. Lee and LaMer (256) published details of the latest forward angle light-scattering camera developed by this group and reviewed the various studies leading to this development. For monodisperse aersols, particle size can be determined by light-scattering methods to around 0.1-micron radius. This has been accomplished in the LaMer laboratory by transmission as a function of wave length, and maxima and minima in intensity of scattered light as a function of angle in terms of (a) wave length (higher order Tyndall spectra), (b) state of circular polarization, and (c) ellipticity of polarization. Studies on growth of monodisperse aerosols (244-6) have made it possible to extend the range of measurement of the methods. Exhaustive attempts to deal with polydisperse preparations by measuring state of polarization at a number of angles failed to provide a size-distribution function. For the present camera smallest particle size detectable is 0.12-micron radius using dioctyl phthalate, with prospects of reaching 0.075-micron radius by optical and photographic modifications. Upper limit of resolution was 0.50 micron. Samples as dense as 10<sup>6</sup> droplets per cc. have been counted. As few as 30 per cc. was the minimum concentration measurable. Studies on growth of aerosols have continued (150, 242).

Other scattering systems for particle size studies on aerosols have been considered in the United States by Aughey and Baum (19) and Katz and coworkers (215). Kerker and Hampton (228) have compared monochromatic and nonmonochromatic light in polarization ratio measurements for size determination of spherical particles. Chin and coworkers (75, 76) have examined polydisperse systems by means of measurements of angular variations of intensity of forward scattered light at very small angles.

A number of counting and sizing devices for particles have been under investigation in other countries. The Nottingham Conference (46) afforded the occasion for a review of progress. Le Bouffant and Soule used intermediate photography and Cooke-Yarborough and Whyward have been dealing with the less imposing problem of red blood cells. Dell has been developing an arrested scan-type microscopic particle counter for use in the pneumoconiosis control program of the National Coal Board. Courshee has been working on a device for measuring the size of black dye spots deposited from agricultural test sprays. An automatic system for obtaining particle size distributions with the aid of the flying spot microscope has been studied by Taylor. A definitive paper by Walton discussed theory and design. Ellison considered the limiting factors in the range of particle sizes which might be estimated by light scattering.

Theoretical aspects and experimental work on light scattering for size determination of polydisperse systems of polymer particles were dealt with by Atherton and Peters (15-17) in England, and by Inagaki and Takahashi (196) in Japan.

A late development in automatized counting is the production of transparent profiles of dust particles by Hamilton and Phelps (172). These are created by metal shadowing done at right angles to the slide on which dust particles are collected. Subsequent removal of dust leaves a precise impression of the geometric projections of particles in the form of holes in the shadowed film. The technique is claimed to solve many optical problems arising from association of automatic counting techniques with high power light microscopy. The technique is based on a suggestion by Whitaker (472).

Other Direct Physical Methods. Vonnegut and Neubauer (456) have proposed a counter based on the cooling of a heated filament when a small vaporizable particle in a passing air stream makes contact. Change in resistance of the filament due to cooling is estimated.

A paper by Cadle and Wiggins (59) on direct photomicrography of airborne particulates offers a technique which avoids the deforming effect of collection on the dispersion of particulates.

Schaefer (385) demonstrated applications of a cloud chamber for studying small particles in the atmosphere. A continuous recording cloud chamber apparatus for counting nuclei in aerosols has been described by Saunders (383). An alternate-type expansion chamber is associated with recording on motion picture film. A simple, accurate method has been devised for counting droplets in a given area of film.

A device called the Sky Scanner (30) has been designed by Beard and Wilhelmsen to trace the movement of radioactive materials leaving stacks of atomic energy installations by remote measurement of gamma intensity. Gamma radiation incident on a sodium iodide crystal induces scintillations which are photomultiplied and recorded. The instrument detects an estimated minimum of 6000 curies of 0.5-m.e.v. gamma emitters at one mile.

### AEROSOL GENERATORS

A comprehensive review of methods for generating solid aerosols appeared early in the year under the authorship of Silverman and Billings (404). Lauterbach, Hayes, and Coelho (253)have published details of a modified aspirator system for aerosol generation on a 6-hour basis.

A contribution on droplet production was made by Dautrebande and coworkers (93) in 1953. Vonnegut and Neubauer (315, 457, 458) and Drozin (107) have conceived electrical atomization procedures. Burgovne and Cohen (52) have adapted the Sinclair-LaMer aerosol generator (243) for production of large droplets. Other droplet devices have been reported (354, 356). Aerosol formation by vapor condensation has been considered mathematically by Corner (87) and experimentally by Amelin and Belyakov (8) who have evolved a technique for regulating dispersion. Aerosol formation from low boiling point substances has been studied (407). A method for uranium fume generation has recently been outlined (142). Generators for simulants of fallout from nuclear detonation were used in experiments by Cohn and associates (84). Kethley and others (229) claim that microorganisms are a valuable tool for testing atomizer performance.

