SOME FACTORS INFLUENCING FLUORESCENCE IN MINERALS

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Abstract

In addition to composition and the exciting wave length, several other factors may have a bearing on the presence of absence of fluorescence in minerals.

A group of sixty-three minerals, of which about one third were fluorescent at room temperature were cooled with dry ice. This treatment served to intensify the fluorescence of many of the luminescent minerals and initiated fluorescence in an additional twenty specimens. The technique of estimating the percentages of minerals in rocks by their fluorescence can be expanded by the use of low temperatures, as is shown by the occurrence of fluorescence in the microcline of certain granites when chilled.

Fluorescence is quenched by heating the mineral, and may also be quenched in certain minerals by crushing. Water added to powdered minerals may either serve to activate fluorescence, or modify an existing fluorescence.

The packing index of ionic minerals is suggested to have some relationship to the presence or absence of fluorescence in minerals. Finally, a summary of the types of luminescence and empirical rules for the occurrence of luminescence are given.

A survey of recent mineralogical and petrological literature on the occurrence of fluorescence in minerals shows that the emphasis has been on the study of two controlling factors. These are the establishment of the exciting wave length, or range of wave lengths, by which the maximum emitted light is produced (Smith and Parsons, 1938), and the activating agent or impurity which is believed to be the cause of the fluorescence (Brown, 1934). However, temperature and agents other than those just noted can control the presence or absence of fluorescence, the intensity of the emitted light, and on occasions, even modify the colour. As early as 1859, Becquerel conducted experiments on several minerals involving temperature changes, and more recently, Pringsheim (1943) has noted the effect of changes in physical conditions in the study of artificial phosphors. Unfortunately, most of the recent studies of naturally occurring fluorescent minerals suggest that the writers have either ignored or have been unfamiliar with many of the techniques developed in the study of phosphors. Two exceptions may be cited, Haburlandt and Köhler (1934) who studied scapolite fluorescence using low temperatures, and Smith (1945) who notes briefly that temperature changes may have a noticeable effect on the fluorescence of minerals. It seems necessary, therefore, to re-emphasize that agents other than composition and wave length can be important in the production of luminescence in minerals. The effect of temperature change should be noted in particular, since under conditions of moderately wide natural temperature ranges, quite different results can be obtained from the same specimens. In addition, a knowledge of this effect may frequently be of use in the somewhat limited practical application of luminescence to mineralogical and petrological studies. Other physical changes which may have an effect on the luminescence of minerals are the reduction of the minerals to a powder and the wetting of powdered minerals with water or other liquids.

The writer has performed a series of experiments which illustrate the effect of temperature, powdering and wetting on a number of minerals. Most of the experiments duplicate work on artificial phosphors, but to the writer's knowledge there has been little or no reference to anything of a similar kind in any of the more recent mineralogical publications in English. Since the experiments were only intended to be exploratory, simple equipment and techniques were used. The exciting radiation was obtained from a Mineralight which supplies ultraviolet light in the vicinity of 2500 Å, with some shorter and longer wave lengths and some visible light. Solid CO_2 (dry ice) with a temperature of about −50° C. provided low temperatures and a bunsen burner provided elevated temperatures. No attempt was made to record exact temperatures.

EFFECT OF TEMPERATURE CHANGE ON FLUORESCENCE

Change of temperature proved the most effective of the techniques employed. In general increasing the temperature of a mineral quenches temporarily any fluorescence which may be present, and reducing the temperature either temporarily increases the intensity of fluorescence if present, or, for a large number of the minerals used, actually initiates an appreciable brief luminescence. As might be expected, there were several exceptions to this general statement, ranging from a permanent quenching when the mineral altered to another form at high temperature, to the non-appearance of fluorescence in certain minerals at low temperature. Whether or not this failure to produce luminescence is a true exception remains somewhat doubtful, since there is a possibility that still lower temperatures might prove effective.

The general results are in accord with an observation by Kröger (1947) that in artificially prepared tungstates and molybdates, with a temperature range between +200° C. and −200° C., the various compounds show a fluorescent response which increases from zero to a maximum with falling temperature. Some of the compounds prepared by him show fluorescence in a range of about 50° above and below room temperature, but others may not develop the phenomena until the temperature is about −100° C.

