THE ANALYSIS OF AIR POLLUTANTS

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Head of the Main Laboratory of

Österrichischen Stickstoffwerke A.G., Linz/Donau

Translated by R. KONDOR

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Preface

Preventing the pollution of the most important part of our environment, the air we breathe, has become a complex and extremely serious problem, because of increases in population density, traffic and industrialization. A large section of the public and specialists in radically different professions — chemists, physicians, engineers, biologists and jurists — are concerned with this problem. But before discussing pollution, and taking the necessary measures, we must have an accurate knowledge of the types and concentrations of the pollutants from various sources. It is the task of physical and chemical analysts to gather evidence of these pollutants at the work site, in industry and trade. The results of air analysis must be known to protect the workers from injury and the products from contamination.

Every chemist, whether in research, industry, or in public institutions, will have to deal with air pollution problems and must be informed about the required techniques.

Information on air analysis is found throughout the literature. Some important methods of investigation are included in the Directives of the Working Group "Prevention of Air Pollution" (Reinhaltung der Luft) in the VDI (Verein deutscher Ingenieure) and the ICI Manual. Collections of methodological directives have been published by international organizations such as the OECD (Organization for Economic Cooperation and Development) and IUPAC (International Union of Pure and Applied Chemistry). The standard formulas of US and British trade organizations are also available. Many books on gas analysis contain sections on air, and papers on air analysis are found in numerous periodicals.

It is an exciting task, for this author, to survey a field of analytical practice which has such great contemporary interest. The task is all the more relevant since the analytical methods, and even the sanitation and legislative angles in many cases are still under discussion. Thus, textbooks in this field cannot present established facts and must include personal opinions to move discussion forward. However, analysis of air pollution cannot be delayed until the theory is worked out since solutions are urgently needed now all over the world.

The author's experience as chief chemist of a large chemical plant on the fringe of a densely populated residential area has given him comprehensive insight into the air pollution problem. He lectures at the University of Vienna and is active in international committees for preventing air pollution.

This book contains detailed instructions for practical analysis, adapting several methods to characteristic problems. These instructions were taken from German, British and American literature. The book is not intended as an introduction to air analysis and is more for the laboratory than the office. Readers should have a basic knowledge of general analytical practice since conventional methods of titration, preparation of titers, colorimetric measurements, etc., are not described in detail.*

Wherever he works, the analyst must not restrict his activity to receiving samples, analyzing them, and handing over results. Thus a book on air analysis dealing only with the theory and practice of analytical methods would be of little value. The analyst participates in preliminary discussions with physicians, engineers, and sometimes jurists, considering the necessity for and the aim of the air analysis. After the purpose of the analysis has been established, the analyst plans the experiments, selecting the suitable method. The analyst has problems in common with the authority ordering the analysis: sanitary survey with the physician; eliminating proven emissions and their consequences with the engineer, and the legal problems with the jurist in civil service.

To facilitate discussions on problems bordering on the analyst's field, and thus to ensure the success of his work with the physician, engineer or jurist, the first chapter of the book contains an introduction to relevant sections of sanitation, technology and legislation. The origin and extent of air pollution, combined with meteorological factors, are weighed against the damage it does to man and plants. From these facts the necessary legal limits (MIK- and MAK-values) are derived. Also, a survey of basic technical possibilities for preventing air pollution is presented.

A general chapter on air analysis gives a comprehensive survey on how to plan the experiments and sampling procedures. We distinguish between long-term procedures and individual determinations. The latter can be carried out either as orientative rapid tests with monitoring instruments or as accurate determinations of the gas levels. In accordance with the modern trend toward continuous recording, we discuss the development of the appropriate instruments first generally; gas chromatography is treated in a separate section. Some sections explain the testing of new analytical methods and the preparation of test mixtures from gases.

In the chapter "Special Topics" we shall discuss first the pollutants in the form of dust and smoke, frequently a no man's land between physics and chemistry, and the numerous analytical methods applied. This chapter includes a short introduction

* The book "Analytische Chemie in der Industriellen Praxis", Frankfurt, 1964, written by the present author, deals with general problems of analytical practice.

to investigating radioactive pollutants. The more important gaseous and vapor pollutants are then comprehensively dealt with. Sections on the more important pollutants discuss their analytical chemistry, their origin, propagation, hygiene and the present state of techniques for waste gas elimination. The analytical treatment should be comprehensive enough to supercede the original literature in most cases. The analytical part facilitates the selection of appropriate methods, it gives the concentration to be expected, the presence of other pollutants and the instrumental means available.

