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ON THE NATURE OF MINERAL INCLUSIONS IN GEMSTONES

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AMONG the many types of inclusions encountered in natural gems, where they are regarded as unequivocal criteria of genuineness and often as welcome identification marks of locality, mineral inclusions are of particular interest; for just as, externally, the mineralogical environments of a gemstone in a specific habitat will furnish valuable evidence of that deposit's evolution, the inner paragenesis—i.e. the various mineral inclusions —will yield valuable information about the chemical and physical conditions that must have existed during the formation of their precious host crystal.   In a great number of cases, such crystalline formation has been rare enough to be considered unique—witness benitoite—in that the chemical constituents, apart from only once being present simultaneously, have been gathered together at the same time in the proper stoichiometrical relation to the relevant crystalline lattice.   During the growth of a gem older minerals, having crystallized during an earlier phase and having temporarily ceased to grow (because of shortage of substance or a phase displacement), were overgrown or fully enclosed by the younger gemstone, which may have grown even more rapidly (Plate A, 1). W. F. Eppler[9] has named such minerals of an older formation "pre-existent mineral inclusions".

149

A
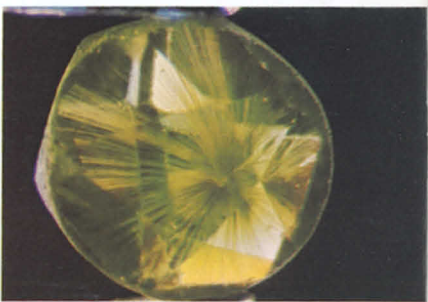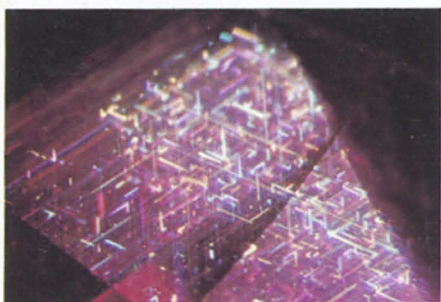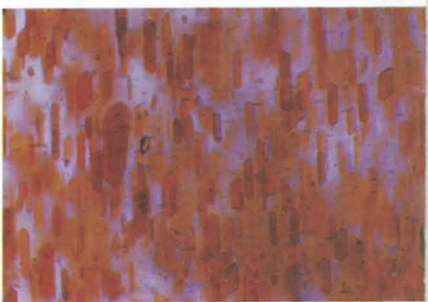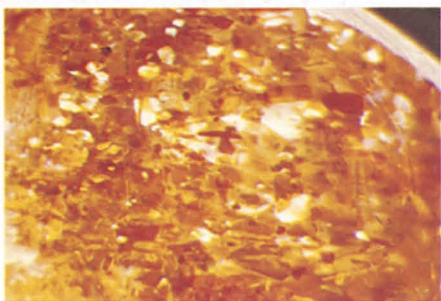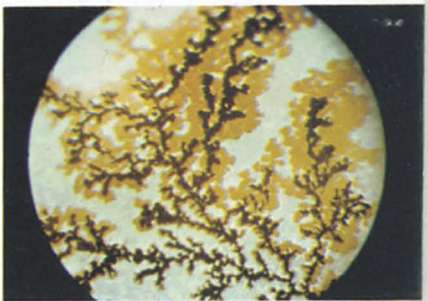
"Syngenetic mineral inclusions" have crystallized, along with the formation of the gem, from a complex solution or melt in which a number of chemical components were present in a dissolved state. Separated molecules, in their function as building blocks of one mineral or another, may link up with one another or with other molecules of their own type, disregarding completely the crystal structure of other minerals forming at the same time; or they may, with the atoms at fairly equal distances apart (structural affinity), enter into a mutual isomorphic relationship (mixed-crystals) or else grow epitaxially upon their growing faces (orientated intergrowth). In the process, one of the mineral species may cease to grow due to a lack of substance supply. Where such a mineral species has developed rapidly—greedily amassing its components in one direction of growth, as it were—slim, stalky crystals have resulted, such as the rutile (Plate A, 2) and tourmaline (Plate A, 3) encountered in many quartzes, or the byssolite fibres included in demantoid garnet (Plate A, 4). Occasionally, these elongated prismatic or fibrous mineral inclusions have grown independently; more often than not, however, they have formed coincidentally with the host crystal. But the crystallizing process of the gem now including them had not ceased by the time the guest minerals suspended growth: in fact it frequently continued to exist in solution. Simultaneous or subsequent solidification, i.e. crystallization of any such substance remaining in solution, ultimately led to the inclusion of foreign minerals that had been settled on the former crystal faces or had grown freely within the solidifying melt.

With atomic spacings (lattice constants) in the guest and host crystals being virtually equal, the mineral embraced will co-ordinate itself in relevant crystallonomic directions pertaining to its host mineral. An example of this is furnished by olivine whose **pinacoidal** face (010) (the face having all but the closest oxygen packing) will

Plate A
1. *Epidote needles embraced by quartz (rock-crystal).*
2. *Rutile needles in rock-crystal.*
3. *Rods of tourmaline in rock-crystal.*
4. *Radiating byssolite fibres in demantoid.*
5. *Exsolved rutile needles in ruby.*
6. *Exsolved platelets of lepidocrocite in iolite.*
7. *Dense concentration of goethite flakes in aventurine-feldspar (Sunstone).*
8. *Secondary, epigenetic formation of psilomelane in dendritic agate.*
9. *Blades of actinolite in rock-crystal.*
10. *Dense felt of amianthus fibres in quartz causing chatoyance.*

grow on the octahedral face (111) (calotte plane with closest C-atom packing) of a diamond in such a way that its c-axis will be in parallel alignment with the octahedral edge. The C-C atomic distance in the primitive cube of the diamond being $a_0 = 3·57A$, the simple calculation 2 a_0. $2\sqrt{2}$ will result in a distance of 10·06A on the octahedral edge, and this is almost equivalent to the lattice constance $c_0 = 10·28A$ of olivine. Other known examples are furnished by the orientated rutile needles in almandine and the exsolved fine rutile needles (silk) in corundum.

As opposed to pre-existent and syngenetic mineral inclusions, "epigenetic mineral inclusions" do not form until after the growth of the host crystal is fully completed. Their formation must be ascribed to the well-known process of exsolution. This is a subsequent separation of one, or even of several components (which may likewise exist, and have in fact been identified, as independent minerals) from a mixed-crystal in which they had co-existed in solid solution until the balance of energy was ultimately upset. Precipitation of minority components has usually been caused by changes in temperature (cooling or heating with or without change of pressure) and has led to crystalline differentiation in that microscopic crystals were formed, being mostly or nearly always coordinated according to the crystallonomic directions of the major component. Typical examples of such exsolved, epigenetic inclusions are provided by the "silk" in corundum i.e. the fine rutile needles with their sagenite pattern of co-ordination in accordance with the three main directions of the base and rhombohedron (Plate A, 5); and the small slabs of lepidocrocite co-ordinated parallel with the main axis in cordierite (dichroite, iolite) (Plate A, 6); also the dense patches of goethite scale in sunstone (aventurine feldspar) (Plate A, 7).

Since the completion of the manuscript a new analysis has been established in that these slabs are not goethite but lepidocrocite (γ—FeOOH), which has the same chemical composition as goethite. The difference is a morphological one in that the goethite inclusions are fibrous to acicular and lepidocrocite tabular.

Alternatively, foreign mineral substance from outside may have penetrated into the interior of a mineral by way of fissures and channels, where it is now present predominantly in the form of skeleton crystals. Most prominent among the minerals involved in the formation of such subsequent inclusions are iron hydroxide,

manganese hydroxide (psilomelane), and also manganese oxide pyrolusite: in the fissures filled by them, all of these have frequently produced magnificent dendritic forms or a host of other beautiful patterns. Patterns thus formed are particularly prized when occurring in types of quartz such as agate and rose-quartz (Plate A, 8)—also in turquoise, rhodonite.

This deliberately brief excursion into the genesis of mineral inclusions in gemstones was intended to point out just how close a relationship exists between mineral inclusions and their host crystals and what interesting conclusions on the formation of gemstones may be drawn from a thorough knowledge of the inner paragenesis. Mineral inclusions have been the most important witnesses of the mysterious processes of crystallization that were involved in the growth of mineral deposits in great depths below the earth's surface. Bearing in mind their immense analytic value for a reliable recon-struction of the evolutionary processes underlying their formation, and thus to the science of mineral deposits, over the past 100 years scientists have devoted more and more attention to the exact determination of mineral inclusions. As long as thin sections could be obtained, or when in any case the object to be tested could be destroyed, the task was easy enough; but being primarily concerned with studying the cut specimens of gemstones, gemmologists have usually had to confine themselves to crystalloptical scrutiny. Despite an almost inconceivable variety of shapes and forms and an all but unlimited multiplicity of possible combinations, a rather reliable criterion is furnished by the characteristic crystal form of a mineral, and it is due to this or to the relevant habit of an idio-morphic crystalline inclusion that its true nature has often been identified. It is thus that in the relatively early stages of crystall-optical research detection and identification were definitely achieved not only of the afore-mentioned mineral inclusions, but of such diverse members of the amphibole family as actinolite shingles (Plate A, 9), amianthus felt (Plate A, 10) and hornblende needles in rockcrystal (Plate B, 1), bamboo-like canes of actinolite in Ural emerald (Plate B, 2) and blades of tremolite in Habachtal and Sandawana emeralds (Plate B, 3); furthermore, irregular structures of green chlorite (Plate B, 4), brown stalks of epidote (Plate A, 1) and tetragonal bipyramids of anatase in rockcrystal (Plate B, 5), radiating sheaths of goethite in amethyst (Plate B, 6), small pseudo-hexagonal slabs and flakes of brown biotite in Transvaal and Ural
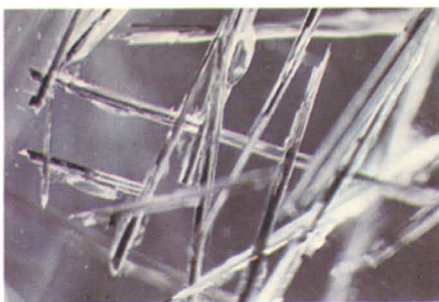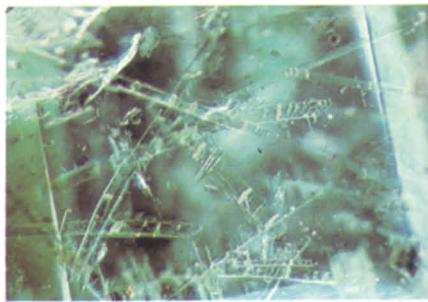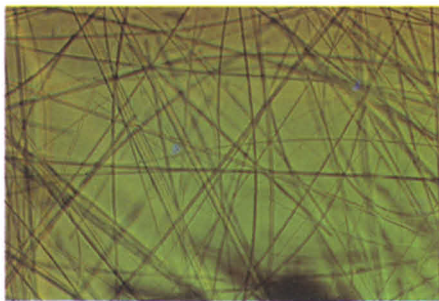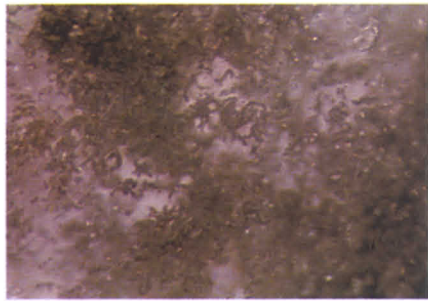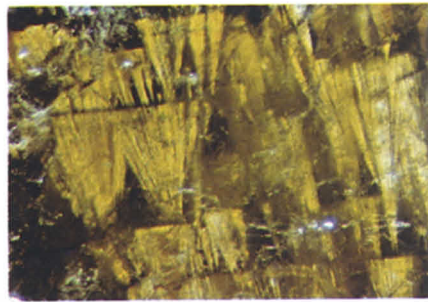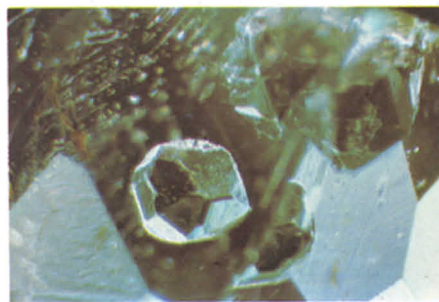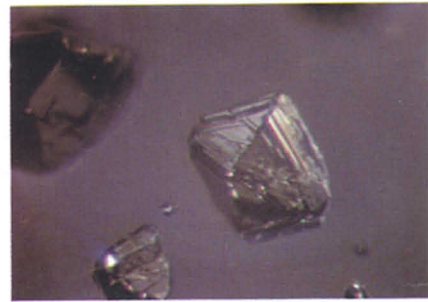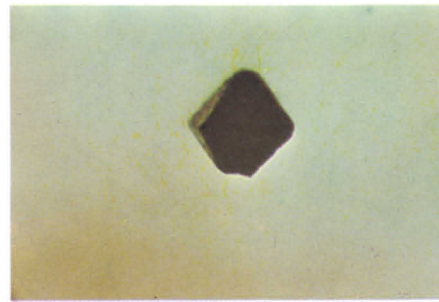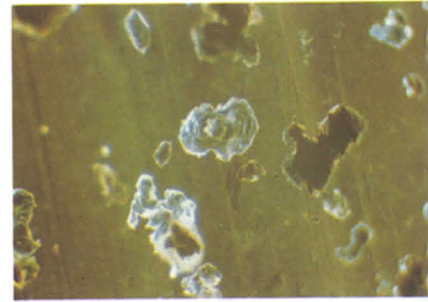
emeralds; also brassy yellow pyrite crystals in both Chivor emerald (Plate B, 7) and fluorite (Plate B, 8), well-developed spinel octahedra in Mogok ruby (Plate D, 3), euhedral octahedra of chromite in williamsite (Plate B, 9), mica platelets in peridot, hematite in diamond, euclase, corundum, quartz, and topaz (Plate B, 10), and diamond and garnet in diamond.

