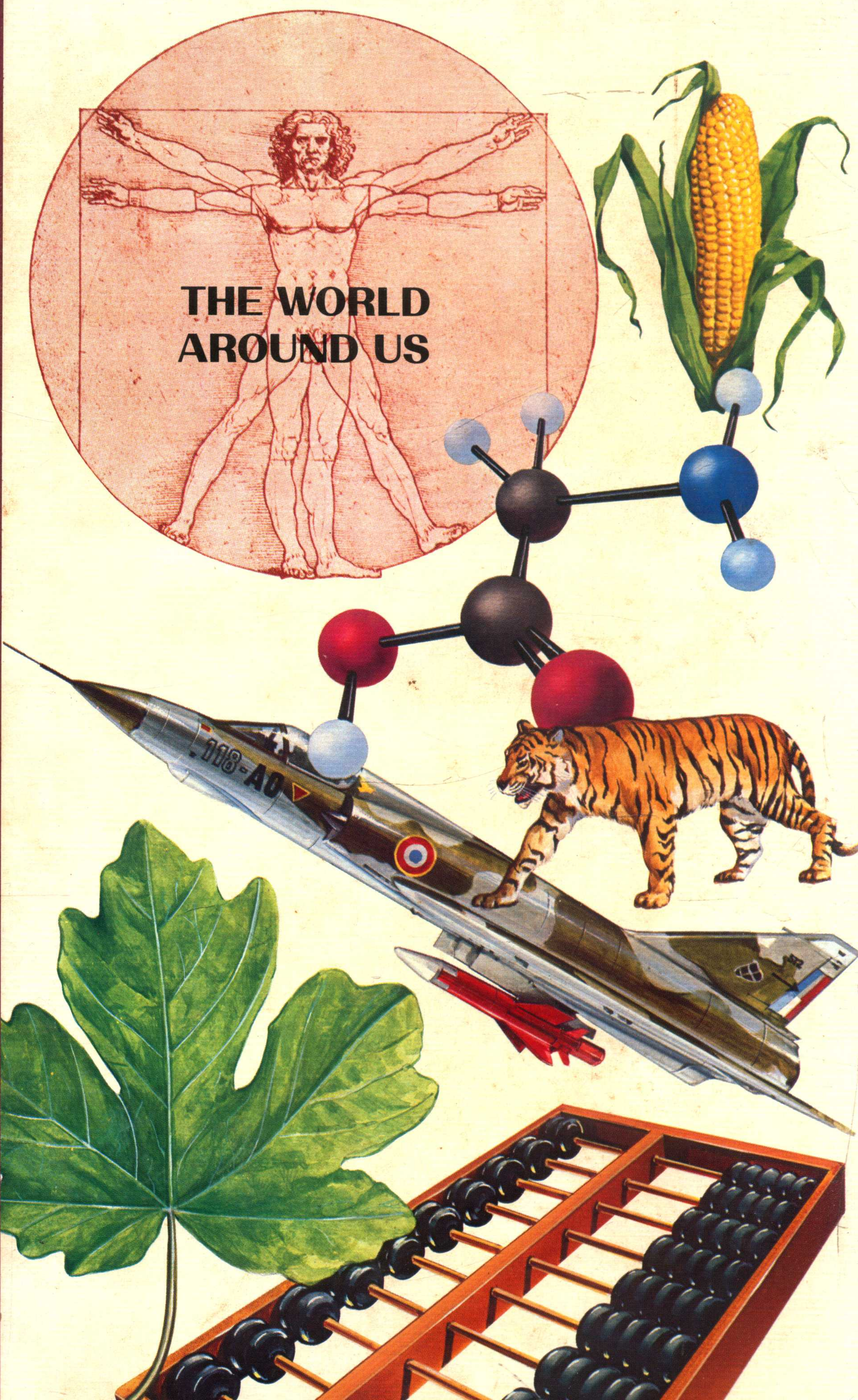
SCIENCE AND TECHNOLOGY ILLUSTRATED

THE WORLD AROUND US



Science Technology

The World Around Us

Library of Congress Catalog Card Number: 84-80129

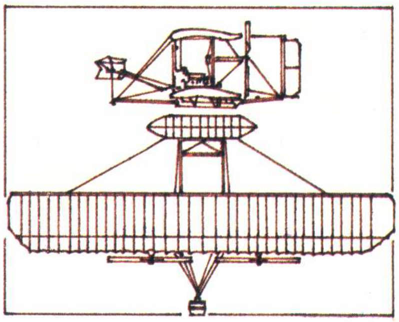
International Standard Book Number: 0-852229-425-5

