

*Crystal Growth
from
High-Temperature
Solutions*

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Online-Edition of the original book
with additional Chapter II and Appendices A and B



Academic Press London New York San Francisco

A Subsidiary of Harcourt Brace Jovanovich, Publishers

Part 005

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2. History of Crystal Growth from Solutions

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2.1. Development of Ideas on Crystals and Crystallization

Gems and crystals have always attracted mankind, and the belief in the virtues of gems and some minerals dates back at least two thousand years. The use of gems for jewellery originates from even earlier times, but in the early stages the colour and transparency were the main attractive factors. Beads were clearly considered of value by Bronze-age chieftains and un-faceted gems of many colours were used in the crowns of emperors and kings. The faceting of gems began only in the 15th century and cutting, cleaving and polishing techniques were subsequently developed in order to enhance the "fire" of gems, that is the inherent optical effects which arise from their high refractive index and dispersion.

Crystals as such were not recognized until comparatively recently although their forms were frequently admired and described. Accordingly, there was no general word for crystals, only specific names for gems and other minerals and for certain salts. According to Marx (1825), Homer (8th century B.C.) in his works "Ilias" and "Odysseus" was the first to use the word "crystallos" in the sense of "ice", and only since the time of Plato (427-347 B.C.) was "crystallos" also used for "rock crystal". The transparency of quartz crystals led Diodorus Siculus (around 30 B.C.) to the idea that they are formed from pure water which solidifies through the power of a godlike fire. Seneca (about A.D. 0-65) and Pliny (A.D. 23-79) believed that rock crystals were formed by "condensation" of water during a period of cold lasting up to several centuries. This belief survived seventeen centuries and was mentioned in an encyclopedia by Gleditsch in 1741. Boyle (1672) and Hottinger (1698) were among the first to prove that rock crystals were not formed from ice.

"Crystal" was for several hundred years identified with quartz = rock crystal (*crystallus montius*), but the dissertations of Hottinger (1698) on

"Krystallogia" and of Capeller (1723) on "Prodromus crystallographia" initiated the generalization of the word crystal. Bartholinus (1669) had previously used "crystal" for other minerals (*crystallus islandicus* = calcite) and Hagendorn in 1671 had used the term for organic crystals like "crystalli benzoës". "Quartz" (first mentioned by Agricola, 1530) became the scientific name for rock crystal, see Tomkeieff (1942), but it took about 200 years before the general term "crystal" gained wide acceptance, and several authors of the 17th and 18th centuries still used expressions like "corpora angula" and "figured stones", while "icicles" was used to describe needle-like crystals.

"Crystallisatio" was used for crystallization from the 17th century, whereas in earlier times expressions like condensation or coagulation were used for the process of crystal formation. It was difficult for the scientists of that time to understand how hard crystals could be formed from a clean and "soft" liquid. The concept of solutions was also not developed although Anselmus Boetius de Boodt (1609) distinguished between the evaporation and slow cooling techniques for crystallization from solution.

According to Schoen and colleagues (1956) mankind has obtained salt from natural deposits as well as by artificial preparation since the earliest recorded history, and Caldwell (1935) reports that artificial evaporation and crystallization of salt is shown on a Chinese print of 2700 B.C. Crystallization of salt was also described on the Egyptian "Papyrus Ebers" of about 1500 B.C. and by Aristoteles (384–322 B.C.). Other early crystallization processes are the preparation around 300 B.C. of sugar from sugarcane syrup in India mentioned by Ray (1956) and the crystallization of cupric sulphate (blue vitriol) and of a few other salts described by Pliny in his work "Naturalis Historia". Similar crystallizations were also reported by Arabian alchemists of the 9th to the 11th century. Birringuccio (1540) described in detail the preparation and crystallization of saltpeter, and Agricola (1546) described the crystallization of salt and stressed the importance of this material for mankind. The knowledge of his time was set down by Libavius in his textbook of alchemy (1597).

The alchemist Geber (whose works according to Darmstaedter (1922) were written in the 12th or 13th century, possibly in South Italy or Spain and who is frequently confused with the Arabian alchemist Dschabir Ibn Haijan of the 9th century) mentioned the purification of several salts by recrystallization, and also described sublimation and distillation. Geber believed in the transformation or "ennobling" of metals and since he applied many high-temperature processes it is probable that he occasionally grew crystals from high-temperature solutions. In his work "Summa Perfectionis Magisterii" he discussed the various opinions of that time on alchemical processes and concluded that special conditions such as a

"medium heat" are required to prepare (crystallize) metals and that the quality of the products depends also on the arrangement of the stars. The belief in the influence of the stars goes back to the Babylonians and was also mentioned by Plato. Furthermore, Geber argues that it must be easier to prepare the minerals and metals despite their dense structure than to imitate animals like oxen and goats because the latter have souls and somehow a higher degree of perfection!

This argument shows clearly that there was a very vague conception of the structure of crystals and thus of the crystallization process until the 17th century. Although Democritus (about 470–400 B.C.) had already expressed his ideas on indivisible particles of matter, the atoms, it was not until the year 1611 that Kepler concluded from the regular hexagonal form of snow crystals (*De nive sexangula*) that they are built up by a regular arrangement of small spherical particles. Thus Kepler was probably the first to describe the principle of order in crystals, caused by attractive forces or by pressure from outside. His ideas were extended by Robert Hooke who wrote in his work "*Micrographia*" in the year 1665:

I think, had I time and opportunity, I could make probable, that all these regular Figures . . . arise only from three or four several positions or postures of *globular* particles, and those the most plain, obvious, and necessary conjunctions of such figur'd particles that are possible. . . . And this I have *ad oculum* demonstrated with a company of bullets, and some few other very simple bodies; so that there was not any regular Figure, which I have hitherto met withall, . . . that I could not with the composition of bullets or globules, and one or two other bodies, imitate, even almost by shaking them together.

Huygens in 1690 postulated flattened spheroid particles of calcite (Niggli, 1946) and Newton (1704) believed that crystals consist of small indivisible particles of "several sizes and figures" (Burke, 1966) which might even be irregular.

In contrast to this regular packing of spheres or similar molecular forms, another point of view on crystalline arrangement was that of Bartholinus (1669), Bergman (1773) and Haüy (1784), who postulated that crystals are built of small geometrical units, cubes in the case of salt and rhombohedra in the case of calcite. This geometrical view of the structure originated not only from corresponding forms of certain as-grown crystals but also from the cleavage characteristics of many crystals. According to Smith (1960), Grignon in 1775 was the first to recognize that metals consist of small crystallites and his dendrite drawing is often reproduced.

The rule of the constancy of angles between crystal faces introduced by Steno (1669) and Bartholinus (1669) became fundamental for the correct recognition of crystal habit (as discussed further in Chapter 5) and thus for the development of the crystallographic and mineralogical sciences,

although it was Guglielmini (1688) who exactly defined the law of constant angles and the relation of the habit to the chemical species. The latter topic was amplified by Cappeller (1723), Leeuwenhoek (1685, 1705) and by Romé de l'Isle (1783). Linnaeus (1768) and especially Werner (1774) contributed much to the classification of minerals by careful observation and description of their external characteristics, not only of their forms but also of their other properties such as colour, manner of cohesion, coldness to the touch, weight, smell and taste, and according to Werner blowpipe tests could be useful for qualitative chemical analysis.

Corresponding to the vague opinions on crystals it is no wonder that a variety of strange ideas on the mechanism of crystallization was held by alchemists (Adams, 1954). The vegetative growth of crystals was especially popular among many chemists and was believed by Paracelse (1493–1541) and still in the 18th century by the French botanist Tournefort in 1702 and by Robinet in 1761. The latter deduced his hypothesis from the concept that nature is uniform in the treatment of all her productions, and consequently there had to be a uniform process for the generation of animal, vegetable and mineral species (see Burke, 1966). Thus it was a major step for Steno (1669) and for Hottinger (1698) to recognize that a crystal does not grow from the interior like a plant or animal (intussusception) but grows by deposition of material on the external faces (juxtaposition). Another question, in connection with the formation of minerals, was whether or not water played a role, and this quarrel between the *plutonists* and the *neptunists* was not settled until the end of the last century.

In the early stages of chemistry, mineralogy and crystallography these sciences were descriptive, and as late as 1822 Mohs expressed the idea that "natural history observes natural products as they are, not how they were formed" (after Michel, 1926).

On the other hand Boyle (1672) believed that gems and minerals were formed from liquids impregnated with various substances by processes analogous to those occurring in his experiments on crystal growth from aqueous solution. Boyle appears to have been the first scientist to apply the techniques of experimental chemistry to the solution of mineralogical problems. In his work "An Essay About the Origine and Virtues of Gems" he proposed the hypothesis that many gems were once fluid bodies or are in part made up of fluid substances. In particular he claimed that gems were formed from solutions and presented the following five "proofs".

1. The transparency of gems: "it is unlikely that bodies that were never fluid should have the arrangement of their constituent parts that is requisite to transparency, which permits easy passage of light through them".