The author has utilized the large store of information and experience in this field available in his firm. He expresses his thanks to the management for their permission to publish these data and for their support. He also wishes to thank his co-workers who helped him in obtaining the necessary data. The author is grateful to Dr. Megay and Dr. Frenzel for information on radioactive pollutants.

Any suggestions and critical remarks from workers in the field of air analysis will be most welcome by the author. It is the author's hope that the book will be useful to many of his colleagues and that repeated analysis will show the right way to prevent air pollution in towns and industrial plants.

Linz, March 1968 *Wolfgang Leithe*

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General

1.1 Introduction

The air around us (the atmosphere) is the most important part of our natural environment. Terminate the supply of breathable air and death follows in short order. Under natural biological conditions, breathing is not at all hazardous, provided the air is of proper and uniform composition. Human activity, however, has polluted the air with biologically harmful substances and it is only in exceptional cases that this pollution is still insignificant. Recently, there have been occasions when the supply of satisfactory air for breathing has been actually endangered. Unfortunately, our senses cannot adequately evaluate the quality of the air.

Our sense of smell does not respond to all harmful air pollutants, such as, for example, carbon monoxide, carbon dioxide, nitrous and nitric oxides. Certain toxic substances, such as hydrocyanic acid, though perceived when present in very low amounts, do not induce defensive reaction mechanisms. Further, the irritative and harmful effects of some gases do not occur simultaneously; in the case of ammonia first the organ is irritated and the harmful effect occurs afterwards, while the opposite trend is observed for SO_2 and NO_2. Moreover, certain limits are imposed on the quantitative evaluation of air by smelling. Apart from the fact that the odor-sensitivity of humans to many air pollutants varies, one can get accustomed to some toxicants, such as hydrogen sulfide or nitric oxide. We have no sensory organ at all for radioactive pollutants, which have become increasingly important over the past decades.

These examples show that with the ever increasing use of toxic substances and the increase in air pollutants, both as far as their type and concentration are concerned, additional means for their evaluation had to be found. Initially, empirical measures were employed, such as the observation of a burning candle which indicated by flickering or extinguishing an excessive carbon monoxide level, or the behavior of song birds in hazardous places which indicated penetration of carbon monoxide.

Later, chemical analysis and physical measurements were used to detect and evaluate pollutants.

The tasks of the investigation of air can be roughly divided into two groups:

a) investigation of the free atmosphere in residential areas in the interest of the whole population, and of the air in agricultural regions in order to assess damages to plants and injuries inflicted upon animals;

b) inspection of work places in factories and workshops subjected to the hazards of waste gases as a means of personnel protection.

It is noteworthy that the air in living rooms, offices, etc., where the city inhabitant usually stays longer than in the open air, is rather infrequently analyzed; it is apparently assumed that the values measured in the open air are also indicative of the enclosed spaces. This assumption is by no means true; thus, for example, a certain measured sulfur dioxide level rapidly decreases in a room where lime-containing paints have been used. The same should apply to nitrogen dioxide, hydrogen fluoride and other gases which form acids with water. Furthermore, the conditions for the dust level in the outside air and the rooms adjacent thereto are by no means the same.

It is noteworthy that modern technology, though it has led to a considerable deterioration of the open air, has effected great improvement in the air quality of living rooms due to the fact that the conditions of lighting, heating and sewage removal have been bettered owing to the higher living standards of large strata of the population. Thus, for example, the influence of the soot originating from candles or oil lamps on the incidence of lung diseases can hardly be assessed today.

As in many other cases of analytical practice the results of air analysis led to numerous consequences in the fields of public sanitation, engineering, etc. On the other hand, the practical requirements imposed on sensitivity, specificity, time saving and frequency of air analysis increased and became more varied. This and the problems of chemical warfare in both world wars gave vital impetus to the systematic development of trace analysis and continuously operating automatic monitoring instruments. This interplay between analysis and practice is continuing at a rapid pace with the result that interesting and important innovations in methods of air analysis are being produced which lead to important new information and technical progress in air pollution prevention.

