An abstract graphic consisting of numerous thin, light blue curved lines that originate from a single point on the left side of the cover and fan out towards the right. A white elliptical line is drawn around the point of origin.

Methods of Air Sampling and Analysis

THIRD EDITION

JAMES P. LODGE, JR., EDITOR

INTERSOCIETY COMMITTEE
AWMA ACS AIChE APWA
ASME AOAC HPS ISA

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PREFACE

This is the third edition of the manual of methods adopted by the Intersociety Committee for a Manual on Methods of Air Sampling and Analysis according to its established procedures. The Intersociety Committee, which was founded in 1960, with its subcommittees consists of several scores of experts currently representing eight major national professional societies concerned with environmental measurements. These experts have volunteered their time and services to evaluate, codify, and publish hundreds of test methods for measurement of air contaminants affecting the health and welfare of industrial workers, as well as of the general public.

While many methods have been published by various agencies, for hundreds of materials agreed-upon methods of adequate accuracy remain to be established. The Committee and subcommittees have served as a workshop and forum for interaction and coordination of professionals from different governmental agencies, academia, and industry, each with knowledge of the methods in use by his organization. In the decade that has passed since the second edition, many changes have occurred in the status of the Committee, its procedures, and the field of atmospheric chemistry.

Financial support from the Environmental Protection Agency and the National Institute for Occupational Safety and Health has ceased, and with it the ability of the Committee to meet frequently, and to convene the subcommittees. Consequently, most of the procedures previously carried out face-to-face have been consigned to mail and, on occasion, telephone. There have been changes in personnel, as noted below. The grand plan for a major program of interlaboratory testing of methods has been replaced by occasional round-robin testing where it was specifically needed,

driven by economic concern for EPA acceptance of commercial instruments, or conducted to resolve dispute. New, more powerful methods have emerged and become widely accepted, and previous concerns have disappeared. Plans to prepublish all methods in "Health Laboratory Science" for comment have not been implemented since the second edition.

While the American Public Health Association had previously given valuable support to the Committee by serving as the contracting and administrating society, we are now an independent organization. Nevertheless, there still appears to be a great need for a manual such as the present one. While instruments for routine air monitoring are today uniquely qualified or disqualified by the Environmental Protection Agency, many local agencies are justifiably reluctant to buy expensive monitoring equipment prior to preliminary tests to assess the nature of their problems. Such tests are generally made with available equipment and manual methods of measurement. For a large number of chemical species there are as yet no approved methods; nevertheless, it is of considerable importance to characterize their concentration in the environment. In the workplace, monitoring instruments are still less common than individual chemical tests on integrated samples spanning a significant portion of the workday. The likelihood that all these methods will be subjected to rigorous interlaboratory comparison is probably declining rather than increasing.

Accordingly, a number of changes have been made in the present manual. The status of methods as "tentative" or "recommended" has been removed throughout. Every method carries statements of precision, accuracy, and interferences from which the reader can quickly learn how much is known about the dependability of

that method. In the absence of sponsorship, the requirement to list method numbers according to the several agencies has also ceased. The given methods are advanced by the subcommittees and, where they are knowledgeable, by members of the parent Committee. They are among the best available, whatever their formal status, and those needing EPA and/or NIOSH method numbers are undoubtedly in position to ascertain them.

For further simplification, certain ubiquitous references have been omitted from the bibliographies. For example, virtually every method requires the use of reagent water of some type or other, as that is defined by the American Society for Testing and Materials, and the use of reagent chemicals, as these are defined by the American Chemical Society. Few analysts are unaware of these requirements, and it seems redundant to enter the appropriate references in every bibliography.

We have kept the previously assigned method numbers within the book, and assigned new numbers to added methods. Thus, some numbers are missing due to the judgment of subcommittees that the old methods were no longer in use, or should not be in use. The previous text, where it is retained, has been carefully edited to remove grammatical and typographical errors. Even in cases where old methods are largely retained, they have been carefully checked to be certain that they were clear and easy to follow; the overall result is that few pages of the second edition have survived without at least minor change. A number of new sections have been added to Part I, on general techniques, with the emergence of new laboratory tools.

In a number of situations it has been necessary to go outside the primary membership to reach truly knowledgeable scientists in the use of particular techniques. To give appropriate credit in these cases, a new convention has been adopted. The actual authors of the technique are listed first, fol-

lowed by the principal reviewers, if any, and then the balance of the subcommittee is listed in alphabetical order. Where changes are minor, the listing of the previous subcommittee, presumably responsible for the original writing, has been preserved.

The original 12 subcommittees have been reduced in number to 9, in effect, by the merger of Subcommittees 4 and 5, and the termination of Committees 7 and 12. The founding chairman of the Intersociety Committee was Dr. Leonard Greenburg. He was succeeded by Dr. E. R. Hendrickson, Professor A. C. Stern, Dr. B. E. Saltzman, Mr. R. F. Toro, Dr. W. T. Ingram, Dr. R. J. Thompson, and Dr. R. S. Saltzman, the present chairman. Successive editors have been Dr. M. D. Thomas, Dr. Morris Katz, and Dr. James P. Lodge, Jr., the editor for this edition. Over the entire history of the Committee, Dr. George J. Kupchik has been Executive Secretary. All correspondence concerning these methods should be directed to him at the School of Health Sciences, BC 612, Hunter College, 425 East 25th Street, New York, NY 10010.

The Intersociety Committee gratefully acknowledges the past support of the U.S. Public Health Service under Grant AP-00256; of the Environmental Protection Agency under Contract Grant 68-02-0004, and of the National Institute for Occupational Safety and Health under Contract number HSM 99-73-89. Acknowledgments of permission to reprint, with editorial modifications, methods from other societies and agencies are duly noted below.

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A KEY TO A CLEANER ENVIRONMENT

Other books have been written about air sampling and analysis, but there are none like this one.

To do any job well, you must use the proper tools, and this book, as with its two predecessor editions, gives you the know-how you need to sample and analyze air. Updated, with new methods added and in a streamlined form for ease of reading and understanding, this new Third Edition is in itself a tool for today's use.