Owing to the slanting position of the angles incurred by the facets of cut gemstones, even the well-defined forms of a crystal will often appear grossly distorted, and this may easily lead to misjudgment. Furthermore, ideal cases of idiomorphic crystal forms are extremely rare; very frequently crystals viewed under the microscope will not give any elucidation on the nature of the mineral inclusion under observation, seeing that the solid body's shape is irregular, contorted, or completely undefined. Not infrequently, such inclusions will be constituted by resorbed or abraded granules, ill-defined slabs, irregularly edged or spiky aggregates, twisted flakes, splintery chips, or other such indefinable fragments or rudimental structures as would no longer permit morphological assessment; they might at best be identified by virtue of their colour or the difference between their R.I./D.R. and those of their host crystal. Nevertheless, such fragmentary mineral inclusions, being characteristic of certain gemstone species or particular localities, are indeed of some diagnostic value; pending discovery of adequate methods of identification, gemmologists therefore contented themselves with giving such exact descriptions of them as they possibly could. While for the purposes of determining genuineness and distinguishing differences in origin gemmological practitioners could reconcile themselves to the mere availability of a phenomenological description of inclusions, this failed to satisfy the gemmologist with a bias towards mineralogy and, even more so, the mineralogist interested in gemmology.

Plate B
1. *Rods of hornblende in rock-crystal.*
2. *Bamboo-like canes of actinolite in emerald from the Ural mountains.*
3. *Needles of tremolite in emerald from Sandawana.*
4. *Dense concentration of chlorite in quartz (often producing moss-agate)*
5. *Clearly euhedral anatase crystals in rock-crystal.*
6. *Divergently radiating fibres of goethite (Cacoxenite) in amethyst.*
7. *Well-developed crystals of pyrite in emerald from Chivor (Colombia).*
8. *Euhedral pyrite in fluorspar.*
9. *Well-defined octahedron of chromite in williamsite.*
10. *Hematite in topaz.*
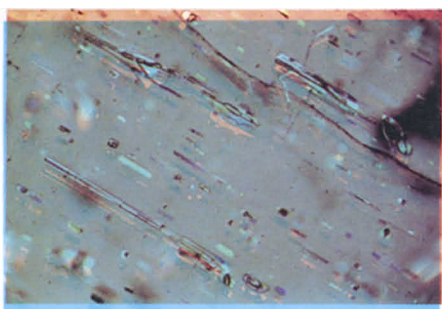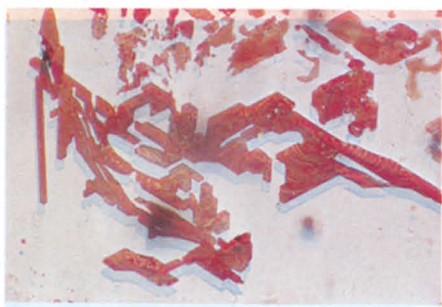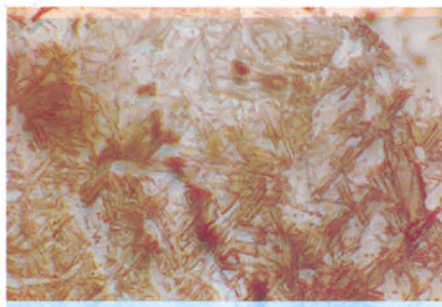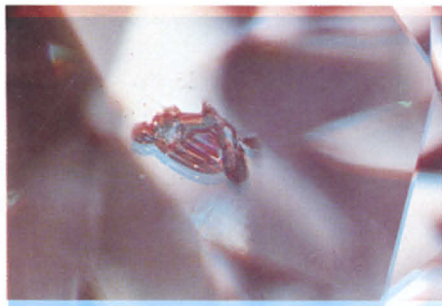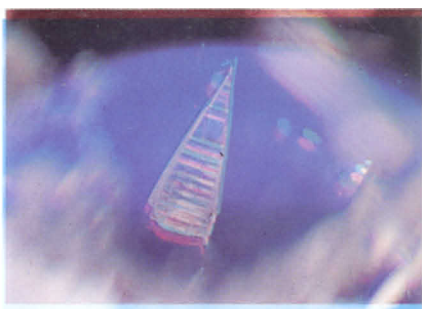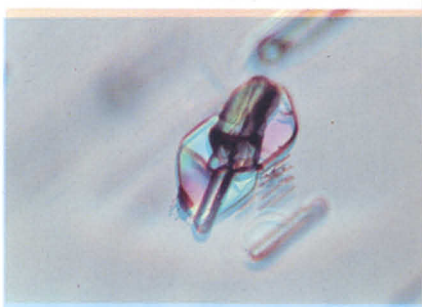
During the past twenty years, ever-growing numbers of scientists have devoted themselves to painstaking research into the field of mineral inclusions in gemstones, and it is to the outstanding merit of W. F. Eppler that he has dedicated himself to this task systematically and consistently. Applying the orthodox method of crystalloptics in some cases, and extricating the minute mineral inclusions for subsequent microscopic and radiographic examinations in others, he has succeeded in identifying beyond any doubt a host of solid foreign bodies included in gemstones. This has not only added a number of enlightening findings to the field of genetic observation, but it has also considerably augmented the diagnostic techniques that are so important to gemmology. Thus, W. F. Eppler managed to corroborate the occurrence of garnet in diamond and to prove the existence of olivine in diamond shortly after it had been observed by R. S. Mitchell and A. A. Giardini[36]. Eppler was the first to discover apatite as being a member of diamond endogenesis[8]. He also reliably identified, in several other gemstones, certain mineral inclusions frequently recurring and thus diagnostically significant, viz. the filling with granulated quartz, apatite, and epidote of tubes parallel with the c-axis in aquamarine (Plate C, 1); also the presence in aquamarine of individual specimens or batches of hematite, mica (fuchsite, muscovite, phlogopite), garnet, ilmenite skeletons and petalite; and other fascinating cases of quartz intergrown with apatite and epidote (Plate C, 2, 3, 4).

The presence of shingles and needles of sphere crystals in star-spinel, causing the latter's asterism, and having previously been detected by this author using purely crystalloptical methods, has also been confirmed by W. F. Eppler[6] (Plate C, 5).

Since the discovery of the rich Siberian diamond deposits in Yakutia, Soviet mineralogists have exercised admirable exactitude in investigating the inner paragenesis of diamonds mined in the U.S.S.R. However, they, too, did not examine the solid bodies

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Plate C

1. *Growth-tubes filled with quartz, apatite and epidote in aquamarine.*
2. *Phlogopite in aquamarine.*
3. *A skeleton of ilmenite in aquamarine.*
4. *Combination of quartz and apatite in aquamarine.*
5. *Orientated arrangement of twinned sphenes in star-spinel.*
6. *Pseudotetragonal olivine prisms epitactically settled on the octahedron face of a diamond.*
7. *Garnet crystal in diamond.*
8. *Combination of distorted octahedrons of chrome-spinel in diamond.*
9. *Emerald-green diopside crystal in diamond.*
10. *Group of pyrrhotine crystals in diamond.*

while still included, but rather, "freed" the mineral inclusions from their host diamond in order to subject these isolated microscopic crystals to crystalloptical and radiographic testing. Proceeding along the same lines as W. F. Eppler, but independent of him, they, too, managed to recognize olivine and garnet as being the most significant and the most frequent accessory minerals in diamonds. The olivine was identified by them as forsterite with approx. 6 per cent fayalite (Plate C, 6) and the garnet as pyralspite with a high content of pyrope (Plate C, 7). They also identified brown chrome-spinel (picotite) (Plate C, 8), green chrome-diopside or chrome-enstatite and diamond (Plate C, 9). An occasional incidence of serpentine, chlorite, biotite, and phlogopite was attributed by them to pseudomorphic changes suffered by mineral inclusions previously syngenetic. Reports of chromite, hematite, ilmenite, magnetite, pyrite, quartz, rutile and zircon—all of them minerals which in earlier works by different authors had been asserted as inclusions in diamonds—were considered doubtful or else refuted by the Soviet mineralogists on the grounds that the purely visual identification method used by earlier authors had given rise to confusion and erroneous identification or that, as in the case of quartz and zircon, it had by no means allowed for the genetic criteria pointing to their pre-existence or syngenetic formation.

The Soviet publications[14, 22, 32, 39] furnish comprehensive information on the findings of research into mineral inclusions with reference to conditions of formation, orientation within the host diamond, R.I. and/or D.R., angles of optical axes, ratios of parameter, lattice constants, and S.G. In places these results are accompanied by illustrative photomicrographs and Laue diagrams. All these findings concerning mineral inclusions in diamond tell that such alkaline minerals as olivine, garnet, chrome-spinel and chrome-diopside (chrome-enstatite) were formed simultaneously with the diamond during the metamorphosis of olivine aggregate into what have been called griquaite nodules (as far as the main mineral constituents are concerned, griquaite is similar to eclogite, but while the first is of deep magmatic, the latter is of metamorphous origin) in intracrustal areas of formation[37], while graphite and serpentine are to be looked upon as pseudomorphoses after diamond and olivine, same as chlorite, biotite and phlogopite, being mutations of garnet, are encountered only as secondary fissure-fillings, having penetrated into cracks and slits at a later stage.

158

Stimulated by these interesting findings of the Soviet mineralogists, W. E. Sharp of the Adamant Research Laboratory, Johannesburg, using the powder method performed x-ray analysis on a great number of industrial diamonds. The diagram thus obtained enabled him to identify as inclusions of ore minerals various iron oxides such as goethite, hematite, magnetite and possibly even wüstite. He, too, holds that the serpentine and chlorite observed are epigenetic alterations of syngenetic olivine or garnet crystals respectively.

In one isolated case, he even succeeded in obtaining the line diagram of graphite from material he had scraped off a fissure plane—of which he informed the author personally. He has thus, we may take it, corroborated the Soviet observation maintaining that graphite may be caused by graphitization of diamond in places where a carbon structure has been grossly disturbed, i.e. tension cracks or cleavage fissures, and also in the region of stress between foreign bodies included and the diamond substance enclosing them. Evaluating the findings of his elaborate x-ray tests, W. E. Sharp arrived at the conclusion that pyrrhotite (Plate C, 10) is an ore inclusion occurring rather frequently in South African diamonds, where it is often found intergrown with other ore phases. Most frequently it was found integrated with pentlandite; occasionally with pentlandite *and* pyrite. Such intergrowth probably resulted from the exsolution of an earlier high-temperature phase that was later embraced by a growing diamond. Individual formations of ilmenite, pyrite, or rutile were not found by Sharp either.

An exceptionally interesting feat of identification was achieved in an entirely unorthodox manner by B. W. Anderson. During the examination of dense batches of yellowish-brown mineral inclusions in a Colombian emerald, his spectroscope revealed a spectrum of rare earths displaying an abundance of lines which indicated parisite[47]. Parisite is a rare fluocarbonate of the cerium metals $((Ce, La, Di)_2 \ Ca[F_2/(CO_3)_3].C_3^4)$—and forming pyramidal to slender prismatic crystals varying in tint between brownish-yellow or yellowish-brown and lilac (Plate D, 1). One of its scarce occurrences being Muzo, Colombia, its presence in emerald may well be considered a welcome locality feature.