2. The external "figuration" (crystal habit) of gems, because "corpuscles of various substances will coagulate in liquids and yield crystals similar to those of gems".
3. The internal textures of gems which resemble those he observed in crystals that were once fluid (e.g. common salt and silver). The interior grain of certain gems and other minerals is visible, and muscovite and diamond, for example, are easily cleaved along the visible plates (cleavages) and along the grain.
4. The fourth "proof", based on the variety of colours in gems, is not convincing, in contrast to the next one:
5. Solid gems may contain heterogeneous matter, and Boyle describes a crystal "in the midst of which there was a drop of water", as evidence that gems grow from solutions.

It is interesting to note that Boyle did not believe in the "architectonick" explanation of crystal habits, although he observed that a solution of "stony stiriae" resulted in a "coagulated mass" when the solvent was quickly evaporated, whereas slow evaporation yielded "distinct crystals, long, transparent, and curiously shaped" (similarly Rouelle described in 1745 the crystallization of salt in truncated pyramids or squares during fast evaporation of the water, whereas slow evaporation resulted in cubes: precursors of experiments on morphological stability!).

The mineral- and gem-forming solutions had, according to Boyle, some special virtues. Although he never saw any great feats performed by those hard and costly stones (such as diamonds, rubies, sapphires), he believed in the testimonies of physicians and patients and tried to explain the medicinal virtues of gems in terms of "effluvia" or "steams", originating from earth fluids and present in the gems. To the objections of other critical scientists that gems will not "part with effluvia or portions of themselves, since they lose not of their weight" Boyle replied that mercury kept in water for a day or two would kill worms without any sensible loss of weight of the mercury. Therefore "effluvia may be of so small specific gravity, as not to make the gem at all heavier in specie than crystal itself". In Boyles' view, before gems solidify the "petrescent" substance unites with a solution, exhalation, or metallic substance, and upon crystallization the gems retain the "virtues" that were added. The medicinal powers are liberated from gems as minute corpuscular "effluvia" in rapid motion. If scientists like Boyle believed in the virtues of gems, it is not surprising that many recipes against illnesses like cholera or tuberculosis contained powdered quartz and other minerals or gems, in addition to saltpeter, red corals, prepared crab eyes and celestial "theriacs" (Wallbergen, 1760). Hottinger (1698) also shared this belief in the virtues of gems and gave

several references on this topic. Even nowadays the belief in some "higher forces" of natural gems is not uncommon (see for instance Adams, 1954), and the value of natural gems in the jewellery industry has continued to rise although synthetic gems like hydrothermal ruby or emerald are available in superior quality.

It is not the purpose here to outline in detail the history of crystallography, mineralogy and crystal growth because there are several excellent works on the former two topics, such as that of Burke (1966), Niggli (1946), Lenz (1861), Kobell (1864), Marx (1825), Sohncke (1879) and Agricola (1546).

2.2. Early Crystallizations from High-temperature Solution

The discussion in Section 2.1 of the historical development of the concepts of crystals and crystallization is included to give a background to early experiments on crystal growth from high-temperature solution. Mention must also be made of flux growth in nature which started during the solidification of the earth's crust. Crystals are constantly growing in the crust, up to 30 km or so below the surface. Water and other volatile compounds are present in the "flux", which is called magma, but they merely act as flux modifiers, not as hydrothermal systems. (For the oldest granitic rocks, which are early precambrian amphibolite facies gneisses from West Greenland, a rubidium-strontium age of 3980 ± 170 million years was reported by Black *et al.*, 1971.)

Nature has not only produced the very high quality crystals used as gems but occasionally single crystals of enormous size, which have grown mainly in pegmatites or hydrothermally in rock fissures. According to Bauer (1932), an aquamarine crystal of good quality weighing 110.5 kg was found in 1910 in a pegmatite quarry in Minas Geraes in Brazil. Topaz crystals up to 135 kg were also found in Brazil, and the largest high-quality topaz "Braganza" of 1680 carats was believed to be diamond and used in the Portuguese crown. Other large gemstones are a star sapphire of 563 carats (Star of India), a ruby of 100 carat (De Longstar ruby), an emerald made into a container for ointments weighing 2680 carats (now in Vienna), and, according to Kunz (1890), an almandine garnet crystal of 4.4 kg was found in 1885 in 35th Street, near Broadway in New York!

Artificial crystallization from high-temperature solutions probably dates from the time when man could produce high temperatures, by the use of fire. Incidental flux growth probably occurred during the preparation of metals and alloys, and of ceramics and glasses, since these crafts were practised by several ancient civilizations. Crystalline products were probably grown from high-temperature solutions during the middle ages when alchemists attempted the synthesis of the "elixir of life" and the

"philosopher's stone" and the "transmutation" of "base" metals into gold, and although little or no attention was given at that time to the resulting crystals.

The alchemists tried to keep for themselves their secrets on "universal processes" by using special symbols as are shown in Fig. 2.1. The process described was discovered by Magister Heinrich Eschenreuter in a hiding place in the monastery Schwartzbach on May 6, 1403, and was hidden again for religious reasons in a hole in the wall of the monastery of St. Marienzell in Thuringia on October 10, 1489. There it was rediscovered by the Benedictine monk Basilius Valentini in the year 1762. This monk, who edited several books on alchemy and the philosopher's stone, published the recipe given in Fig. 2.1 in 1769, during the time of enlightenment, when the Church began to tolerate science. The English translation runs approximately as follows.

Take in the name of God Christ Jesus the son of Mary "minera mercurii" and prepare from this a "blood-red extract" with "spiritus vini", "spiritus nitri" and "spiritus salis", and from this "blood-red extract" make a "vinegar"† with "spiritus salis ammoniaci" and "spiritus tartari" with a strong fire. This "vinegar" add again to the residue "caput mortuum" and treat it until it is transformed into a "deep-red oil or sulphur of the materia philosophorum": Take this "deep-red oil" and put into a container and seal this with Lutum (loam), and let the Lutum dry completely, and when dry put it into a "melt furnace" and apply "moderate fire", primus gradus ignis (of the first grade of heat), and let it stand for forty days that it does not "melt". After these forty days let it stand in "melting" again for forty days so that it will become and look quite black by effervescing, and let it stand in the "melting" until it becomes white, as crystals which look milky, and when it has stood as long as before then you will see that it looks like a glass, and will appear quite dark red transparent, and keep it so long in the "liquid as a water" until there is no more change, then you have prepared the philosophers' tinctura.

More attention was paid to synthetic crystals only from the late 18th century on, when their character and their importance were recognized and when chemical analyses could be performed. Realistic speculations regarding the formation of natural crystals could only then be expressed. Although already at the time of Agricola, in the 16th century, over 100 different salts had been synthesized and crystallized from aqueous solutions, it was not until the early 19th century that the first crystals were deliberately grown from high-temperature solutions.

Around 1800 the crystallization behaviour of lavas from the region of Edinburgh and from Etna and Vesuvius was studied by Hall (1798) and by Watt (1804) who used melts up to 330 kg. Watt found crystals up to 1 mm size on slow cooling (during eight days) and obtained glasses when

† probably "essence" is meant.

Processus Universalis.

Nimm im Namen Gottes Christi Jesu
 Marien Sohn ☿ und mache daraus einen ☿
 durch ♀ ☉ ☽ aus demselben ☿ ma-
 che einen ♀ durch ☿ ☉ und ☿ mit
 starcken Feuer. Denselbigen ♀ thue wieder auf
 das Hinterbliebene ☿ und treibe es so lange bis
 sich das ☿ und ♀ in ein hoch rothes ☿
 verkehret hat: Dasselbige hoch rothe ☿ nimm,
 und thue es in ein Figir-Gesäß und verschliesse es mit
 Luto, und laß das Lutum ganz trocken wer-
 den und wenn es trocken worden ist, so setze es in
 einen ☐ und gieb ☐ ☐ ☐ und laß es
 vierzig Tage stehen, daß es nicht ☐ Wann
 vierzig Tage vorben, so laß es im ☐ stehen
 wieder vierzig Tage, so wird es durch das Gähren
 ganz schwarz werden und aussehen, und laß es
 in dem ☐ stehen bis es weiß wird, wie Cry-
 stallen, die da Milch-färbig aussehen, und wann
 es so lange gestanden als zuvor, so wirst du sehen
 daß es wie ein Glas aussehen wird, und wird ganz
 dunkel durchsichtig roth erscheinen, und halts so
 lang im ☐ bis sichs nicht mehr verändert, so
 hast du der Weißen Tinctur fertig.

FIG. 2.1. Alchemical recipe for preparation of a universal medicine, discovered by Eschenreuter in 1403 and published by Valentini (1769).

the melts were quenched. The crystals were magnetic and had relative densities ranging from 2.743 to 2.949—a good characterization for that time. Among the first syntheses from high-temperature solutions, Wöhler (1823) reported the preparation of tungsten bronze crystals from sodium tungstate flux, and other mineral syntheses (although not by typical flux techniques) were reported by Berthier (1823) and by Mitscherlich (1823). According to Haüy in 1822 apatite was prepared by Saussure from gypsum and phosphoric acid.

The synthesis by Gaudin (1837) of ruby rhombohedra of up to 0.187 g weight by melting potassium alum with potassium chromate attracted many mineralogists and led Böttger (1839) and Elsner (1839) to repeat the experiment. From this time the activity and interest in mineral synthesis began to increase, especially in France, but also in Germany.