Table 1 lists in brief form observations of the effect of the reduction of temperature on the fluorescence of a group of sixty-three minerals. Some were selected because they showed fluorescence at room temperature, but most of the others were chosen in order to include a diverse group of the more common minerals.

TABLE 1

	Mineral	Description	Source	Room Temp.	Low Temp.
Strong Fluorescence at Room Temperature					
1	Calcite (4.0)	Opaque, white	Probably Franklin Furnace	Very bright pink fluor.	No appreciable change.
2	Scapolite (5.2)	Yellow-green	Ontario (?)	Bright yellow-orange fluor.	No appreciable change.
3	Scheelite (5.8)	Creamy-white	Source unknown	Bright bluish white fluor.	No appreciable change.
4	Willemite (4.7)	Light green	Franklin Furnace	Intense green fluor.	No appreciable change.
Moderate Fluorescence at Room Temperature					
5	Calcite (4.0)	Opaque, orange brown	Source unknown	Salmon-pink fluor.	No appreciable change.
6	Fluorite (6.2)	Purple, good penetration twins set in finely xline base	Alston Moor, Cumberland, Eng.	Blue fluor. over most of specimen, some pink fluor. partic. in base.	Blue probably intensified, pink more prevalent, partic. in dihedral angles between twins
7	Scapolite (5.2)	Yellowish-green	Source unknown	Patchy yellowish fluor.	Stronger and more widespread fluor.
Pale Fluorescence at Room Temperature					
8	Albite (4.9)	Greyish white	Lake Athabaska, Sask. (supplied by D. Blake)	Pale yellowish white over parts of xls.	Strong white over most of xls.
9	Anglesite	Dull grey	Eureka Hill, Tintic. Dist., Utah	Greyish yellow fluor.	Intense yellow fluor.
10	Calcite (4.0)	Transparent white	Source unknown	Pale pink fluor.	Slightly brighter fluor.
11	Calcite (4.0)	Grey with some xls. of cinnebar	California	Golden fluor in some parts of calcite, pale pink in others.	Golden fluor, intensified and pink more prevalent.
12	Diopside (5.9)	Dull brown with xls. of titanite	Lewis Co., N.Y.	Pink splotches	Intense pink fluor.
13	Gypsum (4.7 incl. H_2O)	Transparent, pale yellow	Source unknown	Pale green fluor.	May be slightly brighter green, pale green phosphorescence persists for about 1 sec. after U.V. source removed.
14	Sylvite (5.6)	Greyish white	Source unknown	Scattered pink spots	Pink brighter and more prevalent.
15	Wolframite	Vitreous black	Redruth, Cornwall	Patches of yellow fluor.	More extensive golden yellow patches.
Doubtful Fluorescence at Room Temperature					
16	Albite 4.9)	White, opalescent	Source unknown	Very doubtful pink	Bright pink fluor. spots
17	Cassiterite (6.0)	Brown (in quartz with sulphides)	Zinnwald, Bohemia	Faint yellow fluor. assoc. with fract. surfaces of xls.	Strong golden yellow over most of cassiterite xls.
18	Chrysotile (5.4)	Yellow green (fluffed fibres grey white)	Templeton, Ottawa Co., Que.	Doubtful yellowish green fluor. (none on fluffed fibres)	Slightly intensified yellow green fluor.
19	Labradorite (?)	Glassy white, opalescence	Source Unknown	Streaks of pinkish fluor.	Pale pink over entire mineral
20	Orthoclase (Adularia) (5.0)	Semitransparent, glassy white	Arendal, Norway	Very pale pink fluor.	Very strong pink fluor.
21	Perthite	Reddish brown with visible inter growth	Source unknown	Very pale pink fluor.	Very bright pink fluor.
22	Pyromorphite	Orange and yellow green	Source unknown	Doubtful orange fluor. on orange parts of specimen	Orange fluor. brighter
23	Scheelite (5.8)	Grey white	Source unknown	Doubtful bluish pink fluor.	Pronounced pink fluor.
No Fluorescence at Room Temperature					
24	Apatite (5.3)	Brown	Ontario	No fluor.	Pale orange fluor. in streaks parallel to c axis
25	Aragonite (4.3)	Light grey, xline	Herrengrund, Hungary	No fluor.	Doubtful bluish white fluor.
26	Barite (5.1)	Yellowish-white	Brookfield, Colchester Co., N.S.	No fluor.	Pale creamy white fluor.
27	Barite xl. in dolomite (5.1 & 4.3)	Transparent brown in yellow brown	Source unknown	No fluor.	Coppery red fluor. in dolomite around base of barite