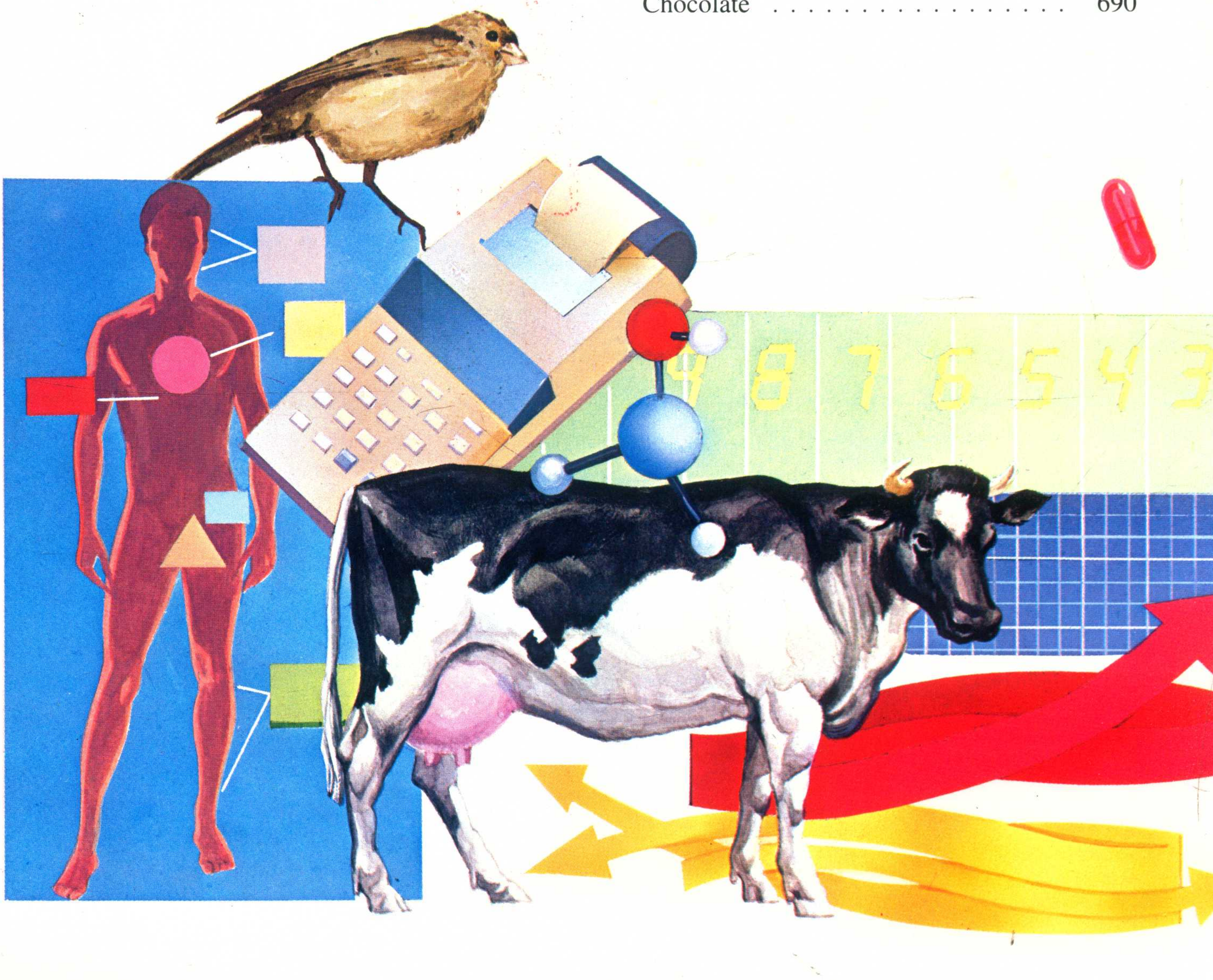
English language edition by license of Gruppo Editoriale Fabbri

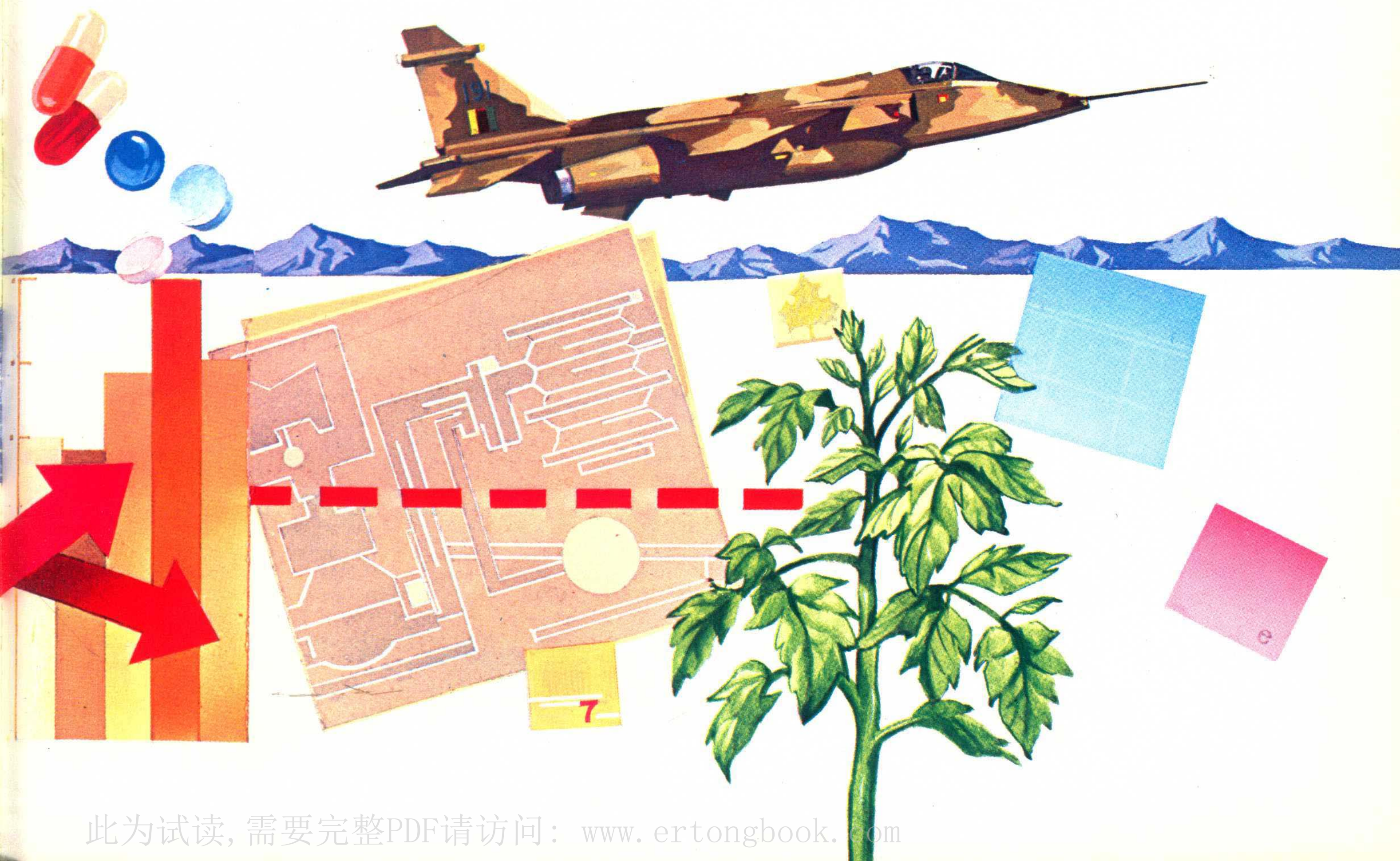
Title page photograph courtesy of Hale Observatories;
California Institute of Technology and
Carnegie Institution of Washington

Printed in U.S.A.