Determination of Charge on Aerosols. Techniques for determining charges on aerosols were summarized by DallaValle and Orr  $(\mathcal{PZ})$  in 1954. A device for this purpose has been designed by Masters  $(\mathcal{PT})$ . Dodd (102) has determined electrical charges on droplets during generation. Jutzi  $(\mathcal{PI}1)$  has used a modified ultramicroscope arrangement in which particles are deflected by an electric field and are photographed to permit calculation of charge and particle size. A variety of materials was studied.

The behavior of particles and droplets in electrical fields and the kinetics of interactions have been fundamentally considered by Russian workers (141, 226, 230, 309). Suspension of aerosols in turbulent atmospheres has lately been considered by Finkelstein and Magram (123).

Chemical Identification of Individualized Particulates. For several years a small group of workers has concentrated on developing techniques for chemical analysis of individual particulates. Cadle (56) has demonstrated a number of chemical identifications using reagents and micromanipulative methods. Seely (390) established a method for detection of submicron chloride particles by collection on reagent-impregnated gelatin. This work was subsequently extended to detection of calcium, sulfate, and nitrate ions by Lodge and Hanoi (269). A study of controlling factors in identification of chloride particles with sensitized gelatin film was reported by Pidgeon (342); it was shown that a number of important factors such as temperature, collection technique, and thickness of film were operative. Following this investigation, Lodge described the use of millipore filters (268) as a medium for collection and analysis in the micron range. The technique involved soaking millipores in suitable reagents. Film impregnated with thymol blue was later used by Gerhard and Johnstone (137) for microdetermination of sulfuric acid aerosol in their experiments on the photochemistry of sulfur oxides in the atmosphere (138). Seely (391) described the detection of a variety of metallic ions in  $10^{-10}$ - to  $10^{-15}$ -gram particles. Farlow (118) developed an improved halide ion-sensitive sampling surface for water aerosols of 25- to 400-micron diameter. This author has also created a quantitative analysis procedure for chloride ion in  $10^{-6}$ - to  $10^{-12}$ -gram particles (117) and a semiquantitative method for mixed soluble halide ions in single  $10^{-10}$ -gram particles (116).

In Canada, a study has been carried out with the object of developing a series of chemical identification tests applicable to submicron particulates mounted for optical or electron microscopy. Gaseous reagents were used so as to subject the arrangement of particulates to least mechanical disturbance. Morphological or color changes observed by microscopy of particular areas of the sample permit the nature of the particles to be identified. The results of these experiments were published by Monkman (305).

Twomey (447) has worked out a chemical identification technique based on measurement of relative humidity at which a hygroscopic particle becomes liquid. Particles were collected on spider webbing. Weisz (469) has provided a technique for determining ions semiquantitatively in single drops of material. Applications of X-Ray Diffraction. A noteworthy development in analysis of collected aerosols has been the increasing exploitation of x-ray diffraction. This technique offers a particular advantage by permitting qualitative and quantitative estimation of crystalline compounds in the chemical form in which they occur in the sample.

Applications of the technique in the environmental field were described by Lennox and Leroux (258). Among these was qualitative determination of levels of crystallites in particulate matter collected from the air of two Canadian cities. The direct quantitative diffraction method of Leroux, Lennox, and Kay (259) has been applied to analysis of a variety of effluents. This work was continued by Leroux and Trillat in France on the air-borne particulates from certain European cities (260). Cholak, Schafer, and Yeager (79) used x-ray diffraction to identify the air-borne effluents from an iron slag processing plant. Katz and Shore (223) used the technique on particulates collected in the Windsor-Detroit study. Hemeon (184) reworked by x-ray diffraction, the water-soluble fraction of particulate matter collected from Donora smog, and identified substantial quantities of zinc ammonium sulfate and zinc sulfate. This study also included similar material collected in London during the smog period in December 1952. Ammonium chloride and calcium sulfate of undetermined significance were the major constituents of the soluble fraction. These findings by workers who have been employing x-ray diffraction demonstrate the potential usefulness of the technique as an aid in delineating the nature of atmospheric reactions involving crystallites.

The use of electron diffraction for identification of chemical composition of individualized particulates in the electron microscope has been attempted, but this approach has not met with great success according to Cartwright (62). Contamination of materials by particles worn off brass specimen holders during mounting has been noted (299).

Spectroscopy on Particulates. Spectrographic methods continue to be used for the analysis of trace elements in collected particulate matter (184, 218). An extension in the range of the technique is represented by the appearance of fluorescent x-ray spectrography, which has been claimed superior to chemical and spectrochemical methods in determination of trace amounts of iron, nickel, and vanadium on catalysts (108). An x-ray photoelectron spectrometer for chemical analysis has also come into use (414). Applications of x-ray emission spectrography to analysis of air-borne dusts have been made (190). The voluminous literature on special applications and new developments in techniques of spectroscopy is comprehensively reviewed each year by the AMERICAN CHEMICAL SOCIETY in ANALYTICAL CHEMISTRY.