TABLE 1 (*Continued*)

	Mineral	Description	Source	Room Temp.	Low Temp.
28	Beryl (5.1)	Brownish green xl.	Source unknown	No fluor.	Patches of yellowish fluor.
29	Carnotite	Light yellow	Montrose Co., Calif.	No fluor.	Doubtful grey-green fluor.
30	Chromite (5.9)	Greenish black	Selukwe S. Rhodesia	No fluor.	Very numerous tiny orange specks
31	Cleavelandite	Creamy white	Source unknown	No fluor.	Pale pink fluor.
32	Corundum (7.2)	Brownish xl.	Transvaal	No fluor.	Patchy pale yellow fluor.
33	Cryolite with galena & chalcopyrite (6.2)	Grey white	W. Greenland	No fluor.	Very pale pink fluor. patches
34	Crysotile in serpentine (5.4)	Yellowish-green in dark green	Thetford Mines, Megantic Co., Que.	No fluor.	Doubtful creamy fluor. in crysotile
35	Dolomite (4.3)	Brownish yellow	Pascalis Township, Que.	No fluor.	Flecks of pinkish fluor.
36	Gypsum (4.7 incl. H_2O)	White, semitranslucent	Source unknown	No fluor.	Streaks of orange yellow fluor. parallel to cleavage
37	Halite (5.8)	White, translucent cube	Source unknown	No fluor.	Doubtful bluish white fluor.
38	Lazulite (5.7)	Dark green	Graves Mt., Georgia	No fluor	Extremely doubtful pink fluor.
39	Oligoclase	White	Source unknown	No fluor.	Pale orange splotches
40	Orthoclase (5.0)	Creamy brown, carlsbad twin	Lewis Co., N.Y.	No fluor.	Patchy pink fluor.
41	Rock salt (5.8)	Brown, semi-translucent	Cheshire, Eng.	No fluor.	Scattered pink fluor. spots
42	Siderite (4.4)	Brown, xline	Source unknown	No fluor.	Scattered pale orange fluor. specks
43	Torbernite	Grey green	Cornwall, Eng.	No fluor.	Scattered greenis fluor. specks
No fluorescence at Room Temperature or Low Temperature					
44	Aragonite (4.3)	Greyish brown, pseudo-hex. twin	Source unknown		
45	Azurite with malachite	Blue and green	Source unknown		
46	Brucite (6.7)	Pale brown, fibrous	Source unknown		
47	Chlorite (5.8)	Green, platy	Source unknown		
48	Chlorite schist with magnetite xls. (5.8 & 5.9)	Grey green	Quebec		
49	Diopside (5.9)	Greenish brown	Source unknown		
50	Fluorite (6.2)	Purple, translucent	Source unknown		
51	Garnet (6.1)	Black	Source unknown		
52	Graphite (1.7)	Black	Source unknown		
	Labradorite	Dark grey, labradorescence	Source unknown		
54	Magnetite (5.9)	Black xls.	Source unknown		
55	Manganite	Black	Source unknown		
56	Penninite	Dark green	Mill Green, Hertford Co., Maryland		
57	Quartz with free gold, pyrite tellurides (5.2)	White, opaque	Bevcourt Mine, Louvicourt Twp., Que		
58	Realgar	Reddish orange	Norris Basin, Yellowstone		
59	Rock salt (5.8)	Greyish white	Cheshire, England		
60	Sphalerite	Greyish black	Source unknown		
61	Stibnite	Metallic black	Japan		
62	Sulphur	Opaque yellow	Lakes Charles, Louisiana		
63	Tourmaline (6.0)	Black xls.	Pierpont, St. Lawrence Co., N.Y.		