Contents







Science and Technology Illustrated

The World Around Us

Science Technology

The World Around Us

and Illustrated

Encyclopaedia Britannica, Inc.
CHICAGO

AUCKLAND • GENEVA
LONDON • MANILA
PARIS • ROME
SEOUL • SYDNEY
TOKYO • TORONTO

Library of Congress Catalog Card Number: 84-80129

International Standard Book Number: 0-852229-425-5

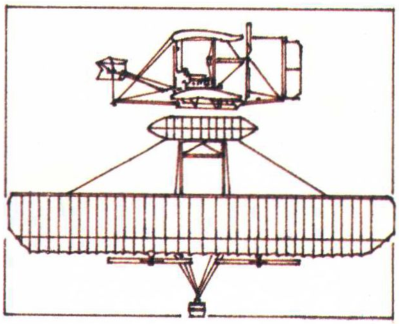
English language edition by license of Gruppo Editoriale Fabbri

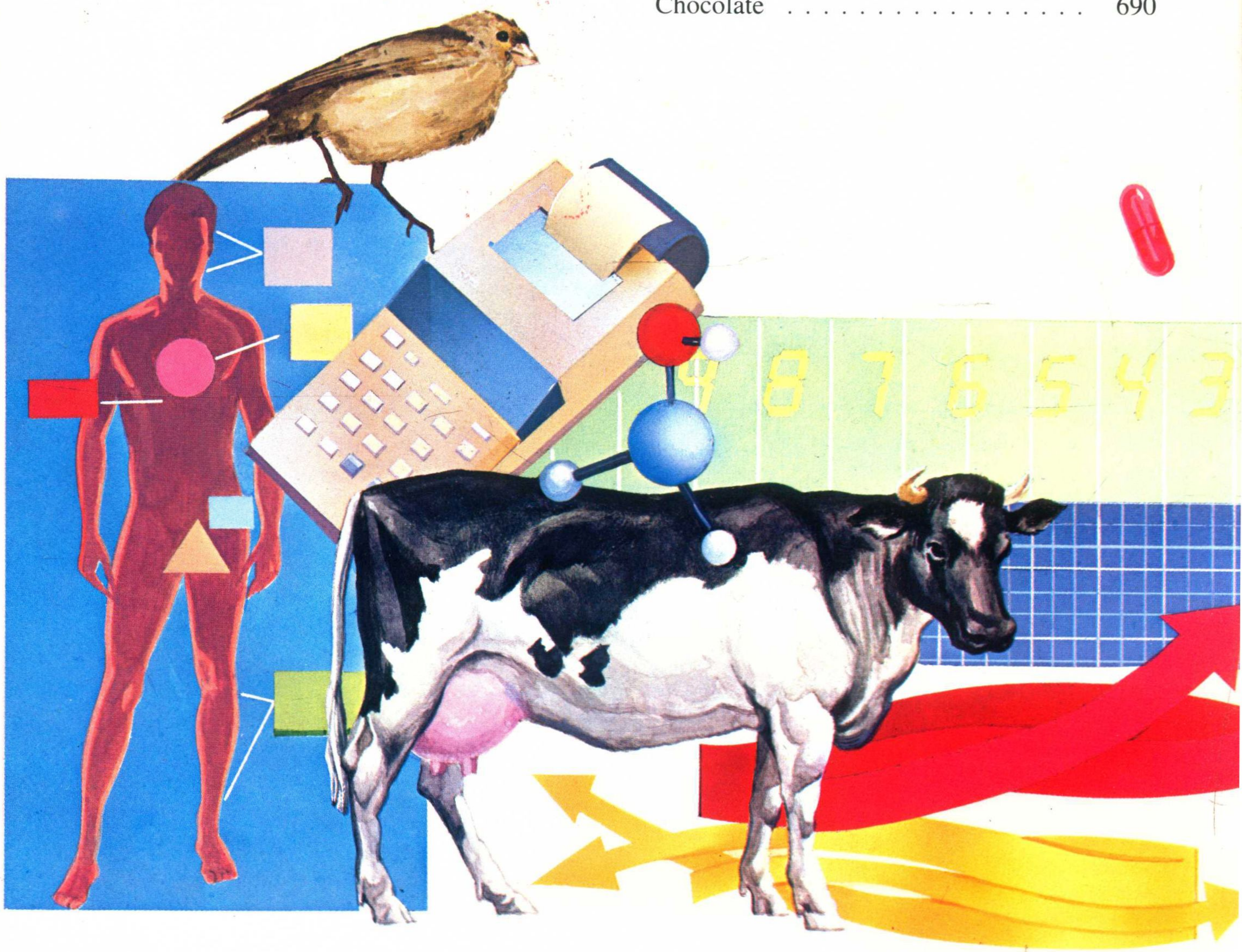
Title page photograph courtesy of Hale Observatories;
California Institute of Technology and
Carnegie Institution of Washington

