

THE AMERICAN MINERALOGIST

JOURNAL OF THE MINERALOGICAL SOCIETY OF AMERICA

VOL. 7

FEBRUARY, 1922

No. 2

NOTES ON THE PREPARATION OF SOME FLUORESCENT AND PHOSPHORESCENT COMPOUNDS¹

W. S. ANDREWS, *General Electric Company, Schenectady, New York*

ARTIFICIAL WILLEMITE OR ZINC ORTHOSILICATE, ($Zn_2 SiO_4$)

Pure zinc orthosilicate has no luminescent properties but the addition of a minute amount of manganese causes it to show a bright green fluorescence when exposed to ultra violet light, cathode rays, or X-rays. Fluorescent natural willemite is found in the zinc mines of Franklin Furnace, N. J. and a few other places, but it is not a common mineral and it is also generally mixed with other earthy matters which tend to dull its luminescent properties. Both natural and artificial willemite often exhibit a more or less persistent phosphorescence after excitation.

A fine quality of artificial willemite may be prepared by pulverizing and mixing thoroughly:

C.P. zinc oxide	100 grams
C.P. silicic acid	50 grams
C.P. black oxide of manganese	0.5 "

This mixture which has a light gray color must be placed in a porcelain crucible and raised gradually to a temperature of about 1200°C. At about 800° to 1000°C. the powder changes from gray to light yellow and finally between 1100° and 1200°C. it becomes snow white, probably owing to the complete solid solution of the manganese. The changes in color from gray to yellow and then to pure white are gradual, depending upon the temperature and time, so that to insure an entire change thruout the mass, the crucible should be kept at a temperature between 1100° and 1200°C. for at least half an hour. If on cooling, the inside of the mass shows a yellow color, it should be ground up in a mortar and fired again until it shows a pure white thruout the mass on cooling.

¹ This series of directions for making artificial compounds showing luminescent properties more or less like those of natural minerals was obtained for this journal thru the interest of Mr. W. L. Lemcke, of Franklin, Pa.

To obtain the best results, all ingredients should be in a fine powder before mixing, so it is well to pass them thru a 200 mesh sieve. The crucible should be covered (altho luting is unnecessary) as the zinc oxide is otherwise liable to distill off before it combines with the silicic acid. When these precautions are observed and the firing is done properly, the resulting zinc silicate will have a bright green fluorescence under ultra violet light from the iron spark. It will also show a brilliant green color under the cathode rays, and will be more or less responsive to X-rays.

Different shades of green fluorescence from dark olive to light greenish yellow may be obtained by varying the amount of manganese content, and by applying different degrees of temperature. The above directions, however, produce a compound that shows a bright green fluorescence which resembles very closely that of the best natural willemite.

If it is desired for use as a paint it should be reground and the fine powder mixed to the proper consistency with a solution of one part, by weight, of gum arabic with twelve parts of water. It is best to make this solution fresh when wanted as it does not keep well.

FLUORESCENT CADMIUM PHOSPHATE

There are but few natural minerals that show a red fluorescence under ultra violet light. A variety of calcite that is found at Franklin Furnace, N. J. exhibits a light red or pink color under the iron spark but good specimens are rare.

It was therefore a welcome discovery to find that when a basic or neutral Cadmium phosphate, $Cd_3(PO_4)_2$, is fused with a small amount of manganese the resulting compound shows a brick red fluorescence under the iron spark, followed by a short period of phosphorescence of a darker red.

This compound may be prepared as follows:

Mix well 120 grams of finely pulverized neutral Cadmium phosphate, with 2 grams of black oxide of manganese, and heat to 900° - 1000° C. in a porcelain crucible. The Cadmium phosphate will melt to a clear liquid which dissolves the manganese without showing any discoloration. Maintain in fusion for about 20 minutes and then pour out upon a clean slab of soap-stone or other suitable material. When cold, the mass will resemble white unglazed porcelain.

Under cathode rays the compound shows a bright golden orange fluorescence with a yellow phosphorescence for a short period. For use as a paint it may be ground very fine and mixed with a weak solution of gum arabic in water as already described.

FLUORESCENT AND PHOSPHORESCENT PHOSPHATE OF
CADMIUM AND ZINC

Mix well the finely pulverized material consisting of:

C.P. basic cadmium phosphate 60 grams

C.P. basic zinc phosphate 60 grams

C.P. black oxide of manganese 2 grams

Fuse the above mixture in a porcelain crucible at a full red heat and maintain in fusion for 15 or 20 minutes. Then pour out to cool on a clean slab of soapstone or porcelain. The fluorescence of this compound under the iron spark is a light pink, and its phosphorescence is a deep red resembling hot coals, and fairly persistent.

CADMIUM PHOSPHATE WITH A PURE WHITE FLUORESCENCE AND
PHOSPHORESCENCE

It is well known that a white or nearly white luminescence can be obtained from a mixture of phosphorescent materials that produce respectively complementary colors, but in the present case it is a single pure salt of Cadmium that possesses the unique property of showing a clear white fluorescence and phosphorescence when exposed to ultra violet light from the iron spark.