Analysis for Beryllium, Arsenic, and Fluorides. A few highly toxic elements occurring as air pollutants in the particulate form are well known. Arsenic trioxide is evolved in some smelting operations and can lead to massive environmental contamination as in a case being dealt with by the author (225). Beryllium is another effluent feared for its serious effects on health (177). Fluorides have preoccupied many investigators during the review period and mention may be made of the contributions of MacIntire (285, 286), and Adams (2), and their associates to knowledge of plant damage and effects on soil fertility. Semrau (393) has provided a review of sources of emission and Lunde (275) surveyed control aspects. The Boyce Thompson Institute workers have pursued plant metabolism studies with special equipment and techniques (162). Phillips (341) reviewed fluorosis in cattle.

Analytical chemistry of beryllium was reviewed by Vinci (455), Toribara, and Sherman (438) in 1952. Two new spectrographic methods have appeared (247, 408). A paper by Gilleson (140)dealt with the continuous automatic analysis of beryllium by a modification of the Aughey-Koppius (18, 234)continuous spectrographic method for lead. There have been other recent contributions to beryllium microdetection (1, 238, 420, 429, 441).

Modifications to the Gutzeit method for arsenic determination continued to be proposed-for instance, a field method for determining traces of arsenic in soils by a confined spot procedure (5). The molybdenum blue method is still being critically examined and modifications have been explored by Berkhout and Jongen (33) among others. Photometric determination of end point in titration of microgram quantities of arsenous acid with ceric sulfate is a procedure conceived by Bricker and Sweetser (45). During the last few years arsenic methods have been the subject of a special study by the Committee on Recommended Analytical Methods of the American Conference of Governmental Industrial Hygienists. As a result of trials of methods by eight collaborating laboratories, a modification of the colorimetric method of Vasak and Sedivec (451), by Monkman, has been selected (9).

The dissatisfaction with methods of analysis for fluorides is evident from the extraordinary number of papers published on this subject. The classical method of Willard and Winters has

been modified by a number of analysts, notably Remmert and Parks (359) and most recently, Gwirtsman, Mavrodineanu, and Coe (162). Deutsch (98) has remodified the Remmert and Parks contribution to eliminate interference of manganese and chlorides in vegetation. Loss of fluoride in ashing through formation of iron fluoride and interference of silica in recovery of the element in the method of Remmert and Parks (359) has been claimed (452, 453). Other papers among many on this subject have involved fluorometry (347, 480), cathode ray polarography (287), spectrophotometry (193, 195, 273), a study of colorimetry of organoferric complexes bleached by low concentration of fluorides (318), electrochemistry (25), ion exchange resins (319), and isotopes (324).

Organic Fraction of Air-Borne Particulates. Exploration of organic chemicals in particulates of Los Angeles air was initiated by Mader and coworkers (291). Among other constituents a variety of products of fuel combustion was found. A survey of this aspect for other United States cities has been reported by Tabor and associates (422). A method for determining free carbon in atmospheric dust has been published (280) and Clayton, Arnold, and Patty (82) have pointed to the possibility of identifying the source of atmospheric carbon in particulates by evaluating the carbon-14 content.

Evidence of a disproportionate rise in incidence of lung cancer in urban populations has been presented both in the United States and England. It has been tempting to seek for the explanation of this phenomenon in the presence of carcinogenic substances in urban air. Waller (464) directed attention to the presence of benzpyrene in the air of certain British cities. Los Angeles air samples were shown to contain carcinogens by Kotin and coworkers (235) in 1954. Kotin, Falk, and Thomas (236) have since identified by ultraviolet absorption spectra, pyrene, benzpyrenes, and other polycyclic aromatic hydrocarbons in the soot produced by gasoline engines. In a later paper, Diesel engine exhaust was studied with comparable findings being made (237). Falk, Markul, and Kotin (115) next investigated the compounds found in atmospheric soot and the stability of such compounds in the atmosphere It was noted that these compounds varied in their stability to light and air. In certain instances the rate of destruction by air in light was decreased when the compounds were adsorbed on soot. Destruction in smog was greater in some instances when the compounds were pure, greater in others when adsorbed on soot. A late paper by Falk and Kotin (114)

discussed carcinogenicity of aliphatic derivatives such as diepoxides.

Tebbens, Thomas, and Mukai (427)have noted that polycyclic hydrocarbons occur in combustion products of hydrocarbon gases. Thomas and coworkers (430) of the same laboratory have identified an extensive range in fuel combustion products. Shore and Katz (398)have estimated polyaromatic compounds in the air of Windsor; Cooper (86) has contributed further evidence of their presence in urban environments of England. Dikun (100) has made similar estimations in Russian cities.

Chromatographic separation and ultraviolet absorption spectroscopy are the techniques of choice in such studies at the present time. Catalytic iodination on activated alumina with subsequent spectrophotometry has been suggested for determining benz(a) pyrene in complex mixtures (448).