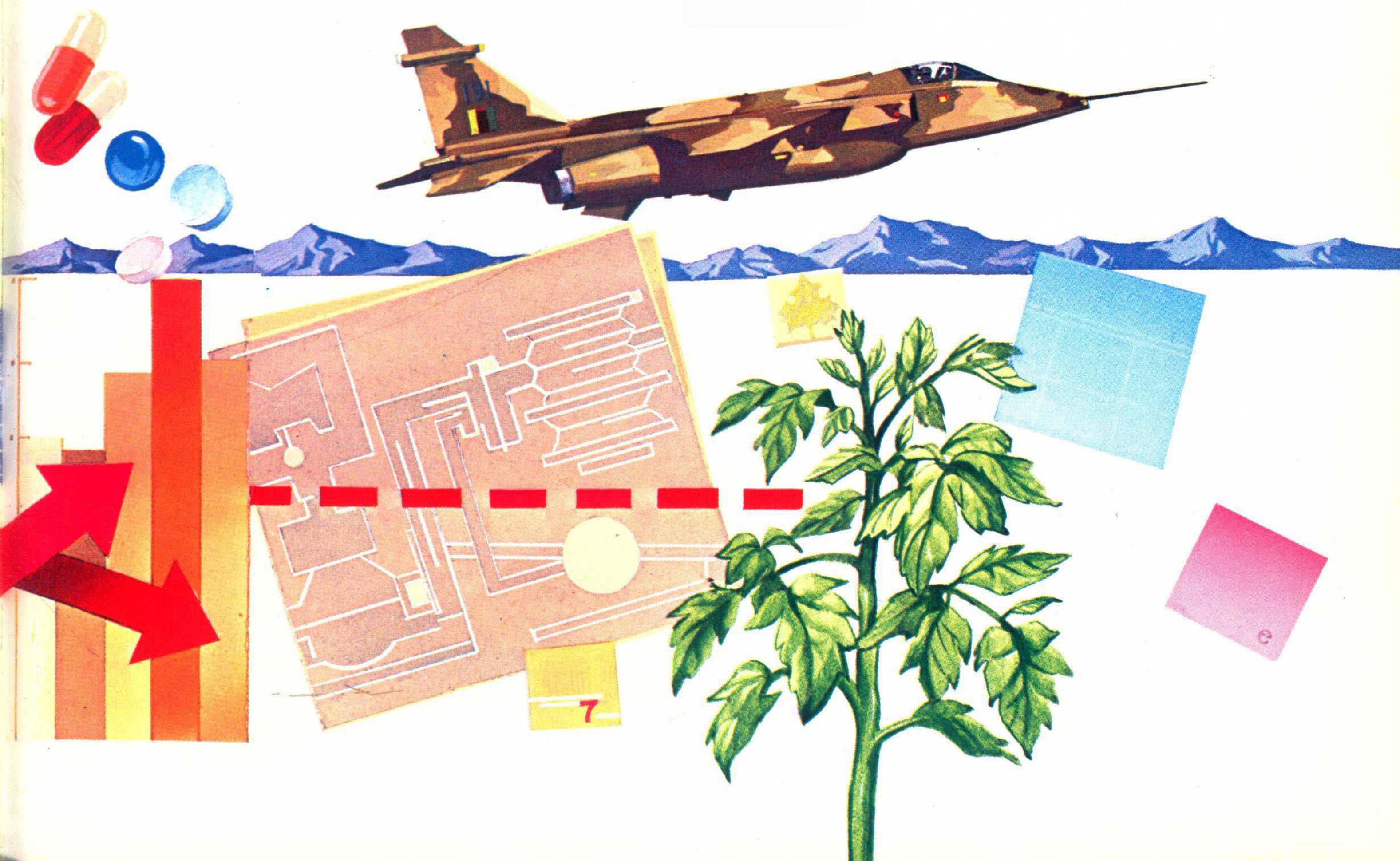
Printed in U.S.A.

Volume 6

Contents

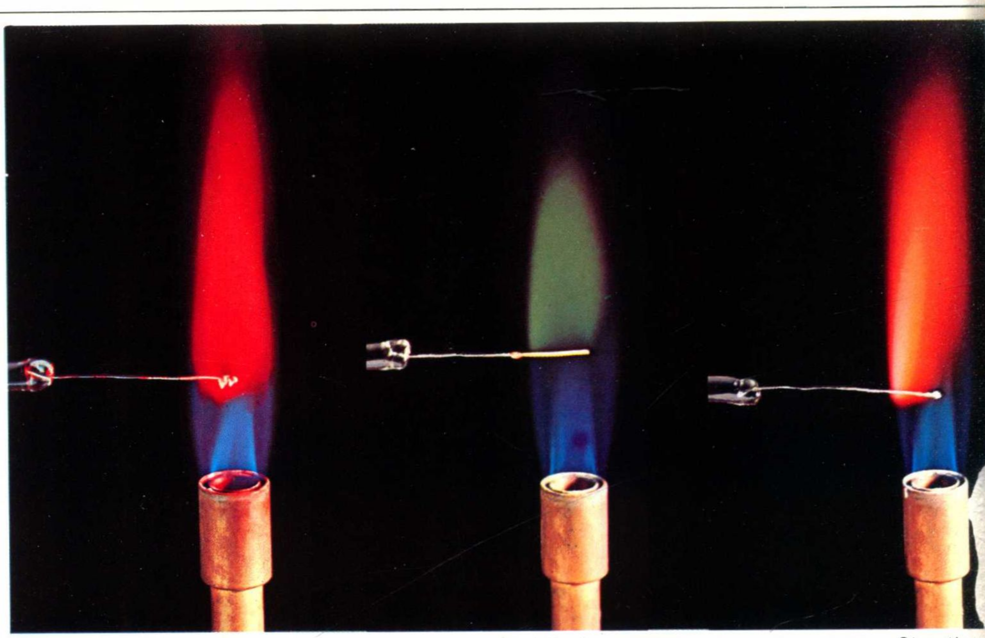




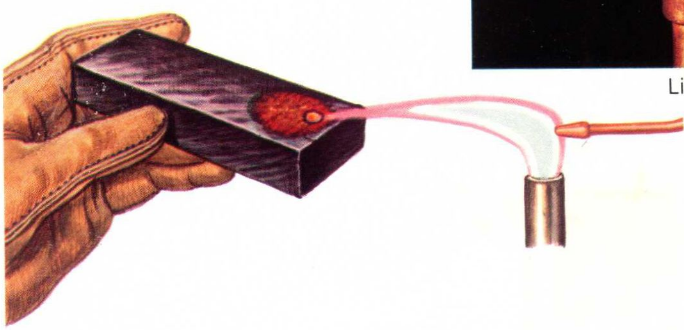


Chemical Analysis

How many atoms are in a molecule? How many elements are in a mixture? Like any other science, chemical analysis requires patience and practice—plus technical expertise. Interest in metals, minerals, and other substances goes back to ancient times. Standard weights, made of stone, were used by the Babylonians as far back as 2600 B.C. Sulfur, carbon, and seven metals—copper, gold, iron, lead, mercury, silver, and tin—were used in antiquity. Identifying substances and the ways in which they could be changed was as important in ancient Egypt, Mesopotamia, and China as it is today. In the 4th century B.C., the purity of gold was determined by the type of yellow streaks made when the sample was rubbed on a touchstone.



Lithium Barium Strontium



Antimony

Bismuth

By the 17th century, the natural philosopher Robert Boyle had introduced the term "analysis" to chemistry. Gas analysis was pioneered in the mid-19th century by Bunsen in Germany and Berthelot in France. Important techniques based on atomic energy have been developed since 1950, along with research using laser and electron probes. Automation and computerization further modernized chemical analysis, which has undergone dramatic changes based on recent advances in the field of electronics.