Realizing the novel technique of combining microscopy with the x-ray powder method, P. C. Zwaan achieved definite analysis
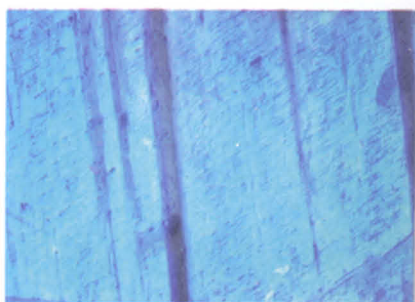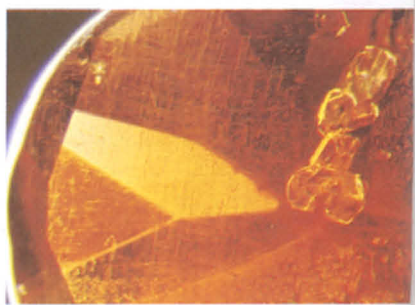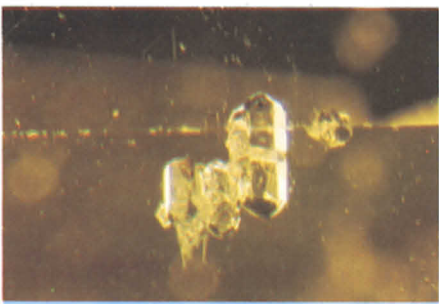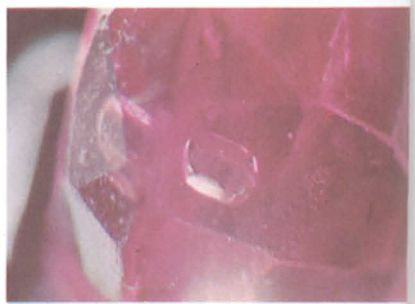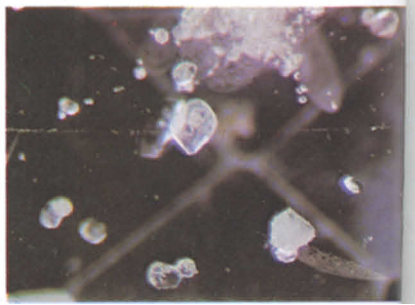
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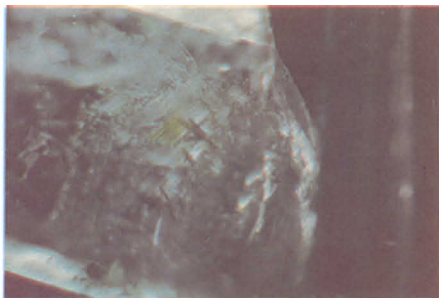
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of a number of internally paragenetic minerals in a variety of gemstones. As early as 1964, several conspicuously hexagonal, prismatic inclusions in a lilac-coloured Ceylon spinel were identified by him as apatite (Plate D, 2)[48], and he has announced the identification of apatite, corundum, phlogopite, pyrrhotite and spinel (the latter confirming earlier observations by this author) in corundum (Plate D, 3) as well as of apatite, muscovite, and rutile (the latter in concurrence with O. Mellis) in almandine[49].

This brief review of the new research techniques developed since the middle of this century, and of the highly noteworthy findings made possible by them with regard to mineral inclusions in gemstones, has—it must be admitted—thrown light on merely a few of the milestones marking the long, often trying, but always fascinating path through this field of investigation: yet it does show how in recent years the most remarkable findings have been made, procuring knowledge which may contribute greatly to elucidating the genesis of gemstones.

Nevertheless, gemmology with its totally axiomatic approach could not rest content with the present state of affairs, since it cannot, in the last analysis, tolerate the destruction of its objects and the great values they so often embody.

Indeed, gemmologists must continue in their search after the techniques and tools that may help them solve their problems without damage to, or even destruction of, their costly materials. In other words, if they wish to abstain from the often destructive methods of mineralogy, they must be concerned with devising instruments that will serve their needs more adequately.

The postulates of gemmology have now been met with to a great extent by the recent invention of the electron microprobe

Plate D

1. *Slender prisms of parisite in emerald from Muzo (Colombia).*
2. *Idiomorphous prisms of apatite in mauve spinel from Ceylon.*
3. *Euhedral octahedron of spinel in Burma ruby.*
4. *Tiny needles of black magnetite in spectrolite from Finland.*
5. *Rutile needles orientated along the edges of the rhombododecahedron in almandine.*
6. *Group of idiomorphous prisms of apatite in almandine from Ceylon (these apatites proved to contain lanthane and thorium).*
7. *Double-ended apatite crystals in kornerupine.*
8. *Stumpy prisms of apatite in ruby (from Ceylon?).*
9. *Slender stalks of apatite in ruby (from Ceylon?).*
10. *Partly xenomorphous, partly resorbed apatite crystals in mauve spinel from Ceylon.*

analysis, which has brought a revolutionary innovation to the field of analytic techniques. Providing the analyst with completely new ways and means of research, this highly impressive new process is of the utmost importance to him by offering, as it does, an exact and non-destructive method of probing into the chemical constituents, and their quantitative ratios, of the substance being tested. It allows a qualitative and quantitative analysis of solid bodies with volumes far beyond the faculties of the unaided human eye, thus obviously including mineral inclusions in gemstones. This very promising instrument is called an electron microprobe and is an elaborate device of considerable dimensions. It is thus highly unlikely that it will ever be included in the equipment of a gemmologist; rather its installation will be restricted to the big research laboratories. More recently, this new device has been employed for mineralogical research tasks in general and for investigating gemstones and their mineral inclusions in particular, and it is to be expected that this will be increasingly so in the future. For this reason a report on the significant results achieved with the new system is preceded by a brief description of the device and its mode of operation.

The first operating electron probe micro-analyser was constructed by R. Castaing shortly before 1949. Designed as a combination of parts of the electron microscope and an x-ray fluorescence unit, it functions as an instrument for the quick and non-destructive qualitative and quantitative analysis of solid bodies with a volume of only a few cubic microns (1 micron = 0·001 mm). Samples to be tested need not be extracted from the substance enclosing them, but they will of course have to be exposed on the surface of it. It is on account of this improved facility of procedure that microprobe analysis proves to be such an advantageous method of investigating mineral inclusions in gemstones. It yields the solutions to many analytical problems which were previously difficult, if not impossible, to solve by conventional, let alone by optical, methods. The electron microprobe emits a finely focused beam of electrons, directing it onto the minute surface of the sample and producing an x-ray reflection characteristic of the chemical elements affected by the electron excitation. The angle of reflection, the intensity and the wavelength of the characteristic x-rays— all of these being factors determined by the structural elements of the material tested—will then be used to furnish the analytical

162

data to determine the elements which are present in the target and their relative mass concentrations. Currently, all such elements can be identified that have atomic numbers upward of 11. In typical cases, the spatial resolving power is between 2 and 3 microns, the sensitivity ranges from $1:10^4$ to $1:10^3$ depending on the composition and expansion of the sample, and the relative accuracy amounts to between 1 and 2 per cent if the concentration is in excess of a few per cent. The highly complex equipment comprises (*a*) an electron beam column, (*b*) a number of x-ray spectrometers, (*c*) a light microscope, (*d*) a high voltage power supply and control circuit, (*e*) one or more vacuum systems, and (*f*) a measuring-and-recording system for the analytical information. Fig. 1 shows the
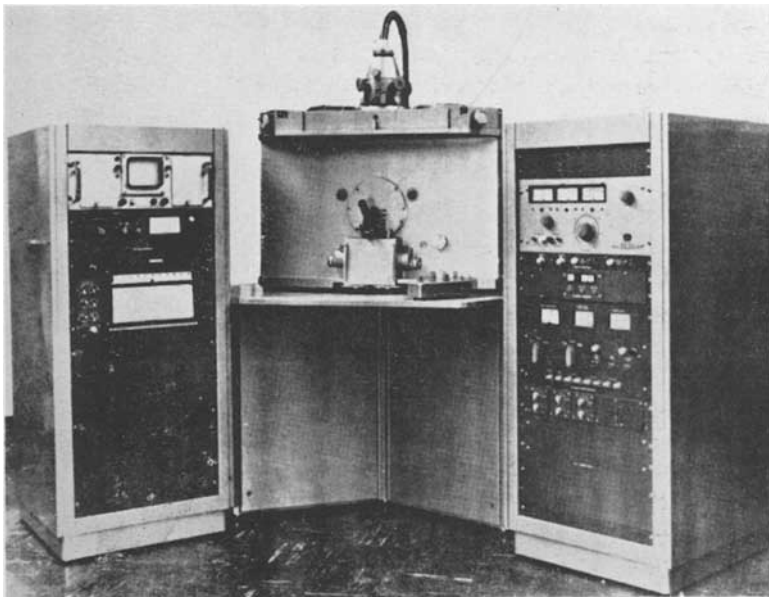
FIG. 1. *A complete electron microprobe laboratory.*

equipment contained in a typical probe laboratory. The most important part of the whole apparatus is the electron beam column housing the electron gun assembly, apertures, magnetic lenses and deflection plates. Fig. 2 depicts a typical example of such an electron beam column. The function of the column is to provide an electron beam of the desired characteristics at the sample surface. The beam must be adjustable in size and density; however, these

1 ELECTRON GUN
2 FILAMENT
3 GRID
4 ANODE
5 ANODE APERTURE
6 CONDENSER LENS
7 CONDENSER LENS APERTURE
8 OBJECTIVE LENS
9 OBJECTIVE APERTURE MASK
10 OBJECTIVE LENS APERTURE AND
   BEAM CURRENT COLLECTOR
11 BEAM DEFLECTION PLATES
12 SAMPLE CHAMBER GATE VALVE
13 SAMPLE

FIG. 2. *Electron beam column with a cross-section diagram.*

parameters must be very stable once established for any programme. The heated filament provides a source of electrons which are accelerated by the field between the anode and source. The acceleration potential may be varied at any time from 2–50 kv, and can be selected by the operator.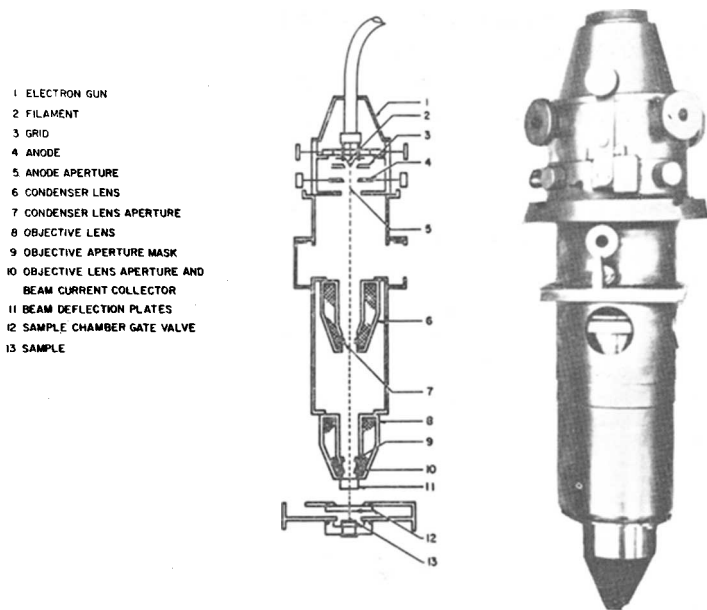 The first magnetic lens, or condensor lens, focuses and diminishes the beam usually at a point well above the objective aperture. The second magnetic lens or objective lens focuses the image of the first lens onto the surface of the sample and provides a further reduction of the image. The beam-current monitor, shown at the position of the objective aperture, is invaluable in duplicating analytical conditions for the measurement of beam stability.