Though full of changes from the edition of 11 years ago, this all-new work leans heavily on the two previous editions. Without the farsightedness of the leaders who gave us the first and second books through the Intersociety Committee for a Manual on Methods of Air Sampling and Analysis (beginning in 1960) this new work could have been created only by starting from "scratch." Even so, it was no mean task for the scores of committees and their editor to come forth with this matchless volume that will guide today's professionals who

work daily to understand and solve the problems of both indoor and outdoor air pollution.

Our hat is off to the experts who labored long and hard—with intelligence and integrity—to make this book a reality. (These pioneers are noted in the front pages of this book and in the committee rosters appended to each method.) We are extremely pleased to be associated with the people who created this worthwhile and useful venture.

Methods of Air Sampling and Analysis, Third Edition is both a timely volume and a book destined for a long life. It describes and evaluates new methods along with the classic. It is a current and up-to-date reference to be regarded as a principal tool in the continuing, expanding battle to help solve the never-ending problem of maintaining clean air and keeping our environment workably pure.

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ACS—American Chemical Society
 AIChE—American Institute of Chemical Engineers
 APWA—American Public Works Association
 ASME—American Society of Mechanical Engineers
 AOAC—Association of Official Analytical Chemists
 AWMA—Air and Waste Management Association
 HPS—Health Physics Society
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James P. Lodge, Jr. attended the University of Illinois, with his undergraduate training there interrupted by two years of service in the U.S. Navy during World War II. After graduation, he studied at the University of Rochester with Professor V. Boeckelheide, receiving his doctorate in 1951. He was Assistant Professor of Chemistry at Keuka College in New York from 1950 to 1952. During the summer of 1952, he joined the Meteorology Department at the University of Chicago, and has worked in the field of Atmospheric Chemistry ever since. From 1955 to 1961, he was Section Head in Chemistry in the Community Air Pollution Program, U.S. Public Health Service, in Cincinnati; this was one of the predecessors of the present U.S. Environmental Protection Agency.

From 1961 to 1974, Dr. Lodge was a Program Scientist at the National Center for Atmospheric Research in Boulder, Colorado, and since then, has been a Consultant in Atmospheric Chemistry. He was chairman of the section on air in the American

Chemical Society report, "Cleaning Our Environment: The Chemical Basis for Action," for which the Board of Directors of ACS awarded him a Special Certificate. He held the same position for the revision, "Cleaning Our Environment, A Chemical Perspective." Dr. Lodge has also received the Distinguished Service Award of the Division of Environmental Chemistry, ACS, and the Frank A. Chambers Award of the Air Pollution Control Association.

As Chairman of the Colorado State Air Pollution Control Commission for six years, Dr. Lodge received several local awards. He has contributed to the fields of air pollution analysis, atmospheric kinetics and photochemistry, atmospheric electricity, and theory of filtration, publishing over 200 papers. In addition to serving as Western Hemisphere Editor of *Atmospheric Environment*, he is its principal book reviewer. Dr. Lodge has also had several papers published in the field of his hobby, the study of theology.

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PART I

GENERAL TECHNIQUES

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PART I

GENERAL TECHNIQUES

1. Physical Precautions

1.1 HOMOGENEITY OF THE SAMPLE. No analytical result regardless of the accuracy and precision of the procedure can be any better than the quality of the sample submitted for analysis. Therefore, the primary concern of the investigator must be directed to the collection of representative samples and the homogeneity of the air mixtures employed to calibrate both the collection and analytical systems. Human sensory perceptions cannot guide the uninitiated with respect to variations of contaminant concentrations in either space or time. Almost without exception gaseous components are either colorless or are present at such low concentrations as to be effectively colorless. The human sense of smell is notoriously deceptive with respect to concentrations of even highly odoriferous components, and therefore cannot be relied upon for estimating relative quality or uniformity of the ambient atmosphere (2,4-Part 3,7,43,44).

The source of the contaminant, air flow direction and velocity whether due to wind or thermal gradients, density of the contaminant, intensity of sunlight, time of day, presence of obstructions such as trees, buildings, partitions, machinery, etc., which act as baffles to produce turbulence, humidity, and half-life of the contaminant together determine the concentration at any given location. That the concentration can vary by several orders of magnitude within a relatively short radius from the point of reference has been amply confirmed by many investigators. Air may flow either in a stream line or turbulent flow and this factor alone can determine the dispersal pattern of the pollutant (2,3,5,7,46).

The density of the pollutant in many cases will counteract diffusion processes to an extent which will establish stratification. This phenomenon prevails in the case of many highly toxic and irritant gases such as phosgene, mustard gas, lewisite, chlorine, etc. (5). Natural convective circulation and diffusion in confined spaces such as silos, coal mines, wells, caves, tanks, etc., is not

sufficient to maintain a normal atmosphere containing approximately 21% oxygen.

The location of sampling sites whether on a temporary or "grab" sample basis or on a fixed station basis for continuous monitoring over long periods of time is often critical and will determine the validity of the conclusions drawn from the results (1,2,3,7,46). In the vicinity of industrial atmospheres the variability may be sufficiently extreme to vitiate the results from fixed station monitors regardless of the care taken in locating the samplers. In an alkyl lead manufacturing facility, for example, no relationship could be established between exposure as indicated by urinary lead excretion and numerous fixed station samplers. In this case, correlation was not established until the monitors in the form of a small filter-microimpinger assembly were actually worn during the entire 8-h shift by the workmen assigned to these areas (1).

Several excellent treatises have been published on planning the air pollution survey and the effects of meteorological and geographical conditions on the validity of the results. This subject is outside of the field of General Techniques and Precautions; therefore, the reader is advised to consult one or more of these publications for full details (2,3,6,7,32,33,34,47).

On a smaller scale, even greater care is required to establish uniform mixtures of pollutants in air for use as calibration mixtures whether the system be dynamic or static in principle (9). Passage through lengths of small-diameter tubing to ensure turbulent mixing is one relatively simple expedient for dynamic mixing (3). Other systems employ some form of baffled chamber inserted between the mixing ports and the delivery point (10). Rotating paddles or even loose pieces of sheet metal or inert plastic to be activated by shaking have been inserted in containers for static mixtures for mixing by turbulence (11). Probably a more effective alternative is to evacuate the container, then mix the contaminant with the air at a uniform rate as it is released into the void (11). (Inject from a hypodermic syringe through a needle inserted through a

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septum.) Diffusion alone cannot be relied upon to produce a homogeneous mixture in most cases.