1.2 Definition of the terms "clean air", "air pollutants", "emissions", and "immissions"

"Clean" air, i.e., air occurring in areas sufficiently distant from places of human activity or other abnormal influences has the following composition in accordance with VDI-Richtlinie (Directive) 2104:

Air component	Vol. %	Air component	Vol. %
N_2	78.10	Krypton	0.0001
O_2	20.93	Neon	0.0018
Argon	0.93	Helium	0.0005
CO_2	0.03–0.04	Xenon	0.00001

The above directive states the hydrogen level as 0.01%. However, according to recent, more accurate data, this level is considerably lower.

With regard to the water content of air (air humidity), see page 137.

The pollutants listed below are also present and occur as traces below 1 ppm in "clean" air (see, e.g., Junge [125]):

Air component	ppm (vol.)	Air component	ppm (vol.)
CO	$(1–20)\cdot 10^{-2}$	N_2O	0.25–0.6
Ozone	$(0.5)\cdot 10^{-2}$	$NO + NO_2$	$(0–3)\cdot 10^{-3}$
H_2	0.4–1	NH_3	$(0–2)\cdot 10^{-2}$
CH_4	1.2–1.5		

These trace substances partly originate from high air layers (O_3) and are partly due to decomposition and putrefaction processes (NH_3, CO, perhaps also N_2O) or weather conditions (NO_2).

Since the number of air components is large and since they are widely distributed in low concentrations (they are often found in uniform amounts independent of the sampling site), it is frequently difficult to define the limitations of the term "air pollutants" or "foreign substances in the air".

The "Technische Anleitung" (Technical Directives) TAL (see page 7) as well as VDI-Richtlinie 2104 define "air pollutants" as "solid, liquid or gaseous substances changing the natural composition of the atmosphere".

Emissions, according to TAL, are air pollutants which enter the atmosphere after leaving the plant. The same directive defines immissions as air pollutants which occur near the source (usually 1.5 m above ground level, the upper limit of vegetation or at a height of 1.5 m above the top of a building).

VDI-Richtlinie 2104 defines emissions as solid, liquid or gaseous substances of all types and origin escaping into the outside air, while immissions are here defined as the discharge of solid, liquid or gaseous pollutants which are permanently or temporarily near ground level.

In daily usage the term "pollution" has a derogatory connotation. Accordingly, in practice we shall speak only about "air pollutants" or "toxicants" when the

substance in question occurs in disadvantageous or evidently harmful concentrations.

In this sense air pollution is defined as follows by the World Health Organization (WHO): "Air pollution occurs when one or several air pollutants are present in such amounts and for such a long period in the outside air that they are harmful to humans, animals, plants or property, contribute to damage or may impair the well-being or use of property to a measurable degree".

1.3 History of air pollution

Air pollution caused by human activity has always existed since fire was used. Further, official measures against strong nuisances of this type were introduced at an early stage. We mention, for example, the banishment of very malodorous trades to the periphery of towns and the prohibition against using "sea" coal for heating purposes in medieval London.

"Smoking stacks" were apparently considered during the earlier stages of the Industrial Revolution as the symbol of industrial activity and prosperity. However, in the second half of the last century the abundance of such stacks and the occurrence of highly repulsive waste gases in the early stages of chemical industry (especially hydrochloric acid and hydrogen sulfide from the Leblanc Soda Process) forced the authorities to take the appropriate steps. The first example of such legislation is the Alcali Act of 1864 in England.

The public and, consequently, the legislative authorities were later compelled, as a result of serious catastrophes, to set up increasingly stringent measures against air pollutions. These mishaps were not caused by the local discharge of concentrated poisonous gases in accidents but by the accumulation of waste gases from industry or heating (up to that time considered as harmless) due to very unfavorable meteorological conditions. The first large-scale catastrophe occurred in 1930 in the Meuse valley near Liège, when under the influence of a smoke blanket caused by an inversion (see page 17) the waste gases from the heavy industry accumulated to such a degree that within a few days several thousand people became sick with respiratory troubles and 60 persons met their death. Under similar conditions 17 persons died in Donora, USA in 1948. The smoke catastrophes in London were considerably larger; thus, in 1952 within 14 days the death toll following acute diseases of the respiratory tracts was about 4000 higher than during comparable intervals of other years. This occurrence was repeated in 1956 with about 1000 deaths.