A stream of electrons, narrowed down to a hair's breadth, is generated by the tube and directed precisely onto the surface of the sample to be analysed—which in the case of mineral inclusions in gemstones is the minute cut surface of the mineral included, which has been exposed on the surface of the gemstone by the cutting of its facets. The electron bombardment produces an x-ray reflection characteristic of the atoms encountered. On the one hand, an angle is formed between the electron beam emitted and the x-ray

164

beam reflected, its size being determined by the structure of the atoms constituting the chemical composition of the sample. A goniometer may be adjusted to the x-ray leaving the surface at various angles, thus measuring the typical angle of reflection for each element. In this way, the constituent elements of the sample may be determined. On the other hand, crystals of a suitable material are used to deflect the x-rays according to their wavelengths, and the deflected x-rays are picked up by x-ray detectors. The photons of these x-rays entering the detector are converted into oscillations of electrical energy. The frequency of these impulses is in direct proportion to the intensity of the x-ray radiation received, and this, in its turn, is in direct proportion to the quantitative ratio of the elements producing it within the substance being analysed (Fig. 3).
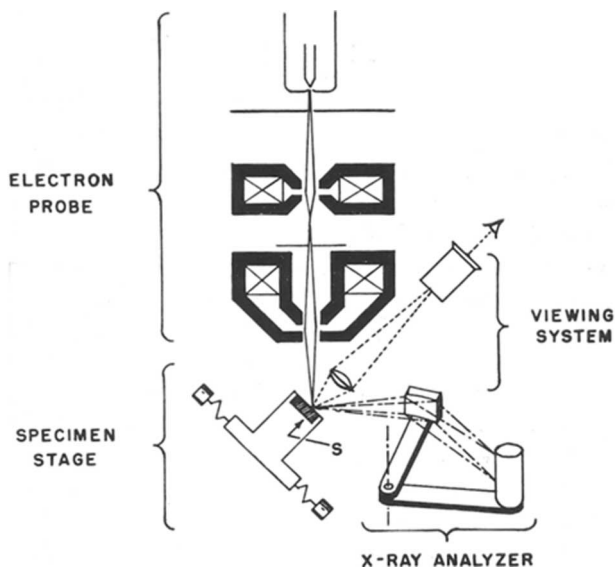
FIG. 3.   *Schematic drawing of an electron micro-analyzer showing the relationship of the electron probe, the specimen stage, the viewing system, and the x-ray analyzer to the specimen, S.*

During the relatively short period that has elapsed since the introduction of this revolutionary innovation in the field of chemical microanalysis, it has already established itself in a wide range of research applications, and it is hardly surprising that gemmologists, too, have not been long in showing interest in it and applying it.

Thus, at the 43rd Annual Congress of the German Mineralogists Association[34], O. Mellis, delivering a lecture about the spectrolite occurring near Ylämaa in Finland, was able to prove the existence of a definite relationship between the chemical composition of the spectrolite and its labradorescent colour changes, as well as to the number of opaque needle-shaped inclusions. He also explained that the process of radiographic determination and the analysis carried out by means of a microprobe identified these same black needle-shaped inclusions (Plate D, 4) as magnetite which was, itself, intergrown with fine ilmenite blades (probably secondarily exsolved from the magnetite—author). Notwithstanding the efforts of eminent mineralogists, it had previously been impossible to determine the true nature of the needles intersecting each other (Plate D, 5) in red garnets of the pyralspite family. However, the combined application of optical and radiographic methods by O. Mellis, in conjunction with electron-beam analysis, has since achieved a definite identification of these needle-shaped inclusions as rutile. He is convinced that its formation is not to be attributed to exsolution but to syngenetic epitaxy and thus represents a phenomenon of growth. These rutile needles are predominantly orientated parallel with the edges of the rhombododecahedron. It is only in Madagascar garnet that they have been observed to be co-ordinated also according to the cube. If parallel to the edges of the rhombododecahedron, the needles are invariably twinned; if aligned, however, with the edges of the hexahedron, they are not pure TiO_2 but are invariably mixed-crystals, which is easily recognized by the difference in interference colours when viewed in polarized light. These reliable findings have considerably increased our knowledge concerning one of the most important and most frequent mineral inclusions in red garnet and are, as such, very much to be welcomed[33, 35].

Almost simultaneously with the author, though without being aware of the coincidence, H. J. Schubnel, using a Castaing microprobe as well as applying a number of combined processes, was analysing several mineral inclusions in a variety of gemstones. He found calcite in ruby and sapphire, spinel in ruby, chloroapatite in spinel from Ceylon, also chloroapatite, pyrite, pyrrhotite and rutile in Ceylon sapphire[40, 41]—thus producing findings happily in accordance with those described in Part II of this study. In a more recent publication, H. J. Schubnel reports that by using the

microprobe analysis he has been able to identify as primarily un-mixed magnetite the black needle-shaped inclusions responsible for the asterism in Indian star-diopside. These are, themselves, inter-grown with secondarily exsolved blades of ilmenite and hercynite [42]. After other analytic methods had failed to detect the colouring chrome jadeite during the examination of jade albite [27, 28, 29, 30] by the author, successful analysis was finally achieved by means of the electron probe following the suggestion of M. Weibel, Professor of Geochemistry at the Institute of Crystallography and Petrology of the Federal College of Advanced Technology in Zurich. The author subsequently obtained permission to use the Institute's AMX electron microprobe to identify the mineral inclusions in a variety of gemstones he had collected over the last 25 years, antici-pating the possible discovery of a suitable method of identification. Since a qualitative evaluation sufficed to diagnose the mineral inclusions in question, the author, in an effort to save time, con-tented himself with a visual assessment of the travel of the graphic indicator, omitting to use the recording attachment which would have traced the relevant spectrograms. A detailed description of the results thus obtained is set out below:

PLATE D, 6 TO 10

Hexagonal prisms of slim to stubby shapes are often encountered in almandine, kornerupine, spinel and ruby from Ceylon. In most cases they are euhedral with a base of medium size. Di-pyramids are rare, but the crystals are frequently double-ended, yet sometimes partly or grossly resorbed. At times, they are sporadic-ally distributed; at others they occur in groups. The microlites are either colourless (i.e. mimicking the colour of the host mineral) or they are olive-green (notably in kornerupines). When viewed between crossed polaroids they are conspicuous owing to straight extinction and vivid interference colours. Their R.I. is distinctly lower than that of their host gems. Examination of the crystal inclusions, exposed on the facet surface, by means of the electron microprobe produced readings definitely indicating the presence of Ca and P.
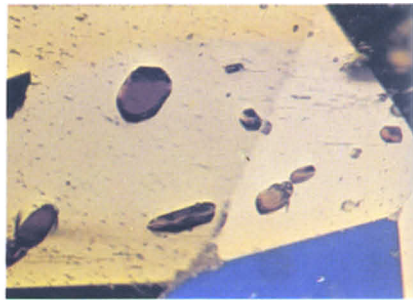
Consequently, these minute crystalline inclusions have been identified as **apatite** $[Ca_5(F, Cl)(PO_4)_3]$. The presence of F cannot be revealed by the microprobe, because its atomic number 9 is below 12, the minimum number detectable by this instrument.
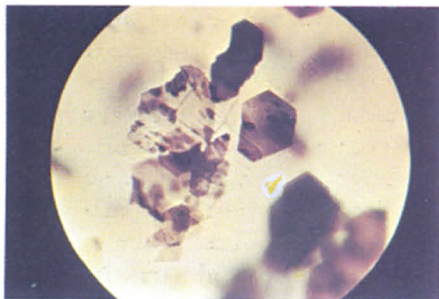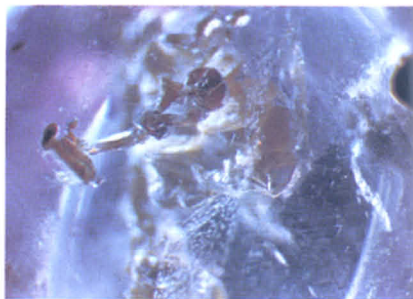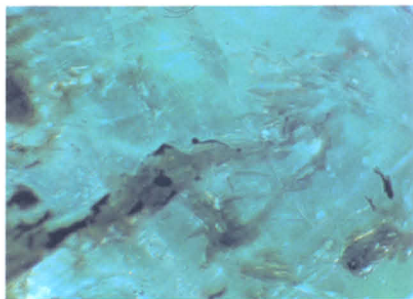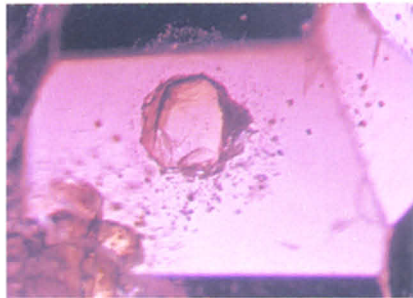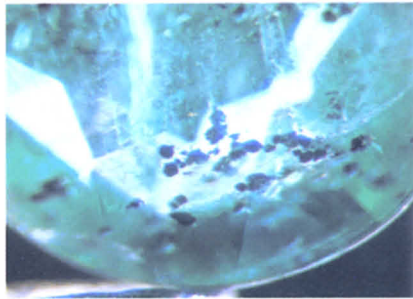
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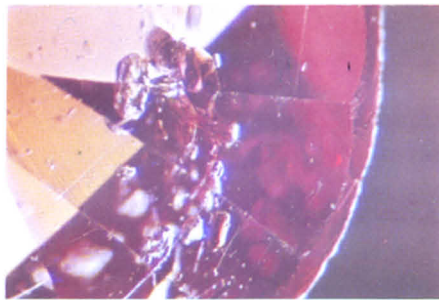
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Apatite crystallizes in the hexagonal system and occurs as long to short columnar or thick tabloid single crystals, or in small crystal groups (as inclusions, they prefer columnar dipyramidal habits). Apatite shows a marked bias for variable chemical composition. Therefore its physical properties fluctuate around the following mean indices: $n = 1 \cdot 642$ and $1 \cdot 646$; S.G. $= 3 \cdot 18$; $H = 5$. Owing its existence to a great variety of formation processes, apatite is widespread as a secondary accessory mineral in a multitude of deposits. It is scarcely ever absent from magmatic rocks and is often to be encountered in pegmatite and pneumatolytic formations, but is also occasionally found in hydrothermal sources (veins and alpine crevasses). In the pegmatite phase, apatite is a primary mineral of early crystallization, though it may also be formed at a later stage of this genetic phase. Accordingly, apatite may occur in gemstones either as a pre-existent or as a syngenetic mineral inclusion.

PLATE E, 1 TO 8

The general appearance of inclusions in almandine, kornerupine, sapphire, spinel from oriental deposits, and also of those in peridot from Zebirget and Burma and those in emerald from the mica-schist deposits (Habachtal, India, Rhodesia, the Urals and Transvaal), is often characterized by varying numbers of inclusions of a mineral ranging in hue between yellowish and reddish-brown or between greyish and dark brown and occurring as isolated, irregularly located scaly flakes—some of them twisted—or in the form of stacked slabs. It is only in peridot that they assume the shape of pseudohexagonal slabs; in other gemstones they are characterized by irregular contours or, at best, by a faint suggestion of imperfect crystalline forms. Their R.Is. are in the region of that

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Plate E
1. *Tabular biotite parcels in almandine.*
2. *Brown biotite platelets in kornerupine from Ceylon.*
3. *Pseudohexagonal and spotted biotite platelets in peridot.*
4. *Dense concentration of biotite in pink sapphire from Ceylon.*
5. *Dark green flakes of biotite in emerald from Transvaal.*
6. *Irregular concentration of biotite flakes from the Habachtal.*
7. *Red-brown phlogopite in sapphire (Ceylon?).*
8. *Tabular phlogopite parcel with broken borders in spinel.*
9. *Microlites of chromite in the centre of a residual fluid drop in peridot.*
10. *Irregular aggregates of chromite in emerald.*

of emerald, and they display a very distinct pleochroism. In polarized light, very vivid interference colours will be seen, while the extinction is usually indistinct. Although optical tests suggested that these guest minerals were members of the mica family, determination of their chemical composition, i.e. the distinction between biotite and phlogopite, with their totally different geological environments, was found to be impossible. The mica family in the restricted sense comprises complex, mostly hydrous silicates of monoclinic pseudohexagonal and marked tabloid crystallization. When scanning these (often rather slim) inclusions bared on the surface of a number of gemstones, the microprobe indicated the presence of the following elements in the case of almandine, kornerupine and sapphire from Ceylon, peridot, and emerald: Fe, Mg, K, Si and Al, and in the case of dark blue sapphire of unknown origin and red spinel from Burma: Al, Mg, K and Si with minute admixtures of Fe and Ti. The constituting elements of **biotite**: $K(Mg, Fe)_3 [(OH)_2(Al, Fe) Si_3O_{10}]$ were identified in the former case, and those of **phlogopite**: $KMg_3 [(F, OH)_2/AlSi_3O_{10}]$ in the latter.

Both these micas crystallize in the monoclinic system and have a marked pseudohexagonal character. The basal pinacoid is the prevalent, often even the only, recognizable form of growth, and this is why the predominantly tabloid to flaky or fine-scaly form is a feature common to all micas. The biotite flakes may also assume an elongated blade-like shape. Except in peridot, the contours are mostly ill-defined or at least indistinct. For all its chemical variability, the optical properties of mica are fairly constant. The only two criteria subject to relatively wide variation are the size of the angle of the optical axes and the type of absorption. But it is these very features, which often differ so enormously within one and the same mica group, that optical methods fail to distinguish between its chemically differing members.

Biotite: common biotites contain, apart from magnesium, not only divalent but also, for the most part, some trivalent Fe, which is responsible for the prevailing dark-brown tints. $n = 1·54–1·64$; S.G. $= 2·8–3·2$; H $= 2·5–3$.