### GAS AND VAPOR ANALYSIS

Definitive methods for oxidants, ozone, and nitrogen dioxide have been studied actively. The petroleum hydrocarbons and the end products of organic combustion have been featured. Because of the essentially photochemical basis upon which Los Angeles atmospheric reactions take place, the research of Haagen-Smit and Fox (166), Taylor and Blacet (426), Johnston (206), Stephens and associates (415), Littman, Ford, and Endow (265), and Gerhard and Johnstone (138) is of great interest to workers in the air pollution field. The kinetic studies of Altshuller (6), and Morriss, Bolze, and King (308) on nitrogen oxides, of Saltzman (382) on oxidants, and the investigations of Goodwin, Bolze, and Morriss (147) on smog formation in large chambers, have provided information bearing on fundamental aspects of the subject. Cholak and others (80) have presented data on oxidant and nitrogen dioxide levels in three United States cities and have discussed evidence for photochemical production of atmospheric oxidant. The definitive toxicity studies of Stokinger (416) on ozone and nitrogen oxides are particularly notable.

The public attitude toward odoriferous pollutants has stimulated work on the mercaptans and other offending compounds. The emphasis on monitoring has continued to grow as indicated in a paper by Romanovsky and others (370) on automatic instruments used in Los Angeles. Friedel (134A) has briefly reviewed applications of spectrometry to studies of organic atmospheric pollutants.

### COLLECTION OF GASES AND VAPORS

New developments in collection of

gases and vapors have been limited, presumably because of the trend toward direct methods of analysis. Treatments of the subject were done by Ingram and Dieringer (197) in 1953 and by Silverman (402) in 1956.

The use of liquid air, oxygen, or nitrogen in mass spectrometry studies of gaseous pollutants was first resorted to in order to trap the range of materials for which the instrument was suited as an analytical tool (396). This method of collection has not been fully satisfactory. As pointed out by Quiram and coworkers (350), yield of hydrocarbons below  $C_3$  is low and liquefied gases are costly. These authors have used the more economical method of adsorption on silica gel in a dry iceisopropyl alcohol bath, water vapor being pretrapped. The problem of retaining lower hydrocarbons has not been solved. Freeze-out trap designs used in oxidant precursor studies have been given by Littman and associates (265).

Procedures for use of activated carbon in collection of gaseous air pollutants and odors have been outlined by Turk and others (442, 444, 445). The condensation trap of Shepherd and associates (396) has also been applied for collection of odoriferous chemicals (328). For organic vapor collection, the use of magnesium perchlorate has been proposed by Bacarella, Dever, and Grunwald (23), who have successfully used the material without explosions (419). A constant flow suction unit has been evolved (386).

For the automatic collection of samples in Greenburg-Smith type impingers, Wilson (483) has designed a simple instrument with a mechanism for discharging and refilling hourly with collecting liquid over 24 hours. The instrument was tested as a scrubber for acid gases and hydrogen fluoride.

### ANALYSIS FOR NITROGEN OXIDES

In the Los Angeles smog investigations, the analytical perspective for nitrogen dioxide called for determination of fractional part per million quantities; existing chemical methods were inadequate for such low levels of concentration (192, 334, 492). As a result, chemical and spectrometric research on the subject has been active.

In 1953, Friedel and coworkers (135) described an improved mass spectrometric method for determination of nitrogen dioxide and related oxides. These workers were able to correct instrument instabilities, which had previously been associated with measurement of the dioxide, by pretreating the spectrometer system with nitrogen dioxide to eliminate reaction impurities normally present in the system. The instrument has been used for analysis of rocket

exhaust gases (40). Norris and coworkers (320) developed an ultraviolet absorption method based on a proposal by Frey and Moore (134) and absorption data described by Hall and Blacet (170). This method permits determination of nitrogen dioxide in mixtures of related oxides at the wave length 394  $m\mu$ , where the other oxides of nitrogen do not absorb. Saier and Pozefsky (378) have conceived an infrared absorption method for nitric oxide and nitrous oxide. Hershberger (188) has explored the possibility of analyzing for nitrogen dioxide and other smog constituents by microwave spectroscopy.

Chemical methods for gas mixtures of nitrogen oxides have been developed by Johnson (204) and Saltzman (381). The latter method uses a modified Griess reagent consisting of 0.5% sulfanilic acid and 20 p.p.m. of 1-naphthylethylenediamine dihydrochloride in 14% acetic acid. Sensitivity to a few parts per billion for a 10-minute sample at 0.4 liter per minute is claimed. This reagent has been employed in a continuous nitrogen dioxide recorder developed in Los Angeles by Stanford Research Institute and the Air Pollution Foundation (363, 435). The changes in color of a continuously flowing absorption solution are measured and yield the nitrogen dioxide levels in the sampled air. Nitric oxide can be estimated concurrently by treating a duplicate air sample with ozone and determining the concentration as nitrogen dioxide with Saltzman reagent. Cholak and associates (80) used a recorder of this design in Cincinnati during the summer of 1956.