Supersaturation in high-temperature solutions was achieved in these early experiments not only by the slow-cooling and evaporation techniques,

TABLE 2.1. Early Reports of Crystals Grown by the Flux-Reaction Technique

Solvent	Reactant	Result	Reference
Na_2WO_4	H_2 (vapour)	Na_xWO_3	Wöhler (1823)
CdCl_2	H_2S (vapour)	CdS	Durocher (1851a)
$\text{K}_2\text{Cr}_2\text{O}_7$	SiO_2 (from crucible)	Cr_2O_3	Svanberg (1854)
Al	SiCl_4 (vapour)	Si	Sénarmont (1856)
AlF_3	B_2O_3 (vapour)	Al_2O_3	Déville and Caron (1858)
$\text{NaCl} + \text{NaAlO}_2$	HCl (vapour)	Al_2O_3	Debray (1861a)
$\text{AlPO}_4 + \text{CaCO}_3$, formation of $\text{Ca}_3(\text{PO}_4)_2 + \text{CaCl}_2$ during the reaction	HCl (vapour)	Al_2O_3	Debray (1861a)
MgCl_2	H_2O (vapour)	MgO	Déville (1861b)
$\text{Ba}(\text{NO}_3)_2$	NO_2 (decomposition)	BaO	Brügelmann (1877, 1878)
$\text{Ca}(\text{NO}_3)_2$	NO_2 (decomposition)	CaO	
$\text{Sr}(\text{NO}_3)_2$	NO_2 (decomposition)	SrO	
NiCl_2	H_2O (vapour)	NiO	Ferrières and Dupont, see Bourgeois (1884)
CoCl_2	H_2O (vapour)	CoO	Bourgeois (1884)
$\text{KF} + \text{ZnF}_2$	H_2O (vapour)	ZnO	Gorgeu (1887c)
Na_2SO_4 or K_2SO_4 (+ ZnSO_4)	H_2O (vapour)	ZnO	Gorgeu (1887c)
$\text{Na}_2\text{SO}_4 + \text{ZnSO}_4$	SiF_4 (vapour)	Zn_2SiO_4	Gorgeu (1887c)
$\text{NaCl} + \text{ZnCl}_2 + \text{SiO}_2$	H_2O (vapour)	Zn_2SiO_4	Gorgeu (1887c)
Na_2SO_4 (+ ZnSO_4 + FeSO_4)	SO_3 (decomposition)	ZnFe_2O_4	Gorgeu (1887d)
BaF_2 , CaF_2 or AlF_3	H_2O (through porous ceramic crucible)	Al_2O_3	Frémy and Verneuil (1888, 1890)
FeCl_2	H_2 (vapour)	Fe	Osmond and Cartaud (1900)
SnS (+ CdCl_2)	CdCl_2 (vapour)	CdS	Viard (1903)
SnS (+ ZnCl_2)	ZnCl_2 (vapour)	ZnS	Viard (1903)

but also by flux-reaction techniques (see Chapter 7). In fact, in one of the first flux-growth experiments Wöhler (1823) passed hydrogen over sodium tungstate melts and obtained crystals of tungsten bronzes. In this case the supersaturation was achieved and maintained by the reduction of Na_2WO_4 to Na_xWO_3 by hydrogen. Further examples of the flux-reaction technique are given in Table 2.1 and further discussion is superfluous except to mention that in most cases constituents of the crystal are transported via the vapour phase to the liquid from which the crystals grow as a result of some reaction. Thus these examples belong to the VLRS technique as discussed in Chapter 7.

Reference to the 19th-century literature also reveals a number of reports of the use of mineralizers. It is interesting that Aristoteles is said to have claimed that solid materials react only in the presence of a liquid. We know today that this statement is only partially true (solid-state reactions), but it was established in ancient technology that the addition of small amounts of fluxes greatly accelerates ceramic, glass-forming and metallurgical processes. The expression "agent minéralisateur" or "mineralizer" was introduced by Beaumont (1849) and by Deville, and, according to Morozewicz (1899), it had a significant influence on the development of mineral synthesis. Vapour and liquids were termed mineralizers when they had an appreciable effect on lowering the melting point, although Friedel (1880) accepted only gaseous mineralizers such as SiF_4 , HCl and HF . Other scientists defined mineralizers as catalysts for crystallization. This view is still shared by several mineralogists today, although in 1899 Morozewicz wrote that mineralizers are nothing else but solvents and that one should abandon the term "mineralizer" in chemical and physical sciences.

When growth is unstable, solvent becomes included in the crystals, and these inclusions play a major role in the identification of natural crystals (see Gübelin, 1953) as well as in the deduction of the growth history of minerals. Material can be included only during growth, either as a liquid initially (from the solution) or as solid material which is captured by the growing crystal; an exception would be the phenomenon of eutectic crystallization but this is rarely found in natural stones. According to Gübelin, the importance of inclusions as a means of investigating the genesis and growth of minerals was emphasised by Sir David Brewster in the early 19th century.

Resistance heating and the use of electric power became popular only in the 20th century, and prior to this many high-temperature experiments were conducted in glass-making or pottery furnaces. For example, Ebelmen made his many syntheses of crystals in the famous porcelain factory at Sèvres in France, Fremy collaborated with the glass factory at St. Gobain and Morozewicz made his experimental investigations on crystallization of

minerals from (synthetic) magmas in a Siemens furnace in the glass factory at Targowerk near Warsaw. A diagram of the furnace used by Morozewicz is shown in Fig. 2.2.

For large melts like the 330 kg mentioned above and the 50 kg melts of Morozewicz, large ceramic crucibles were used as a complement to the small platinum crucibles. Early experiments on flux growth were reviewed

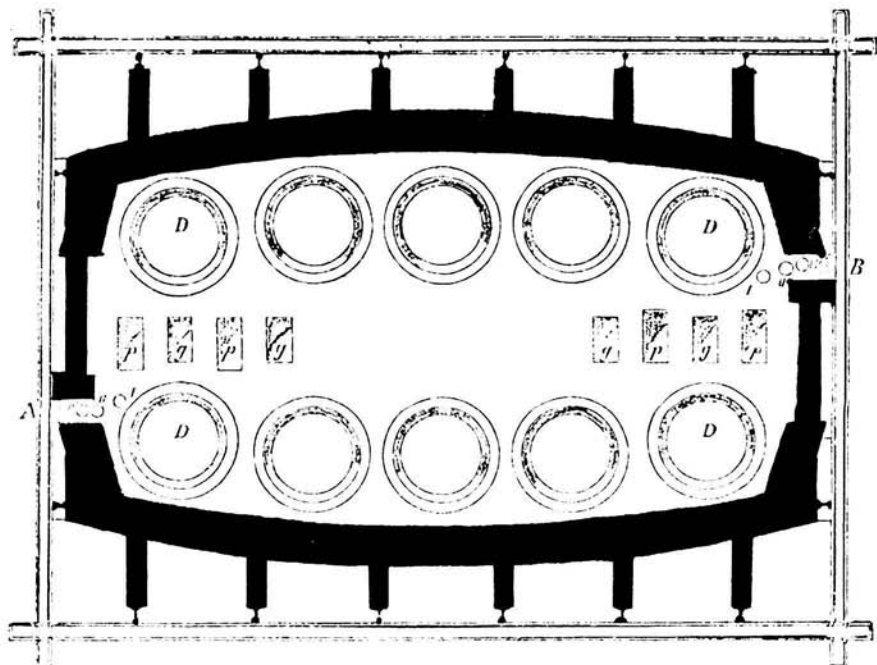


FIG. 2.2. Arrangement of the crucibles containing up to 50 kg melts in a Siemens furnace as used by Morozewicz (1899), with large ceramic crucibles at *D*, air and gas channels at *p* and *g*, and with exhaust gas channels at *A* and *B*.

by Fremy and Verneuil (1888, 1890) and in the book of Fremy (1891). These authors used crucibles up to 50 litre in size and made from porous ceramics in order to facilitate the intrusion of humid air into the fluoride solutions used for crystallization of oxides, mainly of ruby. Large graphite crucibles, of 15 cm height and more than 10 cm diameter, were also used by Hampe for the preparation of aluminium borides, and 1000 kg of coke was required to heat the furnace for six days.

Although such large crucibles were used, and Goethe had already recognized that larger crystals could be grown from larger crucibles, the mineralogists and chemists of the past century did not generally obtain

crystals larger than a few millimetres in diameter. This is due to various factors which are described later, and in particular to the use of impure chemicals, poor temperature control and programming, lack of knowledge of solubility and supersolubility curves and of the stable growth rates, and to uncontrolled evaporation of solvent. As described by Nassau and Nassau (1971) one experiment of Freymy and co-workers yielded up to 24,000 ruby crystals weighing a total of 1200 g from a 12 litre batch. It is not surprising that those mineralogists who were interested in the synthesis of large gem crystals tried other techniques.

Several attempts were made in the second half of the 19th century to crystallize ruby directly in a flame before Verneuil discovered the conditions of crucible-free flame-fusion growth which allowed reproducible growth of large crystals. The Verneuil technique became popular for growth of ruby for watch bearings and gemstones from 1904 onwards, and nowadays about 200 tons are produced per year. The success of the flame-fusion technique led to a decrease in the activity in flux growth during the following forty years and only occasionally was the flux technique used for the preparation of materials. Examples of its use are the crystallization of emerald at IG Farben in the thirties and the synthesis of mica and substitutes for quartz for military applications during World War II.