General Purpose

The purpose of chemical analysis is to answer all questions about the identity and composition of matter. What is it? What is it made of? How pure is it? Does it contain *x* or *y*? Chemical analysis is the attempt to obtain this information, as clearly and completely as possible, through the study of the composition and structure of matter.

Principal Stages

When performing chemical analysis, the following steps are generally necessary:

1. Acquiring a sample of the material to be analyzed
2. Examining the sample
3. Choosing the best analytical method
4. Changing the material into a state to which the selected method may be applied
5. Performing the actual analysis
6. Noting the results of the operation.

Sampling involves using only a small portion of material, but making sure that it has all the physical and chemical properties of the whole from which it was taken. Some materials, such as household amounts of foodstuffs, alcoholic beverages, and gases or liquids in which sameness normally exists, are easy to sample. Other materials, perhaps large amounts of grain or coal, are harder to sample because certain elements vary throughout the batch. For accuracy, it is important that the sample remain unchanged before analysis.

Errors and how they are made are extremely important to chemical analysis. Errors may be indeterminate (errors of chance) or determinate (caused by reactions, impurities, instrument failure, or human error).

Chemical analysis generally requires prior separation of the desired component from others that may also react to the process. Very few tests are specific, or valid for one substance only; quite a few tests are selective, or effective for a small number of substances or elements.

Principles of Method

There are two branches of analytical chemistry: qualitative analysis and quantitative analysis. Before discussing these disciplines, it is important to have an understanding of the following terms:

atom: the smallest particle of an element that can exist either alone or in combination with other elements

molecule: the smallest physical unit of a compound, consisting of two or more atoms bonded together

electron: an elementary charged particle that is a fundamental constituent of matter, existing independently or as the component of the atom outside the nucleus (center)

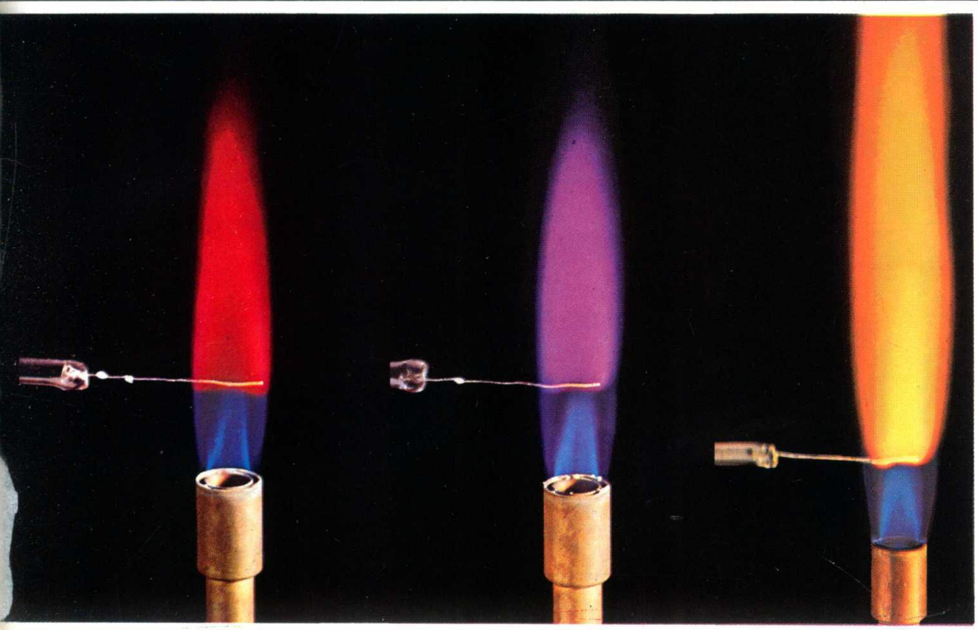
ion: an electrically charged atom or group of atoms, formed by the loss or gain of one or more electrons

cation: a positive ion created by electron loss

anion: a negative ion created by an electron gain

organic chemistry: the branch of chemistry dealing with the compounds of carbon

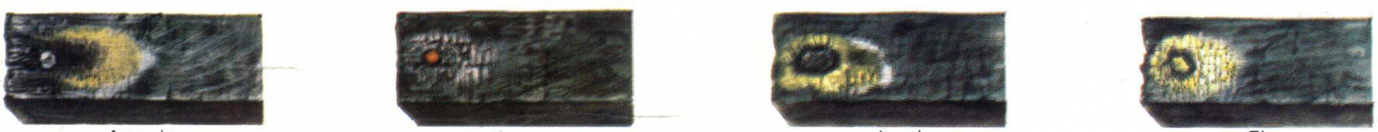
inorganic chemistry: the branch of chemistry dealing with noncarbon compounds



Calcium Potassium Sodium

Many of the alkaline and alkaline earth metals give a characteristic color to the flame of a Bunsen burner when their salts are heated. This type of flame test is performed by wetting an inert platinum wire with hydrochloric acid and then dipping it in the substance to be tested. The wire is then heated, and the metallic chlorides formed by the acid vaporize, coloring the flame and so revealing the identity of the metallic element present.