Examination of this table shows that minerals which are strongly fluorescent at room temperature show no visible change when chilled. The moderately fluorescent specimens may show an intensified fluorescence on cooling, but the slightly fluorescent and doubtfully fluorescent minerals almost invariably exhibit an intensified fluorescence when their temperature is reduced. Among the specimens which show no luminescence at room temperature, about half are fluorescent to some degree when chilled. It will be noted that in several cases the fluorescence at room temperature was observed to be of a patchy or spotty nature, whereas at lower temperatures the patches were found either to increase in size, or the entire surface of the mineral became fluorescent. A similar feature was found upon chilling some of the normally non-fluorescent minerals, when a temporary fluorescence of a patchy nature developed.

Results of particular interest were obtained from specimens 6, 13, 17, and 27. Upon cooling specimen 6 (fluorite), a relationship became apparent between the fluorescence and the twinning, the pink fluorescence becoming intensified at the intersections of the crystals. Specimen 13 (gypsum), developed a short phosphorescence while below room temperature. The initial fluorescence of specimen 17 (cassiterite), showed some relationship to the fractured surfaces of the crystals, but at lowered temperature the emitted light came from the entire crystal surface. In specimen 27 (barite in dolomite), the only fluorescence noted occurred at low temperature, but it was concentrated in the dolomite around the base of the barite crystal.

Because of the possibility that initiation or augmentation of fluorescence in minerals might prove of value in estimating the percentage of minerals in rocks (Quinn, 1935), a few rock specimens of known mineral composition were cooled and examined under ultraviolet light. Most of the results were inconclusive because of the difficulty of identifying, in the hand specimen, the minerals which became luminescent. However, in a specimen of granite from Pascalis township, Quebec, consisting of quartz, oligoclase, microcline, microperthite, biotite and sphene, none of which were fluorescent at room temperature, it was observed that certain of the feldspars developed a bright pink fluorescence at reduced temperature. An uncovered thin section of this rock was prepared and the fluorescent mineral found to be microcline.

Use of a thin section in this fashion presents several problems which may make the results uncertain. Due to the opaqueness of glass to ultraviolet light a cover glass cannot be used, and, as the section is reduced in thickness to a point where the minerals can be identified, it becomes increasingly difficult to distinguish the fluorescent portions of the rock, which, in part, is due to the glass developing an orange fluorescence. If the section is removed from the dry ice to permit close examination it warms so quickly that the fluorescence rapidly disappears. Further, the cooling caused the development of peculiar bubbles in the balsam which resemble the rune-like texture of graphic granite.

A group of hand specimens of granitic rocks from Baffin Island supplied by G. Riley were of particular interest, since they have a very pale pink fluorescence in certain of the feldspars at room temperature, but at low temperature the fluorescence has a definite pink colour, similar to that of calcite from Franklin Furnace, N. J. One of these specimens is known to consist of 27% quartz, 6.5% adularia and anorthoclase, 32% microcline and microperthite, 27% albite, and a little mica, apatite and sphene. About 25% of the surface of the rock fluoresced and Mr. Riley tentatively identified the luminescent mineral as microcline.

TABLE 2

No.	*PART A* Increased Temp.	*PART B* Powdered Mineral at		*PART C* Powdered Mineral Wet with Water
		Room Temp.	Low Temp.	
1	Fluor. becomes paler and disappears, then returns quickly on cooling	Bright pink as in original mineral	Not tested	Not tested
2	Fluor. slowly becomes paler and disappears. Returns on cooling	Yellow-orange fluor. May be slightly paler than original mineral	Intense orange fluor.	Intense orange fluor.
4	Fluor. becomes paler but high temp. req'd to cause quenching. Returns rapidly upon cooling	Intense green fluor. as in original mineral	Similar to fluor. at room temp.	No appreciable change
5	Fluor. becomes paler and disappears. On cooling fluor. returns	Salmon pink as in original mineral	Not tested	Not tested
10	Fluor. quickly becomes paler and disappears. On cooling it returns slowly	Fluor. lost when mineral crushed	Doubtful white fluor.	Doubtful blue fluor.
13	Fluor. disappears very quickly and does not return on cooling	Larger grains retain green fluor. but most of powder is non-fluor.	Similar to effect at room temp.	Larger grains become brighter green, fine material very pale blue.
26	Non-fluor.	Non-fluor.	Doubtful white fluor.	Pale blue-white fluor.
35	Non-fluor.	Not tested	Not tested	Not tested
57	Non-fluor.	Non-fluor.	Non-fluor.	Distinct blue fluor.