Renewed interest in crystal growth from high-temperature solutions began only after the second world war, when the importance of materials research and solid-state physics became obvious. The electronic and the computer industries could not have developed without the discovery and production of transistors for which high-quality crystals were required in large quantities. Flux growth was particularly stimulated by the fabrication of solid-state lasers and by the synthesis and crystallization of ferroelectric barium titanate (see Chapter 1) and of the rare-earth garnets. These recent developments are described elsewhere in this book.

2.3. Comments on the Table of Early References

In view of the large volume of early work on high-temperature solution growth a detailed description of the findings of the various scientists active in this field would be prohibitively long although it would be interesting from a historical viewpoint. A summary of this work is therefore given in the form of a Table. This Table 2.2 is probably incomplete and complex solid solutions or unidentified phases have in general been omitted. For example, the many silicates and solid solutions described in Doelter's extensive work on silicate melts (1904–1908) have not been included, nor have the results described by Vogt (1892–1904) and by Morozewicz (1899). In addition, amalgams which were prepared by Geber around A.D. 1200 (see Darmstaedter, 1922–1969) and studied

extensively in the past centuries, have also been omitted together with the metallic crystals which were prepared by cooling their solutions in mercury (Puschin, 1903). The numerous crystalline phases observed during the investigations of phase diagrams (see Chapter 3) have also been omitted, although mention must be made of the enormous value in crystal growth of the careful phase diagram investigations at the US National Bureau of Standards, the US Geological Survey and elsewhere, from the beginning of the 20th century on.

Some errors may be present in the Table since the contemporary techniques for crystal identification were poorly developed. Characterization relied mainly on morphology, particularly by optical measurement of the angles between faces and hence deduction by stereographic projection of the ratio of the axes and the angles between them in the elementary cell. Additional observations of the optical indicatrix of cleavage, hardness, specific gravity and colour were sometimes reported in addition to chemical analysis of the major constituents.

The data of Table 2.2 were collected from the works of Doelter (1912–1931), Groth (1906), Fouqué and Lévy (1882) and Chirvinskii (1903–1906), which are abbreviated as D (with the volume number), G, FL and C, respectively. Comparison has been made with the reviews of Favre (1856), Gurlt (1857), Fuchs (1872), Daubrée (1879), Friedel (1880), Bourgeois (1884), Meunier (1891a), Michel (1914, 1926) and Grossmann and Neuburger (1918), in addition to reference to the original literature. The Table should be useful in demonstrating the large number of crystals prepared and of solvents used, especially since the early literature is often rather inaccessible.

The first column of Table 2.2 contains the chemical formula of the material crystallized and the second the mineral name, where appropriate. Column three lists the solvent, column four the reference, and column five the source using the abbreviations given above and the volume and page numbers.

TABLE 2.2.

Formula	Name	Solvent	Reference	Source
Ag	Silver	AgI (electrolysis)	Groth (1906)	G 5
AgCl	Chlorargyrite, Cerargyrite	AgI	Lehmann (1877)	G 200
Ag ₂ S		AgCl, SbCl ₃	Doelter (1886a)	DIV/1, 247
		Bi	Rössler (1895, 1898)	G 145
Ag ₂ Se	Naumannite	Bi	Rössler (1895, 1898)	G 145
Ag ₃ AsS ₃	Proustite	S, As ₂ S ₃	Margottet (1877), see also Friedel (1880)	DIV/1, 253
Ag ₃ SbS ₃	Pyrargyrite	S	Margottet (1877)	DIV/1, 247
AlB ₂		B ₂ O ₃	Wöhler and Deville (1857)	
		B ₂ O ₃ , Na ₃ AlF ₆	Hampe (1876)	G 50
AlB ₁₂		B ₂ O ₃	Wöhler and Deville (1857)	
		B ₂ O ₃	Joly (1883)	G 50
Al ₃ B ₄₈ C ₂		B ₂ O ₃	Hampe (1876)	
AlBO ₃	Jeremejewite	CdO + B ₂ O ₃	Ebelmen (1848b, 1888) Mallard (1888a)	DIII/2, 425, DIII/2, G 101
Al ₂ BeO ₄	Chrysoberyl	see BeAl ₂ O ₄		
Al ₂ Be ₃ Si ₆ O ₁₈	Beryl, emerald	see Be ₃ Al ₂ Si ₆ O ₁₈		
Al ₂ SiO ₅	Sillimanite	K ₂ CO ₃ + AlF ₃	Meunier (1890)	
Al ₂ O ₃	Corundum	K ₂ S, K ₂ SO ₄	Gaudin (1837, 1869) Böttger (1839)	DIII/2, 446
Al ₂ O ₃ :Cr	Ruby	Na ₂ B ₄ O ₇ , BaCO ₃ or Na ₂ CO ₃	Ebelmen (1848b, 1851a, b)	DIII/2, G 101
		K ₂ SO ₃ , Ca-phosphate	Debray (1861a, b)	DIII/2, 446
		K ₂ SO ₃ , Ca-phosphate	Elsner (1839)	DIII/2, 446
		K ₂ Cr ₂ O ₇ , K ₂ MoO ₄	Parmentier (1882)	DIII/2, 446
		PbO	Fremy and Feil (1877), Fremy (1891)	DIII/2, G 102

Table 2.2 cont.

Formula	Name	Solvent	Reference	Source
		BaF ₂ , CaF ₂ , AlF ₃	Fremy and Verneuil (1888, 1890)	DIII/2, 446
		AlF ₃	Deville and Caron (1858)	DIII/2, 446
		Na ₃ AlF ₆	Meunier (1887/1888)	DIII/2, 446
		Na ₃ AlF ₆	Gmelin-Kraut II (1911) 617	DIII/2, 446
		Na ₂ B ₄ O ₇	Hönigschmidt (1907, 1910)	DIII/2, 446
		Silicate melts	Morozewicz (1895, 1899)	DIII/2, 446
		Na ₂ S(+ NaAlSiO ₄)	Hautefeuille and Perrey (1890a)	G 101
As ₂ S ₂	Realgar	S, NaHCO ₃	Sénarmont (1851a)	G 154
Au	Gold	Hg	Knafl (1863)	G 6
			Lang (1863)	G 6
B ₆ C		B ₂ O ₃	Joly (1883)	G 50
BaSO ₄	Barite	KCl, NaCl, BaCl ₂	Gorgeu (1887a)	DIV/2, 245
		KCl	Manross (1852)	DIV/2, 245
		MnCl ₂	Gorgeu (1885)	DIV/2, 196
BaSeO ₄		NaNO ₃ + KNO ₃	Diacon (1888)	C 90
BaO		Ba(NO ₃) ₂	Brügelmann (1877, 1878)	G 74
Be		BeCl ₂	Nilson and Petterson (1883)	G 8
		BeCl ₂	Brögger and Flink (1884)	G 8
		KBeF ₃ (electrolysis)	Lebeau (1899)	G 8
BeAl ₂ O ₄	Chrysoberyl	Ca ₂ B ₄ O ₇ , B ₂ O ₃	Ebelmen (1845, 1848a, 1851b, d)	DIII/2, 513; Gurlt (1857)
		BeF ₂ , AlF ₃ , B ₂ O ₃	Deville and Caron (1858)	DIII/2, 513
		Na ₃ AlF ₆	Lacroix (1887)	DIII/2, 513
		Na ₂ S, K ₂ S	Hautefeuille and Perrey (1888a, 1890a)	DIII/2, 513
Be ₃ Al ₂ Si ₆ O ₁₈	Beryl, emerald	B ₂ O ₃	Ebelmen (1848b)	DII/2, 598

		$\text{Li}_2\text{Mo}_2\text{O}_7$	Hautefeuille and Perrey (1888b, 1890b)	DII/2, 598
		LiVO_3	Hautefeuille and Perrey (1888b, 1893)	DII/2, 598
BeNaPO_4 BeO	Beryllonite	$\text{Na}_2\text{SiO}_3 + \text{B}_2\text{O}_3$	Traube (1894)	DII/2, 598
	Bromellite	Na-phosphate	Ouvrard (1890)	DIII/1, 315
		$\text{K}_2\text{CO}_3 + \text{SiO}_2, \text{B}_2\text{O}_3$	Ebelmen (1848b, 1851a, 1851c, 1861)	G 69
		$\text{Na}_2\text{B}_4\text{O}_7 + \text{SiO}_2$		
Be_2SiO_4	Phenacite	$\text{Na}_2\text{S}, \text{K}_2\text{S}$	Hautefeuille and Perrey (1888a, 1890a, 1891, 1893)	G 69
		Silicate melts		
		LiVO_3	Hautefeuille and Perrey (1888b)	
Bi		KNO_3 (decomp.)	Quesneville (1830)	DIII/1, 811
		Hg	Puschin (1903)	G 22
Bi_2O_3	Bismite	KOH	Nordenskiöld (1860, 1861a)	G 109
Bi_2S_3	Bismuthinite	Bi	Werther (1842), Rössler (1895, 1898)	DIV/1, 65; G 160
		Bi or S	Schneider (1854)	DIV/1, 65; G 160
		K_2S	de Sénarmont (1851b)	DIV/1, 65; G 160
			Lagerhjelm (1816)	DIV/1, 65; G 160
			Heintz (1844)	DIV/1, 65; G 160
C	Carbon, graphite, (diamond?)	BiCl_3	Durocher (1851a, 1854)	Gurlt (1857)
		Fe	Deville (1856a)	G 13
		Fe	Moissan (1896, 1905)	G 12
		Olivine	Friedländer (1898, 1900)	G 12
		Silicate melts	Hasslinger (1902, 1903, 1917)	G 12

Table 2.2 cont.