The smog situation (smog is a fabricated word for smoke and fog) in the cities of Los Angeles and San Francisco, which were hitherto notorious for their agreeable weather, is not characterized by a higher mortality rate but by considerable damage to materials and vegetation. Investigation of this phenomenon by research chemists showed that automobile exhaust gases are mainly responsible and not the SO_2-

containing flue gases as in Europe. The olefins and the nitrogen oxides from these gases produced the various fog-forming respiratory irritants, the reactions being catalyzed by solar radiation. However, also in other places, the increasing nuisance caused by waste gases of all types, the damaging of forests, agricultural plants and thus cattle, as well as other objects, such as buildings, metals, textiles, etc., led to much indignation. The damages due to waste gases in the USA are estimated at about 7–10 billion dollars per year in addition to the harm to the health and well-being of the population which cannot be expressed in terms of money.

These catastrophes and inconveniences were widely noted by the public and publication media, and induced the authorities to support large-scale studies and to take preventive steps. First, large-scale research in the field of sanitation and particularly in the field of chemical analysis was conducted which, especially in the USA, was subsidized by public sources. Numerous new, adequately sensitive and rapid methods and instruments for analyzing toxicants were developed and applied. These served for collecting and evaluating comprehensive numerical data in order to establish in cooperation with physicians which qualitative or quantitative composition of the environmental air can be considered as harmless, or at which pollutant levels technical and legal countermeasures are required.

Even before large-scale attempts were made to prevent air pollution, the sanitary conditions at individual work sites were studied in highly industrialized states in order to protect workers against health hazards. The chemist's task was to develop analytical methods by means of which the pollutant concentration could be established and after that compared with the requirements set up by the occupational hygienist.

Germany was spared catastrophes of such an extent, probably because of the different climatic conditions. However, also in this country the air has considerably deteriorated due to industrial expansion, vehicular traffic and higher domestic fuel consumption. About 15 years ago intensive scientific and technical research and development widely supported by public and private sources was initiated. This not only led to stricter legislative measures but also to numerous improvements in technical processes intended to find a compromise reducing the air pollutants to bearable limits without imposing intolerable demands on industrial production or civil requirements.

1.4 Legislation and public measures

The framework of public-legal arguments pertaining to air pollution control reflects the attempts of society to reconcile two legal interests:

a) the right of each person to demand clean air and corresponding reparations for damages to health and property by foreign pollutants;

b) the justifiable requirements for heating, transportation and chances to earn money in industry.

At the present state of technology it is not yet possible to completely satisfy both requirements. Thus the authorities have to find a compromise acceptable to both parties.

In fact, the civil laws and trade regulations in force in the different states permit, in principle, the authorities to take steps against the originators of air pollutions leading to health and material damages or unacceptable nuisance, and to compel them to eliminate the inconvenience and pay for the damage. Plants to be erected require the permission of the Baurechtsämter (Board of Works) and the Gewerbepolizei (a police force controlling infringements by trade and industry). These authorities in conjunction with technical and medical experts examine the possibilities of damages caused by waste gases inside and outside the plant and prescribe the measures to be taken in granting the licenses.

The division of these official functions varies in accordance with the constitution and the legislature in the different states. Sometimes these functions are carried out by a special central authority (for example, in the United Kingdom by the Alkali Inspectorate) or by the different regional, district or local authorities. The problem of the hygienic or economical requirements is mostly left to the discretion of the authorities, who make the decisions, whenever possible, after hearing the opinions of experts; frequently experiences and corresponding decisions of other states are taken into account.