Other effects such as container wall absorption, humidity, volumetric errors, etc., discussed elsewhere in this section must be recognized and appropriate precautions taken to avoid concentration errors derived from these seemingly extraneous sources when attempts are made to produce homogeneous, known concentrations for calibration purposes.

1.2 ABSORPTION EFFECTS ON CONTAINER WALLS AND CONNECTING TUBES. Failure to recognize absorption effects can lead to serious errors both in collection of samples and in preparation of calibration mixtures, especially in static systems. Losses of analyte can occur either by primary adsorption, which may be reversible in response to pressure or temperature changes, or by secondary adsorption, which may involve chemical reaction with another material previously adsorbed (2,4,7). Long-term stability and accuracy of calibration blends have been studied as a function of the container material by several investigators. (48,49,50,51,52). The affinity of glass for water, nitrogen dioxide, benzene, aniline, the isocyanates, etc., has been recognized (8,12,13,31). The interior surface of a 5-gallon (19-L) borosilicate glass carboy can adsorb as much as 50% of the benzene in the 100 to 200 ppm range from a calibration mixture in air within 30 min after preparation. These losses can be reduced to some extent by preconditioning at a concentration higher than the working level but desorption may contribute positive errors (Table 1:I). Losses by adsorption of polynuclear aromatic compounds on glass have also been reported (14).

This adsorption effect can be minimized in the case of collection of air samples in evacuated glass flasks (Shepherd flasks) or sampling tubes by the introduction of a suitable solvent for the vapor, such as isooctane for benzene and xylidine, directly into the container with thorough rinsing of the inner surfaces (31). In some cases, such as the analysis for nitrogen dioxide, the solvent or reagent is introduced before the sample is taken; e.g., 100 mL sample drawn into a syringe containing 10 mL of Saltzman's reagent (15).

Nitrogen oxides are so tenaciously adsorbed on glass surfaces that rinsing with Saltzman's reagent has been found necessary to obtain reliable results. Adsorption of isocyanates on glass, metal or plastic surfaces occurs to such a degree as to preclude preparation of static calibration mixtures even under strictly anhydrous conditions (8).

Positive errors can be included when evacuated double entry gas sampling tubes (fitted with 2 stopcocks) are used for sample collection (7,16). In this case, after release of the vacuum, additional sample is drawn through the tube for 3 min before closing the stopcocks. Recovery of xylidine increased to 120% in the 50 to 70 ppm range under these conditions. This error was increased to 40% by adding glass beads to the sampling flask to increase the surface area (31). These conditions can be avoided by collection of the pollutant in an impinger assembly (Greenburg-Smith, midget or micro).

In dynamic systems, prolonged equilibration is required before dependable calibration can be assured. Calibration of toluene diisocyanate (TDI) in the TLV range required passage of the air-TDI vapor mixture through a mixing chamber composed of a 6' (1.8 m) length of 1/4" (6 mm) copper tubing under strictly anhydrous conditions for 60 to 72 h to reach equilibrium. This phenomenon was not entirely caused by the film of water normally adsorbed on the inner walls of the tubing, as the system had been carefully heated under vacuum to remove volatile impurities before use (8).

Adsorption is a critical factor in making up standard mixtures for phosgene calibration at concentrations below 1 ppm even after meticulous removal of the adsorbed water film. The use of metal cylinders especially must be avoided. In the development of a continuous monitor for phosgene concentrations below 5 ppm, decomposition in metal (steel, stainless steel, copper, aluminum, etc.) or glass tubing was found to be so extensive that their use in the sampling system could not be considered. In this case polyethylene tubing was acceptable. Water adsorbed to the contact surfaces was probably responsible for the losses as HCl was observed to be a by-product. Catalytic de-

Table 1:I Static Calibration Mixture
Benzene Loss by Absorption on Container Walls
Borosilicate Glass Temperature 73 $\pm$ 5°F (24 $\pm$ 3°C)

Time Elapsed Hours	Benzene PPM		Percent Loss	Container Conditioned		Remarks Inside Surface Approx. 800 Sq. In. (0.52 m ²)
	Added	Found		Time Hours	Benzene PPM	
0.5	100	56	44	0	none	
0.5	100	67	33	0	none	
16	200	100	50	0	none	
0.5	100	84	16	16	100	
1	—	78	22	—	—	
0.5	100	87	13	16	200	
0.5	100	107	-7	—	—	Swept, 2nd 100 ppm added
0.5	200	128	36	—	—	3rd 100 ppm added
0.5	50	56	-12	—	—	Swept, 50 ppm added
0.1	100	75	25	0	none	Idle 30 days
0.25	—	72	28	—	—	
0.5	—	55	45	—	—	
1.0	—	55	45	—	—	
4.5	—	48	52	—	—	
24	—	42	58	—	—	
192	—	0	100	—	—	

Note: Five-gallon (19-L) borosilicate glass carboy jacketed with a canvas cover. Calculated quantity of benzene was added and mixed by rotating a polytetrafluoroethylene (PTFE) paddle and sampled at the indicated time after addition. In two cases indicated by 16 h (column 5), equilibrated by standing overnight, then refilled.

composition on anhydrous surfaces cannot be ruled out, however (17).

In a study of the efficiency of midget and micro-impingers by collection of aniline from air, loss by adsorption in the glass batch-type generator (5 to 15% in the 100 μ g range) was encountered unless the system was heated to 60°C in a hot water bath. Even greater were losses due to absorption by gum rubber tubing. Even 3-inch (8-cm) connections introduced significant errors (10% loss) but the losses were not entirely proportioned to hose length. These losses increased as the age and number of uses increased; *e.g.*, recoveries decreased from 83 to 57% over a period of 8 days (18).

Failure to recognize the effect of adsorption on container walls delayed the determination of the half-life of SO₂ during a study of the fate of SO₂ in the atmosphere at Syracuse University. The half-life was found to be unexpectedly short even after correction for adsorption effects (19).