Formula	Name	Solvent	Reference	Source
CaCr_2O_4	see under Cr			
CaF_2	Fluorite	CaCl_2 , KCl, NaCl	Scheerer and Drechsel (1873)	DIV/3, 203; G 206
CaI_2O_6	Lautarite	NaNO_3	de Schulten (1898a)	DIV/3, 448
CaMoO_4	Powellite	NaCl	Hjortdahl (1887)	DIV/2, 783
$\text{Ca}_2\text{Nb}_2\text{O}_6\text{F}$	Pyrochlore	$\text{CaCl}_2 + \text{NaF}$	Holmquist (1896, 1899)	DIII/1, 97
		CaF_2	Joly (1876)	DIII/1, 97
$\text{Ca}_2(\text{Ta, Fe})_2\text{O}_7$	Pyrochlore	K_2CO_3	Ebelmen (1851c)	DIII/1, 97
$\text{Ca}_5(\text{PO}_4)_3(\text{F, Cl})$	Apatite	CaCl_2 , CaF_2	Manross (1852)	DIII/1, 345
		CaF_2	Briegleb (1856)	DIII/1, 345
		NaCl	Forchhammer (1854)	DIII/1, 345
		CaCl_2 , CaF_2	Deville and Caron (1863b)	DIII/1, 345
		NaCl, KCl	Ditte (1883, 1884)	DIII/1, 345
		CaCl_2 , CaF_2	Nacken (1912)	DIII/1, 345
CaSnB_2O_6	Nordenskjöldin	$\text{CaCl}_2(?)$	Ouvrard (1906)	DIII/2, 426
CaSO_4	Anhydrite	KCl , $\text{MgSO}_4 + \text{B}_2\text{O}_3$	Manross (1852)	DIV/2, 196
		NaCl	Simmmler (1859)	DIV/2, 196
		MnCl_2	Gorgeu (1885)	DIV/2, 196
CaSeO_4		$\text{NaNO}_3 + \text{KNO}_3$	Diacon (1888)	C 90
CaTiO_3	Perovskite	Na_2CO_3 , K_2CO_3	Ebelmen (1851a, c)	DIII/1, 40
		$\text{CaCl}_2 + \text{SiO}_2$	Hautefeuille (1864a)	DIII/1, 40
		BaCl_2	Bourgeois (1886)	DIII/1, 40
CaWO_4	Scheelite	$\text{NaCl} + \text{CaCl}_2$	Manross (1852)	DIV/2, 821; FL 186
		$\text{NaCl} + \text{CaCl}_2$	Michel (1879a)	DIV/2; 821; FL 186
		$\text{CaCl}_2(?)$	Debray (1862, 1863)	DIV/2, 821; FL 186
$\text{CaMgSi}_2\text{O}_6$	Diopside	CaF_2 , CaCl_2	Berthier (1835)	
		CaCl_2	Lechartier (1868)	DII/1, 530; FL 106
		$\text{Na}_2\text{B}_4\text{O}_7$	Doelter (1890)	DII/1, 642

		B ₂ O ₃ , Na-phosphate, CaF ₂	Bauer (1899)	DII/1, 642
CaFeSi ₂ O ₆	Hedenbergite	Na ₂ B ₄ O ₇	Doelter (1890)	DII/1, 642
3Na ₃ AlSiO ₄ ·CaSO ₄	Hauynite	Na ₂ SO ₄	Lemberg (1885)	DII/2, 255
		Salts and silicates	Morozewicz (1892, 1893, 1899)	DII/2, 255
CaSiO ₃	Wollastonite	CaCl ₂	Lechartier (1868)	FL 112
			Velain (1878)	
Ca ₃ Al ₂ Si ₃ O ₁₂	Grossularite	CaCl ₂	Gorgeu (1883b)	DII/2, 891
CaTiSiO ₅	Sphene, Titanite	CaCl ₂	Hautefeuille (1865)	DII/1, 62, 65, FL 178
		CaSO ₄	Michel (1892)	DII/1, 62, 65, FL 178
CaAl ₂ Si ₂ O ₈	Anorthite	NaF, KF, CaF ₂ + diorite	Velain (1878), Bauer (1899)	DII/2, 996
		Na ₂ HPO ₄ + B ₂ O ₃ + SnCl ₂	Medanich (1903)	DII/2, 996
(CaAl ₂ Si ₂ O ₈ , NaAlSi ₃ O ₈ s.s.)	Oligoclase	CaF ₂ + NaCl	Petrasch (1903)	DII/3, 226, FL 140
		CaF ₂ + MgF ₂	Medanich (1903)	DII/3, 226, FL 140
		LiCl + Na ₂ MoO ₄		
(CaAl ₂ Si ₂ O ₈ , NaAlSi ₃ O ₈ s.s.)	Labradorite	AlF ₃	Meunier (1890)	DII/3, 284
		Silicates	Petrasch (1903)	DII/3, 284
		Augite	Fouqué and Lévy (1878)	DII/3, FL 61, 139
(Ca, Na) ₂ (Al, Mg)	Melilite	Na ₂ CO ₃	Bourgeois (1883)	DII/2, 963
[(Si, Al) ₂ O ₇]		NaF + CaF ₂ + LiF	Bauer (1899)	DII/2, 963
		MgCl ₂ + CaCl ₂ + NH ₄ Cl		
		Na ₃ PO ₄ + NaVO ₃ + LiCl	Medanich (1903)	DII/2, 963
CaO		Ca(NO ₃) ₂	Brügelmann (1877, 1878)	G 70
CdO	Monteponite	Na ₂ B ₄ O ₇	Florence (1898)	DIII/2, 305
CdS	Greenockite	CaCl ₂ , CaF ₂ , BaS	Déville and Troost (1861)	DIV/1, 347; FL 303, G 150
			Dupont and Ferrières (1882)	FL 304
		K ₂ CO ₃ + S	Schüler (1853)	DIV/1, 347; FL 304

Table 2.2 cont.

Formula	Name	Solvent	Reference	Source
		CdCl ₂	Durocher (1851a)	DIV/1, 347
		Metal halides	Viard (1903)	DIV/1, 348
CdSe	Cadmoselite	CdCl ₂	Fouzes-Diakon (1900)	G 150
CePO ₄	Monazite	CeCl ₃	Radominsky (1875)	DIII/1, 558
CeO ₂		Na ₂ B ₄ O ₇	Nordenskiöld (1860, 1861a)	G 85
		NaCl, Na ₂ B ₄ O ₇	Sterba (1901)	G 85
CoS		BaS + NaCl	Hjortdahl (1867)	DIV/1, 680; G 138
Co ₂ O ₄		CoCl ₂	Schwarzenberg, see Gorgeu and Bertrand (1887)	C 80
Co ₂ SiO ₄		CoCl ₂	Bourgeois (1889)	C 88
CoO		B ₂ O ₃ , Na ₂ B ₄ O ₇	Ebelmen (1851b, c)	G 71
		CoCl ₂ (+ H ₂ O)	Ferrières and Dupont, after Bourgeois (1884)	G 71
		Co ₃ (PO ₄) ₂ + K ₂ SO ₄	Grandeau (1886)	
Cr ₂ O ₃	Eskolaite	Ca-borate	Ebelmen (1847, 1851)	DIV/2, 721; G 103
		K ₂ Cr ₂ O ₇ + SiO ₂	Svanberg (1854)	DIV/2, 721; G 103
CaCr ₂ O ₄		K ₂ CrO ₄	Weyberg (1906)	DIV/2, 705
Li ₂ Cr ₂ O ₄ (?)		Li ₂ CrO ₄	Weyberg (1906)	DIV/2, 705
FeCr ₂ O ₄	Chromite	Na ₃ AlF ₆	Meunier (1887a, 1888)	DIV/2, 705
		B ₂ O ₃	Ebelmen (1851b, d)	DIV/2, 705
MgCr ₂ O ₄		B ₂ O ₃	Ebelmen (1851b, d)	DIV/2, 705
MnCr ₂ O ₄		B ₂ O ₃	Ebelmen (1851b, d)	DIV/2, 705
			Meunier (1887a, 1888)	DIV/2, 705
ZnCr ₂ O ₄		B ₂ O ₃	Ebelmen (1851b)	DIV/2, 705
			Meunier (1887a, 1888)	DIV/2, 705
NaCrS ₂		Na ₂ CO ₃ , Na ₂ CO ₃ -K ₂ CO ₃	Schneider (1873b, 1897)	
FeCr ₂ S ₄	Daubreelite	FeCl ₂ , S	Meunier (1891b)	DIV/2, 656
Cu	Copper		Behrens (1894)	G 3