#### OZONE AND RELATED OXIDANTS

Recent developments in oxidant and ozone determination have been mainly directed toward the design of continuous recording equipment for part per hundred million concentrations. However, a quantitative rubber cracking test for ozone was reported in 1951 by Bradley and Haagen-Smit (43) and has been widely used since that time. It has been lately claimed by Crabtree and Biggs (89) that free radicals also produce similar cracking. A chemical method for oxidant estimation has been developed by Haagen-Smit (164). This is based upon oxidation of phenolphthalin to phenolphthalein. The reaction is primarily sensitive to ozone and partially sensitive to peroxides and nitrogen dioxide. An oxidant method by Todd (436) involving conversion of ferrous thiocyanate to ferric thiocyanate is relatively insensitive to ozone, most sensitive to peroxides, and sensitive to a lesser degree to nitrogen dioxide.

Littman and Benoliel (263) published a description of a continuous oxidant recorder based on the reaction of ozone with potassium iodide in neutral buffered solution. As the reaction was not specific for ozone, the recorder was tested against nitrogen dioxide in typical atmospheric concentrations. The instrument consists of a gas-liquid contactor unit and a recording colorimeter operating at 355.0 m $\mu$ , an intense maximum for iodine.

Littman and Marynowski (266) have reported the results of experiments to determine the proportion of ozone to other oxidants in the Los Angeles atmosphere. Three methods were employed: the rubber cracking test (43), direct spectroscopy for ozone at 255.0 m $\mu$ , and isolation of ozone by adsorption on low temperature silica gel with subsequent absorptiometric estimation. General agreement among the three methods was secured and it was established that ozone accounted for 30 to 45% of the total oxidant.

Spectrometric estimation of ozone in Los Angeles air was first done by Stair (410). Regener (358) later used a long-path ultraviolet instrument and made a number of measurements on a more precise basis. Stair and coworkers (411) made continuous estimations of ozone in Washington with a 1450foot path instrument using the Hartley band at 250 to 260 m $\mu$ . Automatic insertion of filters passing bands centered at 253.7, 365.5, and 405  $m\mu$ provided for correction of effect of other attenuants. An automatic 300foot path ultraviolet spectrometer has since been constructed by Renzetti (360, 361) in Los Angeles. Ozone absorption bands at 265, 280, and 313  $m\mu$ , as established by Vigroux (454), are measured. The ratio of intensities at 265 and 313 m $\mu$  and the ratio at 280 m $\mu$  and 265 m $\mu$  are calculated for the period between midnight and 4 A.M., when photochemical processes are inactive and ozone is minimal. Ratios determined in test periods are compared with the control ratios to yield the ozone concentration. The influence of other attenuants was accounted for by corrections calculated from data of Baum and Dunkelman (27). Another continuous ultraviolet method for ozone has been reported by Fastovskiĭ and Rovinskiĭ (120).

The development of a catalytic atmospheric ozone analyzer has been reported by Olmer (323). Ozone concentration is measured by the temperature differential between two thermistors placed in the gas stream. One is coated with a catalyst promoting the decomposition of ozone. The other is uncoated and used as a gas temperature reference. The two belong to a bridge circuit, output being fed to a recorder. The catalyst employed is Hopcalite, manufactured by the Mine Safety Appliances Co. for carbon monoxide decomposition. It was found that the water vapor of the atmosphere reduced the carbon monoxide sensitivity of the apparatus to the point where 20 p.p.m. was inconsequential. Negative response to other gases was as follows: 20 p.p.m. chlorine, 10 p.p.m. nitrogen dioxide, large unspecified concentrations of hydrocarbons and smoke.

For estimation of low concentrations of ozone, Pring (348) has evolved a method in which the oxidation potential of a bromine-hydrobromic acid electrode is measured. Other oxidants, including nitrogen oxides, do not influence the potential to a substantial extent. Ozonides were determined by infrared spectroscopy in the photochemical investigations of Stephens and coworkers (415).

#### HYDROCARBONS AND RELATED PROBLEMS

Haagen-Smit and coworkers (43, 163, 165, 272) found evidence of an interrelationship of hydrocarbons, ozone, and nitrogen oxides in the production of eve-irritating smog.

Petroleum hydrocarbons and products of combustion were identified in Los Angeles air in 1951 by Shepherd and coworkers (396) who employed mass spectrometry. Among other constituents, a number of saturated and unsaturated olefinic hydrocarbons in the  $C_2$  to  $C_{10}$  range were individually determined. Another air study by mass spectrometry has been detailed by Quiram, Metro, and Lewis (350) who detected 3 p.p.m. of isopentane, 7 p.p.m. of propane, and 8 p.p.m. of methane in the vicinity of a refinery. By the same technique, Weaver (468)has catalogued the contents of air sampled in Los Angeles, Detroit, and Washington and compared the hydrocarbons and other condensable impurities with raw fuels and products of combustion.

A number of investigators have employed mass spectrometry for analysis of automobile exhaust gases (49, 68, 110, 292, 336, 368, 462, 463). Walker and O'Hara (463) among others have described procedures for both individual and continuous estimations. The major problem is in obtaining representative sampling. Theoretical water content of exhaust gases can be computed on the basis of the combustion products. Hence the gasoline and soluble gas content of any sample should be representative if the measured condensable water content is near theoretical. Continuous sampling directly from an operating engine overcomes the representative sampling problem, provided complete vaporization of all condensable gases can be maintained. As intermediate products may no longer be present on final analysis of collected samples, direct measurement is preferable. A

mass spectra interpretation of a complete analysis is provided by these authors.