Tubing through which high concentrations of tetraethyl lead vapor have been drawn will become a source of contamination when "clean" air is passed through the

system. These effects are eliminated by use of all-glass systems connected by standard taper, ball joints, "O" ring seals or polytetrafluoroethylene (Teflon) compression connectors. Chemical transformation occurred when sampling oxides of nitrogen through sampling probes from a simulated combustion source. The species interconversion or loss was a function of temperature and sample probe material and was found to be a function of the air-fuel ratio of the input sample. Glass sampling probes were found to be inert, while the stainless steel probes were found to be reactive (53).

The adsorption of traces of analytes from liquid solution onto container walls is probably somewhat more widely recognized but certainly requires review to reemphasize the importance of this source of errors. Heavy metal ions, especially lead, mercury (22), silver (20,21), cadmium, and antimony, are readily adsorbed by glass and in some cases by plastic surfaces. Lead in urine specimens, for example, is adsorbed to an extent sufficient to introduce significant errors after storage for more than 3 days in borosilicate glass specimen bottles

(23). This effect probably is produced by bacterial fermentation which elevates the pH by ammonia release, and coprecipitates lead with calcium and magnesium phosphates on the glass. In the early stages the precipitate is invisible but nevertheless may contain a significant fraction of the total lead in the sample. In many cases these losses can be held within tolerable limits by reducing the pH to 2 or less with nitric acid. This adsorption loss can be a critical factor in the storage of standard reference solutions and must be taken into account when collaborative testing programs are set up. Efforts to prepare stable aqueous standardizing solutions of metallic mercury met with failure unless excess mercury was maintained in contact with the liquid phase. Furthermore, centrifugation is necessary to remove dispersed microdroplets (or HgO) if shaking is employed to accelerate saturation, and a relatively large temperature coefficient of solubility must be taken into account. In spite of these limitations, this saturated aqueous mercury solution can be employed as a convenient source of mercury for calibration by comparing recovery from a collecting system with direct analysis of the standard (22).

Filtration of samples collected in a liquid medium should in all cases be avoided if the desired contaminant is to be recovered quantitatively in the liquid phase. Not only will the contact surfaces of the filter funnel and the filtrate receiver adsorb trace materials, but the filter medium (paper, membrane, asbestos mat, glass fiber, micro metallic, etc.) may be highly adsorbent (14). Also, the insoluble fraction removed may retain the analyte as in the case when calcium phosphate is separated from a mixture containing traces of soluble lead compounds (24).

The problem of adsorption on glass surfaces previously discussed requires re-emphasis in the case of porous glass used either as a filter for particulate matter or as a diffuser for collection in liquid absorption systems. Filtration of aqueous solutions of phenanthrene, naphthalene, pyrene or anthracene through a porous glass plate reduced the hydrocarbon concentration as much as 40% (14). When ultraviolet (UV) absorption is employed for analysis, errors from background absorption produced

from UV-absorbing materials leached from the filters and light scattering from suspended particles must be taken into account (23). A base-line technique for correction is often required as the solute often alters the light scattering and sedimentation properties with the result that the reference blank and the sample are no longer exactly comparable. If removal of sediment is necessary, filtration through polytetrafluoroethylene (PTFE) mat or decantation after centrifugation will minimize losses. In any case, potential losses at each step must be recognized and determined.

Rust, scale and other metallic corrosion products can introduce serious errors through adsorption of air pollution components and should be meticulously scoured out of systems used for air sampling, storage or analysis. Many of these oxidation and corrosion products are highly catalytic, as illustrated by the rate at which alkyl lead compounds are oxidized by hydrated iron and bismuth oxides (25). Pipe dope used on threaded connections and oils used in cutting threads or lubricating moving parts also can present significant reversible absorption effect. Polytetrafluoroethylene (PTFE) ribbon thread sealant, which has a very low absorption coefficient, is recommended for threaded joints that require lubrication. Moving parts machined from PTFE are self-lubricating and can be sealed with modified PTFE "O" rings.

The reverse effect has been encountered also. The diffusion of HF in Conway dishes is enhanced ten-fold when silicone grease is used as a sealant for the dual concentric chambered dish cover. Fluoride can be removed quantitatively from aqueous HCl by gentle boiling in the presence of silicone fluid (26).

Adsorption of a secondary layer on container walls may completely alter the character of the surface presented to the collection and analytical system. Soaps and synthetic detergents are especially troublesome in this respect. The film of soap on glass is not removed by 5% trisodium phosphate, chromic acid cleaning mixture, or concentrated nitric acid. Only by baking out in an annealing furnace can "chemical sterility" be achieved. In the colorimetric determination of aromatic nitro and amino compounds in air, this soap film reacts with the color-

generating reagents. These adsorbed layers appear to retain nitrous acid or nitrogen oxides in a state that does not react with starch-iodide indicator or sulfamic acid but does react with the coupling agent (1-naphthyl ethylenediamine or Chicago Acid) to produce a deep orange color. This spurious color reaction produces high results, which may be sufficiently uniform to produce false calibration curves (zero intercept at some light transmittance value below 100%). To avoid this nitrite-absorbing contamination and remove residual azo dyes which may be adsorbed on the coupling flasks and spectrophotometer cuvettes, soak all glassware in 5% aqueous trisodium phosphate for several hours and rinse thoroughly with distilled water. Soak Correx cuvettes only in 37% nitric acid wash (27).

The nonabsorption of heavy metal ions on hydrophobic surfaces of PTFE and polyethylene (PE) may be reversed by the presence of certain organic anions. In trace quantities, cesium is adsorbed on glass surfaces by interaction with the layer of adsorbed hydroxyl ions that account for the hydrophilic character of glass surfaces. Addition of a salt of high ionic activity such as sodium nitrate reduces this adsorption effect. The ion exchange capacity of the glass surface can also become a significant factor at very low concentrations. The overall effect is orders of magnitude less (adsorption coefficient increases in the order PTFE-PE-glass) for the nonionic hydrophobic plastic surfaces. However, the absorption coefficients reverse when the tetraphenyl borate (TPB) ion is introduced. This reversal is due to adsorption of TPB ions by van der Waals forces on the hydrophobic PTFE and PE which then sequester cesium ions. On the hydrophilic glass surface the adsorption is ionic in character, which reduces the binding sites for the cesium ions (28).

Lubricants in stopcocks and ground glass joints must be avoided as these materials are a source of contamination and/or absorb those constituents of interest either directly from the air sample or from the liquid reagent system after collection. Teflon plug cocks for stopcocks and sleeves for joints provide seals that require no lubrication or liquid-phase sealant for air-tight joints.