Biotite, as the most common kind of mica, is a widespread constituent of most magmatic rocks—particularly of mica schist—but is also a product of contact and depth metamorphism as well as of pneumatolytic and hydrothermal disintegration. It is there-

fore not surprising that it is encountered in many gemstones. It is mainly found, sometimes in large quantities, in such emeralds as have formed as a result of contact pneumatolytic processes in pegmatite veins of adjacent mica schists (Habachtal, India, Rhodesia, the Urals and Transvaal). The biotite enclosed in such emeralds is of pre-existent descent in that it was transferred—as a relic of resorption—from the mica-schist into the growing emerald and thus incorporated by the latter. The biotite in kornerupine and sapphire from Ceylon is probably to be considered as a by-product of contact metamorphism, while in almandine and peridot it may be safely regarded as a product of depth metamorphism. Thus it settled itself syngenetically in these gemstones.

In sapphire, biotite occasionally contains sparse admixtures of titanium, while in emerald it will display traces of chromium.

Phlogopite differs from biotite in that it contains hardly any iron but it frequently includes fluor. The characteristic fluor may, however, be replaced in part by (OH). Where an abundance of Mg-silicate molecules is combined with Al-silicate, the tints are seldom intense because Mg invariably has a bleaching effect. Although these phlogopites, which often contain more than 30% by weight of MgO, are rich in fluor, the electron microprobe can only determine the absence of iron, but neither the presence of the F nor of the (OH) group. The physical properties of phlogopite differ from those of biotite as follows: $n = 1 \cdot 534 - 1 \cdot 566$: S.G. $= 2 \cdot 75 - 2 \cdot 97$: H $= 2 \cdot 5 - 3$.

Phlogopite is a typical product of the pegmatitic pneumatolytic phase, but it also occurs as a constituent of granular chalks and dolomites of crystalline slate and its contact. Its occurrence in gemstones resulting from formation processes—e.g. the previously mentioned dark blue sapphire and red spinel from Mogok (Burma) —is therefore to be ascribed to syngenetic growth.

PLATE E, 9, 10

Apart from the ill-delineated brown scales, the elongated blades and the stacks of biotite in emerald, and the small biotite tabloids in peridot with their clearly idiomorphous outlines, irregularly formed black granules of ore having a carbonaceous appearance are also frequently present in the internal paragenesis of the two gemstones. In emerald, they occur singly and loosely dispersed or in dense clusters, even in large coarse lumps, whereas in peridot they

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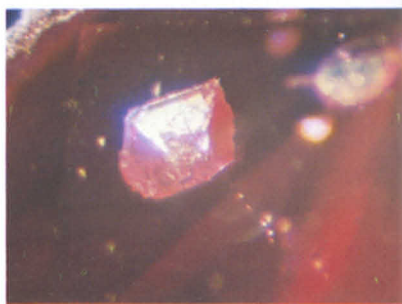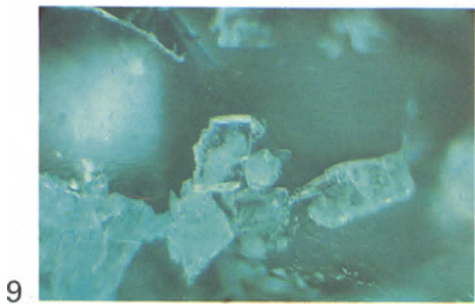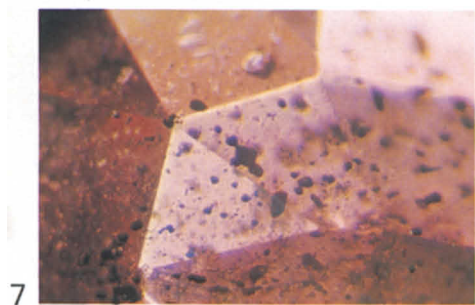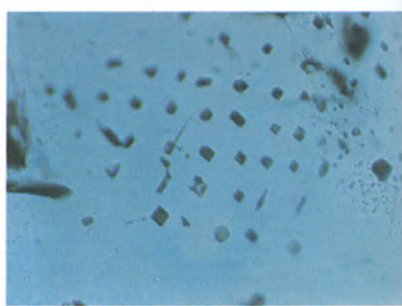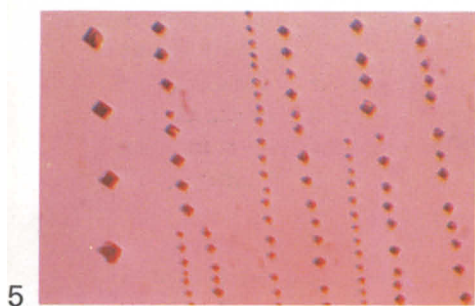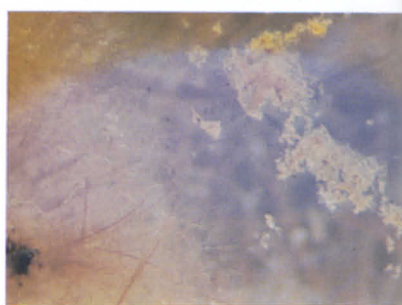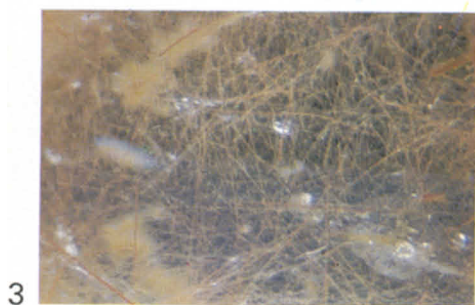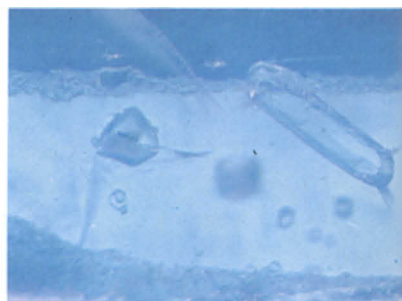
are always found isolated and surrounded by a residual halo. Occasionally minute or flat specimens may appear dark brown. Microprobe analysis identified the chemical elements Cr, Mg and Fe. The ore inclusions thus turned out to be **chromite** of the chemical formula: $(Fe, Mg)Cr_2O_4$. It is sometimes slightly contaminated by other elements such as Al, Mn, Ni, Ti and Zn. Chromite crystallizes holohedrically in the cubic system. It is not magnetic. $n = 2 \cdot 10$; S.G. $= 4 \cdot 5 - 4 \cdot 8$; H $= 5 \cdot 5$.

Chromium ore almost invariably depends on associations rich in olivine and on serpentines originating from them, wherein it occurs as a magmatic precipitate irregularly dispersed or aggregated in clusters. Another indication of its liquid magmatic mode of formation is its occurrence in peridot, in which it was syngenetically produced. The author holds that the halos surrounding each individual granule of chromite are waste liquid drops or dried-up residues of liquid drops which were deposited on growth planes during the growth stages of the peridot, from which small crystals of chromite were subsequently precipitated in the course of a phase displacement. In emerald, however, a syngenetic formation of chromite (which is, after all, a product of magmatic precipitation) would appear to be unlikely since the two minerals are, it must be remembered, separated by a considerable period of genetic construction.

PLATE F, 1

An irregularly shaped mineral fragment of a pronounced light-green colour happened to be exposed in the table of a brilliant-cut diamond, so that one part of the fragment was bared in the surface of the table. This tiny area offered an ideal object for investigation by electron microprobe, and the following elements were detected: Ca, Fe, K, Mg, Cr, Si (no Al). This combination of elements

Plate F
1. *Green fragment of chrome-diopside in diamond.*
2. *Feldspar crystals in sapphire.*
3. *Hair-fine, fox-red fibres of goethite and individual quartz crystals (bright patches) in topaz.*
4. *Dense cluster of quartz grains surrounded by hair-fine fibres of goethite in topaz.*
5. *Tiny octahedra of hercynite arranged in parallel lines in spinel.*
6. *Concentration of hercynite octahedra arranged in parallel array in blue spinel.*
7. *Dense concentration of ilmenite in almandine.*
8. *Idiomorphous calcite crystal displaying a rhombic pattern of lines caused by intertwined twin lamellae in Burma ruby.*
9. *Euhedral crystals, partly well preserved and partly as cleavage fragments, enclosed in emerald from Muzo.*
10. *Calcite, displaying combination of rhombohedron with basal plane, in a red spinel from Burma.*

suggests pyroxene, i.e. augite. Now, pyroxene (augite), having a low alumina and alkaline content with [SiO_4.SiO_2] CaMg as its main molecule, is called **diopside.** In very many cases, Mg is partly replaced by Fe. If chromium is also present, the mineral is called chrome-diopside. Diopside crystallizes in the monoclinic system. The crystals, which are often well-developed, are columnar in shape and are not infrequently found in aggregates of a granular or blade-like structure. $n = 1 \cdot 65 - 1 \cdot 68$; S.G. $= 3 \cdot 3$; H $= 6-7$.

Emerald-green chrome-diopside evolves in olivine rocks and, as the main companion mineral to diamond, is also an important component of its inner paragenesis.

PLATE F, 2

Colourless prismatic or slab-shaped crystals often appear in dark blue sapphires from Burma, Thailand and Australia, embedded either singly or in small groups without bearing any discernible relationship to the crystallonomic directions of their host. Their R.I. is clearly lower than that of the host sapphire, and a more or less oblique extinction can be seen in polarized light. The interference colours change according to the thickness of the small crystals. It is interesting to note that they are almost invariably found in the presence of a red mineral inclusion and black, ore-like grains, which will be mentioned later. Electron microanalysis established the presence of the element groups K, Si and Al, and Ca, Si and Al, thus proving these small bright crystals to be members of the **feldspar** family, more precisely orthoclase $(K[AlSi_3O_8])$ together with another feldspar of the plagioclase species—probably anorthite $(Ca[Al_2SiO_8])$; unfortunately, Na with its atomic number 11 cannot be detected by the electron microprobe. Owing to the considerable mixing ability of feldspars, no absolutely valid constants can be given for these inclusions, only mean values of their physical properties as follows: $n = 1 \cdot 52 - 1 \cdot 58$; S.G. $= 2 \cdot 56 - 2 \cdot 77$; H $= 6-6 \cdot 5$. All feldspars belong to the same morphological type having little inherent variability. Monoclinic and triclinic feldspars reveal the same form of development. Feldspars constitute an extremely important group of rock-forming minerals which originated during various genetic cycles, e.g. the magmatic, the pegmatitic and the contact-metamorphic. Their occurrence in sapphire might be an indication of the pegmatitic origin of this gemstone—a possibility which is reinforced by the

other accompanying minerals of the inner paragenesis of these feld-spar inclusions which were, it may be assumed, syngenetically formed.

Remarkable fox-coloured topazes have turned up in the gem trade from time to time. They are interpenetrated with rust-red needles or hair-like fibres, either scattered sporadically or densely accumulated. On one hand, the linear needles appear to be arranged irregularly, without regard to the crystallonomic direc-tions of topaz, and on the other hand the crooked, twisted and folded fibres form tangled skeins. The more abundantly they occur the more intense is the alien red hue and, of course, the optical turbidity of the host. Apart from the pronounced red tint, crystall-optical analysis can give no positive indication because of the sparse morphological clues and the hair-breadth shape of this type of inclusion. The only possible means of investigation in this case was the highly sensitive and subtle scanning-process of electron analysis. It revealed the presence of one main element, Fe, besides traces of Ti and Ca. The results of this analysis, together with optical examination, suggest the presence of **goethite** (α—FeOOH). This indicates a new and interesting occurrence of goethite in gemstones, particularly since in topaz it is to be attri-buted to syngenetic formation processes and not, as in other cases, to exsolution. Goethite crystallizes holoedrically in the orthor-hombic system, and develops prismatic, short-columnar individuals ranging from needle- to hair-shape, which are elongated after the c-axis. They frequently assemble to form scaly, flaky or coarse aggregates, often displaying a texture of divergent fibres. The optical data is high: $n = 2 \cdot 21 - 2 \cdot 35$; S.G. $= 3 \cdot 8 - 4 \cdot 3$; H $= 5 - 5 \cdot 5$.

Goethite is a characteristic product of the weathering zone and the area of circulating waters. Thus, like limonite, it shows an affinity for the oxydisation zones of iron ore deposits. It is also encountered as an independent mineral with calcite or quartz. Quartz (SiO_2), which occurs either in small, perfectly formed crystals or as tiny sandy grains in loose groups, can be observed in the same topazes paragenetically with goethite. Occasionally, these quartzes are found intergrown with chlorite, for which the electron beam revealed the elements Fe, Si and Al. The syngenetic formation of quartz in topaz is certainly compatible with the pegmatitic genesis of these two minerals, of which either may be the guest of the other.