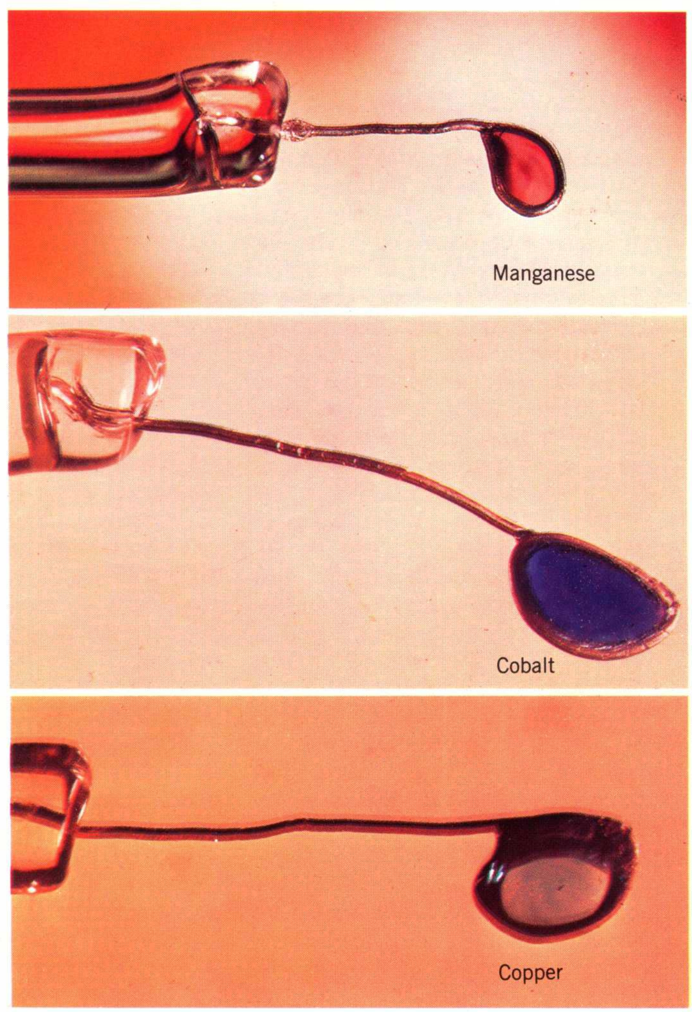
The charcoal blocks immediately below show the results of blowpipe testing of different metal salts. The material to be tested, in powder form, is placed in a hollow on the block's surface and heated with a jet of flame directed by a blowpipe, as in the illustration at left. The traces left on the block after the decomposition of the test substance identify the metallic element on which the material is based.



Arsenic Copper Lead Zinc

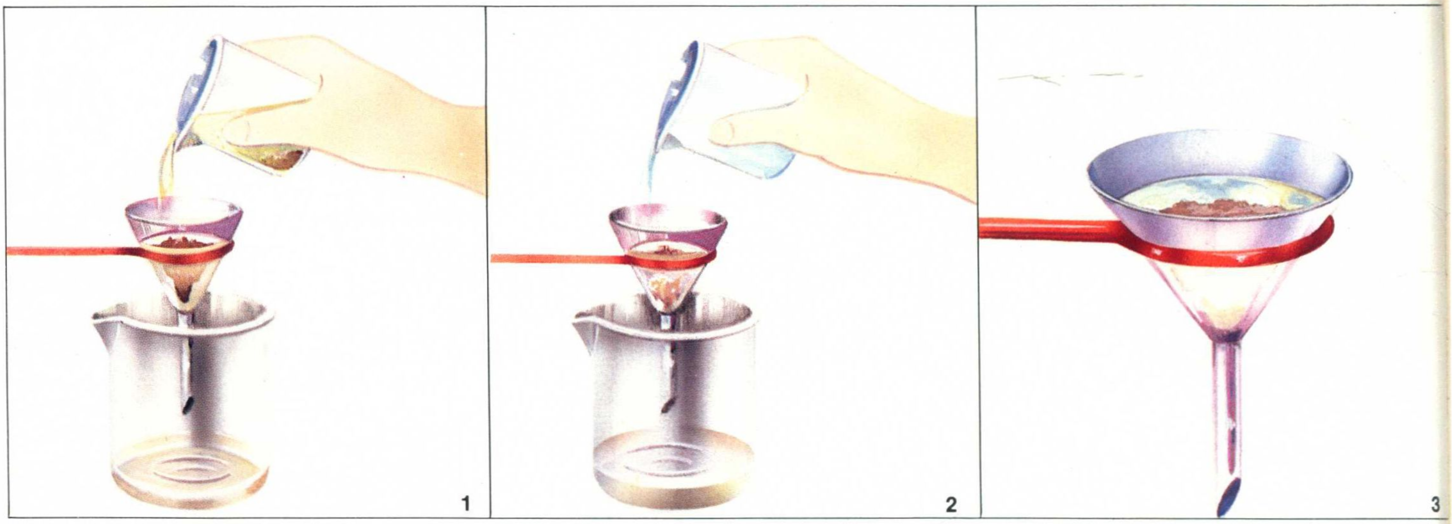
Qualitative Analysis

This process involves identification of the elements in a compound or the components of a mixture of compounds. The science was developed to help in the study of minerals. Its strongest emphasis has always been on common inorganic cations and anions. Certain chemical reactions occur only in the presence of a few ions; by taking a particular set of steps in order to separate a group of ions from a solution, the unknown element is found to exist in smaller and smaller groups, until there is only one possibility left.



Right: Use of borax bead test to identify metal salts. A loop of platinum wire is heated in the flame of a Bunsen burner, dipped in powdered borax, heated again, fusing the borax into a clear bead. The bead is deliberately contaminated with a trace of the substance to be tested and reheated a third time.

On cooling, the color of the bead indicates the metallic element present. Each of the elements that respond to the test forms a characteristic color in the bead, as in the examples shown here. Manganese yields a pale violet-red, cobalt a rich blue, and copper a dull pale blue.



Generally, qualitative analysis takes place using the following steps:

I. *Preliminary study of sample*. This is to determine its form and other physical characteristics, including color, odor, hardness, etc.

II. *Chemical tests*. These include dry tests (those that use the effect of heat) and wet tests (those that dissolve the sample).

1. *Dry Tests:* Colors—their glow and intensities—play a large part in dry tests, which include:

a. *Flame tests*. When heated, compounds of certain elements cause colors to appear. The test is done by putting some of the sample into a flame on a platinum wire wetted with hydrochloric acid. Chlorides are formed by the acid, causing intense colorations depending upon the material used. For example, potassium gives a violet glow; sodium, a bright yellow; calcium, red; and copper, bright green. When looked at through a spectroscope (an optical device for observing a light spectrum), even closer analyses may be made. The various colors given off by rare metals such as indium, thallium, and cesium were discovered by means of this instrument.

b. *Blowpipe and bead tests*. Test material is put into a depression made on a charcoal block, then heated in the flame of a blowpipe, or tube through which gas or air is forced into a flame to concentrate or increase the heating action. The nature of the test material may then be studied from the remains of the fire. With certain substances, this residue may be treated with chemicals and then reheated to gain additional information. Colorless beads are formed by fusing (combining in heat) borax or certain salts on a small loop of platinum wire. These beads dissolve metallic oxides or salts, producing various colors, which enable the tester to make an analysis. Both blowpipe and bead tests have been largely replaced by more modern techniques, but they are still in use to some extent and are important in the history of chemical analysis.