Infrared spectroscopy was applied to analysis of Los Angeles air by Mader and coworkers (290). Twiss and coworkers (446) used infrared instruments in dispersion and nondispersion portable forms for automobile exhaust analysis. Because the method reveals functional groups, an integrated estimate of saturated isomeric hydrocarbons of Ca and above was obtained. The calibrating hydrocarbon was *n*-heptane. Analyses for hydrocarbons were compared with mass spectrometer findings and good agreement was shown. Analysis for  $C_3$  and higher hydrocarbons, carbon dioxide, carbon monoxide, and nitrogen dioxide are most readily handled. Neerman, Parsons, and Bryan (314) have also used a nondispersion infrared analyzer, developed by Parsons and associates (329), for continuous estimation of unburned fuel in automotive exhaust. Littman and Denton (264) have operated a nondispersion instrument for automatic monitoring of hydrocarbons in Los Angeles air. A concurrent air sample is burned to establish a control reference.

MacPhee (288) has suggested the use of molybdates for determining olefinic hydrocarbons occurring as air pollutants. Ruff (376) has achieved measurement of catalytically oxidizable exhaust fumes by a modification of the familiar explosimeter. Weller (470) has created a method for determination of mixture ratios by analysis of engine exhaust gas with an acoustically resonant gas column. The high-temperature reaction of olefins with bromine was explored by Austin (20).

Gas chromatographic separation procedures for hydrocarbons were outlined by Janak (200-2) and by Bradford and coworkers (41) abroad. In the United States, separation procedures for hydrocarbons and automobile exhaust were worked out by Patton and others (331, 332). Lichtenfels and associates (261) succeeded with C<sub>5</sub> to C<sub>8</sub> hydrocarbons. Fredericks and Brooks (133) obtained good resolution through  $C_5$  with helium as carrier gas. The sample was partitioned in a two-stage column first by diisodecylphthalate and then by dimethyl sulfolane. Similar success was attained concurrently in Europe by Van de Craats (88). Drew and coworkers (106) described the application of vapor phase chromatography to mass spectrometric analysis. The utility of the method in the analysis of very small samples was demonstrated by the quantitative separation of 0.05 cc. of a mixture of ethane, propane, and butane. A number of hydrocarbons have been identified in cigarette smoke by gas chromatography (333).

Measurement Methods in Gas Chromatography. The application of gas chromatography to separation of low molecular weight hydrocarbons and other gaseous organics has extended the possibilities of measurement of individual series members. It has permitted the use of the thermal conductivity technique as exemplified by the design of Ray (355) and the density procedure as adopted by James and Martin (199).

The status of the thermal conductivity method, as a general technique for gas analysis, was reviewed by Brown and Dean (48) in 1955. These authors drew attention to the limitations of the method, pointing out that it is restricted to quantitative analysis of mixtures of known constituents in a known range of composition. Except in the case of binary mixtures, the measured constituent must have a thermal conductivity significantly different from other constituents or the other constituents must be present in fixed proportions or vary in a fixed manner. A paper by Dimbat, Porter, and Stross (101) discusses apparatus requirements for quantitative gas-liquid partition chromatography, including thermal conductivity detection. Details of a gage were given by Ambrose and Collerson (7). Strange (417) has recently described a thermal conductivity instrument for multicomponent gas mixtures. Scrubbing out of gases, which obscure the measurement of the selected constituent in the conventional thermal conductivity technique, is not required. Dijkstra (99) has applied gas-liquid chromatography to separation of 4- to 18-carbon aliphatic acids, esters, alcohols, and aldehydes. Using thermal conductivity as little as 10  $\gamma$  of methyl propionate was detected.

Scott (389) has developed a detector based on measurement of the temperature of a flame in which gas fractions are burned with hydrogen as a carrier gas constituent. This is compared with the vapor-density bridge (199) and the thermal conductivity cell (355). Another development in gas chromatographic measurement is the surface potential detector of Griffiths and Phillips (152). In this method two dissimilar plates situated close together are connected by a conductor. The electromotive force set up when one is caused to vibrate is changed if the surfaces are exposed to an adsorbable vapor. The instrument is stated to be nonlinear and slow acting in comparison with the thermal conductivity procedure.