Gemmologists interested in inclusions are well aware of the fact that the small, black octahedra arranged in straight or curved lines are a diagnostic criterion of spinel. They were first mentioned and depicted by B. W. Anderson[1]. For a long time they have aroused the analytical curiosity of a number of scientists concerned with inclusions, and some years ago—because of their appearance and the limited possibilities of optical identification—the author claimed them to be magnetites.

These small octahedra, which are black with a metallic lustre, are more or less idiomorphically developed but often strongly distorted. They seldom occur singly, but are usually arranged in large numbers in straight or crooked lines and, as such, traverse large areas of the interior of the host. Thanks to their fairly widespread occurrence in spinels, it was not particularly difficult to find several specimens in which these little black crystals were exposed on a polished facet. Electron bombardment of the grains thus bared on the surface disclosed the chemical elements to be Fe and Al, as well as slight traces of Si and Ca. The latter may be regarded as insignificant impurities. Thus, these small black octahedra are not magnetite (Fe, Fe_2O_4) but a related member of the spinel group, namely **hercynite** $(Fe\ Al_2O_4)$—a very welcome discovery which somehow fits better into "the chemo-genetic picture". Hercynite is otherwise known under the pseudonym of iron spinel. Its physical properties are exactly those of a high-ranking member of the spinel group: n = approx. 1·80; S.G. = 3·95; H = 7·5–8. Hercynite is predominantly of liquid-magmatic origin and forms coarse crystals with an indication of octahedral development. It often occurs as an accompanying mineral in titano-magnetite deposits and is also widespread as an exsolution-product of magmatically formed titano-magnetite. Its occurrence may well be attributed to unmixing processes in spinel as well. Lamellar hercynite has recently been identified by H. K. Schubnel[42] as being an exsolved by-product in magnetite, which is responsible for the four-rayed asterism in diopside.

A blackish-red almandine of unknown origin was so thickly interspersed with small black and splintery grains of ore with metallic lustre, that the host had almost completely lost its trans-

176

parency and appeared unnaturally dark. Crystalloptical methods were unable to yield information as to the nature of these small ore-like splinters of black, ferrous appearance, although many of them were flush with the surface of the almandine. Careful scanning of the surface by the electron beam revealed the presence of Ti and Fe, thus suggesting the titanic iron ore **ilmenite** ($FeTiO_3$).

The titanium-bearing iron oxide crystallizes in the trigonal-rhombohedral system and is homeotypically akin to hematite. It develops a partly tabular, partly rhombohedral habit. Its R.I. varies according to its Fe_2O_3 content and tends to be higher than $n = 2\cdot72$; S.G. $= 4\cdot5$–5; H $= 5$–6.

Ilmenite is predominantly a liquid-magmatically formed mineral of alkaline igneous rocks; this, in itself, explains its occurrence in the inner paragenesis of garnet, which originated under identical conditions. It originated syngenetically with its host and was enveloped by the latter as a secondary constituent of the same phase of formation. It is also possible that ilmenite is genetically older and was enveloped by garnet, but ilmenite is known to have formed in crevices of the St. Gotthard, so that syngenetic formation can hardly be excluded. Ilmenite in beryls was previously identified by W. F. Eppler, using optical methods[7], to be an inclusion resulting from exsolution. Moreover, ilmenite, as an exsolved mineral, accompanies other iron ores in gemstones[42], e.g. as lamellae in the magnetite needles in star-diopside or in the pyrrhotite in ruby and sapphire.

PLATE F, 8–10

Colourless, transparent crystals of euhedral habit of the rhombohedral system and their cleavages are known as inner-paragenetic features of rubies and spinels from Mogok, and emeralds from the Muzo mine in Colombia, especially when they occur only sporadically and do not predominate in groups or extensive intergrowth. Many of these guest crystals are distinguished by twin lamellae parallel to $< 01\bar{1}2 >$ on whose lamellar planes weak interference colours appear when the gem is revolved. Under extreme magnification two systems of alternately broad and slim lamellae can often be recognized. The R.I. is clearly lower than that of ruby and spinel, but roughly corresponds (with variations on either side) to that of emerald; D.R. is very pronounced. There are two reasons for assuming that the enclosed mineral in question is calcite:

177

the first is the appearance and optical behaviour of the material, the second is the specific occurrence of the host minerals in a source of rock of granular, marble-like limestone, in which they were formed by contact metamorphosis. Since, however, scientific research cannot be content with mere assumptions, the microprobe was the only possible means of obtaining a conclusive answer. All of the exposed samples of the above-mentioned mineral inclusions revealed only Ca. Since C and O cannot be analysed because of their low atomic numbers, **calcite** ($CaCO_3$) is the only possibility here. Calcite is a typical representative of the trigonal-scalenohedral class of crystals.

Its habit and face-development are strongly influenced by accompanying solvents. The number of its various forms of growth, and of their various combinations, is therefore very high. In Burma ruby and in Muzo emerald, the rhombohedral growth and cleavage form would seem to predominate and of the various possible twinnings the most frequent would appear to be poly-synthetic lamellation after $<01\bar{1}2>$. $n = 1 \cdot 48 - 1 \cdot 65$; S.G. $= 2 \cdot 71$; $H = 3$.

As a free mineral, calcite is formed by liquid-magmatic processes under pressure in magmas having a low SiO_2 content, as well as in the last stages of pneumatolytic genesis. Furthermore, sedimentary formations may also supply limestones. Under the influence of metamorphoses the latter may be transformed to marble. Contact metamorphosis of this nature, which took place under the impact of magmatic melts, is the origin of ruby and spinel in Mogok and of emerald in Muzo, and also of the syngenetic precipitation of calcite inclusions in such gemstones.

PLATE G, 1 AND 2

The fact that calcite also occurs in spinel from Mogok may not cause surprise, when it is considered that the growth conditions are the same as for ruby: it came, however, as a surprise when the electron analysis disclosed that calcite does not always occur alone in spinel, but is frequently interspersed with **dolomite** ($CaMg[CO_3]$) and forms interesting intergrowths with the latter. These "double crystals" appear to adopt xenomorphic habits which are either octahedral, hexahedral or even irregularly warped. Their distribution corresponds exactly with the modes of concentration of hercynite, being arranged singly, in groups or in lines. Their

178

appearance frequently seems to suggest that they might be fillings in negative crystals, half of them consisting of striped (twinned) calcite, the other of dolomite.   In polarized light they extinguish alternatingly (i.e. in different positions) and in bright light they reveal vivid interference colours, while the lamellar structure of calcite is conspicuous with strong contrast.   The refractive indices of both foreign crystals is markedly lower than that of spinel. Allowing for the fact that the country rock which was involved in the contact metamorphosis from which the spinel originated consisted of dolomitic limestone, it is easy enough to understand the genetic preconditions leading to the development of these interesting double crystals.   Alternatively, however, the dolomite might have come into being as a result of partial displacement and metasomatosis of the calcite.

PLATE G, 3

Apart from the two afore-mentioned granular inclusions of chromite in emerald, another guest ore having a leaden-grey hue and a high degree of metallic lustre was observed.   It is characterized by a tabular habit with indistinct edges and its appearance distinguishes it unequivocally from all the other ore-like, dark-coloured foreign bodies found in emerald.

The chemical elements Mo and S having been revealed by means of the electron beam, this type of inclusion could be identified as **molybdenite** (MoS_2).

The high R.I., $n = 2\cdot03\text{-}4\cdot33$ is betrayed by the vivid metallic lustre easily perceptible on the inclusion.   S.G. $= 4\cdot75$; $H = 1\text{-}1\cdot5$.

Molybdenite is a mineral characteristic of the perimagmatic to contact-pneumatolytic ranges also being present in pegmatite veins.   Consequently it boasts the same conditions of formation as emerald and, therefore, may well have crystallized syngenetically with its host.

PLATE G, 4

In dark blue sapphires from oriental occurrences—probably from Kyaukpyatthat near Mogok in Burma, Kanburi or Bang-Kha-Cha in Thailand—or from Anakie in Australia, pitchblack microlites of a metallic lustre are occasionally revealed under the microscope.   They are conspicuous due to their well-defined microtabular or columnar habit, and this will be more marked when the

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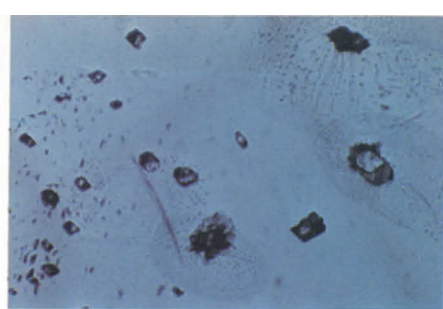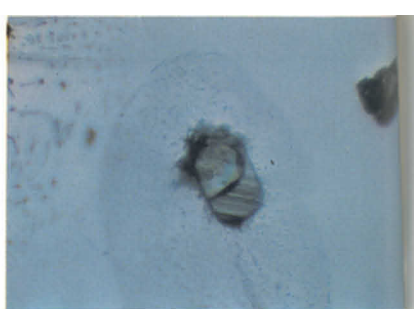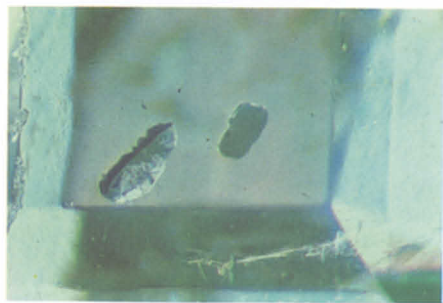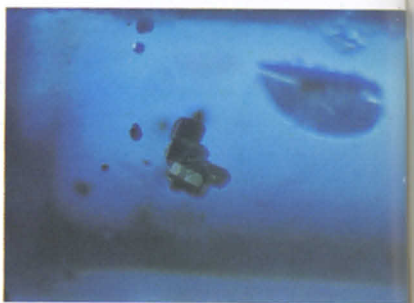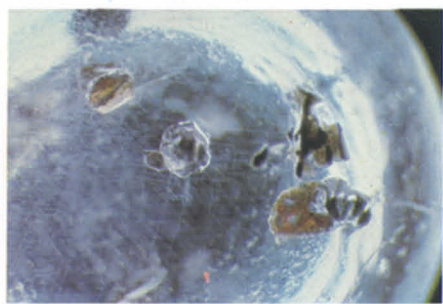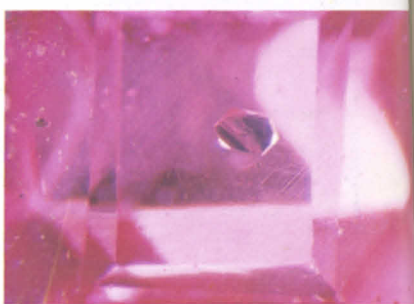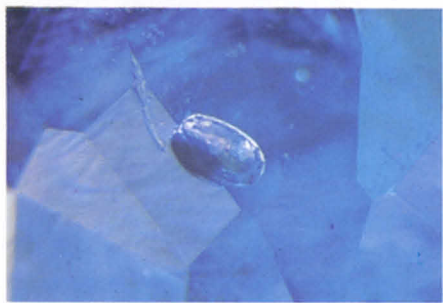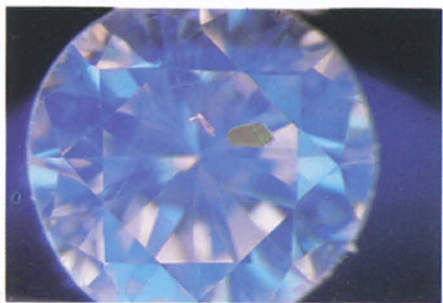
small crystals occur singly or in small groups; as their numbers increase they rather tend to give an impression of isometric shapes. The quality of polish obtained when they are cut in the surface of a sapphire would indicate medium hardness. The crystalloptical method failed to yield a definite identification of the chemical nature of this type of inclusion. Electron bombardment, however, revealed the presence of the elements Nb, Fe, Mn and some Ta (and, in one case, even traces of Ti), so that the mineral could finally be identified as **niobite** (Fe, Mn) (Nb, Ta)$_2$O$_6$. Niobite, with its high content of Nb and low content of Ta, is an end member of the isomorphous series collectively referred to as columbite. The guest mineral, having a low content of Ta, crystallizes dimorphically, i.e. in the tetragonal and rhombic systems, involving a divergent form of development: members rich in Nb will prefer tabular or prismatic habits. The refractive index with a value of 2·45 is much higher than that of sapphire, while its specific gravity, amounting to 5·3, will effect an increase of the density of the host when it is present in large amounts. H = 6.