The effect of the fluorescence due to increased temperature was tried on relatively few minerals, most of which were fluorescent at room temperature. The results are listed in Table 2, Part A, in which the figure in the left hand column refers to the minerals in Table 1. The general effect of increasing the temperature is a temporary loss of fluorescence in the minerals which are luminescent at room temperature, while the non-fluorescent minerals exhibit no change. No thermo-luminescent phenomena were observed in the minerals tested. Specimen 6 (gypsum) which has been previously noted to develop phosphorescence at low temperature permanently lost its fluorescence when heated, presumably due to the loss of water.

CHANGES IN FLUORESCENCE DUE TO CRUSHING

A small group of minerals, some of which are fluorescent at room temperature, were powdered to observe the effect on their luminescing ability when in this condition, both at room temperature and low temperature. The crushing was not performed in a darkened room so that triboluminescence, if any, was not noted. At room temperature, some of the luminescent minerals showed no change when crushed but two lost their ability to emit light. A characteristic of the minerals which became nonfluorescent, which may be an important factor, is that both were transparent crystals in their original form. There was no change in the nonluminescent minerals. When the powdered minerals were cooled there was either no change from the conditions observed at higher temperature, or a very doubtful whitish fluorescence. The results are listed in Table II, Part B.

CHANGES IN FLUORESCENCE DUE TO WETTING POWDERS

It has been stated that some non-luminescent white powders, including crushed quartz, develop a bluish fluorescence when wet with water (Ewles, 1930). Some of the powdered minerals were treated in this fashion to duplicate this effect, if possible, and also to observe the effect on luminescent powders. The results were not uniformly good, but the nonfluorescent powders developed bluish fluorescence of varying intensities and one of the fluorescent powders changed colour to an appreciable degree. The results are listed in Table 2, Part C.

THE "PACKING INDEX" AND FLUORESCENCE

A further observation, the value of which can only be suggested at the present time, but which may be important in the examination of fluorescent minerals, is that the "packing index" (Fairbairn, 1943) appears to bear a relationship to the occurrence of fluorescence in minerals. The packing index applies only to minerals with an ionic type of bonding, but does not apply to such minerals as elements and sulphide minerals which may also show luminescent phenomena.

In Table 1, the bracketed figure beneath most of the mineral names is the packing index of that mineral. It will be noted that the fluorescent or occasionally fluorescent minerals usually have an index which lies somewhere close to 5.0, with some higher and some lower values. Most of the non-fluorescent minerals have either very high or very low values.

A frequency distribution curve of the indices given by Fairbairn has its mode at about 5.8, whereas a similar curve of the indices of minerals which have been reported to show fluorescence occasionally (DeMent, 1949), has its mode at about 4.7. This suggests that a mineral with an index close to 4.7 would be more likely to exhibit fluorescence than one with a higher or lower index. In some cases, lower indices would probably be increased by the addition of an activator, and in others equivalent changes might reduce higher values. Thus there is a possibility that the packing index may also provide an index for the occurrence of fluorescence in ionic minerals.

CAUSES OF LUMINESCENCE

A general statement of the cause of luminescence in solid material is that an "excitant" raises the energy level of the material and the added energy is given off as visible light. These excitants include such parts of the electromagnetic spectrum as infra-red, visible, ultra-violet and x-ray radiations, as well as cathode and radium emanations, chemical reactions, heat, friction and many other factors. Commonly, long and short wave ultra-violet light is used in studying mineral fluorescence. An "activator," which is a small amount of chemical element or impurity which is foreign to the pure mineral is considered a necessary factor for a mineral to exhibit luminescence. In some cases it has been shown that if the per cent of activator is greater or less than a certain optimum range, luminescence cannot be produced (without change in temperature).

In more detail, the types of luminescent processes and empirical rules of the mode of occurrence of luminescence as found from the study of artificial phosphors are summarized below. As given they have been greatly condensed from their original form (Pringsheim, 1943).