		Silicates	Vogt (1892)	G 3
		Silicates	Washington (1894)	G 3
Cu_4FeS_4	Bornite	NaCl	Böcking (1855)	DIV/1, 161
		$\text{Na}_2\text{B}_4\text{O}_7$	Marigny (1864)	DIV/1, 161
Cu_2S	Chalcocite	Pb	Rössler (1895, 1898)	G 143
CuS	Covellite	$\text{ZnSO}_4(?)$	Rogers (1911, 1912)	DIV/1, 98
Cu_2Se	Berzelianite	CuCl	Fouzes-Diakon (1900)	G 145
CuWO_4	Cuprotungstate	NaCl	Michel (1879b)	FL 186
CuZn_2		Zn	Charpy (1896)	G 43
Fe		FeCl_2	Haidinger (1850)	G 41
	Iron	FeCl_2	Osmond and Cartaud (1900)	G 41
		Hg	Nerad (1932)	
FeAs_2	Arsenoferrite	$\text{Na}_2\text{B}_4\text{O}_7$	Berthier (1836, 1837)	DIV/1, 604
$\text{Fe}_3\text{As}_2(? \text{FeAs})$		$\text{Na}_2\text{B}_4\text{O}_7$	Descamp (1878)	DIV/1, 604
FeAsS	Arsenomarcasite	$\text{Na}_2\text{S}, \text{Na}_2\text{S} + \text{Na}_2\text{CO}_3$	de Sénarmont (1851a, b)	DIV/1, 634
FeCr_2O_4	see under Cr			
FeCr_2S_4	see under Cr			
Fe_2O_3	Hematite	$\text{Na}_2\text{B}_4\text{O}_7$	Hauer (1854)	DIII/2, 635
		$\text{Na}_2\text{B}_4\text{O}_7$	Rose (1867a, 1869a)	DIII/2, 635
		$\text{Na}_2\text{B}_4\text{O}_7$	Florence (1898)	DIII/2, 635
Fe_2O_4	Magnetite	Silicate melts	Ebelmen (1851a)	DIII/2, 649
		FeCl_2	Durocher (1851b)	FL 240
		$\text{FeF}_2 + \text{B}_2\text{O}_3$	Deville and Caron (1858)	DIII/2, 649
		$\text{Na}_2\text{B}_4\text{O}_7 + \text{PbO}$	Florence (1898)	DIII/2, 661
Fe_2P		$\text{Na}_2\text{B}_4\text{O}_7$	Hooslef (1856)	DIII/2, 823
$(\text{Fe}, \text{Ni})_3\text{P}$	Schreibersite	Na-phosphate	Faye (1863)	DIII/2, 823
FeS	Pyrrhotite	Sb_2S_3	Morel (1888)	G 146-147
		FeCl_2	Doelter (1886b)	G 146-147, T 101

Table 2.2 cont.

Formula	Name	Solvent	Reference	Source
FeS ₂	Pyrite	K ₂ S _x	Déville, see Friedel (1880)	
Fe ₂ SiO ₄	Fayalite	FeCl ₂	Gorgeu (1883a, 1887b)	DII/1, 719
(Fe, Mg) ₂ SiO ₄	see Mg			
NaFeSi ₂ O ₆	see Na			
FeWO ₄	Ferberite	NaCl	Geuther and Forsberg (1861)	DIV/2, 848
			Michel (1879a)	DIV/2, 848
(Fe, Mn)WO ₄	Wolframite	NaCl	Michel (1879a)	DIV/2, 848
(Fe, Mn)Nb ₂ O ₆	Columbite	FeF ₂ , MnF ₂	Joly (1876)	FL 182
FeAl ₃		Al	Guillet (1902)	G 45
HgS	Cinnabar	HgCl ₂	Durocher (unpublished)	DIV/1, 361
CsHgCl ₃		CsCl	Wells (1892)	G 367
Ir	Iridium	FeS ₂ + Na ₂ B ₄ O ₇	Debray (1882)	G 38
		Ag	Rössler (1900)	G 38
K ₂ Pb(SO ₄) ₂	see Pb			
KAlSiO ₄	Kaliophilite?	KCl	Gorgeu (1887a)	DII/2, 410
	Potash nepheline?			
		KCl, K ₂ SiO ₄	Weyberg (1908)	DII/2, 410
		KCl, K ₂ SO ₄	Ginsberg (1912)	DII/2, 410
		KF	Duboin (1892a)	E 16
(Na, K)AlSiO ₄	Nepheline	Na-vanadate	Hautefeuille (1880a)	DII/2, 220; FL 155;
		Na ₂ CO ₃		E 20, FL 156
KAlSi ₂ O ₆	Leucite	K-vanadate	Hautefeuille (1880b, c, 1888)	DII/2, 470; E 9
		KVO ₃	Hautefeuille and Perrey (1888c)	DII/2, 470, E 9-11
		KF	Duboin (1892a, b)	DII/2, 470
		NaF	Doelter (1897)	DII/2, 470
K-Zn-Silicate		KF, KCl	Duboin (1905)	DII/1, 790

KAlSi_3O_8	Orthoclase†	$\text{WO}_3, \text{Na}_3\text{PO}_4, \text{K}_3\text{PO}_4, \text{K}_2\text{SiF}_6$	Hautefeuille (1876, 1877, 1880f), see also Friedel (1880)	DII/2, 551, FL 132, 141
$\text{KAl}_3\text{Si}_3\text{O}_{10}\text{F}_2$	Fluor-muscovite	$\text{KF} + \text{K}_2\text{SiF}_6$	Doelter (1888)	DII/2, 438, 718
$\text{KMg}_3\text{AlSi}_3\text{O}_{10}\text{F}_2$	Fluor-phlogopite	$\text{KF} + \text{NaF}$	Doelter (1888)	DII/2, 438, 718
$\text{K}(\text{Mg}, \text{Fe})_3\text{AlSi}_3\text{O}_{10}\text{F}_2$	Fluor-biotite	$\text{MgF}_2 + \text{KF}$	Doelter (1888)	DII/2, 438, 718
		$\text{K}_2\text{SiF}_6 + \text{NaF} + \text{MgF}_2$	Chroustchoff (1887)	DII/2, 718
		K_2SiF_6	Hautefeuille and St. Gilles (1887)	DII/2, 718
KFeSi_3O_8	Iron-feldspar(?)	KVO_3	Hautefeuille and Perrey (1888d)	C 86
LiAlSiO_4	Eucryptite	LiVO_3	Hautefeuille and Perrey (1890a)	DII/2, 195
$\text{LiAlSi}_4\text{O}_{10}$	Petalite	LiVO_3	Hautefeuille (1880d)	FL 159, T 100
$\text{Li}_2\text{Cr}_2\text{O}_4$	see under Cr			
MgF_2	Sellaite	KCl, NaCl	Röder (1863), Cossa (1876, 1877)	DIV/3, 188; G 205
MgO	Periclase	$\text{Ca-borate}, \text{B}_2\text{O}_3$	Ebelmen (1851b, c)	DIII/2, 287; G 69
		MgCl_2	Daubrée (1854), Dumas (1859)	DIII/2, 287
		$\text{MgCl}_2 (+ \text{H}_2\text{O})$	Déville (1861b)	G 69
		$\text{MgSO}_4, \text{K}_2\text{SO}_4$	Debray (1861c)	DIII/2, 287; 689
		$\text{MgCl}_2 (+ \text{SiO}_2 + \text{ZrO}_2)$	Hjortdahl (1866)	G 70
		KOH	de Schulten (1898b)	DIII/2, 287
MgAl_2O_4	Spinel	B_2O_3	Ebelmen (1848b)	DIII/2, 522
		$\text{AlCl}_3 + \text{Na}_3\text{AlF}_6$	Meunier (1887b)	
		Silicates	Morozewicz (1895, 1899)	

† The potassium feldspar crystallizing at high temperatures should be called *sanidine* according to the nomenclature of Laves (1960).

Table 2.2 cont.