Niobite is a widespread and typical companion of granite-pegmatites and its accessory occurrence in sapphire indicates that this gemstone from ,the above-mentioned deposits emanated from granite-pegmatitic contact melts. We may safely assume that the formation and inclusions of niobite were syngenetic.

PLATE G, 5

Pyrite, the "Jack-of-all trades" of the mineral world, was identified early on by crystalloptical methods. It is conspicuous

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Plate G

1. *Xenomorphous intergrowths of dolomite and calcite partly filling octahedral cavities inblue spinel.*
2. *Enlarged area of Fig. G/1 exhibits the difference between dolomite and calcite in one of the filled cavities.*
3. *Two tabular fragments of molybdenite in emerald.*
4. *Well-developed microlites of niobite in sapphire.*
5. *In the centre of the picture there is a slightly damaged pyrite accompanied by flakes of biotite (brown) and fine "silk" of rutile needles in sapphire from Ceylon.*
6. *Pyrrhotite of ideal development in ruby. Diagonal lamellae of some exsolved minority component are distinctly seen.*
7. *Resorbed pyrrhotite grain in sapphire.*
8. *Grains of pyrrhotite and pentlandite of varying sizes and irregularly dispersed through na emerald.*
9. *A crystal of olivine flush with the table-facet of a brilliant-cut diamond.*
10. *A fragment of chrome-diopside exposed in the surface of the table-facet of a brilliant-cut diamond.*

not only by its unmistakable shade of brass-yellow and its pro-
nounced metallic lustre, but also by its surface physiognomy and its
growth forms which are, generally, typically paramorphic. The
very fact that it will most frequently precipitate in a variety of
formation cycles ensures that it is encountered in many types of
rocks and, as an inclusion, in various types of gemstones. A
number of completely xenomorphous and pseudomorphous indi-
viduals (such as some partially distorted shapes counterfeiting
yellowish-brown tetragonally prismatic zircons with pyramidal
heads) whose exposed surfaces exhibited a high reflecting power,
were subjected to electron micro-scanning. Since pyrite is noted
for its tendency to form fantastic aggregates, it did not come as a
surprise, on this occasion, when the examination revealed the
presence of Fe and S, thus identifying the inclusions as **pyrite**
(FeS_2).

Pyrite is remarkable for its extraordinary variety of forms.
The habit of its crystals is predominantly cubic, octahedral or
pentagondodecahedral involving a score of combinations. The
crystals are usually well-defined. In addition there is also a
diversity of aggregate formations. S.G. =approx. 5; H = 6–6·5.

Pyrite is known to be ubiquitous among all minerals and can
be formed under almost any conditions. When occurring in such
gemstones as fluorite, sapphire and emerald (from Chivor), which
were likewise generated in completely different formation processes,
pyrite must certainly have crystallized syngenetically with its host.

PLATE G, 6–8

The many varieties of black ore inclusions, ranging in appear-
ance from carbon-dull to an adamantine metallic lustre, do not
usually permit a definite identification by microscopic methods—
although they sometimes display clearly recognizable morphological
characteristics. Fortunately, they are mostly present in such large
quantities that there are almost always a number of specimens
caught on the surface of the host stone, which are ideal objects for
electron microprobe investigation. These ore specimens, ranging
from black to tombac brown in colour, are seldom euhedrally
developed, but are mostly of granular or completely rounded shape:
they can be observed in ruby, sapphire and emerald. They are
normally microscopic, but they may grow to such sizes that they
are perceptible to the naked eye. The microprobe disclosed the

elements Fe and S, occasionally in conjunction with traces of Ni. The ore inclusions examined were thus corroborated as **pyrrhotite** (FeS). S.G. $= 4 \cdot 6$; H $= 4$.

Pyrrhotite crystallizes in the hexagonal system. The rare crystals constitute hexagonal slabs with stumpy prisms, which are frequently characterized by extremely fine, lamellar structure. The low nickel content is present in inserted pentlandite lamellae which were precipitated as a result of exsolution during cooling. The principal home of pyrrhotite is usually the basic plutonic rocks in which it can accumulate in certain areas and form extensive deposits. It is, however, also present in pegmatitic-pneumatolytic phases and in hydrothermal crevices and was thus liable to be precipitated together with the growing corundums and emeralds, and to be included by them.

When the quantitive proportion of nickel is relatively high, the mineral in question is **pentlandite** $(FeNi)_9S_8$—a paragenetic compound which has been shown to be present in ruby and emerald. This nickel ore crystallizes in the cubic system and its lattice is characterized by an extremely close spherical packing of the S atoms. The mineral, which is generally of a coarse texture, very rarely displays clear crystal faces. Unlike pyrrhotite, it is not magnetic, but possesses the same range of colour from bronze to tombac brown. Its hardness is 3–4 and its S.G. $4 \cdot 6$–5. This nickel ore is native to all liquid-magmatic pyrrhotite deposits and its co-existence with pyrrhotite in internal gemstone parageneses may possibly be attributed to pre-existent origin. The former frequently embraces the latter in meshes or cells and is then partly an exsolution product of nickel-bearing pyrrhotite.

PLATE G, 9 AND 10

Crystalloptical and radiographic methods have established beyond doubt [8, 18, 20, 21, 31, 36] that olivine belongs to the family of minerals constituting inclusions in diamond: so that further corroboration would hardly appear to be necessary. Nevertheless, a small colourless crystal found to be cut flush with the table of a brilliant-cut diamond appeared to be such an ideal subject that the author could not resist examining it under the electron microprobe. The little guest-crystal revealed a positively lower R.I. than the diamond. In polarized light it extinguished almost parallel with the c-axis. It also displayed vivid interference colours, and the

183

hardness of its cut surface proved to be rather low. The diagnosis, duly expected in view of these preliminary observations, was confirmed by the electron beam analysis on account of the detection of Mg, Fe and Si: in other words, the crystal included was identified as an **olivine** crystal $(Mg, Fe)_2SiO_4$, which had grown epitaxially and orientated on the octahedron, as do all olivines in the process of simultaneous diamond growth.

Another small crystal of similar appearance and behaviour was located in another brilliant-cut diamond, again having been exposed on the gem's table. It did not, however, live up to expectations, but instead revealed the elements Ca, Mg, Fe and Si, together with traces of Ti and Al, under electron bombardment and is, therefore, a **diopside** $(CaMg\ Si_2O_6)$. This enrichment of the inner mineral-association of the diamond is certainly in accordance with the current hypothesis that diamonds were formed as the byproduct of a high-pressure, high-temperature transformation of olivine rock into so-called griquaite nodules among whose constituent minerals olivine and diopside are to be numbered. Consequently, diopside was also formed syngenetically with diamond. Whenever chromium was present simultaneously, emerald-green chrome-diopside could result from the inclusion of minute traces of the pigmenting transition element.

PLATE H, 1–3

The typical aspect of twinned aggregates of the sagenitic textures of rutile needles precipitated as a result of exsolution in blue and red corundum from Burma and Ceylon have long been known and have also been confirmed beyond doubt by optical and chemical tests. When densely accumulated, they are responsible for the well-known and much-prized asterism in rubies and sapphires. While, when viewed in transmitted light, the disassociated rutile needles appear as brown to black dashes, dark-field or lateral illumination will reveal them as bright lines intersecting according to three principal crystallonomic directions. Other morphological shapes are hardly likely to be familiar to a gemmologist, since he is less well-acquainted with the granular to columnar prismatic habit encountered in nature. On the other hand he will presumably be well aware of the fact that rutile occurs in such different tints as golden-yellow, reddish-brown and fox-colour, but will be less familiar with its crimson, dark-brown and black varieties. Such

individual specimens of **rutile** (TiO_2) in sapphire and coarse aggregates in kyanite—which have certainly not subsequently evolved as a result of exsolution but, rather, simultaneously with the formation of the host crystal from the mother melt—were detected by electron-ray scanning, showing Ti to be the main element with Fe occurring as an occasional impurity. The crimson specimens consist of pure Ti, while the brown and black individual specimens obviously owe their dark shades to an admixture of Fe. Morphologically, rutile belongs to the tetragonal system and generally it prefers elongated prismatic habits of stout columnar as well as needle- to hair-like shapes, forming the quaintest knee-twins and polysynthetic or cyclic multiples. Many of the exsolved twins are conspicuous by their lance-like appearance, whose laws of intergrowth have not yet been determined. $n = 2.616$ and 2.903; S.G. $= 4.25$; H $= 6$. Apart from the fact that rutile frequently occurs as a secondary product of titanium-bearing mineral, it is also widespread as a microscopic secondary constituent assuming the shape of very subtle needles in sedimentary and metamorphic and partly also in igneous rocks. In areas of pronounced metamorphosis, and also in pneumatolytic veins and druses formed hydrothermally, it will occur as large and well-defined crystals. It is certainly this cycle of formation that is responsible for the syngenesis of substantial individuals as well as aggregates of rutile in almandine, disthene, corundum and quartz.

PLATE H, 4 AND 5

Burmese ruby frequently betrays its origin by the pronounced features of its endogenesis, which not only comprise whirly colouring and clustered concentrations of exsolved rutile needles, but also the presence of a diversity of associated minerals. Apart from the guest minerals, already discussed, pale to deep-yellow individual specimens, having a habit reminiscent of the shape of an envelope, have also been encountered. Their broad relief is indicative of a high refractive index, which is considerably above that of the ruby. The high birefringence is expressed in the extinction and the vivid interference colours. In the deep-tinted specimens, the pleochroism is clearly perceptible. The conclusions derived from these observations were confirmed beyond doubt by a number of microlites exposed in the facets of some rubies, in that the electron analysis

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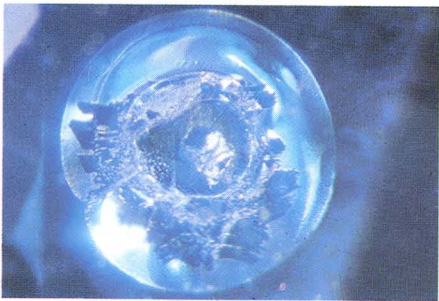
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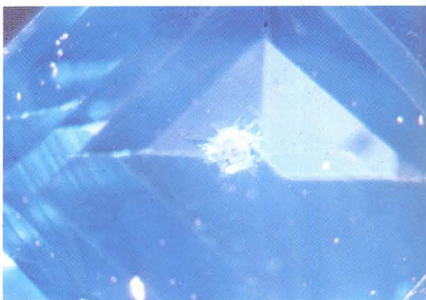
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led to the detection of Ti, Ca, Si (and Al): hence the mineral included is **sphene** ($CaTi[SiO_5]$).

Sphene crystallizes in the monoclinic prismatic system with flat prismatic, tabular, columnar or wedge-shaped habits and yellow, green and brown shades. Its optical properties are quite outstanding: $n = 1 \cdot 90 – 2 \cdot 05$; $\Delta = 0 \cdot 105 – 0 \cdot 135$; S.G. $= 3 \cdot 4 – 3 \cdot 6$; $H = 5 – 5 \cdot 5$. The mineral is a widespread accessory mineral in intermediate igneous rocks, primarily in metamorphic rocks. Its syngenetic precipitation together with ruby is completely congruent with the process of formation of its host, since in the contact-metamorphic melt the necessary constituting elements emanated from limestone-marble, as far as Ca is concerned, but from granite magma with regard to Si and Ti.

PLATE H, 6

At times, the internal paragenesis of dark blue sapphires is very intriguing because of the presence of small red crystals which, by the intensity of their colour, provide a vivid contrast and liven up the endogenetic picture. The microlite inclusions are encountered singly or loosely grouped and are of such positively idiomorphous shape that, in previous studies of mineral inclusions, the small clearly isometric crystals were thought to be spinels or garnets. In some cases they seem to be closely related to adjacent residual fluid drops from which they may have been precipitated. It is interesting to note that the two feldspars previously described were invariably encountered in the company of such small red inclusions.

It came as a particular surprise when electron-beam analysis indicated a complex compound of the elements Nb, Ca, U, Ti, Fe and Ta, with small quantities of both Th and Si, thus revealing that

Plate H
1. *Irregular conglomerations of black and red mineral, whose chemical composition of TiO_2 indicates either anatase or rutile in cyanite.*
2. *Huge prisms of red rutile in sapphire from Ceylon.*
3. *Black rutile resorbed into a ball-like shape and surrounded by a stress-halo in sapphir from Montana, U.S.A.*
4. *Well-developed crystal of sphene in ruby from Burma.*
5. *Group of pale-yellow sphene crystals in Burma ruby.*
6. *Brilliant red and euhedral crystals of uranpyrochlore (hatchettolite) in sapphire (the bright spots are feldspars).*
7. *Rounded grains of metamict zircon surrounded by tension cracks in kornerupine from Ceylon.*
8. *Bright zircon surrounded by tension cracks which contained trace-impurities of anhydrite and hematite in sapphire.*
9. *Idiomorphous, yet slightly resorbed high-zircon in mauve sapphire from Ceylon.*
10. *Crystal of high-zircon—somewhat irregularly developed—in kornerupine from Ceylon.*

these pretty, red individuals were a member of the pyrochlore group. In fact the presence of U identified it more closely as the **uranpyrochlore** mineral answering to the chemical formula (Ca, Fe, U) (Nb, Ca, Ti)$_2$ (O, OH, F)$_7$ and called hatchettolite.