(a) *Types of luminescent processes*

(i) Fluorescence—the spontaneous transfer of a molecule from an excited state and a high energy level to a lower energy level. The mean life is very short and practically independent of temperature.

(ii) Phosphorescence—a fraction of the excited molecules do not begin immediately to emit light by returning from the excited state, but pass instead to a metastable state from which they return to the excited state and emit light only when a quantity of energy ϵ is added by the heat movement of the surrounding medium. Thermoluminescence is a variety of phosphorescence where the value of ϵ is very high and the luminescence can only be released at elevated temperatures.

(iii) Recombination afterglow—the electrons are completely removed from the original molecules by the absorption of light. Light emission occurs when the electrons recombine with one of the excited molecules.

The luminescence of (i) and (ii) decays according to an exponential law, whereas (iii) decays according to a hyperbolic law. The exponential type is independent of the initial intensity, but the steepness of the curve increases with increasing temperature. The hyperbolic type is little affected by the temperature, but a high initial intensity produces a steep curve.

(b) *Rules*

(i) Practically no pure elements and very few simple compounds are luminescent.

(ii) Most substances with a strong luminescence are white or only slightly coloured.

(iii) Most substances become luminescent or their luminescence is strongly increased when they are cooled down to liquid air temperature. Heating to a certain upper limit which usually lies between 100° C. and 400° C. destroys the fluorescence in almost every case.

(iv) Liquid solutions are seldom phosphorescent.

(v) In the solid state many compounds show an afterglow. However, phosphorescence persisting over many hours, and "frozen in" at low temperatures is almost exclusively a property of "crystal phosphors" activated with small traces of an impurity.

(vi) The light-emissive capacity of many materials which are luminescent under normal conditions is diminished or totally suppressed by the presence of some other substance (poisons).

(vii) The question of the importance of grinding and particle size is somewhat uncertain, but it does not seem to be advisable to grind sulphide phosphors to particle sizes less than 0.1 mm, and silicate phosphors are supposed to increase in intensity with particle diameters down to 0.001 mm.

(viii) The number of impurity molecules able to activate phosphors is very large. One condition seems to be that the ionic (or atomic?) radius of the activator must be smaller than the radius of the cation of the basic lattice.

(ix) Activating impurities do not occupy analogous positions in the lattice of all phosphors. In some cases the activator ions are interstitial in the lattice and in others mix crystals are formed. In the second case there is a modification in the size of the crystal lattice and the luminescence may be dependent either on the formation of the mix crystal or the introduction of activators into the modified lattice.

CONCLUSIONS

Luminescence resulting from excitation with short ultra-violet light can be initiated or intensified in many minerals when they are cooled with dry ice. This is in agreement with observations made on artificial phosphors and is directly attributable to the reduction of the activity of the molecules as a result of the reduced temperature. It seems probable that a content of "activator" which is less than the optimum required for fluorescence at room temperature serves to produce luminescence at lower temperatures. Patchy fluorescence is probably caused by widely scattered activator centres. Raising the temperature has an opposite effect and fluorescence is temporarily quenched unless the mineral is altered by the high temperature, in which case the luminescence may be permanently destroyed. Most of the results obtained from a varied group of specimens can be explained in this manner but a few appear to be more complex and additional factors probably have some bearing on the distribution of the fluorescence.

Temporary low temperature luminescence may occasionally prove valuable for the recognition of some minerals in rocks, particularly where megascopic identification is difficult or impossible.

Some minerals when crushed have their luminescence greatly diminished or destroyed. The reason for this is uncertain, but it may be related to the optical properties of the mineral. Wetting of crushed luminescent minerals may change the fluorescent colour, or, in the case of non-fluorescent powders, may produce a distinct blue luminescence.

It is suggested that the packing of ions within the crystal lattice may bear some relationship to the occurrence of luminescence in minerals with an ionic type of bonding.

A fairly complete summary of the types of luminescence and empirical rules for the occurrence of luminescence has been included in the discussion. The summary refers particularly to artificial phosphors and chemical solutions, but in large measure can be applied to minerals as well. No similar modern summary has been found in the easily available mineralogical or petrological literature, and it is believed that it may be of value, particularly as an introduction to the examination of fluorescence in minerals.

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