Fungus growth within an atmospheric

analyzer can be a particularly vexing problem. Fungus within an SO₂ analyzer that uses liquid reagents results in an indicated concentration lower than that actually present. Fungus effect can be minimized in an analyzer by: (a) Keeping the instrument running continually, (b) cleaning periodically, and (c) using a small amount of fungicide in the reagent. A fungus problem will usually be found on dynamic calibration.

1.3 SOLID ADSORBENT COLLECTING SYSTEMS. Extended surface adsorbents such as activated silica gels, alumina gels, charcoals and carbons, glass beads, and spheres offer a very attractive solution to the collection and concentration of gas phase pollutants from atmospheric samples. However, the tenacity of the forces that provide quantitative extraction of the components sought may prove to be self-defeating when quantitative removal of these same components for analysis is attempted (54). Failure to recognize and equate these factors may, and has, produced serious errors. Mercury, either as metallic vapor or as volatile organic derivatives, is not removed quantitatively either by heating or elution with liquid reagents from silica gel. The same deficiency has been encountered in attempts to use activated charcoal for collection of alkyl lead compounds.

Since the polarity of the adsorbed element or compound determines the binding strength on silica and alumina gels, components of high polarity will displace components of lesser polarity—the principle of chromatography. Therefore, in attempting to collect relatively nonpolar compounds such as benzene, the presence of coexisting polar compounds such as phenol, acetic acid or ammonium chloride must be recognized as a source of interference in the use of solid adsorbents for sample collection. Under high humidity conditions the adsorbent may be deactivated by saturation with water vapor (23). Activated charcoal exhibits this effect only to a minor degree. Aromatic hydrocarbons are not removed quantitatively from charcoal (32).

Selective displacement may be taken advantage of in eluting the sample from the adsorbent. An inert carrier gas with or without heat, for example, may be used to separate a mixture of aromatic hydrocarbons from a silica gel column. Proper

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choice of eluting solvent may well serve to separate components by liquid extraction also. An example would be separation of nitrobenzene from aromatic hydrocarbons for analysis by UV spectrophotometry.

Liquid eluents must be chosen and used with care however as decrepitation of the gel may occur. The finely dispersed fragments can produce difficulties in the determination of optical density in the final step of the analysis. This problem is especially acute in UV spectroscopy. Usually this effect is caused by the heat generated when a polar solvent such as ethanol is introduced. Dilution with a nonpolar solvent such as isooctane will usually avoid this difficulty (23). Elution from charcoal is dependent on molecular size rather than polarity (CS_2 , CCl_4 , etc.)

Change in air flow resistance due to either swelling, shrinking or channeling of the adsorbent is sometimes encountered. Collection of alkyl lead vapor in crystals of iodine was plagued with channeling and shrinkage due to iodine sublimation. Addition of a head of fine inert sea sand eliminated the problem by providing enough pressure to keep the iodine crystals compacted without appreciably altering the pressure drop across the column (29).

Many dusts are highly adsorbent and often carry a significant gaseous component present in trace amounts such as SO_2 at concentrations below 0.5 ppm. If a prefilter is installed in the collection of trace contaminants, particles collected on the filter must be analyzed also if a quantitative accounting for the total contaminant present is desired (30). In some cases such as in the analysis for lead in air, the filter would be analyzed separately from the fraction collected in the liquid reagent to provide a differential analysis for organic and inorganic lead. This is especially important in cases where the TLVs for the two forms are different and coefficients must be used for hazard evaluation (1).

The use of pretreatment or guard systems such as solid dehydrating agents to remove moisture, soda lime to remove CO_2 , or sulfuric acid on silica gel to remove NH_3 interference, and liquid scrubbers such as an oxidizing solution to remove reducing interferences such as SO_2 and H_2S before adsorption of the desired pollutant can in-

troduce serious errors (23,35). Silica based and anhydrous CaSO_4 granules may partially or completely adsorb the subject element or compound as well as eliminate the interference in the air sample. Careful calibration of the collection system under use conditions is critical when interferences must be eliminated from the air sample by pretreatment before collection of the desired component.

1.4 DIFFUSION EFFECTS OF PLASTICS. Diffusion outward from the air sample, into, or through plastic containers or tubing probably is one of the most frequent sources of error encountered in air analysis. These irreversible losses are in addition to the reversible surface adsorption effects noted under Section 1.2. Elastomeric materials, which include natural rubber, neoprene and plasticized polyvinyl chloride (PVC) are especially troublesome. (See section 3.1 on Permeation Tubes—Dynamic Calibration.)

The plasticizers employed in the manufacture of flexible tubing from PVC type polymers may act as cosolvents to remove organic components from air samples by absorption. This mechanism is often temperature sensitive and to some extent may be reversible in a fashion resembling gas chromatography column packings. In a study of the recovery of aniline vapor from air, losses as high as 1.7%/cm of exposed gum rubber tubing were encountered (18). Similar experience has been obtained with alkyl lead and mercury compounds, aromatic hydrocarbons (benzene, toluene, etc.), chlorinated hydrocarbons, and aromatic nitro compounds. An all-glass system with joints and connections fitted with PTFE gaskets and the shortest possible path between air sample intake and the collecting medium to a great extent eliminates these losses. However, adsorption effects must not be neglected even in all-glass systems.

The permeability of even the inert plastics exemplified by PTFE has been employed for the constant rate generation of calibration mixtures of certain trace components such as SO_2 , HF, COCl_2 , aromatic hydrocarbons, etc. Although this property would be undesirable in the collection of air samples, the magnitude of loss by diffusion through short inert plastic sampling lines

(PTFE, polyethylene, polypropylene) can be neglected. However, when samples are stored in plastic bottles or bags, diffusion may become a controlling factor. Indication of contamination from fluoropolymer bags used in air pollution studies has also been noted (45). Plastic containers must be used to facilitate collection or transportation; then the diffusion rate factor must be determined for each component sought over the concentration and temperature range to be encountered and a correction factor applied to correct for the time interval between collection and analysis. Aluminum foil-lined polyester bags (Mylar), which materially reduce diffusion losses, are available for CO₂, CO, water vapor and some other gases but are not recommended for the highly polar gases (SO₂, NO₂ and O₃) (36). Polyester film in 2-mil (50 μ m) thickness is considered the best all-around material for sample-bag fabrication. However, polyvinylidene chloride (Saran) film (1-mil [25 μ m] thickness) which has been pre-aged to reduce outgassing of compounds from the plastic into the contents of the bag has been widely accepted on the basis of inertness to many common gases and vapors, typical high tensile strength and heat sealability. Data relative to gas transmission is summarized in the following table.