Uranpyrochlore crystallizes in the octahedral system forming idiomorphous individual crystals. The mineral is predominantly native to granite pegmatite. It is worth-while mentioning that in the course of this study and thanks to the electron microprobe we have come across three typical pegmatite minerals included in sapphire: feldspar, niobite and uranpyrochlore, which leads to the conclusion that at least some sapphires are of pegmatitic origin.

PLATE H, 7—10

The peculiar inclusion characteristics represented by spherical to ellipsoidal grains have been known for some time now and have frequently been the subject of lively controversy. These characteristics constitute a locally typical criterion of the inner paragenesis of almandine, kornerupine, sapphire and ruby from Ceylon. The small, round xenogenetic bodies—traditionally regarded as metamict zircons—may easily be recognized by their single or multifold halos, which B.M. Shaub[44] identified as tension cracks. Isotropization, i.e. the structural disintegration of once-normal high-type zircon inclusions into metamict low-type zircons is always accompanied by an expansion of volume causing fine circular or radiating fissures resulting from internal stress in the embracing gemstone. Under low-power magnification, they faintly resemble the squatty beetle inclusions in amber and they will, occasionally, be distinct from their host crystal by virtue of a pale-yellow colouring and, invariably, because of their broad dark relief. Crystalloptical methods failed to provide better identification of these isotropic mineral inclusions; electron beam microprobe analysis, however, successfully proved the presence of Zr and Si and also, in the fissures emanating from the solid substance, of Fe, Ca and S. Though the mineral inclusion has thus been identified as **zircon** (ZrSiO$_4$), nothing has as yet been established as to its structural condition, i.e. whether it is present in a crystalline or amorphous form. However, it is interesting to find an indication of impurities in the fissures which are indicative of **hematite** (Fe$_2$O$_3$) and **anhydrite** (CaSO$_4$). Parallel with the formation of small fissures, traces of the product of disintegration have obviously penetrated the stress-cracks. This

may also explain the brownish coloration of some of these tension halos in that they probably emanate from minute traces of iron which are almost invariably present in disintegrating mineral inclusions. Bearing in mind that green metamictic zircons have been found rather widespread in the gem places of Ceylon, it is not really surprising that metamict zircons should share among the mineral-association included in a good many Ceylonese gemstones. Indeed, zircon in all its structural conditions, i.e. in the form of low as well as medium and high-type zircon, is one of the principal constituents by volume of the island's gemstone deposits.

By virtue of their lower readings, the physical data of the metamictic zircons are distinct from those of normal zircons: $n =$ approx. 1·8; S.G. = approx. 4; H = approx. 7.

Apart from its metamict form, zircon is also present as crystalline specimens with well-defined crystal shapes and has as such been identified by electron microprobe in almandine, kornerupine and sapphire. Individual specimens are the most frequent, although small crystal groups and irregular inter-growths have also been encountered from time to time. Zircon is also found in large numbers as a microscopic secondary constituent in certain syenitic igneous rocks (magmatites). As an accessory it is rather widespread and hence it is also found in crystalline schists and clastic sedimentary rocks. As a mineral of primary precipitation it occurs as a pre-existent mineral inclusion, but owing to separation from metamorphic formation processes it is also encountered as a syngenetic internal companion of its host gems.

Now, because the mineral inclusions identified by electron-beam microprobe have been discussed individually, readers might be misled into thinking that they had been included in each particular host gemstone in isolation, i.e. without the presence of other minerals. This is not the case: in fact they often constitute the inner paragenesis in conjunction with one another and occasionally in conjunction with other minerals, some of which were identified at an earlier stage, while others still remain to be identified as yet. In the gemstones examined, they were encountered in the following groupings:

189

Almandine:	Apatite, biotite
	Apatite, pyrite
	Ilmenite, quartz, zircon
	Rutile which was found in several almandines in the form of needles, was not considered worthy of further analysis.
Aquamarine:	Quartz with apatite and epidote
	Biotite alone
Diamond:	Chrome-diopside, diamond, garnet, olivine
Disthene:	Potassium feldspar and rutile or anatase
Kornerupine:	Apatite, biotite (chalcopyrite?) (garnet?), zircon
Peridot:	Chromite alone
Ruby:	Apatite, calcite, rutile, spinel, sphene, Pyrrhotite, rutile, sphene
Sapphire:	Biotite (phlogopite), rutile, pyrite, zircon (The mica is often of the titanium-bearing variety.)
	Pyrrhotite and rutile
	Niobite alone
	Feldspar (plagioclase and potassium feldspar), niobite, uranpyrochlore (hatchettolite)
	Rutile alone
	Zircon with hematite and anhydrite.
Emerald:	Biotite, chromite ⎫ The biotite is usually of the
	Biotite, pyrrhotite ⎬ chrome-bearing variety.
	and pentlandite ⎭
	Calcite
	Molybdenite
	Pyrite
Spinel:	Apatite
	Calcite alone or intergrown with dolomite.
	Dolomite intergrown with calcite, hercynite.
	Hercynite alone
	Phlogopite
Topaz:	Goethite and quartz, the latter occasionally with chlorite.

It goes without saying that all the aforementioned mineral inclusions will also constitute an inner hermit-paragenesis on their own, i.e. without the presence of companion minerals.

College of Advanced Technology, Zurich, who by his helpful suggestions, unfailing interest and valuable support made the electron-beam analysis possible, and also wrote the reports of the experimental findings. Thanks are also due to Mr. R. Gubser for kindly agreeing to prepare the samples and carry out the painstaking examination of them, a delicate and time-consuming task to which he devoted a considerable amount of spare time. The author is also most grateful to Professor F. Laves for his permission to use the instruments and for generously placing colleagues at the author's disposal for this purpose.

The author is also indebted to Professor O. Mellis and particularly to Professor K. Chudoba for their thorough and critical examination of the present study and for their valuable suggestions for its improvement.

The findings published in this article constitute a first report on the initial stages of a steadily growing trend towards a comprehensive investigation of mineral inclusions in gemstones. The author is fully aware that certain of his conclusions regarding genetic conditions are in need of more precise scrutiny and corroboration: nevertheless, he hopes that this preliminary report might inspire some scientists interested in mineral inclusions to persevere in their research into the identity of the numerous mineral inclusions that have not yet been identified, so that our incomplete knowledge of the locally typical associations of mineral inclusions may be completed along with our knowledge of their genetic processes.

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ASSOCIATION

N O T I C E S

RECEPTION

The Officers and Council of the Association held a reception at Goldsmiths' Hall, London, on Wednesday, 27th November, 1968, to mark sixty years of gemmology in Great Britain. The principal guests were the members of the Council of the National Association of Goldsmiths. Mr. Norman Harper, Chairman of the Association, presented two pieces of antique silver to the Chairman of the National Association, Mr. Alan Henn, in appreciation of the work of the N.A.G. in promoting and encouraging the study of gemmology since 1908. During the reception, which was attended by two hundred and fifty people, the Arms of both organizations were on display, together with the world's first gemmological diploma, awarded to Mr. S. Barnett in 1913.

A telegram was received from thirty-four Fellows in Finland sending their good wishes for the occasion.

PRESENTATION OF AWARDS

There was a record attendance at Goldsmiths' Hall, London, on 23rd October, 1968, for the annual presentation of awards. The meeting attracted recipients from many parts of the world, including Burma, Holland, Rhodesia, New Zealand and Spain.

In welcoming members, award winners and their guests, Mr. Norman Harper, Chairman of the Association, specially referred to the presence of Professor M. Font-Altaba and Dr. J. M. Bosch, of Barcelona University. Spain had provided the largest overseas centre and the Association was grateful for all their work. There had also been a substantial entry from Burma. The Chairman recalled the early days of gemmology when in 1913 there had been twelve candidates for the preliminary and eight for the diploma examination. This year there had been 380 and 244 candidates for the preliminary and diploma examinations respectively. He paid special tribute to the examiners, who had a most difficult task, not only dealing with varied writing but problems of translations as well. The Chairman congratulated Dr. G. F. Claringbull, the senior examiner, upon his appointment as Director of the British Museum (Natural History). The Association had been fortunate in the many gemmological enthusiasts who had contributed to the success of the Association over the years.

Mr. Harper recalled that Mr. Robert Shipley, who had founded the Gemmological Institute of America in 1931, had studied the N.A.G. gemmological course while he was in Europe, and it could be that some of that knowledge had formed the foundations for that excellent organization.

Mr. Alan Henn, Chairman of the National Association of Goldsmiths of Great Britain and Ireland, was invited to present the awards. After the presentation, Mr. Henn recalled that in July, 1908, the N.A.G. had proposed the establishment of gemmological courses and examinations. The N.A.G. was proud that its daughter organization had done so well and acquired a respected international reputation. The two organizations were closely associated. The G.A. accepted members from every walk of life and from all parts of the world. The N.A.G. restricted its membership to retail jewellers and its educational work was limited to this field. He hoped that the G.A. would always remember that its success stemmed from the jewellery industry and that it would continue to assist with training those connected with it. Students were expected to absorb more knowledge than those who studied many years ago. A high standard was set in the examinations and he congratulated those who had done well. He also congratulated those who had tried but had not been successful.

In addition to the diplomas, certificates for the Association's gem diamond examination were presented. There was a special welcome, too, for Wing Commander and Mrs. Warren Tassell, who were presented with diplomas of the Australian Gemmological Association. Both diplomas were awarded with distinction and in addition Wing Commander Tassell had won the premier Australian award. The Australian Gemmological Association sent greetings to the British Association.

Mr. Philip Riley, Vice-Chairman of the Association, in thanking Mr. Henn, said that there was no one who could have been more welcome amongst them. They had enjoyed the closest support from the N.A.G. over many years and they were indebted to that association for its foresight in establishing gemmology in Britain sixty years ago. It was particularly gratifying that the title Fellow of the Gemmological Association was coveted all over the world. He thanked the Goldsmiths' Company for again lending their splendid Livery Hall for the presentation.

GIFTS TO THE ASSOCIATION

The Council is grateful for the following gifts:

Mr. E. A. Jobbins has kindly presented the Association with an unusual mottled jade cabochon from Burma.

Mrs. B. F. Ames of California has kindly donated two pieces of spectrolite for the Association's collection.

A green actinolite of 1.04 cts from Mr. Max Davis.

SCOTTISH BRANCH

On 3rd December, 1968, Dr. Colin Gribble, of the Department of Geology, Glasgow University, gave a graphic account of his work for prospecting for diamonds in Tanganyika. In 1958, after De Beers had purchased the Williamson Mine, Dr. Gribble was employed with thirty geologists to survey the whole country in two years. Many kimberlite pipes were found but only one is sufficiently rich in diamonds to justify the tremendous costs of modern mining.

194

Dr. Gribble then discussed the Williamson Mine itself, describing all the processes used to extract diamonds from the pipe and gave a brief survey of its future. The quality of questions which followed must be partially due to the high standards of the advanced class on diamonds.

Mr. McWilliam, the Chairman, gave the vote of thanks and presented the diplomas gained by two Scottish students in the recent Gemmological Examinations to Mr. G. M. Turner (distinction) and Mr. D. A. Sadler, so concluding a most enjoyable evening.

GENERAL NOTICES

Course instructor

The Association requires an assistant correspondence course instructor to undertake part-time assessment of papers during the period October to May. Only persons resident in the United Kingdom should apply for further details.

Association tie

The tie referred to in the October, 1968, issue of the *Journal* is restricted to Fellows of the Association. A design for a silver-gilt brooch, which ladies who are Fellows could wear, is in the course of preparation.

iii

v

PLEASE NOTE

A large valuable collection of some eleven hundred cut stones
(other than diamond) and including several hundred cabochons
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stones range from agate to zircon, and from anglesite to
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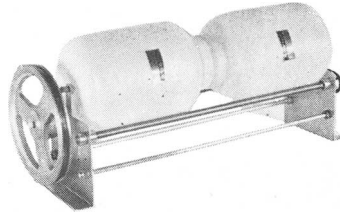
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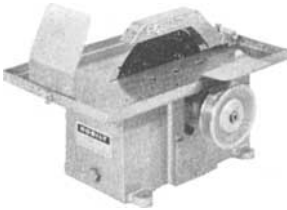
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