Formula	Name	Solvent	Reference	Source
MgCr ₂ O ₄	Magnesiachromite	B ₂ O ₃	Ebelmen (1851b, d)	DIV/2, 705; Gurlt (1857)
MgTiO ₃	Geikielite	MgCl ₂	Ebelmen (1863)	DIII/1, 42
		MgCl ₂	Hautefeuille (1865)	DIII/1, 42; FL 178
		MgCl ₂	Bourgeois (1892)	DIII/1, 42
Mg ₂ PO ₄ F	Wagnerite	MgCl ₂	Deville and Caron (1863a)	DIII/1, 319; FL 107
MgSiO ₃	Enstatite	B ₂ O ₃ , KOH	Ebelmen (1851b, 1855a)	DIII/1, 319; DII/1, 329
		CaCl ₂ , MgCl ₂	Hautefeuille (1865)	DIII/1, 319; FL 98
		MgCl ₂	Lechartier (1868)	DIII/1, 319
Mg ₂ Al ₄ Si ₅ O ₁₈	Cordierite	MgF ₂ + WO ₃	Doelter (unpublished)	DII/2, 626
CaMgSi ₂ O ₄	see under Ca			
(Mg, Fe) ₂ SiO ₄	Olivine	Silicates	Berthier (1823), Reiter (1906)	FL 102, 103 DII/1, 308
Mg ₃ B ₇ O ₁₃ Cl	Boracite	NaCl, MgCl ₂ , B ₂ O ₃	Heintz and Richter (1860)	see Friedel (1880)
MnCr ₂ O ₄		B ₂ O ₃	Ebelmen (1851b, d)	DIV/2, 705; Gurlt (1857)
Mn ₃ O ₄	Hausmannite	Na ₂ B ₄ O ₇	Nordenskiöld (1861a)	DIII/2, 893
		CaCl ₂	Kuhlmann (1861)	DIII/2, 893
		Silicates	Ebell (1876)	DIII/2, 893
(Mn, Fe)WO ₄	Wolframite	NaCl	Michel (1879a)	DIV/2, 851
		NaCl	Geuther and Forsberg (1861)	DIV/2, 851
MnWO ₄	Hübnerite	NaCl	Geuther and Forsberg (1861)	DIV/2, 847
MnSiO ₃	Rhodonite	MnCl ₂	Bourgeois (1885)	DII/1, 730
			Gorgeu (1883a, 1887b)	DII/1, 730
			Doelter and Hussak (1884)	DII/3, 362
Mn ₂ SiO ₄	Tephroite	MnCl ₂	Doelter and Hussak (1884)	DII/1, 712
Mn ₃ Al ₂ Si ₃ O ₁₂	Spessartite	MnCl ₂	Gorgeu (1883b)	DII/3, 362
			Doelter and Hussak (1884)	DII/3, 362

MnO		B ₂ O ₃ , Na ₂ B ₄ O ₇	Ebelmen (1851b, c)	G 70
MoS ₂	Molybdenite	Molybdates	Debray (1904)	DIV/1, 70; G 157
		K-molybdate	de Schulten (1889a, b, 1891)	DIV/1, 70
		MoO ₃	Meunier (1887b, 1890)	DIV/1, 70
		K-molybdate	Guichard (1901)	DIV/1, 70
MoO ₂		K ₂ CO ₃ + B ₂ O ₃ + MoO ₃	Mauro and Panebianco (1881)	G 93-94
		K ₂ MoO ₄	Stevanović (1903)	G 93-94
NaF	Villiaumite	Na ₂ SO ₄ —CaCO ₃	Doelter, see p. 32	DIV/2, 1426
NaBePO ₄	Beryllonite	see Be		
(Na, K)AlSiO ₄	Nepheline	see K		
NaFeSi ₂ O ₆	Acmite, Aegirite	NaCl	Weyberg (1905a, 1907)	DII/2, 339
Na ₄ Al ₃ O ₁₀ ·Na ₂ SO ₄	Nosean	Na ₂ SO ₄	Lemberg (1883)	DII/2, 253
		Na ₂ SO ₄	Morozewicz (1899)	DII/2, 253
		Na ₂ SO ₄	Weyberg (1911, 1914)	DII/2, 253
Na ₂ Al ₂ Si ₂ O ₁₂ ·NaCl	Sodalite	NaCl	Lemberg (1876)	DII/2, 239
		NaCl	Morozewicz (1899)	DII/2, 239
		NaCl	Mügge (1892)	DII/2, 239
		LiBr	Weyberg (1905b)	DII/2, 239
		(→Li ₇ Al ₇ Si ₇ O ₂₈ ·LiBr?)		
NaNbO ₃		Na ₂ B ₄ O ₇	Mallard (1887a, b, 1888a, b, 1889), Knop (1871), Nordenskiöld (1860, 1861a)	G 112
NaAlSi ₃ O ₈	Albite†	WO ₃ , Na ₂ WO ₄ , P ₂ O ₅ , V ₂ O ₅	Hautefeuille (1874), see also Friedel (1880)	DII/2, 401; FL 136
		Na ₂ WO ₄	Wallace (1909)	DII/2, 401
		Na ₂ WO ₄	Day and Allen (1905)	DII/2, 401

† The sodium feldspar crystallizing at high temperatures should be called *analbite* according to the nomenclature of Laves (1960).

Table 2.2 cont.

Formula	Name	Solvent	Reference	Source
Na_xWO_3		$\text{Na}_2\text{WO}_4(+\text{H}_2\text{O})$	Wöhler (1823)	DII/2, 401
Nb_2O_5		B_2O_3	Mallard (1887a, b, 1888a, b, 1889)	G 112
		B_2O_3	Holmquist (1896, 1899)	G 112
NiAs	Nicollite	NiCl_2	Durocher (1851b)	DIV/1, 709; FL 277
Ni_3P (Ni_3P)		Ca-phosphate	Garnier, after Jannetaz (1882)	DIII/2, 823
NiS	Millerite	NiF_2	Poulenc (1882)	DIV/1, 696
Ni_2SiO_4		NiCl_2	Bourgeois (1889)	C 88
NiO		$\text{B}_2\text{O}_3, \text{Na}_2\text{B}_4\text{O}_7$	Ebelmen (1851b, c)	G 70, Gurlt (1857)
		$\text{NiCl}_2(+\text{H}_2\text{O})$	Ferrières and Dupont, after Bourgeois (1884)	G 70
Os		$\text{Ni}_3(\text{PO}_4) + \text{K}_2\text{SO}_4$	Grandeau (1886)	G 70
P		$\text{Na}_2\text{B}_4\text{O}_7 + \text{FeS}_2$	Debray (1882)	G 38
		Pb	Hittorf (1865)	G 17, 19
		P + S	Mitscherlich (1822–1823)	G 17, 19
PbO ($\text{Pb}_3\text{O}_4, \text{PbO}_2$)	Litharge Massicotite Minium	KOH	Michel (1890)	DIII/1, 209; G 77
		$\text{NaNO}_3, \text{KNO}_3$		
		KOH	Geuther (1883), Nordenskiöld (1861a)	DIII/1, 209; G 77, G 76
	Plattnerite	KOH(?)	Becquerel (1857)	DIII/1, 211
PbS	Galena	$\text{Na}_2\text{B}_4\text{O}_7$	Marigny (1864)	DIV/1, 420, G 152
		$\text{PbO}, \text{NH}_4\text{Cl}$	Weinschenk (1890)	DIV/1, 420, G 152
		Pb	Rössler (1895, 1898)	DIV/1, 420, G 152
PbSO_4	Anglesite	CaCl_2	Manross, see Friedel (1880)	DIV/1, 420, G 152
PbSe	Clausthalite	$\text{PbCl}_2, \text{NaCl}$	Michel (1888)	DIV/1, 842
($\text{PbSeO}_4?$)		$\text{NaNO}_3, \text{KNO}_3$	Michel (1888), Diacon (1888)	DIV/1, T 90

PbSe	Clausthalite	Pb	Rössler (1895, 1898)	G 153
PbCrO ₄	Crocoite	KCl	Manross (1852)	DIV/2, 736
Pb ₅ (AsO ₄) ₃ Cl	Mimetite	PbCl ₂	Déville and Caron (1859, 1863b)	DIII/1, 708
		PbCl ₂	Lechartier (1867)	DIII/1, 708
		PbCl ₂	Michel (1887)	DIII/1, 707
Pb ₃ (PO ₄) ₃ Cl	Pyromorphite	NaCl (+ PbCl ₂)	Manross (1852, 1858)	DIII/1, 454
		NaCl (+ PbCl ₂)	Déville and Caron (1863a)	DIII/1, 454
		PbCl ₂	Michel (1887)	DIII/1, 454
Pb ₅ (VO ₄) ₃ Cl	Vanadinite	PbCl ₂	Hautefeuille (1871, 1873, 1883)	DIII/1, 837, FL 183
			Ditte (1883)	DIII/1, 837
PbK ₂ (SO ₄) ₂	Palmierite	Na ₂ SO ₄	Zambonini (1920, 1924)	DIV/2, 634
PbMoO ₄	Wulfenite	NaCl + PbCl ₂	Manross (1852)	DIV/2, 797
		NaCl + PbCl ₂	Schultze (1863)	DIV/2, 797
		NaCl	Michel (1881)	FL 193
PbMoO ₄ :		NaCl	see Doelter	DIV/2, 805
9 wt% Bi ₂ Mo ₃ O ₁₂				
PbMoO ₄ : Y ₂ Mo ₃ O ₁₂		NaCl	see Doelter	DIV/2, 805
PbWO ₄	Stolzite	NaCl + PbCl ₂	Manross (1852)	FL 191
		NaCl	Geuther and Forsberg (1861)	
Pt	Platinum	Silicates	Ebelmen (1851a)	G 41
		NaNO ₃	Köttig (1857)	G 41
PtAs ₂		Na ₂ CO ₃	Murray (1875)	DIV/1, 788
PtTe ₂		Te	Rössler (1897)	G 155
Rh		Pb, Bi	Rössler (1900)	G 38
Ru		Na ₂ B ₄ O ₇ + FeS ₂	Déville and Debray (1879)	DIV/1, 790; G 38

Table 2.2 cont.