2. *Wet Tests*. Various processes are used to dissolve test material so that it can be analyzed, separating cations and anions.

3. *Spot Tests*. A spot test requires only a drop or two of solution and may be done on a slide or on paper. Many confirming tests (those that make sure one element is identified) in qualitative analysis can be performed as spot tests; they quickly identify a substance or compound in which the presence of a suspected element is indicated by color change or precipitation (change brought about by separation).

Instrumental Analysis

Growing rapidly since the 1930s, instrumental procedures have completely changed the face of analytical chemistry. The instruments range from those that duplicate highly automated and technical laboratory processes to those that tell the presence and amount of substances from the way in which unknowns react with different forms of energy. Three forms of instrument analysis are used most often:

1. *Spectroscopic methods*. If an atom or molecule absorbs energy, it is said to be "excited" to a higher level. When it returns to normal, the energy is sent out again as a "line" of radiation. The wavelength of this radiation depends on the difference between the two levels and identifies the atom or molecule. Qualitative analysis is concerned with the wavelength of these lines, and quantitative analysis with the intensity of the lines. (For more information about this procedure, see SPECTROSCOPY.)

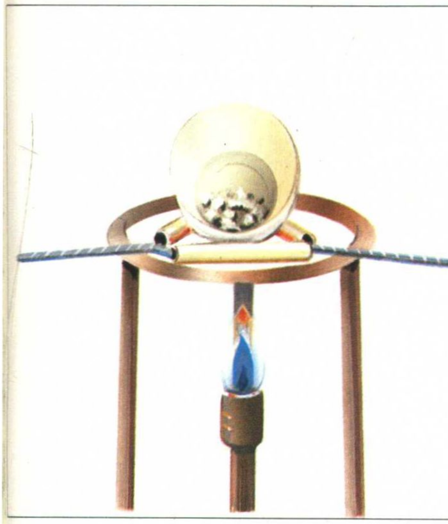
2. *X-ray fluorescence analysis*. X-ray wavelengths emitted when a target is bombarded by X rays identify the elements in the target itself.

3. *Activation analysis*. This is a way to use radioactive detection methods with elements that are not themselves naturally radioactive. Activation analysis has proven valuable in finding impurities and has played a key role in the analysis of "moon rocks" collected by the astronauts.

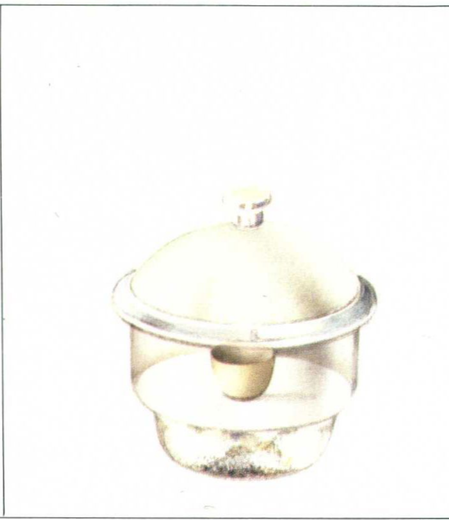
Analytical instruments can be simple devices or complex and expensive elec-

Titration is a method for analyzing the composition of a solution by adding known amounts of a standard reagent or test solution until some given reaction takes place. The reaction may be a change of color, the precipitation of a reaction product, or a change in electrical conductivity.

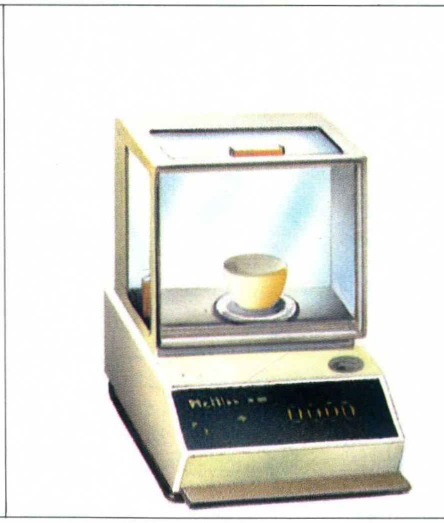
Color titration determines the amount of acid in a solution. The reagent—here an indicator, a substance that changes color in the presence of acids or bases—is allowed to drip slowly into the solution to be tested. By measuring the volume of indicator necessary to trigger a complete color change, it is possible to calculate the volume of acid in the solution.



4



5



6

tronic apparatus. Instrumentation also varies greatly as to the amount of human participation necessary. As the equipment becomes more sophisticated, less attention is usually needed for the analyses. But this means that more training and planning are required at the beginning.

Quantitative Analysis

That area of analytical chemistry dealing with amounts of material in a compound, or of components in a mixture, is known as quantitative analysis. The steps followed in making this type of analysis may be taken according to the specific material studied, the amount of it present, and the method used.

The subject is divided into two general areas:

1. *Gravimetric analysis,* when weight is the overall base for calculation or analysis

2. *Volumetric analysis* (also called titrimetric analysis), which involves solutions of known concentration reacting with the sample to reveal the nature of the unknown

A good illustration of the way these two methods work is a study of the process used to determine chloride. In a gravimetric analysis, the tester treats the sample with silver nitrate solution, which precipitates the chloride as silver chloride. The amount of chloride in the sample is indi-

Above: Sequence of diagrams, from left, illustrates steps in gravimetric analysis. The substance to be examined is precipitated from solution, then filtered and washed to remove impurities. It is then heated in an oven to just above the boiling point of water, to drive off the excess humidity left after washing.

The dry sample, still on the filter paper, is transferred to a crucible, where it will be calcined at high temperature. If the residue cannot be weighed immediately, it is kept in a closed jar with dehumidifying agents to keep it from absorbing moisture from the air. Allowance is also made for the weight of any ash left by the filter paper calcined together with the sample. Finally, weighing the residue matter makes it possible to calculate the quantity (by weight) of the substance originally precipitated from solution.