Gas Transmission: cm³/m² μ m d atm at 23°C

O ₂	32-43
CO ₂	150-236
N ₂	4.8-6.3
Air	8.3-17.3

Water Vapor Transmission: g/m² d

at 24°C—0.31
at 38°C—3.05

For ultimate chemical inertness fluoroplastic film bags are available. Laminated plastics—polyethylene on polyester, polyethylene on cellulose acetate and polypropylene on either base—used in packaging foods offer interesting alternatives for gas-impervious containers and a choice of surface for contact with the air sample. Semiflexible 10-L polyethylene bottles with greater wall thickness also offer a solution

to the diffusion problem and are convenient for preparation of calibration mixtures. (See Static Calibration, section 2.1.)

Inward diffusion also may be encountered and may create problems in shipment or in prolonged storage. Samples taken in a low humidity environment may be altered by inward diffusion of water vapor when the plastic container is stored in a high-humidity climate. Solvent vapors, gasoline vapors, and gasoline and jet engine exhaust components may contaminate the sample in shipment and show up as unexplained peaks in gas chromatographic analysis, or introduce interferences in wet chemical procedures. Shipment in air-tight containers would be a reasonable precaution to avoid contamination from this source.

Although obvious, the testing of all plastic containers, especially thin film bags, for gross leaks before use must be emphasized. The absence of pin holes, gaps in the seams, and leakage around the sampling port and in the retaining valve or septum assembly must be verified before each use. This examination may consist of full inflation under slight positive pressure (2 to 3" water, 0.5–0.8 kPa) under water or in a rigid sealed container with the air line introduced through a bulkhead fitting and connected to a water manometer. Soap solution stabilized with glycerol may be painted or sprayed on for leak detection to pinpoint "micro" leaks.

1.5 MECHANICAL DEFECTS OF SAMPLING EQUIPMENT. As well recognized as this source of error has become, leaks and faulty seals remain one of the major contributors to unreliable results. Any system used to collect air samples, to remove contamination components by absorption, or to calibrate such systems must be checked for leakage, either inward or outward depending upon whether the air stream is pushed or pulled. Perhaps the most expedient procedure involves either an increase or decrease of the pressure in the system within limits of the maximum operational range of the system, by closing off tightly and measuring the rate of loss of either pressure or vacuum with a water manometer. Pinholes in glass seals, especially ring seals; plastic or metal tubing; joints that do not seal perfectly or are insufficiently gasketed; "O" rings that may be cracked, loose

or hardened or lack a sealing lubricant; loose glass-to-rubber or plastic tubing connections; dirt in glass-to-glass joints; glass plugcocks that do not perfectly mate with the stopcock barrels; poor valve stem packing or insufficient sealing pressure; and poorly sealed threaded fittings provide the most frequent leak sources. Pinholes in stainless steel valves, reducing valve diaphragms, and two stage regulators also have contributed their share of difficulties (10). A review of the requirements for gases to be used with gas chromatographic systems and the overall gas sampling requirements is valuable in determining how pollutant standards should be handled (55).

Fouling of rate meters and total volume meters by particulate matter or corrosion products often is not so obvious and requires eternal vigilance to minimize. Orifice type and positive displacement meters and rotameters should be protected by an efficient filter to prevent reduction in orifice opening, erosion of moving parts, or complete plugging. Rotameter calibration can be shifted significantly by dust accumulation, and plastic tube models require protection from solvents which soften or render the bore tacky. Operating characteristics of flow meters were discussed (56). Metal floats are attacked by corrosive gases and some reagents such as iodine may in time render the tube body opaque. Corrosive gases and aerosols must be rigidly excluded from total volume and positive displacement meters to maintain calibration validity and integrity of moving vanes or pistons. Liquid aerosols may be especially troublesome by forming a liquid film that by surface tension and capillary effects reduces air flow in small orifice throats, interferes with the movement of rotameter floats, and increases the resistance to movement of critical components in positive displacement meters. Galvanic corrosion from airborne electrolytes under high-humidity conditions can be a problem with metal components.

Flow meters based on thermal conductivity (hot filament type) also are affected by dust and reactive gases, but in a different fashion. Corrosion will alter the electrical conductivity and leads ultimately to failure of the element, and coating by inert material will reduce heat transfer and ultimately

reduce the response of the instrument. Chlorides and lead are especially destructive. The volumetric meter and the pump or aspirator sub-assembly of any air sampling system should be adequately protected by appropriate traps that are renewed frequently. Activated charcoal granules (5 to 10 mesh) probably offer the most efficient protection for all but a few destructive gases and vapors. Cellulose acetate membrane filters (0.8 μm pore size) will effectively remove over 99% of the dust particles that might affect instrument performance without introducing prohibitive pressure drop (37).

The orifice in impinger-type collecting devices, and the interstices in porous glass diffusers are also vulnerable to partial or complete closure by particulate matter and should be protected by a filter when the solid fraction is not an integral part of the sample. Porous glass diffusers may accumulate particles that are not removed by conventional cleaning procedures after repeated use (10). Also irreversible retention of analyte sometimes occurs. In the collection of alkyl lead vapors from 5 to 10% of known quantities added to calibration mixtures were retained in a pencil-type diffuser substituted for the impinger tube in the standard midget impinger sampler (31). The increase in pressure drop produced by the decrease in free cross-sectional area will contribute to volumetric sampling errors, and may significantly reduce the collection efficiency. In dust collection by impingement, this factor becomes critical.

Pressure drop must be taken into account when a sampling system is calibrated volumetrically. Rotameters and orifice type meters should be calibrated with the pressure reducing or elevating components in place or volume correction made to compensate for the deviations from ambient conditions (38). In systems based on filter collection, the pressure drop across the system may steadily increase as the filter medium loads up with retained particles. Under these conditions an averaged correction may be within tolerable limits of volumetric error. Changes in barometric pressure usually introduce only marginal error if ignored. (Deviation of 2 from 101.3 kPa will introduce a 2% volumetric error).