Aldehydes and Ketones. Shepherd and coworkers (396) identified aldehydes and ketones in air by mass spectrometry. An approach to these substances has been made by infrared spectroscopy (175) as well as by classical chemical procedures (251, 291). Developmental activity in chemical determination of aldehydes and ketones is taking place in several special fields. A review of carbonyl analysis by Mitchell (304) appeared in 1953. Farr (119) reviewed the subject in 1955. New methods for aldehydes have been offered by Siegel and Weiss (399) in modification of the Ponndorf procedure (345). Fabre, Truhaut, and Singerman (113) and MacDonald (283) used a chromatropic acid technique suited to pharmacological and atmospheric applications. Small amounts of ketones have been measured by ultraviolet absorption and the iodoform reaction (13). Buyske and associates (55), in the cigarette smoke research field, have carried out chromatography of the 2.4-dinitrophenvlhvdrazones for separation of some aldehydes and ketones. Ross (372) has provided infrared spectra for 37 such derivatives. Rumpf (377) has given ultraviolet spectra. The polarograph has been exploited for their estimation by Petrova and Novikova (340). Touey (439) employed dimedon, which is converted in an absorption train to bis-dimedon, vielding good efficiency for lower aldehydes in cigarette smoke. For the determination of aldehydes in combustion products, Bailey and Knox (24) designed a method in which the aldehydes are oxidized to the silver salts of the corresponding fatty acids. Separation and determination of individual aldehydes is achieved by chromatography of the acids which can be liberated from the silver salts. Other new methods have involved mercurimetric oxidation (375) and reaction with unsymmetrical dimethylhydrazine (400). Additional procedures for sensitive detection of formaldehyde have been advanced (121, 313, 450, 471A).

Organic Acids. In an early exploratory study by Mader and others (291), organic acids in Los Angeles particulate matter were determined by the classical hydroxamic acid test. Such compounds have been estimated in automobile exhaust gases by Walker and O'Hara (463) using the mass spectrometer. As interest in the identity of particular organic acids developed, chromatographic techniques were introduced. The method of Bulen and associates (51) was employed in plant damage studies (279). Tebbens and Torrey (428) identified formic and acetic acid as urban air pollutants by paper chromatography of the ammonium salts. Thomas and others (430) of the same laboratory have since reported separation of an extensive range of unidentified organic acids in fuel combustion products using paper electrophoresis. Pertinent to identification is a communication by Childers and Struthers (74) which indicates that the sodium salts of the lower aliphatic carboxylic acids can be quantitatively determined by infrared spectroscopy. This approach was found to overcome identification difficulties with the free acids due to hydrogen bonding. Warner and Raptis (466)have offered a procedure for determination of formic acid in the presence of acetic acid by azeotropic distillation with chloroform and subsequent titration of the separated acid.

Mercaptans and other Odoriferous Chemicals. The odoriferous chemicals associated with petroleum refining and the petrochemical industry have been the subject of extensive investigation. Among contributions pertinent to the problem has been an exhaustive treatment of analytical methods for sulfur compounds by Karr (214). The determination of thiols in hydrocarbon gases as a method of estimating stench in commercial propane and other hydrocarbon fuels or refinery gas streams was dealt with by Ellis and Barker (111). Segal and Starkey (392) devised a quantitative technique for methyl mercaptan, dimethyl disulfide, and dimethyl sulfide in gas mixtures according to a procedure of separating mercaptans by mercuric cyanide and absorption of disulfide and sulfide in benzene. Automatic coulometric titration for mercaptans in concentrations as low as 100  $\gamma$  was used by Leisey (257). Mercaptan sulfur in hydrocarbons has been estimated amperometrically by Grimes and others (153) with a modification of the classical method of Kolthoff and Harris (233). Mercaptan sulfur was determined to  $\pm 1$  p.p.m. for liquid hydrocarbons containing 0 to 100 p.p.m. Gaseous hydrocarbons with a content of 0 to 400 p.p.m. could be handled with an accuracy within  $\pm 3$  p.p.m. Organic sulfides, disulfides, thiophene, and tetraethyllead do not interfere, nor free sulfur if present below 0.001%. Landsberg and Escher (248) have outlined the use of commercial sulfur dioxide recording equipment for refinery sulfur compounds.

The problems of odor in many other operations than petroleum processing call for solution, and rational methods of assessment and identification are the key to effective control. Mateson (298) has contributed a review on olfactometry which reveals present frustrations in design of odor assessment apparatus, though an improved example has been evolved. Techniques of assessment and control have been described by Turk (442-5) and Pantaleoni (328). Chapman and Eaton (70) have considered electrical potential changes at surfaces as a means of measuring odorous atmospheric contamination. A tentative standard test procedure on

analysis of odor (12) has been developed by the ASTM Committee D22.

#### OTHER DEVELOPMENTS IN GAS AND VAPOR ANALYSIS

A notable feature of gas and vapor analysis in recent years has been the steady introduction of automatic units from commercial sources. These new entries to the field are reviewed in the monthly columns of ANALYTICAL CHEM-ISTRY and Industrial and Engineering Chemistry. Established continuous devices have been listed by Katz and Clayton (219), Clayton and Giever (83), and by Saint-James (379).

Sulfur Dioxide and Hydrogen Sulfide. A review of analytical methods for acid mist and sulfur dioxide was published in 1953 under the authorship of Lombardo (270).

The shortcomings of the Thomas Autometer instrument for sulfur dioxide monitoring are detailed from time to time (139, 316), but it continues to provide the one gas measurement which is common to most urban air pollution surveys. The Titrilog, dealt with in the previous review (224), has been examined in detail with respect to performance and applications (248). It has been recently adapted in a portable form for automobile and helicopter use (61). Tentative methods for continuous recording of sulfur dioxide (11) were adopted by ASTM Committee D-22 in 1955.