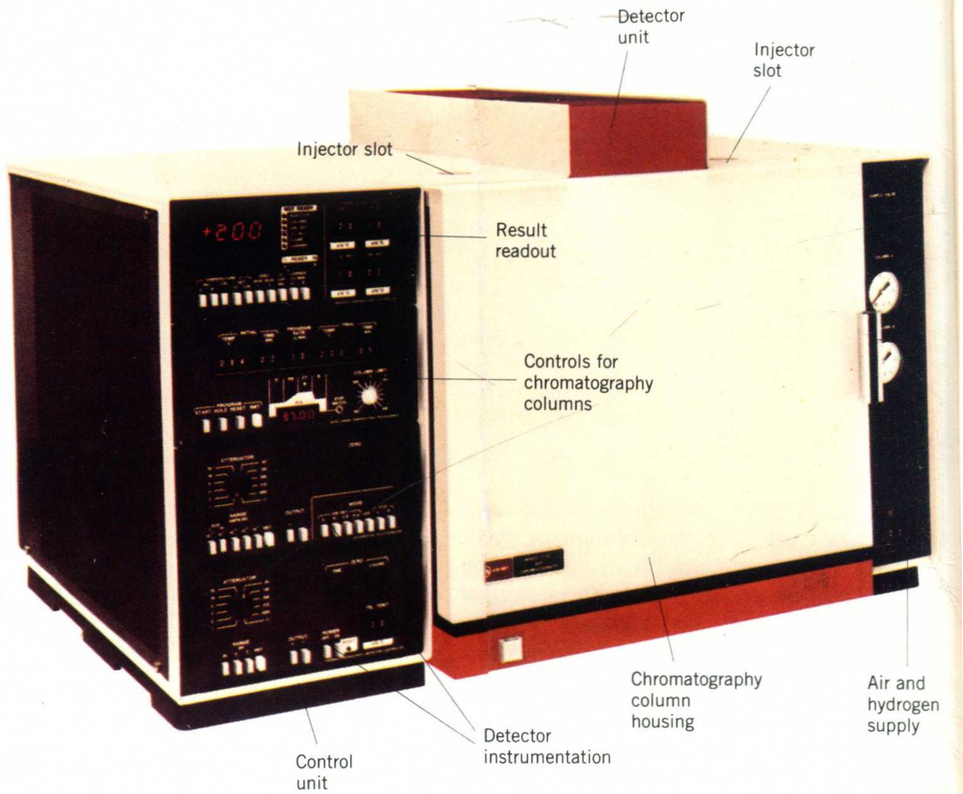
1

2

3

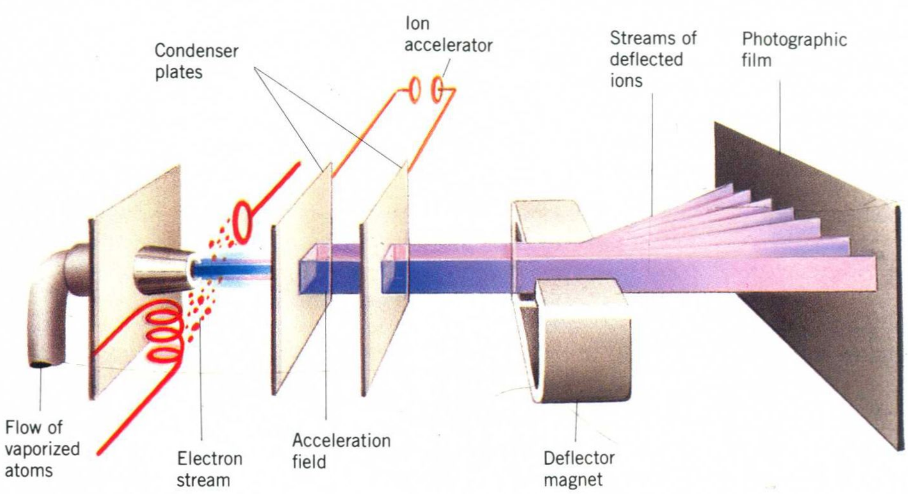
cated by the weight of this silver chloride. In a volumetric analysis, the same sample is mixed with a known quantity of silver nitrate solution; the volume of solution required to react exactly with the amount of chloride present is measured. From this volume and the known amount of solution, the amount of chloride in the sample is determined.

In an ultimate analysis, the extent of one element or compound is determined. In a proximate analysis, specific components are studied as a group of unknown composition. Methods used in quantitative analysis vary according to the nature of the substance in question. For this reason, results of tested methods have been collected to make the task easier. These are used as reference books, covering many areas including metals, food, minerals, and agricultural and technological products.



Additional Methods Used for Analysis

Classical. Both laboratory and manual (by hand) methods of testing are used on a large scale where samples are plentiful.



These methods are cheap to use and important for teaching, industry, and research. Particularly significant modern applications include determining the presence and nature of illicit drug use, studying chlorocarbons in the Earth's atmosphere, and research into use of chemical warfare agents.

Microchemical. These may be used for analyses of very small samples. The apparatus is sophisticated and expensive, and requires great skill to use. New areas of research have been opened up due to these micromethods, making knowledge of composition possible even with samples as tiny as those often used in biochemistry.

Gas Analysis. This depends upon adsorption of the components in a gaseous mixture and measurement of the changes in volume that take place after that adsorption.

Fire Assay. This method is used to determine the amounts of previous metals in ores. The metals are extracted through the use of molten lead, which, on cooling, retains the precious metals as an alloy (metal mixture).

Optical Methods. These concern the measurement of the extent of the interac-

Above: Gas chromatograph. As its name suggests, this device permits the analysis of gases or substances that can be vaporized by heating to a gaseous state. Not purely an analytic tool, the gas chromatograph finds applications in many fields of advanced research in chemistry and biology for its ability to separate extremely complex mixtures into their constituent compounds.

Left: Essential functional elements of a mass spectrometer. The vaporized atoms of the material to be analyzed are first ionized by a stream of electrons and then accelerated by an electrical field through a deflector magnet. The lower the mass of the ions, the greater the deflection. The photographic record made of the patterns of this deflection permits identification of the atoms present in the original sample.

Right: Compact infrared spectrophotometer for chemical analysis.