As the problems of air pollution became increasingly topical and very urgent, it was often necessary to supplement these older, mostly general, regulations with new and more stringent requirements, and to provide numerical data in order to reach, insofar as possible, a unified legal position. In the German Federal Republic this is based on the "Gesetz zur Änderung der Gewerbeordnung and Ergänzung des Bürgerlichen Gesetzbuches" ("Luftreinhaltegesetz") (Law for the Correction of Trade Regulations and Supplement to the Civil Code (Clean Air Maintenance Law)), which went into force on 1 June 1960. The frequently used para. 16 of the Trade Regulations controls the granting of licenses to erect plants, which may lead to inconveniences, risks and nuisances to local residents (the category of such plants was expanded by subsequent legal regulations, for example, those of 4 Aug. 1960). According to para. 25 of this law additional injunctions can be prescribed to licensed plants; these injunctions, however, must have bearing on the corresponding state of technology and be economically feasible for plants of this type. The "Clean Air Maintenance Law" also deals with measures to control emissions by qualified experts and the installation of suitable measuring devices. The plant in question is liable for the costs of such measures.

Another law which went into force on 17 May 1965, provides for the erection of control stations in areas in the German Federal Republic with increased accumula-

tion of air pollutants. This permits the timely detection of hazardous air pollutant concentrations by continuous recording, so that the necessary measures can be taken.

These federal laws are supplemented by state laws for protection against immissions. In this context we mention the law of Nordrhein-Westfalen, the state exposed to the greatest hazards. Finally, we mention the regulations of the "Strassenverkehrsordnung" (Traffic Ordinance), according to which air pollution due to automobile exhaust gases must not exceed the level unavoidable in the present state of technology. Legal regulations in accordance with the US Standards are being drawn up for establishing the maximum permissible concentration.

In order that the most important problems of waste gases could be dealt with by the authorities in the most uniform manner, the competent federal ministers issued a "Technische Anleitung zur Reinhaltung der Luft" (Technical Directives for Clean Air Maintenance) ("TAL"). This serves as a general administrative prescription for plants subject to authorization, according to para. 16 of the Trade Regulations. It contains definitions, general principles for authorization and supplementary directives, restrictions on the emission of smoke and dust, immission limits for dusts, gases and vapors. Furthermore, it contains directives for determining the basic load of SO_2 and for calculating the minimum stack heights. In the minimum requirements for the individual types of plants the corresponding directives of the VDI (Union of German Engineers) are taken into account. This applies, for example, to heating plants, concrete plants, ironworks and to the permissible dust levels in these plants.

In dangerous situations the authorities have the right to undertake immediate steps to reduce immissions. The state Nordrhein-Westfalen has two stages of alert. Stage I is reached when the SO_2-level in the air is 2.5 mg/m^3 at several measuring stations and a further increase is to be expected because of meteorological conditions with poor diffusion. At this stage the competent authorities are warned and further measures are prepared. At alert stage II (SO_2-level above 5 mg/m^3) the vehicular traffic is restricted and fuels poor in sulfur (which have to be in store for this purpose) must be used.

A local agreement between the SO_2-emittent and the authority is illustrated by the example in Linz on the Danube. The Österreichische Stickstoffwerke in this city agreed to interrupt their production of sulfuric acid, when the SO_2-level recorded by the monitoring instruments in the residential area reaches a value of 4 mg SO_2/m^3 for more than 15 min. This situation, however, has not occurred so far.

Immediate measures to be taken when reaching the alert stages are also prescribed in other countries. Thus, for example, in Los Angeles three stages of public alert are specified when the toxicant concentrations listed in the table below are exceeded.

In the first stage preparative measures are taken only. When stage II or III is reached, restrictions on or interruption of certain plants and vehicular traffic must be implemented.

	Stage I, ppm	Stage II, ppm	Stage III, ppm
CO	100	200	300
Sulfur oxides	3	5	10
Nitrogen oxides	3	5	10
Ozone	0.5	1.0	1.5

In the German Federal Republic numerous institutions are dealing with the problem of air pollution control. Air analysis is currently being carried out in the "Institut für Wasser-, Boden- und Lufthygiene" (Institute for Water, Soil and Air Hygiene) at the Federal Health Institute in Berlin, the "Landesanstalt für Immissions- und Bodennutzungsschutz" (State Institute for Protection against Immission and for Soil Utilization) in Essen and Bochum, the "Institut für gewerbliche Wasserwirtschaft und Luftreinhaltung" (Institute for Industrial Water Economy and Clean Air Maintenance) in Cologne, several technical inspection associations, the different working groups of the VDI Committee "Reinhaltung der Luft" (Clean Air Maintenance) and numerous public and private institutions. The "Deutsche Forschungsgesellschaft" (German Research Association) has launched a comprehensive program for determining pollutants at numerous slightly and heavily polluted sites.