This interesting compound may be made by fusing strictly C.P. basic Cadmium phosphate in a porcelain crucible, and pouring out the molten salt on a clean slab of soapstone or porcelain. When cold it is white and brittle like porcelain. It can be used as a paint by grinding fine and mixing with gum arabic or other suitable adhesive.

FLUORESCENT AND PHOSPHORESCENT ANHYDROUS
CADMIUM SULFATE

To prepare this compound dissolve 100 parts by weight of C.P. cadmium sulfate in distilled water and add to it 0.2 parts of manganese sulfate or chloride, also dissolved in water. Stir the mixture well and evaporate to dryness. Then pulverize and calcine at a low red heat for about 30 minutes. When cold, pulverize again and sift. Keep in a well stoppered glass bottle.

Care must be taken in calcining the above not to raise the temperature too high, for at a bright red heat it decomposes and becomes worthless. When properly prepared, it shows a pink fluorescence under the iron spark and a bright yellow phosphorescence which is fairly persistent.

Under cathode rays it shows a bright yellow fluorescence and phosphorescence, but X-rays produce no luminescence. If kept in the open it gradually absorbs moisture, and loses its property of fluorescence which, however, can be restored by calcination.

FLUORESCENT AND PHOSPHORESCENT ANHYDROUS ZINC SULFATE

This compound can be prepared by the same process as that described for making fluorescent cadmium sulfate by substituting the zinc salt for the cadmium. It shows a pink fluorescence under the iron spark and a fairly persistent dark red phosphorescence, giving it the appearance of red hot coals.

Under cathode rays in a vacuum tube the fluorescence and phosphorescence are bright red. Like the anhydrous cadmium sulfate it is not responsive to X-rays.

If exposed to air it absorbs moisture quickly and loses its property of fluorescence, so it must be kept in a well stoppered bottle. However, fluorescence can be restored by recalcination.

PREPARATION OF FLUORESCENT URANYL FLUORIDE

Dissolve 50 grams of uranyl nitrate in 200 C.C. distilled water. In another vessel (which must be protected on the inside with a coating of paraffin or beeswax), dissolve 75 grams of ammonium fluoride in 110 C.C. of distilled water. On mixing these two solutions, a precipitate of uranyl fluoride will be formed, which will settle within a few minutes, and the clear liquid containing ammonium nitrate can then be poured off, draining the precipitate as dry as possible. The precipitate is fairly soluble in cold water and very soluble in hot water, so that if it is necessary to remove the small amount of ammonium nitrate left in the damp precipitate after draining, the washing should be done very carefully with cold water. The precipitate can be completely dried by pouring it out on filter paper and allowing it to stand for a few hours in a warm place. The exact formula for this compound is rather uncertain as it may be a double salt of uranium and ammonium. It is brightly fluorescent under the ultra violet rays from an iron spark

but the period of its phosphorescence is too short to be seen by the unaided eye. It is also weakly responsive to excitation by the X-rays.

TRIBO-LUMINESCENT ZINC SULFIDE

This interesting compound may be prepared as follows:

Zinc carbonate or oxide	100 grams
Flowers of sulfur	30 grams
Black oxide of manganese	0.5 grams

The above ingredients must be in a fine powder and intimately mixed. Place the mixture in a porcelain crucible and calcine at a full red heat for about 30 minutes, or until the mixture assumes a light yellow color thruout when cold.

If a pinch of the above is put on a piece of card and rubbed with the blade of a knife, a stream of yellow scintillations will be produced. It shows no fluorescence or phosphorescence under the ultra violet rays from the iron spark, but cathode rays produce a brilliant yellow fluorescence without any perceptible afterglow. It also shows a yellow fluorescence under X-rays, without noticeable phosphorescence.

EAKLEITE FROM ISLE ROYALE, MICHIGAN¹

WILLIAM F. FOSHAG AND ESPER S. LARSEN, *Washington, D. C.*

A specimen labeled "Wollastonite, Isle Royale, Mich." in the United States National Museum was recently noticed to have the general appearance of the eakleite from the original locality at St. Inez, California, and further study has confirmed the suspicion that the mineral actually is eakleite.

The specimen consists of an aggregation of radiating fibers, with a somewhat silky luster and a pale pink color. The material is exceedingly tough. Microscopic examination showed that it is made up mostly of eakleite, with properties similar to those of the mineral from California. The Isle Royale mineral is finer fibered and is less pure, as it contains several per cent of a weakly birefracting material that is probably a sort of serpentine or chlorite. This foreign material forms nests of small grains in the fibrous masses. The optical properties of this eakleite, compared with those of the original material from California, are:

¹ Published with the permission of the Secretary of the Smithsonian Institution and the Director of the United States Geological Survey.