In the use of cold traps to collect contami-

nated air components (freeze-out technique), consideration must be given to the possible accumulation of ice crystals in the delivery tube. This possibility can be minimized by introducing the sample through a side arm into the chamber and exhausting through the inner tube, i.e., reverse flow (11).

1.6 PARTIAL VAPOR PRESSURE EFFECTS. In collection systems that depend exclusively upon solubility of the component sought in a nonreactive solvent rather than retention by chemical reaction to a nonvolatile derivative, the amount absorbable is determined by the partial pressure of absorbate. The ratio of the air sample volume to the volume of absorbant is a critical factor in collection efficiency and there is a limiting value of the ratio for any system that cannot be exceeded if a given degree of efficiency is to be attained. In the case of acetone in water the limit would be approximately 5 L of air through 20 mL of water aliquoted between two bubblers (39). In the case of mesityl oxide, which has poor solubility in water to begin with, absorption efficiency would be very poor although relative volatility is low. This is especially critical where a low TLV is involved, as large volumes of air would be required. The formation of lower-boiling binary and ternary mixtures also can limit absorption. Failure to collect benzene vapor in ethanol even when chilled undoubtedly can be attributed to revaporization of the higher vapor-pressure ternary alcohol-benzene-water mixture.

In general, the vapor pressure of the volatile analyte should be reduced to the lowest practical limit by refrigeration to promote efficient collection. In many cases a freeze-out trap, without solvent, in solid carbon dioxide, liquid air, or a mixture of liquid air and ethanol proves to be a more effective procedure (11).

Sampling in the region at or close to the vapor saturation limit can produce serious errors by condensation in sampling lines. This condition is most likely to be encountered when collecting samples of air at temperatures above ambient and may involve condensation of water vapor which in turn will entrain or dissolve a significant portion of the components present in the sample under high humidity conditions.

1.7 SOLUBILITY EFFECTS. Limitation may occur in cases where chemical reaction should but actually does not occur readily. The classical example is the failure for SO_3 to be absorbed in pure water. The collection of aniline vapor in dilute aqueous HCl is not quantitative; therefore, chilled alcohol is the medium of choice (13). Until the reaction is initiated by a catalyst, fluorine gas will bubble through distilled water without reacting (to produce F_2O and HF) although once initiated, the reaction may be violent. Other examples that can be cited are failure to obtain absorption of alkyl mercury vapor in acid permanganate, and the less-than-quantitative absorption of alkyl mercury and lead compounds in aqueous iodine reagent (22). The assumption that a given contaminant will be collected based on its reactivity with the reagents can be disastrous if calibration of the system under use conditions is omitted.

If the product from the reaction of the analyte with the collecting reagent has a very low solubility product, losses are to be expected unless care is exercised to avoid the saturation region. The precipitate may adsorb to the container walls, orifice tube, or diffuser surfaces or fail to react in subsequent color development. If the solid phase is finely dispersed and has a refractive index approximately the same as the liquid system, the losses may not be observed; e.g., silica in chloroform.

Conversely, if the collecting train does not include a prefilter, particulate fractions may go into solution and produce false positive results or interferences; e.g., lead dust or fume in the collection of tetra-alkyl lead vapor (30).

1.8 TEMPERATURE EFFECTS. The physical effects of heat on the collection system under normal operating conditions is usually ignored without introducing appreciable difficulties. However, at the design and development stages, attention must be given to the upper and lower temperature limits to be anticipated. At low temperatures components of the liquid reagent may crystallize out or the solvent may freeze. Frequently, a change of solvent will solve this problem; e.g., aqueous KI_3 versus methanolic iodine reagent (22,29). Close attention must be given to possible formation of ice or reagent crystals in orifices and po-

rous glass diffusion beds when operating near the freezing range.

On the other end of the temperature scale, excessive evaporation may limit the choice of reagents, or sampling time may be reduced below practical limits (again alcoholic versus aqueous iodine reagents). Refrigeration usually can be applied in fixed station or mobile systems to alleviate this problem. A portable assembly that derives its sampling suction from the expansion of difluorodichloromethane vapor through an aspirator also provides useful refrigeration by forming the vaporizer coil into a cavity which will receive a midjet impinger (11). In some cases a colorimetric system can be adapted to a nonvolatile solvent, as illustrated by the use of diethyl phthalate as a solvent for the detection of phosgene in trace ranges over extended periods of time—4 to 6 h (40).

As noted in section 1.6 (Partial Vapor Pressure Effects) the temperature coefficient may be quite large and preclude collection by solution in a solvent above a critical temperature, above which no collection will be obtained, and which may be only slightly above room temperature (39). In general, the adsorption capacity of solid adsorbents is decreased exponentially with rising temperature, which may limit the thermal operating range. Temperature coefficients should be determined before sampling is attempted (23).

Excessive evaporation at elevated temperature aggravates the scavenging problem for the metering devices and pump downstream from the collecting unit. Larger activated charcoal cartridges and more frequent replacement must be provided. Generation of volatile components from the reagent will create an even greater problem, *e.g.*, HCl from aqueous iodine monochloride solution (1).

A word of caution with respect to liquid-air cooled freeze-out traps—liquid oxygen from the air sample may condense and create an explosion hazard that may not arise until the trap is removed from the cold bath. In other subambient systems, moisture may be a mixed blessing. Condensation of water vapor often assists with the retention of contaminants, but in the end interferes with the final analysis. Analytical systems that require anhydrous conditions

cannot be employed for air analysis unless dehydration can be employed before the sample enters the collecting system without loss of the analyte; *e.g.*, CO by infrared.

Although temperature extremes sufficient to alter the mechanical performance of collecting systems are not often encountered, this factor cannot be ignored when arctic or tropical desert conditions are encountered. Batteries for power supplies must be chosen with these conditions in mind. Piston-type pumps may bind or blow by; check valves may become inoperable; volumetric factors will require recalibration; metering valve and plug cock tolerances may shift significantly; elastomeric components lose elasticity beyond tolerable limits (tubing, "O"-ring seals, check valves, gaskets, etc.); and diffusion through plastic containers, especially sample collection and storage bags may become excessive.