Formula	Name	Solvent	Reference	Source
RuS ₂	Laurite	Pb	Rössler (1900)	G 38
Sb ₂ S ₃	Stibnite	Na ₂ B ₄ O ₇ + FeS ₂	Deville and Debray (1879)	DIV/1, 790; G 158
		Sb ₂ O ₃ + S + I	Jannasch and Remmler (1893)	DIV/1, 59
Se		SbCl ₃	Durocher (1851a)	Gurlt (1857)
Si		Na ₂ Se _x	Mitscherlich (1855)	G 33
		Al(+ SiCl ₄)	Deville (1856b), after Sénarmont (1856)	G 13
		?	Henry, after Miller (1858)	G 13
		?	Percy, after Miller (1866–1867)	G 13
SiO ₂	Quartz	Na ₂ Si ₂ O ₅ + K ₂ SiF ₆	Friedel (1888)	G 87
		Na ₂ WO ₄ , Li ₂ WO ₄ (+ B ₂ O ₃)	Hautefeuille (1880a)	G 87
		LiCl	Hautefeuille and Margottet (1881)	G 87
SiO ₂	Tridymite	Alkali phosphates	Hautefeuille (1880a)	G 89
SiO ₂	Tridymite	Na ₂ WO ₄ , CaCl ₂		
		LiCl	Hautefeuille and Margottet (1881)	G 87, 89
		Alkali phosphates, Silicates	Rose (1869b)	DIII/1, 192; G 89
		Na ₂ WO ₄	Hautefeuille (1870, 1878)	
		Na ₂ WO ₄ , NaVO ₃ , Na ₃ PO ₄	Hautefeuille, see Friedel (1880)	
SiO ₂	Cristobalite	Na ₂ B ₄ O ₇ (+ BeO)	Ebelmen, see Mallard (1887)	DII/1, 278, 199
SiP ₂ O ₇		HPO ₃	Hautefeuille and Margottet (1883)	G 112–113
Sn	Tin	Pb, Bi	Miller (1843)	DIII/1, 176

SnO ₂	Cassiterite	Hg	Puschin (1903)	G 14-15
		Slags	Abel (1859)	DIII/1, 184
		Slags	Bourgeois (1888)	DIII/1, 184; G 95
		Sn	Vogt (1899)	DIII/1, 184; G 95
SnS SnSe SnP ₂ O ₇	Herzenbergite	Na ₂ B ₄ O ₇	Wunder (1870b)	DIII/1, 184; G 96
		SnCl ₂	Schneider (1855)	G 150
		SnCl ₂	Schneider (1866)	G 140
		HPO ₃	Hautefeuille and Margottet (1886, 1888)	G 113
CaSnB ₂ O ₆	Nordenskjöldin	CaCl ₂ (?)	Ouvrard (1906)	DIII/2, 426
SnSb	Celestite	Sn	Behrens (1898), Stead (1897)	G 61
SrSO ₄		NaCl, SrCl ₂ , KCl	Gorgeu (1883b, 1887b)	DIV/2, 221, 245
		KCl	Manross, see Friedel (1880)	
SrSeO ₄		NaNO ₃ + KNO ₃	Diacon (1888)	C 90
SrO		Sr(NO ₃) ₂	Brügelmann (1877, 1878)	G 74
Ta ₂ O ₅		B ₂ O ₃ (+ BaCO ₃)	Mallard (1887a, b, 1888a, b, 1889)	G 112
ThO ₂	Thorianite	B ₂ O ₃	Holmquist (1896, 1899)	G 112
		B ₂ O ₃ , Na ₂ B ₄ O ₇	Hillebrand (1893, 1896)	DIII/1, 227
		Na ₂ B ₄ O ₇	Nordenskiöld and Chydenius (1860)	DIII/1, G 86
			Rammelsberg (1873)	
		Na ₂ B ₄ O ₇	Bahr (1862, 1863)	
		K ₃ PO ₄	Troost and Ouvrard (1889)	
ThP ₂ O ₇ TiO ₂	Rutile	Na ₂ CO ₃ —ThF ₄	Duboin (1908)	
		HPO ₃	Troost (1885)	G 113
		B ₂ O ₃ , phosphates, alkali silicates	Ebelmen (1851b, d)	DIII/1, 24; G 91

Table 2.2 cont.

Formula	Name	Solvent	Reference	Source
		$\text{Na}_2\text{B}_4\text{O}_7$	Rose (1867a, b, c)	DIII/1, 24; G 91
	Rutile	Phosphates	Doss (1894)	DIII/1, 24; G 91
	Anatase	Phosphates	Doss (1894)	DIII/1, 24; G 91
	Rutile	Na_2WO_4	Hautefeuille (1880e)	DIII/1, 24; G 91
	Rutile (BaTiO_3 ?)	BaCl_2	Bourgeois (1886)	DIII/1, 24; G 91
	Rutile	$\text{SnO}_2, \text{SiO}_2$	Déville and Caron (1861)	G 91
	Brookite	$\text{CaF}_2 + \text{KCl} + \text{SiF}_4 + \text{HCl}$	Hautefeuille (1864b, 1865)	DIII/1, 36, FL 171
		$\text{K}_2\text{SiF}_6 + \text{HCl}$	Hautefeuille (1864b, 1865)	DIII/1, 36, FL 171
FeTiO_3	Ilmenite	FeCl_2	Bourgeois (1892)	DIII/1, 43, 51
		$\text{Na}_2\text{B}_4\text{O}_7$	Rose (1867a)	DIII/1, 51
MgTiO_3	see Mg			
CaTiO_3	see Ca			
TiP_2O_7		HPO_3	Hautefeuille and Margottet (1886)	G 113
CaTiSiO_5	see Ca			
Ti_2O_3		TiNO_3	Thomas (1904)	G 604
UO_2	Uraninite	$\text{Na}_2\text{B}_4\text{O}_7$	Hillebrand (1893)	DIV/2, 934; G 86
$\text{Pb}_3(\text{VO}_4)_3\text{Cl}$	Vanadinite	see Pb		
WO_3		$\text{Na}_2\text{B}_4\text{O}_7$	Nordenskiöld (1861b, 1867)	DIV/2, 811; G 110
		$\text{Na}_2\text{WO}_4 + \text{Na}_2\text{CO}_3 + \text{HCl}$		
$\text{Y}_2\text{Mo}_3\text{O}_{12}$		$\text{Na}_2\text{MoO}_4 + \text{PbCl}_2 +$	Doelter	DIV/2, 805
		$\text{Bi}_2(\text{MoO}_4)_3$		
YPO_4	Xenotime	YCl_3	Radominsky (1875)	
ZnO	Zincite	$\text{Na}_2\text{B}_4\text{O}_7$	Florence (1898)	DIII/2, 299
K-Zn-Silicate	see K			
ZnS	Sphalerite,	ZnCl_2	Durocher (1851a)	Gurlt (1857)
	Wurtzite	$\text{CaF}_2 + \text{BaSO}_4$	Déville and Troost (1861, 1865)	DIV/1, 341, G 148

		$K_2CO_3 + S$	Schneider (1873a)	G 148
		$SnCl_2$	Viard (1903)	DIV/1, 342
Zn_2SiO_4	Willemite	ZnF_2	Deville (1861a)	DII/1, 783
$ZnAl_2O_4$	Gahnite	B_2O_3	Ebelmen (1848b)	DIII/2, 530
		$CaCl_2 + AlCl_3 + ZnCl_2$	Daubrée (1854)	DIII/2, 530
		$B_2O_3 + AlF_3 + ZnF_2$	Deville and Caron (1858)	DIII/2, 530
$ZnCr_2O_4$	Zinc chromite	B_2O_3	Ebelmen (1851b, d)	DIV/2, 705; Gurlt (1857)
$ZnFe_2O_4$	Franklinite	$Na_2B_4O_7 + PbO$	Florence (1898)	DIII/2, 661
		B_2O_3	Ebelmen (1851b)	DIII/2, 661; Gurlt (1857)
$ZnSiO_3$		B_2O_3	Ebelmen (1855b)	DII/1, 786
		B_2O_3	Traube (1894)	DII/1, 786
$ZnSeO_4$		$NaNO_3 - KNO_3$	Diacon (1890)	C 90
$ZnSe$		$ZnCl_2$	Fouzes-Diakon (1900)	G 149
ZrO_2	Baddeleyite (and tetragonal zirconia)	$Na_2B_4O_7$	Nordenskiöld (1860, 1861a)	DIII/1, 132; G 83, 93
		$Na_2B_4O_7$	Knop (1871)	DIII/1, 132; G 83, 93
		Phosphates, $Na_2B_4O_7$	Wunder (1870a, b)	DIII/1, 132; G 83, 93
		Na_2CO_3	Lévy and Bourgeois (1882)	DIII/1, 132; G 83, 93
		$Na_2B_4O_7$, Phosphates	Florence (1898)	DIII/1, 132
		Na_2CO_3	Morozewicz (1909)	DIII/1, 132
$ZrSiO_4$	Zircon	Li_2MoO_4	Hautefeuille and Perrey (1888c)	DIII/1, 148; G 92
		Li_2MoO_4	Stevanovič (1903)	DIII/1, 148; G 92
		Na-borate	Gürtler (1907)	DIII/1, 148; G 92
ZrP_2O_7		HPO_3	Hautefeuille and Margottet (1886)	G 113

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