

A Note on the Identification of "Insoluble" Sulphates

K. F. G. HOSKING, M.Sc., Ph.D., A.M.I.M.M.
and J. P. R. POLKINGHORNE, A.M.I.M.M.

Abstract

THE writers, having demonstrated that the mercuric nitrate test for insoluble sulphates — as described by Feigl — is of little use, describe a modified procedure which enables differentiation to be made between the sulphates of Ca, Sr, Ba and Pb.

Feigl (1954, p.369) makes the following remarks :—" A general test for slightly soluble sulphates (i.e., those of Ca, Sr, Ba and Pb) is to treat a little of the material on a watch glass with a drop of an acid solution of mercuric nitrate [10 g. $\text{Hg}(\text{NO}_3)_2$ plus 1 ml. concentrated HNO_3 , in 100 ml. water]. Yellow basic mercuric sulphate is formed. CaSO_4 and PbSO_4 respond at room temperature ; SrSO_4 and BaSO_4 must be warmed to develop the yellow. If sulphides and halogen compounds are absent, the test is decisive. The test material must be colourless ; otherwise the yellow cannot be seen."

When the writers repeated this test they found that only CaSO_4 developed a yellow tinge at room temperature (c. 60°F.), but, on warming, PbSO_4 and SrSO_4 both became slightly yellow, whilst BaSO_4 remained white.

However, a number of experiments led to the development of the following modified test which has considerably more diagnostic value than that which was claimed (erroneously) for the original :—

1. Add a little mercuric nitrate solution (prepared as for the original test) to the "insoluble" sulphate under examination. Then add double the volume of water and swirl the mixture.

CaSO_4 becomes strongly yellow immediately, whilst SrSO_4 develops a distinct yellow tinge after one or two minutes : PbSO_4 may show a trace of yellow and BaSO_4 remains white.

2. Heat the mixture gently for a few seconds over a small flame.

The CaSO_4 yellow may intensify somewhat and that of SrSO_4 , certainly does so: PbSO_4 develops a yellow hue whose intensity matches that of SrSO_4 , whilst BaSO_4 remains white, or at the most, assumes a very slight yellowish tinge.

Gypsum (and anhydrite) barite, celestite and anglesite behave in precisely the same way as their laboratory equivalents, although gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) is somewhat more reactive than the less soluble anhydrite (CaSO_4).

It is, therefore, an easy matter to differentiate between the "insoluble" sulphates by employing this test alone.

A number of excellent confirmatory tests for these sulphates is described by Feigl (1954, pp.369-370) and also two which enable differentiation to be made between gypsum and anhydrite (pp.414 and 447). The writers, often employ, in addition, the potassium iodide test -- which is not mentioned by Feigl -- to confirm Pb in PbSO_4 . This simply involves adding a small crystal of KI and a few drops of 5N. acetic acid to the substance under test. The presence of lead is indicated by the immediate appearance of an intensely yellow precipitate of PbI_2 .

REFERENCES

FEIGL, F. Spot tests, vol. 1 (London : Elsevier Publishing Co., Ltd., 1954).

Analysis

Analysis of the creature known as Woman — as seen through the eyes of a chemist.

Symbol :	WOE.
Atomic Weight :	120 lbs.
Occurrence :	Found wherever man exists.

Physical Properties :

1. Boils at nothing, and freezes at any moment.
2. Melts when properly treated ; very bitter when not well used.

Chemical Properties :

1. Possesses a great affinity for Gold, Silver, Platinum and Precious Stones.
2. Violent reaction if left alone.
3. Liberates secrets when dipped in conversation.
4. Turns green when placed beside a better specimen.
5. Highly explosive.
6. Highly ornamental.

Uses :

As a tonic for low spirits.
Great equaliser of the distribution of wealth.
Probably the most effective Income Reducing Agent ever known.