Instruments that require electrical circuitry or electronic components for generating analytical data will be affected by temperature extremes and must either be provided with thermal insulation or compensated for altered performance. Extrapolation of response obtained in the range of normal ambient temperature is not a reliable substitute for actual testing under use conditions.

1.9 VOLUMETRIC ERRORS. Rate meters of all types require frequent calibration to detect and correct drift or shifts in reference points, (see Section 1.5). Portable instruments should be checked every 40 to 50 h of operation with at least a secondary standard (wet or dry test meter or glass rotameter) which in turn is compared frequently with a primary standard of the positive displacement total volume type (spirometer).

In volumetric calibration procedures, the pressure drop across the instrument being calibrated and across the system in which it will operate must be determined and appropriate corrections applied (see Section 1.5 for effects of fouling). When filter systems are used, the increasing pressure drop and decreasing sampling rate that develop as the filter loads must also receive consideration. Unnecessary restrictions to free sample flow also produce undesirable pressure drop increments that may limit the range of the sampling system.

Temperature effects do alter volumetric calibration significantly if the system is operated under ambient conditions more than $\pm 20^{\circ}\text{C}$ from the original calibration temperature conditions. A high degree of volumetric accuracy requires determination of the instrumental temperature coefficient as well as correction of the sample volume to standard temperature and pressure (25°C and 101.3 kPa).

Variability in certain types of pumping systems, both between units of the same model or within any given unit, may prevent constant rate sampling within tolerable limits over extended time intervals. Direct current motors are especially vulnerable to rate fluctuations unless effective control circuits are incorporated in the design. Manually operated squeeze-bulb devices seldom deliver reproducible volumes and should be avoided if better than semi-quantitative accuracy is required. Positive displacement syringe-type hand pumps are capable of a high degree of volumetric accuracy and are to be preferred (41).

1.10 OBSERVATIONAL ERRORS. The optical judgment of color shade and optical density is one, if not the most, variable factor to be considered in air analysis. Reasonably accurate results can be attained if the individual using a color comparator is willing to calibrate the instrument for his own personal use, but he must take into account the quality and intensity of the light available for illumination of the comparator, day-to-day variations in his own optical acuity, shade changes introduced by interferences, and stability of the color standards. Field kits based on optical color standards provide a definite, valuable service not available by any other means, but the unavoidable sacrifice of accuracy and precision must be carefully weighed when planning and evaluating investigations based on such equipment (17,29,42).

Optical acuity and judgment also enters as a factor in the detection of air contaminants with the length-of-stain type of detector tubes. Diffusion, migration and trailing may so obscure the stain front as to render accurate reading very questionable. Again individual calibration with knowns can be quite helpful in providing a basis for judgment.

Errors in the application of volumetric glassware are not unique to air sampling

and are recognized by those familiar with analytical laboratory techniques. Such items as "to deliver" versus "to contain," delivery time for pipets, whether or not to "touch off" or "blow out" the drop retained in the pipet tip, capillary effects in meniscus reading, error introduced by a water-repellant film on the glass, and chipped buret and pipet tips should be too well recognized to require further elaboration. The relative accuracy of the original glassware calibration may be overlooked. Most laboratory-ware manufacturers adhere to two standards: precision-grade (within NBS tolerances) and laboratory grade (twice precision-grade tolerances). The choice of buret, volumetric or Mohr pipet or graduated cylinder will be determined by the relative accuracy required in the measurement. That is, volumetric pipets or a buret would be used to measure out standardizing solutions whereas a graduated cylinder is suitable for making up most reagents.

Reading instruments that indicate by mechanical movement, such as a rotameter float, voltmeter needles, etc., involves critical judgment also. Whether or not to read the top, bottom, or center of a rotameter float, parallax effects in observing meter needles (some of the better precision meters provide a mirror background to eliminate this problem), capillary effects on the meniscus of liquid-filled manometers, and scale units, illustrate several of the more common observational errors. The increasing relative error inherent in any instrument as either the minimum or maximum range limits are approached may introduce serious observational error. The upper and lower 10% of any instrument's range should be avoided, and if possible the capacity should be so chosen as to permit operating only in the center 50% of the overall range.

1.11 OPTIMUM SAMPLING RATE. Calibration of any sample collection device should include a sufficient range of rates to delineate the optimum range consistent with the efficiency demanded of the system and to establish the penalty incurred when the rate exceeds or falls below these limits. Failure to include this factor has contributed serious errors (dust collection by impinger) which are inexcusable. Collection

efficiency cannot be assumed; it must be determined by calibration.

The design of the collector in liquid reagent collection systems for gases and vapors is, in most cases, not critical if in addition to acceptable collection efficiency the design provides retention of entrainment (baffles), sufficient freeboard to retain bubbles and foam, and interchangeability of component parts, and the device is easily assembled, dismantled and cleaned (29). Only in those cases that involve surface reactions and adsorption will a "fritted" or porous glass diffusing element perform more efficiently than a standard single-orifice impinger (10,12). Frequently these diffusers create problems such as retention of analyte, excessive frothing and foaming, variable pressure drop, fragility, and particle retention that are not encountered with the use of impinger-type absorbers.

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2nd edition
Subcommittee 9
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2. Calibration Procedures

2.1 PREPARATION OF STATIC CALIBRATION MIXTURES.

2.1.1 Introduction. Static calibration mixtures are prepared by introducing a known weight or volume of contaminant into a given volume of air (1). Generally the mixture is held in a container of fixed dimensions, but flexible chambers may be used.

2.1.2 Measuring the Contaminants. Liquid contaminants are commonly introduced into the calibration system with a microsyringe or micropipet. Gaseous materials are handled with a gas-tight syringe.

A pure gas sample may be obtained from a lecture bottle by either of the methods shown in Figure 2:1. The first technique involves attaching a lecture bottle septum directly to a regulated cylinder (2). Just enough gas to purge the system is bled through the pressure relief valve. The sampling chamber can then be maintained at about two atmospheres with no additional loss of gas to the atmosphere. Contaminant gas is then obtained through the septum using a gas-tight syringe.

In the second method, the pure gas is passed slowly through a T with a septum over one arm. The gas then flows from a dry midget impinger into one containing 10 mL of water. The pure gas is then with-

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