Also in other countries numerous public and private institutions, technical associations and university institutes are dealing with the analysis of air.* In the USA the problem of air pollution is studied very intensively both theoretically and experimentally owing to the high industrial production level and the excessive vehicular traffic on the one hand, and the very unfavorable meteorological conditions in some areas on the other, which lead to very peculiar and annoying pollution phenomena (see page 17).

Among the international corporations the European Council in Strassbourg, the OECD (Organization for Economic Cooperation and Development), the WHO (World Health Organization), the IUPAC (International Union of Pure and Applied Chemistry), the working group "Clean Air Maintenance" of the European Federation of Chemical Engineering and the International Union of Air Pollution Prevention as the head organization are dealing with the different problems of air pollution.

The study group CONCAWE (Conservation of Clean Air and Water, Western Europe), The Hague, established and maintained by the leading petroleum firms, is an early example of industrial cooperation in the field of air pollution control.

* A list of organizations and persons in the European industrial countries dealing with the problem of air pollution was published by the European Federation of Chemical Engineering.

1.5 Books and periodicals on analysis of air and prevention of air pollution

The book by M. B. Jacobs: "The Chemical Analysis of Air Pollutants", Interscience Publishers, New York–London, 1960 is a special monograph on the analysis of air.

Collections of analytical methods published by individual organizations include:

VDI Manual "Reinhaltung der Luft" (Clean Air Maintenance) (VDI-Verlag, Düsseldorf); it contains several VDI directives on air analysis.

ICI Manual "The Determination of Toxic Substances in Air", edited by N. W. Hanson, D. A. Reilly and H. E. Stagg, Heffer Publishing House, Cambridge, 1965. This mainly contains methods for analyzing the air at the work site. Similar problems are dealt with in the collection "Methods for the Determination of Toxic Substances in Air (edited by J. C. Gage, N. Strafford and R. Truhaut; Butterworths, London, 1962), published on behalf of the IUPAC, and in the Booklets 1–16 on "Methods for the Detection of Toxic Substances in Air", published by the British Factory Inspectorate.

The booklet "Methods of Measuring Air Pollution", Paris 1964, contains data on smoke, SO_2, SO_3, hydrocarbons and fluorides.

In the USA the following books were published on the analysis of air: "Manual of Analytical Methods Recommended for Sampling and Analysis of Atmospheric Contaminants", Cincinnati, 1957, published by the American Conference of Governmental Industrial Hygienists, and Volume 23 of the ASTM standards (Industrial Water and Atmospheric Analysis), Philadelphia, 1968.

The following books give general information on problems of air pollution control.

"Die Verunreinigung der Luft" (Air Pollution) (German translation of 14 lectures held at the International Conference of the WHO in 1957), Verlag Chemie, Weinheim.

H. Jung: "Luftverunreinigung und industrielle Staubbekämpfung" (Air Pollution and Industrial Dust Prevention), Akademieverlag, Berlin (East), 1965.

W. Knop and W. Teske: "Technik der Luftreinhaltung" (Techniques of Clean Air Maintenance), Krauskopf-Verlag, Mainz, 1965.

"Air Pollution", edited by A. C. Stern, 3 volumes, Academic Press, New York–London, Volume I, II, 1962; Volume III, 1968.

The book by F. Bayer and G. Wagner: "Gasanalyse" (Gas Analysis) (Volume 39 of the series "Die Chemische Analyse", 3rd Edition), Verlag F. Enke, Stuttgart, 1960, contains a few sections dealing with air analysis.

In the following a survey is given of existing periodicals with articles and reports on the analysis of air and its pollutants.

In Germany: "Staub, Reinhaltung der Luft". [Cover-to-cover translation into English currently published by Israel Program for Scientific Translations, Jerusalem. The English translation (starting with Vol. 25, 1965) is for sale in the area of the United States of America solely by the Clearinghouse for Federal Scientific and Technical Information, U.S. Department of Commerce, Springfield, Va. For sale outside the United States of America solely by VDI-Verlag GmbH, 4 Düsseldorf, Post Box 1139.] Numerous