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The behaviour of secondary lead species when subjected to the tinning test for cassiterite

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Generally, but no invariably, it is possible to detect grains of cassiterite (SnO_2) in a composite sample by subjecting the latter to the 'tinning test'. This test, which has been discussed at some length by Hosking (1974), may be carried out in a variety of ways, but a convenient one is to place the sample, or a representative portion of it, in a zinc tray and then cover the grains with 1:1 HCl. After a short time, which depends on a number of factors, but which is normally less than 2 minutes, and may be much less, any cassiterite grains present are usually coated with a grey matt layer of elemental tin. The reaction involved is generally expressed as follows:

$$SnO_2 + 4H \rightarrow Sn + 2H_2O$$

Recently, when a concentrate of heavy-mineral grains, which had been collected by one of us (K.W.P.O.) from the Klian Intan area of North Perak, was subject to this test, not only were grains tinned that visual examination had suggested were cassiterite, but, in addition, certain bright yellow grains were also coated with a matt grey metallic layer. Initially it was thought that this latter coating might also be tin, but more careful scrutiny indicated that it was not tin-grey in colour, and as the yellow grains were quite unlike any cassiterite any of us had ever seen, it was decided to investigate the matter further.

A powder pattern of the grains in question indicated that they were pyromorphite, $Pb_4(PbCl)(PO_4)_3$, and this was confirmed by appropriate chemical tests. It was also confirmed that the coating was lead by subjecting it to Feigl's sodium rhodizonate test (Feigl, 1954, pp. 385–6).

It was also demonstrated that anglesite, cerussite, mimetite and vanadinite (the only other secondary lead species that were available for study) also reacted to the test in question in much the same way as did pyromorphite in that they all became coated with lead. In addition, after the termination of the test and removal of the grains from the zinc tray, the floor of the latter may be locally dark due to the deposition of elemental lead on it.

In the laboratory, at a room temperature of 24°C, the times in seconds, taken for cassiterite and grains of the lead species, noted above, to become coated, when subject to the test under review, were as follows:

Cassiterite	– 15 seconds
Anglesite	– 25 seconds
Mimetite	– 45 seconds
Vanadinite	– 60 seconds
Pyromorphite	– 50 seconds
Cerussite	– variable. Some grains only partially coated after 2 *minutes*

A further set of tests were carried out primarily with a view to discovering how the available secondary lead minerals behaved when certain other metals were substituted for zinc. Brief details of these tests and of their results are as follows:–

(i) HCl/Al test

When grains of the lead species in question were placed in an aluminium dish and covered with 1:1 HCl they were coated with lead rather more rapidly than when a zinc dish was used. However, partial coating of grains was not uncommon and the degree of coating of grains of a given species during a test varied considerably.

When cassiterite grains are subject to the test they are rapidly coated with tin.

(ii) The HCl/Fe Test

When grains of the lead minerals noted above were placed in an iron dish and covered with 1:1 HCl no metallic coating developed either in the cold or on warming.

Cassiterite also reacted negatively to this test.

(iii) The HCl/Mg test

When grains of the secondary lead species under review together with magnesium ribbon, were placed on a watch-glass, and 1:1 HCl was added, no coating of the grains was observed.

Cassiterite also reacted negatively to this test.

THE PROBLEM OF DIFFERENTIATING BETWEEN COATED CASSITERITE AND COATED SECONDARY LEAD SPECIES

If a given person knows how secondary lead species behave towards the zinc/dilute HCl test, and if he has an average ability to differentiate between greys of different shades, then he will not find much difficulty in assessing the percentages of lead-coated and tin-coated grains in a composite sample that has been subjected to the test in question. The writers are of the opinion that differentiation between lead-coated and tin-coated grains is somewhat easier after the sample has been dried. Of course, the finer the grains the more difficult does this differentiation become. On the other hand, if the coated grains are coarse, then rubbing them with a piece of cloth burnishes the metal coats and this makes it easier to determine whether a given one is lead or tin.

The presence of secondary lead minerals in a sample of grains can be readily confirmed by applying the KI/dil. HNO_3 test (Hosking, 1957). To carry out this test immerse the sample in freshly-prepared, strong KI solution, *then* add 1:7 HNO_3 until the volume is increased by c. ½. Most secondary lead species stain a brilliant yellow almost immediately. Little or no agitation is necessary, and the test can be carried out in the field on a saucer, pan, dulang or vanning shovel.

After a sample has been thus tested it may subjected to the Zn/HCl one after it has been washed to remove the excess of KI and HNO_3. Any yellow-coated lead-containing grains will then be rapidly coated with elemental lead.

In an endeavour to facilitate differentiation between lead-coated and tin-coated grains attempts were made to so stain one or both types of metal coating that the two displayed marked differences in colour. The KI/HNO_3 test, noted above, and the sodium rhodizonate one (see Feigl, 1954) formed the bases of several experiments aimed at achieving this end. Alas, none of these yielded results which were satisfactory.

FURTHER NOTES CONCERNING THE TINNING TEST

It is of interest to note that when grains of the secondary copper minerals that were available to us (namely, malachite, azurite and cuprite) were subject to the ZN/HCl test they all become coated with a dull reddish-brown coating of elemental copper.

Although the silver haloid species (cerargyrite, embolite, etc.) were not available for study, the writers think that were they subjected to the test under consideration they might become coated with elemental silver. This is not entirely a matter of academic interest as such species may be associated, albeit rarely, with cassiterite. Thus, for example, cerargyrite (AgCl) locally occurred with cassiterite at the Perran Silver Mine, Cornwall (Jones, 1925, pp. 128–129).

It is also not irrelevant to remind the reader that grains of tungsten species, excepting those of members of the ferberite-wolframite-hubnerite series, become superficially reduced to a pale-blue compound when subjected to the Zn/HCl test. (Hosking, K.F.G. Unpublished studies.)

CONCLUSIONS

As far as the writers are aware the behaviour of secondary lead and copper species to the tinning test has not hitherto been recorded in the literature. From the points of view of the tin prospector, miner and mineral-dresser the reaction of the secondary lead species to the test in question is important because on superficial examination of a heavy-mineral concentrate it would not be difficult to overlook the presence of certain secondary lead mienrals, for example, cerussite. Were this to happen, then, on application of the tinning test, such lead grains would be counted as cassiterite. It is also to be remembered that lead and tin species are by no means uncommon natural associates. In the southeast Asian Tin Belt, for example, such an association is well-known at Ulu Sokor (Kelantan, Malaya) and at Pinjok and neighbouring mines in South Thailand.

REFERENCES

FEIGL, F., 1954: Spot tests. (Translated by R.E. Oosper) *1*, 385–386. Elsevier Publishing Co., N. York. (518 pages.)

HOSKING, K.F.G., 1957: The identification – essentially by staining techniques – of white and near-white grains in composite samples. Camborne School of Mines Mag., *57*, 5–16.

HOSKING, K.F.G., 1974: Practical aspects of the identification of cassiterite (SnO_2) by the "Tinning Test". Geol. Soc. Malaysia, Bulletin 7, 17–26.

JONES, W.R., 1925: Tinfields of the World. Mining Publications, Ltd., London.