New automatic and semiautomatic variations of the procedure of determining hydrogen sulfide by lead acetate-impregnated paper have been created in various parts of the world by such workers as Sensenbaugh and Hemeon (394), Offutt and Sorg (321), Maslennikow and Kavickaja (296). and Hart (180),

The search for simple and sensitive chemical methods of detection of sulfur dioxide and sulfur trioxide has continued. Color-changing gels based on the Kitagawa patent (231) have been formulated by Patterson and Mellon (330) for sulfur dioxide. Separate determination of sulfur dioxide and sulfuric acid in air is claimed for a method by Alekseeva and Bushtueva (3). Pannetier and Meltzheim (327) have also proposed a method. A spectrophotometric technique for sulfate has been formulated by Jones and Letham (208) using 4 - amino - 4' - chlorodiphenvl.Bleaching of Astrazone pink FG (Bayer) in soaked filter paper has been proposed as a method for sulfur dioxide in smog by Liddell (262). Stratmann (418) described a sulfur dioxide method involving adsorption on silica gel, regeneration, reduction to hydrogen sulfide over platinum catalyst at 700° C., and reaction with ammonium molvbdate to yield a blue color. Sensitivity

to 0.01  $\gamma$  per liter of air is claimed. The errors in iodometric determination of sulfur dioxide and hydrogen sulfide have been assessed (232). Lang and Mader (249) measured atmospheric hydrogen sulfide by absorption in cadmium acetate, and thereafter, iodometrically. Colorimetric and polarographic methods for sulfur dioxide have been compared (335). Moore, Cole, and Katz (306) have found that nitrogen dioxide reduces the response of the fuchsin method for sulfur dioxide. West and Gaeke (471) propose a sulfur dioxide method using disulfitomercurate(II) as fixant.

A study of oxidation of sulfur dioxide at room temperature on certain catalysts such as ferric oxide and carbon is pertinent to the field (96).

Hydrogen Fluoride. Semiautomatic determination of atmospheric fluorides was described by Thomas (434) using thorium nitrate and Chromeazurol-S as indicator. Employing a modification of the Feigl and Heisig test (122), Chaikin and coworkers (65) have set up a continuous analyzer for hydrogen fluoride. Mavrodineanu and Coe (300) developed a procedure for sampling and analyzing air containing as little as one to several hundred parts per billion of gaseous fluorides. A micro apparatus for hydrogen fluoride is the work of Reckendorfer (357) who modified the well-known Feigl and Krumholtz apparatus.

Carbon Monoxide. A practical development in carbon monoxide estimation has been the extension of the range of the Shepherd technique to the low levels occurring in urban air (397). The sensitivity of the iodine pentoxide method has been improved (143). Improvements in blood methods have been attained (4, 77). This gas has been determined in urban air by infrared spectroscopy (370). The oxidation of carbon monoxide by solid silver permanganate has been studied by Katz and his group (151, 220, 221).

Other Instrumentation and Applications. Troy (440) has devised filter systems for ultraviolet photometry. making this spectrum range useful in determining a wide variety of atmospheric pollutants. Broad usefulness is claimed for the infrared instrument of Littman and Denton (264). Yocom and coworkers (494) have analyzed effluents from backyard incinerators by infrared and chemical methods. A series of components of the gas phase of cigarette smoke has been established by infrared spectroscopy (325). Photochemical products from ultraviolet irradiation of biacetyl were identified by mass spectrometry (426) in studies by Taylor and Blacet. Hershberger (188) is experimenting with determination of free radicals by microwave spectroscopy. Newton (317)

has evolved a procedure for using an internal standard gas in mass spectrometric determination of condensable impurities at low concentrations in noncondensable gases, or noncondensable traces in condensable gases. The procedure is more important to the latter case. Pepkowitz (337) designed a continuous oxygen recorder for determining low concentrations in gases. This dewpoint method has been recently adapted to analysis of hydrogen and hydrogenous materials by a change from platinum to copper oxide catalyst (338). Schultz (387) has described positive ion emission techniques for gaseous organic halide tracers in air. A rapid gas analyzer using ionization by alpha particles is the work of Deisler and associates (97). This is a method for analysis based on the ionizing power of polonium in an aged radium D source. Binary mixtures of nitrogen, hydrogen, ethylene, ethane, and others have been handled. A gas detector (311) derived from the work of McCullough and others (281) has been commercially manufactured. These investigators determined carbon monoxide by conversion of red mercuric oxide to mercury which was photoelectrically detected by absorption at 253.7  $m\mu$ . The commercial instrument is claimed to deal with a formidable list of gases and vapors including formaldehyde, hydrogen sulfide, and hydrocarbons. The principles and applications of sonic gas analysis have been defined by Martin (294). Acoustic methods have been surveyed by Lawley (255). Kethley and others (229) have considered the reaction of air-borne microorganisms for sensitive detection of chemical contaminants.

Apparatus for generating gases has been described by Setterlind (395), Saltzman (380), and by Rieders and Hodes (364) who claim a method for generating at the composition of a parent mixture.

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